[Appendix 4]

Monographs, Part 2
(Related to Article 2 Subparagraph 4)

1) Crude Drug and Crude Drug Product

Acanthopanax Root Bark
오가피(五加皮)

Acanthopanacis Cortex

Acanthopanax Root Bark is the bark of the root and stem of *Acanthopanax sessilifolium* (Rupr. et Maxim.) Seeman or other congeneric plants (Araliaceae).

Description Acanthopanax Root Bark is the bark of the root and stem, usually cylindrical or semi-cylindrical, 5 cm to 10 cm in length, 5 mm to 8 mm in diameter and about 1 mm in thickness. The external surface is smooth, exhibiting a yellowish brown to dark gray color. Thorns or their marks are sporadically scattered on the surface of the bark of the stem, The inner surface of the bark exhibits a yellowish white color with a fibrous texture, making it hard to break.
It has a characteristic odor and a slightly bitter taste.

Identification Weigh 1 g of pulverized Acanthopanax Root Bark, add 10 mL of methanol, shake thoroughly to mix, filter, and evaporate the filtrate to dryness. Dissolve the residue in 1 mL of methanol and use this solution as the test solution. Separately, dissolve 1 mg of acanthoside D RS in 1 mL of methanol and use this solution as the standard solution. Spot 10 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of chloroform, methanol and water (70 : 30 : 4) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly sulfuric acid TS for spray on the plate and heat at 105 °C; the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

Purity (1) ***Foreign matter***—(i) Xylem tissue and twig: The amount of the xylem tissue and twigs contained in Acanthopanax Root Bark is less than 2.0%.

(ii) Other foreign matters: The amount of foreign matters other than the xylem tissue and twigs contained in Acanthopanax Root Bark is less than 1.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Extract content ***Water-soluble extract***—NLT 8.0%.

Acid-insoluble ash NMT 1.0%.

Packaging and storage Preserve in well-closed containers.

Achyranthes Root
우슬(牛膝)

Achyranthis Radix

Achyranthes Root is the root of *Achyranthes japonica* Nakai or *Achyranthes bidentata* Blume (Amaranthaceae).

Description (1) ***Achyranthes japonica***— Achyranthes Root of *Achyranthes japonica* has a cylindrical root with numerous long, thin lateral roots and is 5 cm to 20 cm long and 3 mm to 5 mm in diameter. There is a short stem at the top of the root. The outer surface is grayish yellow to pale yellow. The texture is firm but brittle and the fracture surface is horn-like, yellowish white to yellowish brown.
It has a light, characteristic odor and a slightly sweet taste and is viscous.

(2) ***Achyranthes bidentata***—Achyranthes Root of *Achyranthes bidentata* is a long, thin, cylindrical, slightly curved root that is somewhat thick at the top and relatively thin at the bottom, with a length of 15 cm to 50 cm and a diameter of 0.4 cm to 1 cm. The outer surface is grayish yellow to yellowish brown with several longitudinal folds and rarely lateral root scars. The texture is firm but fragile, easy to cut and flexible when soaked in water. The fractured surface is uniform, yellowish brown, coarsely horny and slippery.
Under the microscope, the cross-section shows a cork layer consisting of several rows of cork cells. The cortex is narrow. The central cylinder occupies most of the root. Multiple vascular bundles forming 2 to 4 concentric circles form abnormal vascular bundles. The vascular bundles in the outermost circle are relatively small and those in the third circle on the inner side are relatively large. The xylem consists of vessels and xylem fibers. The vessels are lignified or slightly lignified and sometimes contain debris. The xylem fibers are slightly lignified. A small number of parenchyma cells contain calcium oxalate crystal sands. There are normal vascular bundles in the center, and the primary xylem is diarch.
Achyranthes Root of Achyranthes bidentata has almost no odor and tastes slightly sweet and is mucilaginous.

Identification (1) Weigh 0.5 g of pulverized Achyranthes Root, add 10 mL of water, and shake vigorously to mix; persistent, fine bubbles are formed.

(2) Weigh 2 g of pulverized Achyranthes Root, add 10 mL of methanol, sonicate for 1 hour, and filter. Use the filtrate as the test solution. Separately, weigh 1 mg of 20-hydroxyecdysone RS, dissolve it in 1 mL of methanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a lower layer of a mixture of chloroform, methanol, and water (8 : 2 : 0.5) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 254 nm), or spray sulfuric acid TS for spraying evenly to the plate and heat it at 105 ℃ for 10 minutes; one spot among the several spots obtained from the test solution shows the same color and R_f value as those obtained from the standard solution.

Purity (1) ***Foreign matter***—(i) **Stem**: Achyranthes Root contains NMT 5.0% of the stem.

(ii) **Other foreign matter**: Achyranthes Root contains less than 1.0% of foreign matter other than the stem.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.7 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endosulfan (sum of α, β-endosulfan and endosulfan sulfate): NMT 0.2 ppm.

(vi) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 17.0% (6 hours).

Ash NMT 10.0%.

Acid-insoluble ash NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Prepared Aconite
부자(附子)

Aconiti Lateralis Radix Preparata

Prepared Aconite is the prepared daughter root of *Aconitum carmichaeli* Debeaux (Ranunculaceae). Depending on the method of preparation, they are classified into the following varieties: Yeombuja (Salted Aconite), Bujapyeon (Sliced Aconite) and Pobuja (Boiled Aconite).

Method of preparation (1) ***Yeombuja (Salted Aconite)***—Sort the daughter root of *Aconitum carmichaeli* harvested between June and August by size, and remove the mother root, rootlets and soil. Wash it with water and soak them in brine overnight. Add salt, immerse, remove and dry it in the sun. Repeat the above process and extend the drying time in the sun until a lot of salt crystallizes on the surface and the texture becomes firm. This is known as Yeombuja.

(2) ***Bujapyeon (Sliced Aconite)***—Immerse Yeombuja in water several times to rinse out the salt and cut lengthwise into 3 mm to 5 mm thick slices. Immerse them in water and steam until cooked through. Take them out, let them dry out to the half over a hot flame and then dry them in the sun.

(3) ***Pobuja (Boiled Aconite)***—Immerse Yeombuja in water and change the water 2 to 3 times a day until the salt is completely rinsed out. Boil it with Licorice root and black bean until cooked through. Take it out when it is no longer noticeable on the tongue, remove the periderm and cut it into slices or several pieces and dry it in the sun. This is known as Pobuja.

Description (1) ***Yeombuja (Salted Aconite)***—Yeombuja is the processed daughter root, conical, 4 cm to 7 cm in length and 3 cm to 5 cm in diameter. The outer surface is grayish black and covered with fine salt powder. There are depressed bud scars at the tip surrounded by root tubercles or rootlet scars. The texture is heavy and firm. The transversely cut surface is grayish brown and has small clefts filled with fine salt powder and a polygonal cambium ring. The vascular bundles are irregularly arranged inside the ring.

It has a slight characteristic odor and tastes salty, numbing and pungent.

(2) ***Bujapyeon (Sliced Aconite)***—Bujapyeon is processed Yeombuja, cut lengthwise, wide in the upper part and narrow in the lower part, 17 mm to 50 mm in length, 9 mm to 30 mm in width, 3 mm to 5 mm in thickness, yellowish white and translucent. The texture is hard and brittle. The fractured surface is horn-like.

Bujapyeon occurs as almost odorless and its taste is weak.

(3) ***Pobuja (Boiled Aconite)***—Pobuja is the processed Yeombuja cut into 3 mm to 5 mm thick slices with irregular shapes and sizes. The outer surface is pale brown to dark brown or black. The texture is firm, semi-transparent and slightly lustrous.

Identification Weigh 4 g of the pulverized Prepared Aconite, add 30 mL of ether and 5 mL of ammonia TS, shake for 20 minutes to mix, and filter. Transfer the filtrate to a separatory funnel, add 20 mL of 0.25 mol/L sulfuric acid, shake to mix and allow to stand. Collect the acid layer and determine the spectrum of the solution as directed under the Ultraviolet-visible Spectroscopy; the spectrum exhibits maximum absorption between 231 nm and 274 nm.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(3) ***Sulfur dioxide***—NMT 30 ppm.

(4) ***Aconitine***—Weigh 20 g of the pulverized Prepared Aconite, add 150 mL of ether, and shake for 10 minutes to mix. Add 10 mL of ammonia TS, shake for 30 minutes to mix, allow to stand for 1 to 2 hours, collect the ether layer, and evaporate to dryness. Dissolve the residue in 2 mL of ethanol and use this solution as the test solution. Separately weigh 20 mg of aconitine RS, dissolve in 10 mL of ethanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the standard solution on a thin-layer chromatographic plate of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of cyclohexane, ethyl acetate, triethylamine, and methanol (6 : 2 : 2 : 1) as a developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 254 nm); the spots obtained from the test solution are not more intense than the spots obtained from the standard solution.

Packaging and storage Preserve in well-closed containers.

Akebia Stem
목통(木通)

Akebiae Caulis

Akebia Stem is the stem of *Akebia quinata* Decaisne (Lardizabalaceae) from which the periderm has been removed.

Description Akebia Stem is the stem without the periderm, cylindrical, usually curved twisted, 30 cm to 70 cm in length and 0.5 cm to 2 cm in diameter. The outer surface is yellowish white to yellowish brown with very numerous longitudinal ribs. The nodes may appear dilated and have scars from lateral branches. Those with periderm are grayish brown with cylindrical or elongated ellipsoidal lenticels. The texture is light, firm and difficult to cut. The cut surface is dark grayish brown on both sides. The xylem shows light brown vessels and grayish white medullary rays, which are alternate and radially arranged. The pith is pale grayish yellow and distinct.
Under the microscope, a cross-section shows a wheel-shaped ring consisting mainly of fiber bundles with crystal-bearing cells and stone cell groups, arching around the relatively broad outer surface of the phloem. The medullary rays of the phloem consist of sclerenchymatous cells containing solitary crystals. The cambium is distinct, and the cells around the pith are remarkably thick-walled. Relatively well-developed large and small vessels run radially in the xylem, and the medullary rays are arranged in 4 to 5 rows. The medullary rays of the xylem and the parenchymatous cells around the pith contain solitary crystals of calcium oxalate and starch granules.
It occurs as almost odorless and has a slightly pungent taste.

Identification (1) Weigh 0.5 g of pulverized Akebia Stem, add 10 mL of water, and boil the mixture for a brief while. Then, allow it to cool down and shake vigorously; a persistent fine foam is produced.

(2) Weigh 2 g of pulverized Akebia Stem and Akebia stem RMPM, add 10 mL of methanol to each, and sonicate for 1 hour. Filter and use the filtrates as the test solution and the Akebia stem RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Akebia stem RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography (with fluorescent indicator). Next, develop the plate with a mixture of ethyl acetate, acetic acid (100) and water (81 : 11 : 8) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 365 nm); the several spots obtained from the test solution show the same color and R_f value as the spots from the Akebia stem RMPM standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 10.0%.

Packaging and storage Preserve in well-closed containers.

Alisma Rhizome
택사(澤瀉)

Alismatis Rhizoma

Alisma Rhizome is the tuber of *Alisma orientale* (Sam.) Juz. (Alismataceae), with rootlets and periderm removed.

Description Alisma Rhizome is a tuber, nearly globose, elliptic or ovate, 2 cm to 7 cm in length and 2 cm to 6 cm in diameter. The external surface is yellowish white or pale yellowish brown, with irregular, transverse, ring-shaped shallow furrows and numerous thin, small, protruding scars due to rootlets, sometimes with humpy shoot scars at the lower part. The body is light and the texture is solid. The cut surface is yellowish white, powdery, with several fine pores.
It has a slight, characteristic odor and taste.

Identification Weigh 1 g of pulverized Alisma Rhizome and Alisma rhizome RMPM, add 10 mL of methanol, sonicate for 30 minutes, filter the solutions, and use the filtrates as the test solution and the standard solution of Alisma rhizome RMPM, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution of Alisma rhizome RMPM on a plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of dichloromethane and methanol (17 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly sulfuric acid TS for spraying to this plate and heat at 105 °C; the several spots obtained from the test solution show the same color and R_f value as the spot obtained from the standard solution of Alisma rhizome RMPM.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 1.0 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): NMT 0.2 ppm.
(vi) Endrin: NMT 0.01 ppm.
(vii) Chlorpyrifos: NMT 0.5 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 5.0%.

Packaging and storage Preserve in well-closed containers.

Alpina Katsumadai Seed
초두구(草豆蔻)

Alpiniae Katsumadai Semen

Alpina Katsumadai Seed is the seed of *Alpinia katsumadai*

Hayata (Zingiberaceae), from which the pericarp has been removed.

Description Alpina Katsumadai Seed is a mass of seeds, subspheroidal, 15 mm to 27 mm in diameter. The external surface is grayish brown, with yellowish white septa in central part dividing the masses into three parts, each containing numerous seeds, sticky and agglutinated closely, and the masses of the seeds are smooth. Seeds are ovoid polyhedrons, 3 mm to 5 mm in length, about 3 mm in diameter. The outer layer is covered with a pale brown membranous aril, and the raphe appears as a single longitudinal furrow, with hilum at one end. The texture is hard, and the cut surface along the raphe is oblique-cordate. Endosperm is grayish white.
It has a characteristic odor and a pungent and slightly bitter taste.

Identification Weigh 1 g of pulverized Alpina Katsumadai Seed, add 5 mL of methanol, and warm on a water bath for 5 minutes. After cooling, filter, and use the filtrate as the test solution. Separately, dissolve 2 mg of cardamonin RS in 1 mL of methanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of cyclohexane, ethyl acetate, and acetic acid (100) (14 : 5 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly 5% Iron(III) chloride-ethanol solution on the plate; one of the spots obtained from the test solution shows the same color and R_f value as the spot from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 12.0%.

Packaging and storage Preserve in well-closed containers.

Alpinia Officinarum Rhizome
고량강(高良薑)

Alpiniae Officinari Rhizoma

Alpinia Officinarum Rhizome is the rhizome of *Alpinia officinarum* Hance (Zingiberaceae).

Description Alpinia Officinarum Rhizome is the rhizome, which is cylindrical, usually curved and branched, 5 cm to 9 cm long and 10 mm to 15 mm in diameter. The outer surface is reddish brown to dark brown with fine striped lines, blackish brown nodes with 2 mm to 10 mm in length, and several round scars of rootlet in one side. The texture is hard, tough and difficult to break. The fractured surface is grayish brown to reddish brown and fibrous, and the central cylinder accounts for one third.
Under the microscope, the cross-section shows that the outermost layer consists of the epidermis, and the epidermis cells sometimes contain resinous substances. The cortex and central cylinder are composed of parenchyma separated by the endodermis and vascular bundles are scattered with surrounding fibers. Scattered in the parenchyma are parenchyma cells containing a brown, oily substance, which have solitary calcium oxalate crystals and starch granules. The starch granules are mainly single grained and also has complex starch granules.
It has a characteristic odor and extremely pungent taste.

Identification (1) Weigh 1 g of pulverized Alpinia Officinarum Rhizome, add 10 mL of ether, and evaporate. To the residue obtained by evaporation, add 2 mL of phosphoric acid and heat to dissolve; a yellow color develops. Add 2 mL of water, shake and allow it to stand; the solution become cloudy.

(2) Weigh 1 g each of pulverized Alpinia Officinarum Rhizome and Alpinia officinarum rhizome RMPM, add 10 mL of methanol, sonicate for 60 minutes for extraction. Filter it and use the filtrates as the test solution and the alpinia officinarum rhizome RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL of the test solution and the Alpinia officinarum rhizome RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of toluene and ethyl acetate (7 : 1) as the developing solvent to a distance of about 10 cm, and air dry the plate. Spray evenly vanillin-sulfuric acid-ethanol TS on the plate and heat it at 105 ℃ for 10 minutes; the spots from the test solution and the spots from the Alpinia officinarum rhizome RMPM standard solution show the same color and the same R_f value.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 12.0% (6 hours).

Ash NMT 6.0%.

Acid-insoluble ash NMT 1.5%.

Essential oil content NLT 0.2 mL (50 g).

Extract content ***Dilute ethanol-soluble extract***— NLT 15.0%.

Packaging and storage Preserve in well-closed containers.

Amomum Fruit
사인(砂仁)

Amomi Fructus

Amomum Fruit is the ripe fruit or seed mass of *Amomum villosum* Lourerio var. *xanthioides* T.L.Wu et Senjen or *Amomum villosum* Lourerio (Zingiberaceae).

Description Amomum Fruit is the fruit, ellipsoidal or ovoid, indistinctly 3-margined, 15 mm to 20 mm in length and 10 mm to 15 mm in diameter. The outer surface is pale brown, densely covered with spiny appendages, there are remains of the perianth at the apex, and the base often bears a fruiting pedicel. The pericarp is thin and soft. The seeds are concentrated into a mass with three blunt ridges. The center is divided into 3 loci by white septa, and each locus contains 5 to 26 seeds. The seeds are irregularly polyhedral, about 3 mm in diameter, and the outer surface is reddish brown or dark brown, the outer layer is pale brown and covered with a membranous stigma. The texture is hard and the endosperm are grayish white.
It has a characteristic odor and tastes pungent, cool and slightly bitter.

Identification Dissolve 20 μL of Amomum Fruit essential oil in 1 mL of ethanol and use this solution as the test solution. Alternatively, weigh 1 g of pulverized Amomum Fruit, add 50 mL of ether, heat the mixture after connecting with a reflux condenser on a water bath for 1 hour, filter, and evaporate to dryness. Dissolve the residue in 2 mL of ether and use this solution as the test solution. Separately, dissolve 10 μL of bornyl acetate RS in 1 mL of ethanol and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on a thin-layer chromatographic plate of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of cyclohexane and ethyl acetate (22 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray vanillin-sulfuric acid TS evenly on the plate and heat at 105 °C; one of the several spots obtained from the test solution shows the same color and R_f value as the spot from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 9.0% (seed).

Acid-insoluble ash NMT 3.0% (seed).

Essential oil content NLT 0.6 mL (30.0 g, seed).

Packaging and storage Preserve in well-closed containers.

Amomum Tsao-ko Fruit
초과(草果)

Amomi Tsao-ko Fructus

Amomum Tsao-ko Fruit is the ripe fruit of *Amomum tsao-ko* Crevost et Lemaire (Zingiberaceae).

Description Amomum Tsao-ko Fruit is the long ellipsoidal fruit, 2 cm to 4 cm in length, 10 mm to 25 mm in diameter, with three prominent, dull ridges. External surface is grayish brown to reddish brown with longitudinal furrow and ridge, with round remains of stigma in apex and with a fruit stalk and its remains in base. The pericarp is tough and hard, easily splitting longitudinally. There are loculi divided into three groups by yellowish brown septa, each containing 8 to 11 seeds agglutinated into a mass. Seeds are conical polyhedral, about 5 mm in diameter. External surface is reddish brown, with a long longitudinal furrow in lateral side and concaved hilum in apex, and with grayish white membranous aril. Texture is hard and endosperm is grayish white.
It has a characteristic aroma and a pungent and slightly bitter taste.

Identification Dissolve 50 μL of essential oil obtained in the Essential oil content in 1 mL of ethanol, and use this solution as the test solution. Separately, dissolve 20 μL of cineol RS in 1 mL of ethanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of cyclohexane, ethyl acetate, and formic acid (16 : 2 : 0.5) to a distance of about 10 cm, and air-dry the plate. Spray evenly vanillin-sulfuric acid TS, and heat at 105 °C; one of the several spots obtained from the test solution shows the same color and R_f value as the spot from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.
(4) ***Benzopyrene***—NMT 5 ppb. However, perform the test according to the following procedure. Powder 500 to 600 g of Amomum Tsao-ko Fruit to make it homogeneous, weigh about 5.0 g accurately, add 100 mL of water, and sonicate for 60 minutes to extract. Add 100 mL of hexane and 1 mL of the internal standard solution, mix homogeneously for 5 minutes with a homogenizer, sonicate for 60 minutes to extract, and then centrifuge (3200 g, 10 minutes). Transfer the hexane layer into separatory funnel (I).
To the hexane layer of separatory funnel (I), add 50 mL of a mixture of *N*,*N*-dimethylformamide and water (9 : 1), shake to extract, and transfer the layer of a mixture of *N*,*N*-dimethylformamide and water (9 : 1) into separatory funnel (II) (repeat this procedure 3 times). To separatory funnel (II), add 100 mL of 1% sodium sulfate solution, shake, then add 50 mL of hexane, shake, and allow to stand. Transfer the separated hexane layer into separatory funnel (III). To separatory funnel(II), add 35 mL of hexane, shake to extract, and then combine the hexane layer into separatory funnel (III) (repeat this procedure 2 times). To separatory funnel (III), add 50 mL of water to wash, filter and dehydrate the

hexane layer with about 30 g of anhydrous sodium sulfate, and then concentrate hexane to about 2 mL on a water bath at 45 ℃ in vacuum (70 kPa). Use a Florisil cartridge (1g, 6 mL), previously activated by eluting 10 mL of dichloromethane and 20 mL of hexane in order at the rate of 2 to 3 drops per second. Add the extracted solution into the previously activated cartridge and elute 20 mL of a mixture of hexane and dichloromethane (3: 1) at the rate of 2 to 3 drops per second. Evaporate the eluate on a water bath at below 35 ℃ under nitrogen gas, then dissolve the residue in 1 mL of acetonitrile, filter through a syringe filter (pore size of 0.45 μm), and use the filtrate as the test solution. Separately, weigh accurately an appropriate amount of benzo[a]pyrene RS and 3-methylcholanthrene RS, and dissolve in acetonitrile to make the standard stock solution and the internal standard stock solution containing 1 μg per mL, respectively. Store the standard stock solution and the internal standard stock solution at 5 to 15 ℃, and use within 30 days. Pipet an appropriate amount of the standard stock solution and the internal standard stock solution, and dilute with acetonitrile to make solutions each containing 3, 5, 10, 20, and 40 ng of benzopyrene and 50 ng of the internal standard per mL, respectively, and use this solution as the standard solution. If the concentration of the test solution is outside the range of the calibration curve, adjust the concentration of the standard solution to be within the range of the calibration curve. Perform the test with 10 μL each of the test solution and standard solution as directed under the Liquid Chromatography according to the following conditions. Plot a calibration curve with the ratio [A_S/A_{IS}] of the peak area of benzopyrene to the peak area of the internal standard, obtained from each standard solution, on the Y-axis, and the concentration of benzopyrene on the X-axis. Calculate the concentration of benzopyrene by plotting the ratio [A_{SAM}/A_{SAMIS}] of the peak area of benzopyrene to the peak area of the internal standard in the test solution on the Y-axis.

A_S: Peak area of the reference material in calibration curve standard solution

A_{IS}: Peak area of the internal standard in calibration curve standard solution

A_{SAM}: Peak area of benzopyrene in the test solution

A_{SAMIS}: Peak area of internal standard in the test solution

Internal standard solution—Weigh accurately 3-methylcholantrene RS, and dissolve in acetonitrile to make a solution containing 50 ng per mL.

Reagents and test solutions—Water used for this test must be distilled NLT 3 times, and reagents must be those for residual pesticides or of a higher grade.

Operating conditions

Detectors: Fluoro spectrophotometer (excitation wavelengths 294 nm, fluorescence wavelengths 404 nm)

Column: Supelcosil LC-PAH (4.6 × 250 mm, 5 μm) or equivalent

Column temperature: 37 ℃

Mobile phase: A mixture of acetonitrile and water (4 : 1)

Flow rate: 1.0 mL/min

Loss on drying NMT 12.0%.

Ash NMT 9.0%.

Acid-insoluble ash NMT 3.0%.

Essential oil content NLT 0.3 mL (100.0 g).

Packaging and storage Preserve in well-closed containers.

Anemarrhena Rhizome
지모(知母)

Anemarrhena Rhizome is the rhizome of *Anemarrhena asphodeloides* Bunge (Liliaceae).

Anemarrhena Rhizome contains NLT 0.7% of mangiferin ($C_{19}H_{18}O_{11}$: 422.33), calculated on the dried basis.

Description Anemarrhena Rhizome is the rhizome, slightly flat, thick rod-like, slightly curved, 3 to 15 cm in length and 5 to 15 mm in diameter. The external surface exhibits a yellowish brown to brown color, with a single longitudinal furrow and hair-like remains or scars of leaf sheaths forming fine ring-nodes on the upper surface and with numerous scars of roots in the shape of round and dented spots on the lower surface. Anemarrhena Rhizome has a light texture, making it easy to break. The fractured surface exhibits a pale yellowish brown color. Under a magnifying glass, the transverse section reveals the extremely narrow cortex and the broad central cylinder, irregularly scattered with many vascular bundles, exhibiting a porous appearance due to the mucous cells or groups thereof.

Under a microscope, the transverse section of Anemarrhena Rhizome reveals the phelloderm consisting of several layers of cork cells and several layers of flat, rectangular cells. A small number of leaf-trace vascular bundles are visible in the cortex. The central cylinder is scattered with multiple collateral vascular bundles, and near the central cylinder sheath are transversely long root-trace vascular bundles. The vascular bundle sheath has a slightly thick cell wall, sometimes slightly lignified. Inside the parenchyma are many mucous cells, which are somewhat abundantly distributed in the cortex and contain raphide bundles of calcium oxalate. Many columnar crystal bundles of calcium oxalate are scattered throughout the parenchyma surrounding the vascular bundles. The parenchyma cells contain fatty oil droplets. It has a slight, characteristic odor and a slightly sweet and mucilaginous taste, followed by a bitter taste.

Identification (1) Weigh 0.5 g of pulverized Anemarrhena Rhizome, transfer into a test tube, add 10 mL of water, and shake vigorously to mix; a lasting fine foam is produced. Filter the mixture and to 2 mL of the filtrate, add 1 drop of Iron(III) chloride TS; a blackish green precipitate is formed.

(2) Weigh 0.5 g of pulverized Anemarrhena Rhizome, add 2 mL of acetic anhydride, warm on a water bath for 2 minutes while shaking, filter, add carefully 1 mL of sulfuric acid to the filtrate; a reddish brown color develops at the zone of contact.

(3) Weigh 2 g each of pulverized Anemarrhena Rhizome and Anemarrhena rhizome RMPM, add 20 mL each of ethanol, warm on a water bath after connecting with a reflux condenser for 40 minutes, and filter. To 10 mL each of the filtrates, add 1 mL of hydrochloric acid, warm on a water bath after connecting with a reflux condenser for 1 hour, filter, and vacuum-concentrate the filtrates until they become about 5 mL. To each concentrated solution, add 10 mL of water and 20 mL of toluene to extract and evaporate the toluene layer to dryness. Dissolve each residue in 2 mL of toluene and use these solutions as the test solution and the Anemarrhena rhizome RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Anemarrhena rhizome RMPM standard solution on the thin-

layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of toluene and acetone (9 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray vanillin sulfuric acid TS on the plate and heat at 105 °C for 10 minutes; the color and the R_f values of the several spots obtained from the test solution are the same as those of the spots obtained from the Anemarrhena rhizome bark RMPM standard solution. Of these spots, the spot of sarsasapogenin appears at the R_f value of about 0.4.

Purity (1) ***Foreign matter***—The amount of fiber, originating from leaves, and other foreign matters contained in Anemarrhena Rhizome is less than 3.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(vi) Cypermethrin: NMT 0.5 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 13.0%.

Ash NMT 7.0%.

Acid-insoluble ash NMT 2.5%.

Assay Weigh accurately about 0.05 g of pulverized Anemarrhena Rhizome, add 10 mL of diluted ethanol (7 in 10), sonicate for 1 hour to extract, filter, and use this solution as the test solution. Separately, weigh accurately about 1 mg of mangiferin RS, dissolve in diluted ethanol (7 in 10) to make exactly 10 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions, and determine the peak areas, A_T and A_S, of the test solution and the standard solution, respectively.

$$\text{Amount (mg) of mangiferin } (C_{19}H_{18}O_{11}) = \text{Amount (mg) of mangiferin RS} \times \frac{A_T}{A_S}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 260 nm)

Column: A stainless steel column 4 to 6 mm in internal diameter and 15 to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 to 10 μm in particle diameter).

Column temperature: A constant temperature of about 25 °C

Mobile phase: Control the mobile phases A and B stepwise as directed in the following table or via the concentration gradient.

Mobile phase A: A solution of trifluoroacetic acid (0.5 in 1000)

Mobile phase B: Methanol

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)
0	90	10
4	80	20
6	60	40
10	50	50
16	0	100
22	0	100

Flow rate: 1.0 mL/min

Packaging and storage Preserve in well-closed containers.

Angelica Dahurica Root
백지(白芷)

Angelicae Dahuricae Radix

Angelica Dahurica Root is the root of *Angelica dahurica* Bentham et Hooker f. or *Angelica dahurica* Bentham et Hooker f. var. *formosana* Shan et Yuan (Umbelliferae).

It contains NLT 0.7% in total of oxypeucedanin ($C_{16}H_{14}O_5$: 286.29), imperatorin ($C_{16}H_{14}O_4$: 270.29) and isoimperatorin ($C_{16}H_{14}O_4$: 270.29), calculated on the dried basis.

Description Angelica Dahurica Root is the root and a short main root has many long roots branch. It is almost fusiform, 10 cm to 25 cm in length and 15 mm to 25 mm in diameter. The outer surface is grayish brown to dark brown. A few remnants of leaf sheaths on the crown and ring nodes protruding close to the crown. Longitudinal wrinkles and numerous scars of rootlets which are elongated and protrude laterally are observed in the root. In cross section, the outer area is grayish white and the central area is sometimes dark brown.
Under the microscope, the cross- section shows vessels and medullary rays developing radially from the center, many starch granules and calcium oxalate druse in the parenchyma cells.
It has a characteristic odor and slightly bitter taste.

Identification Weigh 1 g each of pulverized Angelica Dahurica Root and Angelica dahurica root RMPM, add 20 mL of methanol and sonicate for 60 minutes, vacuum-concentrate. To the extracts, add 2 mL of methanol, filter and use these filtrates as the test solution and the Angelica dahurica root RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 2 μL each of the test solution and the Angelica dahurica root RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography (with fluorescent agent). Next, develop the plate with a mixture of hexane and ethyl acetate (2 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 254 nm); of the several spots obtained from the test solution, the spots near the R_f values of 0.45 and 0.7 each show the same color as those from the Angelica dahurica root RMPM standard solution.

Purity (1) ***Foreign matter***—(i) Leaf sheath: Angelica Dahurica Root contains less than 3.0% of leaf sheath.
(ii) Other foreign matter: The amount of foreign matter other than leaf sheath contained in Angelica Dahurica Root does not exceed than 1.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 14.0%.

Ash NMT 7.0%.

Acid-insoluble ash NMT 2.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 25.0%.

Assay Weigh accurately about 1 g of pulverized Angelica Dahurica Root, add 50 mL of methanol, sonicate for 1 hour and filter. Repeat the above procedure with the residue by adding 50 mL of methanol. Combine all of the filtrates, vacuum-concentrate, add 10 mL of methanol and use this solution as the test solution. Separately, weigh accurately about 1 mg each of oxypeucedanin RS, imperatorin RS and isoimperatorin RS (previously dried in a silica gel desiccator for 24 hours), add methanol to make exactly 10 mL and use these solutions as the standard solutions. Perform the test with 10 μL each of the test solution and the standard solutions as directed under Liquid Chromatography according to the following conditions and determine the peak areas, A_{Ta}, A_{Tb} and A_{Tc}, of oxypeucedanin, imperatorin and isoimperatorin in the test solution and the peak areas, A_{Sa}, A_{Sb} and A_{Sc}, of oxypeucedanin, imperatorin and isoimperatorin in the standard solutions.

$$\text{Amount (mg) of oxypeucedanin } (C_{16}H_{14}O_5) = \text{Amount (mg) of oxypeucedanin RS} \times \frac{A_{Ta}}{A_{Sa}}$$

$$\text{Amount (mg) of imperatorin } (C_{16}H_{14}O_4) = \text{Amount (mg) of imperatorin RS} \times \frac{A_{Tb}}{A_{Sb}}$$

$$\text{Amount (mg) of isoimperatorin } (C_{16}H_{14}O_4) = \text{Amount (mg) of isoimperatorin RS} \times \frac{A_{Tc}}{A_{Sc}}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 254 nm)

Column: A stainless column, 4 to 6 mm in internal diameter and 15 to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 to 10 μm in particle diameter).

Column temperature: An ordinary temperature.

Mobile phase: A mixture of methanol and water (65 : 35).

Flow rate: 1.0 mL/min

System suitability

System performance: Proceed with 10 μL of the standard solution according to the above conditions; oxypeucedanin, imperatorin and isoimperatorin are eluted in this order.

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of each peak area of oxypeucedanin, imperatorin and isoimperatorin is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Angelica Gigas Root
당귀(當歸)

Angelicae Gigantis Radix

Angelica Gigas Root is the root of *Angelica gigas* Nakai (Umbelliferae).

Angelica Gigas Root contains NLT 6.0% of the sum of nodakenin ($C_{20}H_{24}O_9$: 408.40 and total decursin [decursin ($C_{19}H_{20}O_5$: 328.36) and decursinol angelate ($C_{19}H_{20}O_5$: 328.36), calculated on the dried basis.

Description Angelica Gigas Root is the root, conical or narrow long conical, usually branched, 15cm to 25 cm long and 2 cm to 5 cm in diameter. The outer surface is pale yellowish brown to blackish brown with irregular longitudinal wrinkles and spot-shaped remains of fibrous roots. The crown is broad, usually with remains of stems and leaves. The texture is firm but fragile. The fractured surface has a pale brown or yellowish brown cortex, relatively sparse with numerous clefts, and the xylem is white or yellowish white.

Under the microscope, the cross-section shows that the cork consists of 5 to 6 cell layers, with the cells oriented transversely and that the parenchyma systematically arranged in a rectangular shape from the primary cortex to the xylem. The cortex has a schizogenic intercellular space, the secretory canal contains yellowish brown substances, and the bast fiber bundles are sparsely scattered. Scalariform or spiral vessels are mainly observed. Numerous starch granules are found in the parenchyma cells.

It has a slight, characteristic odor and a slightly bitter and sweet taste.

Identification Weigh 1 g each of pulverized Angelica Gigas Root and Angelica gigas root RMPM, dissolve them separately in 5 mL of ethanol, and heat on a water bath for 10 minutes. After cooling, filter it and use the filtrates as the test solution and the Angelica gigas root RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the Angelica gigas root RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography (with fluorescent indicator). Next, develop the plate with a mixture of hexane and ethyl acetate (2 : 1) as the developing solvent to a distance of about 10 cm and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 365 nm); two spots among the several spots obtained from the test solution show the same color and R_f value as the spots from the Angelica gigas root RMPM standard solution, and of these, the spot of decrusinol and the spot of decrusin appear at the R_f value of about 0.1 and about 0.4, respectively.

Purity (1) ***Foreign matter***—(i) Stem and woody root: Angelica Gigas Root contains less than 5.0% of the stem and woody root.

(ii) Other foreign matter: Angelica Gigas Root contains less than 1.0% of foreign matter other than the stem and woody root.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.
(iii) Methoxychlor: NMT 1 ppm.
(iv) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(v) Azocyclotin: NMT 0.2 ppm.
(vi) Azoxystrobin: NMT 0.1 ppm.
(vii) Aldrin: NMT 0.01 ppm
(viii) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): NMT 0.2 ppm.
(ix) Endrin: NMT 0.01 ppm.
(x) Terbuconazole: NMT 1.0 ppm.
(xi) Pendimethalin: NMT 0.2 ppm.
(xii) Fenpropathrin: NMT 0.2 ppm.
(xiii) Sethoxydim: NMT 0.2 ppm.
(xiv) Fluazifop-butyl: NMT 0.3 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 12.0%.

Ash NMT 6.0%.

Assay Weigh accurately about 0.5 g of pulverized Angelica Gigas Root, add 20 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour, and filter it. To the residue, add 20 mL of methanol and proceed in the same manner. Combine all the filtrates and add methanol to make exactly 50 mL and use this solution as the test solution. Separately, weigh accurately about 10 mg of nodakenin RS, and dissolve it in methanol to make 20 mL. Pipet 5 mL of this solution. Weigh accurately about 10 mg of decursin RS, add methanol to make exactly 50 mL, and use this solution as the standard solution. Pipet 10 μL each of the test solution and the standard solutions, and perform the test as directed under the Liquid Chromatography according to the following conditions. Determine the peak areas, A_{Ta}, A_{Tb} and A_{Tc}, of nodakenin, decursin and decursinol angelate (the relative retention time to decursin is about 1.02), respectively, of the test solution, and A_{Sa} and A_{Sb}, of nodakenin and decursin, respectively, of the standard solution.

$$\text{Amount (mg) of nodakenin } (C_{20}H_{24}O_9) = \text{Amount (mg) of nodakenin RS} \times \frac{A_{Ta}}{A_{Sa}} \times \frac{1}{4}$$

$$\text{Amount (mg) of total decursin [decursin } (C_{19}H_{20}O_5) \text{ and decursinol angelate } (C_{19}H_{20}O_5)] = \text{Amount (mg) of decursin RS} \times \frac{A_{Tb}+A_{Tc}}{A_{Sb}}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 330 nm)

Column: A stainless column, 4.6 mm in internal diameter and 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: An ordinary temperature.

Mobile phase: Control the mixing ratio of mobile phase A and mobile phase B stepwise or via the concentration gradient as follows.

Mobile phase A: Acetonitrile

Mobile phase B: Water

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)
0	20	80
3	20	80
8	30	70
18	30	70
19	50	50
40	50	50
41	90	10
50	90	10

Flow rate: 1.0 mL/min

System suitability

System performance: Proceed with 10 μL of the standard solution according to the above conditions; nodakenin and decursin are eluted in this order, making adjustments so that the resolution between the peaks are separated completely.

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of each peak area of nodakenin and decursin is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Apricot Kernel
행인(杏仁)

Armeniacae Semen

Apricot Kernel is the well ripe seed of *Prunus armeniaca* Linné var. *ansu* Maximowicz, *Prunus mandshurica* Koehne var. *glabra* Nakai, *Prunus sibirica* Linné or *Prunus armeniaca* Linné (Rosaceae).

Apricot Kernel contains NLT 3.0% of amygdalin ($C_{20}H_{27}NO_{11}$: 457.43), calculated on the dried basis.

Description Apricot Kernel is the flattened ovate seed, 10 mm to 18 mm in length, 8 mm to 13 mm in width and 4 mm to 7 mm in thickness. One end is sharp and pointed and the other end is rounded, thickened and asymmetric. A short, linear hilum is situated on one side of the sharp end and the chalaza at the rounded end. The seed coat occurs as brown and its surface has epidermis cells easily detachable by rubbing, giving a powdery appearance. Several deep brown vein patterns stretch upwards from the chalaza. The seed coat and pale translucent white endosperm easily separate from the cotyledon when softened in hot water. There are two cotyledons, which are milky white and oily.
Under a microscope, the transverse section reveals a single row of epidermis cells on the outside, among which are yellow stone cells protruding. These stone cells are almost uniform in shape, angular orbicular or orbicular, 60 μm to 90 μm in diameter. The cell wall is uniformly thickened, obtusely triangular when viewed laterally, and the cell membrane is conspicuously thickened at the apex. The lower part has cells in a wrinkled nutrient layer with thin, small vascular bundles. The endocuticle consists of a single row containing yellow substances. The perisperm is made up of several rows of degenerated parenchyma and the endosperm consists of a single row of rectangular cells containing aleurone grains and fatty oil.
It occurs as almost odorless and has bitter taste.

Identification Weigh 1 g of pulverized Apricot Kernel, add 10 mL of methanol, heat for 10 minutes on a water bath after connecting with a reflux condenser. After cooling, filter and use this solution as the test solution. Separately, dissolve 2 mg of amygdalin RS in 1 mL of methanol and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test

solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, methanol and water (7 : 3 : l) to a distance of about 10 cm, and air-dry the plate. Spray evenly sulfuric acid TS for spray and heat at 105 °C for 10 minutes; one spot among the several spots obtained from the test solution and a brown to dark brown spot from the standard solution exhibit the same color and have the same R_f value.

Purity (1) ***Foreign matter***—Apricot Kernel does not contain fragments of endocarp and other foreign matter.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxides*—**NMT 30 ppm.

(5) ***Mycotoxins*—**Total aflatoxin (sum of aflatoxins B_1, B_2, G_1 and G_2): NMT 15 ppb (aflatoxin B_1 is NMT 10.0 ppb).

(6) ***Rancidity*—**Grind Apricot Kernel with hot water; no unpleasant odor of rancid oil is perceptible.

Loss on drying NMT 8.0%.

Assay Weigh accurately about 0.5 g of pulverized Apricot Kernel, add 50 mL of methanol, heat after connecting with a reflux condenser for 2 hours, and filter. Repeat the above procedure with the residue using 50 mL of methanol. Combine all the filtrates and evaporate the solvent in vacuum. Add 70 mL of water and 70 mL of hexane to the residue, shake to mix, and discard the hexane layer. Add about 70 mL of ether, shake to mix, and discard the ether layer. Filter the water layer, add water to make exactly 100 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of amygdalin RS (previously dried in a silica gel desiccator for 24 hours), add water to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions, and calculate the peak areas, A_T and A_S, of the test solution and the standard solution.

$$\text{Amount (mg) of amygdalin } (C_{20}H_{27}NO_{11}) = \text{Amount (mg) of amygdalin RS} \times \frac{A_T}{A_S}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 214 nm)

Column: A stainless column 4 to 6 mm in internal diameter and 15 to 25 cm in, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Column temperature: An ordinary temperature

Mobile phase: A mixture of water and methanol (80 : 20).

Flow rate: 1.0 mL/min

System suitability

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak area of amygdalin is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Aralia Continentalis Root
독활(獨活)

Araliae Continentalis Radix

Aralia Continentalis Root is the root of *Aralia continentalis* Kitakawa (Araliaceae).

Aralia Continentalis Root contains NLT 0.4% in total of kaurenoic acid ($C_{20}H_{30}O_2$: 302.45) and continentalic acid ($C_{20}H_{30}O_2$: 302.45), calculated on the dried basis.

Description Aralia Continentalis Root is the root, long cylindrical to rod shaped, 10 cm to 30 cm in length and 5 mm to 20 mm in diameter. External surface exhibits a grayish white to grayish brown color, with longitudinal wrinkles and rootlet scars. The fractured surface is fibrous with the pale yellow pith, and the texture is light and loose.

Under a microscope, the transverse section of Aralia Continentalis Root reveals a small resin canal with secretary cells within the collenchyma. The cambium is clearly formed, consisting of 3 to 5 rows. Xylem fibers are developed around the vessels in the xylem, and the medullary rays consisting of 3 to 5 rows are connected from the pith to the phloem.

It has a characteristic odor and an unpleasant and slightly bitter taste.

Identification (1) Weigh 0.5 g of pulverized Aralia Continentalis Root, add 10 mL of chloroform, extract for 1 hour with shaking, allow to stand for 15 minutes, and filter. Transfer 1 mL of the filtrate into a test tube, add 0.5 mL of acetic anhydride, shake to mix, and carefully add 0.5 mL of sulfuric acid to make two layers; a red to deep red color develops at the zone of contact, and the upper layer exhibits a yellowish red to dark yellowish red color.

(2) Weigh 0.1 g of pulverized Aralia Continentalis Root and Aralia continentalis root RMPM, add 10 mL each of methanol, sonicate for 1 hour to extract, filter, and use the filtrates as the test solution and the Aralia continentalis root RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the Aralia continentalis root RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of n-hexane and ethyl acetate (2 : 1) to a distance of about 10 cm, take out the plate, and air-dry. Spray evenly dilute sulfuric acid TS for spray on the plate and heat at 105 °C for 10 minutes; the color and the R_f values of the several spots obtained from the test solution are the same as those of the spots obtained from the Aralia continentalis root RMPM standard solution. Of these spots, a spot appears at the R_f value of about 0.6.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 12.0% (6 hours).

Ash NMT 9.0%.

Acid-insoluble ash NMT 2.0%.

Assay Weigh accurately about 0.2 g of pulverized Aralia Continentalis Root, add 10 mL of ethanol, sonicate for 1 hour, filter, and use the filtrate as the test solution. Separately, weigh accurately about 2.0 mg each of kaurenoic acid RS and continentalic acid RS (previously dried in a desiccator with silica gel for 24 hours), dissolve in ethanol to make exactly 10 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions, and calculate the peak areas, A_{Ta} and A_{Tb}, of kaurenoic acid and continentalic acid, respectively, from the test solution and the peak areas, A_{Sa} and A_{Sb}, of kaurenoic acid and continentalic acid, respectively, from the standard solution.

$$\text{Amount (mg) of kaurenoic acid } (C_{20}H_{30}O_2) = \text{Amount (mg) of kaurenoic acid RS} \times \frac{A_{Ta}}{A_{Sa}}$$

$$\text{Amount (mg) of continentalic acid } (C_{20}H_{30}O_2) = \text{Amount (mg) of continentalic acid RS} \times \frac{A_{Tb}}{A_{Sb}}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 205 nm)

Column: A stainless steel column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Column temperature: A constant temperature of about 25 °C

Mobile phase: A mixture of acetonitrile, water and trifluoroacetic acid (65 : 35 : 0.1)

Flow rate: 1.5 mL/min

Packaging and storage Preserve in well-closed containers.

Arctium Fruit
우방자(牛蒡子)

Arctii Fructus

Arctium Fruit is the ripe fruit of *Arctium lappa* Linné (Compositae).

Description Arctium Fruit is oblong, obovate, usually flat and slightly curved, 5 mm to 7 mm long, 2 mm to 3.2 mm in diameter, 0.8 mm to 1.5 mm thick, and the outer surface is grayish brown to brown with blackish purple spots. The longitudinal lines are several, with 1 to 2 lines usually distinct in the center. The apex is bluntly round and somewhat broad with circular ring patterns on the upper surface and patches of stigma scars in the center. The lower part is slightly narrower than the upper part, and surface of attachment is relatively pale in color. The pericarp is relatively hard with 2 cotyledons, pale yellowish white, abundantly oily. The mass of 100 Arctium Fruits weighs 1.0 g to 1.5 g.
It has no odor and tastes bitter and oily.

Identification Weigh 0.5 g of pulverized Arctium Fruit, add 20 mL of methanol, shake for 10 minutes, and filter. Use the filtrate as the test solution. With this solution, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL of the test solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of acetone, ethyl acetate and water (15 : 10 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray dilute sulfuric acid TS evenly on the plate and heat at 105 ℃ for 10 minutes; a reddish purple spot appears at an R_f value of about 0.4.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 12.0% (6 hours).

Ash NMT 7.0%.

Acid-insoluble ash NMT 1.0%.

Extract content ***Dilute ethanol–soluble extract***—NLT 15.0%.

Packaging and storage Preserve in well-closed containers.

Areca
빈랑자(檳榔子)

Arecae Semen

Areca is the ripe seed of *Areca catechu* Linné (Palmae), which is collected, boiled in water and removed from the pericarp.

Description Areca is the seed, roundish-conical or flattened almost spherical, 15 mm to 35 mm high and 15 mm to 30 mm in diameter. The hilum is present in the middle of the base and usually forms a dent. The outer surface is grayish reddish brown to grayish yellowish brown, with a pale reticular pattern. The texture is very firm and difficult to crack. The cross-section has a dense texture and shows a marble-like floral pattern of grayish brown seed coat alternating with white albumen. The interior is sometimes hollow.
It has a light, characteristic odor and an astringent, slightly bitter taste.

Identification Weigh 3 g of pulverized Areca and Areca RMPM in a stoppered centrifuge tube, add 30 mL of ether and 5 mL of sodium hydroxide TS, stopper, and shake for 5 minutes to mix. Centrifuge and collect the supernatant. Evaporate ether on a water bath, dissolve the residue in 1.5 mL of methanol, and use

these solutions as the test solution and the Areca RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the Areca RMPM standard solution on a thin-layer chromatographic plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of acetone, water and acetic acid (100) (10 : 6 : 1) as a developing solvent to a distance of about 10 cm and air-dry the plate. Spray iodine TS evenly on the plate; the several spots obtained from the test solution show the same color and R_f value as the spot obtained from the Areca RMPM standard solution.

Purity (1) ***Foreign matter***—(i) Pericarp: Areca contains less than 2.0% of the pericarp.

(ii) Other foreign matter: Areca contains NMT 1.0% of foreign matter other than the pericarp.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

(5) ***Mycotoxins***—Total aflatoxin (sum of aflatoxin B_1, B_2, G_1 and G_2): NMT 15.0 ppb (aflatoxin B_1 is NMT 10.0 ppb).

Ash NMT 2.5%.

Packaging and storage Preserve in well-closed containers.

Areca Peel
대복피(大腹皮)

Arecae Pericarpium

Acera Peel is the pericarp of *Areca catechu* Linné (Palmae) of which the fruit is unripe after boiled. The pericarp from unripe fruit is known as Areca peel, and the one from the ripe fruit is known as Daebokmo.

Description (1) ***Areca peel***—Areca peel is the pericarp, which is usually elliptical or long-ovate, gourd-like shape, 4 cm to 7 cm in length, 20 to 35 cm wide, and 2 to 5 mm thick. The epicarp is deep brown to black, with irregular longitudinal wrinkles, protrudent transverse lines on the surfaces. The style scars are present at the apex, remains of the fruiting stalk and calyx at the base. The endocarp is indented, brown to deep brown, lustrous, smooth and hard-shelled. The texture is light and firm and the fibers of the mesocarp are visible after longitudinally cracked.
It has a light, characteristic odor and a slightly astringent taste.

(2) ***Daebokmo***—Daebokmo is the pericarp, usually elliptical or gourd-shaped. The epicarp is mostly already lost but sometimes remained. The mesocarp is palm hair-shaped, yellowish white or pale brown, sparse and soft. The endocarp is hard and shell-shaped, yellowish brown to deep brown. The internal surface is lustrous, smooth, and sometimes gaps longitudinally.
It has a slight characteristic odor and a light taste.

Identification Weigh 0.5 g of pulverized Areca Peel, add 5 mL of water, shake for 2 to 3 minutes and filter. To 2 mL of the filtrate, add 1 mL of lead acetate TS; the filtrate turns pale yellow with turbidity and yellow precipitates are slowly generated.

Purity (1) ***Foreign matter***—Areca Peel contains less than 10.0% of Areca and foreign matters.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 12.0% (6 hours).

Ash NMT 7.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 5.0%.

Packaging and storage Preserve in well-closed containers.

Arisaema Rhizome
천남성(天南星)

Arisaematis Rhizoma

Arisaema Rhizome is the tuber of *Arisaema amurense* Maximowicz, *Arisaema erubescens* Schott or *Arisaema heterophyllum* Blume (Araceae), from which the cork layer has been completely removed.

Description Arisaema Rhizome is the tuber, irregular oblate, 1.5 to 7 cm in diameter and 1 to 3 cm in height. The external surface occurs as a white to pale brown color. It is relatively smooth with dented stem scars at the top, which are surrounded by pitted root scars, sometimes with small, flattened, globose axillary buds near the tuber. The texture is very hard, not easily broken, and the transverse section is uneven, white and powdery.
It has a slight, pungent odor and a pungent taste.

Identification (1) Weigh 0.5 g of pulverized Arisaema Rhizome, add 10 mL of water to macerate, and shake vigorously; a lasting fine foam is produced.

(2) Weigh 0.2 g of pulverized Arisaema Rhizome, add 2 mL of acetic anhydride, warm on a water bath for 2 minutes, filter, carefully add 0.5 mL of sulfuric acid to the filtrate; a pale brown color develops at the zone of contact.

(3) Apply dilute iodine TS dropwise to the cut surface of Arisaema Rhizome; it exhibits a dark bluish purple color.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2

ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 15.0% (6 hours).

Ash NMT 5.0%.

Acid-insoluble ash NMT 1.0%.

Packaging and storage Preserve in well-closed containers.

Asiasarum Root and Rhizome
세신(細辛)

Asiasari Radix et Rhizoma

Asiasarum Root and Rhizome is the root and rhizome of *Asiasarum heteropoides* F. Maekawa var. *mandshuricum* F. Maekawa or *Asiasarum sieboldii* Miquel var. *seoulense* Nakai (Aristolochiaceae).

Description Asiasarum Root and Rhizome is the root and rhizome, usually rolled to form a single mass. The rhizome stretches crosswise to form an irregular cylinder, short branched, 1 cm to 10 cm in length and 0.2 cm to 0.4 cm in diameter. The external surface of the rhizome exhibits a grayish brown color with ring-shaped nodes, 0.2 cm to 0.3 cm in internode distance, and bowl-shaped stem scars at the branched end. The root is thin and long, packed on the rhizome nodes, 10 cm to 20 cm in length and 0.1 cm in diameter. The external surface of the root exhibits a grayish yellow color, smoothly elongated or longitudinally wrinkled, with rootlets and rootlet scars. The texture is fragile and easy to cut. The cut surface is smooth, exhibiting a yellowish white or white color.

Under a microscope, the transverse section of Asiasarum Root and Rhizome reveals the bark consisting of a single row of cells, longitudinally long or close to transversely long rectangular, with some epidermis cells remaining on the outside. The cortex consists of 10 to 17 layers of cells, and the intercellular space is distinct. Cells in the first row of the external layer are densely packed and include a few cells containing a yellow or yellowish brown substance. The cortex is scattered with numerous oil cells, and the oil cell walls are suberized or slightly suberized. The parenchyma cells of the cortex are filled with starch grains. The endodermal layer is distinct and shows a casparian strip. The central cylinder sheath cells are in 1 to 2 rows. The secondary tissue is not developed, the primary xylem is diarch to triarch, and there are 13 to 27 vessels. 1 to 3 large parenchyma cells surrounded by the phloem cells are visible among the phloem bundles. The diameter of the longer side of the large parenchyma cells is smaller than the diameter of the largest vessel. *Asiasarum sieboldii* var. *seoulense* is very similar to *Asiasarum heteropoides* var. *mandshuricum*, but the former is different from the latter in that it has 25 to 43 vessels at the top of the root and the diameter of its largest vessel is larger than that of the latter.

It has a characteristic odor and a pungent taste slightly numbing the tongue.

Identification Weigh 1 g each of pulverized Asiasarum Root and Rhizome and Asiasarum root and rhizome RMPM, add 10 mL each of methanol, sonicate for 30 minutes to extract, filter, and use the filtrates as the test solution and the Ssiasarum root and rhizome RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Asiasarum root and rhizome RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of toluene and ethyl acetate (4 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly sulfuric acid TS for spray on the plate and heat at 105 ℃ for 10 minutes; the color and the R_f values of the several spots obtained from the test solution are the same as those of the spots obtained from the Asiasarum root and rhizome RMPM standard solution.

Purity (1) ***Foreign matter***—(i) Terrestrial part: No terrestrial part, including leaves and petioles, is contained in Asiasarum Root and Rhizome.

(ii) Other foreign matters: The amount of foreign matters other than the terrestrial part contained in Asiasarum Root and Rhizome is less than 1.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 1.0 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 10.0%.

Acid-insoluble ash NMT 3.0%.

Essential oil content NLT 0.6 mL (30.0 g).

Packaging and storage Preserve in well-closed containers.

Asparagus Tuber
천문동(天門冬)

Asparagi Tuber

Asparagus Tuber is the tuber of *Asparagus cochinchinensis* Merill (Liliaceae), of which outer skin is removed after being boiled or steamed in hot water, followed by drying.

Description Asparagus Tuber is the tuber, fusiform to globular, usually curved, 5 to 15 cm in length and 5 to 20 mm in diameter. The external surface occurs as a pale yellowish brown to pale brown color, semi-translucent, smooth or longitudinally wrinkled at irregular depths and sometimes with the grayish brown epidermis remaining. The texture is hard, smooth, lustrous and viscous. The cut surface is horny, and the central cylinder occurs as a yellowish white color.

Under a microscope, the transverse section of Asparagus Tuber sometimes reveals the remains of the root bark. The epidermis is composed of a single layer of the root bark cells, and the walls are sclerified. The cortex takes up 2/3 of the root, and groups of the stone cells form a ring made of a single row on the outside. The stone cells occurs as a pale yellowish brown color. The mucous cells are scattered in the cortex and contain raphide bundles

of calcium oxalate. The endodermis is distinct with a single row of the pericycle forming a ring immediately below. The protoxylem and sieve tubes are immediately below with the tracheids surrounding the vessels. The parenchyma cells in the pith are scattered with mucous cells, which contain raphide bundles of calcium oxalate.
It has a slight, characteristic odor and a sweet taste at first, followed by a bitter taste.

Identification (1) Weigh 0.5 g of pulverized Asparagus Tuber, add 10 mL of water, warm for 2 to 3 minutes on a water bath, and filter. To 3 mL of the filtrate, add 1 mL of Fehling's solution TS and warm on a water bath; a reddish brown precipitate is formed.

(2) Weigh 1 g each of pulverized Asparagus Tuber and Asparagus tuber RMPM, add 5 mL each of a mixture of butanol and water (40 : 7), shake for 30 minutes to mix, and filter. Use the filtrates as the test solution and the Asparagus tuber RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Asparagus tuber RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate using a mixture of butanol, water and acetic acid (100) (10 : 6 : 3) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS on the plate and heat at 105 ℃ for 2 minutes; the color and the R_f values of the several spots obtained from the test solution are the same as those of the spots obtained from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 18.0% (6 hours).

Ash NMT 3.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 25.0%.

Packaging and storage Preserve in well-closed containers.

Aster Root and Rhizome
자완(紫菀)

Asteris Radix et Rhizoma

Aster Root and Rhizome is the root and rhizome of *Aster tataricus* Linné fil. (Compositae).

Description Aster Root and Rhizome is the root and rhizome. The rhizome is in the shape of irregular masses, varying in size. The top of the rhizome has remains of stems and leaves. The texture is slightly hard. The root consists of many thin roots forming bundles from the rhizome, mostly plaited, 3 to 15 cm in length and 0.1 to 0.3 cm in diameter. The external surface exhibits a reddish purple to grayish red color with longitudinally wrinkles. The texture is relatively soft and tough, and the fractured surface is fibrous.
It has a characteristic odor and a slightly bitter and acrid taste.

Identification (1) Weigh 0.2 g of pulverized Aster Root and Rhizome, add 10 mL of water, shake vigorously to mix, and filter. To 2 mL of the filtrate, add 1 to 2 drops of Iron(III) chloride TS; the resulting solution exhibits a navy blue color.

(2) Weigh 0.5 g of pulverized Aster Root and Rhizome, add 10 mL of water, shake vigorously to mix; a lasting fine foam is produced.

(3) Weigh 1 g of pulverized Aster Root and Rhizome, add 25 mL of methanol, sonicate for 30 minutes to extract, and filter. Vacuum-concentrate the filtrate, dissolve in 2 mL of ethyl acetate, and use this solution as the test solution. Separately, weigh 1 mg of shionone RS, dissolve in 2 mL of ethyl acetate, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 3 μL each of the test solution and the standard solutions on the thin-layer chromatographic plate made of silica gel (with a fluorescent agent) for thin layer chromatography. Next, develop the plate using a mixture of petroleum ether and ethyl acetone (20 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly sulfuric acid TS for spray on the plate and heat at 105 ℃; the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

Purity (1) ***Foreign matter***—The amount of stems and other foreign matters contained in Aster Root and Rhizome is less than 5.0%.
(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 15.0% (6 hours).

Ash NMT 15.0%.

Acid-insoluble ash NMT 8.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 30.0%.

Packaging and storage Preserve in well-closed containers.

Astragalus Root
황기(黃芪)

Astragali Radix

Astragalus Root is the root, with or without periderm, of *Astragalus membranaceus* Bunge or *Astragalus membranaceus* Bunge var. *mongholicus* Hsiao (Leguminosae).

Description Astragalus Root is a thin and long cylindrical root, 30 cm to 100 cm in length and 7 mm to 20 mm in diameter, with small bases of lateral root dispersed on the surface but not branched, twisted near the crown, and ring nodes remained. External surface occurs as pale grayish yellow to pale yellowish brown and covered with irregular, dispersed longitudinal wrinkles and horizontal lenticel-like patterns. The texture is dense and difficult to break, and the fractured surface is fibrous. Under a magnifying glass, a transverse section reveals an outer layer composed of periderm, cortex is pale yellowish white, xylem is pale yellow and zone near the cambium is somewhat yellowish brown. Thickness of the cortex is from about one-third to one-half of the diameter of xylem. White medullary ray runs from xylem to cortex in thin root, but often appears as radiating cracks in thick root. The pith is usually unobservable.
It has a slight, characteristic odor and sweet taste.

Identification Weigh 3 g each of pulverized Astragalus Root and Astragalus root RMPM, add 20 mL each of methanol, heat after connecting with a reflux condenser for 1 hour, filter, and evaporate the filtrates to dryness. Dissolve each of the residues in 0.5 mL of methanol and use these solutions as the test solution and the Astragalus root RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the Astragalus root RMPM standard solution on a thin-layer chromatographic plate made of silica gel (with fluorescence indicator) for thin-layer chromatography. Develop the plate with a mixture of toluene and methanol (9 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 254 nm); the several spots obtained from the test solution show the same color and R_f value as the spots from the Astragalus root RMPM standard solution and of these, one spot among the several spots appears at the R_f value of about 0.2.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Napropamide: NMT 0.1 ppm.
(ii) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(iii) Dieldrin: NMT 0.01 ppm.
(ii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(v) Acetamiprid: NMT 0.1 ppm.
(vi) Azoxystrobin: NMT 0.1 ppm
(vii) Aldrin: NMT 0.01 ppm
(vii) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): NMT 0.2 ppm.
(ix) Endrin: NMT 0.01 ppm.
(x) Imidacloprid: NMT 0.3 ppm.
(xi) Triflumizole: NMT 0.1 ppm.
(xii) Thiamethoxam: NMT 0.1 ppm.
(xiii) Fenarimol: NMT 0.5 ppm.
(xiv) Pymetrozine: NMT 0.05 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.
(4) ***Root of Hedysarum species and others***—Under a microscope, a vertical section of Astragalus Root reveals no crystal-bearing cells containing solitary crystals of calcium oxalate outside edge the fiber bundle.

Loss on drying NMT 13.0% (6 hours).

Ash NMT 5.0%.

Acid-insoluble ash NMT 1.0%.

Packaging and storage Preserve in well-closed containers.

Atractylodes Rhizome
창출(蒼朮)

Atractylodis Rhizoma

Atractylodes Rhizome is the rhizome of *Atractylodes lancea* DC or *Atractylodes chinensis* Koidz. (Compositae).

Description Atractylodes Rhizome is the rhizome, cylindrical and irregularly curved, 3 to 10 cm in length and 10 to 25 mm in diameter. The external surface occurs as a dark grayish brown to dark yellowish brown color. The transverse section reveals a nearly orbicular surface, with pale brown to reddish brown fine dots caused by secretions. After a long-term storage, white cotton-like crystals are produced from Atractylodes Rhizome.
Under a microscope, the transverse section of Atractylodes Rhizome reveals no fiber bundles in the parenchyma of the cortex, and at the end of the medullary rays are the oil cells containing pale brown to yellowish brown substances. In the xylem, fiber bundles that surround vessels in contact with the cambium are arranged radially. The pith and the medullary rays contain the oil cells as in the cortex, and the parenchyma cells contain spherocrystals of inulin and raphides of calcium oxalate.
It has a characteristic odor and a slightly bitter taste.

Identification Weigh 0.5 g each of pulverized Atractylodes Rhizome and Atractylodes rhizome RMPM, add 2 mL each of hexane, sonicate for 15 minutes to extract, filter, and use the filtrates as the test solution and the Atractylodes rhizome RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the Atractylodes rhizome RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of petroleum ether and ethyl acetate (50 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS on the plate and heat at 105 °C; the color and the R_f values of the several spots obtained from the test solution are the same as those of the spots obtained from the Atractylodes rhizome RMPM standard solution. Of these spots, a deep green spot appears at the R_f value of about 0.5.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.7 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(vi) Tolylfluanid: NMT 1 ppm.
(vii) Procymidone: NMT 0.1 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.
(4) ***Atractylodes rhizome White***—Weigh 0.5 g of

pulverized Atractylodes Rhizome, macerate with 5 mL of ethanol by warming on a water bath for 2 minutes, and filter. To 2 mL of the filtrate, add 0.5 mL of vanillin-hydrochloric acid TS and shake immediately to mix; the resulting solution does not exhibit a red to reddish purple color within l minute.

Loss on drying NMT 13.0%.

Ash NMT 7.0%.

Acid-insoluble ash NMT 1.5%.

Essential oil content NLT 0.7 mL (50.0 g).

Packaging and storage Preserve in well-closed containers.

Atractylodes Rhizome White
백출(白朮)

Atractylodis Rhizoma Alba

Atractylodes Rhizome White is the rhizome, with or without periderm, of *Atractylodes japonica* Koidz or *Atractylodes macrocephala* Koidz (Compositae).

Description ***Atractylodes japonica***—Atractylodes Rhizome White of *Atractylodes japonica* is a rhizome in the form of an irregular mass or curved cylinder with a length of 3 cm to 8 cm and a diameter of 2 cm to 3 cm. In those without a peridome, the outer surface occurs as pale grayish yellow to pale yellowish white and grayish brown here and there. When the peridome is retained, the outer surface occurs as grayish brown, sometimes in a protruding nodule, with coarse wrinkles. The texture is difficult to break and the fractured surface is fibrous.

Under the microscope, the cross-section shows stone cell layers in the periderm, often fiber bundles on the outside of the phloem in the parenchyma of the cortex, and oil sacs with pale brown to brown substances at the end of the medullary rays. The xylem shows small, radially lined vessels surrounding the pith and distinct fiber bundles surrounding these vessels. The pith and medullary rays contain oil sacs similar to those in the cortex. The parenchyma tissue contains small inulin crystals and calcium oxalate crystals.

It has a characteristic odor and a somewhat bitter taste.

Atractylodes macrocephala—Atractylodes Rhizome White of *Atractylodes macrocephala* is the rhizome in the form of an irregularly enlarged mass, 3 cm to 13 cm in length and 15 mm to 70 mm in diameter. The outer surface occurs as grayish yellow or dark brown with scattered, cup-like small projections, interrupted longitudinal wrinkles and grooves, and scars of fibrous roots. Remnants of stems and bud scars are attached to the apex. The texture is firm and difficult to break. The fractured surface is not flat and occurs as yellowish white to pale brown and dotted with yellowish brown oil sacs. The fractured surface of dried ones is horny, relatively deeply colored or cracked.

Under the microscope, the cross-section shows a stone cell layer in the periderm, but usually none in the cortex. In the phloem rays and their tips are oil sacs with yellowish brown substances. The cambium ring is distinct. The outer vessels of the xylem are mostly arranged radially in 1 to 3 rows, without xylem fiber bundles in the surrounding areas. Inside, the vessels are closely spaced but form groups with nearby xylem fiber bundles, surrounding the large pith and radiating. The pith and medullary rays also have oil sacs and the parenchyma contains inulin crystals and small acicular crystals of calcium oxalate.

It has a characteristic odor and sweet taste, and viscosity when chewed.

Identification (1) Weigh 0.5 g of pulverized Atractylodes Rhizome White, add 5 mL of ethanol and perform warm extraction on a water bath for 2 minutes and filter. To 2 mL of the filtrate, add 0.5 mL of vanillin-hydrochloric acid TS and shake immediately to mix; a red to reddish purple color develops and persists.

(2) Weigh 0.5 g each of pulverized Atractylodes Rhizome White and Atractylodes rhizome white RMPM, add 2 mL of hexane each and filter the solution with sonication for 15 minutes. Use the filtrates as the test solution and the Atractylodes rhizome white RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Atractylodes rhizome white RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with petroleum ether as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS to the plate and heat at 105 °C; the several spots obtained from the test solution show the same color and R_f value as the spots from the Atractylodes rhizome white RMPM standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.7 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(vi) Captan: NMT 2 ppm.
(vii) Procymidone: NMT 0.1 ppm.

(3) ***Sulfur dioxide***—NMT 30 ppm.

(4) ***Atractylodes Rhizome***—Weigh 2 g of pulverized Atractylodes Rhizome White, add 5 mL of hexane, shake for 5 minutes, filter and use this filtrate as the test solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL of the test solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of hexane and acetone (7 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly 4-dimethylaminobenzaldehyde TS for spraying on the plate and heat at 100 ℃ for 5 minutes; no green to grayish green spot appears between an R_f value of 0.3 and 0.6.

Ash NMT 7.0%.

Acid-insoluble ash NMT 1.0%.

Essential oil content NLT 0.5 mL (50.0 g).

Packaging and storage Preserve in well-closed containers.

Belladonna Root
벨라돈나근

Belladonnae Radix

Belladonna Root is the root of *Atropa belladonna* Linné (Solanaceae).

Belladonna Root contains NLT 0.4% of total alkaloids [as hyoscyamine ($C_{17}H_{23}NO_3$: 289.37)], calculated on the dried basis.

Description Belladonna Root is a cylindrical root, sometimes cut transversely or longitudinally, with a length of 10 cm to 30 cm and a diameter of 5 mm to 40 mm. The outer surface occurs as grayish brown to grayish yellow. The periderm is often removed. Fractured surface occurs as pale yellow to pale yellowish brown and much powdery.
It occurs as almost odorless and has bitter taste.

Identification Weigh 2 g of pulverized Belladonna Root in a glass-stoppered centrifuge tube, add 30 mL of ammonia TS, sonicate for 5 minutes and centrifuge. Pipet the supernatant and transfer it into a separatory funnel, add 40 mL of ethyl acetate, shake, and collect the ethyl acetate layer. Add 3 g of anhydrous sodium sulfate, shake and filter after the solution becomes clear. Evaporate ethyl acetate in vacuum, dissolve the residue in 1 mL of ethanol and use this solution as the test solution. Separately, weigh 2 mg of atropine sulfate RS, dissolve it in 1 mL of ethanol and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of acetone, water and ammonia water (90 : 7 : 3) as the developing solvent to a distance of about 10 cm and dry the plate at 80 ℃ for 10 minutes. Spray evenly Dragendorff TS for spraying on the plate; one spot among the spots obtained from the test solution and a yellowish red spot from the standard solution show the same color and the same R_f value.

Purity (1) ***Foreign matter***—(i) Stem and crown: Belladonna Root contains stem and crown less than 10.0%.

(ii) Other foreign matter: The amount of foreign matter other than stems and crowns contained in Belladonna Root is less than 2.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

Ash NMT 6.0%.

Acid-insoluble ash NMT 4.0%.

Assay Dry the pulverized Belladonna Root at 60 ℃ for 8 hours, weigh accurately about 0.7 g of pulverized Belladonna Root, place it in a glass-stoppered centrifuge tube and moisten with 15 mL of ammonia TS. To this, add 25 mL of ether, stopper the centrifuge tube tightly, shake for 15 minutes, centrifuge and collect the ether layer. With the residue, repeat this operation twice with 25 mL of ether. Combine all extracts and evaporate the ether layer on a water bath. Dissolve the residue in 5 mL of the mobile phase, add 3.0 mL of the internal standard solution and add the mobile phase again to make exactly 25 mL. Filter this solution with a filter paper (NMT 0.8 μm in pore size), discard 2 mL of the first filtrate and use the subsequent filtrate as the test solution. Separately, weigh accurately about 25 mg of atropine sulfate RS (determine loss on drying in advance), dissolve it in the mobile phase to make 25 mL and use this solution as the standard stock solution. Pipet 5.0 mL of the standard stock solution, add 3.0 mL of the internal standard solution, add the mobile phase again to make exactly 25 mL and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and standard solution as directed under the Liquid Chromatography under the following conditions. Determine the peak area ratios, Q_T and Q_S, of hyoscyamine (atropine) to that of the internal standard for the test solution and the standard solution, respectively.

$$\text{Amount (mg) of hyoscyamine } (C_{17}H_{23}NO_3) = \text{Amount (mg) of atropine sulfate RS, calculated on the dried basis} \times \frac{Q_T}{Q_S} \times \frac{1}{5} \times 0.8551$$

Internal standard solution—A solution of brucine in the mobile phase (1 in 2500).

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 210 nm)

Column: Stainless steel tube with an inside diameter of about 4 mm and a length of 15 cm, packed with octadecylsilyl silica gel for liquid chromatography of 5 μm.

Mobile phase: Dissolve 6.8 g of potassium dihydrogen phosphate in 900 mL of water, add 10 mL of triethylamine, adjust the pH with phosphoric acid to 3.5, add water to make 1000 mL and mix this solution with acetonitrile (9 : 1).

Flow rate: Adjust the flow rate so that the retention time of atropine is about 14 minutes.

Selection of column: Proceed with 10 μL of the standard solution according to the above conditions; atropine and the internal standard are eluted in this order with the resolution being NLT 4.

Packaging and storage Preserve in well-closed containers.

Belladonna Extract
벨라돈나엑스

Belladonna Extract contains NLT 0.85% and NMT 1.05% of hyoscyamine ($C_{17}H_{23}NO_3$: 289.37).

Method of preparation Weigh 1000 g of a coarse powder of Belladonna Root, add 4 L of 35 vol% Ethanol, and perform cold extraction for 3 days. Press the mixture, add 2000 mL of 35 vol% Ethanol to the residue, and perform cold extraction again for 2 days. Combine all the extract, allow to stand for 2 days, and filter. Then, prepare the semiliquid extracts as directed under Extracts. An appropriate amount of Ethanol and Purified Water may be used instead of 35 vol% Ethanol.

Description Belladonna Extract occurs as a dark brown and has a characteristic odor and bitter taste.

Identification Add 30 mL of ammonia TS to 0.5 g of Belladonna Extract, shake to mix, transfer the mixture to a separatory funnel, then add 40 mL of ethyl acetate, and shake to mix. Collect the ethyl acetate layer, add 3 g of anhydrous sodium sulfate, shake to mix, and filter after the ethyl acetate solution becomes clear. Evaporate the filtrate to dryness in vacuum, dissolve the residue in 1 mL of ethanol, and use this solution as the test solution. Proceed as directed in the Identification under Belladonna Root.

Purity (1) ***Heavy metals***—Total heavy metals: NMT 30 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

Assay Weigh accurately about 0.4 g of Belladonna Extract, put in a stoppered centrifuge tube, add 15 mL of ammonia TS, and shake to mix. Add 25 mL of ether, stopper, shake for 15 minutes to mix, centrifuge, and take separately the ether layer. Extract the water layer twice, using 25 mL of ether each time. Combine the extracts, evaporate the ether on a water bath, dissolve the residue in 5 mL of the mobile phase, and add exactly 3.0 mL of the internal standard solution. Again, add the mobile phase to make exactly 25 mL, and proceed as directed in the Assay under Belladonna Root.

$$\text{Amount (mg) of hyoscyamine } (C_{17}H_{23}NO_3) = \text{Amount (mg) of atropine sulfate RS, calculated on the dried basis} \times \frac{Q_T}{Q_S} \times \frac{1}{5} \times 0.8551$$

Internal standard solution—A solution of brucine in the mobile phase (1 in 2500).

Packaging and storage Preserve in light-resistant, tight containers in a cold place.

Benzoin
안식향(安息香)

Benzoinum

Benzoin is the resin obtained from *Styrax benzoin* Dryander or *Styrax tonkinensis* Craib ex Hart. (Styracaceae).

Description Benzoin is the resin, a grayish brown to dark reddish brown block of variable size. The white to pale yellowish red granules are embedded in the broken fragments. The texture is firm and tender at ordinary temperature, but softens with heat. It has a characteristic odor and slightly pungent and acrid taste.

Identification (1) Heat a small fragment of Benzoin in a test tube; an irritating vapor develops and a crystalline sublimate is formed.
(2) Weigh 0.5 g of Benzoin, add 10 mL of ether to perform cold extraction. Take 1 mL of the solution on an evaporating dish and add 2 to 3 drops of sulfuric acid; a deep reddish brown to deep reddish purple color develops.

Purity ***Ethanol-insoluble matter***—Weigh 1 g of Benzoin, add 30 mL of ethanol, and boil the mixture on a water bath for 15 minutes after connecting with a reflux condenser. After cooling, collect the insoluble matter through a glass filter and wash 3 times with 5 mL each of ethanol. Dry the residue at 105 ℃ for 4 hours; the residue weighs NMT 0.3 g.

Ash NMT 2.0%.

Acid-insoluble ash NMT 1.0%.

Packaging and storage Preserve in well-closed containers.

Bitter Cardamon
익지(益智)

Alpiniae Oxyphyllae Fructus

Bitter Cardamon is the fruit of *Alpinia oxyphylla* Miquel (Zingiberaceae).

Description Bitter Cardamon is the fruit, spherical to elliptical, with both ends somewhat pointed, 1 cm to 2 cm in length and 7 mm to 10 mm in width. The external surface exhibits a brown to dark brown color, with numerous longitudinal, hump-like protruding lines. Pericarp is 0.3 mm to 0.5 mm in thickness and adheres closely to the seed mass, making it difficult to peel off. The inside of Bitter Cardamon is divided vertically into three loculi by thin membranes, each loculus containing 5 to 8 seeds adhering by the aril. The seed exhibits a brown to dark brown color, irregularly polygonal and about 3.5 mm in diameter, with a hard texture.
It has a characteristic odor and a slightly bitter taste.

Identification Weigh 10 μL of Bitter Cardamon essential oil, dissolve in 1 mL of anhydrous ethanol, and use this solution as the test solution. With this solution, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL of the test solution on the thin-layer chromatographic plate made of silica gel (with fluorescent agent) for thin layer chromatography. Next, develop the plate with a mixture of hexane and ethyl acetate (10 : 1) to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm); a black spot appears at the R_f value of about 0.3.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 10.0%.

Acid-insoluble ash NMT 2.5%.

Essential oil content NLT 0.4 mL (50.0 g).

Packaging and storage Preserve in well-closed containers.

Bupleurum Root
시호(柴胡)

Bupleuri Radix

Buplerum Root is the root of *Bupleurum falcatum* Linné or its varieties (Umbelliferae).

Buplerum Root contains NLT 0.3% of saikosaponin a ($C_{42}H_{68}O_{13}$: 780.98), calculated on the dried basis.

Description Bupleurum Root is the root, thin long cone or columnar form, simple or branched, 10 cm to 15 cm long and 5 mm to 15 mm in diameter. The upper part is thick and the lower part is thin. The crown sometimes has stem and hairy fibers of withered leaves remaining. The outer surface occurs as pale brown to brown, some parts with deep wrinkles. The texture can be easily broken and the fractured surface is somewhat fibrous.
Under the microscope, the cross-section shows a thickness of the cortex reaching 1/3 to 1/2 of the radius, and tangential and long-developed cracks in the cortex. The cortex is scattered by numerous intercellular schizogenic oil channels ranging from 15 μm to 35 μm in diameter. In the xylem, the vessels are arranged radially or in steps and the fiber groups are scattered. The medulla at the crown has the same oil channels as the cortex. The parenchyma cells contain starch granules and oil droplets.
It has a characteristic odor and a slightly bitter taste.

Identification Weigh 1 g of pulverized Bupleurum Root, add 20 mL of methanol, sonicate for 10 minutes and filter. Transfer the filtrate to a separatory funnel, wash with 20 mL of hexane, and evaporate the methanol extract to dryness. Dissolve the residue in 1 mL of methanol and use this solution as the test solution. Separately, weigh 1 mg each of saikosaponin a RS and saikosaponin d RS, dissolve each in 1 mL of methanol and use these solutions as standard solution (1) and standard solution (2), respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution, standard solution (1) and standard solution (2) on the thin layer plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of ethyl acetate, ethanol and water (8 : 2 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS on the plate and heat at 105 °C; two of the several spots obtained from the test solution show the same color and R_f values as the purple spots obtained from the standard solution (1) and the standard solution (2).

Purity (1) ***Foreign matter***—(i) Stem and leaves: Bupleurum Root contains less than 10.0% of the stem and leaves.

(ii) Other foreign matter: Bupleurum Root contains less than 1.0% of foreign matter other than the stems and leaves.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(vi) Pendimethalin: NMT 0.2 ppm.

(vii) Fosthiazate: NMT 0.02 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 11.0%.

Ash NMT 6.5%.

Acid-insoluble ash NMT 2.0%.

Assay Weigh accurately about 0.2 g of pulverized Bupleurum Root, add 50 mL of a solution of ammonium hydroxide in methanol (1 in 20), sonicate for 2 hours and filter. Add methanol to the filtrate to make exactly 50 mL. Pipet 30 mL of this solution exactly and evaporate the solvent. Add methanol to the residue to make exactly 5 mL and use this solution as the test solution. Separately, weigh accurately about 10 mg of saikosaponin a RS (previously dried in a silica gel desiccator for 24 hours), add methanol to make exactly 20 mL and use this solution as the standard solution. Perform the test with 5 μL each of the test solution and the standard solution as directed under Liquid chromatography according to the following conditions and determine the peak areas, A_T and A_S, of the test solution and the standard solution.

$$\text{Amount (mg) of saikosaponin a } (C_{42}H_{68}O_{13}) = \text{Amount (mg) of saikosaponin a RS} \times \frac{A_T}{A_S} \times \frac{5}{12}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 203 nm)

Column: A stainless steel column 4 to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to l0 μm in particle diameter).

Mobile phase: A mixture of water and acetonitrile (65 : 35)

Flow rate: 0.8 mL/min

Packaging and storage Preserve in well-closed containers.

Capsicum
고추(苦椒)

Capsici Fructus

Capsicum is the fruit of *Capsicum annuum* Linné or its varieties (Solanaceae).

Description Capsicum is an elongated, conical to fusiform fruit, often curved fruit, about 3 cm to 10 cm long and about 0.8 cm wide. The outer surface occurs as dark red to dark yellowish red and lustrous, and usually has a calyx and peduncle. The interior is hollow and divided into two loculi, each containing numerous seeds. The seeds are nearly circular and flat, pale yellowish red and about 5 mm in diameter.
It has a characteristic odor and a very pungent taste.

Identification Weigh 2.0 g of pulverized Capsicum, add 5 mL of ethanol, and warm on a water bath for 5 minutes. After cooling, centrifuge and use the supernatant as the test solution. Separately, weigh 1 mg of capsaicin RS, dissolve in 1 mL of ethanol,

and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on the plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of ether and methanol (19 : 1) as the developing solvent to a distance of about 12 cm, and air-dry the plate. Spray evenly 2,6-dibromo-N-chloro-1,4-benzoquinone monoimine TS on the plate and allow it to stand in ammonia gas; one of the spots obtained from the test solution shows the same color and the same R_f value as the spot from the standard solution.

Purity (1) ***Foreign matter***—Less than 1.0%.
(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(3) ***Residual pesticides***—Proceed as directed in "Capsicum (Dried)" in [Attachment 4] MRLs for Agricultural Products in the Public Notification "Standards and Specifications for Food" of the Ministry of Food and Drug Safety (MFDS).

Ash NMT 6.0%.

Acid-insoluble Ash NMT 1.2%.

Extract content ***Ether-soluble extract***—NLT 9.0%.

Packaging and storage Preserve in well-closed containers.

Capsicum Tincture
고추틴크

Method of preparation
100 g of capsicum in moderately fine cutting
An appropriate amount of ethanol

To make 1000 mL

Prepare as directed under Tinctures with the above ingredients.

Description Capsicum Tincture is yellowish red liquid and has extremely pungent taste.
Specific gravity d_{20}^{20} About 0.82.

Identification Proceed as directed in the Identification under Capsicum using Capsicum Tincture as the test solution. Spot 20 μL for the test solution.

Alcohol number NLT 9.7 (Method 2)

Purity (1) ***Heavy metals***—(i) Total heavy metals: NMT 30 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ- and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

Packaging and storage Preserve in light-resistant, tight containers.

Cardamon
소두구(小豆蔻)

Cardamomi Fructus
Cardamon is the ripe fruit of *Elettaria cardamomum* Maton (Zingiberaceae). The capsules are removed from the seeds before use.

Description Cardamon is the fruit, long ellipsoidal, 10 mm to 20 mm in length and 5 mm to 10 mm in diameter. The pericarp is thin, light and fibrous. The external surface occurs as a pale yellow color and has three blunt ridges and many longitudinal lines. At the upper end, a small protrusion is visible. The interior part is longitudinally divided into three loculi by thin membranes, each loculus containing 3 to 7 seeds joining lengthwise by the aril. The seed occurs as a dark brown to blackish brown color, ovoid to long ovoid or irregularly angular and 3 mm to 4 mm in length. The dorsal side is convex, and the ventral side is longitudinally grooved and coarse and small protrusion is presented.
It has a characteristic odor and a pungent and slightly bitter taste. The pericarp occurs as odorless and tasteless.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 6.0% (seed).

Acid-insoluble ash NMT 4.0% (seed).

Essential oil content NLT 1.0 mL (30.0 g, seed).

Packaging and storage Preserve in well-closed containers.

Cassia Seed
결명자(決明子)

Cassiae Semen
Cassia Seed is the ripe seed of *Cassia tora* Linné or *Cassia obtusifolia* Linné (Leguminosae).

Description (1) ***Cassia tora*—**Cassia Seed from *Cassia tora* is a short, cylindrical seed that is relatively small, 3 to 5 mm long and 2 to 3 mm wide. Both sides of the external ridge have a wide, pale yellowish brown band.
When cracked, it has a characteristic odor and taste.
(2) ***Cassia obtusifolia*—**Cassia Seed from *Cassia obtusifolia* is rectangular or short cylindrical seed with both ends sloping in parallel, 3 mm to 7 mm long and 2 mm to 4 mm wide. The external surface is greenish brown or dark brown in color, smooth and lustrous. One end is relatively flat and the other is slanted

and pointed. A crest is prominent at the back and belly. Each side of the crest has a linear, concave pattern with a relatively pale color, symmetrical on a slant. The texture is tough, making it difficult to crack. The seed coat is thin with two yellow cotyledons curved as S shape and overlapped.

Identification Weigh 0.1 g of pulverized Cassia Seed, previously dried in a desiccator (silica gel) for 48 hours, put them on a slide glass, and put a glass ring, 10 mm in both internal diameter and height on it. Then, cover it with moistened filter paper and heat gently the slide glass over a small flame. Take the filter paper when a yellow color has developed on the upper surface of it and place 1 drop of potassium hydroxide TS on the surface of the filter paper where a sublimate is present; a red color develops.

Purity (1) ***Foreign matter***— Less than 1.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): NMT 0.2 ppm.

(vi) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

(5) ***Mycotoxins***—Total aflatoxin (sum of aflatoxin B_1, B_2, G_1 and G_2): NMT 15.0 ppb (aflatoxin B_1 is NMT 10.0 ppb).

Ash NMT 5.0%.

Packaging and storage Preserve in well-closed containers.

Cattle Gallstone
우황(牛黃)

Bovis Calculus

Cattle Gallstone is a stone that forms in the gallbladder of bovine *Bos taurus* Linné var. *domesticus* Gmelin (Bovidae).

Cattle Gallstone contains NLT 20.0% of conjugated bilirubin ($C_{33}H_{36}N_4O_6$: 584.66), calculated on the dried basis.

Description Cattle Gallstone is a stone, often egg-shaped, almost spherical, triangular or square column. The size is not constant, and a few are cylindrical or broken and have a diameter of 0.6 cm to 4.5 cm. The outer surface occurs as red to yellowish brown, sometimes covered with a shiny black film that is called "Ogeumui." Some are coarse, bump-shaped protruding, sometimes with a cracked pattern. The body is light and the texture is fragile and easily peeled off by layers. The cut surface is golden with fine concentric lamellae, sometimes with a narrow white core.

It has a clear aroma and tastes slightly bitter, later sweet and cool. It breaks easily when chewed and does not stick to the teeth.

Identification (1) Weigh 0.1 g of pulverized Cattle Gallstone, add 10 mL of petroleum ether, and shake for 30 minutes to mix. Filter the mixture, and wash the residue with 10 mL of petroleum ether. Weigh 10 mg of the residue, add 3 mL of acetic anhydride, and shake for 1 to 2 minutes to mix. Then, add a mixture prepared by adding 2 drops of sulfuric acid to 0.5 mL of acetic anhydride; the solution turns yellowish red to dark red, then dark reddish purple, and finally dark reddish brown.

(2) Weigh 10 mg of Cattle Gallstone, add 10 mL of chloroform, shake well, and discard chloroform extracts. Add 1 mL of hydrochloric acid and 10 mL of chloroform to the mixture, and shake well to mix. Take separately the chloroform layer when it turns yellowish brown, add 5 mL of barium hydroxide TS, and shake to mix; a yellowish brown precipitate is formed.

Purity (1) ***Curcuma longa Rhizome*** —Weigh 0.1 g of pulverized Cattle Gallstone, add 50 mL of methanol, and warm the mixture after connecting with a reflux condenser on a water bath for 30 minutes. After cooling, filter and concentrate the mixture by evaporation to 1 mL. Use this solution as the test solution. Separately, weigh 1 mg of curcumin RS, dissolve in methanol to make 5 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of chloroform, ethyl acetate, water, and acetic acid (100) (100 : 30 : 3 : 2) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 365 nm); the test solution does not show yellow fluorescent spots in the same position as the spots obtained from the standard solution.

(2) ***Synthetic pigments***—Weigh 2 mg of pulverized Cattle Gallstone, and add 1 mL of dilute hydrochloric acid; the solution does not exhibit a purple color.

(3) ***Starch***—Weigh 5 mg of pulverized Cattle Gallstone, add 2 mL of water, and heat the mixture on a water bath for 5 minutes. After cooling, add 2 to 3 drops of iodine TS; the solution does not exhibit a bluish purple color.

(4) ***Sucrose***—Weigh 20 mg of pulverized Cattle Gallstone, add 10 mL of water, shake for 15 minutes, and filter. To 1 mL of the filtrate, add 2 mL of anthrone TS and shake to mix; the solution does not exhibit a deep bluish green to dark green color.

Ash NMT 10.0%.

Assay The test must be performed quickly, avoiding light as much as possible. Weigh accurately about 0.1 g of pulverized Cattle Gallstone, transfer it to a 300-mL flask, and add 10 mL of dilute hydrochloric acid (1 in 5) and 200 mL of chloroform. Heat the mixture after connecting with a reflux condenser on a water bath at 61 ± 2 °C for 90 minutes. After cooling, transfer the mixture to a separatory funnel. Wash the flask with a small volume of chloroform and add the washing to the separatory funnel. Allow the mixture to stand for 10 minutes and collect the chloroform layer. Extract the aqueous layer again with chloroform. Combine all of the chloroform layer, add 5 g of anhydrous sodium sulfate, shake to mix, and filter. Add chloroform to the filtrate to make exactly 200 mL, and use this solution as the test solution for total bilirubin. Separately, weigh accurately about 0.1 g of pulverized Cattle Gallstone, dissolve it in 200 mL of chloroform, filter, and use the filtrate as the test solution for free bilirubin. Separately, weigh accurately about 20 mg of bilirubin RS, dissolve it in chloroform to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas, A_T and A_S, of the test solution and the standard solution.

Amount (mg) of total bilirubin or free bilirubin
= Amount (mg) of bilirubin RS× $\frac{A_T}{A_S}$ ×2

Amount (mg) of conjugated bilirubin ($C_{33}H_{36}N_4O_6$)
= Amount (mg) of total bilirubin – amount (mg) of free bilirubin

Operating conditions

Detector: A visible spectrophotometer (wavelength: 436 nm)

Column: A stainless steel column about 4 mm to 6 mm in internal diameter and 15 cm to 30 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (particle diameter: 5 μm to 10 μm).

Column temperature: A constant temperature of about 25 ℃

Mobile phase: A mixture of methanol, water, and acetic acid (450 : 49 : 1)

Flow rate: Adjust the flow rate so that the retention time of bilirubin is about 10 minutes.

System suitability

System repeatability: Perform the test 6 times with 10 μL of the standard solution according to the above conditions; the relative standard deviation of the peak areas of bilirubin is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Cibot Rhizome
구척(狗脊)

Cibotii Rhizoma

Cibot Rhizome is the rhizome of *Cibotium barometz* J. Smith (Dicksoniaceae).

Description Cibot Rhizome is the rhizome, irregular, long-mass-shaped, 10 – 30 cm in length and 2 – 10 cm in diameter. The external surface occurs as a deep brown color, with remains of golden hairs. The upper part exhibits several reddish brown woody petioles, and the lower part exhibits remains of black fibrous roots. The texture is hard, making it difficult to break.
It occurs as almost odorless and has a plain and slightly astringent taste.

Identification (1) Weigh 1 g of pulverized Cibot Rhizome, add 10 mL of methanol, heat for 15 minutes on a water bath, and filter. Spot the filtrate on the filter paper and examine under ultraviolet light (365 nm); it exhibits bluish white fluorescence.

(2) Weigh 1 g of pulverized Cibot Rhizome, add 10 mL of water, heat for 15 minutes on a water bath, and filter. To 2 mL of the filtrate, drop 1% Iron(III) chloride solution; the color of the resulting solution changes to dark green.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 13.0%.

Ash NMT 2.5%.

Extract content **Dilute ethanol-soluble extract** NLT 22.0%.

Packaging and storage Preserve in well-closed containers.

Cimicifuga Rhizome
승마(升麻)

Cimicifugae Rhizoma

Cimicifuga Rhizome is the rhizome of *Cimicifuga heracleifolia* Komarov, *Cimicifuga simplex* Wormskjord, *Cimicifuga dahurica* Maximowicz or *Cimicifuga foetida* Linné (Ranunculaceae).

Description Cimicifuga Rhizome is the rhizome, irregular, long masses, often branched, knotted, 10 cm to 20 cm in length and 2 cm to 4 cm in diameter. The outer surface occurs as blackish brown to maroon, slightly rough and uneven. The upper surface has several empty circular holes and stalk scars. The inner wall of the holes has a distinct, slightly indented, reticulated pattern. The lower part has scars from rootlets. The body is light, firm and difficult to cut. The cut surface is uneven with open gaps, fibrous, yellowish green to pale yellowish white.
It occurs as almost odorless and has a bitter and slightly astringent taste.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.
(4) ***Rhizome of Astilbe species***—Under the microscope, powdered Cimicifuga Rhizome does not contain druse in the parenchyma.

Ash NMT 9.0%.

Acid-insoluble ash NMT 1.5%.

Extract content ***Dilute ethanol–soluble extract***—NLT 18.0%.

Packaging and storage Preserve in well-closed containers.

Cinnamon Bark
육계(肉桂)

Cinnamomi Cortex

Cinnamon Bark is the stem bark of *Cinnamomum cassia* J. Presl (Lauraceae), either intact or with a small portion of the pericarp removed.

Cinnamon Bark contains NLT 0.03% of cinnamic acid ($C_9H_8O_2$: 148.16), calculated on the dried basis.

Description Cinnamon Bark is a barrel-shaped or rolled-up stem bark that is 5 cm to 50 cm long, 15 mm to 50 mm in diameter, and 1 mm to 5 mm thick. The outer surface occurs as dark reddish brown, while the inner surface is reddish brown and smooth. The texture is easily broken and the fractured surface is slightly fibrous, reddish brown, and has a light brown, thin layer. Under the microscope, the cross-section shows a primary bark and a secondary bark separated by an almost continuous ring of stone cells. Nearly round fiber bundles are found in the outer region of the ring and the wall of each stone cell is often thickened in a U-shape. The secondary bark has no stone cells and has a small number of sclerenchymatous fibers that are coarsely scattered. The parenchyma tissue is interspersed with oil cells, mucilage cells, and cells with fine calcium oxalate needles, and the parenchyma cells contain starch granules.
It has a characteristic odor and a slightly sweet and bitter taste, which later becomes slightly mucilaginous and astringent.

Identification Weigh 2 g each of pulverized Cinnamon Bark and Cinnamon bark RMPM, add 10 mL of ether, shake for 3 minutes, and filter. Use the filtrate as the test solution and the Cinnamon bark RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Cinnamon bark RMPM standard solution on a thin-layer chromatographic plate made of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of petroleum ether and ethyl acetate (17 : 3) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 254 nm); the spots obtained from the test solution show the same color and R_f value as those obtained from the Cinnamon bark RMPM standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.7 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 15.5% (6 hours).

Ash NMT 5.0%.

Assay Weigh accurately about 1.0 g of Cinnamon Bark, add 50 mL of methanol, sonicate for 1 hour, and filter. Add methanol to make exactly 50 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of cinnamic acid RS (previously dried in a silica gel desiccator for NLT 12 hours), add methanol to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions and determine the peak areas, A_T and A_S, of the test solution and the standard solution.

$$\text{Amount (mg) of cinnamic acid } (C_9H_8O_2) = \text{Amount (mg) of cinnamic acid RS} \times \frac{A_T}{A_S} \times \frac{1}{2}$$

Operating conditions
Detector: An ultraviolet absorption photometer (wavelength: 280 nm)
Column: A stainless column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).
Column temperature: Ordinary temperature
Mobile phase: A mixture of water, acetonitrile, and acetic acid (100) (68 : 30 : 2).
Flow rate: 1.0 mL/min
System suitability
System repeatability: Perform the test 6 times with 10 μL of the standard solution according to the above conditions; the relative standard deviation of the peak areas of cinnamic acid is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Citrii Unshiu Immature Peel
청피(靑皮)

Citri Unshius Pericarpium Immaturus

Citrus Unshiu Immature Peel is the pericarp of unripe *Citrus unshiu* Markovich or *Citrus reticulata* Blanco (Rutaceae).

Description Citrii Unshiu Immature Peel is the pericarp of a fruit, irregularly shaped and about 2 mm in thickness. The external surface occurs as a grayish green to bluish green color, loose and wrinkled, with dented scars associated with oil sacs. The inside occurs as a white to grayish white color. The texture is light and brittle.
It has a characteristic odor and a bitter and slightly pungent taste.

Identification (1) Weigh 0.3 g of pulverized Citrii Unshiu Immature Peel, add 10 mL of methanol, heat after connecting with a reflux condenser for 20 minutes, and filter. To 1 ml of the filtrate, add a small amount of magnesium powder and 3 to 5 drops of hydrochloric acid; the resulting solution slowly exhibits a scarlet color.

(2) Evaporate 5 mL of the filtrate obtained from (1) to dryness, dissolve the residue in 1 mL of methanol, and use this solution as the test solution. Separately, weigh 0.3 g of Citrii unshiu immature peel RMPM, add 10 mL of methanol, heat after connecting with a reflux condenser for 20 minutes, and filter. Then, evaporate 5 mL of the filtrate to dryness, dissolve the residue in 1 mL of methanol, and use this solution as the Citrii unshiu immature peel RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the Citrii unshiu immature

peel RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of ethyl acetate, methanol and formic acid (960 : 100 : 7) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS on the plate and heat at 105 °C; the color and the R_f values of the several spots obtained from the test solution are the same as those of the spots obtained from the Citrii unshiu immature peel RMPM standard solution. Of these spots, a brown spot appears at the R_f value of about 0.2.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(vi) Methidathion: NMT 10 ppm.
(viii) Tetradifon: NMT 25 ppm.
(ix) Triazophos: NMT 5 ppm.
(x) Fenitrothion: NMT 10 ppm.
(x) Phenthoate: NMT 10 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 12.0%.

Ash NMT 5.0%.

Acid-insoluble ash NMT 1.0%.

Essential oil content NLT 0.2 mL (50.0 g).

Extract content ***Dilute ethanol-soluble extract***—NLT 12.0%.

Packaging and storage Preserve in well-closed containers.

Citrus Unshiu Peel
진피(陳皮)

Citri Unshius Pericarpium

Citrus Unshiu Peel is the pericarp of ripe *Citrus unshiu* Markovich or *Citrus reticulata* Blanco (Rutaceae).

Citrus Unshiu Peel contains NLT 4.0% of hesperidin ($C_{28}H_{34}O_{15}$: 610.56), calculated on the dried basis.

Description Citrus Unshiu Peel is the pericarp, irregular, plate-shaped and about 2 mm in thickness. The external surface occurs as a yellowish red to dark yellowish brown color, with numerous small dents associated with oil sacs. The inside occurs as a white to pale grayish brown color. The texture is light and brittle.
It has a characteristic odor and a bitter and slightly pungent taste.

Identification (1) Weigh 0.5 g of pulverized Citrus Unshiu Peel, add 10 mL of methanol, warm for 2 minutes on a water bath, and filter. To 5 mL of the filtrate, add 0.1 g of magnesium and 1 mL of hydrochloric acid and allow to stand; the resulting solution exhibits a reddish purple color.

(2) Weigh 0.5 g each of pulverized Citrus Unshiu Peel and Citrus unshiu peel RMPM, add 10 mL each of methanol, sonicate for 20 minutes to extract, filter, and use these filtrates as the test solution and the Citrus unshiu peel RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Citrus unshiu peel RMPM standard solution on the thin-layer chromatographic plate made of silica gel (with a fluorescent agent) for thin layer chromatography. Next, develop the plate with a mixture of ethyl acetate, methanol and water (100 : 17 : 3) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly aluminum chloride TS on the plate and examine under ultraviolet light (main wavelength: 365 nm); the color and the R_f values of the several spots obtained from the test solution are the same as those of the spots obtained from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(vi) Methoxychlor: NMT 1 ppm.
(vii) Methidathion: NMT 6 ppm.
(viii) Tetradifon: NMT 15 ppm.
(ix) Triazophos: NMT 2 ppm.
(x) Fenitrothion: NMT 10 ppm.
(xi) Phenthoate: NMT 6 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 13.0% (6 hours).

Ash NMT 4.0%.

Assay Weigh accurately about 0.5 g of pulverized Citrus Unshiu Peel, add 60 mL of methanol, heat after connecting with a reflux condenser for 2 hours, and filter. To the residue, add 30 mL of methanol and proceed in the same manner. Combine all the filtrates, add methanol to make exactly 100 mL, and use this solution as the test solution. Separately, weigh accurately about 20 mg of hesperidin RS, dissolve in methanol to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas, A_T and A_S, from the test solution and the standard solution, respectively.

$$\text{Amount (mg) of hesperidin } (C_{28}H_{34}O_{15}) = \text{Amount (mg) of hesperidin RS} \times \frac{A_T}{A_S}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 280 nm)

Column: A stainless steel column 4 to 6 mm in internal diameter and 15 to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 to 10 μm in particle diameter).

Column temperature: An ordinary temperature

Mobile phase: A mixture of water and methanol (60 : 40).

Flow rate: 1.0 mL/min

System suitability

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak areas of hesperidin is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Clove
정향(丁香)

Syzygii Flos

Clove is the flowering bud of *Syzygium aromaticum* Merrill et Perry (Myrtaceae).

Description Clove is the bud, slightly trimmed club-shaped, 1 to 2 cm in length. The corolla is globose, 0.3 to 0.5 cm in diameter. There are 4 petals, wrapped in an overlapping bottle shape, exhibiting a chestnut to yellowish brown color. Inside the petals are the stamen and the style. Numerous fine, yellow, granular pollens are visible on breaking by rubbing. The calyx tube is cylindrical, slightly flat, sometimes slightly curved, 0.7 to 1.4 cm in length and 0.3 to 0.6 cm in diameter. The external surface occurs as a reddish brown or chestnut color. The calyx of the upper part divides into 4 and the lobe is triangular. The texture is solid and highly oily.

Under a microscope, the transverse section of Clove reveals long ovoid oil sacs, irregularly arranged in the outer surrounding surface, and two-layered vascular bundles surrounded by the collenchyma in the inner surface. There are bast fibers in the phloem. Inside the vascular bundles, the spongy tissue with developed aerenchyma is present. The parenchyma cells surrounded by vascular bundles contain aggregate crystals of calcium oxalates and oil droplets of the essential oil.

It has a strong, characteristic odor and a pungent taste, followed by a slight numbing sensation on the tongue.

Identification (1) To 0.1 mL of a mixture of the essential oil, obtained in the Essential oil content, and xylene, add 2 mL of ethanol, shake to mix, and add 1 to 2 drops of Iron(III) chloride TS; the resulting solution exhibits a green to blue color.

(2) Weigh 0.5 g of pulverized Clove, add 5 mL of ethanol, shake to mix, allow to stand for 30 minutes, filter, and use the filtrate as the test solution. Separately, weigh 1 mg of eugenol RS, dissolve in 1 mL of ethanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate using a mixture of petroleum ether and ethyl acetate (9 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly vanillin-sulfuric acid TS on the plate and heat the plate at 105 ℃ for 10 minutes; the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

Purity (1) ***Foreign matter***—(i) Stem: The amount of stems contained in Clove is less than 5.0%.

(ii) Other foreign matters: The amount of foreign matters other than stems is less than 1.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 7.0%.

Essential oil content NLT 1.6 mL (10.0 g).

Packaging and storage Preserve in well-closed containers.

Cnidium Rhizome
천궁(川芎)

Cnidii Rhizoma

Cnidium Rhizome is the rhizome of *Cnidium officinale* Makino or *Ligusticum chuanxiong* Hort. (Umbelliferae). It is used as it is or after being blanched in boiling water.

Description Cnidium Rhizome is the rhizome in the shape of irregular knotted masses, 5 to 10 cm in length and 3 to 5 cm in diameter. The external surface occurs as a grayish brown to dark brown color and is coarse and wrinkled, with numerous parallelly protruding nodal rings. The top part is concave with nearly orbicular stem scars. There are many strumous root scars at the lower part and above the nodal rings.

Under a microscope, the transverse section of Cnidium Rhizome reveals the cork layer composed of about 10 rows of the flat cork cells. The cortex is narrow, scattered with root-trace vascular bundles, the cells are transversely long, and nearly orbicular oil sacs are abundant. The phloem is relatively broad, scattered with groups of sieve tubes. The cambium forms a ring. In the xylem, vessels are polygonal or nearly orbicular, mostly arranged in a single row or in a V-shape. The pith is relatively large, and the parenchyma is scattered with multiple oil sacs. The parenchyma cells contain starch grains and sometimes crystals of calcium oxalate in the shape of nearly orbicular masses or rosette crystals. Cnidium Rhizome from *Cnidium officinale* and Cnidium Rhizome from *Ligusticum chuanxiong* are generally similar.

It has a characteristic odor and a slightly bitter taste.

Identification Weigh about 1 g each of Cnidium Rhizome and Cnidium rhizome RMPM, add 10 mL each of diluted ethanol (7 in 10), sonicate for 60 minutes to extract, filter, and use the filtrates as the test solution and the Cnidium rhizome RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 2 μL each of the test solution and the Cnidium rhizome RMPM standard solution on the thin-layer chromatographic plate made of silica gel (with a fluorescent agent) for thin layer chromatography. Next, develop the plate with a mixture of hexane, ethyl acetate and methanol (10 : 2 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 365nm); the color and the R_f values of several spots obtained from the test solution are the same as those of the spots obtained from the Cnidium rhizome RMPM standard solution. Of these spots, the spot of ligustilide appears at the R_f

value of about 0.6.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Bifenthrin: NMT 0.5 ppm.
(v) Aldrin: NMT 0.01 ppm.
(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): NMT 0.2 ppm.
(viii) Endrin: NMT 0.01 ppm.
(viii) Chlorpyrifos: NMT 0.5 ppm.
(ix) Chlorfenapyr: NMT 2.0 ppm.
(x) Tebufenpyrad: NMT 0.1 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 6.0%.

Acid-insoluble ash NMT 1.0%.

Packaging and storage Preserve in well-closed containers.

Codonopsis Pilosula Root
당삼(黨參)

Codonopsis Pilosulae Radix

Codonopsis Pilosula Root is the root of *Codonopsis pilosula* (Franch.) Nannf, *Codonopsis pilosula* Nannf. var. *modesta* L. T. Shen, or *Codonopsis tangshen* Oliver (Campanulaceae).

Description (1) ***Codonopsis pilosula***—Pilosula Root of *Codonopsis pilosula* consists of the root, long cylindrical, slightly curved, 10 cm to 35 cm long and 4 mm to 20 mm in diameter. The outer surface occurs as yellowish brown to grayish brown. The crown has numerous warty, protruding stalk scars and buds, and the apex of each stalk scar is sunken and dotted. Below the crown are dense, annular transverse grooves that become sparser toward the bottom. These transverse grooves sometimes account for up to half of the total length. The cultured drugs have few horizontal patterns and no hairs. They are interspersed with vertical folds and long horizontal shell buds, and where the lateral roots fall off, there is usually a blackish-brown, sticky substance. The texture is slightly hard or tenacious. The fractured surface has clefts or a radiating pattern, the cortex is pale yellowish white to pale brown, and the xylem is pale yellow.
It has a characteristic odor and a slightly sweet taste.

(2) ***Codonopsis pilosula var. modesta***—Codonopsis Pilosula Root from *Codonopsis pilosula* var. *modesta* consists of the root, long cylindrical in shape, 10 cm to 35 cm in length and 5 mm to 25 mm in diameter. The outer surface is yellowish white to grayish yellow. Below the crown are dense transverse grooves that often extend more than half the length of the root. The fractured surface has more clefts and the cortex is grayish white or pale brown.

(3) ***Codonopsis tangshen***—Codonopsis Pilosula Root of *Codonopsis tangshen* consists of a long, cylindrical root 10 cm to 45 cm long and 5 mm to 20 mm in diameter. The outer surface is grayish yellow to yellowish brown, with distinct longitudinal folds. The texture is relatively soft and tenacious. The fractured surface has fewer clefts, and the cortex is yellowish white.

Identification (1) Weigh 0.5 g of pulverized Codonopsis Pilosula Root, add 10 mL of water, boil and allow to cool down. Pour this solution to a test tube and shake vigorously; the fine persistent foam rises.

(2) Weigh 1 g of pulverized Codonopsis Pilosula Root, add 25 mL of methanol, sonicate for 30 minutes, filter and use the filtrate as the test solution. Separately, dissolve 1 mg of lobetyolin RS in 5 mL of methanol and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of *n*-butanol, acetic acid (100), and water (14 : 2 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS on the plate and heat it at 105 ℃. Examine the plate under ultraviolet light (main wavelength: 365 nm); one spot among the several spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): NMT 0.2 ppm.
(vi) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 16.0%.

Ash NMT 6.0%.

Acid-insoluble ash NMT 2.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 35.0%.

Packaging and storage Preserve in well-closed containers.

Coix Seed
의이인(薏苡仁)

Coicis Semen

Coix Seed is the ripe seed of *Coix lacryma-jobi* var. *mayuen* (Rom.Caill.) Stapf (Gramineae).

Description Coix Seed is the seed, ovoid to broad ovoid, about 6 mm in length and about 5 mm in width, with the slightly dented apex and base. The dorsal side is round and distended, while the ventral side is longitudinally and deeply furrowed in the center. The external surface occurs as a reddish brown color. After the seed coat is removed, the external surface exhibits an almost white color, with a powdery texture. Under a magnifying glass, the transverse section reveals the white endosperm on the dorsal

side and the pale yellow scutellum in the hollow on the ventral side.
It has a slight, characteristic odor and a slightly sweet taste. It adheres to the teeth on chewing.

Identification (1) Add iodine TS dropwise to a transverse section of Coix Seed; the endosperm occurs as a dark reddish brown color, and the scutellum occurs as a dark gray color.

(2) Place a small amount of Coix Seed on a slide glass, add a few drops of iodine TS, and examine under a microscope; nearly equi-diameter and obtuse polygonal simple starch grains, typically 10 μm to 15 μm in diameter, and complex starch grains occur as a reddish brown color, while small starch grains, coexisting with fatty oils and aleurone grains within the parenchyma cells, occur as a bluish purple color.

(3) Weigh 1 g of pulverized Coix Seed, add 10 mL of methanol, warm on a water bath for 10 minutes, and filter. Concentrate the filtrate until it becomes 2 mL and use this solution as the test solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL of the test solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of petroleum ether, ethyl acetate and acetic acid (10 : 3 : 0.1) to a distance of about 10 cm, and air-dry the plate. Expose the plate to iodide steam; a yellow spot appears at the R_f value of about 0.63.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—Proceed with Coix Seed as directed in 'Coix Seed' described in [Attachment 4] Tolerances for Pesticide Residues for Agricultural Products provided in the Public Notice "Standards and Specifications for Food" of the Ministry of Food and Drug Safety (MFDS).
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 14.0% (6 hours).

Ash NMT 3.0%.

Packaging and storage Preserve in well-closed containers.

Condurango
콘두란고

Condurango Cortex
Condurango is the bark of the trunk of *Marsdenia condurango* Reichenbach fil. (Asclepiadaceae).

Description Condurango is cylindrical or semicylindrical bark of stems, 4 cm to 15 cm in length and 1 mm to 6 mm in thickness. External surface occurs as grayish brown to dark brown, nearly smooth and with numerous lenticels, or more or less scaly and rough. Inner surface occurs as pale grayish brown and longitudinally striated. Fractured surface is longitudinal and fibrous on the outer side and generally granular on the inner side.
Under a microscope, a transverse section reveals a cork layer composed of several layers of epithelial cells. Primary cortex has numerous stone cell groups and secondary cortex adjoins the starch sheath consisting of a single layer and contains phloem fiber bundles scattered. Articulate latex tubes are scattered in both cortices. Parenchyma cells contain starch grains or druses of calcium oxalate.
It has a slight, characteristic odor and bitter taste.

Identification Weigh 1 g of pulverized Condurango, add 5 mL of water for cold extraction, filter, and heat the clear filtrate; the solution becomes turbid, but clear again upon cooling.

Purity (1) ***Foreign matter***—Condurango contains xylem and other foreign matter less than 2.0%.
(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm
(iv) Cadmium: NMT 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
Ash NMT 12.0%.

Packaging and storage Preserve in well-closed containers.

Condurango Fluid Extract
콘두란고유동엑스

Method of preparation Take moderately fine powder of Condurango, and prepare Condurango Fluid Extract as directed under the Fluid Extracts by using a mixture of purified water, ethanol and glycerin (5 : 3 : 2) as the first menstruum, and a mixture of purified water and ethanol (3 : 1) as the second menstruum.

Description Condurango Fluide Extract is a brown liquid, has a characteristic odor and a bitter taste.

Identification Mix 1 mL of Condurango Fluid Extract with 5 mL of water, filter, if necessary, and heat the clear solution; the solution is turbid, but becomes almost clear again upon cooling.

Purity (1) ***Heavy metals***—Total heavy metals: NMT 30 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

Packaging and storage Preserve in tight containers.

Coptis Rhizome
황련(黃連)

Coptidis Rhizoma
Coptis Rhizome is the rhizome of *Coptis japonica* Makino, *Coptis chinensis* Franch., *Coptis deltoidea* C.Y.Cheng et P.K.Hsiao or *Coptis teeta* Wallich, from which the roots have been removed.
Coptis Rhizome contains NLT 4.2% of berberine [as berberine

chloride ($C_{20}H_{18}ClNO_4$: 371.81)], calculated on the dried basis.

Description Coptis Rhizome is rhizome, irregular and cylindrical in shape, 2 cm to 4 cm in length, occasionally up to 10 cm, and 2 mm to 7 mm in diameter, which is slightly curved and often cracked. External surface occurs as grayish yellowish brown, with ring nodes and with numerous remains of rootlets. Remains of petioles occasionally are found at one end. Fractured surface is rather fibrous. Cork layer occurs as pale grayish brown, cortex and pith occur as yellowish brown to reddish yellowish brown, and xylem occurs as yellow to reddish yellow.
Under a microscope, a transverse section of Coptis Rhizome reveals a cork layer, composed of thin-walled cork cells. Parenchyma cell of the cortex usually exhibits groups of stone cells near the cork layer and yellow phloem fibers near the cambium. Xylem consists chiefly of vessels, tracheae and xylem fibers, and meullary ray is distinct. Pith is large. In pith, stone cells or stone cells with thick and lignified cells are sometimes recognized. Parenchyma cells contain minute starch grains.
It has a slight, characteristic odor, and very bitter, persistent taste, and colors the saliva yellow.

Identification (1) Weigh 0.5 g of pulverized Coptis Rhizome, add 10 mL of water, allow to stand for 10 minutes with occasional shaking, and filter. To 2 to 3 drops of the filtrate, add l mL of hydrochloric acid and l to 2 drops of hydrogen peroxide TS, and shake to mix; the solution exhibits a reddish purple color.

(2) Weigh 0.5 g of pulverized Coptis Rhizome and Coptis rhizome RMPM, add 20 mL of methanol each, shake for 2 minutes to mix, and filter. Use the filtrate as the test solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the Coptis rhizome RMPM standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of n-butanol, water and acetic acid (100) (7 : 2 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine this plate under ultraviolet light (main wavelength: 365 nm); the several spots obtained from the test solution show the same color and R_f value as the spots from the Coptis rhizome RMPM standard solution and of these, one spot appears a yellow to yellowish green, fluorescent spot at the R_f value of about 0.4.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 1.0 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 11.0% (180 °C, 6 hours).

Ash NMT 4.0%.

Acid-insoluble ash NMT 1.0%.

Assay Weigh accurately about 0.5 g of pulverized Coptis Rhizome, add 30 mL of a mixture of methanol and dilute hydrochloride acid (100 : 1), heat after connecting with a reflux condenser for 30 minutes, and filter. Repeat the above procedure twice with the residue, using 30 mL and 20 mL of a mixture of methanol and dilute hydrochloric acid (100 : 1). To the final residue, add 10 mL of methanol, shake well to mix, and filter. Combine all the filtrates, add methanol to make exactly 100 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of Berberine Chloride RS (previously dried in a silica gel desiccator for 24 hours), dis- solve in methanol to make exactly 100 mL and use this solution as the standard solution. Perform the test with 20 μL each of the test solution and the standard solution as directed under Liquid chromatography according to the following operating conditions and determine the peak areas, A_T and A_S, in the test solution and the standard solution.

Amount (mg) of berberine [berberine chloride ($C_{20}H_{18}ClNO_4$)]
= Amount (mg) of berberine chloride RS× $\frac{A_T}{A_S}$

Operating conditions
Detector: An ultraviolet absorption photometer (wavelength: 345 nm)
Column: A stainless column, 4 to 6 mm in internal diameter and 15 to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 to 10 μm in particle diameter).
Column temperature: A constant temperature of about 40 °C.
Mobile phase: Dissolve 3.4 g of potassium dihydrogen phosphate and 1.7 g of sodium lauryl sulfate in 1000 mL of a mixture of water and acetonitrile (1 : 1).
Flow rate: Adjust the flow rate so that the retention time of berberine is about 10 minutes.
System suitability
System performance: Dissolve 1 mg each of berberine RS and palmatine RS in methanol to make 10 mL. Proceed with 20 μL of this solution according to the above operating conditions; palmatine and berberine are eluted in this order with clearly dividing each peak.
System repeatability: Repeat the test 6 times with 20 μL of the standard solution according to the above conditions; the relative standard deviation of the peak area of berberine is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Cornus Fruit
산수유(山茱萸)

Corni Fructus
Cornus Fruit is the ripe fruit of *Cornus officinalis* Siebold et Zuccarini (Cornaceae) from which the seeds have been removed.
Cornus Fruit contains NLT 1.2% in total of loganin ($C_{17}H_{26}O_{10}$: 390.38) and morroniside ($C_{17}H_{26}O_{11}$: 406.38), calculated on the dried basis.

Description Cornus Fruit is the fruit without seeds, irregular pieces or sac-shaped, 15 mm 20 mm in length, and about 1 cm in width. The outer surface occurs as dark reddish purple to dark purple, lustrous and with coarse folds. A scar forms when the seed is removed. A calyx scar is present in the upper part and a pedicel scar at the base. The texture is soft.
It has a slight odor and sour and slightly sweet taste.

Identification Weigh 1 g each of pulverized Cornus Fruit and Cornus Fruit RMPM, add 10 mL each of ethanol, shake for 5 minutes to mix, filter and use these solutions as the test solution and the Cornus Fruit RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Cornus Fruit RMPM standard solution on a thin-layer chromatographic plate of silica gel for thin-layer chromatography. Develop the plate with a lower layer of a mixture of dichloromethane, methanol and water (60 : 35 : 15) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 254 nm); the spots obtained from the test solution show the same color and R_f value as the spot from the Cornus Fruit RMPM standard solution.

Purity (1) ***Foreign matter***—The amount of the pedicel and other foreign matter contained in Cornus Fruit is less than 2.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Methoxychlor: NMT 1 ppm.
(iv) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(v) Aldrin: NMT 0.01 ppm.
(vi) Endrin: NMT 0.01 ppm.
(vii) Myclobutanil: NMT 2.0 ppm.
(vii) Triforine: NMT 0.2 ppm.
(ix) Triflumizole: NMT 0.2 ppm.
(x) Hexaconazole: NMT 0.3 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 5.0%.

Assay Weigh accurately about 2 g of pulverized Cornus Fruit, add 100 mL of methanol, heat after connecting with a reflux condenser on a water bath for 2 hours, cool, and filter. To the residue, add 100 mL of methanol and proceed in the same manner. Combine all of the filtrates, concentrate under vacuum, add methanol to make exactly 50 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg each of loganin RS and morroniside RS (previously dried in a silica gel desiccator for 24 hours), dissolve each in methanol to make exactly 50 mL, and use these solutions as the standard solutions. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas, A_{Ta} and A_{Tb}, of loganin and morroniside in the test solution and the peak areas, A_{Sa} and A_{Sb}, of loganin and morroniside in the standard solution.

$$\text{Amount (mg) of loganin } (C_{17}H_{26}O_{10}) = \text{Amount (mg) of loganin RS} \times \frac{A_{Ta}}{A_{Sa}}$$

$$\text{Amount (mg) of morroniside } (C_{17}H_{26}O_{11}) = \text{Amount (mg) of morroniside RS} \times \frac{A_{Tb}}{A_{Sb}}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 240 nm)

Column: A stainless steel column, 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica ge1 (5 μm to 10 μm in particle diameter).

Mobile phase: A mixture of diluted acetic acid (0.1 in 100), acetonitrile and methanol (85 : 10 : 5)

Flow rate: 0.5 mL/min

System suitability

System performance: Proceed with 10 μL of the standard solution under the above operating conditions; morroniside and loganin are eluted in this order.

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution under the above operating conditions; the relative standard deviation of each peak area of morroniside and loganin is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Corydalis Tuber
현호색(玄胡索)

Corydalis Tuber

Corydalis Tuber is the tuber of *Corydalis ternata* Nakai or *Corydalis yanhusuo* W.T.Wang (Papaveraceae).

Corydalis Tuber from *Corydalis ternata* Nakai contains NLT 0.03% of coptisine ($C_{19}H_{14}NO_4$: 320.32) and NLT 0.02% of berberine [berberine chloride ($C_{20}H_{18}ClNO_4$: 371.81)], and Corydalis Tuber from *Corydalis yanhusuo* W.T.Wang contains NLT 0.03% of coptisine ($C_{19}H_{14}NO_4$: 320.32) and NLT 0.05% of tetrahydropalmatine ($C_{21}H_{25}NO_4$: 355.43), calculated on the dried basis.

Description (1) ***Corydalis ternata***—Corydalis Tuber from *Corydalis ternata* Nakai is mostly uneven flattened globose or polygonal tuber, 1 cm to 2 cm in diameter. Stem scar is observed at one end and with several warty protrusion at the base. External surface occurs as grayish yellow to grayish brown, texture is hard, and the fractured surface is smooth or granular and yellow to grayish yellowish brown.
It occurs as almost odorless and has a bitter taste.

(2) ***Corydalis yanhusuo***—Corydalis Tuber from *Corydalis yanhusuo* W.T.Wang is mostly uneven flattened globose tuber, 0.5 cm to 1.5 cm in diameter. External surface occurs as yellow to yellowish brown, the texture is hard and crunchy with irregular reticular wrinkles, and the cut surface is yellow and horny with a wax-like luster.
It occurs as almost odorless and has a bitter taste.

Identification Weigh 0.5 g of pulverized Corydalis Tuber, add 10 mL of dilute acetic acid, warm on a water bath for 3 minutes with occasional shaking, and filter. To 5 mL of the filtrate, add 3 drops of Mayer's TS; a yellowish brown, flocculent precipitate is formed.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

(3) ***Sulfur dioxide***—NMT 30 ppm.

(4) ***Mycotoxins***—Total aflatoxin (sum of aflatoxins B_1, B_2, G_1 and G_2): NMT 15.0 ppb (aflatoxin B_1 is NMT 10.0 ppb).

Loss on drying NMT 15.0%.

Ash NMT 3.0%.

Assay Weigh accurately about 1 g of pulverized Corydalis Tuber, add 10 mL of diluted methanol (7 in 10), sonicate for 20 minutes, filter, and use the filtrate as the test solution. Separately, weigh accurately about 10 mg each of coptisine RS, berberine chloride RS (previously dried in a silica gel desiccator for 24 hours) and tetrahydropalmatine RS and dissolve in diluted methanol (7 in 10) to make exactly 100 mL, respectively. Pipet 25 mL each of the coptisine solution and the berberine chloride solution, add diluted methanol (7 in 10) to make exactly 100 mL, and use this solution as the standard solution for Corydalis Tuber from *Corydalis ternata* Nakai. Pipet 25 mL of the coptisine solution and 50 mL of the tetrahydropalmatine solution, add diluted methanol (7 in 10) to make exactly 100 mL, and use this solution as the standard solution for Corydalis Tuber from *Corydalis yanhusuo* W.T.Wang. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions. Determine the peak areas, A_{Ta}, A_{Tb} and A_{Tc}, of coptisine, berberine chloride and tetrahydropalmatine in the test solution and A_{Sa}, A_{Sb} and A_{Sc}, of coptisine, berberine chloride and tetrahydropalmatine in the reference standards, respectively.

$$\text{Amount (mg) of coptisine } (C_{19}H_{14}NO_4) = \text{Amount (mg) of coptisine} \times \frac{A_{Ta}}{A_{Sa}} \times \frac{1}{40}$$

$$\text{Amount (mg) of berberine [berberine chloride } (C_{20}H_{18}ClNO_4)] = \text{Amount (mg) of berberine chloride RS} \times \frac{A_{Tb}}{A_{Sb}} \times \frac{1}{40}$$

$$\text{Amount (mg) of tetrahydropalmatine } (C_{21}H_{25}NO_4) = \text{Amount (mg) of tetrahydropalmatine RS} \times \frac{A_{Ta}}{A_{Sa}} \times \frac{1}{20}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 280 nm)

Column: A stainless steel column 4 – 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Column temperature: A constant temperature of about 25 °C.

Mobile phase: Control the mixing ratio of mobile phase A and mobile phase B stepwise or via the concentration gradient as follows.

Mobile phase A – Adjust the pH of a mixture of a solution obtained by dissolving 0.77 g of ammonium acetate in water to make 1000 mL and triethylamine (1000 : 1) to 6.0 with acetic acid.

Mobile phase B – Acetonitrile

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)
0	75	25
20	70	30
30	5	95
35	5	95
40	75	25

Flow rate: 1.0 mL/min

System suitability

System performance: Proceed with 10 μL of the standard solution according to the above operating conditions; coptisine, berberine and tetrahydropalmatine are eluted in this order with the resolution being NLT 3.

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution under the above operating conditions; the relative standard deviation of each peak area of coptisine, berberine and tetrahydropalmatine is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Croton Seed
파두(巴豆)

Crotonis Semen

Croton Seed is the seed of *Croton tiglium* Linné (Euphorbiaceae). From which the testa has been removed.

Description Croton Seed is the seed, slightly flattened elliptic, 12 mm to 15 mm in length and 7 mm to 9 mm in diameter. The external surface occurs as brown or grayish brown with the hilum and caruncle or small caruncle scar remaining at one end. The seed coat is thin and hard but brittle. The seed kernel occurs as yellowish white and very oily.
It has a slight odor and tastes soft and oily at first but strongly pungent later.

Identification Weigh 0.1 g of pulverized Croton Seed, add 10 mL of petroleum ether, sonicate for 20 minutes, filter, and use the filtrate as the test solution. With this solution, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL of the test solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of petroleum ether, ethyl acetate, and formic acid (10 : 1 : 0.5) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly sulfuric acid TS for spraying on the plate and heat at 105 °C; two yellowish brown spots appear at the R_f value of about 0.2.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Mycotoxins***—Total aflatoxin (sum of aflatoxins B_1, B_2, G_1 and G_2): NMT 15.0 ppb (However, aflatoxin B_1 is NMT 10.0 ppb).

Ash NMT 3.5%.

Extract content ***Dilute ethanol-soluble extract***—NLT 18.0%.

Packaging and storage Preserve in well-closed containers.

Curcuma Longa Rhizome
강황(薑黃)

Curcumae Longae Rhizoma

Curcuma Longa Rhizome is the rhizome, boiled or steamed thoroughly, and dried, of *Curcuma longa* Linné (Zingiberaceae). Curcuma Longa Rhizome contains NLT 3.2% of the sum of curcumin ($C_{21}H_{20}O_6$: 368.38), demethoxy curcumin ($C_{20}H_{18}O_5$: 338.35), and bis-demethoxy curcumin ($C_{19}H_{16}O_4$: 308.33), calculated on the dried basis.

Description Curcuma Longa Rhizome consists of primary rhizome, and often secondary rhizome. The rhizome is irregularly ovoid, cylindrical or fusiform, 2 cm to 5 cm long and 1 cm to 3 cm in diameter. The secondary rhizome is cylindrical with two blunt ends, slightly curved, about 1 cm in diameter and 2 cm to 6 cm in length, with ring nodes. The rhizome with cork layer occurs as yellowish red and lustrous. The rhizome without cork layer occurs as dark yellowish red and powder is stuck to the root. The texture is firm and difficult to break and the fractured surface occurs as yellowish brown to yellowish red horny and lustrous like wax. Under the magnifying glass, the endodermis ring is clearly visible and the parenchyma is scattered in a star-like shape.
Under the microscope, the outermost layer usually consists of the cortex with 4 to 10 layers or part of the cortex. A layer of endodermis separates the cortex and the central cylinder. The cortex and central cylinder consist of parenchyma tissue and scattered vascular bundles. Oil cells are scattered in the parenchyma tissue. Yellow substances, sand crystals or single calcium oxalate crystals, and gelatinized starch granules are found in the parenchyma cells.
It has a characteristic odor and bitter and irritating taste and makes the color of saliva yellow.

Identification (1) Weigh 0.5 g of pulverized Curcuma Longa Rhizoma, add 1 drop each of sulfuric acid and ethanol and mix them on the glass; a reddish purple color develops.

(2) Take a small amount of pulverized Curcuma Longa Rhizoma and add 1 drop each of ethanol and ether on the filter paper. Removed with powder when the filter paper is dried; the filter paper becomes yellow. Add 1 drop of saturated boric acid solution and heat; a reddish orange color develops. Then, add 1 drop of ammonia TS to the mixture; a dark blue-black color develops immediately, gradually changes into brown, and returns to reddish orange when it keeps long.

(3) Weigh 1 g of pulverized Curcuma Longa Rhizome and Curcuma longa rhizome RMPM, add 20 mL of methanol, sonicate for 1 hour. Then, filter the solution, vacuum-concentrate, dissolve it in 2 mL of methanol and use these solutions as the test solution and the curcuma longa rhizome RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot10 μL each of the test solution and the Curcuma longa rhizome RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography (with fluorescent agent). Next, develop the plate with a mixture of dichloromethane, methanol and formic acid (94 : 4 : 0.7) as the developing solvent to a distance of about 10 cm and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 365 nm); the spots obtained from the test solution show the same color and R_f value as the spots from the Curcuma longa rhizome RMPM standard solution and of these, the spots of bis-demethoxy curcumin, demethoxy curcumin, and curcumin appear at the R_f values of about 0.3, 0.4 and 0.6, respectively.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 16.0%.

Ash NMT 7.0%.

Acid-insoluble ash NMT 1.0%.

Assay Weigh accurately about 0.1 g of pulverized Curcuma Longa Rhizome, add 25 mL of diluted methanol (7 in 10), sonicate for 30 minutes and filter. To the residue, add 20 mL of diluted methanol (7 in 10) and proceed in the same manner. Combine all the filtrates, add diluted methanol (7 in 10) to make exactly 50 mL, and use this solution as the test solution. Separately, weigh accurately about 1 mg each of curcumin RS, demethoxy curcumin and bis-demethoxy curcumin RS (previously dried in a silica gel desiccator for 24 hours), add diluted methanol (7 in 10) to make exactly 50 mL, and use these solutions as the standard solutions. Perform the test with 10 μL each of the test solution and the standard solutions as directed under Liquid Chromatography according to the following conditions and determine the peak areas, A_{Ta}, A_{Tb}, and A_{Tc}, of curcumin, demethoxy curcumin, and bis-demethoxy curcumin in the test solution, and the peak areas A_{Sa}, A_{Sb}, and A_{Sc}, of curcumin, demethoxy curcumin and bis-demethoxy curcumin in the standard solutions.

$$\text{Amount (mg) of curcumin } (C_{21}H_{20}O_6) = \text{Amount (mg) of curcumin RS} \times \frac{A_{Ta}}{A_{Sa}}$$

$$\text{Amount (mg) of demethoxy curcumin } (C_{20}H_{18}O_5) \setminus \text{Amount (mg) of demethoxy curcumin RS} \times \frac{A_{Tb}}{A_{Sb}}$$

$$\text{Amount (mg) of bis-demethoxy curcumin } (C_{19}H_{16}O_4) = \text{Amount (mg) of bis-demethoxy curcumin RS} \times \frac{A_{Tc}}{A_{Sc}}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 420 nm)

Column: A stainless column, 4 to 6 mm in internal diameter and 15 to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 to 10 μm in particle diameter).

Column temperature: An ordinary temperature.

Mobile phase: A mixture of acetonitrile and diluted acetic acid (2 in 100) (65 : 35).

Flow rate: 1.0 mL/min

System suitability

System performance: Proceed with 10 μL of the standard solution according to the above conditions; bis-demethoxy curcumin, demethoxy curcumin, and curcumin are eluted in this order.

System repeatability: Repeat the test 6 times with 10 μL

each of the standard solution according to the above conditions; the relative standard deviation of the peak area of each of bisdemethoxy curcumin, demethoxy curcumin, and curcumin is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Curcuma Root
울금(鬱金)

Curcumae Radix

Curcuma Root is the tuberous root of *Curcuma wenyujin* Y. H. Chen et C. Ling, *Curcuma longa* Linné, *Curcuma kwangsiensis* S. G. Lee et C. F. Liang, or *Curcuma phaeocaulis* Val. (Zingiberaceae), which is intact dried or steamed and dried with the pericarp removed.

Description (1) ***Curcuma wenyujin***—Curcuma Root of *Curcuma wenyujin* is a tuberous root with a long round or oval shape, 35 mm to 70 mm long and 12 mm to 25 mm in diameter. The outer surface occurs as grayish brown, with uneven longitudinal folds. The color of protruded part of longitudinal folds occurs as relatively pale brown. The texture is hard and a cross section occurs as grayish brown with horn, and the endodermis ring pattern is distinct.
It has a characteristic order and a slightly bitter taste.

(2) ***Curcuma longa***—Curcuma Root of *Curcuma longa* is a spindle-shaped tuberous root with a length of 25 mm to 45 mm and a diameter of 10 mm to 15 mm, with one side thin and long. The outer surface occurs as grayish brown or grayish yellow with longitudinal folds. The cross-section occurs as orange and the outer surface occurs as yellowish brown to reddish brown.
It has a characteristic odor and very pungent taste.

(3) ***Curcuma kwangsiensis***—Curcuma Root of *Curcuma kwangsiensis* is a tuberous root, long conical or elongated circular, with a length of 20 mm to 65 mm and a diameter of 10 mm to 18 mm. The outer surface is shallow longitudinally or coarsely reticulate wrinkled.
It has a characteristic odor and a slightly pungent and bitter taste.

(4) ***Curcuma phaeocaulis***—Curcuma Root of *Curcuma phaeocaulis* is a tuberous root with a long oval shape, 15 mm to 35 mm in length, 10 mm to 12 mm in diameter, which is relatively thick and large.
It has a light characteristic odor and a plain taste.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.
(4) ***Mycotoxins***—Total aflatoxins (sum of aflatoxins B_1, B_2, G_1 and G_2): NMT 15.0 ppb (aflatoxin B1 is NMT 10.0 ppb).

Loss on drying NMT 16.0%.

Ash NMT 9.0%.

Packaging and storage Preserve in well-closed containers.

Cynomorium Herb
쇄양(鎖陽)

Cynomorii Herba

Cynomorium Herb (Herba Cynomorii) is the succulent stem of *Cynomorium songaricum* Ruprecht (Cynomoriaceae).

Description Cynomorium Herb is the stem, flat cylindrical, slightly curved, 5 cm to 20 cm long and 2 cm to 5 cm in diameter. The outer surface occurs as brown to maroon, coarse with distinct longitudinal furrows and irregular pits, sometimes with triangular blackish brown scales. The body is heavy and the texture is firm and barely broken. The fractured surface occurs as pale brown to maroon. The vascular bundles occur as yellow and appear triangular.
Cynomorium Herb has a light aroma and a slightly bitter and astringent taste.

Identification (1) Weigh 1 g of pulverized Cynomorium Herb, add 10 mL of water, and allow the mixture to stand for 30 minutes. Filter it and use the filtrate as the test solution. Separately, weigh 2 mg of L-proline RS, dissolve it in 1 mL of water, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Drop 10 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of propanol, water, acetic acid (100) and ethanol (4 : 2 : 1 : 1) as a developing solvent to a distance of about 10 cm, and air-dry the plate. Spray 2% ninhydrin-ethanol solution evenly on the plate and heat at 105 °C; the test solution shows a purple spot at an R_f value of about 0.5. In addition, one of the spots obtained from the test solution shows the same color and R_f value as the spot obtained from the standard solution.

(2) Weigh 1 g of pulverized Cynomorium Herb, add 20 mL of ethyl acetate, sonicate for 30 minutes, and filter. Concentrate the filtrate to 1 mL and use it as the test solution. Separately, weigh 0.5 mg of ursolic acid RS, dissolve it in 1 mL of methanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of hexane, ethyl acetate and formic acid (15 : 5 : 0.5) as a developing solvent to a distance of about 10 cm, and air-dry the plate. Spray sulfuric acid TS for spraying evenly on the plate and heat at 105 °C; one of the several spots obtained from the test solution shows the same color and R_f value as the spot obtained from the standard solution.

Purity (1) ***Foreign matter***—Cynomorium Herb contains less than 5.0% of the flower stalk and other foreign matter.
(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2

ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 12.0%.

Ash NMT 8.0%.

Extract content ***Dilute ethanol–soluble extract***—NLT 25.0%.

Packaging and storage Preserve in well-closed containers.

Cyperus Rhizome
향부자(香附子)

Cyperi Rhizoma

Cyperus Rhizome is the rhizome of *Cyperus rotundus* Linné (Cyperaceae), with rootlets removed.

Description Cyperus Rhizome is the rhizome, mainly fusiform, 15 mm to 35 mm in length and 5 mm to 10 mm in diameter. The external surface occurs as maroon to blackish brown, longitudinally wrinkled, sometimes with remains of the stem at the apex. It has 5 to 10 irregular ring nodes protruding and those not trimmed have scars of brown root hair and rootlets. Those with the root hair already removed are relatively smooth and ring nodes are not distinct. The texture is hard. The cut surface of those that have been steamed or boiled is yellowish brown or reddish brown and horny, and those that have been dried is white and distinctly powdery. The endodermis has distinct ring patterns and the central cylinder has a relatively intense color and is scattered with spot-like vascular bundles.
It has a characteristic aroma and slightly bitter taste.

Identification Weigh 1 g of pulverized Cyperus Rhizome and Cyperus Rhizome RMPM, add 5 mL of ether, respectively, shake for 1 hour to mix, filter, and evaporate the filtrate to dryness. Add 0.5 mL of methanol to each of the residue and use these solutions as the test solution and the standard solution of Cyperus Rhizome RMPM, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the standard solution of cyperus Rhizome RMPM on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography (with fluorescent indicator). Develop the plate with a mixture of hexane, ethyl acetate and formic acid (14 : 4 : 0.5) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (wavelength: 254 nm). The one spot among the several spots obtained from the test solution and one of the several spots from the standard solution of Cyperus Rhizome RMPM exhibit blackish brown color at the R_f value of about 0.7. Spray evenly dilute sulfuric acid TS on the plate and heat at 105 °C: the color and R_f values of the several spots from the test solution and the spots from the standard solution of Cyperus Rhizome RMPM are the same.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.7 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): NMT 0.2 ppm.
(vi) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 3.0%.

Acid-insoluble ash NMT 1.5%.

Essential oil content NLT 0.3 mL (50.0 g, 1 mL of silicon resin).

Packaging and storage Preserve in well-closed containers.

Dictamnus Root Bark
백선피(白鮮皮)

Dictamni Radicis Cortex

Dictamnus Root Bark is the root bark of *Dictamnus dasycarpus* Turczaininov (Rutaceae).

Description Dictamnus Root Bark is the root bark, cylindrically curled up, 5 cm to 15 cm, 1 cm to 2 cm in diameter and 2 mm to 5 mm thick. The outer surface occurs as grayish white or grayish yellow, longitudinally wrinkled, with root scars and often with small, protruding granular dots. The inner surface occurs as almost pale yellow. The texture is weak and easily broken and powdery. The fractured surface is uneven, somewhat lamellar, and when the outer layer is peeled off, numerous glistening small spots can be seen when exposed to light.
Under the microscope, the cross-section shows the remaining cork layers, which consist of about 3 to 10 rows of flat rectangular or elliptical cork cells. The cortex is narrow and consists of elliptical parenchyma cells that normally contain oil droplets. There is a large transverse intracellular space. The phloem is broad and occupies most of the entirety. The cells are contracted circularly or nearly so, are sparsely arranged, and have large lacerated gaps. The phloem rays are curved and consist of 1 to 3 rows of cells. The fibers are mostly scattered singly in the cortex and phloem, have a polygonal or rectangular shape, and very thick cell walls that have the shape of stone cells. The parenchyma cells contain small starch granules, calcium oxalate druse and oil droplets.
It has a characteristic odor and slightly bitter taste.

Identification (1) Weigh 0.5 g of pulverized Dictamnus Root Bark, add 10 mL of diluted acetic acid, heat it on a water bath for 3 minutes and filter it. Add 1 to 2 drops of Mayer's reagent to 5 mL of the filtrate; a yellowish white precipitate is produced.

(2) Weigh 1 g of pulverized Dictamnus Root Bark, add 50 mL of methanol, sonicate for 30 minutes, and filter it. Vacuum-concentrate the filtrate, dissolve it in 1 mL of methanol, and use this solution as the test solution. Separately, weigh 1 mg of fraxinellone RS, dissolve it in 1 mL of methanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer

chromatography. Next, develop the plate with a mixture of cyclohexane, toluene and ethyl acetate (2 : 2: 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly 5% vanillin-sulfuric acid TS to the plate and heat it at 105 °C; one of the several spots obtained from the test solution shows the same color and R_f value as the spot obtained from the standard solution.

Purity (1) ***Foreign matter***—Dictamnus Root Bark contains less than 5.0% of the xylem tissue and other foreign matter.
(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 12.0%.

Ash NMT 13.0%.

Acid-insoluble ash NMT 1.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 14.0%.

Packaging and storage Preserve in well-closed containers.

Dioscorea Rhizome
산약(山藥)

Dioscoreae Rhizoma

Dioscorea Rhizome is the dried rhizome or the dried steamed rhizome (rhizophore) of *Dioscorea batatas* Decaisne or *Dioscorea japonica* Thunberg (Dioscoreaceae), from which periderm has been removed.

Description Dioscorea Rhizome is the rhizome, usually cylindrical, sometimes cut transversely or longitudinally, 5 cm to 30 cm in length and 1 cm to 6 cm in diameter. The outer surface occurs as white to pale yellow, longitudinally grooved, longitudinally wrinkled with root hair scars or pale brown bark. The body is heavy and the texture is firm and difficult to cut. The cut surface occurs as white, powdery and sometimes horny.
Under the microscope, the cross-section shows almost circular mucous cells in the basic tissue, which contain calcium oxalate raphides. The vascular bundles are collateral, surrounded by a series of parenchymatous vascular bundles. The resin canals are distributed between the parenchyma cells and are fully filled with brown resinous substances. There are many starch granules. The starch granules are close to circular, elongated circular or triangular ovoid, with distinct streaks in the large ones.
It occurs as almost odorless, tastes weak and slightly sour and sticky when chewed.

Identification (1) Weigh 0.5 g of pulverized Dioscorea Rhizome, add 2 mL of chloroform, warm on a water bath for 2 to 3 minutes, and filter. To the filtrate, add 0.5 mL of acetic anhydride, shake well to mix, and add 0.5 mL of sulfuric acid carefully to form two layers; a very pale red to reddish brown color appears at the zone of contact and bluish green to green color at the upper layer.
(2) Weigh 0.5 g of pulverized Dioscorea Rhizome, add 10 mL of water, boil carefully for about 5 minutes, and filter. To the filtrate, add 1 drop of dilute iodine TS; a blue color develops.
(3) Weigh 1 g each of pulverized Dioscorea Rhizome and Dioscorea rhizome RMPM, add 50 mL of ethanol and 5 mL of acetic acid, and heat after connecting with a reflux condenser for 30 minutes. Filter and evaporate on a water bath to dryness. To each residue, add 2 mL of ethanol and use each solution as the test solution and the Dioscorea rhizome RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Dioscorea rhizome RMPM standard solution on a thin-layer chromatographic plate of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of cyclohexane and ethyl acetate (3 : 1) as a developing solvent to a distance of about 10 cm, and air-dry the plate. Spray dilute sulfuric acid TS evenly on the plate and heat at 105 ℃ for 10 minutes; the several spots obtained from the test solution show the same colors and the same R_f values as the spots obtained from the Dioscorea rhizome RMPM standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—Proceed with Dioscorea Rhizome as directed in "Dioscorea Rhizome (dried)" described in [Attachment 4] Tolerances for Pesticide Residues for Agricultural Products provided in the Public Notice "Standards and Specifications for Food" of the Ministry of Food and Drug Safety (MFDS).
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 14.0% (6 hours).

Ash NMT 6.0%.

Packaging and storage Preserve in well-closed containers.

Dolichos Seed
백편두(白扁豆)

Dolichoris Semen

Dolichos Seed is the ripe seed of *Dolichos lablab* Linné (Leguminosae).

Description Dolichos Seed is the seed, flat-ellipsoid or flat-ovoid, 8 mm to 12 mm long, 6 mm to 9 mm in diameter and 4 mm to 7 mm thick. The outer surface occurs as yellowish white, smooth, lustrous, with a white, prominent, eyebrow-shaped caruncle on the edge of one side. The texture is firm. The testa is thin and horny, with 2 plump, yellowish white cotyledons inside. It has a characteristic odor and tastes light and has a bean-like smell when chewed.

Identification Weigh 1 g of pulverized Dolichos Seed, add 10 mL of diluted ethanol (7 in 10) and warm on a water bath for 20 minutes. Cool, filter, concentrate the filtrate to make 2 mL and use as the test solution. Separately, dissolve 1 mg of arginine RS in 1 mL of diluted ethanol (7 in 10) and use this solution as the

standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of butanol, acetic acid and water (3 : 1 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly ninhydrin-ethanol solution (2 in 100) on the plate and heat at 105 °C; the test solution shows a purple spot at the R_f value of 0.3. One of the spots obtained from the test solution shows the same color and R_f value as the spot from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) **Residual pesticides**—Proceed with Dolichos Seed as directed in "Other Legumes" described in [Attachment 4] Tolerances for Pesticide Residues for Agricultural Products provided in the Public Notice "Standards and Specifications for Food" of the Ministry of Food and Drug Safety (MFDS).
(3) ***Sulfur dioxide***—NMT 30 ppm.
(4) ***Mycotoxins***—Total aflatoxin (sum of aflatoxins B_1, B_2, G_1 and G_2): NMT 15.0 ppb (aflatoxin B_1 is NMT 10.0 ppb).

Loss on drying NMT 12.0%.

Ash NMT 5.0%.

Acid-insoluble ash NMT 1.0%.

Extract content ***Water-soluble extract***—NLT 14.0%.

Packaging and storage Preserve in well-closed containers.

Drynaria Rhizome
골쇄보(骨碎補)

Drynariae Rhizoma

Drynaria Rhizome is the rhizome of *Drynaria foutunei* J. Smith (Polyodiaceae); it is used as it is or after removing the scaly pieces (ramenta) by burning them off.

Description Drynaria Rhizome is the rhizome, flattened cylindrical, mostly curved, branched, 5 – 15 cm in length, 10 – 15 mm in width and 2 – 5 mm in thickness. The external surface is densely covered with deep brown to dark brown small ramenta and is soft like flocky hair. The seared one occurs as reddish brown or dark brown, of which external surfaces of the upper part and both sides are evenly marked by depressed or raised circular leaf scars with occasional remnants of petioles or fibrous roots. The body is light and has a fragile texture, making it easy to break. The fractured surface occurs as reddish brown, exhibiting vascular bundles in the shape of yellow dots forming a ring pattern.
It has a slight, characteristic odor and a plain and slightly astringent taste.

Identification Weigh 0.5 g of pulverized Drynaria Rhizome, add 30 mL of methanol, sonicate to extract, and filter. Evaporate the filtrate to dryness, dissolve the residue in 1 mL of methanol, and use this solution as the test solution. Separately, dissolve 5 mg of naringin RS in l mL of methanol and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel (with a fluorescent agent) for thin layer chromatography. Next, develop the plate with the upper layer of the mixture of ethyl acetate, methanol, acetic acid and water (9:1:1:0.2) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly a solution of 1% aluminum chloride in ethanol on the plate and examine under ultraviolet light (main wavelength: 365 nm); the color and the *Rf* value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Packaging and storage Preserve in well-closed containers.

Ephedra Herb
마황(麻黃)

Ephedrae Herba

Ephedra Herb is the terrestrial stem of *Ephedra sinica* Stapf, *Ephedra intermedia* Schrenk et C. A. Meyer or *Ephedra equisetina* Bunge (Ephedraceae).

Ephedra Herb contains NLT 0.7% in total of alkaloids [as ephedrine ($C_{10}H_{15}NO$: 165.23) and as pseudoephedrine ($C_{10}H_{15}NO$: 165.23)], calculated on the dried basis.

Description Ephedra Herb is the terrestrial stem, thin cylindrical to long cylindrical, 5 cm to 25 cm in length, 1 mm to 2 mm in diameter and 3 cm to 5 cm in length between the nodes. The external surface occurs as a pale green to yellowish green color, with numerous longitudinal furrows in parallel and usually scaly leaves at the node. The leaves are 2 to 4 mm in length, pale brown to brown, usually being opposite at every node, adhering at the base to form a tubular sheath around the stem. Under a magnifying glass, the transverse section of the stem reveals a circular to elliptical shape, exhibiting a grayish brown to yellowish green color in the surrounding area and the center filled with a reddish purple substance or hollow. The fractured surface is fibrous on the marginal area and easy to split lengthwise.
It has a slight odor and an astringent and slightly bitter taste, which slightly paralyzes sensation on the tongue.

Identification Weigh 0.5 g each of pulverized Ephedra Herb and ephedra herb RMPM, add 10 mL each of methanol, shake for 2 minutes to mix, filter, and use the filtrates as the test solution and the Ephedra herb RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Ephedra herb RMPM standard solution on the thin-layer

chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate using a mixture of 1-butanol, water and acetic acid (100) (7 : 2 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly 2% ninhydrin-ethanol solution on the plate and heat at 105 °C for 5 minutes; the color and the *Rf* values of several spots obtained from the test solution are the same as those of the spots obtained from the Ephedra herb RMPM standard solution.

Purity (1) ***Foreign matter***—**Woody stem:** Ephedra Herb contains less than 5.0% of woody stems.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 11.0%.

Acid-insoluble ash NMT 2.0%.

Assay Weigh accurately about 5 g of pulverized Ephedra Herb, previously dried in a desiccator (silica gel) for 24 hours, transfer into a stoppered centrifuge tube, add 20 mL of diluted methanol (1 in 2), shake for 30 minutes to mix, centrifuge, and take the supernatant. Add 20 mL of diluted methanol (1 in 2) to the residue and repeat this procedure twice. Combine all the extracts, add diluted ethanol (1 in 2) to make exactly 100 mL, and use this solution as the test solution. Separately, weigh accurately about 50 mg of ephedrine hydrochloride RS, previously dried at 105 °C for 3 hours, and dissolve in diluted methanol (1 in 2) to make exactly 20 mL. Pipet 2 mL of this solution, add diluted methanol (1 in 2) to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 10 µL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and calculate the peak areas, A_{Ta} and A_{Tb}, of ephedrine and pseudoephedrine (of which relative retention time to ephedrine is about 0.9), respectively, from the test solution and the peak area, A_S, of ephedrine from the standard solution.

$$\begin{aligned}&\text{= Amount (mg) of total alkaloids}\\&\text{= Amount (mg) of ephedrine hydrochloride RS}\\&\times\frac{A_{Ta}+A_{Tb}}{A_S}\times\frac{1}{10}\times 0.819\end{aligned}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 210 nm)

Column: A stainless steel column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 µm to 10 µm in particle diameter).

Column temperature: A constant temperature of about 45 ℃

Mobile phase: A mixture of a solution of sodium lauryl sulfate (1 in 128), acetonitrile and phosphoric acid (640 : 360 : 1)

Flow rate: Adjust the flow rate so that the retention time of ephedrine is about 14 minutes.

System suitability

System performance: Weigh 1 mg of ephedrine hydrochloride RS and 4 mg of atropine sulfate RS, dissolve each in diluted methanol (1 in 2) to make 10 mL. Proceed with 10 µL of this solution under the above conditions; ephedrine and atropine are eluted in this order with clear separation of each peak.

System repeatability: Repeat the test 6 times with 10 µL each of the standard solution according to the above conditions; the relative standard deviation of the peak areas of ephedrine is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Epimedium Herb
음양곽(淫羊藿)

Epimedii Herba

Epimedium Herb is the aerial part of *Epimedium koreanum* Nakai, *Epimedium brevicornum* Maxim., *Epimedium pubescens* Maxim., *Epimedium wushanense* T. S. Ying or *Epimedium sagittatum* Maxim.(Berberidaceae).

Epimedium Herb contains NLT 0.3% of icariin ($C_{33}H_{40}O_{15}$: 676.66), calculated on the dried basis.

Description (1) ***Epimedium koreanum***—Epimedium Herb from *Epimedium koreanum* is the aerial part, composed of a stem and biternate compound leaves. The stem is thin and long, cylindrical and 20 cm to 30 cm in length. It has a longitudinal ridge and is easy to cut. The lower part is hollow in the middle, the upper-middle part has the white pith, and the external surface occurs as a brown or yellowish brown color. The leaflet is ovoid cordate, 4 cm to 9.5 cm in length and 3 cm to 8.5 cm in width. The apex is long-acute, the leaf base is cordate, the outer lobe of the leaflets on both sides is larger than the inner lobe, and the edge of the leaf occurs as a yellowish brown color with a thorn-like serra. The external surface is smooth and lustrous, exhibiting a deep green or yellowish green color. The rear surface occurs as a grayish green color, with the leaf veins protruding and sparsely pilose with soft yellowish brown hairs, and the hairs of the middle vein are relatively dense. The leaf is thin, and the texture is paper-like.

(2) ***Epimedium brevicornum***—Epimedium Herb from *Epimedium brevicornum* is composed of biternate compound leaves, similar to Epimedium Herb from *Epimedium koreanum*, but it is different from Epimedium Herb from *Epimedium koreanum* in that the texture of its leaf is close to leathery.

(3) ***Epimedium pubescens***—Epimedium Herb from *Epimedium pubescens* is composed of ternate compound leaves, and the base and the petiole of the leaf are densely covered with soft, ciliary hairs

(4) ***Epimedium wushanense***—Epimedium Herb from *Epimedium wushanense* is composed of ternate compound leaves, the leaflet is lanceolate to narrow lanceolate, 9 cm to 23 cm in length and 1.8 cm to 4.5 cm in width with the length being 5 to 6 times the width.

(5) ***Epimedium sagittatum***—Epimedium Herb from *Epimedium sagittatum* is composed of ternate compound leaves, and the leaflet is gradually acute towards the apex, 4 cm to 12 cm in length and 2.5 cm to 5 cm in width. The base of the leaflets on both sides is distinctly slanting to one side, and the outside is sagittate. The lower part of the leaf has a sparse growth of short hairs or is hairless, and the texture of the leaf is leathery.

It has a slight, characteristic odor and a slightly bitter taste.

Identification Weigh 2 g of pulverized Epimedium Herb, add 20 mL of methano1, shake for 15 minutes to mix, filter, and use the filtrate as the test solution. Separately, weigh 1 mg of icariin RS, dissolve in 1 mL of methanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solutions on the thin-layer chromatographic plate made of silica gel (with a fluorescent agent) for thin layer chromatography. Next, develop the plate with a mixture of ethyl acetate, ethanol and water (8 : 2 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 254 nm); the color and the R_f value of a spot among the several spots obtained from the test solution are the same as those of a dark violet spot obtained from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 12.0% (6 hours).

Ash NMT 8.0%.

Acid-insoluble ash NMT 1.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 15.0%.

Assay Weigh accurately about 1 g of pulverized Epimedium Herb, add 50 mL of diluted ethanol (7 in 10), heat after connecting with a reflux condenser for 1 hours, and filter. To the residue, add 40 mL of diluted ethanol (7 in 10) and proceed in the same manner. Combine all the filtrates, add diluted ethanol (7 in 10) to make exactly 100 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of icariin RS and dissolve in methanol to make exactly 100 mL. Pipet 5 mL of this solution, add diluted ethanol (7 in 10) to make exactly 20 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas, A_T and A_S, from the test solution and the standard solution, respectively.

$$\text{Amount (mg) of icariin } (C_{33}H_{40}O_{15}) = \text{Amount (mg) of icariin RS} \times \frac{A_T}{A_S} \times \frac{1}{4}$$

Operating conditions
Detector: An ultraviolet absorption photometer (wavelength: 214 nm)
Column: A stainless column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).
Mobile phase: A mixture of water and acetonitrile (72 : 28)
Flow rate: 1.0 mL/min
System suitability
System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak areas of icariin is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Eriobotrya Leaf
비파엽(枇杷葉)

Eriobotryae Folium
Eriobotrya Leaf is the leaf of *Eriobotrya japonica* Lindley (Rosaceae).

Description Eriobotrya Leaf is the leaf, oblong to obovate, 12 cm to 30 cm in length and 4 cm to 9 cm in width. The apex is pointed, the margin of leaf is sparsely serrate and entire near the base. The upper surface occurs as grayish brown, yellowish brown to greenish brown, lustrous, and smooth. The lower surface is pale colored and densely yellow tomentose. The petioles are very short and have yellowish brown tomentose. The texture is leathery and easily broken.
Under the microscope, the cross- section shows a thin cuticle, 4 to 5 layers of palisade tissue, and sparsely distributed large cells without chloroplasts. In the midrib, the vascular bundle appears laterally, bent into the xylem tissue forming an interrupted ring, and pericyclic fiber bundles arranged in the phloem. The upper part of the small vascular bundle consists of lignified tissue surrounded by a single calcium oxalate crystal. The solitary and druses appear in the mesophyll. The tomentum is unicellular, curved, 25 μm in thickness and about 1.5 mm in length.
It is almost odorless and has a slightly bitter taste.

Identification (1) Weigh 0.5 g of pulverized Eriobotrya Leaf, add 10 mL of water, shake for 2 to 3 minutes to mix, and filter. Add 0.5 mL of lead acetate TS to 2 mL of the filtrate; a pale yellowish brown precipitation is produced.
(2) Weigh 0.3 g of pulverized Eriobotrya Leaf, add 10 mL of methanol, and heat for 5 minutes on a water bath. After cooling, filter, and use the filtrate as the test solution. Separately, weigh 5 mg of ursolic acid RS, dissolve in 5 mL of methanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on a thin-layer chromatographic plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of hexane and ethyl acetate (1 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray sulfuric acid TS for spraying evenly on the plate with and heat at 105 ℃ for 10 minutes; one of the spots obtained from the test solution shows the same color and the R_f value as the spot obtained from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2

ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 15.0% (6 hours).

Ash NMT 10.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 15.0%.

Packaging and storage Preserve in well-closed containers.

Eucommia Bark
두충(杜仲)

Eucommiae Cortex

Eucommia Bark is the stem bark of *Eucommia ulmoides* Oliver (Eucommiaceae), from which the periderm is removed.

Eucommia Bark contains NLT 0.05% of pinoresinol diglucoside ($C_{32}H_{42}O_{16}$: 682.67), calculated on the dried basis.

Description Eucommia Bark is the stem bark, flattened, with two edges slightly curved inwards, varying in length and width and 3 mm to 7 mm in thickness. The external surface occurs as a pale brown or grayish brown color, sometimes with distinct wrinkles or longitudinal cracks or sometimes relatively thin. Those with coarse bark remaining have distinct lenticels. The inner surface is flat and smooth, exhibiting a brown or dark brown color, with fine longitudinal wrinkles. The inner texture is fragile and easy to break. When fractured, Eucommia Bark produces fine, dense, silvery white and very elastic resinous threads.

Under a microscope of cut surface, the outermost layer of Eucommia Bark reveals a thick rhytidome layer. Several layers of cork cells are regularly arranged inside the rhytidome layer. The cell walls of these cells are lignified, underneath of which the phelloderm is present. The phloem takes up most of the area with stone cell rings in a transverse arrangement of 5 to 7 rows, each ring made of 3 to 5 stone cells. The medullary rays consist of 2 to 3 rows of cells, located close to the cork layer, sometimes leaning to one side. Parenchyma cells, containing white gutta percha, can be observed near the pith. These parenchyma cells are particularly numerous inside the phloem.

It has a characteristic odor and a slightly bitter taste.

Identification Weigh 1 g each of pulverized Eucommia Bark and Eucommia bark RMPM, add 10 mL each of methanol, sonicate for 60 minutes to extract, filter, and use the filtrates as the test solution and the Eucommia bark RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Eucommia bark RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of chloroform, methanol and water (10 : 5 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS on the plate and heat at 105 ℃ for 10 minutes; the color and the R_f value of several spots obtained from the test solution are the same as those of the spots obtained from the Eucommia bark RMPM standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 10.0%.

Ash NMT 8.0%.

Acid-insoluble ash NMT 6.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 9.0%.

Assay Weigh accurately about 1.0 g of pulverized Eucommia Bark, add 20 mL of diluted ethanol (75 in 1.0), sonicate for 30 minutes to extract, filter, and use the filtrate as the test solution. Separately, weigh accurately about 10 mg of pinoresinol diglucoside RS and dissolve in a mixture of diluted ethanol (75 in 100) to make exactly 100 mL. Pipet 10 mL of this solution, add diluted ethanol (75 in 100) to make exactly 50 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and calculate the peak areas, A_T and A_S, from the test solution and the standard solution, respectively.

$$\text{Amount (mg) of pinoresinol diglucoside } (C_{32}H_{42}O_{16}) = \text{Amount (mg) of pinoresinol diglucoside} \times \frac{A_T}{A_S} \times \frac{1}{25}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 230 nm)

Column: A stainless steel column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Column temperature: A constant temperature of about 35 ℃

Mobile phase: Control the mobile phases A and B stepwise as directed in the following table or via the concentration gradient.

Mobile phase A: Diluted formic acid (1 in 1000)

Mobile phase B: Acetonitrile

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)
0	95	5
20	80	20
25	80	20
30	95	5

Flow rate: 1.0 mL/min

Packaging and storage Preserve in well-closed containers.

Euryale Seed
검인(芡仁)

Euryales Semen

Euryale Seed is the ripe seed of *Euryale ferox* Salisbury (Nymphaeaceae).

Description Euryale Seed is a seed, nearly spherical with a diameter of 5 mm to 8 mm. It sometimes breaks to form small masses. The intact Euryale Seed has an outer surface (endotesta) in the form of a thin membrane, closely adhered to the endosperm, and occurs as a reddish brown or dark purple color, sometimes with a white and irregular reticular mesh. One end is pale yellow and takes up about one third of the whole. It has a concave hilum mark and removing the inner seed coat reveals a distinct white color. The cut surface is white and powdery.
It occurs as odorless and has week taste.

Purity (1) ***Foreign matter***—The amount of husk and foreign matter contained in Euryale Seed is less than 2.0%.
(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm
(iv) Cadmium: NMT 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 14.0%.

Ash NMT 2.0%.

Packaging and storage Preserve in well-closed containers.

Evodia Fruit
오수유(吳茱萸)

Evodiae Fructus

Evodia Fruit is the fruit of *Evodia rutaecarpa* Bentham, *Evodia rutaecarpa* Bentham var. *officinalis* Huang or *Evodia rutaecarpa* Bentham var. *bodinieri* Huang (Rutaceae), harvested before it is fully ripe and open.

Evodia fruit contains NLT 0.1% in total of evodiamine ($C_{19}H_{21}N_3O$: 307.39) and rutaecarpine ($C_{18}H_{13}N_3O$: 287.32), calculated on the dried basis.

Description Evodia Fruit is the fruit, flattened spherical or slightly pentagonal-spherical flattened, and 2.5 mm to 5 mm in diameter. The external surface occurs as a dark brown to grayish brown color, with numerous oil sacs appearing as hollow pits. Sometimes, it has a fruit stalk, 2 mm to 5 mm in length and covered densely with hairs. The pericarp of a ripe fruit is split to reveal five loculi with each loculus containing seeds. The seed is lustrous, obovoid or globular in shape, and occurs as a brown to blackish brown or bluish black color.
Under a microscope, the transverse section of Evodia Fruit reveals hard hairs in the epidermis of the pericarp.
It has a characteristic odor and a pungent taste, followed by a lasting bitter taste.

Identification (1) Weigh 1 g of pulverized Evodia Fruit, add 20 mL of methanol, heat for 5 minutes on a water bath, cool, and filter. Evaporate the filtrate to dryness, add 3 mL of dilute acetic acid to the residue, warm for 2 minutes on a water bath, cool, and filter. Perform the following test using the filtrate as the test solution. To 0.2 mL of the test solution, add 0.8 mL of dilute acetic acid. To this solution, carefully add 2 mL of 4-dimethylaminobenzaldehyde TS and warm on a water bath; a purplish brown ring is formed at the zone of contact.

(2) Weigh 1 g of pulverized Evodia Fruit, add 20 mL of methanol, heat for 5 minutes on a water bath, cool, filter, and use the filtrate as the test solution. Separately, weigh 1 mg of evodiamine RS and 1 mg of rutaecarpine RS, dissolve each in 1 mL of methanol, and use these solutions as the standard solution (1) and the standard solution (2), respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 2 μL each of the test solution and the standard solutions on the thin-layer chromatographic plate made of silica gel (with a fluorescent agent) for thin layer chromatography. Next, develop the plate with a mixture of hexane and ethyl acetate (3 : 2) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly sulfuric acid TS for spray on the plate, heat at 105 ℃ for 10 minutes, and examine under ultraviolet light (main wavelength: 365 nm); the color and the R_f values of two spots among the several spots obtained from the test solution are the same as those of the spots from the standard solutions (1) and (2).

Purity (1) ***Foreign matter***—(i) Fruit stalk: The amount of fruit stalks contained in Evodia Fruit is less than 5.0%.
(ii) Other foreign matters: The amount of foreign matters other than fruit stalks contained in Evodia Fruit is less than 1.0%.
(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): NMT 0.2 ppm.
(vi) Endrin: NMT 0.01 ppm.
(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 15.0%.

Ash NMT 8.0%.

Assay Weigh accurately about 0.5 g of pulverized Evodia Fruit, add 25 mL of methanol, sonicate for 1 hour to extract, and filter. To the residue, add 20 mL of methanol and proceed in the same manner. Combine all the filtrates, add methanol to make exactly 50 mL, and use this solution as the test solution. Separately, weigh accurately 10 mg of evodiamine RS and 10 mg of rutaecarpine RS and add methanol to make exactly 50 mL. Pipet 2 mL each of these solutions, add methanol to make exactly 20 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution

as directed under the Liquid Chromatography according to the following operating conditions, and determine the peak areas, A_{Ta} and A_{Tb} from the test solution and the peak areas, A_{Sa} and A_{Sb} from the standard solution.

$$\text{Amount (mg) of evodiamine } (C_{19}H_{21}N_3O) = \text{Amount (mg) of evodiamine RS} \times \frac{A_{Ta}}{A_{Sa}} \times \frac{1}{10}$$

$$\text{Amount (mg) of rutaecarpine } (C_{18}H_{13}N_3O) = \text{Amount (mg) of rutaecarpine RS} \times \frac{A_{Tb}}{A_{Sb}} \times \frac{1}{10}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 254nm)

Column: A stainless steel column about 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Column temperature: An ordinary temperature

Mobile phase: A mixture of acetonitrile and water (1 : 1)

Flow rate: 1.0 mL/min

System suitability

Proceed with 10 μL of the standard solution under the above conditions; evodiamine and rutaecarpine are eluted in this order with clear separation of each peak.

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviations of each peak area of evodiamine and rutaecarpine are NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Farfarae Flower
관동화(款冬花)

Farfarae Flos

Farfarae Flower is the flower bud of *Tussilago farfara* Linné (Compositae).

Description Farfarae Flower is the flower bud, long, clavate, solitary or 2 to 3 accreted at the base, 10 – 25 mm in length and 5 – 10 mm in diameter. The upper end is relatively broader, and the lower part is gradually tapered or has short stems. On the external surface, numerous scaly bracts are attached. The external side of the bract occurs as a reddish purple to pale red color, and the internal side is densely covered with white flocky hairs. The body is light, showing white hairs inside when divided into two parts.

Under a microscope, a transverse section reveals spherical pollen grains, the rectangle epidermis of the calyx, and the thickened cell walls in the shape of strung beads in succession.

It is aromatic and has a slightly bitter and pungent taste.

Identification Weigh 1 g of pulverized Farfarae Flower, add 20 mL of ethanol, sonicate for 1 hour to extract, and filter. Vacuum-concentrate the filtrate, dissolve in 1 mL of ethyl acetate, and use this solution as the test solution. Separately, weigh 3 mg of tussilagone RS, dissolve in 1 mL of ethyl acetate, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel (with a fluorescent agent) for thin layer chromatography. Next, develop the plate using a mixture of petroleum ether and acetone (9 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly sulfuric acid TS for spray on the plate, heat the plate at 105 ℃, and examine under ultraviolet light (main wavelength: 365 nm); the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

Purity (1) ***Foreign matter***—Farfarae Flower contains less than 2.0% of foreign matters, including residual pedicels and shells.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 12.0%.

Ash NMT 8.0%.

Acid-insoluble ash NMT 2.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 18.0%.

Packaging and storage Preserve in well-closed containers.

Fennel
회향(茴香)

Foeniculi Fructus

Fennel is the ripe fruit of *Foeniculum vulgare* Miller (Umbelliferae).

Fennel contains NLT 1.4% of trans-anethole ($C_{10}H_{12}O$: 148.2), calculated on the dried basis.

Description Fennel is cylindrical cremocarp fruit, 3 mm to 8 mm in length, 1 mm to 3 mm in width and occasionally attached with 2 mm to 10 mm of a fruit stalk. The external surface occurs as grayish yellowish green to grayish yellow. Two mericarps are closely attached with each other, and with five longitudinal ridges. Under a microscope, a transverse section reveals ridges on the left and right sides of the edge are far protruded than others. One large oil canal is present between each ridge and two oil canals are on the side in which mericarp is attached to the fruit stalk.

It has a characteristic odor and sweet taste followed by bitterness.

Identification Weigh 0.5 g each of pulverized Fennel and Fennel RMPM, add 10 mL of hexane, shake to mix, allow to stand for 5 minutes, and filter. Use the filtrate as the test solution and the Fennel RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the fennel RMPM standard solution on a thin layer plate made of silica gel (with fluorescence indicator) for thin layer chromatography. Develop the plate with

a mixture of hexane and ethyl acetate (20 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine this plate under ultraviolet light (main wavelength: 254 nm); the several spots obtained from the test solution show the same color and R_f value as the spots from the Fennel RMPM standard solution.

Purity (1) ***Foreign matter***—(i) Fruit stalk: The amount of peduncles contained in Fennel is less than 3.0%.

(ii) Other foreign matter: The amount of foreign matter other than fruit stalks contained in Fennel is less than 1.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

(5) ***Estragole***—To a mixture of xylene and the essential oil obtained from the Essential oil content, add xylene to make 5.0 mL, and use this solution as the test solution. Separately, dissolve exactly 5 mg of estragole RS in 0.5 mL of xylene and use this solution as the standard solution. Perform the test with 1 μL each of the test solution and the standard solution as directed under the Gas Chromatography according to the following operating conditions. Determine the peak areas, A_T and A_S, in the test solution and the standard solution, respectively. Fennel contains NMT 10.0% of estragole ($C_{10}H_{12}O$: 148.20) in the essential oil.

$$\text{Amount (mg) of estragole } (C_{10}H_{12}O) = \text{Amount (mg) of estragole RS} \times \frac{A_T}{A_S} \times 10$$

Operating conditions

Detector: A flame ionization detector

Column: A capillary column about 0.3 mm in internal diameter and about 30 m to 60 m in length, coated with polyethylene glycol 20 M for gas chromatography.

Column temperature: Maintain the temperature at 60 °C for 4 minutes, raise from 60 °C up to 170 °C for 22 minutes, and then keep at 170 °C for 15 minutes.

Sample injection port temperature: A constant temperature of about 220 °C.

Detector temperature: A constant temperature of about 270 °C.

Carrier gas: Nitrogen

Flow rate: 0.4 mL/min

Ash NMT 10.0%.

Acid-insoluble ash NMT 1.5%.

Essential oil content NLT 0.7 mL (50.0 g).

Assay Weigh accurately about 0.5 g of pulverized Fennel, add 25 mL of ethyl acetate, sonicate and filter. To the filtrate, add ethyl acetate to make exactly 25 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of trans-anethole RS, add methanol to make exactly 25 mL, and use this solution as the standard solution. Perform the test with 2 μL each of the test solution and the standard solution as directed under Gas Chromatography according to the following operating conditions and determine the peak areas, A_T and A_S, in the test solution and the standard solution.

$$\text{Amount (mg) of trans-anethole } (C_{10}H_{12}O) = \text{Amount (mg) of trans-anethole RS} \times \frac{A_T}{A_S}$$

Operating conditions

Detector: A flame ionization detector

Column: A capillary column about 0.32 mm in internal diameter and 30 m to 60 m in length, coated with 5% phenyl methylsiloxane polymer.

Column temperature: Maintain at 145 °C for 20 minutes.

Sample injection port temperature: 230 °C

Detector temperature: 230°C

Carrier gas: Nitrogen

Flow rate: 1.0 mL/min

System suitability

System repeatability: Repeat the test 6 times with 2 μL of the standard solution according to the above operating conditions; the relative standard deviation of the peak areas of trans-anethole is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Forsythia Fruit
연교(連翹)

Forsythiae Fructus

Forsythia Fruit is the fruit of *Forsythia viridissima* Lindley or *Forsythia suspensa* Vahl (Oleaceae). The greenish fruit collected when it is beginning to ripen, steamed and dried is called Chunggyo. The fruit collected when it is ripened perfectly is called Nogyo.

Forsythia Fruit of *Forsythia virdissima* contains NLT 0.4% of arctigenin ($C_{21}H_{24}O_6$: 372.42) and Forsythia Fruit of *Forsythia suspensa* contains NLT 0.25% of forsythiaside ($C_{29}H_{36}O_{15}$: 624.59), calculated on the dried basis.

Description (1) ***Forsythia virdissima***—Forsythia Fruit of *Forsythia virdissima* is the fruit, approximately ovoid, slightly broad, flat, 10 mm to 17 mm long and 5 mm to 12 mm in diameter. The tip is very pointed and open like a bird's beak. The lower part is slightly round, with the peduncle preserved or fallen off. The outer surface is brown or green, slightly convex and unevenly wrinkled.

It has a slight, characteristic odor and bitter taste.

(2) ***Forsythia suspensa***—Forsythia Fruit of *Forsythia suspensa* is the fruit, long ovate to ovate, slightly flat, 15 mm to 25 mm in length and 5 mm to 13 mm in diameter. The outer surface has irregular longitudinal wrinkles and several small, protruding spots. Each side has a distinct longitudinal furrow. The apex is dull and pointed with a small fruit stalk at the base or already fallen off.

Identification (1) Weigh 0.2 g of pulverized Forsythia Fruit, add 2 mL of acetic anhydride, shake well, allow the mixture to stand for 2 minutes, and filter. To 1 mL of the filtrate, add gently 0.5 mL of sulfuric acid; a reddish purple color develops at the zone of contact.

(2) Weigh 1 g of pulverized Forsythia Fruit, add 10 mL of methanol, heat the mixture on a water bath for 2 minutes, and

filter. To 5 mL of the filtrate, add 0.1 g of magnesium and 1 mL of hydrochloric acid and allow the mixture to stand; a pale red to yellowish red color develops.

Purity (1) ***Foreign matter***—(i) Branchlets: Forsythia Fruit contains less than 5.0% of branchlets.

(ii) Other foreign matter: Forsythia Fruit contains less than 1.0% of foreign matter other than branchlets.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 11.0%.

Ash NMT 5.0%.

Extract content ***Dilute ethanol–soluble extract***—NLT 10.0%.

Assay (1) ***Arctigenin***—Weigh accurately about 0.1 g of pulverized Forsythia Fruit, add 50 mL of diluted methanol (1 in 2), sonicate for 30 minutes, and filter. Use the filtrate as the test solution. Separately, weigh accurately about 10 mg of arctigenin RS, dissolve it in methanol to make exactly 50 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under Liquid Chromatography according to the following conditions and determine the peak areas, A_T and A_S, of the test solution and the standard solution.

$$\text{Amount (mg) of arctigenin } (C_{21}H_{24}O_6) = \text{Amount (mg) of arctigenin RS} \times \frac{A_T}{A_S}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 280 nm)

Column: A stainless steel column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (particle diameter: 5 μm to 10 μm).

Column temperature: A constant temperature of about 25 °C

Mobile phase: A mixture of diluted acetic acid (100) (3 in 1000) and methanol (11 : 9)

Flow rate: 1.0 mL/min

(2) ***Forsythiaside***—Weigh accurately about 0.1 g of pulverized Forsythia Fruit, add 50 mL of diluted methanol (1 in 2), sonicate for 30 minutes, and filter. Use the filtrate as the test solution. Separately, weigh accurately about 10 mg of forsythiaside RS, dissolve it in methanol to make exactly 50 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under Liquid Chromatography according to the following conditions and determine the peak areas, A_T and A_S, of the test solution and the standard solution.

$$\text{Amount (mg) of forsythiaside } (C_{29}H_{36}O_{15}) = \text{Amount (mg) of forsythiaside RS} \times \frac{A_T}{A_S}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 280 nm)

Column: A stainless steel column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (particle diameter: 5 μm to 10 μm).

Column temperature: A constant temperature of about 25 °C

Mobile phase: A mixture of diluted acetic acid (100) (3 in 1000) and methanol (11 : 9)

Flow rate: 1.0 mL/min

Packaging and storage Preserve in well-closed containers.

Fritillaria Bulb
천패모(川貝母)

Fritillariae Cirrhosae Bulbus

Fritillaria Bulb is the scaly bulb of *Fritillaria cirrhosa* D. Don, *Fritillaria unibracteata* Hsiao et K. C. Hsia, *Fritillaria prezewalskii* Maximowicz or *Fritillaria delavayi* Franchet (Liliaceae). Fritillaria Bulb is divided into Songpae or Cheongpae by appearance.

Description (1) ***Songpae***—Songpae is the scaly bulb of Fritillaria Bulb, conical to nearly globose, 3 to 8 mm in height and 3 to 8 mm in diameter. Its external surface occurs as a white color. There are two scaly leaves on the outside, different in size, with the large scaly leaf closely wrapping the small one, and the unwrapped part is crescent-shaped. The apex is closed, with cylindrical and slightly tapering buds and 1 to 2 scales inside. The apex is round or slightly pointed, the base is flat, and there is a single scaly bulb disk, concave and grayish brown in color, at the central part. Occasionally, the remains of fibrous roots are visible. The texture is hard and fragile, and the transverse section is white and powdery.
It is nearly odorless and has a slightly bitter taste.

(2) ***Cheongpae***—Cheongpae is the scaly bulb of Fritillaria Bulb, slightly flattened oblate, 4 to 11 mm in height and 4 to 16 mm in diameter. There are two scaly leaves on the outside, almost similar in size, wrapping each other. The apex is open, and there are buds, 2 to 3 small scaly leaflets and the slender cylindrical remains of the stem inside.

Identification (1) Weigh 0.5 g of pulverized Fritillaria Bulb, add 5 mL of acetic anhydride, shake well for 5 minutes to mix, and filter. To 2 mL of the filtrate, carefully add 1 mL of sulfuric acid; a red color develops at the zone of contact. Allow it to stand for a while; the upper layer exhibits a green color.

(2) Weigh 0.5 g of pulverized Fritillaria Bulb, add 10 mL of dilute acetic acid, shake well for 5 minutes to mix, and filter. To 2 mL of the filtrate, carefully add 1 to 2 drops of Mayer's TS; the resulting solution becomes turbid. Allow it to stand for a while; a white precipitate is formed.

(3) Weigh 2 g of pulverized Fritillaria Bulb, add 20 mL of methanol, warm for 5 minutes on a water bath with occasional shaking, cool, and filter. Evaporate the filtrate to dryness, add 3

mL of dilute hydrochloride to the residue, warm for 2 minutes on a water bath, cool, and filter. Apply 1 drop of this filtrate on the filter paper, air-dry, and spray Dragendorff's TS on the filter paper and stand for a while; a yellowish red color appears.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

Loss on drying NMT 15.0%.

Ash NMT 5.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 9.0%.

Packaging and storage Preserve in well-closed containers.

Fritillaria Thunbergii Bulb
절패모(浙貝母)

Fritillariae Thunbergii Bulbus

Fritillaria Bulb is the scaly bulb of *Fritillaria thunbergii* Miquel (Liliaceae).

The large Fritillaria Thunbergii Bulb, from which the central bud has been removed, is called Daepae, the smaller one, from which the central bud is not removed, is called Jupae. The Fritillaria Thunbergii Bulb, from which the central bud has been removed and then cut into thick slices is called Jeolpaepyeon.

Description (1) ***Daepae***—Daepae is the scaly bulb, and the single scaly leaf on the outer layer of the scaly bulb is close to a crescent shape, 1 to 2 cm in height and 20 to 35 mm in diameter. The external surface occurs as a milky white to pale yellow color, while the inner surface occurs as a white or pale brown color, covered with white powder. Texture is hard, yet brittle. The fractured surface occurs as a white to yellowish white color and is very powdery.
It has a characteristic odor and a slightly bitter taste.

(2) ***Jupae***—Jupae is the scaly bulb, round oblate, 10 to 15 mm in height and 10 to 25 mm in diameter. The external surface occurs as a milky white color, there are 2 plump and fleshy scaly leaves on the outer layer, usually in reniform, overlapping with each other, and small stem remains with 2 to 3 dried, shrunken, small scaly leaflets present inside.
It has a characteristic odor and a slightly bitter taste.

(3) ***Jeolpaepyeon***—Jeolpaepyeon is the scaly bulb, from which the single scaly leaf on the outer layer has been cut, elliptical or close to circular and 1 to 2 cm in diameter, with the external surface of the edge exhibiting a pale yellow color. The texture is fragile and easy to cut. The fractured surface occurs as a powdery white color and is rich in powder.
It has a characteristic odor and a slightly bitter taste.

Identification (1) Weigh 0.5 g of Fritillaria Thunbergii Bulb, add 5 mL of acetic anhydride, shake well for 5 minutes to mix, and filter. To 2 mL of the filtrate, carefully add 1 mL of sulfuric acid; a red color develops at the zone of contact. Allow it to stand for a while; the upper layer exhibits a green color.

(2) Weigh 5 g of pulverized Fritillaria Thunbergii Bulb, add 5 mL of ammonia water and 20 mL of dichloromethane, allow to stand for 24 hours, and filter. Vacuum-concentrate the filtrate, dissolve in 1 mL of methanol, and use this solution as the test solution. Separately, dissolve 1 mg of verticine (peimine) RS in 1 mL of dichloromethane and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of n-butanol, acetic acid (100) and water (6 : 2 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly Dragendorff's TS on the plate and heat at 105 °C; the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): NMT 0.2 ppm.
(vi) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 15.0%.

Ash NMT 5.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 9.0%.

Packaging and storage Preserve in well-closed containers.

Gambir
아선약(阿仙藥)

Gambir is the dried aqueous extract obtained from the leaves and young twigs of *Uncaria gambir* Roxburgh (Rubiaceae).

Description Gambir is the aqueous extract obtained from the leaves and young twigs, which are in irregular masses. The outer surface occurs as brown to dark brown and the interior occurs as pale brown. The texture is brittle and easily broken.
It has a slight characteristic odor and an extremely astringent and bitter taste.

Identification (1) Weigh 0.2 g of pulverized Gambir, add 10 mL of water, heat on a water bath for 5 minutes with occasional shaking and filter. After cooling, add 2 to 3 drops of gelatin TS to the filtrate; the solution becomes white and turbid or a white precipitate is formed.

(2) Weigh 0.1 g of pulverized Gambir, dissolve in 20 mL

of dilute ethanol, shake to mix for 2 minutes, and filter. Mix 1 mL of the filtrate with 9 mL of dilute ethanol. To 1 mL of the resulting mixture, add 1 mL of vanillin-hydrochloric acid TS; the solution shows a pale red to reddish brown color.

Ash NMT 6.0%.

Acid-insoluble ash NMT 1.5%.

Extract content ***Dilute ethanol–soluble extract***—NLT 70.0%.

Packaging and storage Preserve in well-closed containers.

Gamisoyosan Extract Granules
가미소요산엑스 과립

Gamisoyosan Extract Granules contains NLT 3.3 mg of total paeoniflorin ($C_{23}H_{28}O_{11}$: 480.46) in Peony root and Moutan root bark, 2.6 mg of glycyrrhizic acid ($C_{42}H_{62}O_{16}$: 822.93) in licorice and 8.0 mg of geniposide ($C_{17}H_{24}O_{10}$: 388.37) in gardenia fruit for a dose (one sachet).

Method of preparation For a dose (one sachet)

Angelica gigas root, Atractylodes rhizome white, Poria, Bupleurum root, Peony root	1.00 g
Licorice, Moutan root bark, Gardenia fruit	0.67 g
Ginger, Mentha herb	0.33 g

Pulverize the above crude drugs to coarse powder, weigh each crude drugs, put them into the extractor, add eight- to ten-fold volume of water, extract for 2 to 3 hours at 80 – 100 ℃, and filter. Vacuum-concentrate the filtrate under 60℃ until it becomes 1.64 – 2.45 g of semiliquid extract or concentrate in a suitable method until it becomes 0.91 – 1.36 g of dry extract. Prepare Gamisoyosan Extract Granules as directed under Granules.

Identification (1) ***Angelica gigas root***—Pulverize Gamisoyosan Extract Granules, weigh an amount, equivalent to 1 g of Angelica gigas root, add 10 mL of water, and shake for 5 minutes to mix. Then, add 100 mL of methanol, connect with a reflux condenser, heat for 1 hour, and filter. Vacuum-concentrate the filtrate until it becomes 10 mL and use this solution as the test solution. Separately, weigh 1 g of pulverized Angelica gigas root, add 100 mL of methanol, connect with a reflux condenser, heat for 1 hour, and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of toluene, ether, water and acetic acid (500 : 500 : 5 : 2) as the developing solution to a distance of about 10 cm, and air-dry the plate. Spray evenly vanillin-sulfuric acid TS on the plate; the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

(2) ***Atractylodes rhizome White***—Pulverize Gamisoyosan Extract Granules, weigh an amount, equivalent to 1 g of Atractylodes rhizome white, add 10 mL of water, and shake for 5 minutes to mix. Then, add 100 mL of methanol, connect with a reflux condenser, heat for 1 hour, and filter. Vacuum-concentrate the filtrate until it becomes 10 mL and use this solution as the test solution. Separately, weigh 1 g of pulverized Atractylodes rhizome white, add 100 mL of methanol, connect with a reflux condenser, heat for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for Thin Layer Chromatography. Next, develop the plate using a mixture of hexane and acetone (7 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly the solution, prepared by dissolving 5 g of 4-dimethylaminobenzaldehyde in 100 mL of dilute sulfuric acid, on the plate and heat the plate at 105 ℃ for 10 minutes; the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

(3) ***Poria***—Pulverize Gamisoyosan Extract Granules, weigh an amount, equivalent to 1 g of Poria, add 10 mL of water, and shake for 5 minutes to mix. Then, add 100 mL of methanol, connect with a reflux condenser, heat for 1 hour, and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the test solution. Separately, weigh 1 g of Poria powder, add 100 mL of methanol, connect with a reflux condenser, heat for 1 hour, and filter. Vacuum-concentrate the filtrate until it becomes 10 mL and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel (with a fluorescent agent) for thin layer chromatography. Next, develop the plate using a mixture of hexane and acetone (7 : 3) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly *p*-anisaldehyde-sulfuric acid TS on the plate and heat the plate at 105 ℃ for 10 minutes. Examine under ultraviolet light (main wavelength: 254 nm); the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

(4) ***Bupleurum root***—Pulverize Gamisoyosan Extract Granules, weigh an amount, equivalent to 1 g of Bupleurum root, add 10 mL of water, and shake for 5 minutes to mix. Then, add 100 mL of methanol, connect with a reflux condenser, heat for 1 hour, and filter. Vacuum-concentrate the filtrate until it becomes 10 mL and use this solution as the test solution. Separately, weigh 1 g of Bupleurum root RMPM powder, add 100 mL of methanol, connect with a reflux condenser, heat for 1 hour, and filter. Vacuum-concentrate the filtrate until it becomes 10 mL and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of chloroform, methanol and water (30 : 10 : 1) as the developing solution to a distance of about 10 cm, and air-dry the plate. Spray evenly sulfuric acid TS for spray on the plate and heat the plate at 105 ℃ for 10 minutes; the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

(5) ***Ginger***—Pulverize Gamisoyosan Extract Granules, weigh an amount, equivalent to 1 g of ginger, add 10 mL of water, and shake for 5 minutes to mix. Then, add 100 mL of methanol, connect with a reflux condenser, heat for 1 hour, and filter. Vacuum-concentrate the filtrate until it becomes 10 mL and use this solution as the test solution. Separately, weigh 1 g of Ginger powder, add 100 mL of methanol, connect with a reflux condenser, heat for 1 hour, and filter. Vacuum-concentrate the filtrate

until the filtrate becomes 10 mL and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of hexane and ethyl acetate (85 : 15) as the developing solution to a distance of about 10 cm, and air-dry the plate. Spray evenly vanillin-sulfuric acid TS on the plate and heat the plate at 105 ℃ for 10 minutes; the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

(6) ***Mentha herb***—Pulverize Gamisoyosan Extract Granules, weigh an amount, equivalent to 1 g of Mentha herb, add 10 mL of water, and shake for 5 minutes to mix. Then, add 100 mL of methanol, connect with a reflux condenser, heat for 1 hour, and filter. Vacuum-concentrate the filtrate until it becomes 10 mL and use this solution as the test solution. Separately, weigh 1 g of Mentha herb powder, add 100 mL of methanol, connect with a reflux condenser, heat for 1 hour, and filter. Vacuum-concentrate the filtrate until it becomes 10 mL and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of ethyl acetate and acetone (10 : 3) as the developing solution to a distance of about 10 cm, and air-dry the plate. Spray evenly vanillin-sulfuric acid TS on the plate and heat the plate at 105 ℃ for 10 minutes; the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

(7) ***Peony root, Licorice, Moutan root bark, and Gardenia fruit***—Perform the test as directed under the Assay; the peaks obtained from the test solution and the standard solution have the same retention time.

Purity (1) ***Heavy metals***—(i) Total heavy metals: NMT 30 ppm.

(ii) Lead: NMT 5 ppm.

(iii) Arsenic: NMT 3 ppm.

Disintegration Meets the requirements.

Particle size distribution estimation by analytical sieving Meets the requirements.

Uniformity of dosage units (distribution) Meets the requirements.

Microbiological examination of non-sterile products Meets the requirements.

Assay (1) ***Total paeoniflorin in Peony root and Moutan root bark***—Take NLT 20 sachets of Gamisoyosan Extract Granules, weigh accurately the mass, and pulverize them. Weigh accurately the powder, equivalent to about 10 mg of paeoniflorin, add 10 mL of water, and shake for 5 minutes to mix. Then, add 100 mL of methanol, connect with a reflux condenser, heat for 1 hour, and take the supernatant, and filter. To the residue, add 100 mL of methanol, repeat extraction twice, combine and vacuum-concentrate all the filtrates until it becomes 50 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of paeoniflorin RS (previously dried in a desiccator with silica gel for 24 hours), dissolve in methanol to make exactly 50 mL, and use this solution as the standard solution. Perform the test with 20 μL each of the test solution and the standard solution as directed under the Liquid Chromatography, and calculate the peak areas, A_T and A_S, of paeoniflorin, respectively.

$$\text{Total amount (mg) of paeoniflorin } (C_{23}H_{28}O_{11}) = \text{Amount (mg) of paeoniflorin RS} \times \frac{A_T}{A_S}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 254 nm)

Column: A stainless steel column about 4 – 6 mm in internal diameter and 15 – 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 – 10 μm in particle diameter).

Mobile phase: A mixture of methanol and water (60 : 40).

Flow rate: 1.0 mL/min

(2) ***Glycyrrhizic acid in Licorice***—Take NLT 20 sachets of Gamisoyosan Extract Granules, weigh accurately the mass, and pulverize them. Weigh accurately the powder, equivalent to about 10 mg of glycyrrhizic acid, add 50 mL of water, connect with a reflux condenser, heat on a water bath for 3 hours, add 50 mL of 3 mol/mL sulfuric acid TS, and hydrolyze on a water bath for 1 hour. After cooling, add 50 mL of chloroform, connect with a reflux condenser, and warm for 30 minutes. After cooling, transfer the mixture into a separatory funnel, take the chloroform layer to extract three more times each time with 30 mL of chloroform, combine the chloroform layers, and filter through anhydrous sodium sulfate. Vacuum-concentrate the filtrate, dissolve the residue in methanol to make exactly 50 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of glycyrrhizic acid RS (previously dried in a desiccator with silica gel for 24 hours), prepare the solution in the same manner as in the preparation of the test solution, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography, and calculate the peak areas, A_T and A_S, of glycyrrhizic acid, respectively.

$$\text{Amount (mg) of glycyrrhizic acid } (C_{42}H_{62}O_{16}) = \text{Amount (mg) of glycyrrhizic acid RS} \times \frac{A_T}{A_S}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 254 nm).

Column: A stainless steel column about 4 – 6 mm in internal diameter and 15 – 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 – 10 μm in particle diameter).

Mobile phase: A mixture of methanol, water and acetic acid (78 : 19 : 3).

Flow rate: 1.0 mL/min

(3) ***Geniposide in gardenia fruit***—Take NLT 20 sachets of Gamisoyosan Extract Granules, weigh accurately the mass, and pulverize them. Weigh accurately the powder, equivalent to about 10 mg of geniposide, add 10 mL of water, and shake for 5 minutes to mix. Then, add 100 mL of methanol, connect with a reflux condenser, heat for 1 hour, and take the supernatant, and filter. To the residue, add 100 mL of methanol and repeat extraction twice, combine and vacuum-concentrate all the filtrates until it becomes 50 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of geniposide RS

(previously dried in a desiccator with silica gel for 24 hours), add methanol to make exactly 50 mL, and use the solution as the standard solution. Perform the test with 20 μL each of the test solution and the standard solution as directed under the Liquid Chromatography, and calculate the peak areas, A_T and A_S, of geniposide, respectively.

$$\text{Amount (mg) of geniposide } (C_{17}H_{24}O_{10}) = \text{Amount (mg) of geniposide RS} \times \frac{A_T}{A_S}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 254 nm)

Column: A stainless steel column about 4 – 6 mm in internal diameter and 15 – 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 – 10 μm in particle diameter).

Mobile phase: A mixture of water, methanol and acetic acid (85 : 15 : 1)

Flow rate: 1.0 mL/min

Packaging and storage Preserve in tight containers.

Gardenia Fruit
치자(梔子)

Gardeniae Fructus

Gardenia Fruit is the ripe fruit of *Gardenia jasminoides Ellis* (Rubiaceae), dried as it is or dried after passing through boiling water or steamed in water.

Gardenia Fruit contains NLT 3.0% of geniposide ($C_{17}H_{24}O_{10}$:388.37), calculated on the dried basis.

Description Gardenia Fruit is the ovate to long ovate fruit, 1 cm to 3.5 cm in length and 10 mm to 15 mm in width. The external surface occurs as yellowish brown to reddish brown, usually with 5 to 7 distinct wing-shaped longitudinal ridges. At the top, it has the calyx or its scar and the lower part is slightly sharp and sometimes has the fruit stalk remaining. The pericarp is thin and easy to break. The inside of the cut surface is relatively not intense in color but is lustrous, with 2 to 3 rows of protruding membranes, which contain seeds. The seeds are flattened ovate, several seeds gathered together to form masses. They are deep red or yellowish red, with a dense arrangement of thin, small bump-like protrusion on the external surface.
It has a slight characteristic odor and bitter taste.

Identification (1) Weigh 1 g of pulverized Gardenia Fruit, previously dried in a silica gel desiccator for 24 hours, add 100 mL of warm water, warm the mixture at 60 ℃ to 70 ℃ for 30 minutes with frequent shaking, and filter after cooling. To 1.0 mL of the filtrate, add water to make 10 mL. The color of the resulting solution is yellow and is not lighter than that of the following control solution.

Control solution—Dissolve 2 mg of potassium dichromate in water to make exactly 10 mL.

(2) Weigh 1 g each of pulverized Gardenia Fruit and Gardenia Fruit RMPM, add 20 mL of methanol, warm for 3 minutes on a water bath, cool, and filter. Use the filtrates as the test solution and the standard solution of Gardenia Fruit RMPM, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Gardenia Fruit RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate and methanol (3 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly p-anisaldehyde-sulfuric acid TS on the plate, and then heat at 105 ℃ for 10 minutes; the several spots obtained from the test solution show the same color and R_f value as the spots obtained from the Gardenia Fruit RMPM standard solution. Among them, the spot of geniposide appears at the R_f values of about 0.5.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 10.0%.

Ash NMT 6.0%.

Assay Weigh accurately about 1.0 g of pulverized Gardenia Fruit, add 50 mL of diluted methanol (7 in 10), sonicate for 1 hour to extract, and then filter. Use the filtrate as the test solution. Separately, weigh accurately about 1.0 mg of geniposide RS, add diluted methanol (7 in 10) to make exactly 1 mL, and use this solution as the standard solution. Perform the test with 20 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions, and determine the peak areas, A_T and A_S, of geniposide in the test solution and the standard solution, respectively.

$$\text{Amount (mg) of geniposide } (C_{17}H_{24}O_{10}) = \text{Amount (mg) of geniposide RS} \times \frac{A_T}{A_S} \times 50$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 254 nm).

Column: A stainless steel column, 4.6 mm in internal diameter and 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 35 ℃

Mobile phase: Control the mixing ratio of mobile phase A and mobile phase B stepwise or via the concentration gradient as directed in the following table.

Mobile phase A – Diluted acetic acid (1 in 100).

Mobile phase B – A mixture of acetonitrile and acetic acid (100) (99 : 1).

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)
0	90	10
8	85	15
35	85	15

40	80	20
45	0	100

Flow rate: 0.6 mL/min

Packaging and Storage Preserve in well-closed containers.

Gastrodia Rhizome
천마(天麻)

Gastrodiae Rhizoma

Gastrodia Rhizome is the steamed and dried rhizome of *Gastrodia elata* Bluem (Orchidaceae).

Gastrodia Rhizome contains NLT 0.20% in total of gastrodin ($C_{13}H_{18}O_7$: 286.28) and gastrodigenin ($C_7H_8O_2$: 124.14), calculated on the dried basis.

Description Gastrodia Rhizome is the rhizome, slightly curved, flattened cylindrical to fusiform, 3 to 15 cm in length, 1.5 to 5 cm in width and 0.5 to 2 cm in thickness. The external surface occurs as a pale yellowish white to yellowish brown color, with irregular longitudinal wrinkles and several turns of transverse rings, originating from the latent bud. Sometimes, chestnut-colored, root-shaped hyphae are visible. The top part has reddish brown to deep brown shoots shaped like a parrot's beak or remains of stems, and the other end has a round, umbilicated scar. The texture is very hard and difficult to cut. The cut surface is relatively flat and horny, exhibiting a yellowish white to pale brown color.

Under a microscope, the transverse section of Gastrodia Rhizome sometimes reveals the epidermal tissue remaining on the outermost layer, which is pale brown in color. Cells in the cortex are transversely long, the cell walls of cells present in the first a few rows from the outermost layer are slightly thickened, and pits are visible, even if rarely. The central cylinder has relatively large parenchyma cells, nearly orbicular or polygonal, sometimes with pitted patterns observed. The vascular bundles are collateral or amphicribral and scattered, and the vessels form groups of two or more, polygonal in shape. The parenchyma cells have polysaccharide masses and sometimes contain raphide bundle of calcium oxalate.

It has a slight, characteristic odor and a sweet taste.

Identification (1) Weigh 0.5 g of pulverized Gastrodia Rhizoma, add 10 mL of water, warm on a water bath for 5 minutes, and filter. To the filtrate, add 2 to 4 drops of iodine TS; the resulting solution exhibits a reddish purple color.

(2) Weigh 0.5 g each of pulverized Gastrodia Rhizoma and Gastrodia Rhizoma RMPM, add 5 mL each of diluted methanol (7 in 10), warm on a water bath after connecting with a reflux condenser for 1 hour, filter, and use the filtrates as the test solution and the Gastrodia Rhizoma RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the Gastrodia Rhizoma RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate using a mixture of dichloromethane, methanol and water (7 : 2.5 : 0.25) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS on the plate and heat at 105 ℃ for 10 minutes; the color and the R_f values of the several spots obtained from the test solution are the same as those of the spots obtained from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 13.0%.

Ash NMT 6.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 17.0%.

Assay Weigh accurately about 1.0 g of pulverized Gastrodia Rhizoma, add 100 mL of diluted methanol (7 in 10), sonicate for 45 minutes to extract, and filter. To the filtrate, add diluted methanol (7 in 10) to make exactly 100 mL, and use this solution as the test solution. Separately, weigh accurately about 20 mg of gastrodin RS, dissolve in diluted methanol (7 in 10) to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions, and determine the peak areas, A_{Ta} and A_{Tb}, of gastrodin and gastrodigenin, respectively, from the test solution and the peak areas, A_{Sa} and A_{Sb}, of gastrodin and gastrodigenin, respectively, from the standard solution.

$$\text{Amount (mg) of gastrodin } (C_{13}H_{18}O_7) = \text{Amount (mg) of gastrodin RS} \times \frac{A_{Ta}}{A_{Sa}}$$

$$\text{Amount (mg) of gastrodigenin } (C_7H_8O_2) = \text{Amount (mg) of gastrodigenin RS} \times \frac{A_{Tb}}{A_{Sb}}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 270 nm)

Column: A stainless steel column 4 to 6 mm in internal diameter and 15 to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 to 10 μm in particle diameter).

Column temperature: 30 ℃

Mobile phase: Control the mobile phases A and B stepwise as directed in the following table or via the concentration gradient.

Mobile phase A: A solution of formic acid (1 in 1000).

Mobile phase B: A mixture of formic acid and acetonitrile (1 in 1000).

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)
0	98	2
25	90	10
30	85	15

Flow rate: 1.0 mL/min

System suitability

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviations of each peak area of gastrodin and gastrodigenin are NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Gentian
겐티아나

Gentianae Luteae Radix et Rhizoma

Gentian is the root and rhizome of *Gentiana lutea* Linné (Gentianaceae).

Description Gentian consists of the root and rhizome, nearly cylindrical, 10 cm to 50 cm in length and 2 cm to 4 cm in diameter. The outer surface occurs as dark brown with lateral roots and sometimes longitudinally divided. The rhizome is short with fine transverse folds and sometimes with buds and remains of leaves on the upper edge. The root is longitudinally and deeply wrinkled and slightly twisted. The fractured surface is flat and yellowish brown, and the cortex and xylem is dark brown at the cambium.

Under the microscope, a cross-section of the root shows several layers of collenchyma connected internally with 4 to 6 layers of thin-walled cork. The secondary cortex of the parenchyma consists of irregularly distributed phloem. The xylem consists mainly of parenchyma with single or clustered vessels and tracheids. A small number of sieve tubes are present in the xylem. The parenchyma cells of the cortex and xylem contain oil droplets and minute calcium oxalate crystals. Starch granules are very rare.

It has a characteristic odor and an initially sweet, later lasting bitter taste.

Identification (1) Weigh 0.1 g of pulverized Gentian, previously dried in a desiccator (silica gel) for 48 hours, put them on a slide glass. Put a glass ring, 10 mm in both internal diameter and in height, on it, then cover with another slide glass and heat gently and gradually; a pale yellow crystals are sublimed on the upper slide glass. These crystals are insoluble in water or ethanol, but soluble in potassium hydroxide TS.

(2) Weigh 0.5 g of pulverized Gentian, add 10 mL of methanol, shake for 5 minutes to mix. Then, filter it and use the filtrate as the test solution. Separately, dissolve 1 mg of gentiopicroside RS in 1 mL of methanol and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography (with fluorescent agent). Next, develop the plate with a mixture of ethyl acetate, anhydrous ethanol and water (8 : 2 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 254 nm); one spot among the several spots from the test solution and a dark purple spot from the standard solution show the same color and the same R_f value.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

Ash NMT 6.0%.

Acid-insoluble ash NMT 3.0%.

Packaging and storage Preserve in well-closed containers.

Gentian Root and Rhizome
용담(龍膽)

Gentianae Scabrae Radix et Rhizoma

Gentian Root and Rhizome is the root and rhizome of *Gentiana scabra* Bunge, *Gentiana triflora* Pallas or *Gentiana manshurica* Kitagawa (Gentianaceae).

Description Gentian Root and Rhizome is the root and rhizome, the latter of which is in the shape of irregular masses, 1 cm to 3 cm in length and 0.3 cm to 1 cm in diameter. The external surface occurs as a dark grayish brown or deep brown color, with stem scars or remains of stems at the top. There are several thin, long roots attached around the top and bottom. The root is cylindrical, slightly curved twisted, 10 cm to 20 cm in length and 0.2 cm to 0.5 cm in diameter. The external surface occurs as a pale yellow to yellowish brown color, with numerous distinct transverse wrinkles at the top with some longitudinal wrinkles. The texture is fragile and easy to cut. The cut surface is slightly smooth, the cortex occurs as a yellowish white or pale yellowish brown color, and the xylem exhibits a relatively pale color with spot-shaped rings.

Under a microscope, the transverse section of Gentian Root and Rhizome reveals the bark cells, close to circular or flattened circular and the slightly thick outer wall, usually containing droplets of fatty oil. The cortex is narrow, and the endodermis is distinct. Each parent cell contains 2 to 10 daughter cells. The phloem is relatively wide, and most cells have already degenerated, broken with many torn clefts. A small number of the sieve tube groups are scattered in the direction of the diameter. The cambium usually forms discontinuous rings. In the xylem, 3 to 10 vessels form groups, sometimes forming the shape of two thighs. The medullary rays are wide, and the pith takes up 1/3 of the root. The parenchyma has many longitudinally and transversely torn spaces, and a small number of parenchyma tissues contain droplets of fatty oil and needle or prismatic calcium oxalate crystals. Starch grains are not visible.

It has a slight, characteristic odor and an extremely bitter taste that lasts for a long time.

Identification Weigh 0.5 g of pulverized Gentian Root and Rhizome, add 10 mL of methanol, shake for 20 minutes to mix, and filter. Perform the test using this solution as the test solution as directed under the Thin Layer Chromatography. Separately, dissolve 1 mg of gentiopicroside RS in 1 mL of methanol and use this solution as the standard solution. Spot 10 μL each of the test solution and the standard solutions on the thin-layer chromatographic plate made of silica gel (with a fluorescent agent) for thin layer chromatography. Next, develop the plate with a mixture of ethyl acetate, dehydrated ethanol and water (8 : 2 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the

plate. Examine the plate under ultraviolet light (main wavelength: 254 nm); the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of the dark violet spot from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.7 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 7.0%.

Acid-insoluble ash NMT 3.0%.

Packaging and storage Preserve in well-closed containers.

Geranium Herb
현초(玄草)

Geranii Herba

Geranium Herb is the aerial part collected before or when flowering of *Geranium thunbergii* Siebold et Zuccarini (or Geraniaceae).

Description Geranium Herb is the aerial part, consisting of the stem and opposite leaves. Stem is slender and long, greenish brown. Stem and leaf are covered with soft hairs. Its leaf is a palm-like shape and divided into 3 to 5 lobes, 2 cm to 4 cm in length. It has a long petiole, grayish yellowish green to grayish brown. Each lobe is oblong to obovate and has a dull serrate.
It occurs as nearly odorless and has an astringent taste.

Identification Weigh 0.1 g of Geranium Herb, add 10 mL of water to boil, and filter. To the filtrate, add 1 drop of Iron(III) chloride TS; the solution exhibits a blackish blue.

Purity (1) ***Foreign matter***—Geranium Herb contains less than 2.0% of the root and other foreign matter.
(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): NMT 0.2 ppm.
(vi) Endrin: NMT 0.01 ppm.

Ash NMT 10.0%.

Acid-insoluble ash NMT 1.5%.

Extract content ***Dilute ethanol-soluble extract***— NLT 15.0%.

Packaging and storage Preserve in well-closed containers.

Ginger
건강(乾薑)

Zingiberis Rhizoma

Ginger is the dried rhizome of *Zingiber officinale* Roscoe (Zingiberaceae).

Ginger contains NLT 0.4% of 6-gingerol ($C_{17}H_{26}O_4$: 294.39), calculated on the dried basis.

Description Ginger consists of a rhizome and has the shape of flat, irregular masses with finger-shaped branches. Ginger is 2 cm to 4 cm long and has a diameter of 1 cm to 2 cm. The outer surface occurs as grayish white to pale grayish brown with white powder and with or without pale grayish yellow periderm. The branched parts are slightly compressed and slightly curved ovate or oblong-ovate, sometimes with pendant buds at both ends, swollen warty. The texture is firm. The cut surface is slightly fibrous, powdery and yellowish white or grayish white. Under the magnifying glass, the cross section shows a distinct ring pattern in the endodermis with scattered vascular bundles and yellow oil spots.
Under the microscope, the cross section of Ginger shows a cork layer consisting of several rows of flat cork cells. Several leaf vascular bundles are scattered in the cortex, and oil cells are visible throughout. The endodermis is distinct and shows a casparian strip. The central cylinder occupies most of the underground stems and is scattered with collateral vascular bundles. The vascular bundles near the cylinder are small and relatively densely arranged. Unwoody fibers and oil cells are found within and around the xylem. The parenchyma contains starch granules.
It has a characteristic odor and an extremely pungent taste.

Identification Weigh 2 g of pulverized Ginger, add 5 mL of acetone, shake for 3 minutes to mix. Then, filter it and use the filtrate as the test solution. Separately, dissolve 1 mg of 6-gingerol RS in 1 mL of acetone and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of hexane, acetone, and acetic acid (100) (10 : 7 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly 2,4-dinitrophenylhydrazine TS on the plate and heat it at 105 ℃ for 10 minutes; one spot of the several spots from the test solution and a brown spot from the standard solution show the same color and R_f value.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—Proceed as directed in "Ginger" in [Attachment 4] MRLs for Agricultural Products in the Public Notification "Standards and Specifications for Food" of the Ministry of Food and Drug Safety (MFDS).
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 14.0%.

Ash NMT 8.0%.

Assay Weigh accurately about 2 g of pulverized Ginger, add 60 mL of methanol, connect with a reflux condenser, heat for 2 hours, and filter it. To the residue, add 30 mL of methanol and proceed in the same manner. Combine all the filtrates, add methanol to make exactly 100 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of 6-gingerol RS, dissolve it in methanol to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas, A_T and A_S, of 6-gingerol in the test solution and the standard solution, respectively.

$$\text{Amount (mg) of 6-gingerol } (C_{17}H_{26}O_4) = \text{Amount (mg) of 6-gingerol RS} \times \frac{A_T}{A_S}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 280 nm)

Column: A stainless steel column, about 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Mobile phase: A mixture of water and acetonitrile (55 : 45)

Flow rate: Adjust the flow rate so that the retention time of 6-gingerol is about 7 minutes.

System suitability

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak area of 6-gingerol is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Ginkgo Leaf
은행엽(銀杏葉)

Ginkgo Folium

Ginkgo Leaf is the leaf of *Ginkgo biloba* Linné (Ginkgoaceae).

Ginkgo Leaf contains NLT 0.5% of total flavonoids, calculated on the dried basis.

Description Ginkgo Leaf is the leaf, mostly crinkled or broken, and the whole ones are fan-shaped, 3 cm to 12 cm in length and 5 cm to 15 cm in width. The external surface occurs as green, the top margin of the leaf is curved, irregularly wavy and sometimes concave in the middle, and deeply concave ones may reach 4/5 the length of the leaf. The leaf vein is dichotomous, branched parallel into 2 tridents. It is smooth and hairless, with the end of the leaf margin divided into 3. It is easy to be torn longitudinally. The petiole is cuneate, 2 cm to 8 cm in length.
It has a characteristic odor and a slightly astringent taste.

Identification Weigh 0.5 g of pulverized Ginkgo leaf, add 10 mL of ethanol, warm to extract, and filter. To 1 mL of the filtrate, add a small amount of magnesium and 1 drop of hydrogen chloride; the resulting solution exhibits a red color.

Purity (1) ***Foreign matter***—The amounts of stems and other foreign matters contained in Ginkgo Leaf are NMT 5.0% and NMT 2.0%, respectively.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

Loss on drying NMT 11.0% (1.0 g, between 100 ℃ and 105 ℃, 2 hours)

Ash NMT 11.0%.

Assay Weigh accurately about 2.5 g of pulverized Ginkgo Leaf, add 50 mL of diluted acetone (3 in 5), heat after connecting with a reflux condenser for 30 minutes, and filter. To the residue, add 40 mL of diluted acetone (3 in 5) and proceed in the same manner. Combine the filtrates and add diluted acetone (3 in 5) to make exactly 100 mL. Take 50 ml of this solution, evaporate the acetone in vacuum, and rinse with 30 ml of methanol. Add 4.4 ml of hydrochloric acid and water to make exactly 50 mL. After centrifugation, transfer 10 ml of the supernatant into a 10 ml brown-glass vial, stopper the vial, and warm on a water bath for 25 minutes. After cooling, use this solution as the test solution. Separately, weigh accurately about 10 mg of quercetin dihydrate RS, dissolve in 20 ml of methanol, add 15 ml of dilute hydrochloric acid, 5 ml of water and methanol to make exactly 50 ml, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas, A_{Ta}, A_{Tb} and A_{Tc}, of quercetin, kaempferol (the relative retention time to quercetin is about 1.4) and isorhamnetin (the relative retention time to quercetin is about 1.5), respectively, from the test solution and the peak area, A_S, of quercetin from the standard solution.

$$\text{Amount (mg) of total flavonoids} = \text{Amount (mg, as quercetin) of quercetin dehydrate RS} \times \frac{A_{Ta} + A_{Tb} + A_{Tc}}{A_S} \times 2 \times 2.514$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 370 nm)

Column: A stainless steel column 4 mm in internal diameter and 12.5 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: 25 ℃

Mobile phase: Control the mobile phases A and B stepwise as directed in the following table or via the concentration gradient.

Mobile phase A: Dissolve 0.3 of phosphoric acid in 1000 mL of water and adjust the pH to 2.0 with phosphoric acid.

Mobile phase B: Methanol

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)
0	60	40
1	60	40
20	45	55
21	0	100

Flow rate: 1.0 mL/min

System suitability

System performance: Adjust the conditions so that the retention time of quercetin is about 12.5 minutes with the resolution between the peaks of kaempferol and of isorhamnetin being NLT 1.5.

Packaging and storage Preserve in well-closed containers.

Ginseng
인삼(人蔘)

Ginseng Radix

Ginseng is the root of *Panax ginseng* C. A. Meyer (Araliaceae), used as it is or after removing the fine roots and the cork layer.

Ginseng contains NLT 0.10% of ginsenoside Rg_1 ($C_{42}H_{72}O_{14}$: 801.01) and NLT 0.20% of ginsenoside Rb_1 ($C_{54}H_{92}O_{23}$: 1109.29), calculated on the dried basis.

Description Ginseng is the root, thin and long cylindrical to fusiform, often branching 2 to 5 lateral roots from the middle. It is 5 to 20 cm in length, and the main root is 5 to 30 mm in diameter. The external surface occurs as a pale yellowish brown to pale grayish brown color, with longitudinal wrinkles and scars of rootlets, The crown is slightly curved, with short remains of the rhizome. The fractured surface is practically flat, exhibiting a light yellowish brown color, and the area close to the cambium exhibits a brown color.

Under a microscope, the transverse section of Ginseng reveals the thin-walled parenchyma cells filled with starch grains and the cortex scattered with secret vessels filled with yellow to yellowish red secretion. Aggregate crystals of calcium oxalate are observed in the parenchyma cells in the phloem.

It has a characteristic odor and a slightly sweet taste at first, followed by a slight bitter taste.

Identification (1) Apply dilute iodine TS dropwise to the cut surface of Ginseng; it exhibits a dark blue color.

(2) Weigh 2 g of pulverized Ginseng, add 20 mL of methanol, heat after connecting with a reflux condenser on a water bath for 15 minutes, filter, and use the filtrate as the test solution. Separately, dissolve 1 mg of ginsenoside Rg_1 RS in 1 mL of methanol and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of ethyl acetate, methanol and water (14 : 5 : 4) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly sulfuric acid TS for spray on the plate and heat the plate at 105 ℃ for 10 minutes; the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

Purity (1) ***Foreign matter***—The amount of stems and other foreign matters contained in Ginseng is less than 2.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—Proceed with Ginseng as directed in 'Dried Ginseng' described in [Attachment 4] Tolerances for Pesticide Residues for Agricultural Products provided in the Public Notice "Standards and Specifications for Food" of the Ministry of Food and Drug Safety (MFDS).

(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 15.0% (6 hours).

Ash NMT 5.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 14.0%.

Assay (1) ***Ginsenoside Rg_1***—Weigh accurately about 1 g of pulverized Ginseng, put in a stoppered centrifuge tube, add 30 mL of diluted methanol (3 in 5), shake well for 15 minutes to mix, centrifuge, and take the supernatant. Add 15 mL of diluted methanol (3 in 5) to the residue and repeat the procedure in the same manner, combine the supernatant with that obtained above, and add diluted methanol (3 in 5) to make exactly 50 mL. Pipet 10 mL of this solution, add 3 mL of dilute sodium hydroxide TS, allow to stand for 30 minutes, add 3 mL of 0.1 mol/L hydrochloric acid TS, add diluted methanol (3 in 5) to make exactly 20 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of ginsenoside Rg_1 RS (previously dried in a desiccator with silica gel for 24 hours), add diluted methanol (3 in 5) to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions, and determine the peak areas, A_T and A_S, of the test solution and the standard solution, respectively.

$$\text{Amount (mg) of ginsenoside } Rg_1 \ (C_{42}H_{72}O_{14}) = \text{Amount (mg) of ginsenoside } Rg_1 \text{ RS} \times \frac{A_T}{A_S}$$

Operating conditions

Detector: Ultraviolet absorption photometer (measurement wavelength: 203 nm)

Column: A stainless steel column 4.6 mm in internal diameter and 15 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 30 ℃

Mobile phase: A mixture of water and acetonitrile (4 : 1)

Flow rate: Adjust the flow rate so that the retention time of ginsenoside Rg_1 is about 25 minutes.

System suitability

System performance: Dissolve 1 mg each of ginsenoside Rg_1 RS and ginsenoside Re in diluted methanol (3 in 5) to make 10 mL. Proceed with 10 μL of this solution under the above operating conditions; ginsenoside Rg_1 and ginsenoside Re are eluted in this order with the resolution between these peaks being NLT 1.5.

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak areas of ginsenoside Rg_1 is NMT 1.5%.

(2) ***Ginsenoside Rb₁***—Use the test solution obtained in (1) as the test solution. Separately, weigh accurately about 10 mg of ginsenoside Rb_1 RS (previously dried in a desiccator with silica gel for 24 hours), add diluted methanol (3 in 5) to make exactly100 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas, A_T and A_S.

$$\text{Amount (mg) of ginsenoside } Rb_1 \ (C_{54}H_{92}O_{23}) = \text{Amount (mg) of ginsenoside } Rb_1 \text{ RS} \times \frac{A_T}{A_S}$$

Operating conditions

Detector: Ultraviolet absorption photometer (measurement wavelength: 203 nm)

Column: A stainless steel column 4.6 mm in internal diameter and 15 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 40 ℃

Mobile phase: A mixture of water and acetonitrile (7 : 3)

Flow rate: Adjust the flow rate so that the retention time of ginsenoside Rb_1 is about 20 minutes.

System suitability

System performance: Dissolve 1 mg each of ginsenoside Rb_1 RS and ginsenoside Rc in diluted methanol (3 in 5) to make 10 mL. Proceed with 10 μL of this solution under the above operating conditions; ginsenoside Rb_1 and ginsenoside Rc are eluted in this order with the resolution between these peaks being NLT 3.

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak areas of ginsenoside Rb_1 is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Red Ginseng
홍삼(紅蔘)

Ginseng Radix Rubra

Red Ginseng is the root of *Panax ginseng* C. A. Meyer (Araliaceae), after being steamed.

Red Ginseng contains NLT 0.10% of ginsenoside Rg_1 ($C_{42}H_{72}O_{14}$: 801.01) and NLT 0.20% of ginsenoside Rb_1 ($C_{54}H_{92}O_{23}$: 1109.29), calculated on the dried basis.

Description Red Ginseng is a steamed, thin and long cylindrical to fusiform root, often branching out into 2 to 5 lateral roots from the middle, 5 cm to 25 cm in length and main root is 5 mm to 30 mm in diameter. External surface occurs as pale yellowish brown to reddish brown and translucent, has longitudinal wrinkles and thin marks of root. Crown is slightly distorted with short remains of stem. Curved surface is flat, and the texture is horny and hard.

It has a characteristic odor, and the taste is at first slightly sweet, followed by a slight bitterness.

Identification (1) Weigh 0.2 g of pulverized Red Ginseng, add 2 mL of acetic anhydride, warm on a water bath for 2 minutes, and filter. To 1 mL of the filtrate, add gently 0.5 mL of sulfuric acid; a reddish brown color develops at the zone of contact.

(2) Weigh 2 g of pulverized Red Ginseng, add 20 mL of methanol, heat after connecting with a reflux condenser for 15 minutes, cool, and filter. Use the filtrate as the test solution. Separately, weigh 1 mg of ginsenoside Rg_1 RS, dissolve in 1 mL of methanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a lower layer of a mixture of chloroform, methanol and water (13 : 7 : 2) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly sulfuric acid TS for spray on the plate, and heat at 105 °C for 5 minutes; one spot among the several spots obtained from the test solution and the spot from the standard solution show the same color and the same R_f value.

Purity (1) ***Foreign matter***—The amount of the stems and other foreign matter contained in Red Ginseng is less than 2.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—Proceed as directed in "Red Ginseng" described in [Attachment 4] Tolerances for Pesticide Residues for Agricultural Products provided in the Public Notice "Standards and Specifications for Food" of the Ministry of Food and Drug Safety (MFDS).

(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 15.5% (6 hours).

Ash NMT 4.5%.

Extract content ***Dilute ethanol-soluble extract***—NLT 18.0%.

Assay (1) ***Ginsenoside Rg₁***—Weigh accurately about 1 g of pulverized Red Ginseng and perform the test as directed in the Assay under Ginseng.

(2) ***Ginsenoside Rb₁***—Use the test solution obtained in (1) as the test solution. Perform the test as directed in the Assay under Ginseng.

Packaging and storage Preserve in well-closed containers.

Gleditsia Spine
조각자(皂角刺)

Gleditsiae Spina

Gleditsia Spine is the thorn of *Gleditsia japonica* Miquel or *Gleditsia sinensis* Lamark (Leguminosae).

Description (1) ***Gleditsia japonica***—Gleditsia Spine from *Gleditsia japonica* consists of main spines and primary to secondary branched small spines. The main spines are flattened, long conical or conical, 3 to 15 cm in length or longer and 3 to 10 mm in width. Branched small spines are pointed at the apex, 1 to 6 cm in length. The external surface occurs as a purplish brown to deep brown color. The body is light, and the texture is hard and difficult to fracture. The fractured surface is 1 to 3 mm in thickness, and the tip of the spine is sharp and thin. The xylem occurs as a yellowish white color, the pith is loose, exhibiting a

pale reddish brown color, and the texture is fragile, making it easy to fracture.
It occurs as odorless and has a plain taste.

(2) ***Gleditsia sinensis***—The main and small spines of Gleditsia Spine from *Gleditsia sinensis* are generally more circular and harder than those of Gleditsia Spine from *Gleditsia japonica.*

Purity (1) ***Foreign matter***—The amount of stems and other foreign matters contained in Gleditsia Spine is less than 3.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 10.0%.

Ash NMT 2.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 10.0%.

Packaging and storage Preserve in well-closed containers.

Glehnia Root
해방풍(海防風)

Glehniae Radix

Glehnia Root is the root of *Glehnia littoralis* Fr. Schmidt ex Miquel (Umbelliferae).

Description Glehnia Root is the slender, long cylindrical root, 10 cm to 20 cm in length and 5 mm to 15 mm in diameter. The external surface occurs as pale yellowish white, frequently with the epidermis remaining, and the external surface with the epidermis not removed occurs as yellowish brown. The entire root has thin longitudinal wrinkles and longitudinal furrows, yellowish brown in color, with spot-like scars of thin roots. The yellowish brown rhizome usually remains at the apex, the upper part is slightly slender, the middle part slightly thick, gradually tapering towards the lower part. The texture is hard is but easy to break. The fractured surface is powdery and the cortex occurs as pale white to pale yellow, sometimes cracked, brown secretory canals scattered as small dots. The xylem occurs as pale yellow and the texture is dense.
It has a slight, characteristic odor and a slightly sweet taste.

Identification Weigh 1 g of pulverized Glehnia Root and Glehnia Root RMPM, add 10 mL of acetone, sonicate for 20 minutes, filter, and evaporate the filtrate to dryness. Take each residue, dissolve in 1 mL of ethanol, and use each solution as the test solution and the standard solution of Glehnia Root RMPM. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the standard solution of Glehnia Root RMPM on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of cyclohexane and ethyl acetate (2 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS on the plate and heat at 105 ℃ for 10 minutes; the several spots from the test solution and the spots from standard solution of Glehnia Root RMPM show the same color and have the same R_f value.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

(3) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 6.0%.

Acid-insoluble ash NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Hawthorn Fruit
산사(山楂)

Crataegi Fructus

Hawthorn Fruit is the ripe fruit of *Crataegus pinnatifida* Bunge and its variants (Rosaceae).

Description Hawthorn Fruit is a circular or elongated fruit with a diameter of 1 cm to 2.5 cm. The outer surface occurs as reddish brown to dark red with sparse white round spots. There is a persistent calyx at the apex, which is deeply concave and has a pedicel scar at the bottom. Most are processed and cut transversely or longitudinally, 2 mm to 6 mm in thickness, wrinkled and uneven. There are 4 to 5 seeds, rarely 3, most are precipitated, the texture is hard and long kidney-shaped, the dorsal side is approximately round with a valley and two peaks in the center.
It has a slight characteristic aroma and sour taste.

Identification (1) Weigh 1 g of pulverized Hawthorn Fruit, add 10 mL of ether, and shake for 2 minutes to mix, and filter. After removing the ether layer from the filtrate, dissolve the residue in 1 mL of acetic anhydride, and add 1 to 2 drops of sulfuric acid; a reddish purple color develops.

(2) Weigh 1 g of pulverized Hawthorn Fruit, add 4 mL of ethyl acetate, sonicate for 15 minutes, filter, and use the filtrate as the test solution. Separately, weigh 1 mg of ursolic acid RS, dissolve in 1 mL of ethyl acetate, and use this solution as standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 4 μL each of the test solution and the standard solution on a thin-layer chromatographic plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of hexane, ethyl acetate and formic acid (20 : 10 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray sulfuric acid TS for spraying evenly on the plate and heat at 105 ℃ for 10 minutes; one of the several spots obtained from the test solution shows the same color and

the R_f value as the reddish purple spot obtained from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 6.0%.

Packaging and storage Preserve in well-closed containers.

Hyeonggaeyeongyotang Extract Granules
형개연교탕엑스 과립

Hyeonggaeyeongyotang Extract Granules contains NLT 2.3 mg of glycyrrhizic acid ($C_{42}H_{62}O_{16}$: 822.93) in Licorice, 1.7 mg of peoniflorin ($C_{23}H_{28}O_{11}$: 480.46) in Peony Root, 5.0 mg of geniposide ($C_{17}H_{24}O_{10}$: 388.37) in Gardenia Fruit, and 7.9 mg of baicalin ($C_{21}H_{18}O_{11}$: 446.36) in Scutellaria Root for a dose (one sachet).

Method of preparation **For a dose (one sachet)**

Platycodon Root, Angelica Dahurica Root, Bupleurum Root	0.83 g
Licorice, Angelica Gigas Root, Saposhnikovia Root, Forsythia Fruit, Peony Root, Poncirus Immature Fruit, Cnidium Rhizome, Gardenia Fruit, Schizonepeta Spike, Scutellaria Root	0.50 g

Pulverize the above crude drugs to coarse cutting, weigh each crude drugs, put into the extractor, and add eight to ten fold of purified water. Extract for 2 to 3 hours at 80 to 100 ℃, filter the extract, and concentrate the filtrate under vacuum below 60 ℃ until it becomes 1.94 g to 2.75 g of semiliquid extract or concentrate in a suitable method until it becomes 0.92 g to 1.30 g of dry extract. Then, prepared as directed under Granules.

Identification (1) ***Platycodon Root***—Pulverize Hyeonggaeyeongyotang Extract Granules, weigh an amount equivalent to 1 g of platycodon root, add 10 mL of water, and shake for 5 minutes to mix. Add 100 mL of methanol, heat after connecting with a reflux condenser for 1 hour, and filter. Concentrate the filtrate under vacuum until the filtrate becomes 10 mL, and use this solution as the test solution. Separately, weigh accurately about 1 g of pulverized Platycodon Root, add 100 mL of methanol, heat after connecting with a reflux condenser for 1 hour, and filter. Concentrate the filtrate under vacuum until the filtrate becomes 10 mL and, use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel (with fluorescent indicator) for thin-layer chromatography. Develop the plate with a lower layer of a mixture of chloroform, methanol and water (65 : 35 : 10) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly sulfuric acid TS for spraying on the plate, and heat at 105 ℃ for 10 minutes; one spot among the several spots from the test solution and the spot from the standard solution show the same colors and R_f values.

(2) ***Angelica Dahurica Root***—Pulverize Hyeonggaeyeongyotang Extract Granules, weigh an amount equivalent to 1 g of Angelica Dahurica Root, add 10 mL of water, and shake for 5 minutes to mix. Add 100 mL of methanol, heat after connecting with a reflux condenser for 1 hour, and filter. Concentrate the filtrate under vacuum until the filtrate becomes 10 mL, and use this solution as the test solution. Separately, weigh accurately about 1 g of pulverized Angelica Dahurica Root, add 100 mL of methanol, heat after connecting with a reflux condenser for 1 hour, and filter. Concentrate the filtrate under vacuum until the filtrate becomes 10 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of hexane and ethyl acetate (1 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 365 nm); one spot among the several spots from the test solution and spots from the standard solution show the same color and the R_f values.

(3) ***Bupleurum Root***—Pulverize Hyeonggaeyeongyotang Extract Granules, weigh an amount equivalent to 1 g of bupleurum root, add 10 mL of water, and shake for 5 minutes to mix. Add 100 mL of methanol, heat after connecting with a reflux condenser for 1 hour, and filter. Concentrate the filtrate under vacuum until the filtrate becomes 10 mL, and use this solution as the test solution. Separately, weigh 1 g of pulverized Bupleurum Root RMPM, add 100 mL of methanol, heat after connecting with a reflux condenser for 1 hour, and filter. Concentrate the filtrate under vacuum until the filtrate becomes 10 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate using a mixture of dichloromethane, methanol and water (30 : 10 : 1) as a developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly sulfuric acid TS for spraying on the plate, and heat at 105 ℃ for 10 minutes: one spot among the several spots from the test solution and the spot from the standard solution show the same color and the R_f values.

(4) ***Angelica Gigas Root***—Pulverize Hyeonggaeyeongyotang Extract Granules, weigh an amount equivalent to 1 g of Angelica Gigas root, add 10 mL of water, and shake for 5 minutes to mix. Add 100 mL of methanol, heat after connecting with a reflux condenser for 1 hour, and filter. Concentrate the filtrate under vacuum until the filtrate becomes 10 mL, and use this solution as the test solution. Separately, weigh accurately about 1 g of pulverized Angelica Gigas Root, add 100 mL of methanol, heat after connecting with a reflux condenser for 1 hour, and filter. Concentrate the filtrate under vacuum until the filtrate becomes 10 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of toluene, ether, water and acetic acid (500 : 500 : 5 : 2) as the developing solvent to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with vanillin-sulfuric acid TS: one spot among the several spots from the test solution and the spot from the standard solution show the same

colors and the same R_f values.

(5) ***Saposhnikovia Root***—Pulverize Hyeonggaeyeongyotang Extract Granules, weigh an amount equivalent to 1 g of Saposhnikovia Root, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate under vacuum until the filtrate becomes 10 mL, and use this solution as the test solution. Separately, weigh accurately about 1 g of pulverized Saposhnikovia Root, add 100 mL of methanol, heat after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate under vacuum until the filtrate becomes 10 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of hexane and ethyl acetate (1 : 1) as the developing solvent to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 365 nm): one spot among the several spots from the test solution and the spot from the standard solution show the same colors and the R_f values.

(6) ***Forsythia Fruit***—Pulverize Hyeonggaeyeongyotang Extract Granules, weigh an amount equivalent to 1 g of Forsythia Fruit, add 10 mL of water, and shake for 5 minutes to mix. Add 100 mL of methanol, heat after connecting with a reflux condenser for 1 hour, and filter. Concentrate the filtrate under vacuum until the filtrate becomes 10 mL, and use this solution as the test solution. Separately, weigh 1 g of pulverized Forsythia Fruit , add 100 mL of methanol, heat after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate under vacuum until the filtrate becomes 10 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel (with fluorescent indicator) for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, methyl ethyl ketone, formic acid, and water (5 : 3 : 1 :1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 365 nm): one spot among the several spots from the test solution and the spot from the standard solution show the same colors and the R_f values.

(7) ***Poncirus Immature Fruit***—Pulverize Hyeonggaeyeongyotang Extract Granules, weigh an amount equivalent to 1 g of Poncirus Immature fruit, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate under vacuum until the filtrate becomes 10 mL, and use this solution as the test solution. Separately, weigh 1 g of pulverized Poncirus Immature Fruit RMPM, add 100 mL of methanol, heat after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate under vacuum until the filtrate becomes 10 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel (with fluorescent indicator) for thin-layer chromatography. Then, develop the plate with a mixture of ethyl acetate and hexane (1 :1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly the plate with p-anisaldehyde-sulfuric acid TS and heat at 105 ℃ for 10 minutes and examine under ultraviolet light (main wavelength: 365 nm): one spot among the several spots from the test solution and the spot from the standard solution show the same colors and the R_f values.

(8) ***Cnidium Rhizome***—Pulverize Hyeonggaeyeongyotang Extract Granules, weigh an amount equivalent to 1 g of Cnidium Rhizome, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate under vacuum until the filtrate becomes 10 mL, and use this solution as the test solution. Separately, weigh 1 g of pulverized Cnidium Rhizome, add 100 mL of methanol, heat after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate under vacuum until the filtrate becomes 10 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel (with fluorescent indicator) for thin-layer chromatography. Develop the plate with a mixture of cyclohexane and ethyl acetate (9 : 1) as the developing solvent to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with sulfuric acid TS for spraying and heat at 105 ℃ for 10 minutes and examine under ultraviolet light (main wavelength: 365 nm): one spot among the several spots from the test solution and the spot from the standard solution show the same colors and the R_f values.

(9) ***Schizonepeta Spike***—Pulverize Hyeonggaeyeongyotang Extract Granules, weigh an amount equivalent to 1 g of Schizonepeta Spike, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate under vacuum until the filtrate becomes 10 mL, and use this solution as the test solution. Separately, weigh 1 g of pulverized Schizonepeta Spike, add 100 mL of methanol, heat after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate under vacuum until the filtrate becomes 10 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test Solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of hexane and ethyl acetate (17 : 3) as the developing solvent to a distance of about 10 cm and air-dry the plate. Spray evenly vanillin-sulfuric acid TS on the plate, and heat at 105 ℃ for 10 minutes: one spot among the several spots from the test solution and the spot from the standard solution show the same colors and the R_f values.

(10) ***Licorice, Peony Root, Gardenia Fruit, and Scutellaria Root***—Perform the test as directed in the Assay; the test solution shows peaks at the same retention time as the standard solution.

Purity (1) ***Heavy metals***—(i) Total heavy metals: NMT 30 ppm.

(ii) Lead: NMT 5 ppm.

(iii) Arsenic: NMT 3 ppm

Disintegration Meets the requirements.

Particle size distribution estimation by analytical sieving Meets the requirements.

Uniformity of dosage units (distribution) Meets the requirements.

Microbial Limit Meets the requirements.

Assay (1) ***Glycyrrhizic Acid of Licorice***—Weigh accurately the mass of NLT 20 sachets of Hyeonggaeyeongyotang Extract Granules and pulverize. Weigh accurately an amount equivalent to 10 mg of glycyrrhizic acid, add 50 mL of water, heat after

connecting with a reflux condenser for 3 hours, add 50 mL of 3 mol/L of sulfuric acid TS, and hydrolyze on a water bath for 1 hour. After cooling, add 50 mL of chloroform, heat after connecting with a reflux condenser for 30 minutes. After cooling, take the chloroform layer in separatory funnel, extract three times with 30 mL each of chloroform repetitively, combine chloroform layers, and filter through anhydrous sodium sulfate. Concentrate the filtrate under vacuum, dissolve the residue in methanol to make exactly 50 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of glycyrrhizic acid RS (previously dried in a silica gel desiccator for 24 hours), proceed in the same manner as the test solution, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions and determine the peak areas, A_T and A_S, of the test solution and the standard solution.

Amount (mg) of glycyrrhizic acid ($C_{42}H_{62}O_{16}$)
= amount (mg) of glycyrrhizic acid RS × $\frac{A_T}{A_S}$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 254 nm).

Column: A stainless steel column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Mobile phase: A mixture of methanol, water, and acetic acid (100) (78 : 19 : 3).

Flow rate: 1.0 mL/min

(2) ***Paeoniflorin of Peony Root***—Weigh accurately the mass of NLT 20 sachets of Hyeonggaeyeongyotang Extract Granules and pulverize. Weigh accurately an amount equivalent to about 10 mg of paeoniflorin, add 10 mL of water, and shake for 5 minutes to mix. Add 100 mL of methanol, heat after connecting with a reflux condenser for 1 hour, take the supernatant, and filter. To the residue, add 100 mL of methanol, extract twice repetitively, combine the filtrates, concentrate the filtrate under vacuum until the filtrate becomes 50 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of paeoniflorin RS (previously dried in a silica gel desiccator for 24 hours), dissolve in methanol to make exactly 50 mL, and use this solution as the standard solution. Perform the test as directed in the Assay under *Peony Root*.

(3) ***Geniposide of Gardenia Fruit***—Weigh accurately the mass of NLT 20 sachets of Hyeonggaeyeongyotang Extract Granules and pulverize. Weigh accurately an amount equivalent to about 50 mg of geniposide, add 70 mL of methanol, and heat after connecting with a reflux condenser for 1 hour. After cooling, filter, add methanol to make exactly 100 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of geniposide RS (previously dried in a silica gel desiccator for 24 hours), add methanol to make exactly 20 mL, and use this solution as the standard solution. Perform the test with 20 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions, and determine the peak areas, A_T and A_S, of geniposide.

Amount (mg) of geniposide ($C_{17}H_{24}O_{10}$)
= Amount (mg) of geniposide RS × $\frac{A_T}{A_S}$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 254 nm)

Column: A stainless steel column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Mobile phase: A mixture of water, methanol and acetic acid(100) (85 : 15 : 1)

Flow rate: 1.0 mL/minute

(4) ***Baicalin of Scutellaria Root***—Weigh accurately the mass of NLT 20 sachets of Hyeonggaeyeongyotang Extract Granules and pulverize. Weigh accurately an amount equivalent to about 50 mg of baicalin. Perform the test as directed under the Assay of Scutellaria Root.

Packaging and storage Preserve in tight containers.

Imperata Rhizome
모근(茅根)

Imperatae Rhizoma

Imperata Rhizome is the rhizome of *Imperata cylindrica* Beauvois var. *koenigii* Durand et Schinz ex A. Camus (Gramineae), from which rootlets and scale leaves have been removed.

Description Imperata Rhizome is the rhizome, long and thin cylindrical, 30 cm to 60 cm in length and 2 mm to 4 mm in diameter. The outer surface occurs as yellowish white or pale yellow, slightly lustrous and longitudinally wrinkled. The nodes are distinct and slightly protruding, irregularly spaced, but usually between 1.5 cm and 3 cm. The body is light and the texture is somewhat brittle. The cut surface has a white cortex and several clefts. The central cylinder occurs as pale yellow, and the outer cortex is easily removed and detached. It occurs as almost odorless and tastes slightly sweet.

Under the microscope, the cross-section shows single-rowed, almost quadrangular, small epidermis cells, often containing silicon masses. The hypodermal fibers are in 1 to 3 rows and the cell walls are thickened and lignified. The epidermis is relatively broad with about 10 leaf-trace vascular bundles. The vascular bundles are closed and collateral and usually surrounded by clefts. The endodermal cells are thickened and contain silicon masses. Several closed collateral vascular bundles are scattered inside the central cylinder, the fibers of the vascular bundle sheath are annular and lignified, and the outer vascular bundles and fibers are interconnected to form a ring.

It occurs as odorless and the taste is light at first, but slightly sweet later.

Identification (1) Weigh 1.0 g of pulverized Imperata Rhizome, add 20 mL of hexane, allow the mixture to stand for 30 minutes with occasional shaking and then filter it. Evaporate the filtrate to dryness, dissolve the residue in 5 mL of chloroform, transfer 0.5 mL of this solution to a test tube and mix with 0.5 mL of acetic anhydride by shaking and add slowly 0.5 mL of sulfuric acid along the inner wall; a reddish brown color develops at the contact surface and the upper layer produces a blue-green to blue-purple color.

(2) Weigh 2 g of pulverized Imperata Rhizome, add 20 mL of a mixture of ethanol and water (95:5), sonicate for 1 hour and

filter. Concentrate the filtrate to 5 mL and use this solution as the test solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL of the test solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of hexane and acetone (9:1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS on the plate and heat at 105 ℃; the spot with the R_f value of about 0.3 exhibits a purple color.

Purity (1) ***Foreign matter***—(i) Rootlet and scaly leaf: Imperata Rhizome contains of rootlet and scaly leaf less than 3.0%.

(ii) Other foreign matter: The amount of foreign matter other than rootlets and scaly leaves is less than 1.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 5.0%.

Acid-insoluble ash NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Ipecac
토근(吐根)

Ipecacuanhae Radix et Rhizoma

Ipecac is the root and rhizome of *Cephaelis ipecacuanha* A. Richard or *Cephaelis acuminata* Karsten (Rubiaceae).

Ipecac contains NLT 2.0% of the total alkaloids [as emetine ($C_{29}H_{40}N_2O_4$: 480.64) and cephaeline ($C_{28}H_{38}N_2O_4$: 466.61)], calculated on the dried basis.

Description Ipecac is the root and rhizome. The root is thin and long cylindrical, 3 cm to 15 cm in length and 3 mm to 9 mm in diameter. The external surface occurs as gray, dark gray or reddish brown and in the shape of irregular nodal rings. Most are twisted and curved, sometimes branched. In the fractured surface, the cortex is easily separable from the xylem, the cortex occurs as grayish brown and the xylem occurs as pale brown. The thickness of the cortex is up to two-thirds of the diameter in the thickened part. The rhizome is cylindrical and scars of opposite leaves are observed.

Under a microscope, a transverse section of Ipecac reveals a cork layer, consisting of brown thin-walled cork cells. Parenchyma cells are filled with starch grains and sometimes with raphide bundles of calcium oxalate. In the cortex, sclerenchyma cells are absent. In the xylem, sclerenchymatous vessels and tracheids are arranged alternately with medullary rays containing starch grains.

Ipecac has a slight, characteristic odor and the taste is slightly bitter and unpleasant. The powder of Ipecac irritates the mucous membrane of the nose.

Identification Weigh 0.5 mg of pulverized Ipecac, add 2.5 mL of hydrochloric acid, allow to stand for 1 hour with occasional shaking, and filter. Collect the filtrate into an evaporation dish and add small pieces of chlorinated lime: circumference turns red.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm

(iv) Cadmium: NMT 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

Loss on drying NMT 12.0% (6 hours).

Ash NMT 5.0%.

Acid-insoluble ash NMT 2.0%.

Assay Weigh accurately about 0.5 g of pulverized Ipecac, transfer to a stoppered centrifuge tube, add 30 mL of 0.01 mol/L hydrochloric acid TS, shake for 15 minutes to mix, centrifuge, and take separately the supernatant. Repeat this procedure twice with the residue using 30 mL each of 0.01 mol/L hydrochloric acid TS. Combine all the extracts, add 0.01 mol/ L hydrochloric acid TS to make exactly 100 mL and use this solution as the test solution. Separately, weigh accurately about 10 mg of emetine hydrochloride RS (previously dried in a silica gel desiccator for 24 hours), dissolve in 0.01 mol/L hydrochloric acid TS to make exactly 100 mL, and use this solution as the standard solution. Pipet 10 μL of the test solution and the standard solution and perform the test as directed under the Liquid Chromatography according to the following operating conditions. Determine the peak areas, A_{Ta} and A_{Tb}, of emetine and cephaeline, respectively, in the test solution and the peak area, A_S, of emetine in the standard solution.

$$\text{Amount (mg) of total alkaloids (emetine and cephaeline)} = \text{Amount (mg) of emetine hydrochloride RS} \times \frac{A_{\mathrm{Ta}} + A_{\mathrm{Tb}} \times 0.971}{A_{\mathrm{S}}} \times 0.868$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 283 nm)

Column: A stainless column 4 mm to 6 mm in internal diameter and 10 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Column temperature: A constant temperature of about 50 ℃

Mobile phase: Dissolve 2 g of sodium 1-heptanesulfonate in 500 mL of water, add acetic acid (100) to adjust the pH to 4.0, and add 500 mL of methanol.

Flow rate: Adjust the flow rate so that the retention time of emetine is about 14 minutes.

System suitability

System performance: Dissolve 1 mg of emetine hydrochloride RS and 1 mg of cephaeline hydrofluoric acid in 0.01 mol/L hydrochloric acid TS to make 10 mL respectively. Proceed with 10 μL of this solution according to the above conditions; cephaeline and emetine are eluted in this order with clear separation of each peak.

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak areas of emetine is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Jujube
대추(大棗)

Zizyphi Fructus

Jujube is the ripe fruit of *Zizyphus jujuba* Miller var. *inermis* Rehder or *Zizyphus jujuba* Miller var. *hoonensis* T. B. Lee (Rhamnaceae).

Description Jujube is an ellipsoid or spherical fruit with a length of 2 cm to 3 cm and a diameter of 1 cm to 2 cm. The outer surface occurs as reddish brown to dark red, fine wrinkled and lustrous. Both ends are slightly dented, with a style scar at one end and a fruit stalk on the other. The epicarp is thin and leathery. The mesocarp is thick, dark grayish brown, spongy, soft and sticky. The endocarp is extremely hard, fusiform and divided into two loci containing flat and ovoid seeds, and the texture is firm. Jujube has a slight, characteristic odor and sweet taste.

Identification Weigh 1 g of pulverized Jujube, add 50 mL of ethyl acetate, sonicate for 1 hour, and cool. Then, filter it, concentrate the filtrate until it becomes 2 mL and use it as the test solution. Separately, dissolve 1 mg of oleanolic acid RS in 1 mL of ethanol and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of hexane, ethyl acetate and formic acid (15 : 5 : 0.5) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray sulfuric acid TS for spraying evenly on the plate and heat it at 105 °C; one of the several spots obtained from the test solution shows the same color and R_f value as the spot obtained from the standard solution.

Purity (1) ***Rancidity***—Jujube has no unpleasant, rancid odor and taste.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—Proceed as directed in "Jujube (dried)" described in [Attachment 4] Tolerances for Pesticide Residues for Agricultural Products provided in the Public Notice "Standards and Specifications for Food" of the Ministry of Food and Drug Safety (MFDS).

(4) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 3.0%.

Packaging and storage Preserve in well-closed containers.

Juncus Medulla
등심초(燈心草)

Junci Medulla

Juncus Medulla is the stem pith of *Juncus effusus* Lnné (Juncaceae).

Description Juncus Medulla is the stem pith, slenderly cylindrical like noodles, 30 cm to 60 cm in length and about 2 mm in diameter. The external surface occurs as a milky white to pale yellowish white color, flattened on pressing and with fine longitudinal wrinkles. Under a magnifying glass, the transverse section of the cut surface reveals numerous fine pits and a loose and light fractured surface like sponge.
Under a microscope, the transverse section of Juncus Medulla reveals the aerenchyma that occupies the entire area. Each cell is nearly quadrilateral or rectangular, splitting into several branches. The Lacuna of cells forms a triangular or quadrilateral shape.
It occurs as nearly odorless and has a plain taste.

Identification Weigh 1 g of pulverized Juncus Medulla, add 100 mL of methanol, heat after connecting with a reflux condenser on a water bath for 1 hour, filter, and evaporate the filtrate to dryness. Wash the residue with 2 mL of ether, dissolve the residue in 1 mL of anhydrous ethanol, use this solution as the test solution. With this solution, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL of the test solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of cyclohexane and ethyl acetate (10 : 7) as the developing solvent to a distance of about 10 cm, and air-dry the plate; Spray evenly dilute sulfonic acid TS on the plate and heat at 105 °C for 10 minutes; a violet spot appears at the R_f value of about 0.5.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 11.0%.

Ash NMT 5.0%.

Packaging and storage Preserve in well-closed containers.

Kalopanax Bark
해동피(海桐皮)

Kalopanacis Cortex

Kalopanax Bark is the bark of *Kalopanax pictus* Nakai (Araliaceae).

Description Kalopanax Bark is the long plate-like or semi-cylindrical bark, varying in length, and 1 mm to 4 mm in thickness. The external surface occurs as grayish white to grayish brown, coarse, with grayish black longitudinal clefts and transversely open patterns. Scattered yellow, round, and spot-like lenticels are observed, but not distinct. The cortex has nail-like spines, 1 cm to 3 cm in length, 1 cm to 1.7 in diameter at the base, longitudinally oblong. The inner bark with the spines fallen off occurs as yellow. The inner surface occurs as yellowish brown or purplish brown, smooth, with a distinct fine longitudinal pattern. The texture is hard, tough and difficult to be cut. The cut surface occurs as grayish brown on the outside, grayish yellow on the inside, very fibrous with distinct lamellae.
It has a slight aroma and bitter taste.

Identification (1) Weigh 0.5 g of pulverized Kalopanax Bark, add 5 mL of acetic anhydride, shake for 5 minutes to mix, and filter. Add slowly 1 mL of sulfuric acid to 2 mL of the filtrate; a reddish purple color develops at the zone of contact and when allowed to stand, the upper layer exhibits a green color.

(2) Weigh 1 g of pulverized Kalopanax Bark, add 10 mL of a mixture of methanol and water (7 : 3), sonicate for 20 minutes, filter, and use the filtrate as the test solution. Separately, weigh 1 mg of hederacoside C, dissolve in 1 mL of methanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of butanol, water and acetic acid (6 : 3 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly vanillin-sulfuric acid TS and heat at 105 °C; one of the several spots obtained from the test solution shows the same color and R_f value as the spot obtained from the standard solution.

Purity (1) ***Foreign matter***—Kalopanax Bark contains less than 1.0% of the cork layer and other foreign matter.
(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Cadmium: NMT 0.3 ppm.
(iv) Mercury: NMT 0.2 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(4) ***Sulfur dioxide***—NMT 30 ppm

Loss on drying NMT 9.0%.

Ash NMT 10.0%.

Extract content ***Dilute ethanol–soluble extract***—NLT 8.0%.

Packaging and storage Preserve in well-closed containers.

Kochia Fruit
지부자(地膚子)

Kochiae Fructus

Kochiae Fruit is the ripe fruit of *Kochia scoparia* Schrader(Chenopodiaceae).

Description Kochia Fruit is the fruit, round oblate, five-pointed star-shaped and 1 to 3 mm in diameter. The outside is covered with the persistent perianth, and the external surface occurs as a grayish green to pale brown color, surrounded by five membranous winglets. The center of the dorsal surface has a slightly prominent, dotted fruit stalk scar and 5 to 10 radial veins. When the perianth is stripped off, the translucent membranous pericarp is visible. The seed is flattened ovoid, about 1 mm in length, exhibiting a black color.
It has a slight, characteristic odor and a slightly bitter taste.

Identification Weigh 2 g of pulverized Kochia Fruit, add 20 mL of ethanol and 1.5 mL of hydrochloric acid, heat after connecting with a reflux condenser for 2 hour, and filter. Concentrate the filtrate until it becomes 5 mL, add 10 mL of water, transfer into a separatory funnel, and extract with 20 mL of petroleum ether. Evaporate the ether layer to dryness, dissolve the residue in 2 mL of ethanol, and use this solution as the test solution. Separately, weigh 5 mg of oleanolic acid RS, dissolve in 5 mL of ethanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 2 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of cyclohexane, acetone and ethyl acetate (5 : 2 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS on the plate and heat at 105 °C; the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 10.0%.

Ash NMT 10.0%.

Acid-insoluble ash NMT 5.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 15.0%.

Packaging and storage Preserve in well-closed containers.

Leonurus Herb
익모초(益母草)

Leonuri Herba

Leonurus Herb is the aerial part, collected before or on flowering of *Leonurus japonicus* Houttuyn (Labiatae).

Leonurus Herb contains NLT 0.05% of leonurine ($C_{14}H_{21}N_3O_5$: 311.33), calculated on the dried basis.

Description Leonurus Herb is the aerial part, composed of square stems and cauline leaves and flowers. Stems are 30 cm to 60 cm in length and 1 mm to 5 mm in diameter. The external surface occurs as a yellowish green to greenish brown color, densely covered with white and short hairs. The fractured surface of the stem reveals the huge white pith, of which texture is pliable. Leaves are opposite, palmately ternate to lobed-ternate, the upper surface occurs as a pale yellow color, and the lower surface is densely pubescent with white hairs, exhibiting a grayish green color. Flowers are pubescent verticillately on the axil, exhibiting a pale green to greenish brown color, and the calyx is cylindrical, usually 5-lobed at the apex.
It has a slight, characteristic odor and a bitter and astringent taste.

Identification Weigh 3 g each of pulverized Leonurus Herb and Leonurus herb RMPM, add 30 mL each of methanol, sonicate for 1 hour to extract, filter, and use these filtrates as the test solution and the Leonurus herb RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution of Leonurus herb RMPM on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of butanol, formic acid and water (4 : 1 : 0.5) to a distance of about 10 cm, and air-dry the plate. Spray evenly Bismuth potassium iodide TS to the plate; the color and the R_f values of several spots obtained from the test solution are the same as those of the spots obtained from the Leonurus herb RMPM standard solution. Of these, the spot of stachydrine hydrochloride appears at the R_f value of about 0.15.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 13.0% (6 hours).

Ash NMT 10.0%.

Acid-insoluble ash NMT 2.0%.

Extract content *Dilute ethanol-soluble extract*—NLT 8.0%.

Assay Weigh accurately about 1 g of pulverized Leonurus Herb, add 50 mL of diluted methanol (7 in 10), sonicate for 1 hour to extract, filter, and use the filtrate as the test solution. Separately, weigh accurately about 10 mg of leonurine RS and dissolve in diluted methanol (7 in 10) to make exactly 100 mL. Pipet 10 mL of this solution, add diluted methanol (7 in 10) to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions, and determine the peak areas, A_T and A_S, of the test solution and the standard solution, respectively.

$$\text{Amount (mg) of leonurine } (C_{14}H_{21}N_3O_5) = \text{Amount (mg) of leonurine RS} \times \frac{A_T}{A_S} \times \frac{1}{20}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 270 nm)

Column: A stainless steel column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Column temperature: A constant temperature of about 30 ℃

Mobile phase: Control the mobile phases A and B stepwise as directed in the following table or via the concentration gradient.

Mobile phase A: A mixture of diluted trifluoroacetic acid (1 in 1000) and methanol (95 : 5)

Mobile phase B: A mixture of methanol and diluted trifluoroacetic acid (1 in 1000) (95 : 5)

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)
0	75	25
30	45	55
35	75	25

Flow rate: 1.0 mL/min

System suitability

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak areas of leonurine is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Licorice
감초(甘草)

Glycyrrhizae Radix et Rhizoma

Licorice is the root and rhizome of *Glycyrrhiza uralensis* Fisher, *Glycyrrhiza glabra* Linné, *Glycyrrhiza inflata* Batal. Or *Glycyrrhiza korshinskyi* Grig. (Leguminosae) with or without the periderm.

Licorice contains NLT 2.5% of glycyrrhizic acid ($C_{42}H_{62}O_{16}$: 822.93) and NLT 0.7% of liquiritigenin ($C_{15}H_{12}O_4$: 256.27), calculated on the dried basis.

Description (1) ***Glycyrrhiza uralensis***—Licorice from *Glycyrrhiza uralensis* consists of the root and the rhizome. The root is cylindrical, 25 – 100 cm in length and 5 – 35 mm in diameter. The external surface occurs as reddish brown to yellowish brown with distinct longitudinal wrinkles, dents and lenticels, and has

sparse, thin root scars. The texture is hard. The transverse section is fibrous, yellowish white and very powdery, with distinct rings of cambium; the medullary rays are radiated and often have clefts. The rhizome is cylindrical with externally bud scars, and the center of the transverse section has the pith.
Under a microscope, the transverse section of unpeeled Licorice from *Glycyrrhiza uralensis* reveals several layers of yellowish brown cork layers, inside of which the cork cortex layer consisting of 1 – 3 layers is present. Groups of bast fiber bundles with thickened, but incompletely lignified walls are present in the cortex, and these fiber bundles mainly form rows of crystal-bearing cells. The phloem is clearly visible, but in old roots, it is not clearly visible due to overall deterioration and not being close to the cambium. The medullary rays are radial and penetrate through the cambium to reach the cortex, and the medullary ray cells are filled with starch grains. The vessels are large and radiated between medullary rays solitarily or in groups. Xylem fiber bundles surrounded by crystal-bearing cells are scattered between vessels. The rhizome has the pith, and the parenchyma cells in the cortex and the xylem contain solitary crystals of calcium oxalate and starch grains. Peeled Licorice sometimes lacks the periderm or part of the phloem.

(2) ***Glycyrrhiza glabra***—Licorice from *Glycyrrhiza glabra* Linné consists of the root and the rhizome. It has a woody texture and is thick, hard and sometimes branched. The external peel is not coarse and is largely grayish brown. The lenticel is slender and not distinct.

(3) ***Glycyrrhiza inflata***—Licorice from *Glycyrrhiza inflat*e Batal. Consists of the root and the rhizome. Its texture is relatively tough and is sometimes branched. The external peel is rough and largely grayish brown. The lenticel is slender and not distinct.

(4) ***Glycyrrhiza korshinskyi Grig***—Licorice from *Glycyrrhiza korshinskyi* Grig. Consists of the root and the rhizome. It has a woody texture, which is hard, and is sometimes branched. The external peel is not coarse and occurs as a reddish brown or grayish brown color with distinct lenticels.
Licorice has a slight, characteristic odor and a sweet taste.

Identification Weigh 2 g of pulverized Licorice, add 10 mL of methanol, sonicate for 5 minutes, filter, and use the filtrate as the test solution. Separately, weigh 5 mg of glycyrrhizic acid RS, dissolve in 1 mL of methanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 2 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel (with a fluorescent agent) for thin layer chromatography. Next, develop the plate with a mixture of ethyl acetate, water, formic acid and acetic acid (100) (15 : 2 : 1 : 1) as the developing solution to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 254 nm); the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Difenoconazole: NMT 0.05 ppm.
(iv) Methoxychlor: NMT 1 ppm.
(v) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(vi) Azoxystrobin: NMT 0.05 ppm.
(vii) Aldrin: NMT 0.01 ppm.
(viii) Endrin: NMT 0.01 ppm.
(ix) Acetamiprid: NMT 0.1 ppm
(x) Imidacloprid: NMT 0.1 ppm.
(xi) Chlorothalonil: NMT 0.05 ppm.
(xii) Thiamethoxam: NMT 0.1 ppm.
(xiii) Fenpyroximate: NMT 0.1 ppm.
(xiv) Pymetrozine: NMT 0.5 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.
(4) ***Mycotoxins***—Total aflatoxins (sum of aflatoxins B_1, B_2, G_1 and G_2): NMT 15.0 ppb (However, aflatoxin B_1 is NMT 10.0 ppb).

Loss on drying NMT 12.0% (6 hours).

Ash NMT 7.0%.

Acid-insoluble ash NMT 2.0%.

Assay (1) ***Glycyrrhizic acid***—Weigh accurately about 0.5 g of powdered Licorice, add 40 mL of diluted ethanol (7 in 10), sonicate for 1 hour to extract, and filter. To the residue, add 30 mL of diluted ethanol (7 in 10) and proceed in the same manner. Combine all the filtrates, add diluted ethanol (7 in 10) to make exactly 100 mL, and use this solution as the test solution. Separately, weigh accurately about 20 mg of glycyrrhizic acid RS (previously dried in a desiccator with silica gel for 24 hours), dissolve in diluted ethanol (7 in 10) to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 20 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and calculate the peak areas, A_T and A_S, from the test solution and the standard solution, respectively.

$$\text{Amount (mg) of glycyrrhizic acid } (C_{42}H_{62}O_{16}) = \text{Amount (mg) of glycyrrhizic acid RS} \times \frac{A_T}{A_S}$$

Operating conditions
Detector: An ultraviolet absorption photometer (wavelength: 254 nm)
Column: A stainless steel column about 4 – 6 mm in internal diameter and 15 – 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 – 10 μm in particle diameter).
Mobile phase: A mixture of diluted acetic acid (1 in 15) and acetonitrile (3 : 2)
Flow rate: Adjust the flow rate so that the retention time of glycyrrhizic acid is about 10 minutes.
System suitability
System performance: Dissolve 5 mg of glycyrrhizic acid RS and 1 mg of propylparaben in diluted ethanol (7 in 10) to make 20 mL. Proceed with 20 μL of this solution under the above conditions; glycyrrhizic acid and propylparaben are eluted in this order with clear separation of each peak.
System repeatability: Repeat the test 6 times with 20 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak areas of glycyrrhizic acid is NMT 1.5%.

(2) ***Liquiritigenin***—Weigh accurately about 0.5 g of pulverized Licorice, add 100 m L of 2 mol/L hydrochloric acid, connect with a reflux condenser, and heat at 90 °C for 1 hour. To the extract, add 100 mL of dichloromethane, connect with a reflux

condenser, and heat at 40 °C for 30 minutes. Transfer the extract to a separatory funnel and take the dichloromethane layer. Add 50 mL of dichloromethane, shake, and take the dichloromethane layer. Repeat this process 2 times. Collect and combine the dichloromethane layers, vacuum-concentrate, dissolve in 50 mL of methanol, and use this solution as the test solution. Weigh accurately about 10 mg of liquiritigenin RS (previously dried in a desiccator with silica gel for 24 hours), add methanol to make exactly 50 mL, and use this solution as the standard solution. Perform the test with 20 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and calculate the peak areas, A_T and A_S, from the test solution and the standard solution, respectively.

$$\text{Amount (mg) of liquiritigenin } (C_{15}H_{12}O_4) = \text{Amount (mg) of liquiritigenin RS} \times \frac{A_T}{A_S}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 276 nm)

Column: A stainless steel column about 4 – 6 mm in internal diameter and 15 – 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 – 10 μm in particle diameter).

Mobile phase: A mixture of diluted acetic acid (1 in 100) and acetonitrile (75 : 25)

Flow rate: 1.0 mL/min

System suitability

System repeatability: Repeat the test 6 times with 20 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak areas of liquiritigenin is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Glycyrrhiza Extract
감초엑스

Glycyrrhiza Extract contains NLT 4.5% of glycyrrhizic acid ($C_{42}H_{62}O_{16}$: 822.93).

Method of preparation Weigh 1 kg of offine cutting Licorice, add 5 L of Tap Water or Purified Water, macerate for 2 days, and filter the macerated solution through a cotton-cloth filter. Then, add another 3 L of Tap Water or Purified Water, macerate for 12 hours, and filter the macerated solution through a cotton-cloth filter. Evaporate the combined filtrates until the whole volume becomes 3 L. After cooling, add 1000 mL of ethanol and allow to stand in a cold place for 2 days. Filter and evaporate the filtrate to a semiliquid extract.

Description Glycyrrhiza Extract occurs as brown to blackish brown, semiliquid extract and has a characteristic odor and a sweet taste. It dissolves in water, forming a clear or slightly turbid solution.

Identification Weigh 0.8 g of Glycyrrhiza Extract, add 10 mL of a mixture of ethanol and water (7 : 3), shake for 2 minutes to mix, centrifuge, and use the supernatant as the test solution. Hereinafter, proceed as directed in the Identification under Licorice.

Purity (1) ***Insoluble matter***—Dissolve 2.0 g of Glycyrrhiza Extract in 18 mL of water, filter, and add 5 mL of ethanol to the filtrate; the resulting solution is clear.

(2) ***Heavy metals***—Total heavy metals: NMT 30 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

Assay Weigh accurately 0.15 g of Glycyrrhiza Extract, place in a glass-stoppered tube for centrifugation, add 25 mL of dilute ethanol, and heat at 50 °C for 30 minute with occasional shaking to mix. After cooling, centrifuge and take the supernatant. To the residue, add 20 mL of dilute ethanol and proceed in the same manner. Combine the extracts, add dilute ethanol to make exactly 100 mL, and use this solution as the test solution. Separately, weigh accurately about 20 mg of glycyrrhizic acid RS (previously dried in a desiccator with silica gel for 24 hours), dissolve in dilute ethanol to make exactly 100 mL, and use this solution as the standard solution. Proceed as directed in the Assay (1) Glycyrrhizic acid under Licorice.

$$\text{Amount (mg) of glycyrrhizic acid } (C_{42}H_{62}O_{16}) = \text{Amount (mg) of glycyrrhizic acid RS} \times \frac{A_T}{A_S}$$

Packaging and storage Preserve in tight containers.

Crude Glycyrrhiza Extract
감초조엑스

Licorice Extract

Crude Glycyrrhiza Extract contains NLT 6.0% of glycyrrhizic acid ($C_{42}H_{62}O_{16}$: 822.93).

Method of preparation Boil coarse powder of Licorice in Tap Water or Purified Water, filter the solution under pressure, and evaporate the filtrate.

Description Crude Glycyrrhiza Extract is a lustrous, dark yellowish red to blackish brown plate, rod or mass. It is dissolved in water with turbidity. It is brittle when it is cold. The fractured surface occurs as a dark yellowish red color and is lustrous like a seashell. It is flexible when it is warm.
It has a characteristic odor and a sweet taste.

Identification Weigh 0.6 g of Crude Licorice Extract, add 10 mL of a mixture of ethanol and water (7 : 3), and dissolve by warming, if necessary. After cooling, centrifuge and use the supernatant as the test solution. Hereinafter, proceed as directed in the Identification under Licorice.

Purity (1) ***Insoluble substances***—Boil 5.0 g of pulverized Crude Glycyrrhiza Extract in 100 mL of water. After cooling, filter the mixture through a filter paper of known mass, wash the residue with water, and dry the residue at 105 ℃ for 5 hours; the amount of the residue is NMT 1.25 g

(2) ***Starch***—Weigh about 1 g of pulverized Crude Glycyrrhiza Extract, add water to make 20 mL, shake well to mix, and filter. Under a microscope, the residue on the filter paper contains

no starch grains.

(3) ***Foreign matter***—The filtrate obtained in (1) does not have any strong bitter taste.

(4) ***Heavy metals***—Total heavy metals: NMT 30 ppm.

(5) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

Ash NMT 12.0% (1 g, proceed as directed in the Ash under the Crude Drugs Test).

Assay Weigh accurately about 0.15 g of Crude Glycyrrhiza Extract and proceed as directed in the Assay under Glycyrrhiza Extract.

Amount (mg) of glycyrrhizic acid ($C_{42}H_{62}O_{16}$)
= Amount (mg) of glycyrrhizic acid RS× $\frac{A_T}{A_S}$

Packaging and storage Preserve in tight containers.

Lindera Root
오약(烏藥)

Linderae Radix

Lindera Root is the root of *Lindera strychnifolia* Fernandez-Villar (Lauraceae).

Description Lindera Root is the root, spindle-shaped, slightly curved, constricted in the middle to form a bead-like shape, 10 cm to 15 cm in length and 10 mm to 25 mm in diameter. The external surface occurs as a yellowish brown to brown color, with lateral wrinkles and sporadically scattered scars of rootlets. The texture is hard and difficult to break. The fractured surface is powdery. Under a magnifying glass, the transverse section occurs as a yellowish white to light yellowish brown color, revealing radiating medullary rays and annual rings with a relatively darker color in the center.

Under a microscope, the transverse section of Lindera Root reveals a cork layer partially consisting of cork stone cells and the oil cells and fibers in the cortex. In the xylem, vessels, xylem fibers and medullar rays are arranged alternately. Parenchyma cells in the cortex and the xylem contain sandy and columnar crystals of calcium oxalate and starch grains. Starch grains consist of simple grains, 1 μm to 15 μm in diameter, and compound starch grains made of 2 to 4 grains.

It has an aroma and a slightly bitter and pungent taste with a refreshing sensation.

Identification (1) Weigh 1 g of pulverized Lindera Root, add 10 mL of chloroform and 1 mL of ammonia TS, allow to stand for 1 hour with occasional shaking, and filter. Transfer the filtrate into a separatory funnel, add 2 mL of dilute hydrochloric acid, shake well to mix, and allow to stand. Then, take the aqueous layer and add 1 to 2 drops of Mayer's reagent; the resulting solution becomes turbid in white.

(2) Weigh 1 g of pulverized Lindera Root, add 30 mL of petroleum ether, allow to stand for 30 minutes, sonicate for 10 minutes to extract, filter, and evaporate the filtrate to dryness. Dissolve the residue in 1 mL of ethyl acetate and use this solution as the test solution. Separately, dissolve 1.5 mg of linderane RS in 2 mL of ethyl acetate and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate using a mixture of hexane and acetone (9 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS for spray on the plate and heat at 105 ℃; the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 1.0 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 14.0% (6 hours).

Ash NMT 2.5%.

Extract content ***Dilute ethanol-soluble extract***—NLT 7.0%.

Packaging and storage Preserve in well-closed containers.

Linseed
아마인(亞麻仁)

Lini Semen

Linseed is the ripe seed of *Linum usitatissimum* Linné (Linaceae).

Description Linseed is the seed, flattened ovoid, bluntly round at one end and pointed and slightly flat and oblique at the other. It is 4 mm to 6 mm long and 2 mm to 3 mm wide. The outer surface occurs as reddish brown to grayish brown, slightly smooth and lustrous. The hilum is located at the slightly indented part of the pointed end, the dorsal ridge of the seed is pale brown and is located on the edge of one side.

Under the microscope, the cross-section shows relatively large epidermis cells, which are almost rectangular. The cell walls contain mucilage that expands on contact with water, making the lamellae distinct. The outside is covered by a cuticular layer. The hypodermis consists of 1 to 5 rows of parenchyma cells and the cell walls are somewhat thick. The fibrous layer consists of a row of dense fibrous cells. The degenerate layer has no clear cell boundary. The pigment layer consists of a layer of flat parenchyma cells containing reddish brown substances inside. The endosperm and cotyledon cells are polygonal containing fatty oil, aleurone granules and 1 to 2 pseudocrystals.

It occurs as almost odorless and produces mucilage when immersed in water.

Identification Weigh 0.5 g of pulverized Linseed, add 5 mL of dichloromethane, perform cold extraction for 20 minutes, and filter. Use the filtrate as the test solution. With this solution, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL of the test solution on the thin layer plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of hexane, ether and acetic acid (7 : 3 : 0.1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Expose the plate to iodine vapor; the test solution shows 2 yellow spots at R_f values of 0.3 and 0.7.

Purity (1) ***Foreign matter***—Linseed contains less than 2.0% of foreign matter.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 5.0%.

Extract content ***Ether-soluble extract***—NLT 30.0%.

Packaging and storage Preserve in well-closed containers.

Liriope Tuber
맥문동(麥門冬)

Liriopis seu Ophiopogonis Tuber

Liriope Tuber is the tuber of the *Liriope platyphylla* Wang et Tang or *Ophiopogon japonicus* Ker-Gawler (Liliaceae).

Description (1) ***Liriope platyphylla***—Liriope Tuber from *Liriope platyphylla* consists of the tuber, with the shape of long square column or round square column, 12 mm to 40 mm in length and 4 mm to 9 mm in diameter. The external surface occurs as a pale yellow color, with longitudinal wrinkles. The texture is soft, but tough. The fractured surface occurs as a yellowish white color, slightly transparent. The central cylinder is a thin, strong, tough woody core.
Under a microscope, the transverse section of Liriope Tuber from *Liriope platyphylla* reveals the epidermis cells in a rectangular to polygonal shape, consisting of cells of a single row. The exodermal cells are lined in 1 to 2 rows, bigger than the epidermis cells, and lignified. The cortex is very broad, composed of about 30 rows of cells containing a mucilaginous substance and calcium oxalate raphide bundles. The outer layer of the endodermis is composed of 1 to 3 rows of stone cells. The endodermal cells have evenly thickened cell walls and have passage cells. The vascular bundles are the radial type, and there are 12 to 20 phloem bundles, each of which is located in the bow-like dents among the xylem bundles.
The lignified tissue of the xylem bundles' are connected to each other, forming a ring pattern. The pith is small.
Liriope Tuber from *Liriope platyphylla* has a slight, characteristic odor and a slightly sweet and sticky taste.

(2) ***Ophiopogon japonicus***—Liriope Tuber from *Ophiopogon japonicus* consists of the tuber, with a shape of long square column or fusiform, 10 mm to 25 mm in length, 3 mm to 9 mm in diameter, and pointy at one end and slightly rounded at the other. The external surface occurs as a pale yellow to pale yellowish brown color with longitudinal wrinkles of various sizes. Liriope Tuber from *Ophiopogon japonicus* is easy to break, and the fractured surface occurs as a yellowish white color, slightly transparent. The central cylinder is a thin, strong, tough woody core.
Under a microscope, the transverse section of Liriope Tuber from *Ophiopogon japonicus* reveals the epidermis cells in a rectangular to polygonal shape, consisting of cells of a single row. The exodermal cells are lined in 3 to 5 rows, with lignified cell walls. The cortex is very broad, composed of about 14 to 27 rows of cells containing a mucilaginous substance and calcium oxalate raphide bundles. The outer layer of the endodermis is composed of stone cells in a single row. The endodermal cells have evenly thickened cell walls and have passage cells. The central cylinder is very small, and the central cylinder sheath is composed of 1 to 2 rows of parenchyma cells. The vascular bundles are the radial type, and there are 13 to 22 phloem bundles, each of which is located in the bow-like dents among the xylem bundles. The lignified tissue of the xylem bundles is connected to each other, forming a ring pattern. The pith is small.
Liriope Tuber from *Ophiopogon japonicus* has a slight, characteristic odor and a slightly sweet and sticky taste.

Identification Weigh 2 g each of pulverized Liriope Tuber and Liriope tuber RMPM, add 20 mL of methanol, and allow to stand for 3 hours. Then, sonicate each for 30 minutes to extract, filter, and evaporate each filtrate to dryness. Dissolve each residue in 1 mL of methanol and use these solutions as the test solution and the Liriope tuber RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Liriope tuber RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate using a mixture of toluene, methanol and acetic acid (100) (800 : 50 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS for spray on the plate and heat at 105 °C; the color and the R_f values of the several spots obtained from the test solution are the same as those of the spots obtained from the Liriope tuber RMPM standard solution. Of these spots, a reddish brown spot appears at the R_f value of about 0.3.

Purity (1) ***Foreign matter***—Liriope Tuber contains less than 1.0% of rootlets.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(vi) Pendimethalin: NMT 0.2 ppm.

(6) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 3.0%.

Packaging and storage Preserve in well-closed containers.

Lithospermum Root
자근(紫根)

Lithospermi Radix

Lithospermum Root is the root of *Lithospermum erythrorhizon* Siebold et Zuccarini, *Arnebia euchroma* Johnst or *Arnebia guttata* Bunge (Boraginaceae).

Description (1) ***Lithospermum erythrorhizon***—Lithospermum Root from *Lithospermum erythrorhizon* is the root, slightly slender and long conical, often branched, 6 to 10 cm in length and 5 to 15 mm in diameter. The external surface occurs as a dark violet to purplish brown color, and the cortex is coarse, easily peeled by thin. Lithospermum Root is mostly with twisted, deep longitudinal furrows, which sometimes reach to the xylem. Sometimes, remains of stems are present at the crown. Lithospermum Root is easily broken, and the fractured surface is granular with numerous clefts. Under a magnifying glass, the transverse section reveals the cortex, of which outside occurs as a dark violet color and of which inside occurs as a pale brown color, with an irregular arrangement, and the xylem exhibiting a yellowish color. The center of the crown is often cracked and the surrounding part occurs as a reddish purple color.
Lithospermum Root from *Lithospermum erythrorhizon* has a slight, characteristic odor and a slight sweet taste.

(2) ***Arnebia euchroma***—Lithospermum Root from *Arnebia euchroma* is the root, irregular, cylindrical, mostly twisted, 7 to 20 cm in length and 10 to 25 mm in diameter. The external surface occurs as a reddish purple to purplish brown color, and the cortex is loose, bar-shaped, typically overlapped with ten layers and easily peeled off. The apex sometimes bears branched remains of stems. The texture is loose, soft and light. Under a magnifying glass, the transverse section reveals an uneven surface and the relatively small xylem, exhibiting a yellowish white to yellow color.
Lithospermum Root from *Arnebia euchroma* has a characteristic odor and a slight bitter and astringent taste.

(3) ***Arnebia guttata***—Lithospermum Root from *Arnebia guttata* is the root, conical to cylindrical, twisted, 6 to 20 cm in length and 15 to 40 mm in diameter. The crown is usually large, with the apex bearing one or more remains of stems, and covered with short and stiff hairs. The external surface occurs as a reddish purple or dark violet color, and the cortex is mostly thin, typically overlapped with several layers and easily peeled off. The texture is hard, fragile and easily cut. Under a magnifying glass, the transverse section reveals a relatively clear surface, the cortex exhibiting a reddish purple color and the relatively small xylem, exhibiting a yellowish white color.
Lithospermum Root from *Arnebia guttata* has a characteristic odor and an astringent taste.

Identification (1) Weigh 0.5 g of pulverized Lithospermum Root and heat in a test tube; a red vapor is produced, which condenses into reddish brown oil drops on the upper wall of the tube.

(2) Weigh 0.5 g of pieces or powder of Lithospermum Root, add 1 mL of ethanol, and shake to mix; the resulting solution exhibits a red color. To this solution, add 1 drop of sodium hydroxide TS; the red color changes to a bluish purple color. To this solution, add l to 2 drops of dilute hydrochloric acid; the resulting solution exhibits a red color.

(3) Weigh 0.5 g of pulverized Lithospermum Root, add 5mL of ethanol, shake for 30 minutes to mix, and filter. Vacuum-concentrate the filtrate at the temperature not exceeding 40 ℃, then add 1 mL of ethanol, and use this solution as the test solution. With this solution, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL of the test solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of ethyl acetate and ethanol (3 : l) to a distance of about 10 cm, and air-dry the plate; a reddish purple spot appears at the R_f value of about 0.75.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(vi) Thifluzamide: NMT 1.0 ppm.
(vii) Pencycuron: NMT 1.0 ppm.
(viii) Hexaconazole: NMT 0.2 ppm
(3) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 11.0%.

Acid-insoluble ash NMT 3.5%.

Packaging and storage Preserve in well-closed containers.

Longan Arillus
용안육(龍眼肉)

Longan Arillus

Longan Arillus is the aril of *Dimorcarpus longan* Loureiro (Sapindaceae).

Description Longan Arillus is the aril, in the shape of longitudinally broken and irregular thin slices, frequently several slices agglutinated, Longan Arillus is 2 cm to 4 cm in length, 1 cm to 2 cm in width and 2 mm to 4 mm in thickness. The external surface occurs as a dark reddish brown to blackish brown color and is semi-translucent. One side is wrinkled and uneven, while the other side is lustrous, with longitudinal wrinkles. The texture is soft and sticky.
It has a slight, characteristic odor and a sweet taste.

Identification Weigh 1 g of Longan Arillus, add 10 mL of water, shake well, and filter. To 3 mL of the filtrate, add 3 mL of Fehling's TS and warm on a water bath; a red precipitate is formed.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2

ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 15.0%.

Ash NMT 5.0%.

Packaging and storage Preserve in well-closed containers.

Longgu
용골(龍骨)

Fossilia Ossis Mastodi

Longgu is the ossified bone of large mammals, mainly composed of calcium carbonate.

Description Longgu is the ossified bone of large mammals in the shape of irregular masses, fragments or occasionally cylindrical masses. The external surface occurs as a pale grayish white color, sometimes scattered with grayish black or yellowish brown spots. The outer part consists of a layer, dense in texture and 2 mm to10 mm in thickness, while the inner part is made of pale brown porous substances. The texture is heavy and hard, but somewhat fragile. When crushed, it reduced to pieces and powder.

It occurs as odorless, tasteless and strongly adhesive to the tongue on licking.

Identification (1) Weigh 0.5 g of pulverized Longgu and dissolve in 10 mL of dilute hydrochloric acid; the resulting solution generates gas and becomes turbid in pale brown. Pass the gas through calcium hydroxide TS; a white precipitate is formed.

(2) The turbid solution, obtained in (1), has a characteristic odor. Filter this solution and add ammonia TS to the filtrate to neutralize; the resulting solution responds to the Chemical Identification reactions for calcium salt.

(3) Weigh 0.1 g of pulverized Longgu, dissolve in 5 mL of nitric acid by warming, and add ammonium molybdate TS; a yellow precipitate is formed.

Purity (1) ***Heavy metals***—Weigh 2 g of pulverized Longgu, add 5 mL of water, shake to mix, carefully add 6 mL of hydrochloric acid, and evaporate to dryness on a water bath. Dissolve the residue in 50 mL of water and filter. To 25 mL of the filtrate, add 2 mL of dilute acetic acid, 1 drop of ammonia TS and water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution as follows: evaporate 3 mL of hydrochloric acid to dryness on a water bath, add 2 mL of dilute acetic acid and 2.0 mL of lead standard solution, and add water to make 50 mL (NMT 20 ppm).

(2) ***Arsenic—***Weigh 0.2 g of pulverized Longgu and perform the test according to Method 2 (NMT 10 ppm).

Packaging and storage Preserve in well-closed containers.

Lonicera Flower
금은화(金銀花)

Lonicerae Flos

Lonicera Flower is the flower bud or flower starting to bloom of *Lonicera japonica* Thunberg (Caprifoliaceae).

Description Lonicera Flower is the flower bud or flower just starting to bloom. The flower bud is small clavate or conical in shape and the flower is lip-shaped. It is 15 – 35 mm in length and about 3 mm in diameter in the upper part and 1.5 mm in diameter in the lower part. The external surface occurs as a yellowish white or greenish white color, gradually darken on storage. Under a magnifying glass, pale brownish hair is densely pubescent, the calyx is green, 5-lobed at the apex, and the lobes are pubescent, about 2 mm in length. There are 5 stamens, one pistil and a glabrous ovary.

It has a slight, characteristic odor and a plain and slightly bitter taste.

Identification (1) Weigh 0.5 g of pulverized Lonicera Flower, add 10 mL of water, heat to boil, and filter. To the filtrate, add 1 to 2 drops of Iron(III) chloride TS; the resulting solution exhibits a navy blue color.

(2) Weigh 2.0 g of pulverized Lonicera Flower, add 10 mL of ethanol, heat to boil for 2 minutes on a water bath, and filter. To the filtrate, add 0.1 g of magnesium and 2 to 3 drops of hydrochloric acid: the resulting solution exhibits a yellowish brown to reddish brown color.

(3) Weigh 0.1 g of pulverized Lonicera Flower and Lonicera flower RMPM, add 10 mL each of diluted ethanol (7 in 10), sonicate for 60 minutes to extract, filter, and use these solutions as the test solution and the Lonicera flower RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 2 μL each of the test solution and the Lonicera flower RMPM standard solution on the thin-layer chromatographic plate made of silica gel (with fluorescent agent) for thin layer chromatography. Next, develop the plate with a mixture of ethyl acetate, methanol and water (8:2:1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wave- length: 365 nm); the color and the R_f values of several spots obtained from the test solution and the Lonicera flower RMPM standard solution are the same. Of these spots, the spot of chlorogenic acid appear at the R_f values of about 0.3.

Purity (1) ***Foreign matter—Stems and leaves***: Lonicera Flower contains less than 5.0% of stems and leaves

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 15.0% (6 hours).

Ash NMT 9.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 16.0%.

Packaging and storage Preserve in well-closed containers.

Lonicera Leaf and Stem
인동(忍冬)

Lonicerae Folium et Caulis

Lonicera Leaf and Stem is the leaves and climbing stems of *Lonicera japonica* Thunberg (Caprifoliaceae).

Lonicera Leaf and Stem contains NLT 0.1% of loganin ($C_{17}H_{26}O_{10}$: 390.38), calculated on the dried basis.

Description Lonicera Leaf and Stem is the leaves and climbing stems. The leaf is round and entire, 3 cm to 7 cm in length and 1 cm to 3 cm in width, with a short petiole. The upper surface occurs as a greenish brown color, while the lower surface occurs as a pale grayish green color. Under a magnifying glass, both surfaces are covered with soft hairs. The stem is long cylindrical, frequently branched, usually forming tangled bundles and 1.5 mm to 6 mm in diameter. The external surface occurs as a reddish brown to dark brown color, sometimes a grayish green color. The epidermis is easily peeled and fallen off. There are many nodes on the branch, with an internodal distance of 6 cm to 9 cm. The texture is fragile, making it easy to cut. The cut surface occurs as a yellowish white color, hollow in the middle.

Under a microscope, the transverse section of Lonicera Leaf and Stem reveals the medullary rays in the stem, consisting of 1 to 2 rows of cells. The vessels are up to about 160 μm in diameter and contain yellowish brown or reddish brown substances. The xylem fiber is polygonal, and the cell wall is very thick. The xylem parenchyma cells have very thick and lignified cell walls, sometimes containing prismatic crystals of calcium oxalate. The parenchyma cells in the pith are irregular polygonal, varying in size, of which walls are slightly lignified and pitted.

It has a slight, characteristic odor and a slightly astringent taste, followed by a bitter taste.

Identification Weigh 1 g each of Lonicera Leaf and Stem and Lonicera leaf and stem RMPM, add 10 mL of diluted methanol (1 in 2), sonicate for about 10 minutes to extract, filter, and use the filtrates as the test solution and the Lonicera leaf and stem RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Lonicera leaf and stem RMPM standard solution on the plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of ethyl acetate, methanol and formic acid (96 : 10 : 0.7) to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS and heat at 105 °C; the color and the R_f values of the several spots obtained from the test solution are the same as those of the spots obtained from the Lonicera leaf and stem RMPM standard solution. Of these, the spot of loganin appears at the R_f value of about 0.25 from both the test solution and the Lonicera leaf and stem RMPM standard solution.

Purity (1) ***Foreign matter***—The stems of over 5 mm in diameter is not contained in Lonicera Leaf and Stem.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 12.0% (6 hours).

Ash NMT 9.0%.

Acid-insoluble ash NMT 1.0%.

Extract content *Dilute ethanol-soluble extract*—NLT 12.0%.

Assay Weigh accurately about 1.0 g of pulverized Lonicera Leaf and Stem, add 10 mL of diluted methanol (7 in 10), sonicate for 30 minutes to extract, filter, and use the filtrate as the test solution. Separately, weigh accurately about 10 mg of loganin RS, dissolve in methanol to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under Liquid Chromatography according to the following operating conditions, and determine the peak areas, A_T and A_S, of the test solution and the standard solution, respectively.

$$\text{Amount (mg) of loganin } (C_{17}H_{26}O_{10}) = \text{Amount (mg) of loganin RS} \times \frac{A_T}{A_S} \times \frac{1}{10}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 254nm)

Column: A stainless steel column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Mobile phase: Control the mobile phases A and B stepwise as directed in the following table or via the concentration gradient.

Mobile phase A: A mixture of water, methanol and formic acid (90 : 10 : 0.1)

Mobile phase B: A mixture of methanol, water and formic acid (90 : 10 : 0.1)

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)
0	100	0
15	70	30
25	30	70
30	30	70
35	100	0

Flow rate: 1.0 mL/min

Packaging and storage Preserve in well-closed containers.

Lycium Fruit
구기자(枸杞子)

Lycii Fructus

Lycium Fruit is the dried fruit of *Lycium chinense* Miller or *Lycium barbarum* Linné (Solanaceae).

Lycium Fruit, when dried, contains NLT 0.5% of betaine ($C_5H_{11}NO_2$: 117.15).

Description Lycium Fruit is the fruit, nearly fusiform or elliptical, 6 – 20 mm in length and 3 – 10 mm in diameter. The external surface occurs as a red to dark red color, with a scar of the pistil stalk like a small bump at the end and a white scar of the fruit stalk on the base. The pericarp is soft, tough and crumpled. The sarcocarp is pulpy, soft and tender. There are 20 to 50 seeds inside. The seed is kidney-shaped, nearly flat, about 2 mm in length and 1 – 2 mm in width. The external surface of the seed occurs as a pale yellow or yellowish brown color.
It occurs as a slight, characteristic odor and a sweet taste.

Identification Weigh 1 g of pulverized Lycium Fruit, add 5 mL of ethyl acetate, shake for 15 minutes to extract, filter, and use the filtrate as the test solution. With this solution, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL of the test solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of hexane and ethyl acetate (10 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate; it shows one yellowish spot at the R_f value of about 0.6.

Purity (1) ***Foreign matter***—Lycium Fruit contains less than 3.0% of foreign matters, including branches and fruit stalks.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—Proceed with Lycium Fruit as directed in "Lycium Fruit (Dried)" described in [Attachment 4] Tolerances for Pesticide Residues for Agricultural Products provided in the Public Notice "Standards and Specifications for Food" of the Ministry of Food and Drug Safety (MFDS).

(4) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 8.0%.

Assay Weigh accurately about 1 g of pulverized Lycium Fruit, add 50 mL of diluted methanol (1 in 2), connect with a reflux condenser, heat for 2 hours, and filter. To the residue, add 50 mL of diluted methanol (1 in 2) and proceed in the same manner. Combine all filtrates, evaporate to dryness in vacuum, add 30 mL of deionized water to the residue, and adjust the pH to 3.0 with dilute hydrogen chloride. Load this solution to the column I, slowly elute with 60 mL of deionized water, and discard the eluents. To the column I, load 15 mL of diluted ammonia TS (2 in 5) and 15 mL of deionized water in this order, collect the eluate, and vacuum-concentrate the eluate until it becomes 5 mL. Load the concentrated solution to the column II and elute with 10 mL of deionized water. Combined all the eluates and evaporate the solvent in vacuum to dryness. Dissolve the residue in 1 mL of water, and use this solution as the test solution. Separately, weigh accurately about 10 mg of betaine RS, dissolve in 1 mL of water, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and calculate the peak areas, A_T and A_S, from the test solution and the standard solution, respectively.

$$\text{Amount (mg) of betaine } (C_5H_{11}NO_2) = \text{Amount (mg) of betaine RS} \times \frac{A_T}{A_S}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 210 nm)

Column: A stainless steel column 4 – 6 mm in internal diameter and 15 – 25 cm in length, packed with dimethylaminopropylsilyl silica gel for liquid chromatography (5 – 10 μm in particle diameter).

Mobile phase: A mixture of acetonitrile and water (85 : 15)

Flow rate: 1.0 mL/min

Column I: A glass column 10 – 12 mm in internal diameter and 10 cm in length, packed 5 cm high with strongly acidic cationic ion-exchange resin (H+ form).

Column II: A glass column 10 – 12 mm in internal diameter and 10 cm in length, packed 5 cm high with a mixture of weakly acidic cationic ion-exchange resin (H+ form) and strongly basic anionic ion-exchange resin (OH- form) (1 : 2).

System suitability

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak areas of betaine is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Lycium Root Bark
지골피(地骨皮)

Lycii Radicis Cortex

Lycium Root Bark is the root bark of *Lycium chinense* Miller or *Lycium barbarum* Linné (Solanaceae).

Description Lycium Root Bark is the root bark, cylindrical to semi-cylindrical or fragmentary, 3 to 10 cm in length, 5 to 15 mm in width and 1 to 3 mm in thickness. The external surface occurs as a grayish yellow to amber yellow color, coarse with irregular longitudinally-torn patterns. The periderm is scale-shaped and easy to peel off. The inner surface occurs as a yellowish white to grayish yellow color, relatively smooth with fine longitudinal striations. The body is light, and the texture is fragile, making it easy to fracture. The fractured surface occurs as a grayish white to yellowish brown color, is not fibrous and has a light and coarse texture.
Under a microscope, the transverse section of Lycium Root Bark reveals the rhytidome on the outermost layer. The rhytidome consists of 2 to 3 bands of the cork tissue layers, and the innermost layer forms a whole and even ring, which originates from deep inside the phloem. Degenerated sieve tubes and the medullary ray cells are visible in the rhytidome tissue. The phloem takes up half of the thickness of the root bark, the medullary rays consist of a single row of cells, and the parenchyma cells contain sand crystals of calcium oxalate and starch grains. Scattered fibers and the stone cells are visible. Fibers exist individually or in bundles, and cell walls are lignified or slightly lignified.
It has a characteristic odor and a slightly sweet taste, followed by a bitter taste.

Identification (1) Weigh 0.5 g of pulverized Lycium Root Bark, add 10 mL of acetic anhydride, warm on a water bath for 2 minutes, and filter. To 2 mL of the filtrate, carefully add 1 mL of sulfuric acid; a reddish brown color appears at the zone of contact, and after allowing it to stand, the upper layer exhibits a green color.

(2) Weigh 0.5 g of pulverized Lycium Root Bark, add 10 mL of water, heat on a water bath for 5 minutes, and filter. To 2 mL of the filtrate, add 1 mL of ninhydrin TS and heat on a water bath for 2 to 3 minutes; the resulting solution exhibits a violet color.

(3) Weigh 1 g of pulverized Lycium Root Bark, add 10 mL of methanol, shake for 15 minutes, filter, and use the filtrate as the test solution. With this solution, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL of the test solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of 1-butanol, water, pyridine and acetic acid (3 : 1 : 1 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly Dragendorff's TS on the plate, heat at 105 °C for 3 minutes, and develop the color with sodium nitrite TS; a brown spot appears at the R_f value of about 0.5.

Purity (1) ***Foreign matter***—The amount of the xylem and other foreign matters contained in Lycium Root Bark is less than 5.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 12.0%.

Ash NMT 18.0%.

Acid-insoluble ash NMT 3.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 8.0%.

Packaging and storage Preserve in well-closed containers.

Lycopus Herb
택란(澤蘭)

Lycopi Herba

Lycopus Herb is the aerial part, before flowering, of *Lycopus lucidus* Turczaininov (Labiatae).

Description Lycopus Herb is the terrestrial part, a rectangular cylindrical stem with a few branches, 50 cm to 100 cm in length and 2 mm to 6 mm in diameter. The external surface occurs as yellowish green or violet with a distinct violet color and white hair at the nodes and equally shallow longitudinal furrows at the four sides of the stem. The texture is fragile, the transverse section occurs as yellowish white and the center of the pith is hollow. Leaves are opposite and the petiole is short, mostly crumpled, lanceolate or long orbicular when unfolded, 5 cm to 10 cm in length. The upper surface occurs as blackish green and the lower surface occurs as grayish green. Glandular dots are densely distributed and both surfaces are covered in equally short hairs. Apex is pointed and margin is serrate. Flowers are aggregated in leaf axils in verticillate cymes, corolla is mostly fallen off, and bracts and calyx are yellowish brown Lycopus Herb occurs as odorless and has a week taste.

Identification Weigh 1 g of pulverized Lycopus Herb, add 30 mL of acetone, sonicate for 30 minutes, filter, and evaporate the filtrate to dryness. To the residue, add 10 mL of petroleum ether, macerate for 2 minutes, remove the petroleum ether layer and evaporate to dryness. Dissolve the residue in 2 mL of ethanol and use this solution as the test solution. Separately, weigh 0.5 mg of ursolic acid RS, dissolve in 1 mL of ethanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of hexane, ethyl acetate and formic acid (15 : 5 : 0.5) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly the plate with sulfuric acid TS for spraying and heat at 105 °C; one spot among the several spots obtained from the test solution shows the same color and R_f value as the spot obtained from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 10.0%.

Ash NMT 8.0%.

Acid-insoluble ash NMT 2.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 14.0%.

Packaging and storage Preserve in well-closed containers.

Magnolia Bark
후박(厚朴)

Magnoliae Cortex

Magnolia Bark is the stem bark of *Magnolia obovata* Thunb., *Magnolia officinalis* Rehder et Wilson, or *Magnolia officinalis* Rehder et Wilson var. *biloba* Rehder et Wilson (Magnoliaceae).

Magnolia Bark contains NLT 1.0% of the sum of magnolol ($C_{18}H_{18}O_2$: 266.33) and honokiol ($C_{18}H_{18}O_2$: 266.33), calculated on the dried basis.

Description Magnolia Bark is plate-like or semicylindrical bark, 2 mm to 7 mm in thickness. The external surface occurs as grayish white to grayish brown and rough, sometimes occurs as reddish brown with a cork layer removed. Interior surface occurs as pale brown to dark purplish brown. Cut surface is extremely fibrous and it occurs as pale reddish brown to purplish brown.
Under a microscope, a transverse section reveals a thick cork layer or several thin cork layers. The cork layer internally adjoins the circular tissue of stone cells of approximately equal in diameter. The primary cortex is narrow and has fiber groups scattered

in the pericycle. Phloem fibers are lined step-wise between medullary rays in the secondary cortex and then these tissues show a latticework. Oil cells are scattered in the primary and secondary cortex, but sometimes observed in the narrow medullary rays.
It has a slight odor and bitter taste.

Identification Weigh 1 g of pulverized Magnolia Bark, add 10 mL of methanol, and shake for 10 minutes to mix. Then, centrifuge it and use the supernatant as the test solution. Separately, weigh 1 mg each of Magnolol RS and Honokiol RS, dissolve separately in 1 mL of methanol and use these solutions as standard solution (1) and standard solution (2). With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution, the standard solution (1) and the standard solution (2) on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of n-hexane and ethyl acetate (3 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly vanillin-sulfuric acid TS and heat at 105 °C for 10 minutes; two of the several spots obtained from the test solution show the same color and R_f value as the spots obtained from standard solution (1) and standard solution (2).

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 15.0%.

Ash NMT 6.0%.

Assay Weigh accurately about 0.1 g of pulverized Magnolia Bark, add 100 mL of diluted methanol (7 in 10), sonicate for 20 minutes and filter. Combine all the filtrates, add diluted methanol (7 in 10) to make exactly 100 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg each of magnolol RS and honokiol RS (previously dried in a silica gel desiccator for NLT 1 hour), add a mixture of methanol and water (7 : 3) to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under Liquid chromatography according to the following operating conditions and determine the peak areas of magnolol and honokiol, A_{Ta} and A_{Tb}, in the test solution and the peak areas of magnolol and honokiol, A_{Sa} and A_{Sb}, in the standard solution.

$$\text{Amount (mg) of magnolol } (C_{18}H_{18}O_2) = \text{Amount (mg) of magnolol RS} \times \frac{A_{Ta}}{A_{Sa}}$$

$$\text{Amount (mg) of honokiol } (C_{18}H_{18}O_2) = \text{Amount (mg) of honokiol RS} \times \frac{A_{Tb}}{A_{Sb}}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 289 nm)

Column: A stainless column, 4 to 6 mm in internal diameter and 15 to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 to 10 μm in particle diameter).

Column temperature: A constant temperature of about 20 °C.

Mobile phase: A mixture of acetonitrile, water and acetic acid (100) (70 : 30 : 1)

Flow rate: 0.3 mL/min

System suitability

System performance: Weigh 1 mg each of magnolol RS and honokiol RS, dissolve each in diluted methanol (7 in 10) to make 10 mL. Proceed with 10 μL of this solution under the above operating conditions; honokiol and magnolol are eluted in this order with the resolution being NLT 5.

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above operating conditions; the relative standard deviation of the peak areas of magnolol and honokiol is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Mentha Herb
박하(薄荷)

Menthae Herba

Mentha Herb is the aerial part of *Mentha arvensis* Linné var. *piperascens* Malinvaud ex Holmes (Labiatae).

Description Mentha Herb is the aerial part, consisting of stem and stem with leaves grown opposite. The stem is a quadrangular column, 15 cm to 40 cm long and 0.2 cm to 0.4 cm in diameter. The outer surface occurs as purplish brown to pale green with short hairs near the edge and a distance of 2 cm to 5 cm between the nodes. The texture is weak. The cut surface occurs as white and the pith is hollow in the center. The leaves are opposite with short petioles, and the leaf lobes are bruised and curled up. A whole leaf, when spread out, is elliptical to ovate, 2 cm to 7 cm in length and 1 cm to 3 cm in width. The front of the leaf occurs as deep green, the back is grayish green, rarely covered with short hairs and with concave, spot-like scales. The flowers are in axillary verticils, the calyx is bell-shaped and divides into 5 parts at the tip, and the corolla is light purple.
It has a characteristically refreshing aroma when rubbed by hand, and tastes pungent and cool.

Identification (1) Take 1 mL of the mixture of essential oil and xylene, obtained in the Essential oil content, and slowly add 2 mL of sulfuric acid to form two layers; a deep red to reddish brown color develops at the contact surface.

(2) Weigh 0.5 g of pulverized Mentha Herb, add 5 mL of petroleum ether, shake it to mix, and let it stand for 30 minutes. Then, filter it and use the filtrate as the test solution. Separately, dissolve 1 mg of *l*-menthol RS in 1 mL of petroleum ether and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of cyclohexane and ethyl acetate (3 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly vanillin-sulfuric acid TS to the plate and heat it at 105 °C for 10 minutes; one spot among the several spots obtained from the test solution and a spot from the standard solution show the

same color and the same R_f value.

Purity (1) ***Foreign matter***—Mentha Herb contains less than 2.0% of the root and other foreign matter.
(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Methoxychlor: NMT 1 ppm.
(iv) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(v) Aldrin: NMT 0.01 ppm.
(vi) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): NMT 0.2 ppm.
(vii) Endrin: NMT 0.01 ppm.
(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 15.0% (6 hours).

Ash NMT 11.0%.

Acid-insoluble ash NMT 2.5%.

Essential oil content NLT 0.4 mL (50.0 g, 1 mL of silicon resin).

Packaging and storage Preserve in well-closed containers.

Morinda Root
파극천(巴戟天)

Morindae Radix

Morinda Root is the root of *Morinda officinalis* How (Rubiaceae), pressed flatly and dried with the rootlets removed.

Description Morinda Root is compressed cylindrical root, 5 mm to 20 mm in diameter, somewhat bent and varying in length. External surface occurs as grayish yellow or dark gray, with longitudinal wrinkles and transverse cracks. Some cortex is transversely broken and xylem is exposed. The texture is tough. The transverse section has a thick cortex, purple or pale purple and easily to separate from the xylem. Xylem is hard, yellowish brown or yellowish white, 1 mm to 5 mm in diameter.
Under a microscope, a transverse section reveals a cork layer composed of several rows of cork cells containing calcium oxalate raphide bundles. The cortex is narrow and contains stone cells, intermittently arranged individually or in groups to form a ring. The parenchyma cells contain calcium oxalate raphide bundles. The phloem is relatively broad and areas near the cambium contain calcium oxalate raphide bundles. The cambium is distinct. In the xylem, the vessels are scattered individually or gathered in groups of 2 to 3. The xylem fiber is relatively developed, the xylem rays consist of 1 to 3 rows of cells and sometimes groups of unlignified xylem parenchyma cells are observed.
It occurs as odorless and has a sweet, slightly astringent taste.

Identification Weigh 2 g of pulverized Morinda Root, add 15 mL of ethanol, warm on a water bath for 1 hour after connecting with a reflux condenser, and filter. Evaporate the filtrate to dryness, dissolve the residue in 1 mL of ethanol, and use this solution as the test solution. With this solution, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL of the test solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of hexane and ethyl acetate (3 : 2) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS on the plate and heat at 105 °C for 10 minutes; a violet spot appears at the R_f value of about 0.6.

Purity (1) ***Foreign matter***—Xylem: The amount of the xylem contained in Morinda Root is less than 35.0%.
(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(3) ***Residual pesticides—***(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 13.0%.

Ash NMT 6.0%.

Acid-insoluble ash NMT 1.0%.

Extract content ***Dilute ethanol–soluble extract***—NLT 52.0%.

Packaging and storage Preserve in well-closed containers.

Moutan Root Bark
목단피(牡丹皮)

Moutan Radicis Cortex

Moutan Root Bark is the root bark of *Paeonia suffruticosa* Andrews (Paeoniaceae).

Moutan Root Bark contains NLT 1.0% of paeonol ($C_9H_{10}O_3$: 166.17), calculated on the dried basis.

Description Moutan Root Bark is the root bark, cylindrical to semi-cylindrical, slightly curved inward or open longitudinally when split vertically, 5 cm to 20 cm in length, 0.5 cm to 1.2 cm in diameter, and 0.1 cm to 0.4 cm in thickness. The outer surface occurs as grayish brown to yellowish brown with several transverse long lenticels and scars of rootlets. Those without an outer bark are pink on the outside. The inside occurs as pale grayish yellow or brown, not dark, usually with sparkling crystals. The texture is firm and easy to cut. The cut surface occurs as pale pink and powdery.
It has a characteristic odor and a slightly pungent and bitter taste.

Identification Weigh 2 g each of pulverized Moutan Root Bark and Moutan root bark RMPM, add 10 mL of hexane, shake for 3 minutes, filter and use the filtrates as the test solution and the Moutan root bark RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL of the test solution and the Moutan root bark RMPM standard solution on the thin-layer chromatographic

plate made of silica gel for thin-layer chromatography (with fluorescent indicator). Next, develop the plate with a mixture of hexane and ethyl acetate (1 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 254 nm); several spots obtained from the test solution and the spots from the moutan root bark RMPM standard solution shows the same color and the same R_f value.

Purity (1) ***Foreign matter***—(i) Xylem: Less than 5.0%.
(ii) Other foreign matter: The amount of foreign matter other than xylem contained in Moutan Bark is less than 1.0%.
(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(vi) Chlorpyrifos: NMT 0.5 ppm.
(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 13.0%.

Ash NMT 6.0%.

Acid-insoluble ash NMT 1.0%.

Assay Weigh accurately about 0.3 g of pulverized Moutan Bark, add 40 mL of methanol, heat the mixture after connecting with a reflux condenser on a water bath for 30 minutes, cool and filter. Repeat the above procedure by adding 40 mL of methanol to the residue. Combine the whole filtrates, add methanol to make exactly 100 mL, then pipet 10.0 mL of this solution, add methanol to make exactly 25 mL and use this solution as the test solution. Separately, Weigh accurately about 10 mg of paeonol RS (previously dried in a desiccator with calcium chloride for drying for more than 1 hour), dissolve it in methanol to make exactly 100 mL, then pipet 10.0 mL of this solution, add methanol to make exactly 50 mL and use this solution as the standard solution. Perform the test with 20 μL each of the test solution and the standard solution as directed under Liquid chromatography according to the following conditions and determine the peak areas, A_T and A_S, of paeonol in the test solution and the standard solution, respectively.

$$\text{Amount (mg) of paeonol } (C_9H_{10}O_3) = \text{Amount (mg) of paeonol RS} \times \frac{A_T}{A_S} \times \frac{1}{2}$$

Operating conditions
Detector: An ultraviolet absorption photometer (wavelength: 274 nm)
Column: A stainless steel column, 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).
Column temperature: A constant temperature of about 20 °C
Mobile phase: A mixture of water, acetonitrile and acetic acid (100) (65 : 35 : 2).
Flow rate: Adjust the flow rate so that the retention time of paeonol is about 14 minutes.
System suitability
System performance: Dissolve 1 mg of paeonol RS and 5 mg of butyl p-hydroxybenzoate in 25 mL of methanol each. Proceed with 10 μL of this solution according to the above conditions; paeonol and butylparaben are eluted in this order with the resolution being NLT 2.
System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak area of paeonol is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Mulberry Root Bark
상백피(桑白皮)

Mori Radicis Cortex

Mulberry Root Bark is the root bark of *Morus alba* Linné (Moraceae), from which the periderm has been removed.

Description Mulberry Root Bark is the root bark, tubular, semi-tubular or banded, often in fine lateral cuttings, 1 mm to 6 mm in thickness. The external surface occurs as a white to pale yellowish brown color. Those with the periderm, which is easy to peel, occurs as a yellowish brown color with numerous longitudinal, fine wrinkles and numerous reddish brown lenticels. The inner surface occurs as a yellowish white or grayish yellow color with a fine longitudinal pattern. The body is light and tough, strongly fibrous and difficult to cut. The cut surface occurs as a white to pale brown color and is fibrous.
Under a microscope, the transverse section of Mulberry Root Bark reveals distinct medullary rays, consisting of 2 to 6 rows of cells. The lactiferous tubes are scattered throughout, and the cell walls are slightly thick. Fibers are solitary or in groups. The parenchyma cells contain starch grains and prismatic crystals and rhomboid crystals of calcium oxalate. Root barks from old-aged Mulberry contain a small number of stone cell groups, with most cell cavities containing prismatic crystals. Stone cell groups are intermittently arranged inside the phloem, forming a ring shape. It has a slight, characteristic odor and occurs as nearly tasteless.

Identification (1) Weigh 1 g of pulverized Mulberry Root Bark, add 20 mL of hexane, boil after connecting with a reflux condenser on a water bath for l5 minutes, and filter. Then, evaporate the filtrate to dryness and dissolve the residue in l0 mL of chloroform. Transfer 0.5 mL of this solution into a test tube, add 0.5 mL of acetic anhydride, shake to mix, and carefully add 0.5 mL of sulfuric acid; a reddish brown color develops at the zone of contact.
(2) Weigh 1.0 g each of pulverized Mulberry Root Bark and Mulberry root bark RMPM, add 10 mL each of methanol, heat on a water bath for 30 minutes, cool, and filter. Then, evaporate the filtrates to dryness and add1 mL of ethanol to each residue to prepare the test solution and the Mulberry root bark RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Mulberry root bark RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of cyclohexane and ethyl acetate (3 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS for spray on the

plate and heat at 105 °C for 10 minutes; the color and the R_f values of the several spots obtained from the test solution are the same as those of the spots obtained from the Mulberry root bark RMPM standard solution. Of these spots, a violet spot appears at the R_f value of about 0.5.

Purity (1) ***Foreign matter***—The amount of the root xylem and other foreign matters contained in Mulberry Root Bark is less than 1.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 11.0%.

Acid-insoluble ash NMT 2.0%.

Packaging and storage Preserve in well-closed containers.

Mume Fruit
오매(烏梅)

Mume Fructus

Mume Fruit is the unripe fruit of *Prunus mume* Siebold et Zuccarini (Rosaceae), dried in an appropriate method.

Description Mume Fructus is the fruit, close to spherical or flattened spherical, 20 mm to 30 mm in length and 15 mm to 20 mm in diameter The external surface occurs as a black to blackish brown color, wrinkled and with a circular fruit stem scar at the bottom. The core is elliptic, 10 mm to 14 mm in length, about 10 mm in diameter and about 5 mm in thickness, exhibiting a yellowish brown color. The external surface is very hard, with numerous dented spots. The seed is flattened ovoid, exhibiting a pale yellow color.
It has a characteristic odor and an acidic taste.

Identification (1) Weigh 0.5 g of pulverized Mume Fructus, add 10 mL of water, shake for 5 minutes to mix, and filter. Add dilute hydrochloride to the filtrate to acidify, evaporate, dissolve the residue in water, and add lead acetate TS; a white precipitate is formed.

(2) Weigh 5 g of pulverized Mume Fruit, add 30 mL of methanol, sonicate for 30 minutes to extract, and filter. Vacuum-concentrate the filtrate, dissolve in 2 mL of methanol, and use this solution as the test solution. Separately, dissolve 1 mg of ursolic acid RS in 1 mL of methanol and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 2 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of cyclohexane, ethyl acetate and formic acid (200 : 100 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly sulfuric acid TS for spray on the plate and heat at 105 °C; the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

(3) ***Sulfur dioxide***—NMT 30 ppm.

(4) ***Benzo(a)pyrene***—NMT 5 ppm. Perform the test according to the following procedure. Pulverize and homogenize NLT 500 g and NMT 600 g of Mume Fruit. Then, accurately weigh about 5.0 g of the homogenized Mume Fruit, add 100 mL of water, and sonicate for 60 minutes to extract. Add 100 mL of hexane and 1 mL of the internal standard solution, mix homogeneously for 5 minutes with a homogenizer, sonicate for 60 minutes to extract, centrifuge (3,200 g, 10 minutes), and transfer the hexane layer to the separatory funnel (I).
To the hexane layer in the separatory funnel (I), add 50 mL of a mixture of *N*,*N*-dimethylformamide and water (9 : 1), shake to extract, and then transfer the *N*,*N*-dimethylformamide-water mixture (9 : 1) layer to the separatory funnel (II) (repeat this procedure 3 times). Add 100 mL of 1% sodium sulfate solution into the separatory funnel (II) and shake, then add 50 mL of hexane, shake, and allow it to stand. Then, transfer the separated hexane layer to the separatory funnel (III). Add 35 mL of hexane to the separatory funnel (II), shake to extract, and then add the hexane layer to the separatory funnel (III) to combine (repeat this procedure twice). Wash the hexane layer in the separatory funnel (III) with 50 mL of water, filter the hexane layer with dehydration using about 30 g of anhydrous sodium sulfate, and then vacuum-concentrate (70 kPa) on a water bath at 45°C until the hexane becomes about 2 mL. Activate the Florisil cartridge (1 g, 6 mL) by sequentially flowing 10 mL of dichloromethane and 20 mL of hexane at a rate of 2 to 3 drops per second before use. Add the extraction solution to the activated cartridge and elute 20 mL of a mixture of hexane and dichloromethane (3 : 1) at a rate of 2 to 3 drops per second. Evaporate the eluted solution under nitrogen gas on a water bath below 35°C, dissolve the residue in 1 mL of acetonitrile, filter through a 0.45 μm syringe filter, and use the filtrate as the test solution. Separately, weigh accurately appropriate amounts of benzo(a)pyrene RS and 3-methylcholanthrene RS, dissolve each in acetonitrile to make the standard stock solution and the internal standard stock solution, respectively, having known concentration of 1 μg/mL. Store these standard stock solution and the internal standard stock solution at 5 °C to 15 °C and use them within 30 days. Pipet appropriate amounts of the standard stock solution and the internal standard stock solution and dilute them with acetonitrile to prepare the standard solutions having known concentrations of 3, 5, 10, 20 and 40 ng of benzo(a)pyrene per mL and the internal standard solution having known concentration of 50 ng of the internal standard per mL, respectively. If the concentration of the test solution is outside the range of the calibration curve, adjust the concentrations of the standard solutions so that the concentration of the test solution is within the range of the calibration curve. Perform the test with 10 μL each of the test solution and the standard solutions as directed

under the Liquid Chromatography according to the following conditions. Create a calibration curve with the peak area ratio [A_S/A_{IS}] of benzo(a)pyrene to the internal standard obtained from each standard solution as the Y-axis and the concentration of benzo(a)pyrene as the X-axis, and then calculate the concentration of benzo(a)pyrene by substituting the peak area ratio [A_{SAM}/A_{SAMIS}] of benzo(a)pyrene to the internal standard obtained from the test solution on the Y axis.
A_S: Peak area of the reference standard from the standard solution on the calibration curve
A_{IS}: Peak area of the internal standard from the standard solution on the calibration curve
A_{SAM}: Peak area of benzo(a)pyrene from the test solution
A_{SAMIS}: Peak area of the internal standard from the test solution

Internal standard solution—Accurately weigh 3-methylcholanthrene RS and dissolve in acetonitrile to make a solution having known concentration of 50 ng/mL.

Reagents and reagent solutions—The water used in this test should be tertiarily distilled or higher quality, and the reagents used in this test should be those for Pesticide residue testing or with higher quality.

Operating conditions
Detectors: A fluorescence spectrophotometer (excitation wavelengths: 294 nm, fluorescence wavelengths: 404 nm)
Column: Supelcosil LC-PAH (4.6 × 250 mm, 5 μm) or an equivalent column
Column temperature: 37 °C
Mobile phase: A mixture of acetonitrile and water (4 : 1)
Flow rate: 1.0 mL/min

Loss on drying NMT 19.0% (6 hours).

Ash NMT 5.0%.

Acid-insoluble ash NMT 1.5%.

Extract content ***Dilute ethanol-soluble extract***—NLT 27.0%.

Packaging and storage Preserve in well-closed containers.

Myrrh
몰약(沒藥)

Myrrha
Myrrh is the gum-resin obtained from *Commiphora myrrha* Engler or *Commiphora molmol* Engler (Burseraceae). The former is known as Gum Myrrh, and the latter as Gum Opoponax.

Description (1) ***Gum Myrrh***—Gum Myrrh occurs as an irregular, granular mass that varies in size, with the larger ones being 6 cm or more in diameter. The outer surface occurs as yellowish brown or reddish brown, those that are nearly translucent are blackish brown, and covered with yellow dust. The texture is brittle and the fractured surface is uneven and opaque.
It has a characteristic aroma and tastes bitter and slightly pungent.
(2) ***Gum Opoponax*—**Gum Opoponax appears as an irregular, granular mass, usually attached to each other, and varies in size, with the larger ones reaching 6 cm in diameter. The outer surface occurs as yellowish brown to maroon and opaque. The texture is firm or sparse.

Identification (1) Add water to Myrrh and mash; a light yellowish brown emulsion is formed.
(2) Weigh 1 g of pulverized Myrrh, add 3 mL of ether, shake to mix and filter. Evaporate the filtrate to dryness and add nitric acid to the residue; it becomes a yellow liquid. Shake the mixture to mix; it turns purplish brown.

Purity ***Ethanol-insoluble matter***—Weigh accurately about 2 g of pulverized Myrrh, add 30 mL of ethanol and perform warm extraction for 30 minutes with occasional shaking. Weigh a small filter and filter the extract with it. Repeat warm extraction 3 times with the residue in 15 mL of ethanol for 5 minutes each time. After thoroughly washing the residue in the filter with 5 mL of warmed ethanol, dry the insoluble matter at 100 °C for 1 hour. Let it cool in a desiccator (silica gel) and weigh it; the amount of the insoluble matter is NMT 70%.

Ash NMT 15.0%.

Acid-insoluble ash NMT 5.0%.

Packaging and storage Preserve in well-closed containers.

Nelumbo Seed
연자육(蓮子肉)

Nelumbinis Semen
Nelumbo Seed is the ripe seed of *Nelumbo nucifera* Gaertner (Nymphaeaceae), usually used as it is or after removing the green embryo.

Description Nelumbo Seed is the seed, usually elliptic or close to spherical, 12 mm to 18 mm in length and 8 mm to 14 mm in diameter. The external surface occurs as a pale yellowish brown to reddish brown color, with fine longitudinal striations and relatively wide vein patterns. The center of one end is papillate, deep brown, mostly with many cracks and somewhat dented around the edge. The seed coat is thin, yellowish brown and hard to peel off. There are two yellowish white, plump cotyledons inside the seed coat, with the green embryo at the empty space between the two cotyledons.
It occurs as nearly odorless and has a slightly oily and sweet taste. Its green embryo occurs as extremely bitter.

Identification (1) Weigh 1.0 g of pulverized Nelumbo Seed, add 10 mL of dilute acetic acid, heat for 3 minutes on a water bath with occasional stirring to mix, cool, and filter. To 2 mL of the filtrate, add 2 to 3 drops of Dragendorff's TS; an amber yellow precipitate is formed.
(2) Add an appropriate amount of water to a small quantity of pulverized Nelumbo, mix thoroughly, and add several drops of iodide TS; the resulting solution exhibits a bluish purple color, which disappears gradually upon heating, but returns to the bluish purple color, if cooled again.
(3) Weigh 0.5 g of pulverized Nelumbo Seed, add 0.5 mL of water, shake for 5 minutes to mix, and centrifuge. To 0.5 mL of the supernatant, add 1 drop of 1-naphthol TS, mix well, and slowly add 1 mL of sulfuric acid; a violet ring appears at the zone of contact between the two liquid layers.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.
(4) ***Mycotoxins***—Total aflatoxins (sum of aflatoxins B_1, B_2, G_1 and G_2): NMT 15.0 ppb (However, aflatoxin B_1 is NMT 10.0 ppb).

Ash NMT 5.5%.

Extract content ***Dilute ethanol-soluble extract***—NLT 12.0%.

Packaging and storage Preserve in well-closed containers.

Nutmeg
육두구(肉豆蔻)

Myristicae Semen

Nutmeg is the ripe seed of *Myristica fragrans* Houttuyn (Myristicaceae), with the seed coat removed.

Description Nutmeg is an ovoid or oval seed with a length of 2 cm to 3 cm and a diameter of 1.5 cm to 2.5 cm. The outer surface occurs as yellowish brown to grayish yellow, the outer shell is sometimes covered with white powder. The entire seed has longitudinal furrows of pale color and irregular reticulate furrows. The hilum is at the end of the broad area and appears as a circular protrude of pale color, and the chalaza is indented and appears dark. The seed ridge is a longitudinal furrow and the single furrow connecting the two ends is shallow and broad. Thin, narrow, reticulate furrows are visible throughout. The embryo is small and slightly shrunken near the hilum. Under the magnifying glass, a transverse section shows a thin, dark brown perisperm. The perisperm has a series of irregular projections and is connected to the pale yellowish white endosperm to form marble-like patterns. The embryo is small and slightly shrunken near the hilum.
Under the microscope, the perisperm is divided into the inner layer and outer layer. The outer layer consists of flat cells containing brown substances. The inner layer consists of rectangular cells with reddish brown substances that protrude into the endosperm and form an irregular tissue. This tissue has a vascular bundle and is dotted with numerous oil cells. The oil cells contain essential oil. The endosperm cells are polygonal and contain large amounts of fatty oil, starch granules and aleurone grains. The aleurone grains contain pseudocrystals. The endosperm is dotted with cells containing brown substances.
It has a characteristic odor and tastes pungent and slightly bitter.

Identification (1) Weigh 1 g of pulverized Nutmeg, add 10 mL of methanol, heat the mixture on a water bath for 3 minutes, and filter. Allow the filtrate to stand in ice water for 10 minutes; a white precipitate is formed.

(2) Dissolve the precipitate produced in (1) with 5 mL of chloroform and use this solution as the test solution. With this solution, perform the test as directed under the Thin Layer Chromatography. Spot 2 μL of the test solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of hexane and acetone (4 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Expose the plate to iodine vapor; a yellow spot appears at an R_f value of about 0.3.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.
(4) ***Mycotoxins***—Total aflatoxins (sum of aflatoxins B_1, B_2, G_1 and G_2): NMT 15.0 ppb (aflatoxin B1 is NMT 10.0 ppb).

Ash NMT 3.0%.

Essential oil content NLT 0.5 mL (10.0 g).

Packaging and storage Preserve in well-closed containers.

Nux Vomica
마전자(馬錢子)

Strychni Semen

Nux Vomica is the ripe seed of *Strychnos nux-vomica* Linné (Loganiaceae).

Nux Vomica contains NLT 1.05% of strychnine ($C_{21}H_{22}N_2O_2$: 334.42), calculated on the dried basis.

Description Nux Vomica is button-shaped, prominent at one side and slightly dented at the other side, 10 mm to 30 mm in diameter and 3 mm to 5 mm in thickness, exhibiting a pale grayish green to pale grayish brown color. The external surface is covered densely with lustrous suppressed hairs radiating from the center to the circumference. The marginal and central parts on both sides are slightly bulged. The dot-like micropyle is situated at one side on the margin, from which a raised line often runs to the center on one side. The texture is extremely hard. When cracked after being soaked in water, the seed coat is thin, and the interior consists of two horny, pale grayish yellow endosperms and the central part with a narrow, hollow cavity. There occurs as a white embryo, about 7 mm in length, on one side of the inner endosperm.
It has a slight odor and a very bitter and long-lasting taste.

Identification (4) Weigh 3 g of pulverized Nux Vomica, add 3 mL of ammonia TS and 20 mL of chloroform, macerate for 30 minutes with occasional shaking, and filter. Remove most of the chloroform from the filtrate by warming on a water bath. To the remaining filtrate, add 5 mL of diluted sulfuric acid (1 in 10) and warm on a water bath while shaking well to mix until the odor of chloroform is no longer perceptible. After cooling, filter the resulting solution through a pledget of absorbent cotton and add 2 mL of nitric acid to 1 mL of the filtrate; the resulting solution exhibits a red color.

(2) Weigh 1 g of pulverized Nux Vomica, add 10 mL of

ethanol, sonicate for 1 hour to extract, filter, and use the filtrate as the test solution. Separately, weigh 1 mg of strychnine RS, dissolve in 1 mL of methanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of ethyl acetate, isopropyl alcohol and ammonia water (10 : 5 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 254 nm); the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot obtained from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

Ash NMT 3.0%.

Assay Weigh accurately about 1 g of pulverized Nux Vomica, previously dried at 60 °C for 8 hours, place in a stoppered centrifuge tube, and moisten with l mL of strong ammonia water. To this solution, add 20 mL of ether, stopper the tube tightly, shake for 15 minutes to mix, centrifuge, and take the supernatant. Repeat this procedure three times with the residue each time with 20 mL of ether. Combine all the extracts and evaporate ether on a water bath. Dissolve the residue in 10 mL of the mobile phase, add exactly 10 mL of the internal standard solution, and add more of the mobile phase to make exactly 100 mL. Filter this solution through a membrane filter with a pore size not exceeding 0.8 μm, discard the first 2 mL of the filtrate, and use the subsequent filtrate as the test solution. Separately, weigh exactly about 75 mg of strychnine nitrate RS (of which loss on drying is previously determine) and dissolve in the mobile phase to make exactly 50 mL. Pipet 10 mL of this solution, add exactly 10 mL of the internal standard solution, add more of the mobile phase to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 5 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and calculated the peak area ratio, Q_T and Q_S, of strychnine to the internal standard solution, respectively, from each solution.

Amount (mg) of strychnine ($C_{21}H_{22}N_2O_2$)
= Amount (mg) of strychnine nitrate RS, calculated on the dried basis $\times \frac{Q_T}{Q_S} \times \frac{1}{5} \times 0.8414$

Internal standard solution—A solution of barbital sodium in the mobile phase (l in 500).

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 210 nm)

Column: A stainless steel column about 4 mm in internal diameter and about 15 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm in particle diameter).

Mobile phase: Dissolve 6.8 g of potassium dihydrogen phosphate in water to make 1000 mL, mix this solution with acetonitrile and triethylamine (45 : 5 : 1), and adjust the pH of the mixture to 3.0 with phosphoric acid.

Flow rate: Adjust the flow rate so that the retention time of strychnine is about 17 minutes.

Selection of column: Proceed with 5 μL of the standard solution according to the above conditions. At this time, use a column from which the internal standard and strychnine are eluted in this order with the peaks being fully separated from each other.

Packaging and storage Preserve in well-closed containers.

Nux Vomica Extract
마전자엑스

Strychni Semen Extract

Nux Vomica Extract contains NLT 6.15% and NMT 6.81% of Strychnine ($C_{21}H_{22}N_2O_2$: 334.41).

Method of preparation After defatting 1000 g of coarse powder of Nux Vomica with hexane, proceed with percolation as directed under the Percolation using a mixture of 750 mL of ethanol, 10 mL of acetic acid and 240 mL of purified water as the first solvent and diluted ethanol (7 in 10) as the second solvent. Combine all the extracts and prepare the dry extract as directed under Extracts. However, appropriate amounts of ethanol and purified water can be used for the preparation.

Description Nux Vomica Extract occurs as a yellowish brown powder. It has a characteristic odor and an extremely bitter taste.

Identification To 0.5 g of Nux Vomica Extract, add 0.5 mL of ammonia TS and 10 mL of chloroform, extract with shaking occasionally, filter the chloroform extract layer, evaporate most of the chloroform from the filtrate by warming on a water bath. Proceed as directed in the Identification under Nux Vomica.

Purity (1) ***Heavy metals***—(i) Total heavy metals: NMT 30 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

Assay Weigh accurately about 0.2 g of Nux Vomica Extract, transfer into a stoppered centrifuge tube, add 15 mL of ammonia TS, and shake to mix. To this solution, add 20 mL of ether, stopper the tube, shake for 15 minutes to mix, centrifuge, and take the ether layer. Repeat this procedure 3 times with the water layer, each time with 20 mL of ether. Combine all the extracts and evaporate ether on a water bath. Dissolve the residue in 10 mL of the mobile phase, add exactly 10 mL of the internal standard solution, and add more of the mobile phase to make exactly 100 mL. Proceed as directed in the Assy under Nux Vomica.

Amount (mg) of strychnine ($C_{21}H_{22}N_2O_2$)
= Amount (mg) of strychnine nitrate RS, calculated on the dried basis

$$\times \frac{Q_T}{Q_S} \times \frac{1}{5} \times 0.8414$$

Internal standard solution—A solution of barbital sodium in the mobile phase (l in 500).

Packaging and storage Preserve in light-resistant, tight containers

Ostericum Root
강활(羌活)

Osterici seu Notopterygii Radix et Rhizoma

Ostericum Root is the root of *Ostericum koreanum* Maximowicz, or the rhizome or root of *Notopterygium incisum* Ting or *Notopterygium forbesii* H. Boissieu (Umbelliferae).

Description (1) ***Ostericum koreanum***—Ostericum Root from *Ostericum koreanum* is the root, conical or long conical in shape with several branches. It is 15 - 30 cm in length and 2 - 5 cm in diameter. The external surface occurs as yellowish brown to brown with sparse rootlets or rootlet scars. Horizontal patterns forming rings are present near the crown. The crown is relatively broad, usually with remains of stem bases or petiole bases. The texture is hard, but fragile. The transverse section occurs as the pale brown or yellowish brown cortex, which is relatively sparse with several clefts, and the white or yellowish white xylem.
Under a microscope, the transverse section of Ostericum Root reveals the outermost layer of the root consisting of 3 to 4 rows of cork cells and underneath is the collenchymas consisting of 4 to 8 layers. Secretory canals are sparsely arranged in the collenchymas or in the phloem. There are 1 to 3 rows of medullary rays, radiating from the secondary xylem to the cortex. The cambium consists of 3 to 4 rows. Intercellular spaces are particularly abundant in the cortex and the parenchyma is filled with starch grains.
Ostericum Root from Ostericum koreanum has a characteristic odor and a sweet and cooling taste at first, which is followed by a slight bitter taste.

(2) ***Notopterygium incisum***—Ostericum Root from *Notopterygium incisum* consists of the rhizome and the root, which have a slightly curved, cylindrical shape with occasionally branches. It is 4 - 13 cm in length and 6 - 25 mm in diameter. The external surface occurs as a chestnut to blackish brown color, and the peeled area occurs as a yellow color. Those with short nodes and tightly packed ring patterns giving the appearance of a silkworm are called Jamgang (silkworm-like root), and those with long nodes giving the appearance of bamboo joints are called Jukjeolgang (bamboo node-like root). The nodes have marks of roots protruding in the shape of a spots or a nodule, with numerous broken scale pieces exhibiting a brown color. The body is light and has a fragile texture, making it easy to cut. The transverse section is irregular with several clefts. The cortex occurs as a yellowish brown to dark brown color and is slippery with brown oil drops. The xylem occurs as a yellowish white color with clear medullary rays. The pith occurs as a yellow to yellowish brown color.
Under a microscope, the transverse section of Ostericum Root from *Notopterygium incisum* reveals the cork layer consisting of about 10 rows of cork cells. The cortex is narrow with several clefts in the phloem, and the cambium forms rings. There are a relatively large number of xylem vessels. The oil sacs are large and particularly abundant in the phloem, and they are also present in the pith and in the medullary rays. The oil sacs contain a yellowish brown oil-like substance.
Ostericum Root from *Notopterygium incisum* has an aroma and a slightly bitter and pungent taste.

(3) ***Notopterygium forbesii***—Ostericum Root from *Notopterygium forbessii* is the rhizome and the root. The rhizome is close to a cylindrical shape, and the top of the rhizome has scars of stems or leaf sheaths. The root is conical with longitudinal wrinkles and lenticels. The external surface occurs as a chestnut color with relatively dense circular patterns near the rhizome. Those with roots 8 - 15 cm in length and 1 - 3 cm in diameter are called Jogang (twig-like root). Those with thick, large, irregularly knotted rhizome with several stem bases at the top and relatively thin roots are called daedugang (large-headed root). Texture is not dense and fragile, making it easy to cut. The cut surface is slightly flat, the cortex occurs as a pale brown color, and the xylem occurs as a yellowish white color.
Under a microscope, the traverse section of Ostericum Root from *Notopterygium forbessii* reveals the outermost layer of the root consisting of 3 to 4 rows of cork cells and the collenchymas consisting of 4 to 8 layers underneath the cork cells. The secretory canals are present in the collenchymas. Sparsely arranged secretory canals are also present in the phloem. The medullary rays are arranged in 1 to 3 rows, radiating from the secondary xylem to the cortex. The cambium has 3 to 4 rows. Intercellular spaces are particularly abundant in the cortex, and the parenchyma is filled with starch grains.
Ostericum Root from *Notopterygium forbessii* has a slight odor and a relatively plain taste.

Identification Weigh 1 g of pulverized Ostericum Root, add 10 mL of ether and extract at the ordinary temperature. Examine the extract solution under ultraviolet light; the extract solution exhibits strong fluorescence.

Purity (1) ***Foreign matters***—Ostericum Root contains less than 5.0% of the stem bases and other foreign matters.
(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(i) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): NMT 0.2 ppm.
(vi) Endrin: NMT 0.01 ppm.
(vii) Oxolinic acid: NMT 7.0 ppm.
(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 13.0%.

Ash NMT 10.0%.

Acid-insoluble ash NMT 2.0%.

Extract Content ***Dilute ethanol-soluble extract***—NLT 20.0%.

Packaging and storage Preserve in well-closed containers.

Oyster Shell
모려(牡蠣)

Ostreae Testa

Oyster Shell is the shell of *Ostrea gigas* Thunberg, *Ostrea talienwhanensis* Crosse or *Ostrea rivularis* Gould (Ostreidae).

Description (1) ***Ostrea gigas***—Oyster Shell of Ostrea gigas is the shell composed of long pieces with dorsal and ventral edges nearly parallel, 10 cm to 50 cm long and 4 cm to 15 cm high. The right valve is relatively small and the scales are firm and thick, layered or lamellar. The outer surface is flat or has several dents, occurs as pale purple, grayish white or yellowish brown, and the inner surface occurs as white, with both apexes without fine serrations. The left valve is deeply concave, the scales of the right valve are relatively coarse and large, the apexes of the shell are connected to each other in a small area. The texture is brittle and the cross section forms layers that are white.
Oyster Shell occurs as almost odorless and tastes slightly salty.

(2) ***Ostrea talienwhanensis***—Oyster shell of Ostrea talienwhanensis is the shell and more like a triangle, and dorsal and ventral edges are inverted V-shaped. The outer surface of the right valve occurs as pale yellow and the concentric scales are loosely arranged, and the bottom of the scales is wavy. The inner surface occurs as white. The left valve bears strong and thick concentric scales. The radiating dorsal heads emanating from the apex of the oyster shell are numerous and distinct. The inner side has a downward concave box shape, and the surfaces where they cross and join are small.

(3) ***Ostrea rivularis***—Oyster shell of Ostrea rivularis is round, oval or triangular. The outer surface of the right valve is uneven, gray, purple, brown, and yellow, with concentric scales forming a ring. It is thin and fragile when immature and overlaps after a few years of growth. The inner surface occurs as white and sometimes its edge occurs as pale purple.

Identification (1) Weigh 1 g of pulverized Oyster Shell, dissolve it in 10 mL of dilute hydrochloric acid while heating; it evolves a gas and forms a very slightly red, turbid solution in which a transparent, thin suspended matter remains. Pass the evolved gas through calcium hydroxide TS; a white precipitate is produced.

(2) The turbid solution obtained from (1) has a slight, characteristic odor. Filter this solution and neutralize it with ammonia TS; the solution responds to the Chemical identification reactions for calcium salt.

(3) Ignite 1 g of pulverized Oyster Shell; it first turns blackish brown and develops a characteristic odor. Ignite it further; it becomes almost white.

Purity ***Barium salt***—Weigh 1 g of pulverized Oyster Shell, dissolve it in 10 mL of dilute hydrochloric acid; the solution does not respond to the Chemical identification reaction (1) for barium salt.

Loss on drying NMT 4.0% (1 g, 180 ℃, 4 hours).

Packaging and storage Preserve in well-closed containers.

Peach Kernel
도인(桃仁)

Persicae Semen

Peach Kernel is the ripe seed of *Prunus persica* (L.) Batsch or *Prunus davidiana* (*Carrière*) Franch (Rosaceae).

Peach Kernel contains NLT 0.5% of amygdalin ($C_{20}H_{27}NO_{11}$: 457.43), calculated on the dried basis.

Description (1) ***Prunus persica***—Peach Kernel from *Prunus persica* is the seed, flattened, long ovoid, 12 mm to 20 mm in length, 6 mm to 12 mm in width and 3 mm to 7 mm in thickness. The external surface occurs as a pale brown to reddish brown and appears as if it has been sprinkled with powder due to the epidermis cells made of stone cells. The shape of the seed is somewhat pointy at one end, convex in the middle, and round at the other end with the chalaza, from which several longitudinal wrinkles are originated. The seed coat is thin, and there are 2 cotyledons, close to white and very oily.
Under a microscope, the seed coat reveals stone cells protruding externally, polygonal, long polygonal or obtuse triangular depending on the position, and the cell membrane is mostly evenly thickened.
It has a characteristic odor and a slightly bitter taste.

(2) ***Prunus davidiana***—Peach Kernel from *Prunus davidiana* is the seed, flattened, ovoid, about 9 mm in length, about 7 mm in width and about 5 mm in thickness.

Identification Weigh 1 g each of pulverized Peach Kernel or Peach kernel RMPM, add 10 mL each of methanol, and heat after connecting with a reflux condenser on a water bath for 10 minutes. After cooling, filter each solution and use these solutions as the test solution and the Peach kernel RMPM standard solution. Perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Peach kernel RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of ethyl acetate, methanol and water (12 : 2 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS for spray on the plate and heat at 105 °C for 10 minutes; the color and the R_f values of the several spots obtained from the test solution are the same as those of the spots obtained from the peach kernel RMPM standard solution. Of these spots, the spot of amygdalin appears at the R_f value of about 0.3.

Purity (1) ***Foreign matter***—Peach Kernel does not contain broken pieces of the endocarp or other foreign matters.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(vi) Chlorothalonil: NMT 0.1 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

(5) ***Mycotoxins***—Total aflatoxins (sum of aflatoxins B_1, B_2, G_1 and G_2): NMT 15.0 ppb (However, aflatoxin B_1 is NMT 10.0 ppb).

(6) ***Rancidity***—Grind Peach Kernel in boiling water; no odor of rancid oil is perceptible.

Loss on drying NMT 8.0%.

Assay Weigh accurately about 2.0 g of pulverized Peach Kernel, add 50 mL of diluted methanol (1 in 2), heat after connecting with a reflux condenser for 1 hours, filter, and use the filtrate as the test solution.
Separately, weigh accurately about 10 mg of amygdalin RS (previously dried in a desiccator with silica gel for 24 hours), dissolve in water to make exactly 20 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and calculate the peak areas, AT and AS, from the test solution and the standard solution, respectively.

$$\text{Amount (mg) of amygdalin } (C_{20}H_{27}NO_{11}) = \text{Amount (mg) of amygdalin RS} \times \frac{A_T}{A_S} \times 2.5$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 210 nm)

Column: A stainless steel column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Column temperature: A constant temperature of about 30 ℃

Mobile phase: Control the mixing ratio of mobile phase A and mobile phase B stepwise or via the concentration gradient as follows.

Mobile phase A: Water
Mobile phase B: Methanol

Time (min)	Mobile Phase A (vol%)	Mobile Phase B (vol%)
0	95	5
30	0	100

Flow rate: 1.0 mL/min

System suitability

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak areas of amygdalin is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Peony Root
작약(芍藥)

Paeoniae Radix

Peony Root is the root of *Paeonia lactiflora* Pallas or other congeneric and allied plants in the same genus (Paeoniaceae).

Peony Root contains NLT 2.3% in total of albiflorin ($C_{23}H_{28}O_{11}$:480.46) and paeoniflorin ($C_{23}H_{28}O_{11}$: 480.46), calculated on the dried basis.

Description Peony Root is the root, cylindrical, sometimes curved, 5 to 20 cm in length and 10 to 25 mm in diameter, and a large root is cut lengthwise. The external surface occurs as white or brown color, smooth with distinct longitudinal wrinkles, often dented with wrinkles or scars of rootlets and with horizontally distinct lenticels. The upper part of the root often has remains of scars of stems or unremoved brown peel. The texture is hard and difficult to break. Under a magnifying glass, the transverse section reveals a granular surface, very dense, with the distinct cambium, exhibiting milky white or brown color, the radial medullar rays and the cambium.
It has a characteristic odor and a slightly sweet taste at first, followed by an astringent and slight bitter taste.

Identification (1) Weigh 0.5 g of pulverized Peony Root, add 30 mL of ethanol, shake for 15 minutes to mix, and filter. To 3 mL of the filtrate, add 1 drop of Iron(III) chloride TS and shake to mix; the resulting solution exhibits a bluish purple to bluish green color, which changes to a dark bluish purple to dark green color.

(2) Weigh 2 g each of pulverized Peony Root and Peony root RMPM, add 10 mL each of methanol, warm on a water bath for 5 minutes, cool, filter, and use the filtrates as the test solution and the Peony root RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Peony root RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of acetone, ethyl acetate and acetic acid (100) (10 : 10 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly p-anisaldehyde-sulfuric acid TS on the plate and heat at 105 °C for 10 minutes; the color and the R_f values of the several spots obtained from the test solution are the same as those of the spots obtained from the Peony root RMPM standard solution. Of these, a violet spot appears at the R_f value of about 0.4.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Napropamide: NMT 0.1 ppm.
(ii) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'- DDT and p,p'-DDT): NMT 0.1 ppm.
(iii) Dieldrin: NMT 0.01 ppm.
(iv) Myclobutanil: NMT 0.1 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(vi) Cyprodinil: NMT 0.1 ppm.
(vii) Aldrin: NMT 0.01 ppm.
(viii) Endrin: NMT 0.01 ppm.
(ix) Iminoctadine: NMT 0.3 ppm.
(x) Carbendazim: NMT 0.05 ppm.
(xi) Triadimenol: NMT 0.1 ppm.
(xii) Triadimefon: NMT 0.5 ppm.
(xiii) Triforine: NMT 0.1 ppm.
(xiv) Triflumizole: NMT 1.0 ppm.
(xv) Pendimethalin: NMT 0.2 ppm.
(xvi) Propineb: NMT 0.2 ppm.
(xvii) Fludioxonil: NMT 0.1 ppm.
(xviii) Dithianon: NMT 0.3 ppm.
(xix) Azoxystrobin: NMT 0.1 ppm.
(xx) Cadusafos: NMT 0.01 ppm.
(xxi) Terbufos: NMT 0.05 ppm.
(xxii) Thiram: NMT 0.2 ppm.
(xxiii) Fenarimol: NMT 0.1 ppm.
(xxiv) Fosthiazate: NMT 0.01 ppm.

(xxv) Prochloraz: NMT 0.1 ppm.

(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 14.0% (6 hours).

Ash NMT 6.5%.

Assay Weigh accurately about 0.5 g of pulverized Peony Root, add 50 mL of diluted methanol (1 in 2), heat after connecting with a reflux condenser on a water bath for 30 minutes, cool, and filter. To the residue, add 30 mL of diluted methanol (1 in 2) and proceed in the same manner. Combine all the extracts, add diluted methanol (1 in 2) to make exactly 100 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg each of paeoniflorin RS (previously dried in a desiccator with silica gel for more than 24 hours) and albiflorin RS (previously dried in a desiccator with silica gel for more than 24 hours), add diluted methanol (1 in 2) to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 20 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions, and determine the peak areas, A_{Ta} and A_{Tb}, from the test solution and the peak areas, A_{Sa} and A_{Sb}, from the standard solution.

$$\text{Amount (mg) of albiflorin } (C_{23}H_{28}O_{11}) = \text{Amount (mg) of albiflorin RS} \times \frac{A_{Ta}}{A_{Sa}}$$

$$\text{Amount (mg) of paeoniflorin } (C_{23}H_{28}O_{11}) = \text{Amount (mg) of paeoniflorin RS} \times \frac{A_{Tb}}{A_{Sb}}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 230 nm)

Column: A stainless steel column 4 to 6 mm in internal diameter and 15 to 25 cm in length, packed with octadecylsilyl silica gel (5 to 10 μm in diameter).

Column temperature: An ordinary temperature

Mobile phase: Control the mobile phases A and B stepwise as directed in the following table or via the concentration gradient.

Mobile phase A: Water

Mobile phase B: Acetonitrile.

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)
0	90	10
15	90	10
30	80	20
45	65	35
48	50	50
55	50	50

Flow rate: 1.0 mL/min

System suitability

System performance: Proceed with 10 μL of the standard solution according to the above conditions. Adjust the concentration-gradient condition so that albiflorin and paeoniflorin are eluted in this order with clear separation of each peak.

System repeatability: Repeat the test 6 times with 20 μL each of the standard solution according to the above conditions; the relative standard deviations of each peak area of paeoniflorin and albiflorin are NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Perilla Leaf
자소엽(紫蘇葉)

Perillae Folium

Perilla Leaf is the leaf and twig of *Perilla frutescens* Britton var. *acuta* Kudo or *Ferilla frutescens* Britton var. *crispa* Decaisne (Labiatae).

Description Perilla Leaf is the leaf and twig. Both surfaces of the leaf occur as a brownish violet color, or the front surface occurs as a grayish green to greenish brown color, while the rear surface exhibits a brownish violet color. When an evenly-shaped leaf is smoothened while immersing it in water, the lamina is ovate to obcordate, 5 to 12 cm in length and 5 to 8 cm in width, with the slightly acute apex and the serrated margin. The base is broadly cuneate and has the petiole, 3 to 5 cm in length. The transverse sections of the stem and the petiole are square. Under a magnifying glass, the leaf reveals sparsely scattered hairs on both surfaces, more abundantly on the vein, and small glandular hairs on the rear surface.
It has a characteristic odor and a slightly bitter taste.

Identification Take 0.3 mL of a mixture of essential oil, obtained in the Essential oil content, and xylene, add 1 mL of acetic anhydride, shake to mix, and add 1 drop of sulfuric acid; the resulting solution exhibits a reddish purple to dark reddish purple color.

Purity (1) ***Foreign matter***—(i) Stem: The amount of stems with over 3 mm in diameter contained in Perillae Leaf is less than 3.0%.

(ii) Other foreign matters: The amount of foreign matters other than stems contained in Perillae Leaf is less than 1.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 13.0% (6 hours).

Ash NMT 16.0%.

Acid-insoluble ash NMT 2.5%.

Essential oil content NLT 0.2 mL (50.0 g, 1 mL of silicon resin).

Packaging and storage Preserve in well-closed containers.

Pharbitis Seed
견우자(牽牛子)

Pharbitidis Semen

Pharbitis Seed is the ripe seed of *Pharbitis nil* Choisy or *Pharbitis purpurea* Voigt (Convolvulaceae).

Description Pharbitis Seed is in the form of a seed bead, vertically divided into 4 to 6 equal parts, with a length of 6 to 8 mm and a width of 3 to 5 mm. External surface occurs as black to grayish reddish brown or grayish white, smooth or slightly shrunken. The transverse section is generally fan-shaped, pale yellowish brown to pale grayish brown in color, and dense in texture. Under a magnifying glass, the surface of the seed coat reveals dense, short hairs, dented hilum at the bottom of the ridge. The seed coat is thin; the outer layer occurs as dark gray, and the inner layer is pale gray. At one end are two irregularly folded cotyledons in cross section. Between them, there are two thin septa extending from the middle of the dorsum to the ridge. In contrast, the umbilicus is located at the other end, and no septum is seen. In the cross section of the cotyledon, there are dark gray secretion pits.
It has a characteristic odor when cracked, tastes similar to oil, and is slightly irritating.

Identification Weigh 1 g of pulverized Pharbitis Seed, add 10 mL of methanol, extract by shaking, filter and evaporate to dryness. Dissolve the residue in 1 mL of methanol and use this solution as the test solution. Separately, dissolve 1 mg of caffeic acid RS in 1 mL of methanol and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of dichloromethane, methanol and formic acid (93 : 9 : 4) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly phosphomolybdic acid-anhydrous ethanol TS (1 in 20) and heat at 105 °C; one spot among the spots obtained from the test solution shows the same color and R_f value as the dark purple spot from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 6.0%.

Packaging and storage Preserve in well-closed containers.

Phellodendron Bark
황백(黃柏)

Phellodendri Cortex

Phellodendron Bark is the bark of *Phellodendron amurense* Ruprecht or *Phellodendron chinense* Schneider (Rutaceae), from which the periderm has been removed.
Phellodendron Bark contains NLT 0.6% of berberine [as berberine chloride ($C_{20}H_{18}ClNO_4$: 371.81)], calculated on the dried basis.

Description Phellodendron Bark is plate-shaped or semi-tubular pieces of bark, 2 mm to 4 mm in thickness. The external surface occurs as grayish yellowish brown to grayish brown, with numerous traces of lenticels. The internal surface occurs as yellow to dark yellowish brown, with fine vertical lines, and smooth. The fractured surface is fibrous and bright yellow. Under a magnifying glass, the transverse section reveals a thin and yellow outer cortex, scattered with stone cells appearing as yellowish brown dots. Inner cortex is thick. Primary medullary rays expand its width towards the outer end. The phloem appears as a nearly triangular part between these medullary rays in secondary cortex and many secondary medullary rays radiating and gathering to the tip of the triangle. Brown phloem fiber bundles lined in stepwise, crossed over the medullary rays and then these tissues show a latticework.
It has a slight, characteristic odor and extremely bitter taste and is mucilaginous. Phellodendron Bark colors the saliva yellow.

Identification (1) To the pulverized Phellodendron Bark, add water and stir to mix; the solution develops a gel phase due to mucus
(2) Weigh 1 g of pulverized Phellodendron Bark, add 10 mL of ether, allow to stand for 10 minutes with occasional shaking, and filter. Collect the powder on the filter paper, add 10 mL of ethanol, allow to stand for 10 minutes with occasional shaking, and filter. Use the filtrate as the test solution. Separately, dissolve 1 mg of berberine chloride RS in 1 mL of methanol and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel (with fluorescence indicator) for thin layer chromatography. Next, develop the plate with a mixture of n-butanol, water and acetic acid (100) (7 : 2 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 365 nm); one spot among the several spots from the test solution and a spot with yellow to yellowish green fluorescence from the standard solution show the same color and the same R_f value.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 11.0% (105 °C, 6 hours).

Ash NMT 7.5%.

Assay Weigh accurately about 0.5 g of pulverized Phellodendron Bark and perform the test as directed in the Assay under Coptis Rhizome.

Packaging and storage Preserve in well-closed containers.

Picrasma Wood
고목(苦木)

Picrasmae Lignum

Picrasma Wood is the heartwood of *Picrasma quassioides* Bennet (Simaroubaceae).

Description Picrasma Wood is the heartwood, which consists of chips, slices or short pieces of wood. The external surface occurs as pale yellow and the texture is dense. Under a magnifying glass, there are distinct annual rings and thin medullary rays on the transverse section.
Under a microscope, the medullary rays of Picrasma Wood consists of rows of 1 to 5 cell layers in width, and the longitudinal section consists of 5 to 50 cell layers in height. The vessels of the earlywood have a diameter of up to 150 μm, whereas those of the latewood have only one fifth of this diameter. These vessels are located either singly or in groups in the xylem parenchyma and are in contact with each other. The xylem fibers are distinctly thickened, the water glands and xylem parenchyma cells contain calcium oxalate crystals or starch granules. The vessels sometimes contain vivid yellow or reddish brown recinous substances.
It occurs as odorless and has an extremely bitter and persistent taste.

Identification Weigh 1 g of pulverized Picrasma Wood, add 10 mL of methanol, perform cold extraction overnight, filter and evaporate to dryness. Dissolve the residue in 1 mL of methanol and use this solution as the test solution. With this solution, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL of the test solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography (with fluorescent agent). Next, develop the plate with a mixture of ethyl acetate, methanol and water (100 : 13.5 : 10) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 365 nm): a spot with yellowish green fluorescence is observed at the R_f value of about 0.2.

Purity (1) ***Foreign matter***— Less than 1.0%.
(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm
(iv) Cadmium: NMT 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

Ash NMT 4.0%.

Acid-insoluble Ash NMT 1.0%.

Packaging and storage Preserve in well-closed containers.

Pinellia Tuber
반하(半夏)

Pinelliae Tuber

Pinellia Tuber is the tuber of *Pinellia ternata* Breitenbach (Araceae) from which the periderm has been removed.

Description Pinellia Tuber is the tuber that has the periderm completely removed, is slightly flattened spherical to irregular spherical, 7 mm to 25 mm in diameter and 7 mm to 15 mm in height. The outer surface occurs as white to grayish white-yellow. The upper end is dented where the stem has been removed and has root scars that are densely dented as numerous small spots around the circumference. The texture is dense and difficult to cut. The cross-section is white and powdery.
Under the microscope, the cross-section shows that cork cells remain on the outside. The 10 to 12 layers of parenchyma cells near the cork cells contain a relatively small amount of starch granules or sometimes none at all, but the parenchyma cells inside are filled with starch granules. The parenchyma cells contain raphide bundles of calcium oxalate and mucilage. The vascular bundles are collateral and amphivasal, distributed transversely and longitudinally. The vessels are mostly spiral, sometimes with annular vessels.
It occurs as almost odorless and the taste is initially light, slightly mucilaginous, but leaves a strong acrid taste.

Identification Weigh 1 g of pulverized Pinellia Tuber and Pinellia tuber RMPM, add 10 mL of methanol, sonicate for 60 minutes, filter the solutions and use the filtrates as the test solution and the Pinellia tuber RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Pinellia tuber RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of chloroform and methanol (95 : 0.5) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS to the plate and heat it at 105 °C for 10 minutes; the several spots obtained from the test solution show the same color and R_f value as the spots from the Pinellia tuber RMPM standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.
(4) ***Mycotoxins***—Total aflatoxin (sum of aflatoxins B_1, B_2, G_1 and G_2): NMT 15.0 ppb (aflatoxin B_1 is NMT 10.0 ppb).

Loss on drying NMT 14.0% (6 hours).

Ash NMT 3.5%.

Packaging and storage Preserve in well-closed containers.

Plantago Seed
차전자(車前子)

Plantaginis Semen

Plantago Seed is the ripe seed of *Plantago asiatica* Linné or *Plantago depressa* Willdenow (Plantaginaceae).

Description Plantago Seed is the seed, flattened ellipsoidal, 2 to 2.5 mm in length, 0.7 to 1 mm in width and 0.3 to 0.5 mm in thickness. The external surface is lustrous, exhibiting a brown to yellowish brown color. 100 seeds of Plantago Seed weigh about 50 mg. Under a magnifying glass, the surface of the seed is practically smooth, with the dorsal side protruding like a bow and with the ventral side slightly dented. The micropyle and the raphe are not visible.

Under a microscope, a transverse section of Plantago Seed reveals the outermost mucous layer and the very thin cell wall, which swells on contact with water, below which is the pigment layer. The cells in the pigment layer are trapezoid with a straight lower surface and contain brown pigments. The endosperm cells consist of several rows with slightly thick cell walls, are close to rectangular in a mosaic arrangement, and contain fatty oil droplets. The cotyledon cells are regularly arranged and contain aleurone grains.

It is odorless and tastes slightly bitter and mucilaginous.

Identification (1) Weigh 1 g of Plantago Seed, add 2 mL of warm water, and allow to stand for 10 minutes; the seed coat swells to discharge mucilage

(2) Weigh 1 g of Plantago Seed, boil gently with 10 mL of dilute hydrochloric acid for 2 minutes, and filter. Neutralize the filtrate with sodium hydroxide TS. To 3 mL of this solution, add 1 mL of Fehling's TS, and warm the mixture; a red precipitate is formed.

(3) Weigh 1 g of pulverized Plantago Seed, add 10 mL of methanol, sonicate for 30 minutes to extract, and filter. Vacuum-concentrate the filtrate, dissolve in 2 mL of methanol, and use this solution as the test solution. Separately, weigh 1 mg of geniposidic acid RS and 1 mg of acteoside RS, dissolve each in 1 mL of methanol, and use these solutions as the standard solution (1) and the standard solution (2), respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the standard solutions (1) and (2) on the thin-layer chromatographic plate made of silica gel (with a fluorescent agent) for thin layer chromatography. Next, develop the plate with a mixture of ethyl acetate, methanol, formic acid and water (36 : 4 : 3 : 2) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 254 nm); the color and the R_f values of two spots among the several spots obtained from the test solution are the same as those of the spots obtained from the standard solutions (1) and (2).

Purity (1) ***Foreign matter***—The amount of foreign matters contained in Plantago Seed is less than 2.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Methoxychlor: NMT 1 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(v) Aldrin: NMT 0.01 ppm.
(vi) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 5.5%.

Acid-insoluble ash NMT 2.0%.

Packaging and storage Preserve in well-closed containers.

Platycodon Root
길경(桔梗)

Platycodonis Radix

Platycodon Root is the root of *Platycodon grandiflorum* A. De Candolle (Campanulaceae), with or without the periderm.

Description Platycodon Root is the root, thin, long fusiform or conical, often branched. The main root is 10 - 15 cm in length and 1 - 3 cm in diameter. The external surface occurs as a grayish brown, pale brown or white color. The upper end of the root has a dented scar of the removed stem, near of which there are fine horizontal wrinkles and longitudinal furrows. Most part of the root, except the crown, is covered with coarse longitudinal wrinkles, horizontal furrows and lenticel-like horizontal lines. The texture is hard, but easy to break. The transverse section is not fibrous, the cortex is slightly thinner than the xylem, almost white with scattered gaps. The area close to the cambium exhibits a brown color. The xylem occurs as a white to pale brown color, and the tissue is slightly denser than the cortex.

Under a microscope, the transverse section reveals a yellowish brown cork layer, most of which has been removed. The phloem is wide, the phloem rays on the outside are bent, and the phloem bundles are mostly compressed and degenerated. The lactiferous tubes are scattered in bundles and contain a yellowish brown granular substance. The bundles of the lactiferous tubes are arranged inside of the phloem along with the sieve tubes. The cambium forms a ring pattern. The xylem has wide medullary rays and polygonal vessels, solitary or several gathered together in a radiating arrangement.

It has a slight, characteristic odor and has a taste plain at first, and acrid and bitter later.

Identification (1) Weigh 0.5 g of pulverized Platycodon Root, boil with 10 mL of water, allow to cool, and shake the mixture vigorously; a lasting fine foam is produced.

(2) Weigh 0.2 g of pulverized Platycodon Root, warm with 2 mL of acetic anhydride on a water bath for 2 minutes, and filter. To l mL of the filtrate, carefully add 0.5 mL of sulfuric acid to make two layers; a red to reddish brown color develops at the zone of contact, and the upper layer exhibits a bluish green to green color.

(3) To 1 g each of pulverized Platycodon Root and Platycodon root RMPM, add 50 mL each of methanol, connect with a reflux condenser, heat for 1 hour, and filter. Vacuum-concentrate each filtrate until they become 5 mL, add 20 mL each of ether, collect the precipitate, dissolve the precipitate each in 2 mL of ethanol, and use these solutions as the test solution and the Platycodon root RMPM standard solution. With these solutions,

perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Platycodon root RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of cyclohexane and acetone (4:1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS on the plate and heat at 105 °C for 10 minutes; the color and the R_f values of the several spots obtained from the test solution are the same as those of the spots obtained from the Platycodon root RMPM standard solution. Of these spots, reddish brown spots appear at the R_f values of about 0.25 and 0.4.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Napropamide: NMT 0.1 ppm.
(ii) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'- DDT and p,p'-DDT): NMT 0.1 ppm.
(iii) Dieldrin: NMT 0.01 ppm.
(iv) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(v) Aldrin: NMT 0.01 ppm.
(vi) Endrin: NMT 0.01 ppm.
(vii) Thiomethionate: NMT 0.3 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 6.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 25.0%.

Packaging and storage Preserve in well-closed containers.

Platycodon Fluid Extract
길경유동엑스

Method of preparation Weigh coarse powder of Platycodon Root and prepare the fluid extract as directed under Fluid Extracts using 25 vol% ethanol. However, an appropriate quantity of Ethanol and Purified Water may be used in place of 25 vol% Ethanol.

Description Platycodon Fluid Extract occurs as a reddish brown liquid. It is miscible with water, producing slight turbidity. It has a mild taste at first, followed by an acrid and bitter taste.

Identification (1) Add 0.5 mL of Platycodon Fluid Extract to 10 mL of water and shake vigorously to mix; a lasting fine foam is produced.
(2) Dissolve 1 drop of Platycodon Fluid Extract in 2 mL of acetic anhydride and carefully add 0.5 mL of sulfuric acid; the zone of contact exhibits a red to reddish brown color.

Purity (1) ***Starch***—Mix 1 mL of Platycodon Fluid Extract with 4 mL of water and add 1 drop of dilute iodine TS; the resulting solution does not exhibit a purple or blue color.
(2) ***Heavy metals***—Total heavy metals: NMT 30 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

Residue on evaporation Transfer 5 mL of Platycodon Fluid Extract in a beaker, previously weighed accurately, evaporate to dryness on a water bath, and dry at 105 °C for 5 hours; the amount of residue is NLT 0.50 g.

Packaging and storage Preserve in light-resistant, tight containers.

Pogostemon Herb
광곽향(廣藿香)

Pogostemonis Herba
Pogostemon Herb is the aerial part of *Pogostemon cablin* Bentham (Labiatae).

Description Pogostemon Herb is the aerial part, consisting of the stem and the opposite leaves on the stem. Stems are square-columnar, frequently branched, 30 - 60cm in length and 0.2 - 0.7 cm in diameter. The external surface is covered with soft hairs. The texture is fragile and easy to break. The center of the fractured surface reveals the pith. Leaves are opposite, crumpled into masses, ovate or elliptical, when unfolded, 4 - 9 cm in length and 3 - 7 cm in width. The leaves are covered with grayish white fine soft hairs on both surfaces. The apex of the leaf is short-acute or obtuse-rounded, the base is wedge-shaped or obtuse-rounded, the margin of the leaf is irregular in size and bluntly serrated. The petioles are slender, 2 - 5 cm in length, coated with soft hairs.
It has a characteristic odor and a slightly bitter taste.

Identification Weigh 0.5 g each of pulverized Pogostemon Herb and pogostemon herb RMPM, add 5 mL each of methanol, shake for 3 minutes to mix, filter, and use each filtrate as the test solution and the pogostemon herb RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate using a mixture of hexane and acetone (9 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly vanillin-sulfuric acid TS on the plate and heat the plate at 105 ℃ for 10 minutes; the color and the R_f value of the several spots obtained from the test solution are the same as those of spots from the pogostemon herb RMPM standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 13.0% (6 hours).

Ash NMT 13.0%.

Essential oil content NLT 0.3 mL (50.0 g).

Packaging and storage Preserve in well-closed containers.

Polygala Root
원지(遠志)

Polygalae Radix

Polygala Root is the root of *Polygala tenuifolia* Willdenow (Polygalaceae).

Polygala Root contains NLT 2.0% of tenuifolin ($C_{36}H_{56}O_{12}$: 680.37), calculated on the dried basis.

Description Polygala Root is a cylindrical, thin, long and curved root. The main root is 10 cm to 20 cm long and 2 mm to 10 mm in diameter. The outer surface occurs as pale grayish yellow to grayish brown with a relatively dense and deeply indented transverse folds, longitudinal folds and open crevices. Older roots have relatively dense transverse folds that are even more deeply indented and slightly knotted. The texture is hard, brittle and easy to cut. The cut surface has a yellowish brown cortex and a yellowish white xylem. The cortex and xylem are separate and easily detached, sometimes the core is already removed.

Under the microscope, the cross section shows a cork layer consisting of about 10 rows of cork cells. The cells in the 1 to 2 outer rows are rectangular. The cortex is narrow and the phloem is relatively wide with open gaps throughout. The cambium forms a ring. Those in which the core has not been removed have a xylem. Several vessels form groups and are scattered, surrounded by lignified xylem fiber bundles. The xylem rays consist of 1 to 3 rows of cells. Most parenchyma cells contain fatty oil droplets, sometimes calcium oxalate druse or single crystals.

It has a slight, characteristic odor and slightly acrid taste.

Identification (1) Weigh 0.5 g of pulverized Polygala Root, add 10 mL of water, and shake vigorously to mix; persistent, fine bubbles are formed.

(2) Weigh 0.5 g of pulverized Polygala Root, add 2 mL of acetic anhydride, and shake well to mix. Allow the mixture to stand for 2 minutes, filter, and gently add 1 mL of sulfuric acid to the filtrate; the contact surface shows a reddish brown color, and the supernatant shows a light bluish green to brown color.

(3) Weigh 1 g each of pulverized Polygala Root and Polygala root RMPM, add 20 mL of a solution of hydrochloric acid in ethanol (1 in 10), heat the mixture after connecting with a reflux condenser for 30 minutes, and filter. To the filtrate, add 30 mL of water and extract twice with 20 mL of ethyl acetate. Combine the extracts and evaporate to dryness. Dissolve the residue in 1 mL of ethyl acetate, filter, and use each of the filtrates as the test solution and the Polygala root RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the Polygala root RMPM standard solution on a thin-layer chromatographic plate made of silica gel (with fluorescent indicator) for thin layer chromatography. Develop the plate with a mixture of ethyl acetate, hexane, and formic acid (10 : 4 : 0.5) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray dilute sulfuric acid TS evenly to the plate, heat it at 105 ℃ for 10 minutes, and examine the plate under ultraviolet light (wavelength: 365 nm); the spots obtained from the test solution show the same color and R_f value as those obtained from the standard solution.

Purity (1) ***Foreign matter***—(i) Stem: Polygala Root contains less than 10.0% of the stem.

(ii) Other foreign matter: Polygala Root contains less than 1.0% of foreign matter other than the stem.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): NMT 0.2 ppm.

(vi) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

(5) ***Mycotoxins***—Total aflatoxins (sum of aflatoxins B_1, B_2, G_1 and G_2): NMT 15.0 ppb (aflatoxin B1 is NMT 10.0 ppb).

Loss on drying NMT 12.0%.

Ash NMT 6.0%.

Assay Weigh accurately about 1 g of pulverized Polygala Root, add 50 mL of a mixture of methanol and water (7 : 3), sonicate for 1 hour, and filter. Add a mixture of methanol and water (7 : 3) to the filtrate to make exactly 50 mL. Pipet 25 mL of this solution and evaporate to dryness. Add 50 mL of 10% sodium hydroxide to the residue, heat the mixture after connecting with a reflux condenser for 2 hours, and filter. After cooling down the filtrate, add hydrochloric acid to adjust the pH to 4 to 6. Extract the mixture three times with 50 mL of water-saturated butanol, combine the water-saturated butanol layer, and concentrate it by evaporation. To the residue, add methanol to make exactly 25 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of tenuifolin RS (previously dried in a silica gel desiccator for NLT 12 hours), add methanol to make exactly 25 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas, A_T and A_S, of the test solution and the standard solution.

$$\text{Amount (mg) of tenuifolin } (C_{36}H_{56}O_{12}) = \text{Amount (mg) of tenuifolin RS} \times \frac{A_T}{A_S} \times 2$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 210 nm)

Column: A stainless steel column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (particle diameter: 5 μm to 10 μm).

Column temperature: A constant temperature of about 25 °C.

Mobile phase: A mixture of methanol and a solution prepared by dissolving 1.2 g of sodium dihydrogen phosphate in 1000 mL of water and adjusting the pH to 2.0 with phosphoric acid (7 : 3).

Flow rate: 0.5 mL/min

System suitability

System repeatability: Perform the test 6 times with 10

μL of the standard solution according to the above conditions; the relative standard deviation of the peak areas of tenuifolin is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Polygonatum Rhizome
황정(黃精)

Polygonati Rhizoma

Polygonatum Rhizome is the steamed rhizome of *Polygonatum sibiricum* Redoute, *Polygonatum falcatum* A. Gray, *Polygonatum kingianum* Coll. et Hemsley or *Polygonatum cyrtonema* Hua (Liliaceae).

Description Polygonatum Rhizome is the irregular, cylindrical or massive rhizome, 3 cm to 10 cm in length and 5 mm to 30 mm in diameter, occasionally branched. The external surface occurs as yellowish brown to blackish brown, transverse nodes, semi-transparent. The upper part of the node shows orbiculate stem scar with a dented circumference. The lower part bears prominent root scars, several scale nodes and thin longitudinal wrinkles. Texture is hard and tenacious, fractured surface is pale brown, translucent and horny with numerous yellowish white small spots.
Under a microscope, a transverse section reveals epidermis is covered with cuticle, parenchyma tissue lies inside of epidermis. Numerous vascular bundles and mucilage cells are scattered in parenchyma tissue. Vascular bundles are collateral or amphivasal bundles. Mucilage cells contain raphides of calcium oxalate.
It has a slight sweet odor and sweet taste, and sticky when chewed.

Identification (1) Weigh 0.5 g of pulverized Polygonatum Rhizome, add 2 mL of acetic anhydride, warm on a water bath for 2 minutes, and filter. To 1 mL of the filtrate, add carefully 0.5 mL of sulfuric acid; a reddish brown color appears at the zone of contact.

(2) Weigh 1 g of pulverized Polygonatum Rhizome, add 10 mL of dilute hydrochloric acid, heat slowly for 2 minutes, and filter. Neutralize the filtrate with sodium hydroxide TS. To 3 mL of the filtrate, add 1 mL of Fehling's TS and warm; a red precipitate is formed.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 5.0%.

Acid-insoluble ash NMT 1.0%.

Packaging and storage Preserve in well-closed containers.

Polygonum Multiflorum Root
하수오(何首烏)

Polygoni Multiflori Radix

Polygonum Multiflorum Root is the tuber of *Polygonum multiflorum* Thunberg (Polygonaceae).
Polygonum Multiflorum Root contains NLT 0.75% of 2,3,5,4'-tetrahydroxy-stilbene-2-O- β-D-glucoside ($C_{20}H_{22}O_9$: 406) and NLT 0.10% in total of emodin ($C_{15}H_{10}O_5$: 270.24) and physcion ($C_{16}H_{12}O_5$: 284.27), calculated on the dried basis.

Description Polygonum Multiflorum Root is the tuber in fusiform or in masses, 5 cm to 15 cm in length, 3 cm to 10 cm in diameter. External surface occurs as reddish brown to blackish brown, with slightly bumpy, uneven, shallow furrows, irregular wrinkles and longitudinal furrows. It has transversely long lenticels or continuous stripes. Both ends have a distinct cut scar and fibrous vascular bundles exposed. The texture is tough, hard and difficult to be cut. The cut surface occurs as pale yellowish brown or pale reddish brown, powdery, with 4 to 11 nearly orbicular hetero-vascular bundle rings gathered to form a floral pattern known as "Geummun." The xylem in the center is relatively large, sometimes with the core observed.
Under a microscope, the transverse section reveals a cork layer consisting of several rows of cells, which contain brown substances inside. The phloem is relatively broad and is scattered with 4 to 11 nearly orbicular hetero-vascular bundles, that is, complex vascular bundles. Separately, a type of vascular bundle is present at the center of the root, and this vascular bundle is solitary. All vascular bundles are lateral. The cambium forms a ring. In the xylem, the vessels are relatively few and are surrounded by tracheids and a small number of xylem fibers. The primary xylem is at the center of the root. The parenchyma cells contain calcium oxalate druses and starch grains.
It occurs as odorless and tastes slightly bitter and astringent.

Identification (1) Drop the ammonia TS to the pulverized Polygonum Multiflorum Root; a deep red color appears.

(2) Weigh about 2 g of Polygonum Multiflorum Root, add 10 mL of water, heat and filter. Add 1 to 2 droplets of the Iron(III) chloride TS to 1 mL of the filtrate: a purple to blue color develops

(3) Weigh 1 g each of pulverized Polygonum Multiflorum Root and Polygonum Multiflorum Root RMPM, add 10 mL of methanol, sonicate for 60 minutes, filter and use the filtrates as the test solution and the Polygonum Multiflorum Root RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution of Polygonum Multiflorum Root RMPM on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography (with fluorescent indicator). Next, develop the plate with a mixture of ethyl acetate, methanol, water and acetic acid (200 : 10 : 10 : 3) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 365 nm): the several spots obtained from the test solution show the same color and R_f value as the spots from the Polygonum Multiflorum Root RMPM standard solution and among these, the spot of 2,3,5,4'- tetrahydroxystilbene-2-O-β-D-glucoside appears at the R_f value of about 0.35.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(2) *Residual pesticides*—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(3) Sulfur dioxide—NMT 30 ppm.

Loss on drying NMT 14.0%.

Ash NMT 5.0%.

Acid-insoluble Ash NMT 1.5%.

Extract Content ***Dilute ethanol-soluble extract***—NLT 17.0%

Assay (1) ***2,3,5,4'-Tetrahydroxystilbene-2-O-β-D- glucoside***—Weigh accurately 0.2 g of pulverized Polygonum Multiflorum Root, add 50 mL of methanol, sonicate for 60 minutes, filter and use the filtrate as the test solution. Separately, weigh accurately about 10 mg of 2,3,5,4'-tetrahydroxystilbene-2-O-β-D-glucoside RS and dissolve in methanol to make exactly 100 mL. Pipet 25 mL of this solution, add methanol to make exactly 100 m L and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under Liquid Chromatography according to the following operating conditions and determine the peak areas, A_T and A_S, of the test solution and the standard solution.

Amount (mg) of 2,3,5,4'-tetrahydroxystilbene-2-O-β-D-glucoside ($C_{20}H_{22}O_9$)
= Amount (mg) of 2,3,5,4'-tetrahydroxystilbene-2-O-β-D-glucoside RS × A_T/A_S × 1/8

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 320 nm)

Column: A stainless steel column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Column temperature: A constant temperature about 30 ℃.

Mobile phase: Control the mixing ratio of mobile phase A and mobile phase B stepwise or via the concentration gradient as follows.

Mobile phase A: A mixture of water and acetic acid (200 : 1).

Mobile phase B: A mixture of acetonitrile and acetic acid (200 : 1).

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)
0	85	15
35	60	40
40	85	15

Flow rate: 1.0 mL/min

System suitability

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution under the above operating conditions; the relative standard deviation of the peak area of 2,3,5,4'-tetrahydroxystilbene-2-O-β-D-glucoside is NMT 1.5%.

(2) ***Emodin and physcion***—Weigh accurately 1 g of pulverized Polygonum Multiflorum Root, add 50 mL of methanol, heat after connecting with a reflux condenser for 1 hour and filter. Pipet 25 mL of the filtrate, evaporate to dryness, add 20 mL of hydrochloric acid solution (8 in 100) and sonicate for 5 minutes. To the extract, add 20 mL of chloroform, heat after connecting with a reflux condenser for 1 hour and transfer to a separatory funnel. Wash the container with a small amount of chloroform, add the washing to the separatory funnel, shake and separate the chloroform layer. Extract the hydrochloric acid layer with three times with 15 mL each of chloroform, combine the chloroform layers and concentrate under vacuum. To the residue, add methanol to make exactly 10 mL and use this solution as the test solution. Separately, weigh accurately about 10 mg of emodin RS, dissolve in methanol to make exactly 10 mL, weigh accurately about 10 mg of physcion RS and dissolve in methanol to make exactly 100 mL. Pipet 5 mL of the emodin solution and 25 mL of the physcion solution, add methanol to make exactly 50 mL and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under Liquid chromatography according to the following operating conditions and determine the peak areas, A_{Ta} and A_{Tb}, of emodin and physcion in the test solution and the peak areas, A_{Sa} and A_{Sb}, of emodin and physcion in the standard solution.

$$\text{Amount (mg) of emodin } (C_{15}H_{10}O_5) = \text{Amount (mg) of emodin RS} \times \frac{A_{Ta}}{A_{Sa}} \times \frac{1}{5}$$

$$\text{Amount (mg) of physcion } (C_{16}H_{12}O_5) = \text{Amount (mg) of physcion RS} \times \frac{A_{Tb}}{A_{Sb}} \times \frac{1}{10}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 254 nm)

Column: A stainless steel column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Column temperature: A constant temperature about 30 ℃.

Mobile phase: A mixture of acetonitrile, water and phosphoric acid (850 : 150 : 1)

Flow rate: 0.5 mL/min

System suitability

System performance: Proceed with 10 μL of the standard solution according to the above conditions; emodin and physcion are eluted in this order with the resolution being NLT 3.

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of each peak area of emodin and physcion is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Polyporus Sclerotium
저령(猪苓)

Polyporus

Polyporus Sclerotium is the sclerotium of *Polyporus umbellatus* Fries (Polyporaceae).

Description Polyporus Sclerotium is the sclerotium, rod-

shaped, close to circular or flattened masses, sometimes branched, 5 to 25 cm in length and 2 to 6 cm in diameter. The external surface occurs as a black, gray or blackish brown color and has wrinkled or hump-like protrusion. The body is light, and the texture is hard. The cut surface occurs as a near white or yellowish white color, usually in a granular shape.
It occurs as odorless and tasteless.

Identification (1) Weigh 0.5 g of pulverized Polyporus Sclerotium, add 5 mL of acetone, warm on a water bath for 2 minutes, while shaking to mix, and filter. Evaporate the filtrate to dryness, dissolve the residue in 5 drops of acetic anhydride, and add 1 drop of sulfuric acid; the resulting solution exhibits a reddish purple color, which immediately changes to a dark green color.

(2) Weigh 1 g of pulverized Polyporus Sclerotium, add 20 mL of methanol, sonicate for 30 minutes to extract, filter, and use this solution as the test solution. Separately, weigh 1 mg of ergosterol RS, dissolve in 5 mL of methanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate using a mixture of petroleum ether and ethyl acetate (3 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly 2% vanillin-sulfuric acid TS on the plate and heat at 105 °C; the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 1.0 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 16.0%.

Acid-insoluble ash NMT 4.0%.

Packaging and storage Preserve in well-closed containers.

Poncirus Immature Fruit
지실(枳實)

Ponciri Fructus Immaturus

Poncirus Immature Fruit is the unripe fruit of *Poncirus trifoliata* Rafinesque (Rutaceae).

Poncirus Immature Fruit contains NLT 2.0% of poncirin ($C_{28}H_{34}O_{14}$: 594.28) and NLT 0.7% of naringin ($C_{27}H_{32}O_{14}$: 580.55), calculated on the dried basis.

Description Poncirus Immature Fruit is the fruit, almost spherical and 1 to 2 cm in diameter. The external surface occurs as a brown to deep brown color and is rough with numerous dented spots of oil sacs and pale green hairs. The epidermal side of the transverse section occurs as a yellowish brown color, while the inner side occurs as a pale grayish brown color, of which center consists of about 8 small loculi radiating out. Each loculus is dried out, exhibiting a yellowish brown color, and dented, sometimes containing unripe seeds.
It has a characteristic odor and a bitter taste.

Identification (1) Weigh 0.5 g each of pulverized Poncirus Immature Fruit and Poncirus immature fruit RMPM, add 10 mL each of ethanol, shake well to mix, allow to stand for 30 minutes, filter, and use the filtrates as the test solution and the poncirus immature fruit RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Poncirus immature fruit RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate using a mixture of dichloromethane, methanol and water (60 : 21 : 2) as a developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS and heat at 105 °C for 10 minutes; the color and the R_f values of the several spots obtained from the test solution are the same as those of the spots obtained from the Poncirus immature fruit RMPM standard solution. Of these spots, two yellow spots appear at the R_f values of about 0.45 and about 0.6 from both the test solution and the Poncirus immature fruit RMPM standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): NMT 0.2 ppm.
(vi) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 12.0%.

Ash NMT 7.0%.

Assay Weigh accurately about 0.1 of pulverized Poncirus Immature Fruit, add 50 mL of diluted methanol (7 in 10), sonicate for 1 hour to extract, and filter. Combine all the filtrates, add diluted methanol (7 in 10) to make exactly 50 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg each of poncirin RS and naringin RS (previously dried in a desiccator with silica gel for 24 hours), add diluted methanol (7 in 10) to make exactly 50 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions, and determine the peak areas, A_{Ta} and A_{Tb}, of poncirin and naringin, respectively, from the test solution and the peak areas, A_{Sa} and A_{Sb}, of poncirin and naringin, respectively, from the standard solution.

$$\text{Amount (mg) of poncirin } (C_{28}H_{34}O_{14}) = \text{Amount (mg) of poncirin RS} \times \frac{A_{Ta}}{A_{Sa}}$$

Amount (mg) of naringin ($C_{27}H_{32}O_{14}$)

$$= \text{Amount (mg) of naringin RS} \times \frac{A_{Tb}}{A_{Sb}}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 313 nm)

Column: A stainless steel column 4 to 6 mm in internal diameter and 15 to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 to 10 μm in particle diameter).

Mobile phase: Control the mobile phases A and B stepwise as directed in the following table or via the concentration gradient.

Mobile phase A: Diluted acetic acid (1 in 100)

Mobile phase B: A mixture of acetonitrile and acetic acid (100 : 1)

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)
0	90	10
30	35	65
35	10	90
40	10	90
45	90	10

Flow rate: 1.0 mL/min

System suitability

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak areas of poncirin is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Poria
복령(茯苓)

Poria Sclerotium

Poria is the sclerotium of *Poria cocos* Wolf (Polyporaceae).

Description Poria is the sclerotium, in masses, usually as broken or fragmented pieces, unbroken ones are 10 cm to 30 cm in diameter and weigh 0.1 kg to 2 kg. The remaining outer layer occurs as dark brown to dark reddish brown, coarse, fissured. The inside occurs as white or pale reddish white. The texture is firm, but brittle.

It occurs as almost odorless, the taste is weak and slightly mucous.

Identification (1) Weigh 1 g of pulverized Poria, add 5 mL of acetone, warm on a water bath for 2 minutes while shaking, and filter. Evaporate the filtrate to dryness, dissolve the residue in 0.5 mL of acetic anhydride, and add 1 drop of sulfuric acid; the resulting solution exhibits a pale red color, which changes immediately to dark green.

(2) Take a section or powder of Poria and add 1 drop of iodine TS; a deep reddish brown color is produced.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 18.0%.

Ash NMT 1.0%.

Packaging and storage Preserve in well-closed containers.

Prunella Spike
하고초(夏枯草)

Prunellae Spica

Prunella Spike is the spike of *Prunella vulgaris* Linné var. *lilacina* Nakai or *Prunella vulgaris* Linné (Labiatae).

Description Prunella Spike is the spike, nearly cylindrical with many bracts and calyxes attached, 3 cm to 6 cm in length and 10 mm to 15 mm in diameter. External surface occurs as grayish brown to reddish brown, and the texture is light. Corollas are often remained on the upper part and stems are remained on the lower part. A calyx usually enclosed four mericarps. Bract is cordate to eccentric, and exhibits white hairs on the calyx and the vein.

It occurs as almost odorless and tasteless.

Identification Weigh 1 g of pulverized Prunella Spike, add 20 mL of ethanol, warm on a water bath for 1 hour after connecting with a reflux condenser, filter, and evaporate the filtrate to dryness. To the residue, add 15 mL of petroleum ether, shake to mix for 2 minutes, and discard the petroleum ether layer. Dissolve the residue in 1 mL of ethanol, and use this solution as the test solution. Separately, weigh 1 mg of ursolic acid RS, dissolve in 1 mL of ethanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 2 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate and methanol (40 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly the plate with dilute sulfuric acid TS and heat at 105 °C; one spot among the several spots obtained from the test solution shows the same color and R_f value as the reddish purple spot obtained from the standard solution.

Purity (1) ***Foreign matter***—(i) Stem: Prunella Spike contains less than 5.0% of the stems.

(ii) Other foreign matter: The amount of foreign matter other than the stems contained in Prunella Spike is less than 1.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(4) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 13.0%.

Acid-insoluble ash NMT 5.0%.

Packaging and storage Preserve in well-closed containers.

Pueraria Root
갈근(葛根)

Puerariae Radix

Pueraria Root is the root of *Pueraria lobata* Ohwi (Leguminosae) with or without periderm.

Pueraria Root contains NLT 2.0% of puerarin ($C_{21}H_{20}O_9$: 416.38) and NLT 0.3% of daidzin ($C_{21}H_{20}O_9$: 416.38), calculated on the dried basis.

Description Pueraria Root is the root and is cut into thick rectangular pieces or cut vertically into small masses. The former are 20 - 30 cm in length and about 1 cm in thickness, and the latter are close to hexahedrons of irregular sizes. External surface occurs as grayish white to pale brown, longitudinally wrinkled and coarse. It is easily breakable lengthwise. Under a magnifying glass, the transverse section is fibrous and shows concentric annulate ring or part of it formed by abnormal growth of the cambium. The phloem occurs as a pale grayish yellow color, and the xylem exhibits numerous vessels appearing as small dots. The medullary rays occur as a pale grayish yellow color and are slightly dented.

When the traverse section of Pueraria Root is examined under a microscope, most of the cortex has been removed. In the xylem, the medullary rays consist of 3 to 8 rows of cells, and several vessels form groups in an alternating arrangement with xylem fiber bundles. There are many fiber bundles, usually with several tens of bundles arranged in a ring shape. The parenchyma cells of the xylem contain solitary crystals of calcium oxalate and a small amount of starch grains

It has a slight odor and a slightly sweet taste.

Identification Weigh 2 g each of pulverized Pueraria Root and pueraria root RMPM, add 10 mL each of methanol, shake for 3 minutes to mix, filter, and use each filtrate as the test solution and the pueraria root RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 2 μL each of the test solution and the pueraria root RMPM standard solution on the plate of silica gel (with a fluorescent agent) for thin layer chromatography. Next, develop the plate with a mixture of ethyl acetate, methanol and water (12 : 2 : l) as the developing solution to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wave- length: 254nm); the color and the R_f values of several spots obtained from the test solution and the pueraria root RMPM standard solution are the same. Of these spots, the spots of puerarin and daidzin appear at the R_f values of about 0.5 and 0.55, respectively.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): NMT 0.2 ppm.
(vi) Endrin: NMT 0.01 ppm.
(vii) Captan: NMT 2 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 13.0% (6 hours).

Ash NMT 6.0%.

Assay Weigh accurately about 2 g of pulverized Pueraria Root, add 60 mL of methanol, connect with a reflux condenser, heat for 2 hours, and filter. To the residue, add 30 mL of methanol and proceed in the same manner. Combine all the filtrates and add methanol to make exactly 100 mL. Take 10 mL of this solution, add methanol to make exactly 100 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg each of puerarin RS and daidzin RS (previously dried in a desiccator with silica gel for 24 hours), add methanol to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions, and calculate the peak areas, A_{Ta} and A_{Tb}, of puerarin and daidzin, respectively, from the test solution and the peak areas, A_{Sa} and A_{Sb}, of puerarin and daidzin, respectively, from the standard solution.

$$\text{Amount (mg) of puerarin } (C_{21}H_{20}O_9) = \text{Amount (mg) of puerarin RS} \times \frac{A_{Ta}}{A_{Sa}} \times 10$$

$$\text{Amount (mg) of daidzin } (C_{21}H_{20}O_9) = \text{Amount (mg) of daidzin RS} \times \frac{A_{Tb}}{A_{Sb}} \times 10$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 254 nm)

Column: A stainless steel column 4 - 6 mm in internal diameter and 15 - 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 - 10 μm in particle diameter).

Column temperature: An ordinary temperature

Mobile phase: Control the mobile phases A and B stepwise as directed in the following table or via the concentration gradient.

Mobile phase A: Methanol
Mobile phase B: Water

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)
0	25	75
20	25	75
30	45	55
40	55	45
45	25	75
50	25	75

Flow rate: 1.0 mL/min
System suitability
System performance: Proceed with 10 μL of the standard solution according to the above conditions. However, adjust the concentration-gradient condition so that puerarin and daidzin are eluted in this order with clear separation of each peak.
System repeatability: Repeat the test 6 times with 10 μL each of the standard solution under the above conditions; the relative standard deviations of each peak area of puerarin and daidzin are NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Raphanus Seed
내복자(萊菔子)

Raphani Semen
Raphanus Seed is the ripe seed of *Raphanus sativus* Linné (Cruciferae).

Description Raphanus Seed has an oval or nearly oval shape, is slightly flat, and 2.5 to 4 mm long and 2 to 3 mm wide. The outer surface occurs as yellowish brown to reddish brown or grayish brown, with a dark brown, rounded hilum at one end and several rows of vertical grooves at the other end. The seed coat is thin and crumbles easily. There are two cotyledons, which are yellowish white and oily.
Under the microscope, the cross-section shows a pigment layer adhering to the palisade layer and atrophying, with a reddish brown substance inside and the endosperm cells flattened in one line, with starch grains inside.
It occurs as almost odorless and has a light, slightly bitter and spicy taste.

Identification Weigh 1 g each of pulverized Raphanus Seed and Raphanus seed RMPM, dissolve separately in 50 mL of diluted methanol (4 in 5), and heat after connecting with a reflux condenser for 1 hour. Then, filter and evaporate the filtrates to dryness. Dissolve each of the residues in 2 mL of methanol and use these solutions as the test solution and the Raphanus seed RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the Raphanus seed RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of ethyl acetate, water, and formic acid (10 : 3 : 2) as the developing solvent to a distance of about 10 cm and air-dry the plate. Spray p-anisaldehyde-sulfuric acid TS evenly on the plate and heat at 105 °C; the several spots obtained from the test solution show the same color and R_f value as spots from the Raphanus seed RMPM standard solution, and of these, a blue-green spot appears at the R_f value of about 0.5.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) **Sulfur dioxide**—NMT 30 ppm.

Loss on drying NMT 8.0%.

Ash NMT 7.0%.

Acid-insoluble ash NMT 1.0%.
Extract content ***Ether-soluble extract***—NLT 31.0%.

Packaging and storage Preserve in well-closed containers.

Rehmannia Root
지황(地黃)

Rehmanniae Radix
Rehmannia Root is the root of *Rehmannia glutinosa* Liboschitz ex Steudel (Scrophulariaceae).

Description Rehmannia Root is the root, cylindrical to fusiform, often curved, 5 to 15 cm in length and 5 to 15 mm in diameter. The external surface occurs as a yellowish brown to blackish brown color, with deep, longitudinal wrinkles and transverse scars of lateral roots and lenticels. The texture is soft and easily breakable. Under a magnifying glass, the transverse section reveals a yellowish brown to blackish brown surface, where the cortex exhibits a darker color than the xylem and the pith is hardly visible.
Under a microscope, the transverse section of Rehmannia Root reveals the cork layer consisting of several rows of the cork cells. The cortex has a sparse arrangement of parenchyma cells and is scattered with numerous secretory cells which contain amber yellow oil droplets. The stone cells, nearly orbicular, are visible at the upper part of the tuberous root. The phloem has relatively fewer secretory cells. The cambium forms a ring. The xylem rays are broad, consisting of 2 to 4 rows. Vessels are rare, radiating in an intermittent and sparse arrangement.
It has a characteristic odor and a slightly sweet taste at first, followed by a slight bitter taste.

Identification Weigh 2 g each of pulverized Rehmannia Root and Rehmannia root RMPM, add 20 mL each of methanol, warm after connecting with a reflux condenser for 1 hour, filter, and evaporate the filtrates to dryness. Dissolve the residues in 5 mL each of methanol, and use these solutions as the test solution and the Rehmannia root RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and Rehmannia root RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate using a mixture of dichloromethane, methanol and water (16 : 6 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS on the plate and heat at 105 °C; the color and the R_f values of the several spots obtained from the test solution are the same as those of the spots obtained from the Rehmannia root RMPM standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD,

p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(vi) Difenoconazole: NMT 0.3 ppm.
(ix) Iminoctadine: NMT 0.1 ppm.
(viii) Kresoxim-methyl: NMT 0.1 ppm.
(xxii) Thiram: NMT 0.5 ppm.
(x) Pyrimethanil: NMT 0.2 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.
(4) ***Benzo(a)pyrene***—NMT 5 ppm.

Ash NMT 6.0%.

Acid-insoluble ash NMT 2.0%.

Packaging and storage Preserve in well-closed containers.

Prepared Rehmannia Root
숙지황(熟地黃)

Rehmanniae Radix Preparata

Prepared Rehmannia Root is the processed root of *Rehmannia glutinosa* Liboschitz ex Steudel (Scrophulariaceae).

Prepared Rehmannia Root contains NLT 0.1% of 5-hydroxymethyl-2-furaldehyde ($C_6H_6O_3$: 126.11), calculated on the dried basis.

Method of preparation Select well cleaned Rehmannia glutinosa. Steam and dry them repeatedly in the sun using alcohol, Amomum fruit and Citrus unshiu peel as adjuvants until the inside and outside become black and shiny and the texture is soft, tender and sticky.

Description Prepared Rehmannia Root is the steamed root as an irregular mass, with different size. The outer surface occurs as black, lustrous and sticky. The texture is soft and flexible, difficult to break and the cut surface occurs as black and lustrous.
It has a light characteristic odor and sweet taste.

Identification Weigh 1 g of the Prepared Rehmannia Root, add 20 mL of water or dilute ethanol, shake to mix, and filter. Add 10 mL of Fehling's TS to the filtrate, and heat for a while; a reddish purple to reddish brown precipitate is formed.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.
(4) ***Benzopyrene***—NMT 5 ppb.

Loss on drying NMT 17.0%.

Ash NMT 6.0%.

Acid-insoluble ash NMT 2.5%.

Assay Cut Prepared Rehmannia Root as finely as possible, weigh accurately about 2 g, and add 100 mL of diluted methanol (1 in 2). Heat the mixture for 3 hours after connecting with a reflux condenser and filter. Add 100 mL of diluted methanol (1 in 2) to the residue and operate in the same manner. Combine all filtrates and extract twice with 200 mL of hexane each and discard the hexane layer. Concentrate the remaining water layer in vacuum to less than half its volume and extract twice with 100 mL of ethyl acetate. Combine the extracts and evaporate the solvent in vacuum. Dissolve the residue in methanol to make 20 mL and use this solution as the test solution. Separately, weigh accurately about 10 mg of 5-hydroxymethyl-2-furaldehyde RS, dissolve it in methanol to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under Liquid Chromatography according to the following conditions and determine the peak areas, A_T and A_S, of the test solution and the standard solution.

$$\text{Amount (mg) of 5-hydroxymethyl-2-furaldehyde } (C_6H_6O_3) = \text{Amount (mg) of 5-hydroxymethyl-2-furaldehyde RS} \times \frac{A_T}{A_S} \times \frac{1}{5}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 280 nm)

Column: A stainless column with an inner diameter of 4 mm to 6 mm and a length of 15 cm to 25 cm, packed with octadecylsilyl silica gel for liquid chromatography (particle diameter: 5 μm to 10 μm).

Column temperature: 25 ℃

Mobile phase: A mixture of water and acetonitrile (95 : 5)

Flow rate: 1.0 mL/min

System suitability

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution under the above operating conditions; the relative standard deviation of the peak area of 5-hydroxymethyl-2-furaldehyde is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Rhubarb
대황(大黃)

Rhei Radix et Rhizoma

Rhubarb is usually the root and rhizome of *Rheum palmatum* Linné, *Rheum tanguticum* Maximowicz ex Balf. and *Rheum officinale* Baillon (Polygonaceae), from which periderm has been removed.

Rhubarb contains NLT 0.02% of sennoside A ($C_{42}H_{38}O_{20}$: 862.74), and NLT 1.5% in total of aloe emodin ($C_{15}H_{10}O_5$: 270.24), rhein ($C_{15}H_8O_6$: 284.23), emodin ($C_{15}H_{10}O_5$: 270.24), chrysophanol ($C_{15}H_{10}O_4$: 254.25) and physcion ($C_{16}H_{12}O_5$: 284.27), calculated on the dried basis.

Description Rhubarb is the root and rhizome, ovoid, oblong-ovoid or cylindrical, often cut and trim transversely or longitudinally, 5 to 15 cm long and 4 to 10 cm in diameter. The bark is mostly removed on the outside. Rhubarb without most of the bark has a yellowish brown to pale brown exterior with white and fine

reticulations. The texture is thick and firm. In Rhubarb with a cork layer, the outside occurs as dark brown or blackish red with coarse wrinkles, and the texture is rough and brittle. The cross-section is not fibrous. It occurs as pale grayish brown or brown and has complex patterns of blackish brown tissue interspersed with white and pale brown tissue. This pattern sometimes radiates near the cambium. The pith consists of whorls-shaped tissue radiating from the center of a small brown circle 1 to 3 mm in diameter, arranged in a ring or irregularly scattered.

Under the microscope, the cross-section of Rhubarb of Rheum palmatum shows that the cork layer of the rhizome and the cortex have mostly been removed and sometimes partially preserved. The phloem rays are arranged in 3 to 4 rows, relatively linear and contain brown substances. The cambium consists of flat cells. The xylem rays are relatively dense, consist of 2 to 4 rows of cells and contain deep brown substances. The vessels are rare, sparse and arranged towards the center. The pith part is broad and consists mainly of parenchyma cells with several complex vascular bundles in a single fence or scattered. The complex vascular bundles have a ring-shaped cambium with the phloem in the center, sometimes with visible mucous cavities near the cambium. Outside the cambium is the xylem with medullary rays that spread out in a star shape and contain deep brown substances inside. The parenchyma cells contain many starch grains and large druses of calcium oxalate. In Rhubarb of Rheum tanguticum, the phloem rays of the rhizome consist of 2 to 3 rows that are curved in a wave-like manner. The phloem has many mucilage cavities arranged in concentric rings, there are no xylem rays. Many mucilage cavities are in the star spot. Rhubarb of Rheum officinale has phloem rays of the rhizome consisting of 1 to 2 rows, linear, without mucilage cavities in the xylem, xylem rays and mucilage cavities in the star spot.

It has a characteristic odor and an astringent and bitter taste. When chewed, it tastes gritty and colors the saliva yellow.

Identification Weigh 2 g of pulverized Rhubarb, add 40 mL of a mixture of tetrahydrofuran and water (7 : 3), shake for 30 minutes to mix and centrifuge. Transfer the supernatant to a separatory funnel, add 13 g of sodium chloride and shake it for 30 minutes to mix. Collect the tetrahydrofuran layer and adjust the pH to 1.5 by adding 1 mol/L hydrochloric acid TS. Transfer this solution to another separatory funnel, add 30 mL of tetrahydrofuran, shake for 10 minutes to mix, collect the tetrahydrofuran layer, and use this solution as the test solution. Separately, dissolve 1 mg of sennoside A RS in 4 mL of a mixture of tetrahydrofuran and water (7 : 3) and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 40 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography (with fluorescent indicator). Develop the plate with a mixture of ethyl acetate, n-propanol, water and acetic acid (100) (40 : 40 : 30 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 365 nm); one of the several spots obtained from the test solution and a red fluorescent spot from the standard solution show the same color and the same R_f value.

Purity (1) ***Rhaponticin***—Weigh 0.5 g of pulverized Rhubarb, add 10 mL of ethanol, heat the mixture on a water bath with a reflux condenser for 10 minutes and filter. Use the filtrate as the test solution. With the test solution, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL of the test solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography (with fluorescent indicator). Next, develop the plate with a mixture of isopropyl ether, n-butanol and methanol (26 : 7 : 7) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 365 nm); no spot with blue-violet fluorescence is observed at an R_f value between 0.3 and 0.6, though a bluish white fluorescence may appear.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 13.0% (6 hours).

Ash NMT 13.0%.

Acid-insoluble ash NMT 2.0%.

Assay (1) ***Sennoside A***—Weigh accurately about 0.5 g of pulverized Rhubarb, add exactly 50 mL of sodium bicarbonate solution (1 in 1,000), shake for 30 minutes to mix, filter and use the filtrate as the test solution. Separately, weigh accurately about 10 mg of sennoside A RS (previously dried in a silica gel desiccator for 24 hours), dissolve it in sodium bicarbonate solution (1 in 1,000) to make exactly 50 mL. Pipet 5.0 mL of this solution, add sodium bicarbonate solution (1 in 1,000) to make exactly 20 mL and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under Liquid chromatography according to the following conditions, and determine A_T and A_S of the peak area of each solution.

$$\text{Amount (mg) of sennoside A } (C_{42}H_{38}O_{20}) = \text{Amount (mg) of sennoside A RS} \times \frac{A_T}{A_S} \times \frac{1}{4}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 340 nm)

Column: A stainless column, 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Column temperature: A constant temperature of about 40 °C

Mobile phase: A mixture of diluted acetic acid (1 in 80) and acetonitrile (4 : 1).

Flow rate: Adjust the flow rate so that the retention time of sennoside A is about 15 minutes.

System suitability

System performance: Dissolve 1 mg each of sennoside A RS and naringin RS in sodium bicarbonate solution (1 in 1,000) to make 10 mL. Proceed with 20 μL of this solution according to the above conditions; sennoside A and naringin are eluted in this order with the resolution being NLT 3.

System repeatability: Repeat the test 6 times with 20 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak area of sennoside A is NMT 1.5%.

(2) ***Aloe emodin, rhein, emodin, chrysophanol and physcion***—Weigh accurately about 0.15 g of pulverized Rhubarb, add 25 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour and filter. Pipet 5 mL of the filtrate, concentrate it in vacuum, add 10 mL of 8% hydrochloric acid, and sonicate the mixture for 2 minutes. Add 10 mL of chloroform and heat after connecting with a reflux condenser for 1 hour. Transfer the mixture to a separatory funnel, wash the flask with a small amount of chloroform, combine the washings into the separatory funnel, shake it and collect the chloroform layer. Extract the hydrochloric acid layer with three 10 mL volumes of chloroform, combine the chloroform layers and concentrate in vacuum. Add methanol to the residue to make exactly 10 mL and use this solution as the test solution. Separately, weigh accurately about 10 mg each of aloe emodin, rhein, emodin, chrysophanol and physcion RS and dissolve them separately in methanol to make exactly 100 mL. Pipet 10 mL each of the aloe emodin, rhein, emodin and chrysophanol solutions and 5 mL of the physcion solution, add methanol to make exactly 50 mL and use these solutions as the standard solutions. Perform the test with 10 μL each of the test solution and the standard solutions as directed under Liquid chromatography according to the following conditions. Determine the peak areas, A_{Ta}, A_{Tb}, A_{Tc}, A_{Td} and A_{Te}, of aloe emodin, rhein, emodin, chrysophanol and physcion in the test solution and the peak areas, A_{Sa}, A_{Sb}, A_{Sc}, A_{Sd}, and A_{Se}, of aloe emodin, rhein, emodin, chrysophanol and physcion RS in the standard solution.

$$\text{Amount (mg) of aloe emodin } (C_{15}H_{10}O_5) = \text{Amount (mg) of aloe emodin RS} \times \frac{A_{Ta}}{A_{Sa}} \times \frac{1}{10}$$

$$\text{Amount (mg) of rhein } (C_{15}H_8O_6) = \text{Amount (mg) of rhein RS} \times \frac{A_{Tb}}{A_{Sb}} \times \frac{1}{10}$$

$$\text{Amount (mg) of emodin } (C_{15}H_{10}O_5) = \text{Amount (mg) of emodin RS} \times \frac{A_{Tc}}{A_{Sc}} \times \frac{1}{10}$$

$$\text{Amount (mg) of chrysophanol } (C_{15}H_{10}O_4) = \text{Amount (mg) of chrysophanol RS} \times \frac{A_{Td}}{A_{Sd}} \times \frac{1}{10}$$

$$\text{Amount (mg) of physcion } (C_{16}H_{12}O_5) = \text{Amount (mg) of physcion RS} \times \frac{A_{Te}}{A_{Se}} \times \frac{1}{20}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 254 nm)

Column: A stainless steel column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Column temperature: A constant temperature of about 30 °C

Mobile phase: A mixture of methanol, water and phosphoric acid (850 : 150 : 1)

Flow rate: 0.5 mL/min

System suitability

System performance: Proceed with 10 μL of the standard solution according to the above conditions; aloe emodin, rhein, emodin, chrysophanol and physcion are eluted in this order with the resolution being NLT 3.

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of each peak area of aloe emodin, rhein, emodin, chrysophanol and physcion is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Rhus Galls
오배자(五倍子)

Galla Rhois

Rhus Galls is the gall produced mainly by parasitic aphids of *Schlechtendalia chinensis* Bell (Pemphigidae), on the leaf of *Rhus javanica* Linné, *Rhus potaninii* Maximowicz or *Rhus punjabensis* Stew. var. *sinica* Rehder et Wilson (Anacardiaceae). According to its appearance, it is divided into Dubae and Gakbae.

Description (1) ***Dubae***—Dubae of Rhus Galls is the gall, oblong or spindle-globular, 25 mm to 90 mm in length and 15 mm to 40 mm in diameter. The external surface is slightly pubescent, exhibiting a grayish brown color. The texture is hard, fragile and easy to break. The fractured surface is lustrous and horny in shape, and the gall wall is 2 mm to 3mm in thickness. The inner wall is smooth and soft, containing blackish brown dead aphids and their gray powdery excreta.
It has a characteristic odor and an astringent taste.

(2) ***Gakbae***—Gakbae of Rhus Galls is the rhombic gall with irregular obtuse branches. Gakbae of Rhus Galls is distinctly pubescent, and the gall walls are relatively thin.

Identification (1) Weigh 0.5 g of pulverized Rhus Gallas, macerate with 10 mL of warm water, and filter. Add Iron(III) chloride TS to the filtrate; the resulting solution exhibits a navy blue color.

(2) Weigh 5 g of pulverized Rhus Gallas, add 5 mL of methanol, sonicate for 30 minutes to extract, filter, and use this solution as the test solution. Separately, weigh 1 mg of gallic acid RS in 1 mL of methanol and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of dichloromethane, ethyl formate and formic acid (7 : 5 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 254 nm); the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

Ash NMT 5.0%.

Packaging and storage Preserve in well-closed containers.

Rosa Fruit
금앵자(金櫻子)

Rosae Laevigatae Fructus

Rosa Fruit is the ripe fruit of *Rosa laevigata* Michaux (Rosaceae).

Description Rosa Fruit is the fruit, obovoid, 20 - 35 mm in length and 1 - 2 cm in diameter. The external surface occurs as a

yellowish red to reddish brown color, with scars of fallen thorns appeared as small, brown bumpy dots. A dish-shaped scar of the calyx is remained at the upper part, a yellow stalk base is remained in the middle part, and the lower part is gradually tapered. The texture is hard. The cut surface reveals the wall of the calyx, 1 - 2 mm in thickness, several small achenes that are hard inside, and light yellow tomenta attached to the inner membrane and the achene.
Rosa Fruit has a slight odor and a sweet and slightly astringent taste.

Purity (1) ***Foreign matter***—Rosa Fruit contains less than 2.0% of the fruit stalks and thorns.
(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(4) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 5.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 34.0%.

Packaging and storage Preserve in well-closed containers.

Round Amomum Fruit
백두구(白豆蔻)

Amomi Fructus Rotundus
Round Amomum Fruit is the ripe fruit of *Amomum kravanh* Pierre ex Gagnep. or *Amomum compactum* Solander ex Maton (Zingiberaceae).

Description (1) ***Amomum kravanh***—Round Amomum Fruit of Amomum kravanh is a fruit, nearly spherical, and 1 cm to 2 cm in diameter. The outer surface occurs as yellowish white to pale yellowish brown with three relatively deep longitudinal grooves, prominent stigmata at the apex, an indented scar of the fruit stalk in the lower part, and a uniform growth of pale brown cilia at both ends. The pericarp is thin and light and easily split longitudinally. The inside of the pericarp is divided into 3 loculi, each containing about 10 seeds. The seeds are irregularly polyhedral with wrinkles and remaining aril.
Round Amomum Fruit of Amomum kravanh is aromatic, has a slight camphor-like odor and tastes pungent and cool.
(2) ***Amomum compactum***—Round Amomum Fruit of Amomum compactum is smaller than Amomum kravanh. The outer surface occurs as yellowish white, sometimes purplish brown. The pericarp is relatively thin and the seeds are thin and desiccated.
Round Amomum Fruit of Amomum compactum has a weaker odor and taste compared to Amomum kravanh.

Identification Dissolve 20 μL of the essential oil of Round Amomum Fruit in 1 mL of ethanol and use this solution as the test solution. Separately, dissolve 10 μL of cineole RS in 1 mL of ethanol and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of cyclohexane, ethyl acetate and formic acid (16 : 2 : 0.5) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly vanillin-sulfuric acid TS to the plate and heat it at 105 °C; one of the spots obtained from the test solution shows the same color and R_f value as the spot from the standard solution.

Purity (1) ***Foreign matter***—The amount of fruit stalk and other foreign matter is contained in Round Amomum Fruit less than 2.0%.
(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 12.0% (seed).

Acid-insoluble ash NMT 5.0% (seed).

Essential oil content NLT 0.4 mL (50.0 g) (seed).

Packaging and storage Preserve in well-closed containers.

Rubus Fruit
복분자(覆盆子)

Rubi Fructus
Rubus Fruit is the unripe fruit of *Rubus coreanus* Miquel (Rosaceae).

Description Rubus Fruit is the fruit as an aggregate, consisting of numerous small drupes, mostly round and 7 mm to 9 mm in diameter. The outer surface occurs as pale green, grayish brown or reddish brown to reddish purple, surrounded by numerous drupelets, almost glabrous. The calyx divides into 5 parts nearby the flower stalk and occurs as green to greenish brown, with a fruit stalk scar at the lower end. The individual small drupelets are easily separated, crescent and has style remained. The lower part is relatively flat and has a diameter of about 2 to 3 mm.
Rubus Fruit occurs as almost odorless and tastes sour and sweet.

Identification Weigh 0.5 g of pulverized Rubus Fruit, add 10 mL of ethanol, heat the mixture for about 2 minutes, and filter. To 5 mL of the filtrate, add a small amount of magnesium powder and 2 to 3 drops of hydrochloric acid; the resulting solution exhibits a deep red color.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(2) **Residual pesticides**—Proceed with Rubus Fruit as directed in "Rubus Fruit" described in [Attachment 4] Tolerances for Pesticide Residues for Agricultural Products provided in the Public Notice "Standards and Specifications for Food" of the Ministry of Food and Drug Safety (MFDS).

(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 17.0%.

Ash NMT 8.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 20.0%.

Packaging and storage Preserve in well-closed containers.

Safflower
홍화(紅花)

Carthami Flos

Safflower is the tubulous flower of *Carthamus tinctorius* Linné (Compositae).

Safflower contains NLT 0.05% of kaempferol ($C_{15}H_{10}O_6$: 286.23), calculated on the dried basis.

Description Safflower is the tubulous flower without ovary directly attached, 1 cm to 2 cm in length. The external surface occurs as red to reddish brown. The corolla tube is elongated with 5 narrow lobes, which are in the shape of a narrow cord, 5 mm to 8 mm in length. It has 5 stamens, and yellowish white anthers gather to form a barrel shape. The stigma is long, cylindrical and slightly branches at the apex in the shape of a fork. The texture is flexible.
It has a characteristic odor and a slightly bitter taste.

Identification (1) Weigh 0.2 g of pulverized Safflower, add 10 mL of dilute ethanol, heat after connecting with a reflux condenser for 15 minutes, and then filter. Put 3 mL of the filtrate in a glass vessel about 30 mm in both internal diameter and height, and hang a piece of filter paper 2 cm in width and 30 cm in length, so that one end of the filter paper reaches the bottom of the vessel and allow the paper to soak up the liquid for 1 hour. Take out and immediately hang the paper in another glass vessel of the same type, containing 3 mL of water, and allow the paper to soak up the water for 1 hour; most of the upper part of the paper is tinted with pale yellow and the lower part with pale red.

(2) Weigh 0.5 g each of pulverized Safflower and Safflower RMPM, add 5 mL of diluted acetone (8 in 10), shake for 15 minutes to mix, and filter. Use the filtrates as the test solution and the Safflower RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the Safflower RMPM standard solution on a thin-layer chromatographic plate made of silica gel (with fluorescence indicator) for thin layer chromatography. Develop the plate with a mixture of ethyl acetate, water, formic acid and methanol (35 : 15 : 10 :2) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine this plate; the several spots obtained from the test solution show the same color and R_f value as the spots from the Safflower RMPM standard solution.

Purity (1) ***Foreign matter***—The amount of ovaries, stems, leaves and other foreign matter contained in Safflower is less than 2.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

(vi) Quintozen (sum of quintozene, pentachloroaniline and methyl pentachlorophenylsulfide): NMT 0.1 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

(5) ***Mycotoxins***—Total aflatoxin (sum of aflatoxins B_1, B_2, G_1 and G_2): NMT 15.0 ppb (aflatoxin B_1 is NMT 10.0 ppb).

Ash NMT 18.0%.

Assay Weigh accurately about 1 g of pulverized Safflower, add 50 mL of a mixture of ethanol, water and hydrochloric acid (25 : 10 : 4), heat after connecting with a reflux condenser for 135 minutes, filter, and then add ethanol to make exactly 50 mL. Use this solution as the test solution. Separately, weigh accurately about 10 mg of kaempferol RS (previously dried in a silica gel desiccator for NLT 12 hours), and dissolve in methanol to make exactly 100 mL. Pipet 10 mL of this solution exactly, add the mobile phase to make exactly 50 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under Liquid chromatography according to the following operating conditions and determine the peak areas, A_T and A_S, in the test solution and the standard solution.

$$\text{Amount (mg) of kaempferol } (C_{15}H_{10}O_6) = \text{Amount (mg) of kaempferol RS} \times \frac{A_T}{A_S} \times \frac{1}{10}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 365 nm)

Column: A stainless steel column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Column temperature: A constant temperature about 30 ℃.

Mobile phase: A mixture of methanol and 0.4% phosphoric acid (13 : 12)

Flow rate: 1.0 mL/min

System suitability

System repeatability: Repeat the test 6 times with 10 μL of the standard solution according to the above operating conditions; the relative standard deviation of the peak area of kaempferol is NMT 1.5%.

Packaging and storage Preserve in light-resistant, well-closed containers.

Saffron
사프란

Crocus

Saffron is the stigma of *Crocus sativus* Linné (Iridaceae).

Description Saffron is the stigma, thin cord-like in shape, 20 mm to 35 mm long, tripartite or separate. It occurs as dark yellowish red to reddish brown overall, and the upper part is relatively broad and slightly flat. The apex is tooth-shaped with an uneven margin and has a short lacunae on the inside. A piece of small and yellow style sometimes remains on the lower part. The body is light and the texture is soft and lackluster. When dried, the texture is brittle and can be easily cut.
It has a strong and characteristic odor, bitter taste and colors saliva yellow.

Identification (1) Add 1 drop of sulfuric acid to Saffron; the color changes to a dark blue, gradually turning purple to reddish brown.

(2) **Crocin**—Dry Saffron in a desiccator (silica gel) for 24 hours and powder it. Weigh 0.1 g of pulverized Saffron, add 150 mL of warm water, heat the mixture between 60 ℃ and 70 ℃ for 30 minutes with frequent shaking, and filter after cooling. To 1 mL of the filtrate, add water to make 10 mL. The color of the solution shall not be more intense than the following control solution.

Control solution—Weigh 5 mg of potassium dichromate, dissolve in water to make exactly 10 mL.

Purity (1) ***Foreign matter***—(i) Glycerin, sugar or honey: Saffron has no sweet taste and leaves no stains when pressed between pieces of paper.

(ii) Yellow portion of the style: Saffron contains less than 10.0% of the yellow portion of the style.

(2) ***Aniline pigment***—Add 10 mL of chloroform to 50 mg of Saffron and shake to mix; the resulting solution is colorless or very pale yellow even if it is yellow.

(3) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(4) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

Loss on drying NMT 12.0% (6 hours).

Ash NMT 7.5%.

Packaging and storage Preserve in light-resistant, well-closed containers.

Salvia Miltiorrhiza Root
단삼(丹參)

Salviae Miltiorrhizae Radix

Salviae Miltiorrhizae Root is the root of *Salvia miltiorrhiza* Bunge (Labiatae).

Salvia Miltiorrhiza Root contains NLT 4.1% of salvianolic acid B ($C_{36}H_{30}O_{16}$: 718.62), calculated on the dried basis.

Description Salvia Miltiorrhiza Root is the root, long cylindrical in shape, 10 to 20 cm in length and 3 to 15 mm in diameter. The root consists of 1 to 2 or more branches that are slightly bent, some with thin, hair-like rootlets. The outer surface is coarse, reddish brown or dark reddish brown, wrinkled longitudinally. The bark of the old roots is soft and tender. The texture is firm and fragile. The cut surface is soft with clefts or slightly uniform and dense, with reddish-brown cortex and grayish-yellow or purplish-brown xylem, which has bundles of yellowish white vessels arranged radially.
Under the microscope, the cross-section shows a cork layer consisting of 4 to 6 rows of cork cells. The cortex is broad and the phloem is semicircular. The cambium forms rings and the interfascicular cambium is not very distinct. The xylem consists of 8 to 10 bundles arranged radially. The vessels are concentrated near the cambium and merge into a single row toward the center. The xylem fibers are distributed in bundles near the primary xylem in the center.
It has a light, characteristic odor and slightly bitter and astringent taste.

Identification (1) Weigh 1 g of pulverized Salvia Miltiorrhiza Root, add 10 mL of ethanol, boil the mixture shortly and filter it; the filtrate shows a brownish yellow color. Add 1 mL of dilute sulfuric acid and 0.5 g of zinc powder; the filtrate turns to yellow after short stand.

(2) Weigh 2 g of pulverized Salvia Miltiorrhiza Root, add 10 mL of methanol, sonicate for 1 hour, filter it and use the filtrate as the test solution. Separately, dissolve 1 mg of tansinone IIA RS in 1 mL of methanol and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography (with fluorescent agent). Next, develop the plate with a mixture of hexane and ethyl acetate (4 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 254 nm) or spray evenly sulfuric acid TS for spraying to the plate and heat; one spot among the several spots obtained from the test solution and a spot from the standard solution show the same color and the same R_f value.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 12.0%.

Ash NMT 10.0%.

Extract content *Dilute ethanol-soluble extract*—NLT 25.0%.

Assay Weigh accurately about 0.3 g of pulverized Salvia Miltiorrhiza Root, add 5 mL of diluted methanol (75 in 100), sonicate for 30 minutes, filter and use the filtrate as the test solution.

Separately, weigh accurately about 1 mg of salvianolic acid B RS (previously dried in a silica gel desiccator for 24 hours), add diluted methanol (75 in 100) to make exactly 50 mL and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under Liquid chromatography according to the following conditions and determine the peak areas, A_T and A_S, of the test solution and the standard solution.

Amount (mg) of salvianolic acid B ($C_{36}H_{30}O_{16}$)
= Amount (mg) of salvianolic acid B RS$\times \frac{A_T}{A_S} \times 10$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 280 nm)

Column: A stainless column, 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (particle diameter: 5 μm to 10 μm).

Column temperature: An ordinary temperature.

Mobile phase: Control the step or concentration gradient by mixing the mobile phases A and B as directed below.

Mobile phase A: Diluted acetic acid (1 in 100)

Mobile phase B: A mixture of methanol, acetonitrile and acetic acid (100 : 75 : 1)

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)
0	75	25
25	75	25
40	60	40
65	35	65
89	11	89
100	75	25

Flow rate: 1.0 mL/min

System suitability

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak area of salvianolic acid B is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Saposhnikovia Root
방풍(防風)

Saposhnikoviae Radix

Saposhnikovia Root is the root of *Saposhnikovia divaricata* Schischkin (Umbelliferae).

Description Saposhnikovia Root is a root that is thin, long conical, and thinning downward, 15 cm to 20 in length and 7 mm to 15 mm in diameter. The outer surface occurs as pale brown. There are several longitudinal wrinkles and root scars. The body part is light, and its texture soft and easy to cut. The cut surface shows a pale brown cortex and many lacunae, and the xylem occurs as pale yellow.

Under the microscope, the cross-section shows a cork layer consisting of several rows of cork cells, and the phelloderm is narrow. The cortex shows an irregular and relatively large elliptical lactiferous tube. The phloem is relatively broad with several lactiferous tubes that are almost circular, with 4 to 8 secretory cells nearby, and the lactiferous tubes are filled with a golden yellow oil-like substance. The medullary rays are curved and usually separated from the phloem tissue to form a lacuna. The cambium is annular and distinct. The xylem has very many vessels, radiating individually or in groups of 2 to 3. The medullary rays of the xylem consist of 1 to 2 rows of cells with several lacunae and radiating. In the middle of the crown is the pith.

It has a characteristic odor and a slightly sweet taste.

Identification Weigh 1 g each of pulverized Saposhnikovia Root and Saposhnikovia root RMPM, add 10 mL of methanol, and sonicate for 60 minutes. Filter the solutions and use the filtrates as the test solution and the Saposhnikovia root RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Saposhnikovia root RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography (with fluorescent indicator). Next, develop the plate using a mixture of dichloromethane, methanol and water (45 : 10 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 254 nm); the several spots obtained from the test solution show the same color and R_f value as the spots from the Saposhnikovia root RMPM standard solution.

Purity (1) ***Foreign matter***—Saposhnikovia Root contains less than 2.0% of the stem and other foreign matter.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(vi) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): NMT 0.2 ppm.
(vii) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 7.0%.

Acid-insoluble ash NMT 1.5%.

Extract content ***Dilute ethanol-soluble extract***—NLT 20.0%.

Packaging and storage Preserve in well-closed containers.

Sappan Wood
소목 (蘇木)

Sappan Lignum

Sappan Wood is the heartwood of *Caesalpinia sappan* Linné (Leguminosae).

Description Sappan Wood is the heartwood pieces, long cylindrical, semi-cylindrical or stick-like in shape. The external surface occurs as an amber red to grayish brown color, sometimes

with traces of sapwood in pale brown to grayish brown. It is often cut transversely or longitudinally. The texture is hard, but those cut longitudinally are easy to be broken. The transversely cut surface reveals distinct annual rings.
Under a microscope, the transverse section of Sappan Wood reveals the medullary rays consisting of 1 to 2 rows of cells. The vessels are about 160 μm in diameter and contain a yellowish brown or reddish brown substance. The xylem fiber is polygonal and very thick-walled. The xylem parenchyma cells are very thick-walled and lignified, sometimes containing prismatic crystals of calcium oxalate. The parenchyma cells of the pith are irregularly polygonal, varying in size, and the walls are slightly lignified with pitting.
It occurs as nearly odorless and has a slightly astringent taste.

Identification (1) Weigh 0.5 g of pulverized Sappan Wood, add 10 mL of dilute ethanol, shake to mix, and filter. To 5 mL of the filtrate, add 2 to 3 drops of sodium hydroxide TS; the resulting solution exhibits a deep red color.

(2) Weigh 1 g of pulverized Sappan Wood, add 10 mL of ethanol, sonicate for 30 minutes to extract, filter, and use the filtrate as the test solution. Separately, weigh 1 mg of brazilin RS, dissolve in 1 mL of methanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of dichloromethane, acetone and formic acid (15 : 5 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 254 nm); the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

Purity (1) ***Foreign matter***—(i) Sapwood: The amount of sapwood other than the heartwood contained in Sappan Wood is less than 3.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

(5) Put a small piece of Sappan Wood in calcium hydroxide TS; the resulting solution does not exhibit a purplish blue color.

Loss on drying NMT 13.0% (6 hours).

Ash NMT 2.5%.

Extract content ***Dilute ethanol-soluble extract***—NLT 5.0%.

Packaging and storage Preserve in well-closed containers.

Schisandra Fruit
오미자(五味子)

Schisandrae Fructus

Schisandra Fruit is the ripe fruit of *Schisandra chinensis* Baillon (Schisandraceae).

Schisandra Fruit contains NLT 0.7% in total of schisandrin ($C_{24}H_{32}O_7$: 432.51), gomisin A ($C_{23}H_{28}O_7$: 416.46) and gomisin N $C_{23}H_{28}O_6$: 400.47), calculated on the dried basis.

Description Schisandra Fruit is the fruit, irregularly spherical to flattened spherical and 5 mm to 8 mm in diameter. The external surface occurs as a dark red to blackish brown color, with wrinkles and occasionally with white powder. The flesh is pliant, and when peeled off, it contains 1 to 2 kidney-shaped seeds. The seed is 2 mm to 5mm in length, and its external surface occurs as a lustrous yellowish brown to dark reddish brown color with a distinct raphe on the dorsal side.
It has a slight odor and an acidic taste at first, followed by an astringent and bitter taste.

Identification Weigh 1.0 g each of pulverized Schisandra Fruit and Schisandra fruit RMPM, add 20 mL each of dichloromethane, heat after connecting with a reflux condenser on a water bath for 30 minutes, filter, and evaporate the filtrates to dryness. Add 1 mL of methanol to each residue to prepare the test solution and the Schisandra fruit RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 2 μL each of the test solution and the Schisandra fruit RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with the upper layer of a mixture of petroleum ether, ethyl formate and formic acid (15 : 5 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS and heat at 105 °C; the color and the R_f values of the several spots obtained from the test solution are the same as those obtained from the Schisandra fruit RMPM standard solution. Of these, the spots of schisandrin, gomisin A and gomisin N appear at the R_f values of about 0.2, 0.25 and 0.45, respectively.

Purity (1) ***Foreign matter***—The amount of fruit stalks and other foreign matters contained in Schisandra Fruit is less than 1.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—Proceed with Schisandra Fruit as directed in 'Schisandra Fruit (Dried)' described in [Attachment 4] Tolerances for Pesticide Residues for Agricultural Products provided in the Public Notice "Standards and Specifications for Food" of the Ministry of Food and Drug Safety (MFDS).

(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 16.0%.

Ash NMT 5.0%.

Assay Weigh accurately about 0.5 g of pulverized Schisandra Fruit, add 20 mL of methanol, sonicate for 20 minutes to extract, and filter. To the residue, add 20 mL of methanol and proceed in the same manner. Combine all the filtrates, add methanol to make exactly 50 mL, and use this solution as the test solution.

Separately, weigh accurately 10 mg of schisandrin RS, 10 mg of gomisin A RS and 10 mg of gomisin N RS and add methanol to each to make exactly 25 mL. Pipet 2 mL each of these solutions, add methanol to make exactly 20 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions, and determine the peak areas, A_{Ta}, A_{Tb} and A_{Tc} from the test solution and the peak areas, A_{Sa}, A_{Sb} and A_{Sc} from the standard solution.

$$\text{Amount (mg) of schisandrin } (C_{24}H_{32}O_7) = \text{Amount (mg) of schisandrin RS} \times \frac{A_{Ta}}{A_{Sa}} \times \frac{1}{5}$$

$$\text{Amount (mg) of gomisin A } (C_{23}H_{28}O_7) = \text{Amount (mg) of gomisin A RS} \times \frac{A_{Tb}}{A_{Sb}} \times \frac{1}{5}$$

$$\text{Amount (mg) of gomisin N } (C_{23}H_{28}O_6) = \text{Amount (mg) of gomisin N RS} \times \frac{A_{Tc}}{A_{Sc}} \times \frac{1}{5}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 254 nm)

Column: A stainless steel column about 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Mobile phase: A mixture of acetonitrile, water and formic acid (70 : 30 : 0.1)

Flow rate: 0.6 mL/min

System suitability

System performance: Proceed with 10 μL of the standard solution according to the above conditions. Adjust the flow rate so that schisandrin, gomisin A and gomisin N are eluted in this order with clear separation of each peak and the resolution between each peaks is NLT 1.6.

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviations of each peak area of schisandrin, gomisin A and gomisin N are NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Schizonepeta Spike
형개(荊芥)

Schizonepetae Spica

Schizonepeta Spike is the spike of *Schizonepeta tenuifolia* Briquet (Labiatae).

Description Schizonepeta Spike is a thin, long barley ear-like shaped spike, which occurs as purple greenish brown to greenish brown, 5 cm to 10 cm in length. It has calyx tubes with small labiate flower or often fruits. Also, short milky white hairs are observed at the wholepoles.
It has a characteristic odor and slightly cool feeling on keeping in the mouth.

Identification (1) Weigh 2 g of pulverized Schizonepeta Spike, add 20 mL of water, shake well to mix, and distill. To 3 mL of the distillate, add 2 or 3 drops of 2,4-dinitrophenylhydrazine-ethanol TS; the solution exhibits an orange red color.

(2) Weigh 0.8 g of pulverized Schizonepeta Spike and Schizonepeta Spike RMPM, add 20 mL of petroleum ether and seal. Allow to stand at room temperature for 10 hours with occasional shaking. Filter and evaporate the filtrate to dryness. Dissolve the residue in 1 mL of petroleum ether and use this solution as the test solution and the standard solution of Schizonepeta Spike RMPM. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard schizonepeta spike solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of hexane and ethyl acetate (17 : 3) to a distance of about 10 cm and dry the plate in shades. Spray evenly sulfuric acid TS for spraying on the plate and examine under ultraviolet light (main wavelength: 365 nm); the color and R_f values of the several spots obtained from the test solution and those from the standard solution of Schizonepeta Spike RMPM are the same, and one of the several spots exhibits green fluorescence at the R_f value of about 0.3.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 11.0%.

Acid-insoluble ash NMT 3.0%.

Extract Content ***Dilute ethanol-soluble extract—***NLT 8.0%

Packaging and storage Preserve in well-closed containers.

Scopolia Rhizome
스코폴리아근

Scopoliae Rhizoma

Scopolia Rhizome is the rhizome of *Scopolia japonica* Maximowicz or *Scopolia carniolica* Jacquin (Solanaceae).

Scopolia Rhizome contains NLT 0.3% of total alkaloids [as hyoscyamine ($C_{17}H_{23}NO_3$: 289.37) and scopolamine ($C_{17}H_{21}NO_4$: 303.35)], calculated on the dried basis.

Description Scopolia Rhizome is the rhizome, mainly irregularly branched, slightly curved, about 5 cm to 15 cm long and 1 cm to 3 cm in diameter. Constrictions make the rhizome appear nodular. Rarely, the stem base is present at one end. Stem stigmas are on the upper side of each node, and root or radicle stigmas are on the underside of the rhizome. The outer surface occurs as grayish brown to blackish brown, with wrinkles. The fractured surface occurs as grayish white to pale brown, granular and compact, with lighter colored cortex.
Under the microscope, the cross-section shows the xylem with groups of vessels arranged in a step-like manner and

accompanied by xylem sieve tubes in medullary rays. The parenchyma cells contain starch granules and sometimes sand crystals of calcium oxalate.
It has a characteristic odor and a slightly sweet, later slightly bitter taste.

Identification (1) Weigh 1 g of pulverized Scopolia Rhizome, add 10 mL of ether and 0.5 mL of ammonia TS, shake for 30 minutes and filter. Wash the residue with 10 mL of ether, transfer the filtrate and the washing to a separatory funnel, add 20 mL of diluted sulfuric acid (1 in 50), shake well and collect the acid extract into another separatory funnel. Render the solution slightly alkaline with ammonia TS, add 10 mL of ether, shake well, and collect the ether layer. Transfer the ether layer to a porcelain dish and evaporate the ether on a water bath. To the residue, add 5 drops of fuming nitric acid and evaporate the mixture on a water bath to dryness. After cooling, dissolve the residue in 1 mL of dimethylformamide and add 5 to 6 drops of tetraethylammonium hydroxide TS; the solution shows a reddish purple to purple color.

(2) Weigh 2 g of pulverized Scopolia Rhizome, transfer it to a stoppered centrifuge tube, add 30 mL of ammonia TS, sonicate for 5 minutes, filter and centrifuge the filtrate. Transfer the supernatant to a separatory funnel, add 40 mL of ethyl acetate, shake, and collect the ethyl acetate layer. Then, add 3 g of anhydrous sodium sulfate, shake and filter after the ethyl acetate solution becomes clear. Evaporate ethyl acetate in vacuum, dissolve the residue in 1 mL of ethanol, and use this solution as the test solution. Separately, weigh 2 mg of atropine sulfate RS and 1 mg of scopolamine hydrobromide RS, dissolve each in 1 mL of ethanol and use these solutions as the standard solutions (1) and (2). With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the standard solutions on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of acetone, water and ammonia solution (28) (90 : 7 : 3) to a distance of about 10 cm and dry the plate at 80 ℃ for 10 minutes. After cooling, spray Dragendorff's TS for spraying evenly to the plate; two principal spots obtained from the test solution and each yellowish red spot obtained from the standard solutions (1) and (2) show the same color and the same R_f value.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

Ash NMT 7.0%.

Assay Weigh accurately about 0.7 g of pulverized Scopolia Rhizome, previously dried at 60 ℃ for 8 hours, transfer it in a stoppered centrifuge tube and moisten with 15 mL of ammonia TS. Add 25 mL of ether, cap the centrifuge tube tightly, shake for 15 minutes, centrifuge, and collect the ether layer. Repeat this procedure twice with the residue using 25 mL of ether each time. Combine all the extracts and evaporate the ether on a water bath. Dissolve the residue in 5 mL of the mobile phase, add 3.0 mL of the internal standard solution, and add the mobile phase again to make exactly 25 mL. Filter the resulting solution through a filter having a porosity of NMT 0.8 μm, discard the first 2 mL of the filtrate, and use the subsequent filtrate as the test solution. Separately, weigh accurately about 25 mg of atropine sulfate RS (determined the loss on drying before use), dissolve it in the mobile phase to make exactly 25 mL, and use this solution as the standard stock solution(1). Weigh accurately about 25 mg of scopolamine hydrobromide RS (determined the loss on drying before use), dissolve it in the mobile phase to make exactly 25 mL, and use this solution as the standard stock solution(2). Pipet 5 mL of the standard stock solution (1) and 1 mL of the standard stock solution (2), add 3 mL of the internal standard solution, then add the mobile phase to make exactly 25 mL, and use this solution as the standard solution Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions. Determine the ratios Q_{TS} and Q_{SA} of the peak area of hyoscyamine (atropine) to that of the internal standard, and the ratios Q_{TS} and Q_{SS} of the peak area of scopolamine to that of the internal standard for the test solution and the standard solution, respectively. Calculate the amount of hyoscyamine and scopolamine using the following equations and designate the total as the amount of total alkaloids.

$$\text{Amount (mg) of hyoscyamine } (C_{17}H_{23}NO_3) = C \times \frac{Q_{TA}}{Q_{SA}} \times \frac{1}{5} \times 0.8551$$

$$\text{Amount (mg) of scopolamine } (C_{17}H_{21}NO_4) = C_2 \times \frac{Q_{TS}}{Q_{SS}} \times \frac{1}{25} \times 0.7894$$

C_1: Amount (mg) of atropine sulfate RS, calculated on the dried basis
C_2: Amount (mg) of scopolamine hydrobromide RS, calculated on the dried basis

Internal standard solution—A solution of brucine in the mobile phase (1 in 2500).

Operating conditions
Detector: An ultraviolet absorption photometer (wavelength: 210 nm)
Column: A stainless steel tube about 4 mm in internal diameter and 15 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (particle diameter: 5 μm).
Mobile phase: Dissolve 6.8 g of potassium dihydrogen phosphate in 900 mL of water, add 10 mL of triethylamine, and adjust the pH with phosphoric acid to 3.5. Add water to make 1000 mL and mix this solution with acetonitrile (9 : 1).
Flow rate: Adjust the flow rate so that the retention time of scopolamine is about 8 minutes.
Selection of column: Proceed with 10 μL of the standard solution under the above conditions. Use a column that elutes scopolamine, atropine, and the internal standard in this order and clearly separates the individual peaks.

Packaging and storage Preserve in well-closed containers.

Scopolia Extract
스코폴리아엑스

Scopolia Extract contains NLT 0.90% and NMT 1.09% of total alkaloid [as hyoscyamine ($C_{17}H_{23}NO_3$: 289.37) and scopolamine ($C_{17}H_{21}NO_4$: 303.35)].

Method of preparation Prepare a semiliquid extract of the coarse powder of scopolia rhizome with 35 vol% ethanol, tap water, or purified water as menstruum, as directed under the Extracts.

Description Scopolia Extract occurs as brown to dark brown in color and has a characteristic odor and bitter taste.
It is soluble in water with a slight cloudiness.

Identification (1) Dissolve 4 g of Scopolia Extract in 10 mL of water, add 8 mL of ammonia TS and 80 mL of ether, stopper tightly, and shake for 1 hour to mix. Add 2.5 g of Tragacanth gum powder and shake the mixture vigorously to mix. Allow the mixture to stand for 5 minutes until the layers are clearly separated and collect the ether layer. Evaporate the ether layer on a water bath, add 5 drops of fuming nitric acid, and evaporate on a water bath to dryness. After cooling, dissolve the residue in 1 mL of *N*,*N*-Dimethylformamide and add 5 to 6 drops of tetraethylammonium hydroxide TS; the solution shows a reddish purple to purple color.

(2) To 0.5 g of Scopolia Extract, add 30 mL of ammonia TS, shake to mix and transfer the mixture to a separatory funnel. Add 40 mL of ethyl acetate and shake to mix. Collect the ethyl acetate layer separately, add 3 g of anhydrous sodium sulfate, shake to mix and filter after the solution becomes clear. Collect the filtrate, evaporate the ethyl acetate in vacuum, dissolve the residue in 1 mL of ethanol and use this solution as the test solution. Proceed as directed under the Identification (2) of Scopolia Rhizome.

Purity (1) ***Heavy metals***—(i) Total heavy metals: NMT 30 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

Assay Weigh accurately about 0.4 g of Scopolia Extract, place it in a stoppered centrifuge tube, add 15 mL of ammonia TS and shake to mix. To this solution, add 25 mL of ether, stopper tightly, shake to mix for 15 minutes, centrifuge and collect the ether layer separately. Repeat this procedure twice with the water layer, using 25 mL of ether each time. Combine the extracts and evaporate the ether on a water bath. Dissolve the residue in 5 mL of the mobile phase, add 3 mL of the internal standard solution, add the mobile phase again to make exactly 25 mL. Proceed as directed under the Assay of Scopolia Rhizome.

$$\text{Amount (mg) of hyoscyamine } (C_{17}H_{23}NO_3) = C \times \frac{Q_{TA}}{Q_{SA}} \times \frac{1}{5} \times 0.8551$$

$$\text{Amount (mg) of scopolamine } (C_{17}H_{21}NO_4) = C_2 \times \frac{Q_{TS}}{Q_{SS}} \times \frac{1}{25} \times 0.7894$$

C_1 : Amount (mg) of atropine sulfate RS, calculated on the dried basis

C_2 : Amount (mg) of scopolamine hydrobromide RS, calculated on the dried basis

Internal standard solution—A solution of brucine in the mobile phase (1 in 2,500).

Packaging and storage Preserve in light-resistant, tight containers in a cold place.

10% Scopolia Extract Powder
스코폴리아엑스 10 배산

10% Scopolia Extract Powder contains NLT 0.09% and NMT 0.11% of total alkaloid [as hyoscyamine ($C_{17}H_{23}NO_3$: 289.37) and scopolamine ($C_{17}H_{21}NO_4$: 303.35)].

Method of preparation

Scopolia Extract	100 g
Starch, lactose, or their mixture	a sufficient quantity
To make	1,000 g

Add 100 mL of purified water to Scopolia Extract and heat the mixture while stirring and softening. After cooling, add 800 g of starch, lactose or their mixture gradually in small amounts and shake to mix well. Dry the mixture preferably at low temperature and dilute it with sufficient amount of starch, lactose or their mixture to make 1,000 g of homogeneous powder.

Description 10% Scopolia Extract Powder occurs as a brownish yellow to grayish-yellowish brown powder and has a slight characteristic odor and a slightly bitter taste.

Identification (1) Add 15 mL of water and 8 mL of ammonia TS to 20 g of 10% Scopolia Extract Powder, and mix well to make the mixture homogeneous. Add 100 mL of ether and 7 g of sodium chloride, stopper tightly, shake for 1 hour, add 5 g of Tragacanth gum powder and shake vigorously. Allow the mixture to stand for 5 minutes until it becomes clearly separated and filter. Proceed with the filtrate as directed under the Identification (1) of Scopolia Extract.

(2) Add 5 g of 10% Scopolia Extract Powder to a stoppered centrifuge tube, add 30 mL of ammonia TS, sonicate for 5 minutes, filter and centrifuge the filtrate. Transfer the supernatant to a separatory funnel, add 40 mL of ethyl acetate, shake to mix, and collect the ethyl acetate layer separately. Add 3 g of anhydrous sodium sulfate, shake to mix and filter after the ethyl acetate layer becomes clear. Evaporate the ethyl acetate in vacuum, dissolve the residue in 1 mL of ethanol and use this solution as the test solution. Proceed as directed under the Identification (2) of Scopolia Rhizome.

Purity (1) ***Heavy metals***—(i) Total heavy metals: NMT 30 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

Assay Weigh accurately about 4.0 g of 10% Scopolia Extract Powder, place it in a glass-stoppered centrifuge tube, add 10 mL of ammonia TS and shake. Add 25 mL of ether, stopper tightly, shake for 15 minutes and collect the ether layer. Repeat this procedure three times with the water layer, using 25 mL of ether each time. Combine the extracts and evaporate the ether on a water bath. Dissolve the residue in 5 mL of the mobile phase, add

exactly 3 mL of the internal standard solution, and add the mobile phase again to make exactly 25 mL. Proceed as directed under the Assay of Scopolia Rhizome.

$$\text{Amount (mg) of hyoscyamine } (C_{17}H_{23}NO_3) = C \times \frac{Q_{TA}}{Q_{SA}} \times \frac{1}{5} \times 0.8551$$

$$\text{Amount (mg) of scopolamine } (C_{17}H_{21}NO_4) = C_2 \times \frac{Q_{TS}}{Q_{SS}} \times \frac{1}{25} \times 0.7894$$

C_1: Amount (mg) of atropine sulfate RS, calculated on the dried basis

C_2: Amount (mg) of scopolamine hydrobromide RS, calculated on the dried basis

Packaging and storage Preserve in tight containers.

Scrophularia Root
현삼(玄參)

Scrophulariae Radix

Scrophularia Root is the root of *Scrophularia buergeriana* Miquel or *Scrophularia ningpoensis* Hemsley (Scrophulariaceae).

Description Scrophularia Root is irregularly curved, long cylindrical or spindle-shaped root, 4 cm to 20 cm in length and 1 cm to 3 cm in diameter. External surface occurs as yellowish brown to brown, with rough longitudinal wrinkles, transverse long lenticels and sparse rootlet scars. Texture is compact and hard to be fractured, and the fractured surface occurs as black to blackish brown.
It has a characteristic odor like burnt sugar and tastes slightly sweet, later slightly bitter.

Identification (1) Weigh 0.5 g of pulverized Scrophularia Root, add 10 mL of water, heat on a water bath for 3 minutes, and filter. To 4 mL of the filtrate, add 2 mL of Fehling's TS and heat on a water bath; a red precipitate is produced.

(2) Weigh 0.1 g of pulverized Scrophularia Root, add 10 mL of methanol, warm for 2 to 3 minutes on a water bath, and filter. Evaporate the filtrate to dryness, add 4 mL of acetic anhydride to the residue, warm for 2 minutes, and filter. After cooling the filtrate, add carefully 1 mL of sulfuric acid; the zone of contact exhibits a reddish brown color.

(3) Weigh about 1 g of pulverized Scrophularia Root, add 10 mL of diluted ethanol (7 in 10), sonicate for 60 minutes, filter, and use the filtrate as the test solution. Separately, dissolve 1 mg of E-harpagoside RS in 1 mL of diluted ethanol (7 in 10) and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 2 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of butanol, water and acetic acid (7 : 2 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly the plate with vanillin-sulfuric acid TS and heat at 105 ℃ for 10 minutes; one of the several spots from the test solution and the pink spot from the standard solution have the same color and the R_f value.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 17.0% (6 hours).

Ash NMT 6.0%.

Acid-insoluble ash NMT 2.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 24.0%

Packaging and storage Preserve in well-closed containers.

Scutellaria Root
황금(黃芩)

Scutellariae Radix

Scutellaria Root is the root or the root from which the periderm has been removed of *Scutellaria baicalensis* Georgi (Labiatae).

Scutellaria Root contains NLT 10.0% of the sum of baicalin ($C_{21}H_{18}O_{11}$: 446.37), baicalein ($C_{15}H_{10}O_5$: 270.24), and wogonin ($C_{16}H_{12}O_5$: 284.28), calculated on the dried basis.

Description Scutellaria Root is a root, which is conical, twisted and curved, 8 cm to 25 cm in length and 1 cm to 3 cm in diameter. The external surface occurs as yellowish brown or deep yellow, sparsely scattered with humpy rootlet scars. The upper part has relatively coarse, twisted, curved longitudinal wrinkles or an irregular reticular patterns. The texture is hard but brittle, and easy to cut. The cut surface occurs as yellow, and reddish brown at the center. Older roots are decayed or hollow in the middle, and show dark brown or reddish brown.
It occurs as nearly odorless and has a slightly bitter taste.

Identification (1) Weigh 0.5 g of pulverized Scutellaria Root, add 20 mL of ether, heat after connecting with a reflux condenser for 5 minutes, cool, and then filter. Dissolve the residue obtained by evaporating the filtrate in 10 mL of ethanol. To 3 mL of the resulting solution, add 1 to 2 drops of dilute iron(III) chloride TS; the solution exhibits grayish green and later turns purplish brown.

(2) Weigh 1 g each of pulverized Scutellaria Root and Scutellaria root RMPM, respectively, add 30 mL of a mixture of ethyl acetate and methanol (3 : 1), warm on a water bath for 30 minutes after connecting with a reflux condenser, and filter. Dissolve the residues in 5 mL of methanol and use these solutions as the test solution and the Scutellaria root RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 2 μL each of the test solution and the Scutellaria root RMPM standard solution on a thin layer plate made of silica gel (with fluorescence indicator) for thin layer chromatography. Next, develop the plate with a mixture of ethyl acetate, methanol and water (100 : 17 : 13) as the developing solvent to a distance of about 10 cm, and air-dry the

plate. Spray evenly dilute sulfuric acid TS and heat at 105 °C; the several spots obtained from the test solution show the same color and R_f value as the spots from the Scutellaria root RMPM standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 15.0%.

Ash NMT 6.0%.

Acid-insoluble ash NMT 1.0%.

Assay Weigh accurately about 0.5 g of pulverized Scutellaria Root, add 40 mL of diluted ethanol (7 in 10), heat after connecting with a reflux condenser for 1 hour, and filter. To the residue, add 40 mL of diluted ethanol (7 in 10) and proceed in the same manner. Combine the filtrates, add diluted ethanol (7 in 10) to make exactly 100 mL, and use this solution as test solution 1. To 2 mL of this solution, add diluted ethanol (7 in 10) to make exactly 20 mL and use this solution as test solution 2. Separately, weigh accurately about 10 mg of baicalin RS (previously dried in a silica gel desiccator for NLT 24 hours), about 10 mg of baicalein RS (previously dried in a silica gel desiccator for NLT 24 hours) and about 10 mg of wogonin RS (previously dried in a silica gel desiccator for NLT 24 hours), and dissolve each in methanol to make exactly 20 mL. Pipet 2 mL each of these solutions, add diluted ethanol (7 in 10) to make exactly 20 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution 1, the test solution 2, and the standard solution as directed under Liquid chromatography according to the following operating conditions, and determine the peak areas of baicalein and wogonin, A_{Tb} and A_{Tc}, in the test solution 1, the peak area of baicalin, A_{Ta}, in the test solution 2, and the peak areas of baicalin, baicalein and wogonin, A_{Sa}, A_{Sb} and A_{Sc}, in the standard solution.

$$\text{Amount (mg) of baicalin } (C_{21}H_{18}O_{11}) = \text{Amount (mg) of baicalin RS} \times \frac{A_{Ta}}{A_{Sa}} \times 5$$

$$\text{Amount (mg) of baicalein } (C_{15}H_{10}O_5) = \text{Amount (mg) of baicalein RS} \times \frac{A_{Tb}}{A_{Sb}} \times \frac{1}{2}$$

$$\text{Amount (mg) of wogonin } (C_{16}H_{12}O_5) = \text{Amount (mg) of wogonin RS} \times \frac{A_{Tc}}{A_{Sc}} \times \frac{1}{2}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 277 nm)

Column: A stainless column, about 4 to 6 mm in inner diameter and 15 to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 to 10 μm in particle diameter).

Column temperature: 40 °C.

Mobile phase: Control the mixing ratio of mobile phase A and mobile phase B stepwise or via the concentration gradient as follows.

Mobile phase A: Diluted acetic acid (1 in 100).

Mobile phase B: A 1% acetic acid solution of a mixture of acetonitrile and methanol (7 : 3)

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)
0	75	25
10	68	32
20	55	45
24	55	45
35	52	48
40	75	25
45	75	25

Flow rate: 1.0 mL/min

System suitability

System performance: Dissolve 2 mg each of baicalin RS, baicalein Rs, wogonin RS and methyl p-hydroxybenzoate in methanol to make 100 mL. Proceed with 10 μL of this solution according to the above operating conditions. Adjust the concentration gradient conditions so that methyl p-hydroxybenzoate, baicalin, baicalein and wogonin are eluted in this order, with clearly dividing each peak.

System repeatability: Repeat the test 6 times with 10 μL each of the standard solutions according to the above operating conditions; the relative standard deviation of the peak areas of baicalin, baicalein and wogonin is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Senega
세네가

Senegae Radix

Senega is the root of *Polygala senega* Linné or *Polygala senega* Linné var. *latifolia* Torrey et Gray (Polygalaceae).

Description Senega is the root, slender, conical and slightly twisted. The main root is 3 cm to 10 cm in length and 5 mm to 15 mm in diameter. The external surface occurs as a pale grayish brown to grayish brown color, mostly with a longitudinal pattern and protruding lines. The crown is tuberously enlarged, with remains of stems and red buds. Branched rootlets are twisted and curved. The transverse section reveals the grayish brown cortex and the nearly white xylem, usually circular, occasionally dented cuneate to semi-circular. The cortex on the opposite side is thick. Under a microscope, the transverse section of Senega reveals the cork layer of the main root consisting of several layers of pale grayish brown cork cells followed by slightly transversely long parenchyma cells. The secondary cortex is composed of parenchyma cells and sieve tubes, transverse by 1 to 3 rows of the medullary rays, all containing an oil droplet-like substance. The sieve tubes are gathered only on the outside of the normally developed xylem. The xylem is usually circular, occasionally cuneate to semi-circular, with the cortex on the opposite side forming a thick ridge. The cuneate part is filled with unlignified

parenchyma cells, with the membrane wall usually not lignified in *Polygala senega* and slightly lignified in *Polygala senega* var. *latifolia.* The medullary rays are difficult to distinguish from other tissues, but consist of slightly thin membranes radiating, with no pith visible. The parenchyma cells of Senega contain oil droplets. Starch grains and calcium oxalate crystals are absent.
It has a characteristic odor, resembling the aroma of methyl salicylate. The taste is sweet at first, but leaving an acrid taste.

Identification (1) Weigh 0.5 g of pulverized Senega, add 10 mL of water, and shake vigorously; a lasting fine foam is produced.

(2) Weigh 0.5 g of pulverized Senega, add 30 mL of water, shake for 15 minutes to mix, and filter. Take 1 mL of the filtrate, mix with 50 mL of water, and determine the absorption spectrum of the solution as directed under the Ultraviolet-visible Spectroscopy; it exhibits a maximum absorption at the wavelength of about 317 nm.

Purity (1) ***Foreign matter***—(i) Stem: The amount of stems contained in Senega is less than 2.0%.

(ii) Other foreign matters: The amount of foreign matters other than stems contained in Senega is less than 1.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

Loss on drying NMT 13.0% (6 hours).

Ash NMT 5.0%.

Acid-insoluble ash NMT 2.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 30.0%.

Packaging and storage Preserve in well-closed containers.

Senna Leaf
센나엽

Sennae Folium

Senna Leaf is the leaflets of *Cassia angustifolia* Vahl or *Cassia acutifolia* Delile (Leguminosae).

Senna Leaf contains NLT 1.0% of total sennosides [as sennoside A ($C_{42}H_{38}O_{20}$: 862.74) and sennoside B ($C_{42}H_{38}O_{20}$: 862.74)], calculated on the dried basis.

Description Senna Leaf is the leaflet, elongated ovate to ovate lanceolate, 15 mm to 50 mm in length and 4 mm to 20 mm in width, The external surface occurs as a pale grayish yellow to pale grayish yellowish green color, with the leaf being entire and the apex being acute pointed. The phyllopodium is asymmetric, and primary lateral veins are running toward the apex along the margin and joining together. The upper surface is flat, while the lower surface has slight hairs with the veins of the lower surface protruding. The petiole of the leaflet is short.
Under a microscope, a transverse section of Senna Leaf reveals the epidermis with thick cuticles on both sides, with numerous stomata and thick-walled, warty unicellular hairs. The epidermis cells are often separated into two loculi by a septum, which is in parallel with the surface of the leaf, and contain a mucilaginous substance in the inner loculus. There is a single layer of the palisade parenchyma under the epidermis on both the upper and lower sides. The spongy tissue is consisted of 3 to 4 layers and contains clustered and solitary crystals of calcium oxalate. Cells adjacent to the vascular bundle form crystal-bearing cells.
It has a slight characteristic odor and a bitter taste.

Identification (1) Weigh 0.5 g of pulverized Senna Leaf, add 10 mL of ether, macerate for 2 minutes, and filter. Then, add 5 mL of ammonia TS to the filtrate; the aqueous layer exhibits a yellowish red color. To the residue of ether-extraction, add 10 mL of water, macerate for 2 minutes, and filter. Add 5 mL of ammonia TS to the filtrate; the aqueous layer exhibits a yellowish red color.

(2) Weigh 2 g of pulverized Senna Leaf, add 40 mL of a mixture of tetrahydrofuran and water (7 : 3), shake for 30 minutes to mix, and centrifuge. Transfer the supernatant to a separatory funnel, add 13 g of sodium chloride, and shake for 30 minutes to mix. Separate the water layer together with the undissolved sodium chloride and adjust the pH to 1.5 by adding 1 mol/L hydrochloric acid TS. Transfer this solution to another separatory funnel, shake with 30 mL of tetrahydrofuran for 10 minutes to mix, take the tetrahydrofuran layer, and use this solution as the test solution. Separately, weigh 1 mg of sennoside A RS, dissolve in 1 mL of a mixture of tetrahydrofuran and water (7 : 3), and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel (with a fluorescent agent) for thin layer chromatography. Next, develop the plate with a mixture of ethyl acetate, n-propanol, water and acetic acid (100) (40 : 40 : 30 : 1) as the developing solvent to a distance of about 15 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 365 nm); the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

Purity (1) ***Foreign matter***—(i) Petioles and fruits: The amount of petioles and fruits contained in Senna Leaf is less than 5.0%.

(ii) Other foreign matters: The amount of foreign matters other than petioles and fruits contained in Senna Leaf is less than 1.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.

(ii) Arsenic: NMT 3 ppm.

(iii) Mercury: NMT 0.2 ppm.

(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.

(ii) Dieldrin: NMT 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.

(iv) Aldrin: NMT 0.01 ppm.

(v) Endrin: NMT 0.01 ppm.

Loss on drying NMT 12.0% (6 hours).

Ash NMT 12.0%.

Acid-insoluble ash NMT 2.0%.

Assay Weigh accurately about 0.5 g of pulverized Senna Leaf in stoppered centrifuge tube, add 25 mL of diluted methanol (7 in 10), shake for 30 minutes to mix, centrifuge, and take the supernatant. To the residue, add 10 mL of diluted methanol (7 in l0), shake for 10 minutes to mix, centrifuge, and take the supernatant. Repeat this process twice. Combine all the extracts, add diluted methanol (7 in l0) to make exactly 50 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of sennoside A RS (previously dried in a desiccator with silica gel for NLT 24 hours), dissolve in a solution of sodium bicarbonate (1 in 100) to make exactly 20 mL, and use this solution as the standard stock solution (1). Weigh accurately about 10 mg of sennoside B RS (previously dried in a desiccator with silica gel for NLT 24 hours), dissolve in a solution of sodium bicarbonate (1 in 100) to make exactly 20 mL, and use this solution as the standard stock solution (2). Pipet 5 mL of the standard stock solution (1) and 10 mL of the standard stock solution (2), add methanol to make exactly 50 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions, and determine the peak areas, A_{Ta} and A_{Tb}, from the test solution and the peak areas, A_{Sa} and A_{Sb}, from the standard solution. Calculate the amounts of sennoside A and sennoside B according to the following equations; the sum is the total amount of sennosides.

$$\text{Amount (mg) of sennoside A } (C_{42}H_{38}O_{20}) = \text{Amount (mg) of sennoside A RS} \times \frac{A_{Ta}}{A_{Sa}} \times \frac{1}{4}$$

$$\text{Amount (mg) of sennoside B } (C_{42}H_{38}O_{20}) = \text{Amount (mg) of sennoside B RS} \times \frac{A_{Tb}}{A_{Sb}} \times \frac{1}{2}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 340 nm)

Column: A stainless steel column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Column temperature: A constant temperature of about 50 ℃

Mobile phase: Dissolve 2.45 g of tetra-n-heptylammonium bromide in l000 mL of a mixture of 1 mol/L acetic acid-sodium acetate buffer solution (1 in 10) (pH 5.0) and acetonitrile (17 : 8).

Flow rate: Adjust the flow rate so that the retention time of sennoside A is about 26 minutes.

System suitability

Proceed with 10 μL of the standard solution under the above conditions; sennoside A and sennoside B are eluted in this order with clear separation of each peak.

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak areas of sennoside A is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Sinomenium Stem and Rhizome
방기(防己)

Sinomeni Caulis et Rhizoma

Sinomenium Stem and Rhizome is the climbing stem and rhizome of *Sinomenium acutum* Rehder et Wilson (Menispermaceae).

Description Sinomenium Stem and Rhizome is the climbing stem and rhizome, long cylindrical, slightly curved, 20 cm to 70 cm in length and 0.5 cm to 2 cm in diameter. The outer surface occurs as greenish brown, maroon or grayish brown and the nodes are slightly expanded. The body is light and the texture is firm but brittle and easy to cut. The cut surface occurs as grayish yellow or pale grayish brown, the cortex is narrow, medullary rays radiate into the xylem and the pith is pale yellowish white or yellowish brown.

Under the microscope, the cross-section shows the epidermis of the outermost layer, which is covered by a thick cuticular layer or consists of a cork layer. The cortex is interspersed with fibers and stone cells. The fiber bundles of the peduncle sheath are crescent-shaped with 2 to 5 rows of stone cells on the inner side. The stone cells extend outward to join the stone cell bundles in the medullary rays to form a ring. The vascular bundles are collateral. In the phloem, the medullary rays become broader outward and the stone cells are wedge-shaped or branched. The phloem cells are mostly degenerated, sometimes scattered with 1 to 3 fibers. The xylem is scattered with single vessels or multiple vessels connected longitudinally. The pith cells have thick cell walls and distinct pits. The parenchyma cells contain starch granules and calcium oxalate crystals.

It occurs as almost odorless and has a bitter taste.

Identification (1) Weigh 0.5 g of pulverized Sinomenium Stem and Rhizome, add 10 mL of dilute acetic acid, heat for 2 minutes on a water bath with frequent shaking, cool and filter. To 5 mL of the filtrate, add 2 drops of Dragendorff's TS; an orange-yellow precipitate is produced immediately.

(2) Weigh 2 g each of pulverized Sinomenium Stem and Rhizome and Sinomenium stem and rhizome RMPM, add 25 mL of methanol, heat after connecting with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrates, dissolve in 1 mL of ethanol and use these solutions as the test solution and the Sinomenium stem and rhizome RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the Sinomenium stem and rhizome RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography (with fluorescent indicator). Next, develop the plate with a mixture of methanol and ammonium hydroxide (65 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 254 nm); the spots obtained from the test solution show the same color and R_f value as the several spots from the Sinomenium stem and rhizome RMPM standard solution.

Purity (1) Weigh 2 g of pulverized Sinomenium Stem and Rhizome, add 25 mL of ethanol, sonicate for 1 hour and filter. Evaporate the filtrate to dryness, dissolve the residue in 1 mL of ethanol and use this solution as the test solution. Separately, weigh 1 mg of aristolochic acid RS, dissolve it in 1 mL of ethanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the standard

solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography (with fluorescent agent). Next, develop the plate with a mixture of toluene, ethyl acetate, methanol and formic acid (20 : 10 : 1 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 254 nm) or spray evenly aluminum chloride TS on the plate and examine it under ultraviolet light (main wavelength: 365 nm); The several spots obtained from the test solution and a spot from the standard solution do not show the same color and the same R_f value.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 6.0%.

Packaging and storage Preserve in well-closed containers.

Sophora Flower
괴화(槐花)

Sophorae Flos

Sophora Flower is the flower bud and flower of *Sophora japonica* Linné (Leguminosae). The former is known as Koemi, and the latter Koehwa

Description (1) ***Koemi***—Koemi is the flower bud, ovoid or elliptical, 2 - 6 mm in length, about 2 mm in diameter. The lower part of the calyx has several longitudinal patterns, while the upper part of the calyx has unblown yellowish white petals. The stalk is thin and small. The body is light and breaks upon rubbing by hand.
It has a slight, characteristic odor and a slightly bitter and astringent taste.

(2) ***Koehwa***—Koehwa is the flower, wrinkled and rolled. The calyx occurs as campanulate and yellowish green, and the apex is shallowly divided into 5 pieces. There are 5 petals, yellow or yellowish white. One of the petals is relatively larger, nearly round, with the slightly concave apex. The other 4 petals are long and round. There are 10 stamens, 9 of which are accreted at the base. The stalk of the stamen is thin and long. The pistil is cylindrical and curved.
It has a characteristic odor and a slightly bitter taste.

Identification (1) Weigh 0.5 g of pulverized Sophora Flower, add 10 mL of methanol to extract, filter, and use the filtrate as the test solution. Spot 2 to 3 drops of the filtrate on the filter paper and drop 1% alum solution onto the filtrate; the area where the two liquids contact exhibits a yellow color. Under ultraviolet light, the area where the test solution is present exhibits a yellowish brown color, and the area where the two liquids contact exhibits radiant yellow fluorescence.

(2) Weigh 0.2 g of pulverized Sophora Flower, add 5 mL of methanol, shake for 10 minutes to mix, filter, and use the filtrate as the test solution. Weigh 8 mg of rutin RS, add 1 ml of methanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel (with a fluorescent agent) for thin layer chromatography. Next, develop the plate with a mixture of ethyl acetate, formic acid and water (8 : 1 : l) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly aluminum chloride TS on the plate and examine under ultraviolet light (main wavelength: 365 nm); the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

Purity (1) ***Foreign matter***—Sophora Flower contains less than 10.0% of the peduncles and other foreign matters.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 9.0%.

Packaging and storage Preserve in well-closed containers.

Sophora Root
고삼(苦參)

Sophorae Radix

Sophora Root is the root of *Sophora flavescens* Solander ex Aiton (Leguminosae), intact or without periderm.

Sophora Root contains NLT 1.0% of the sum of oxymatrine ($C_{15}H_{24}N_2O_2$: 264.36) and matrine ($C_{15}H_{24}N_2O_2$: 264.36), calculated on the dried basis.

Description Sophora Root is cylindrical root, 51 cm to 20 cm in length and 2 cm to 3 cm in diameter. The outer surface occurs as dark brown to yellowish brown, with distinct longitudinal wrinkles and with laterally elongated lenticels. The outer surface of the root without periderm occurs as yellowish white. Fractured surface is a somewhat fibrous surface. The cross-section occurs as pale yellowish brown. The cortex is 1 mm to 2 mm thick, slightly darkened near the cambium, and forms a fissure between the xylem.
It has a characteristic odor and has an extremely bitter and persistent taste.

Identification (1) Weigh 0.5 g of pulverized Sophora Root, add 10 mL of dilute acetic acid, heat on a water bath for 3 minutes with occasional shaking, cool, and filter. To 5 mL of the filtrate, add 2-3 drops of Dragendorff's TS; an orange yellow precipitate is produced immediately.

(2) Weigh 1 g of pulverized Sophora Root and sophora root RMPM, add 50 mL of methanol, connect with a reflux condenser, heat the mixture for 1 hour, and filter. Vacuum-concentrate the

filtrate, dissolve it in 1 mL of methanol, and use these solutions as the test solution and the sophora root RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 4 μL each of the test solution and the sophora root RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of cyclohexane, acetone and ethyl acetate (5 : 2 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS on the plate and heat it at 105 °C for 10 minutes; the spots obtained from the test solution show the same color and R_f value as the spots from the sophora root RMPM standard solution and of these, a reddish brown spot is observed at the R_f value of about 0.7.

Purity (1) ***Foreign matter***—(i) Stem: NMT 10.0%.
(ii) Other foreign matter: Sophora Root contains less than 1.0% of foreign matter other than stems.
(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm
(iv) Cadmium: NMT 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 11.0%.

Ash NMT 6.0%.

Acid-insoluble Ash NMT 1.5%.

Assay Weigh accurately about 4 g of pulverized Sophora Root, add 50 mL of diluted ethanol (3 in 4), sonicate for 20 minutes, and filter. To the residue, add 50 mL of diluted ethanol (3 in 4), and proceed in the same manner. Combine all the filtrates, evaporate to concentration in vacuum, and dissolve in water completely, followed by the addition of 10% hydrochloride solution to adjust to pH 2. Add 50 mL of dichloromethane to wash, collect the aqueous layer, add 50 mL of dichloromethane and proceed in the same manner. Add potassium carbonate to aqueous layer to adjust pH to 10, add 50 mL of dichloromethane, and extract by shaking. Add 50 mL of dichloromethane to aqueous layer, and proceed in the same manner. Combine all the extracts, evaporate to dryness in vacuum, dissolve the residue in methanol to make exactly 10 mL, and use this solution as the test solution. Separately, weigh accurately about 20 mg of oxymatrine RS and about 10 mg of matrine RS, dissolve in methanol to make exactly 10 mL, and use this solution as the standard solutions. Perform the test with 10 μL each of the test solution and the standard solution as directed under Liquid chromatography according to the following conditions, and determine the peak areas, A_{Ta} and A_{Tb}, of the test solution and the peak areas, A_{Sa} and A_{Sb}, of the standard solutions.

$$\text{Amount (mg) of oxymatrine } (C_{15}H_{24}N_2O_2) = \text{Amount (mg) of oxymatrine RS} \times \frac{A_{Ta}}{A_{Sa}}$$

$$\text{Amount (mg) of matrine } (C_{15}H_{24}N_2O) = \text{Amount (mg) of matrine RS} \times \frac{A_{Tb}}{A_{Sb}}$$

Operating conditions
Detector: An ultraviolet absorption photometer (wavelength: 205 nm)
Column: A stainless column, 4.6 mm in internal diameter and 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm in particle diameter).
Column temperature: An ordinary temperature
Mobile phase: A mixture of potassium phosphate buffer solution (pH 6.0) and acetonitrile (91 : 9).
Flow rate: 1.0 mL/min
System suitability
System performance: Proceed with10 μL of the standard solution according to the above conditions; oxymatrine and matrine are eluted in this order and the individual peaks are completely separated.
System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak area of oxymatrine and matrine is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Sparganium Rhizome
삼릉(三棱)

Sparganii Rhizoma
Sparganium Rhizome is the tuber of *Sparganium stoloniferum* Buchanan-Hamilton (Sparganiaceae).

Description Sparganium Rhizome is a tuber, conical, slightly flattened, 2 cm to 6 cm in length and 2 cm to 4 cm in diameter. The external surface occurs as a yellowish white or grayish yellow color, with marks pared with a knife and small dot-like fibrous rootlet scars arranged transversely in a ring pattern. The body is heavy, and the texture is compact.
Under a microscope, the transverse section of Sparganium Rhizome reveals the cortex consisting of the aerenchyma tissue and the parenchyma cells irregular in shape with large cavities between cells. Cells are tightly arranged in the endodermis. Parenchyma cells of the central cylinder are close to a circular shape, of which cell walls are mostly thickened, and starch grains are present inside. The vascular bundles are collateral and amphivasal, and scattered, and the vessels are not lignified. Secretory cells are evenly scattered in the cortex and the central cylinder and contain a reddish brown secretory substance.
It occurs as almost odorless and has a plain and slightly astringent taste slightly numbing on chewing.

Identification Weigh 2 g each of pulverized Sparganium Rhizome and Sparganium rhizome RMPM, add 30 mL each of ethanol, warm after connecting with a reflux condenser for 10 minutes on a water bath, filter, and evaporate the filtrates to dryness. To each residue, add 2 mL of ethanol to prepare the test solution and the Sparganium rhizome RMPM standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the Sparganium rhizome RMPM standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of cyclohexane and ethyl acetate (4 : 1) as the developing solvent to a

distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS on the plate and heat at 105 ℃ for 10 minutes; the color and the R_f values of the several spots obtained from the test solution are the same as those of the spots obtained from the Sparganium rhizome RMPM standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 10.0%.

Ash NMT 5.0%.

Acid-insoluble ash NMT 1.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 11.0%.

Packaging and storage Preserve in well-closed containers.

Ssanghwatang Extract Granules
쌍화탕엑스 과립

Ssanghwatang Extract Granules contains NLT 12.2 mg of paeoniflorin ($C_{23}H_{28}O_{11}$: 480.46) in Peony Root and 4.7 mg of glycyrrhizic acid ($C_{42}H_{62}O_{16}$: 822.93) in Licorice per dose (one sachet).

Method of preparation for a dose (one sachet)

Peony Root	3.13 g
Angelica Gigas Root, Prepared Rehmannia Root, Cnidium Rhizome, Astragalus Root	1.25 g
Licorice, Cinnamon Bark	0.94 g
Jujube	0.67 g
Ginger	0.50 g

Pulverize the above crude drugs into coarse powder, weigh each crude drug, put it into the extractor, add eight to ten times the amount of water, extract for 2 to 3 hours at 80 to 100 ℃ and filter. Concentrate the filtrate at 60 ℃ under vacuum to give 3.37 g to 5.05 g of semiliquid extract, or concentrate by an appropriate method to give 1.32 g to 1.98 g of dry extract. Prepare Ssanghwatang Extract Granules as directed under the Granule.

Identification (1) ***Angelica Gigas Root***—Pulverize Ssanghwatang Extract Granules and weigh an appropriate amount corresponding to 1 g of Angelica Gigas Root, dissolve it in 10 mL of water, and shake for 5 minutes. Then, add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour, and filter. Concentrate the filtrate in vacuum to give 10 mL, and use this solution as the test solution. Separately, weigh 1 g of pulverized Angelica Gigas Root, add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate in vacuum to give 10 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of toluene, ether, water, and acetic acid (500 : 500 : 5 : 2) as a developing solvent to a distance of about 10 cm, and air-dry the plate. Spray vanillin-sulfuric acid TS evenly on the plate and heat at 105 ℃ for 10 minutes; one spot among the several spots obtained from the test solution and a spot obtained from the standard solution show the same color and the same R_f value.

(2) ***Prepared Rehmannia Root***—Pulverize Ssanghwatang Extract Granules and weigh an appropriate amount corresponding to 1 g of Prepared Rehmannia Root, dissolve it in 10 mL of water, and shake for 5 minutes. Then, add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour, and filter. Concentrate the filtrate in vacuum to give 10 mL, and use this solution as the test solution. Separately, weigh 1 g of pulverized Prepared Rehmannia Root, add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate in vacuum to give 10 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of chloroform and methanol (20 : 1) as a developing solvent to a distance of about 10 cm, and air-dry the plate. Spray 2,4-dinitrophenylhydrazine TS evenly on the plate. Examine the plate under ultraviolet light (maim wavelength: 254 nm); one spot among the several spots obtained from the test solution and a spot obtained from the standard solution show the same color and the same R_f value.

(3) ***Cnidium Rhizome***—Pulverize Ssanghwatang Extract Granules and weigh an appropriate amount corresponding to 1 g of Cnidium Rhizome, dissolve it in 10 mL of water, and shake for 5 minutes. Then, add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour, and filter. Concentrate the filtrate in vacuum to give 10 mL, and use this solution as the test solution. Separately, weigh 1 g of pulverized Cnidium Rhizome, add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate in vacuum to give 10 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel (with fluorescent indicator) for thin layer chromatography. Develop the plate with a mixture of cyclohexane and ethyl acetate (9 : 1) as a developing solvent to a distance of about 10 cm, and air-dry the plate. Spray sulfuric acid TS for spraying evenly to the plate and heat at 105 ℃ for 10 minutes. Examine the plate under ultraviolet light (main wavelength: 365 nm); one spot among the several spots obtained from the test solution and a spot obtained from the standard solution show the same color and the same R_f value.

(4) ***Astragalus Root***—Pulverize Ssanghwatang Extract Granules and weigh an appropriate amount corresponding to 1 g of Astragalus Root, dissolve it in 10 mL of water, and shake for 5 minutes. Then, add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour, and filter. Concentrate the filtrate in vacuum to give 10 mL, and use this solution as the test solution. Separately, weigh 1 g of pulverized Astragalus Root, add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate in vacuum to give 10 mL, and use this solution

as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel (with fluorescent indicator) for thin layer chromatography. Develop the plate with a mixture of toluene and methanol (93 : 7) to a distance of about 10 cm, and air-dry the plate. Spray vanillin-sulfuric acid TS evenly to the plate and heat at 105 ℃ for 10 minutes. Examine the plate under ultraviolet light (main wavelength: 365 nm); one spot among the several spots obtained from the test solution and a spot obtained from the standard solution show the same color and the same R_f value.

(5) ***Cinnamon Bark***—Pulverize Ssanghwatang Extract Granules and weigh an appropriate amount corresponding to 1 g of Cinnamon Bark, dissolve it in 10 mL of water, and shake for 5 minutes. Then, add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour, and filter. Concentrate the filtrate in vacuum to give 10 mL, and use this solution as the test solution. Separately, weigh 1 g of pulverized Cinnamon Bark, add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate in vacuum to give 10 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of hexane and ethyl acetate (85 : 15) as a developing solvent to a distance of about 10 cm, and air-dry the plate. Spray saturated *o*-dianisidine-acetic acid (100) solution (prepare before use) evenly to the plate; one spot among the several spots obtained from the test solution and a spot obtained from the standard solution show the same color and the same R_f value.

(6) ***Jujube***—Pulverize Ssanghwatang Extract Granules and weigh an appropriate amount corresponding to 1 g of Jujube, dissolve it in 10 mL of water, and shake for 5 minutes. Then, add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour, and filter. Concentrate the filtrate in vacuum to give 10 mL, and use this solution as the test solution. Separately, weigh 1 g of pulverized Jujube, add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate in vacuum to give 10 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of chloroform and methanol (6 :1) as a developing solvent to a distance of about 10 cm, and air-dry the plate. Spray *p*-anisaldehyde-sulfuric acid TS evenly to the plate and heat at 105 ℃ for 10 minutes; one spot among the several spots obtained from the test solution and a spot obtained from the standard solution show the same color and the same R_f value.

(7) ***Ginger***—Pulverize Ssanghwatang Extract Granules and weigh an appropriate amount corresponding to 1 g of Ginger, dissolve it in 10 mL of water, and shake for 5 minutes. Then, add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour, and filter. Concentrate the filtrate in vacuum to give 10 mL, and use this solution as the test solution. Separately, weigh 1 g of Ginger, add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate in vacuum to give 10 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of hexane and ethyl acetate (17 : 3) as a developing solvent to a distance of about 10 cm, and air-dry the plate. Spray vanillin-sulfuric acid TS evenly to the plate and heat at 105 ℃ for 10 minutes; one spot among the several spots obtained from the test solution and a spot obtained from the standard solution show the same color and the same R_f value.

(8) ***Peony Root, Licorice***—Proceed as directed under the Assay; the test solution shows a peak at the same retention time as the standard solution.

Purity (1) ***Heavy metals***—(i) Total heavy metals: NMT 30 ppm.

(ii) Lead: NMT 5 ppm.

(iii) Arsenic: NMT 3 ppm.

Disintegration Meets the requirements.

Particle size distribution estimation by analytical sieving Meets the requirements.

Uniformity of dosage units (distribution) Meets the requirements.

Microbial limit Meets the requirements.

Assay (1) ***Paeoniflorin of Peony root***—Take NLT 20 sachets of Ssanghwatang Extract Granules, weigh accurately and pulverize. Weigh accurately an amount equivalent to about 10 mg of paeoniflorin, add 10 mL of water, and shake for 5 minutes, Add 100 mL of methanol, heat the mixture with a reflux condenser for 1 hour, take the supernatant and filter. To the residue, add 100 mL of methanol and extract twice repetitively. Combine the filtrate, concentrate the filtrate in vacuum until it gives 50 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of paeoniflorin RS (previously dried in a silica gel desiccator for 24 hours), dissolve it in methanol to make exactly 50 mL, and use this solution as the standard solution. Perform the test as directed in the Assay of Peony Root.

(2) ***Glycyrrhizic acid in licorice***—Take NLT 20 sachets of Ssanghwatang Extract Granules, weigh accurately and pulverize. Weigh accurately an amount equivalent to 10 mg of glycyrrhizic acid, add 50 mL of water, and heat the mixture after connecting with a reflux condenser for 3 hours. Add 50 mL of 3 mol/L of sulfuric acid TS and hydrolyze on a water bath for 1 hour. After cooling, add 50 mL of chloroform, heat the mixture after connecting with a reflux condenser for 30 minutes. After cooling, transfer the mixture to a separatory funnel, and collect the chloroform layer. Extract three times with 30 mL of chloroform, combine all chloroform layers and filter through anhydrous sodium sulfate. Concentrate the filtrate in vacuum, dissolve the residue in methanol to make exactly 50 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of glycyrrhizic acid RS (previously dried in a silica gel desiccator for 24 hours). Operate in the same manner as the test solution, and use the resulting solution as the standard solution. Pipet 10 μL each of the test solution and the standard solution and perform the test as directed under Liquid Chromatography according to the following conditions. Determine the peak areas, A_T and A_S, of glycyrrhizic acid in the test solution and the standard solution, respectively.

$$\text{Amount (mg) of glycyrrhizic acid } (C_{42}H_{62}O_{16}) = \text{Amount (mg) of glycyrrhizic acid RS} \times \frac{A_T}{A_S}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 254 nm)

Column: A stainless steel column, 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (particle diameter: 5 μm to 10 μm in).

Mobile phase: A mixture of methanol, water and acetic acid (100) (78 : 19 : 3)

Flow rate: 1.0 mL/min

Packaging and storage Preserve in tight containers.

Ssanghwatang Extract Solutions
쌍화탕 액

Ssanghwatang Extract Solution contains NLT 17.1 mg of paeoniflorin ($C_{23}H_{28}O_{11}$: 480.46) in Peony Root and 5.6 mg of glycyrrhizic acid ($C_{42}H_{62}O_{16}$: 822.93) in Licorice per dose (one bottle).

Method of preparation for a dose (one bottle)

Peony Root	3.13 g
Angelica Gigas Root, Prepared Rehmannia Root, Cnidium Rhizome, Astragalus Root	1.25 g
Licorice, Cinnamon Bark	0.94 g
Jujube	0.67 g
Ginger	0.50 g
Purified water	An appropriate amount
	To make 100 mL

Pulverize the above crude drugs into coarse powder, weigh each crude drug, put it into the extractor, add eight to ten times the amount of water, extract for 2 to 3 hours at 80 to 100 ℃ and filter. Prepare Ssanghwatang Extract Solution with the filtrate as directed under the Liquids.

Identification (1) ***Angelica Gigas Root***—Weigh an appropriate amount of Ssanghwatang Extract Solution corresponding to 1 g of Angelica Gigas Root, dissolve it in 100 mL of methanol, shake to mix, and filter. Concentrate the filtrate in vacuum to give 20 mL, and use this solution as the test solution. Separately, weigh 1 g of pulverized Angelica Gigas Root, add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate in vacuum until the filtrate becomes 20 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of toluene, ether, water, and acetic acid (500 : 500 : 5 : 2) as a developing solvent to a distance of about 10 cm and air-dry the plate. Spray vanillin-sulfuric acid TS evenly on the plate and heat at 105 ℃ for 10 minutes; one spot among the several spots obtained from the test solution and a spot obtained from the standard solution show the same color and the same R_f value.

(2) ***Prepared Rehmannia Root***—Weigh an appropriate amount of Ssanghwatang Extract Solution corresponding to 1 g of Prepared Rehmannia Root, dissolve in 100 mL of methanol, shake to mix, and filter. Concentrate the filtrate in vacuum to give 20 mL, and use this solution as the test solution. Separately, weigh 1 g of pulverized Prepared Rehmannia Root, add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate in vacuum until the filtrate becomes 20 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel (with fluorescent indicator) for thin layer chromatography. Develop the plate with a mixture of chloroform and methanol (20 : 1) as a developing solvent to a distance of about 10 cm, and air-dry the plate. Spray 2,4-dinitrophenylhydrazine TS evenly on the plate. Examine the plate under ultraviolet light (maim wavelength: 254 nm); one spot among the several spots obtained from the test solution and a spot obtained from the standard solution show the same color and the same R_f value.

(3) ***Cnidium Rhizome***—Weigh an appropriate amount of Ssanghwatang Extract Solution corresponding to 1 g of Cnidium Rhizome, dissolve it in 100 mL of methanol, shake to mix, and filter. Concentrate the filtrate in vacuum to give 20 mL, and use this solution as the test solution. Separately, weigh 1 g of pulverized Cnidium Rhizome, add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate in vacuum until the filtrate becomes 20 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel (with fluorescent indicator) for thin layer chromatography. Develop the plate with a mixture of cyclohexane and ethyl acetate (9 : 1) as a developing solvent to a distance of about 10 cm, and air-dry the plate. Spray sulfuric acid TS for spraying evenly to the plate and heat at 105 ℃ for 10 minutes. Examine the plate under ultraviolet light (main wavelength: 365 nm); one spot among the several spots obtained from the test solution and a spot obtained from the standard solution show the same color and the same R_f value.

(4) ***Astragalus Root***—Weigh an appropriate amount of Ssanghwatang Extract Solution corresponding to 1 g of Astragalus Root, dissolve it in 100 mL of methanol, shake to mix, and filter. Concentrate the filtrate in vacuum to give 20 mL, and use this solution as the test solution. Separately, weigh 1 g of pulverized Astragalus Root, add 100 mL of methanol, heat the mixture with a reflux condenser for 1 hour and filter. Concentrate the filtrate in vacuum until the filtrate becomes 20 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel (with fluorescent indicator) for thin layer chromatography. Develop the plate with a mixture of toluene and methanol (93 : 7) as a developing solvent to a distance of about 10 cm, and air-dry the plate. Spray vanillin-sulfuric acid TS evenly to the plate and heat at 105 ℃ for 10 minutes. Examine the plate under ultraviolet light (main wavelength: 365 nm); one spot among the several spots obtained from the test solution and a spot obtained from the standard solution show the same color and the same R_f value.

(5) ***Cinnamon Bark***—Weigh an appropriate amount of Ssanghwatang Extract Solution corresponding to 1 g of Cinnamon Bark, dissolve it in 100 mL of methanol, shake to mix, and filter. Concentrate the filtrate in vacuum to give 20 mL, and use this solution as the test solution. Separately, weigh 1 g of pulverized Cinnamon Bark, add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour and filter.

Concentrate the filtrate in vacuum until the filtrate becomes 20 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of hexane and ethyl acetate (85 : 15) as a developing solvent to a distance of about 10 cm, and air-dry the plate. Spray saturated *o*-dianisidine-acetic acid (100) solution (prepare before use) evenly to the plate; one spot among the several spots obtained from the test solution and a spot obtained from the standard solution show the same color and the same R_f value.

(6) ***Jujube***—Weigh an appropriate amount of Ssanghwatang Extract Solution corresponding to 1 g of Jujube, dissolve it in 100 mL of methanol, shake to mix, and filter. Concentrate the filtrate in vacuum to give 20 mL, and use this solution as the test solution. Separately, weigh 1 g of pulverized Jujube, add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate in vacuum until the filtrate becomes 20 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of chloroform and methanol (6 : 1) as a developing solvent to a distance of about 10 cm, and air-dry the plate. Spray *p*-anisaldehyde-sulfuric acid TS evenly to the plate and heat at 105 ℃ for 10 minutes; one spot among the several spots obtained from the test solution and a spot obtained from the standard solution show the same color and the same R_f value.

(7) ***Ginger***—Weigh an appropriate amount of Ssanghwatang Extract Solution corresponding to 1 g of Ginger, dissolve it in 100 mL of methanol, shake to mix, and filter. Concentrate the filtrate in vacuum to give 20 mL, and use this solution as the test solution. Separately, weigh 1 g of Ginger, add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate in vacuum until the filtrate becomes 20 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of hexane and ethyl acetate (17 : 3) as a developing solvent to a distance of about 10 cm, and air-dry the plate. Spray vanillin-sulfuric acid TS evenly to the plate and heat at 105 ℃ for 10 minutes; one spot among the several spots obtained from the test solution and a spot obtained from the standard solution show the same color and the same R_f value.

(8) ***Peony Root, Licorice***—Proceed as directed under the Assay; the test solution shows a peak at the same retention time as the standard solution.

Purity (1) ***Heavy metals***—(i) Total heavy metals: NMT 30 ppm.

(ii) Lead: NMT 5 ppm.

(iii) Arsenic: NMT 3 ppm.

pH 3.0 to 5.0

Specific gravity d_{20}^{20}: 0.980 to 1.080

Uniformity of dosage units (distribution) Meets the requirements.

Microbial limit Meets the requirements.

Assay (1) ***Paeoniflorin in Peony root***—Weigh accurately an amount of Ssanghwatang Extract Solution equivalent to about 10 mg of paeoniflorin, add 10 mL of water, and shake for 5 minutes. Add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour, collect the supernatant and filter. To the residue, add 100 mL of methanol and extract twice repetitively. Combine the filtrate, concentrate the filtrate in vacuum until it gives 50 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of paeoniflorin RS (previously dried in a silica gel desiccator for 24 hours), dissolve it in methanol to make exactly 50 mL, and use this solution as the standard solution. Perform the test as directed in the Assay of Peony Root.

(2) ***Glycyrrhizic acid in Licorice*** —Weigh accurately an amount of Ssanghwatang Extract Solution equivalent to about 10 mg of glycyrrhizic acid, and heat it after connecting with a reflux condenser for 3 hours. Add 50 mL of 3 mol/L sulfuric acid TS, and hydrolyze the mixture on a water bath for 1 hour. After cooling, add 50 mL of chloroform, heat the mixture after connecting with a reflux condenser for 30 minutes. After cooling, transfer the mixture to a separatory funnel, and collect the chloroform layer. Extract three times with 30 mL of chloroform, combine all chloroform layers and filter through anhydrous sodium sulfate. Concentrate the filtrate in vacuum, dissolve the residue in methanol to make exactly 50 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of glycyrrhizic acid RS (previously dried in a silica gel desiccator for 24 hours), and add 50 mL of water. Operate in the same manner as the test solution, and use the resulting solution as the standard solution. Pipet 10 μL each of the test solution and the standard solution and perform the test as directed under Liquid Chromatography according to the following conditions. Determine the peak areas, A_T and A_S, of glycyrrhizic acid in the test solution and the standard solution, respectively

$$\text{Amount (mg) of glycyrrhizic acid } (C_{42}H_{62}O_{16}) = \text{Amount (mg) of glycyrrhizic acid RS} \times \frac{A_T}{A_S}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 254 nm)

Column: A stainless steel column, 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (particle diameter: 5 μm to 10 μm in).

Mobile phase: A mixture of methanol, water and acetic acid (100) (78 : 19 : 3)

Flow rate: 1.0 mL/min

Packaging and storage Preserve in tight containers.

Star Anis Fruit
팔각회향(八角茴香)

Illici Veri Fructus

Star Anis Fruit is the fruit of *Illicium verum* Hook. Fil. (Illiciaceae), dried as it is or dried after passing through hot water.

Description Star Anis Fruit is mostly aggregate fruit of 8 mericarps radiated from the central axis. Each mericarp is 10 mm to

20 mm in length, 3 mm to 5 mm in width, and 6 mm to 10 mm in height. External surface occurs as reddish brown, irregularly wrinkled, with summit beaked and upper part mostly dehiscent. Inner surface occurs as pale brown, smooth and lustrous. Texture is hard and brittle. Fruit stalk is 30 mm to 40 mm in length, connected to the base of the fruit, curved, and usually deciduous. Each mericarp contains one seed. The seeds are flat ovoid in shape, about 6 mm in length, reddish brown to yellowish brown in color, lustrous, with a hilum at the end. Endosperm is white and oily.
It has a characteristic aroma and tastes pungent and sweet.

Identification Weigh 1 g of pulverized Star Anis Fruit, add 10 mL of hexane, shake to mix, and allow to stand for 5 minutes. Then, filter and use the filtrate as the test solution. Separately, weigh 1 mg of anethole RS and 1 mg of anisaldehyde RS, dissolve each in 1 mL of ethanol and use these solutions as the standard solutions (1) and (2). With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel (with fluorescence indicator) for thin layer chromatography. Next, develop the plate with a mixture of hexane and ethyl acetate (20 : 1) to a distance of about 10 cm and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 254 nm); Two spots among the several spots obtained from the test solution show the same color and R_f values as the spots obtained from the standard standards (1) and (2).

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 11.0%.

Ash NMT 4.0%.

Essential oil content NLT 0.4 mL (10.0 g).

Extract Content Dilute ethanol-soluble extract—NLT 15.0%.

Packaging and storage Preserve in well-closed containers.

Swertia Herb
당약(當藥)

Swertiae Herba

Swertia Herb is the whole plant of *Swertia japonica* Makino (Gentianaceae) collected during the flowering season.

Swertia Herb contains NLT 2.0% of swertiamarin ($C_{16}H_{22}0_{10}$: 374.34), calculated on the dried basis.

Description Swertia Herb is the whole plant and consists of flowers, opposite leaves, stems and usually short woody roots, reaching a length of about 20 cm. The Leaves and stems occur as dark green to dark purple or light brown, and the flowers are white to milky white. The stems are cylindrical, about 2 mm in diameter, occasionally with branches. The root occurs as yellowish brown. The leaves are wrinkled and when smoothed by immersion in water, the leaves are linear to narrowly lanceolate, 1 to 4 cm long, 1 to 5 mm wide. The leaf margins are not serrated and there are no petioles. The corolla is deeply divided into 5 parts, the lobes are narrow and long oval, and the peduncle is distinct. There are 5 stamens attached to the tube of the corolla and arranged alternately with the corolla lobes. Under the magnifying glass, the corolla reveals two elliptical nectary juxtaposed at the base of the inner surface, and the surroundings of the lobe have the shape of eyelashes.
It has a light, characteristic odor and extremely bitter and persistent taste.

Identification Weigh 2 g of pulverized Swertia Herb, add 10 mL of ethanol, shake for 5 minutes to mix. Then, filter it and use the filtrate as the test solution. Separately, weigh 2 mg of swertiamarin RS, dissolve it in 1 mL of ethanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 3 μL of the test solution and 5 μL of the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography (with fluorescent indicator). Next, develop the plate with a mixture of ethyl acetate, n-propanol, and water (6 : 4 : 3) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet rays (254 nm); one spot among the several spots obtained from the test solution and a spot from the standard solution show the same color and the same R_f value.

Purity (1) ***Foreign matter***—The amount of the straw and other foreign matters contained in Swertia Herb is less than 1.0%.
(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

Loss on drying NMT 12.0% (6 hours).

Ash NMT 6.5%.

Assay Weigh accurately about 1 g of pulverized Swertia Herb in a stoppered centrifuge tube, add 40 mL of methanol, and shake it for 15 minutes to mix. Then, centrifuge the mixture and collect the supernatant. Add 40 mL of methanol to the residue and proceed in the same manner. Combine the extracts, and add methanol to make exactly 100 mL. Pipet 5.0 mL of this solution, add the mobile phase to make exactly 20 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of swertiamarin RS (previously dried in a silica gel desiccator for 24 hours), and add methanol to make exactly 20 mL. Pipet 5 mL of this solution, add the mobile phase to make exactly 20 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as

directed under Liquid chromatography according to the following conditions, and determine A_T and A_S of the peak area of each solution.

$$\text{Amount (mg) of swertiamarin } (C_{16}H_{22}O_{10}) = \text{Amount (mg) of swertiamarin RS} \times \frac{A_T}{A_S} \times 5$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 238 nm)

Column: A stainless steel tube about 4.6 mm in internal diameter and about 15 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 50 °C

Mobile phase: A mixture of water and acetonitrile (91 : 9)

Flow rate: Adjust the flow rate so that the retention time of swertiamarin is about 12 minutes

System suitability

System performance: Weigh 1 mg each of swertiamarin RS and theophylline and dissolve each in the mobile phase to make 10 mL. Proceed with 10 μL of these solutions according to the above conditions; theophylline and swertiamarin are eluted in this order with each of the peaks completely separated.

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak area of swertiamarin is NMT 1.5%.

Packaging and storage Preserve in well-closed containers.

Terminalia Fruit
가자(訶子)

Terminaliae Fructus

Terminalia Fruit is the ripe fruit of *Terminalia chebula* Retzius or *Terminalia chebula* var. *tomentella* (Kurz) C.B.Clarke (Combretaceae).

Description (1) ***Terminalia chebula*** It is a fruit, 20 - 40 mm in length and 15 - 25 mm in diameter, with an oblong to ovoid shape. External surface occurs as yellowish brown to dark brown, often lustrous, with 5 - 6 longitudinal ridges and irregular wrinkles. At the bottom, there is a round fruit stalk mark. The texture is hard. Sarcocarp is yellowish brown, 2 - 4 mm in thickness. The putamen is pale yellow, 15 - 25 mm in length and 10 - 15 mm in diameter, of which surface is rough and hard. The seed is narrow and ellipsoidal, about 10 mm in length and 2 - 4 mm in diameter. The seed coat is yellowish brown. The number of cotyledons is two, which are white, overlapped to each other and rolled.
It has a slight, characteristic odor and sour, pungent and later sweet taste.

(2) *Terminalia chebula var. tomentella* It is a fruit. Compared to *Terminalia chebula* Retzins, the longitudinal ridges of *Terminalia chebula* var. *tomentella* (Kurz) are not clear and there are many irregular wrinkles.

Identification Weigh 0.5 g of pulverized Terminalia Fruit, add 10 mL of water, shake well to mix, and filter. To the filtrate, add 1 to 2 drops of Iron(III) chloride TS; the resulting solution exhibits a dark violet color.

Purity (1) ***Foreign matters***—Terminalia Fruit contains less than 2.0% of the peduncle and other foreign matters.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 12.0% (6 hours).

Ash NMT 5.0%.

Extract Content ***Dilute ethanol-soluble extract*** NLT 35.0%.

Packaging and storage Preserve in well-closed containers.

Thuja Seed
백자인(柏子仁)

Thujae Semen

Thuja Seed is the seed of *Thuja orientalis* Linné (Cupressaceae) from, which the seed coat is removed.

Description Thuja Seed is a seed, long ovoid or long elliptical, 4 to 7 mm long and 1.5 to 3 mm in diameter. The outer surface occur as yellowish white or pale yellowish brown and covered with a membranous tegmen. The apex is slightly pointed, with a small deep brown spot, and the base is bluntly rounded. The texture is soft and oily.
Under the microscope, the cross-section shows a tegmen on the outer surface consisting of a single row. The cells are long rod-shaped and usually connected with hypodermal cells containing a brown pigment. The endosperm is close to polygonal or close to circular, and the cell cavities are filled with relatively large aleurone grains and fat oil droplets. The aleurone grains leaving a mesh pattern after dissolution. The cotyledon cells are rectangular, and the cell cavities are filled with relatively small aleurone grains and fat oil droplets. The structure of the cotyledon shows 2 vascular bundles.
Thuja Seed has a characteristic odor and light taste.

Purity (1) ***Foreign matter***—Thuja Seed contains less than 1.0% of the endocarp and other foreign matter.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

(5) ***Mycotoxins***—Total aflatoxin (sum of aflatoxins B_1, B_2, G_1 and G_2): NMT 15.0 ppb (aflatoxin B_1 is NMT 10.0 ppb).

Loss on drying NMT 7.0%.

Ash NMT 6.0%.

Acid-insoluble ash NMT 1.0%.

Extract content ***Ether-soluble extract***—NLT 50.0%.

Packaging and storage Preserve in well-closed containers.

Toad Venom
섬수(蟾酥)

Bufonis Venenum

Toad Venom is a collection of secretions from the poisonous glands of *Bufo bufo gargarizans* Cantor or *Bufo melanostictus* Schneider (Bufonidae).

Toad Venom contains NLT 5.8% of bufosteroid, calculated on the dried basis.

Description Toad Venom is a collection of secretions, flat circular masses or pieces, varying in length and width. The external surface occurs as a chestnut or reddish brown color. Those in a shape of masses are hard and difficult to cut. The cut surface occurs as a chestnut color, horny and slightly lustrous. Those in a shape of pieces are fragile and easily broken. The cut surface occurs as a reddish brown color and is semi-translucent.
It has a fish-like odor and tastes sweet at first, later tingling with a lasting sensation of numbness. Pulverized Toad Venom stimulates the olfactory sense, causing sneezing.

Identification (1) Weigh 0.l g of pulverized Toad Venom, add 5 mL of chloroform, warm after connecting with a reflux condenser on a water bath for l0 minutes, and filter. Then, evaporate 1 mL of the filtrate to dryness on a water bath. Dissolve the residue in 25 mL of methanol and proceed with this solution as directed under the Ultraviolet-visible Spectroscopy; it exhibits a maximum absorption at the wavelength of about 300 nm.

(2) Weigh 0.1 g of pulverized Toad Venom, add 5 mL of a solution of tartaric acid (1 in 100), warm for l0 minutes on a water bath, and filter. To 1 mL of the filtrate, slowly add 1 mL of 4-dimethylaminobenzaldehyde TS, heat for l0 minutes on a water bath, and add l0 mL of water; the resulting solution exhibits a blue color.

(3) Weigh 2 g of pulverized Toad Venom, add 10 mL of methanol, sonicate for 30 minutes to extract, filter, and use the filtrate as the test solution. Separately, weigh 1 mg of cinobufagin RS, dissolve in 1 mL of methanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL of the test solution and 5 μL of the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate using a mixture of cyclohexane, dichloromethane and acetone (4 : 3 : 3) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly dilute sulfuric acid TS on the plate and heat at 105 ℃ for 10 minutes; the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

Ash NMT 5.0%.

Acid-insoluble ash NMT 2.0%.

Assay Weigh accurately about 0.5 g of pulverized Toad Venom, previously dried in a desiccator (silica gel) for 24 hours, add 50 mL of methanol, heat after connecting with a reflux condenser on a water bath for 1 hour, cool, and filter. Wash the residue with 30 mL of methanol and combine the solution used for washing with the filtrate. To this solution, add methanol to make exactly l00 mL. Pipet 10 mL of this solution, add exactly 5 mL of the internal standard solution, add methanol to make exactly 25 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of bufalin RS (previously dried in a desiccator with silica gel for 24 hours), about 20 mg of cinobufagin RS (previously dried in a desiccator with silica gel for 24 hours), and about 20 mg of redibufogenin RS (previously dried in a desiccator with silica gel for 24 hours), and dissolve each in methanol to make exactly 100 mL. Pipet 10 mL of this solution, proceed in the same manner as in the preparation of the test solution, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions. Determine the peak area ratios, Q_{TB} and Q_{SB}, of bufalin to the internal standard, the peak area ratios, Q_{TC} and Q_{SC}, of cinobufagin to the internal standard and the peak area ratios, Q_{TR} and Q_{SR}, of redibufogenin to the internal standard from each solution, and then calculate the amounts of bufalin, cinobufagin and redibufogenin according to the following equations, of which sum total is the amount of bufosteroid.

$$\text{Amount (mg) of bufalin } (C_{24}H_{34}O_4) = \text{Amount (mg) of bufalin RS} \times \frac{Q_{TB}}{Q_{SB}}$$

$$\text{Amount (mg) of cinobufagin } (C_{26}H_{34}O_6) = \text{Amount (mg) of cinobufagin RS} \times \frac{Q_{TC}}{Q_{SC}}$$

$$\text{Amount (mg) of redibufogenin } (C_{24}H_{32}O_4) = \text{Amount (mg) of redibufogenin RS} \times \frac{Q_{TR}}{Q_{SR}}$$

Internal standard solution—A solution of indometacin in methanol (1 in 4000).

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 300 nm)

Column: A stainless steel column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Column temperature: A constant temperature of about 40 ℃

Mobile phase: A mixture of dilute phosphoric acid (1 in 1000) and acetonitrile (11 : 9)

Flow rate: Adjust the flow rate so that the retention time of the internal standard is 16 to 19 minutes.

Selection of column: Proceed with 10 μL of the standard solution according to the above conditions. At this time, use a column from which bufalin, cinobufagin, resibufogenin and the internal standard are eluted in this order with the peaks being fully separated from each other.

Packaging and storage Preserve in well-closed containers.

Tribulus Fruit
질려자(蒺藜子)

Tribuli Fructus

Tribulus Fruit is the ripe fruit of *Tribulus terrestris* Linné (Zygophyllaceae).

Description Tribulus Fruit is the fruit, five-pointed star-shaped, with five mericarps arranged radially and 7 to 12 mm in diameter. Each mericarp is ax-shaped, 3 to 6 mm in length. The dorsal part occurs as a yellowish green color and is protrudent, with a longitudinal edge and several small spines. The spines are in symmetric pairs of longer and shorter spines. The two lateral sides are coarse and reticulated, exhibiting a grayish white color. The texture is hard.
Under a microscope, the transverse section of Tribulus Fruit reveals several triangular mericarps connected by the pericarp. The pericarp is composed of the single-layered epidermis, the mesocarp is composed of the parenchyma and the sclerenchyma layer, and the endocarp is composed of several layers of fiber cells. A single layer of cells between the mesocarp and the endocarp contain solitary crystals of calcium oxalate. The vascular bundles are thin, small and scattered throughout. Among the three edges of the mericarp, the two outside-facing edges contain large, conical fiber bundles, below which groups of the stone cells are present. The seed coat consists of a single layer of cells in a tight arrangement. The parenchyma cells of the cotyledon contain oil droplets. It occurs as nearly odorless and has a mild taste at first, followed by a bitter taste.

Identification Weigh 0.2 g of pulverized Tribulus Fruit, add 3 mL of acetic anhydride, warm on a water bath for 2 minutes, and filter. To 1 mL of the filtrate, add carefully 1 mL of sulfuric acid; a reddish brown color appears at the zone of contact, and the upper layer exhibits a bluish purple to green color.

Purity (1) ***Foreign matter***—(i) Fruit stalk: The amount of fruit stalks contained in Tribulus Fruit is less than 4.0%.
(ii) Other foreign matters: The amount of foreign matters other than fruit stalks contained in Tribulus Fruit is less than 1.0%.
(2) ***Heavy metals***—(i) Lead: NMT 5 ppm
(ii) Arsenic: NMT 3 ppm
(iii) Mercury: NMT 0.2 ppm
(iv) Cadmium: NMT 0.3 ppm
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 9.0%.

Ash NMT 13.0%.

Acid-insoluble ash NMT 2.5%.

Extract content ***Dilute ethanol-soluble extract***—NLT 11.0%.

Packaging and storage Preserve in well-closed containers.

Trichosanthes Root
괄루근(栝樓根)

Trichosanthis Radix

Trichosanthes Root is the root of *Trichosanthes kirilowii* Maximowicz or *Trichosanthes rosthornii* Harms (Cucurbitaceae), from which the cortex has been removed.

Description Trichosanthes Root is irregular cylindrical, fusiform or plate-like masses, 8 - 16 cm in length and 1.5 - 5.5 cm in diameter. The external surface occurs as a yellowish white or pale yellowish brown color with longitudinal wrinkles and slightly concave, horizontally long lenticels, and some remains of chestnut-colored outer shells. The texture is solid. The cut surface occurs as a white or pale yellow color and very powdery. Under a magnifying glass, the transverse section reveals the wide medullary rays and yellowish brown spots or small holes formed by vessels.
It has a slight, characteristic odor and a slightly bitter taste.

Identification Weigh 2 g of pulverized Trichosanthes Root, add 20 mL of diluted ethanol (1 in 2), sonicate for 30 minutes to extract, filter, and use the filtrate as the test solution. Separately, dissolve 1 mg of L-citrulline RS in 1 mL of diluted ethanol (1 in 2) and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 2 μL of the test solution and 1 μL of the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of 1-butanol, water, ethanol and acetic acid (100) (8 : 3 : 2 : 2) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly ninhydrin TS on the plate and heat at 105 °C; the color and the R_f value of one spot among the several spots obtained from the test solution are the same as those of a spot from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): NMT 0.2 ppm.
(vi) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 4.0%.

Packaging and storage Preserve in well-closed containers.

Trichosanthes Seed
괄루인(栝樓仁)

Trichosanthis Semen

Trichosanthes Seed is the seed of ripe *Trichosanthes kirilowii* Maximowicz or *Trichosanthes rosthornii* Harms (Cucurbitaceae).

Description (1) ***Trichosanthes kirlowii*—**Trichosanthes Seed from *Trichosanthes kirilowii* is flat, elliptical, 12 - 15 mm in length, 6 - 10 mm in width and about 3.5 mm in thickness. The external surface occurs as a pale brown to deep brown color, flat, smooth, with dented scars surrounding the edge of the seed. The upper part is relatively pointy with a hilum, and the base is obtusely round or relatively narrow. The seed coat is tough and hard. The inner seed coat is membranous, exhibiting a grayish green color. Two yellowish white cotyledons are very oily.
Tricosanthes Seed has a characteristic odor and a slightly bitter taste.

(2) ***Trichosanthes rosthornii*—**Trichosanthes Seed from *Trichosanthes rosthornii* is a relatively large and flat seed, 15 - 19 mm in length, 8 - 10 mm in width and about 2.5 mm in thickness. The external surface occurs as a deep brown color, with distinctly dented scars surrounding the edge in a ring shape. This pattern is relatively wide. The upper part appears as if it has been cut.

Identification Weigh 0.1 g of pulverized Trichosanthes Seed, add 2 mL of acetic anhydride, heat for 2 minutes on a water bath while shaking to mix, and filter. Add gently 0.5 mL of sulfuric acid to the filtrate; the boundary between the two liquid layers exhibits a reddish brown to red color.

Purity (1) ***Foreign matter***—Trichosanthes Seed contains less than 1.0% of fragmented seed coats.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

(5) ***Mycotoxins***—Total aflatoxins (sum of aflatoxins B_1, B_2, G_1 and G_2): NMT 15.0 ppb (However, aflatoxin B_1 is NMT 10.0 ppb).

Ash NMT 4.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 3.0%.

Packaging and storage Preserve in well-closed containers.

Valerian Root and Rhizome
길초근(吉草根)

Valerianae Radix et Rhizoma

Valerian Root is the root and rhizome of *Valerianae faurei* Briquet or other congeneric allied plants of the same genus (Valerianaceae).

Description Valerian Root and Rhizome is the root and rhizome, usually with numerous fine, long roots on the short rhizome. The rhizome is obovoid, 1 - 2 cm in length and 1 - 3 mm in diameter. The upper part of the rhizome has buds and the remains of the stem, which may be flanked by thick and short stolons. The stolons are thick and short or thin and long, sometimes having extremely small, scaly leaves. The texture is hard and difficult to break. The root is nearly conical, 10 - 15 cm in length and 1 - 3 mm in diameter. The external surface has fine, longitudinal wrinkles and is easy to bend. Under a magnifying glass, the transverse section of the root reveals a thick, pale grayish brown cortex and a grayish brown stele.
It has a strong, characteristic odor and a slightly bitter taste.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.

Ash NMT 10.0%.

Acid-insoluble ash NMT 5.0%.

Essential oil content NLT 0.3 mL (50.0 g, 1 mL of silicon resin).

Packaging and storage Preserve in well-closed containers.

Vitex Fruit
만형자(蔓荊子)

Viticis Fructus

Vitex Fruit is the ripe fruit of *Vitex rotundifolia* Linné fil. or *Vitex trifolia* Linné (Verbenaceae).

Description Vitex Fruit is the fruit, spheroidal to flattened spheroidal and 4 mm to 6 mm in diameter. The external surface occurs as a grayish brown to blackish brown color, covered with grayish white frost-like hairs, bearing 4 longitudinal shallow furrows. The apex is slightly concave, with the old grayish-white calyx and the short fruit stalk at the base. The calyx is 1/3 to 2/3 length of the fruit, with 5 crenatures, two of which is relatively deep. The whole calyx is densely pubescent. The body is light, and the texture is tough, making it hard to break. The transverse section reveals 4 loculi, each with a white seed.
It has a characteristic aroma and a plain and slightly pungent taste.

Identification Weigh 0.5 g of pulverized Vitex Fruit, add 10 mL of ethanol, shake to mix, and filter. To 5 mL of the filtrate, add 0.1 g of magnesium and 0.3 mL of hydrochloric acid; the resulting solution exhibits a pale red to reddish purple color.

Purity (1) ***Foreign matter***—(i) Fruit stalks and leaves: Vitex Fruit contains less than 4.0% of fruit stalks and leaves.

(ii) Other foreign matters: Vitex Fruit contains less than 1.0% of foreign matters other than fruit stalks and leaves.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(4) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 12.0% (6 hours).

Ash NMT 9.0%.

Acid-insoluble ash NMT 3.5%.

Extract content ***Dilute ethanol-soluble extract***—NLT 8.0%.

Packaging and storage Preserve in well-closed containers.

Xanthium Fruit
창이자(蒼耳子)

Xanthii Fructus

Xanthium Fruit is the ripe fruit of *Xanthium strumarium* Linné (Compositae).

Description Xanthium Fruit is the fruit, fusiform or ovoid, 10 to 15 mm in length and 4 to 7 mm in diameter. The external surface occurs as a yellowish brown to yellowish green color and has spines all over, or the spines are removed. The apex has two relatively thick spines, separated or linked up, and the base has a scar of the fruit stalk. The texture is hard and rigid. The center of the transverse section shows two loculi, separated by a vertical septum, each having an achene. The achene is usually fusiform, relatively even at one side, and the apex of an achene has the single protruding style. The pericarp is thin, exhibiting a grayish black color, with longitudinal patterns. The seed coat is membranous, exhibiting a pale gray color, and there are two cotyledons, which are oily.
It has a characteristic odor and a slightly bitter taste.

Identification (1) Weigh 0.5 g of pulverized Xanthium Fruit, macerate with 10 mL of warm water, filter, and add 1 drop of Iron(III) chloride TS to the filtrate; the resulting solution exhibits a grayish green color.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): NMT 0.2 ppm.
(vi) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Loss on drying NMT 7.0%.

Ash NMT 7.0%.

Acid-insoluble ash NMT 1.0%.

Extract content ***Dilute ethanol-soluble extract***—NLT 10.0%.

Packaging and storage Preserve in well-closed containers.

Yukmijihwangtang Extract Granules
육미지황탕엑스 과립

Yukmijihwangtang Extract Granules contain NLT 1.4 mg of paeniflorin ($C_{23}H_{28}O_{11}$: 480.46) of Moutan root bark per dose (one sachet).

Method of preparation For a dose (one sachet)

Prepared Rehmannia root	2.00 g
Moutan root bark, Poria, Corni fructus, Dioscorea rhizome, Alismatis rhizome	1.00 g

Pulverize the above crude drugs into coarse powder, weigh each crude drug, put it into the extractor, add 8 to 10 times of purified water of the amount, extract for 2 to 3 hours at 80 ℃ to 100 ℃ and filter. Concentrate the filtrate at 60 ℃ in vacuum to give 1.57 g to 2.34 g of semiliquid extract, or concentrate by an appropriate method to give 0.82 g to 1.22 g of powdered extract. Prepare Yukmijihwangtang Extract Granules with the extract as directed under Granules.

Identification (1) ***Prepared Rehmannia root***—Pulverize Yukmijihwangtang Extract Granules and weigh an appropriate amount corresponding to 1 g of prepared rehmannia root. Add 100 mL of methanol and heat the mixture after connecting with a reflux condenser for 1 hour. After cooling, filter the mixture and evaporate to dryness. Dissolve the residue in water, transfer the resulting solution to a separatory funnel, and add 30 mL of ethyl acetate to extract. Collect the ethyl acetate layer and evaporate to dryness. Add 2 mL of ethanol to the residue to dissolve and use this solution as the test solution. Separately, weigh accurately about 1 g of pulverized Prepared Rehmannia Root and operate it in the same manner as the test solution. Use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel (with fluorescent indicator) for thin layer chromatography. Develop the plate with a mixture of chloroform and methanol (20 :1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray 2,4-dinitrophenylhydrazine TS evenly on the plate and examine it under ultraviolet light (main wavelength: 254 nm); one spot among the several spots obtained from the test solution shows the same color and R_f value as the spot obtained from the standard solution.
(2) ***Poria***—Pulverize Yukmijihwangtang Extract Granules and weigh an appropriate amount corresponding to 1 g of Poria. Add 10 mL of water and shake for 5 minutes to mix. Then, add 100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour, and filter. Concentrate the filtrate in vacuum to give 10 mL, and use this solution as the test solution. Separately, weigh accurately about 1 g of pulverized Poria, add

100 mL of methanol, heat the mixture after connecting with a reflux condenser for 1 hour and filter. Concentrate the filtrate in vacuum to give 10 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel (with fluorescent indicator) for thin layer chromatography. Develop the plate with a mixture of hexane and acetone (7 : 3) as a developing solvent to a distance of about 10 cm, and air-dry the plate. Spray *p*-anisaldehyde-sulfuric acid TS evenly to the plate and heat it at 105 ℃ for 10 minutes. Examine the plate under ultraviolet light (main wavelength: 254 nm); one spot among the several spots obtained from the test solution shows the same color and R_f value as the spot obtained from the standard solution.

(3) ***Corni fructus***—Pulverize Yukmijihwangtang Extract Granules and weigh an appropriate amount corresponding to 1 g of Corni fructus. Add 100 mL of methanol and shake well to mix. Then, filter the mixture and concentrate the filtrate by evaporation to give about 2 mL. Use this solution as the test solution. Separately, weigh 1 g of pulverized Corni fructus RMPM and operate it in the same way as the test solution. Use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a lower layer of a mixture of dichloromethane, methanol, and water (60 : 35 : 15) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray *p*-anisaldehyde-sulfuric acid TS evenly to the plate and heat at 105 ℃ for 10 minutes; one spot among the several spots obtained from the test solution shows the same color and R_f value as the spot obtained from the standard solution.

(4) ***Dioscorea rhizome***—Pulverize Yukmijihwangtang Extract Granules and weigh an appropriate amount corresponding to 1 g of Dioscorea rhizome. Add 50 mL of ethanol and 5 mL of acetic acid. Heat the mixture after connecting with a reflux condenser for 1 hour. After cooling, filter the mixture and evaporate the filtrate to dryness. Dissolve the residue in 2 mL of ethanol, and use this solution as the test solution. Separately, weigh accurately about 1 g of pulverized Dioscorea rhizome RMPM, operate it in the same manner as the test solution and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of cyclohexane and ethyl acetate (3 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray sulfuric acid TS for spraying evenly to the plate and heat it at 105 ℃ for 10 minutes; one spot among the several spots obtained from the test solution shows the same color and R_f value as the spot obtained from the standard solution.

(5) ***Alismatis rhizome***—Pulverize Yukmijihwangtang Extract Granules and weigh an appropriate amount corresponding to 1 g of Alismatis rhizome. Transfer it to a soxhlet extractor, add 100 mL of ether, and heat the mixture after connecting with a reflux condenser for 1 hour. After cooling, filter the mixture and concentrate the filtrate by evaporation to give 2 mL. Use this solution as the test solution. Separately, weigh accurately about 1 g of pulverized Alismatis Rhizome and operate it in the same manner as the test solution. Use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel (with fluorescent indicator) for thin layer chromatography. Develop the plate with a mixture of chloroform and acetone (1 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray *p*-anisaldehyde-sulfuric acid TS evenly to the plate and heat it at 105 ℃ for 10 minutes; one spot among the several spots obtained from the test solution shows the same color and R_f value as the spot obtained from the standard solution.

(6) ***Moutan root bark***—Proceed as directed under the Assay; the test solution shows a peak at the same retention time as the standard solution.

Purity (1) ***Heavy metals***—(i) Heavy metals: NMT 30 ppm.
(ii) Lead: NMT 5 ppm.
(iii) Arsenic: NMT 3 ppm.

Disintegration Meets the requirements.

Particle size distribution estimation by analytical sieving Meets the requirements.

Uniformity of dosage units (distribution) Meets the requirements.

Microbial limit Meets the requirements.

Assay (1) ***Paenioflorin of Moutan root bark***—Take NLT about 20 sachets of Yukmijihwangtang Extract Granules, weigh accurately and pulverize. Weigh accurately an amount equivalent to about 5 mg of paenioflorin, add 40 mL of water, extract by sonication for 30 minutes, and filter. Extract the filtrate with chloroform, remove the chloroform layer, add water to the aqueous layer to make 50 mL, and use this solution as the test solution. Separately, weigh accurately about 5 mg of paeoniflorin RS (previously dried in a silica gel desiccator for 24 hours), dissolve in water to make exactly 50 mL, and use this solution as the standard solution. Perform the test with 20 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas A_T and A_S of paeoniflorin, respectively.

$$\text{Amount (mg) of paenioflorin } (C_{23}H_{28}O_{11}) = \text{Amount (mg) of paenioflorin RS} \times \frac{A_T}{A_S}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 254 nm)

Column: A stainless steel column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Mobile phase: A mixture of water, acetonitrile, and acetic acid (86 : 14 : 1).

Flow rate: 1.0 mL/min

Packaging and storage Preserve in tight containers.

Zanthoxylum Peel
산초(山椒)

Zanthoxyli Pericarpium

Zanthoxylum Peel is the pericarps of the ripe fruit of

Zanthoxylum piperitum De Candolle, *Zanthoxylum schinifolium* Siebold et Zuccarini or *Zanthoxylum bungeanum* Maximowicz (Rutaceae)

Description (1) ***Zanthoxylum piperitum***—Zanthoxylum Peel from *Zanthoxylum piperitum* is the pericarps of the ripe fruit, consisting of 2 to 3 mericarps, each of which is flattened spheroidal, dehiscent in 2 pieces and about 5 mm in diameter. The external surface of the pericarp occurs as a dark yellowish red to dark red color, with numerous small, dented spots originated from oil sacs, and the inner surface occurs as a pale yellowish white color. Under a microscope, the transverse section of Zanthoxylum Peel from *Zanthoxylum piperitum* reveals the external epidermis and the adjoined unicellular layer containing reddish brown tannin, and the inside of the pericarp holds oil sacs about 500 μm in diameter. Vascular bundles consisting mainly of spiral vessels are sporadically dispersed, the endocarp consists of stone cell layers, and there are only a small number of epidermis cells inside.
Zanthoxylum Peel from *Zanthoxylum piperitum* has a characteristic odor and a pungent taste numbing the sensation on the tongue.

(2) ***Zanthoxylum schinifolium***—Zanthoxylum Peel from *Zanthoxylum schinifolium* is the pericarp, consisting of 2 to 3 small mericarps, which are apocarpous at the upper part and grouped on the fruit stalk. The mericarps are spherical, splitting along the ventral suture and 3 mm to 4 mm in diameter. The external surface occurs as a grayish green to dark green color, scattered with numerous oil dots and covered with reticulated and raised fine wrinkles. The inner surface is smooth, exhibiting an almost white color. The endocarp is commonly separated from the pericarp at the base. The remaining seeds are ovoid, 3 mm to 4 mm in length, 2 cm to 3 cm in diameter, with black and lustrous outer surface.
Zanthoxylum Peel from *Zanthoxylum schinifolium* has a characteristic odor and a slightly sweet and pungent taste.

(3) ***Zanthoxylum bungeanum***—Zanthoxylum Peel from *Zanthoxylum bungeanum* is the pericarp, consisting of mostly a single mericarp, and 4 mm to 5 mm in diameter. The external surface occurs as a reddish purple to reddish brown color, scattered with numerous wart-like oil dots, 0.5 mm to 1 mm in diameter, which appear translucent when observed against light. The inner surface exhibits a pale yellow color.
Zanthoxylum Peel from *Zanthoxylum bungeanum* has a strong and characteristic odor and a long-lasting pungent taste numbing the sensation on the tongue.

Identification Weigh 0.5 g of pulverized Zanthoxylum Peel, add 10 mL of dilute ethanol (7 in l0), shake to mix, filter, and use the filtrate as the test solution. With this solution, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL of the test solution on the thin-layer chromatographic plate made of silica gel (with a fluorescent agent) for thin layer chromatography. Next, develop the plate with a mixture of ethyl acetate, ethanol and water (8 : 2 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 365 nm); one spot showing a grayish red to red color appears at the R_f value of about 0.7.

Purity (1) ***Foreign matter***—(i) Seed: The amount of seeds contained in Zanthoxylum Peel is less than 20.0%.

(ii) Fruit stalk and twig: The amount of fruit stalks and twigs contained in Zanthoxylum Peel is less than 5.0%.

(iii) Other foreign matters: The amount of foreign matters other than seeds, fruit stalks and twigs contained in Zanthoxylum Peel is NMT 1.0%.

(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Dieldrin: NMT 1 ppm.
(iv) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(v) Aldrin: NMT 0.01 ppm.
(vi) Endrin: NMT 0.01 ppm.

(4) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 6.0%.

Acid-insoluble ash NMT 1.5%.

Essential oil content NLT 0.5 mL (30.0 g).

Packaging and storage Preserve in well-closed containers.

Zedoary
아출(莪朮)

Curcumae Rhizoma

Zedoary is the rhizome of *Curcuma phaeocaulis* Val., *Curcuma kwangsiensis* S. G. Lee et C. F. Liang or *Curcuma wenyujin* Y. H. Chen et C. Ling (Zingiberaceae), which is intact dried or dried after steaming.

Description (1) ***Curcuma phaeocaulis***—Zedoary of Curcuma phaeocaulis is the rhizome, ovoid, oblong ovoid, conical or oblong conical, 2 cm to 8 cm long, 15 mm to 40 mm in diameter. The outer surface occurs as grayish yellow to grayish brown, the upper part is often pointed and blunt, and the base is blunt and round. The upper annulated segment is protruded, circular, with slightly indented rootlet scars or rootlet remnants. Some have a series of dented shoot scars on both sides and nearly circular outer rhizome scars, sometimes with knife marks remaining. The body is heavy and the texture is firm. The cross-section is waxy, grayish brown to bluish brown, usually with grayish brown powder. The cortex and central cylinder are easily detached, and the inner layer has a dark brown ring pattern.
Zedoary of Curcuma phaeocaulis has a slight characteristic odor and a slightly bitter and pungent taste.

(2) ***Curcuma kwangsiensis***—Zedoary of Curcuma kwangsiensis is the rhizome, slightly protrude-annulated. The cut surface occurs as yellowish brown to brown, with mostly light yellow powder. The ring pattern of the inner layer occurs as yellowish white.

(3) ***Curcuma wenyujin***—Zedoary of Curcuma wenyujin is the rhizome and its cut surface occurs as yellowish brown to dark brown, with mostly pale yellow to yellowish brown powder.
Zedoary of Curcuma wenyujin has a slight characteristic odor.

Identification Weigh 1 g each of pulverized Zedoary and Zedoary RMPM, add 50 mL of petroleum ether, and sonicate for 1 hour. Filter the mixtures and evaporate the filtrates to dryness. Dissolve the residue in 2 mL of petroleum ether and use these solutions as the test solution and the Zedoary RMPM standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each

of the test solution and the Zedoary RMPM standard solution on the thin layer plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of petroleum ether and ethyl acetate (9 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly vanillin-sulfuric acid TS on the plate and heat at 105 °C for 10 minutes; the spots obtained from the test solution show the same color and R_f values as the spots obtained from the Zedoary RMPM standard solution, and one of the spots appears purple near the R_f value of about 0.7.

Purity (1) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.7 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(3) ***Sulfur dioxide***—NMT 30 ppm.

Ash NMT 7.0%.

Essential oil content NLT 0.5 mL (50.0 g, 1 mL of silicon resin).

Packaging and storage Preserve in well-closed containers.

Zizyphus Seed
산조인(酸棗仁)

Zizyphi Semen

Zizyphus Seed is the ripe seed of *Zizyphus jujuba* Miller var. *spinosa* Hu ex H. F. Chou (Rhamnaceae).

Description Zizyphus Seed is the seed, flattened round or flattened elliptical, 5 mm to 9 mm in length, 5 mm to 7 mm in width and 3 mm in thickness. The outer surface occurs as reddish purple or purple-brown, smooth, lustrous, sometimes with open patterns. One side is relatively even and the other side is slightly bumpy with a protruded longitudinal line in the center. There is a concave, linear hilum at one end and a small, protruded chalaza at the other. The testa is relatively flexible and encloses the grayish endosperm and pale yellow cotyledons.
It has a slightly oily odor and a weak taste.

Identification (1) Weigh 0.5 g of Zizyphus Seed, add 5 mL of ether, shake for 2 minutes to mix, and filter. Evaporate the filtrate, add 0.5 mL of acetic anhydride to the residue and add 1 drop of sulfuric acid; a pale red color is developed at first and slowly changes to reddish purple.

(2) Weigh 1 g of pulverized Zizyphus Seed, add 30 mL of methanol, heat the mixture after connecting with a reflux condenser on a water bath for 1 hour, filter and evaporate to dryness. Dissolve the residue in 0.5 mL of methanol and use this solution as the test solution. Separately, weigh 10 mg of jujuboside A RS, dissolve in 10 mL of methanol, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the standard solution on a thin-layer chromatographic plate of silica gel for thin-layer chromatography. Next, develop the plate with water-saturated butanol as a developing solvent to a distance of about 10 cm, and air-dry the plate. Spray vanillin-sulfuric acid-ethanol TS evenly on the plate and heat at 105 ℃ for 10 minutes; one spot among the several spots obtained from the test solution show the same color and the same R_f value as the spot obtained from the standard solution.

Purity (1) ***Foreign matter***—Zizyphus Seed contains NMT 3% of the endocarp and other foreign matter.
(2) ***Heavy metals***—(i) Lead: NMT 5 ppm.
(ii) Arsenic: NMT 3 ppm.
(iii) Mercury: NMT 0.2 ppm.
(iv) Cadmium: NMT 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): NMT 0.1 ppm.
(ii) Dieldrin: NMT 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): NMT 0.2 ppm.
(iv) Aldrin: NMT 0.01 ppm.
(v) Endrin: NMT 0.01 ppm.
(4) ***Sulfur dioxide***—NMT 30 ppm.
(5) ***Mycotoxins***—Total aflatoxin (sum of aflatoxin B_1, B_2, G_1 and G_2): NMT 15.0 ppb (aflatoxin B_1 is NMT 10.0 ppb).

Ash NMT 7.0%.

Packaging and storage Preserve in well-closed containers.

2) Biological Preparations, etc.

Adsorbed Diphtheria-Tetanus Combined Vaccine for Adult
성인용 흡착 디프테리아 및 파상풍 혼합백신

Adsorbed Diphtheria-Tetanus Combined Vaccine for Adult is a liquid preparation obtained by adding aluminum salt to adsorb and mix detoxified toxoid solutions of diphtheria toxin and tetanus toxin.

Adsorbed Diphtheria-Tetanus Combined Vaccine for Adult meets the requirements in the section of the Adsorbed Diphtheria-Tetanus Combined Vaccine for Adults under the Specifications and Test Methods for Biologicals, specified separately.

Adsorbed Diphtheria-Tetanus-Acellular Pertussis Combined Vaccine
흡착 디프테리아, 파상풍 및 정제 백일해 혼합백신

Adsorbed Diphtheria-Tetanus-Acellular Pertussis Combined Vaccine is a liquid preparation obtained by adding aluminum salt to adsorb and mix detoxified toxoid solutions of diphtheria toxin and tetanus toxin and a solution containing purified, inactivated pertussis antigens.

Adsorbed Diphtheria-Tetanus-Acellular Pertussis Combined Vaccine conforms to the requirements of Adsorbed Diphtheria-Tetanus-Acellular Pertussis Combined Vaccine in the Specifications and Test Methods for Biologicals, specified separately.

Adsorbed Diphtheria-Tetanus-Acellular Pertussis-Enhanced Inactivated Poliomyelitis Combined Vaccine
흡착 디프테리아, 파상풍, 정제 백일해 및 개량 불활화 폴리오 혼합백신

Adsorbed Diphtheria-Tetanus-Acellular Pertussis-Enhanced Inactivated Poliomyelitis Combined Vaccine is a liquid preparation prepared by adsorbing and mixing aluminum salts in toxoid liquor in which diphtheria toxin and tetanus toxin have been inactivated, purified pertussis antigen has been isolated, purified, and inactivated, and polioviruses of types 1, 2, and 3 have been inactivated using appropriate methods.

Adsorbed Diphtheria-Tetanus-Acellular Pertussis-Enhanced Inactivated Poliomyelitis Combined Vaccine meets the requirements in the section of the Adsorbed Diphtheria-Tetanus-Acellular Pertussis-Enhanced Inactivated Poliomyelitis Combined Vaccine under the Specifications and Test Methods for Biologicals, specified separately.

Clostridium Botulinum Toxin Type A
클로스트리디움 보툴리눔 독소 A형

Clostridium botulinum Toxin Type A is a freeze-dried preparation containing *Clostridium botulinum* toxin type A. It becomes a liquid preparation on the addition of a solvent.

Clostridium botulinum Toxin Type A meets the requirements of *Clostridium botulinum* Toxin Type A under the Specifications and Test Methods for Biologicals, specified separately

Enhanced Inactivated Poliomyelitis Vaccine
개량 불활화 폴리오 백신

Enhanced Inactivated Poliomyelitis Vaccine is a liquid preparation. obtained by culturing poliovirus of type I, II and III followed by inactivation, mixing and addition of a preservative.

Enhanced Inactivated Poliomyelitis Vaccine meets the requirements in the section of the Enhanced Inactivated Poliomyelitis Vaccine under the Specifications and Test Methods for Biologicals, specified separately.

Erythropoietin Concentrated Solution (rDNA)
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Erythropoietin Concentrated Solution (rDNA) is a solution containing recombinant glycoproteins which are indistinguishable from naturally occurring human erythropoietin (urinary erythropoietin) in terms of amino acid sequence (165 amino acids) and glycosylation pattern, at a concentration of 0.5 mg/mL to 10 mg/mL. It has an effect of increasing the number of reticulocytes.

Erythropoietin Concentrated Solution (rDNA) has a potency of NLT 100000 IU per mg of protein determined using the conditions described under Assay and in the test for protein.

Description Erythropoietin Concentrated Solution (rDNA) occurs as a clear or slightly turbid, colorless liquid.

Identification (1) ***Biological identification***—Proceed as directed under the Assay of potency; an increase in the red blood cell count is observed.

(2) ***Capillary electrophoresis***

Test solution: Dilute Erythropoietin Concentrated Solution (rDNA) with water to adjust the concentration of protein to 1 mg/mL. Desalt 0.25 mL of this solution by passing it through a centrifuge cartridge with a molecular weight cut-off of NMT 10,000. Add 0.2 mL of water to the sample and repeat this

process twice. Determine the protein content as directed under the Assay, dilute the desalted solution with water to adjust the concentration of protein to 1 mg/mL, and use this solution as the test solution.

Standard solution: Dilute erythropoietin RS with water to adjust the concentration of protein to 1.0 mg/mL. Proceed with desalting in the same manner as in the preparation of the test solution, and use this solution as the standard solution.

Proceed with the test solution and the standard solution as directed under the Capillary electrophoresis according to the following conditions, and compare the electropherograms obtained from the test solution and the standard solution; peaks of 8 isoforms are detected in the electropherogram obtained from the test solution in comparison with the peaks obtained from the standard solution. The contents of each isoform are within the following ranges.

Isoform	Content (%)
1	0 - 15
2	0 - 15
3	1 - 20
4	10 - 35
5	15 - 40
6	10 - 35
7	5 - 25
8	0 - 15

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 214 nm)

Capillary: An uncoated silica capillary about 50 μm in internal diameter and about 100 cm in effective length

Column temperature: A constant temperature of about 35 °C

Keep the test solution and the standard solution at 4 °C during the analysis.

Concentrated capillary electrophoresis buffer solution: Dissolve 0.584 g of sodium chloride, 1.792 g of tricine and 0.820 g of anhydrous sodium acetate in water to make 100 mL.

1 mol/L putrescine solution: Dissolve 0.882 g of putrescine in water to make 10 mL.

Capillary electrophoresis buffer solution: Dissolve 21.0 g of urea in 25 mL of water at 30 °C. To a mixture of 5 mL of the concentrated capillary electrophoresis buffer solution and 125 μL of 1 mol/L putrescine solution, add water to make 50 mL. Adjust the pH to 5.55 with dilute acetic acid at 30 °C and filter through a membrane filter (pore size: 0.45 μm).

Step	Solution	Time (min)	Condition
Capillary equilibration	0.1 mol/L sodium hydroxide solution	60	Under pressure
	Capillary electrophoresis buffer solution	60	Under pressure
	Capillary electrophoresis buffer solution	720	20 kV
Between-run washing	Water	10	Under pressure
	0.1 mol/L sodium hydroxide solution	5	Under pressure
	Capillary electrophoresis buffer solution	10	Under pressure
Injection	Injection of the test solution and the standard solution	-	Under pressure or under vacuum
Separation	Capillary electrophoresis buffer solution	80	143 V/cm (15.4 kV)

System suitability

System performance: Each peak obtained from the standard solution is fully separated, and the height of the tallest peak is NLT 50 times the height of the baseline noise. If necessary, adjust the injection volume so that peaks of sufficient heights are obtained. In the eletropherogram of standard solution, peaks corresponding to isoforms 1 to 8 are observed.; Isoform 1 may not be visible, but isoform 8 is always detected. The height of the peak of isoform 6 is the tallest, and the resolution between the peaks of isoforms 5 and 6 is NLT 1.

System repeatability: Repeat the test NLT 3 times with the standard solution; the baseline is stable showing almost no fluctuations, and the relative standard deviation of the retention time of the peak corresponding to isoform 2 is less than 2%.

(3) ***Polyacrylamide gel electrophoresis and immunoblotting***

(a) ***Polyacrylamide gel electrophoresis***

Dilution buffer solution: Weigh 1.89 g of Tris, 5.0 g of sodium dodecyl sulfate and 50 mg of bromophenol blue, dissolve in water, add 25.0 mL of glycerol, and add more water to make 100 mL. Adjust the pH of this solution to 6.8 with hydrochloric acid and add water to make 125 mL.

Coomassie Brilliant Blue staining solution: Dissolve 1.25 g of Coomassie Brilliant Blue R-250 in 1 L of a mixture of water, anhydrous methanol and acetic acid (100) (5 : 4 : 1).

Test solution (a): Dilute Erythropoietin Concentrated Solution (rDNA) with water to adjust the concentration of protein to 1.0 mg/mL. Mix equal volumes of this solution and the dilution buffer solution.

Test solution (b): Dilute Erythropoietin Concentrated Solution (rDNA) with water to adjust the concentration of protein to 0.1 mg/mL. Mix equal volumes of this solution and the dilution buffer solution.

Standard solution (a): Dissolve erythropoietin RS in water to adjust the concentration of protein to 1.0 mg/mL. Mix equal volumes of this solution and the dilution buffer solution.

Standard solution (b): Dissolve erythropoietin RS in water to adjust the concentration of protein to 0.1 mg/mL. Mix equal volumes of this solution and the dilution buffer solution.

Standard solution (c): Use a solution of molecular weight markers suitable for calibrating sodium dodecyl sulfate-polyacrylamide gel electrophoresis in the molecular weight range of 10000 to 70000.

Standard solution (d): Use a solution of molecular weight markers suitable for calibrating sodium dodecyl sulfate-polyacrylamide gel electrophoresis and for blotting on the membrane in the molecular weight range of 10000 to 70000.

Boil the test solutions and the standard solutions on a water bath for 2 minutes. Load 20 μL of each samples in the order of standard solution (c), standard solution (a), test solution (a), standard solution (b), test solution (b) and standard solution (d) to the wells of a 12% acrylamide gel, 0.75 mm in thickness and about 16 cm^2 in area. Put an empty well between the test solution (a) and the standard solution (b). After the electrophoresis is completed, cut the empty well between the test solution (a) and the standard solution (b) to make two gel pieces. Stain the gel containing the standard solution (c), the standard solution (a) and the test solution (a) with the Coomassie Brilliant Blue staining

solution; the test solution (a) shows a single diffuse band, of which position and intensity are the same as those of the band from the standard solution (a).

(b) ***Immunoblotting***

After the polyacrylamide gel electrophoresis is completed, transfer the gel containing the standard solution (b), the test solution (b) and the standard solution (d) onto a membrane suitable for protein immobilization and start the electrotransfer. After the electrotransfer is completed, incubate the membrane in a blocking solution containing 50 g/L of dry skim milk or 10 vol% bovine serum albumin for 1 to 2 hours, while shaking the container. Incubate the membrane in a solution containing the anti-erythropoietin antibodies diluted with the same blocking solution for 1 to 14 hours, while shaking the container. Detect the erythropoietin-bound antibodies using secondary antibodies labeled with an enzyme, such as alkaline phosphatase, or with a radioactive material; the test solution (b) shows a single diffuse band, of which position and intensity are the same as those of the band from the standard solution (b).

System suitability

System performance: The molecular weight markers of the standard solution (d) are resolved into discrete bands having a linear relationship between the distance migrated and the logarithmic value of the molecular weight.

(4) ***Peptide map***

Test solution: Dilute Erythropoietin Concentrated Solution (rDNA) with tris-acetic acid buffer solution (pH 8.5) to adjust the concentration of protein to 1.0 mg/mL, Equilibrate this solution with tris-acetate buffer solution (pH 8.5) using a suitable procedure, such as dialysis and membrane filtration, and then transfer the equilibrated solution to a polypropylene test tube. To 0.25 mL of this solution, add 5 μL of a freshly prepared 1 mg/mL trypsin solution. Cap the tube and incubate at 37 °C for 18 hours, and then stop the reaction immediately by freezing.

Standard solution: Dissolve erythropoietin RS in water to adjust the concentration of protein to 1.0 mg/mL. Proceed in the same manner as in the preparation of the test solution.

Perform the test with 50 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions; the profiles of the chromatograms obtained from the test solution and the standard solution are the same.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 214 nm)

Column: A stainless steel tube about 4.6 mm in internal diameter and about 25 cm in length, packed with end-blocked butylsilyl silica gel for liquid chromatography (about 5 μm to10 μm in particle diameter).

Mobile phase: Control the mobile phases A and B stepwise as directed in the following table or via the concentration gradient.

Mobile phase A: A mixture of trifluoroacetic acid and water (999.4 : 0.6)

Mobile phase B: A mixture of acetonitrile, water and trifluoroacetic acid (899.4 : 100 : 0.6)

Time (min)	Flow rate (mL/min)	Mobile phase A (vol%)	Mobile phase B (vol%)	Chromatographic conditions
0 - 10	0.75	100	0	Constant concentration
10 - 125	0.75	100 → 39	0 → 61	Linear concentration gradient
125 - 135	1.25	39 → 17	61 → 83	Linear concentration gradient
135 - 145	1.25	17 → 0	83 → 100	Linear concentration gradient
145 - 150	1.25	100	0	Constant concentration

Equilibration of column: Equilibrate the column by flowing the mobile phase A for NLT 15 minutes.

System suitability

System performance: The chromatograms obtained from the test solution and the standard solution are qualitatively similar.

(5) ***N-terminal sequence analysis***

To 50 μg of Erythropoietin Concentrated Solution (rDNA), add 1 mL of diluted trifluoroacetic acid (1 in 1000). Desalt this solution by passing it through a C_{18} reverse-phase cartridge, previously equilibrated with diluted trifluoroacetic acid (1 in 1000). Wash the cartridge successively with diluted trifluoroacetic acid (1 in 1000), a mixture of diluted trifluoroacetic acid (1 in 1000) and acetonitrile (90 : 10) and a mixture of diluted trifluoroacetic acid (1 in 1000) and acetonitrile (50 : 50), and then discard the flow-through. Elute the cartridge with a mixture of water and acetonitrile (50 : 50), collect the eluent, and lyophilize the collected eluent.

Dissolve the lyophilized sample in diluted trifluoroacetic acid (1 in 1000) and run 15 cycles using an amino acid sequence analyzer. Use the reaction conditions for proline when running the second and third cycles.

Identify the amino acids released at each cycle as directed under the Reverse-phase liquid chromatography; the order of amino acids is Ala-Pro-Pro-Arg-Leu-Ile-(unsequenced amino acid)-Asp-Ser-Arg-Val-Leu-Glu-Arg-Tyr. Use the amino acids RS to set the analytical conditions for the reverse-phase liquid chromatography so that all amino acids are separated.

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Purity (1) ***Dimer and the related substance of higher molecular weight***

Test solution: Dilute Erythropoietin Concentrated Solution (rDNA) with the mobile phase to adjust the concentration of protein to 0.2 mg/mL.

Resolution test solution—Dilute the system suitability RS for the size-exclusion chromatography of erythropoietin with the mobile phase to adjust the concentration to 0.2 mg/mL.

Standard solutions—To 0.02 mL of the resolution test solution, add 1 mL of the mobile phase.

Perform the test with 100 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions; the sum of the peak areas of any peaks detected before the major peak obtained from the test solution is not greater than the peak area of the major peak obtained from the standard solution.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 214 nm)

Column: A stainless steel column about 7.5 mm in internal diameter and about 60 cm in length, packed with hydrophilic silica gel for liquid chromatography, which is suitable for fractionation of globular proteins with the molecular weight range of 20000 to 200000.

Mobile phase: Dissolve 1.15 g of anhydrous sodium monohydrogen phosphate, 0.2 g of potassium dihydrogen phosphate and 23.4 g of sodium chloride in water to make 1000 mL. If necessary, adjust the pH to 7.4.

Flow rate: 0.5 mL/min

Time span of measurement: About 2 times the retention time of erythropoietin monomer

System suitability

System performance: The area of the major peak obtained from standard solution is 1.5% to 2.5% of the area of the major peak obtained from the test solution.

Resolution: The resolution between the dimer and the monomer of erythropoietin is NLT 1.5 in the chromatogram obtained from the resolution test solution.

(2) ***Sialic acid***

Test solution (a): Dilute Erythropoietin Concentrated Solution (rDNA) with the mobile phase used in the 'Dimer and the related substance of higher molecular weight' to adjust the concentration of protein to 0.3 mg/mL.

Test solution (b): To 0.5 mL of test solution (a), add 0.5 mL of the mobile phase used in the 'Dimer and the related substance of higher molecular weight'.

Standard solution (a): Dissolve a suitable amount of *N*-acetylneuraminic acid in water to adjust the concentration to 0.1 mg/mL.

Standard solution (b): To 0.8 mL of the standard solution (a), add 0.2 mL of water.

Standard solution (c): To 0.6 mL of the standard solution (a), add 0.4 mL of water.

Standard solution (d): To 0.4 mL of the standard solution (a), add 0.6 mL of water.

Standard solution (e): To 0.2 mL of the standard solution (a), add 0.8 mL of water.

Standard solution (f): Use water.

Prepare three sets each of the standard solution and the test solution. Transfer 100 μL each of the test solutions and the standard solutions into 10 mL-glass-stoppered test tubes and add 1.0 mL of resorcinol to each test tube. Stopper the tubes and heat at 100 °C for 30 minutes. Cool on ice and add 2.0 mL of a mixture of butyl acetate and butanol (48 : 12) to each tube. Mix vigorously and allow to stand until the solution separates into two layers. After confirming that the supernatant is completely clear, carefully take the supernatant to avoid getting any liquid from the lower layer. Determine the absorbances of the test solutions and the standard solutions at 580 nm as directed under the Ultraviolet-visible Spectroscopy. Then, calculate the contents of sialic acid in the test solutions (a) and (b) and the mean values. Calculate the relative molar ratio of sialic acid to erythropoietin assuming that the molecular weights of erythropoietin and *N*-acetylneuraminic acid are 30600 and 309, respectively; it is NLT 10 moles of sialic acid per mole of erythropoietin.

System suitability

System performance: The resulting value obtained from the standard solution (a) is 1.5 to 3.3 times that obtained from the test solution (a).

System repeatability: Repeat the analysis of the standard solutions and the test solutions 3 times; the difference in the analysis results between the standard solutions and the test solutions is within 10% each time.

Bacterial endotoxins Less than 20 EU per volume of Erythropoietin Concentrated Solution (rDNA) equivalent to 100000 U of erythropoietin.

Assay (1) ***Protein content***—Dilute Erythropoietin Concentrated Solution (rDNA) with a solution prepared by dissolving 4 g of ammonium bicarbonate in 1000 mL of water, and use this solution as the test solution.

Determine the absorbance at 250 to 400 nm as directed under the Ultraviolet-visible Spectroscopy and make a correction for light scattering up to 400 nm; it exhibits a maximum between 276 nm and 280 nm. Calculate the concentration of erythropoietin using the specific absorbance; it is NLT 80% and NMT 120% of the labeled concentration.

$$\text{Content (mg/mL) of erythropoietin} = \frac{A_{280} \times D}{EC}$$

A_{280}: Absorbance at the wavelength of 280nm

D: Dilution factor

EC: 0.743 (extinction coefficient of erythropoietin)

(2) ***Potency***—The activity of Erythropoietin Concentrated Solution (rDNA) is expressed in International Units (IU) in comparison with that of erythropoietin RS.

Concentrated staining solution: Prepare a solution of thiazole orange, 2-fold concentrated than is suitable for counting reticulocytes.

Test solution (a): Dilute Erythropoietin Concentrated Solution (rDNA) with bovine serum albumin-saline solution to adjust the concentration to 80 IU/mL.

Test solution (b): Mix equal volumes of the test solution (a) and bovine serum albumin-saline solution.

Test solution (c): Mix equal volumes of the test solution (b) and bovine serum albumin saline solution.

Standard solution (a): Dilute erythropoietin RS with bovine serum albumin-saline solution to adjust the concentration to 80 IU/mL.

Standard solution (b): Mix equal volumes of the standard solution (a) and bovine serum albumin-saline solution.

Standard solution (c): Mix equal volumes of the standard solution (b) and bovine serum albumin-saline solution.

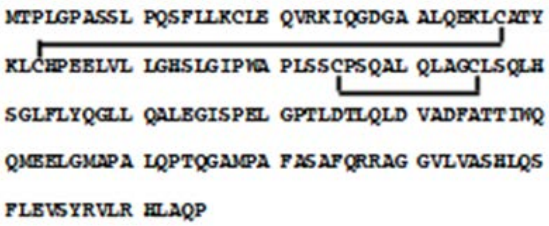
The exact concentrations of the test solutions and the standard solutions may be modified according to the response range of the animals tested.

At the beginning of the assay, randomly distribute mice of a suitable age and strain (for example, 8-week-old B6D2F1 mice), and assign 6 mice in each group. Inject each animal subcutaneously with 0.5 mL each of the test solution or the standard solutions (one kind of solution for each group). Collect blood samples from the animals 4 days after the injections.

Dilute the whole blood samples collected with a 500-fold volume of the buffer used to prepare the thiazole orange staining solution. Mix equal volumes of this solution and the concentrated staining solution. Stain the samples for 3 to 10 minutes, and then determine the reticulocyte count with a flow cytometer. Determine the percentage of reticulocytes with a biparametric histogram using the number of cells and red fluorescence (620 nm) as the two parameters. Calculate the potency by the parallel line assay method; the potency calculated is NLT 80% and NMT 125% of the labeled potency. The confidence interval (P = 0.95) of the calculated potency is NLT 64% and NMT 156% of the labeled potency.

Packaging and storage Preserve in hermetic containers (below -20 °C). Avoid repeated freezing and thawing.

Filgrastim Concentrated Solution (rDNA)
필그라스팀 농축액 (유전자재조합)



$C_{845}H_{1339}N_{223}O_{243}S_9$: 18799

Filgrastim solution for Injection (rDNA) is an aqueous solution of a protein in which a methionine is added to the *N*-terminus of the primary structure of human granulocyte colony-stimulating factor (G-CSF). Compared to natural products, Filgrastim concentrated solution (rDNA) is not glycosylated.

Human granulocyte colony-stimulating factor is produced and secreted by endothelial cells, monocytes, and other immune cells. This protein stimulates the differentiation and proliferation of leukocyte stem cells into mature granulocytes.

Filgrastim concentrated solution (rDNA) contains NLT 1.0×10^8 IU per mg of protein.

Description Filgrastim concentrated solution (rDNA) occurs as a colorless, transparent or slightly yellowish clear liquid.

Identification

(1) ***Potency*—**Meets the requirements when tested as directed under the Assay.

(2) ***Purity*—**Proceed with Filgrastim concentrated solution (rDNA) as directed under the charge variants of the Purity; the position of the main band obtained from the electropherogram of the test solution and the position of the main band obtained from the electropherogram of standard solution (a) are similar.

(3) ***Purity*—**Proceed with Filgrastim concentrated solution (rDNA) as directed under the high molecular weight proteins of the Purity; the retention time of the main peak obtained from the chromatogram of the test solution and the retention time of the main peak obtained from the chromatogram of the standard solution are similar.

(4) ***Purity*—**Proceed with Filgrastim concentrated solution (rDNA) as directed under the molecular-weight variants (reducing and non-reducing gel electrophoresis) of the Purity; the position of the main band obtained from the electropherogram of the test solution (a) and the position of the main band obtained from the electropherogram of standard solution (b) are similar.

(5) ***Peptide map***

Test solution: Take an appropriate amount of Filgrastim concentrated solution (rDNA) to contain 25 μg as protein and transfer it to a polypropylene tube. Add 25 μL of 0.1 mg/mL glutamyl endopeptidase and add 0.02 mol/L sodium phosphate buffer solution to make 100 μL. Cap the tube and allow to react at 37 °C for 17 hours, then stop the reaction at 2 to 8 °C.

Standard solution: Prepare the filgrastim RS at the same time as the test solution and in the same manner.

Perform the test with 10 μL each of the test solution and the standard solutions as directed under Liquid chromatography according to the following conditions; the chromatogram pattern obtained from the test solution is consistent with the chromatogram pattern obtained from the standard solution.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 215 nm)

Column: A stainless steel tube about 2.1 mm in internal diameter and about 10 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 60 °C

Mobile phase: Use mobile phases A and B to control a step or concentration gradient as follows.

Mobile phase A: A mixture of water, acetonitrile, and trifluoroacetic acid (950 : 50 : 0.5)

Mobile phase B: A mixture of water, acetonitrile, and trifluoroacetic acid (50 : 950 : 0.5)

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)	Chromatographic conditions
0 - 8	97 → 94	3 → 6	Linear concentration gradient
8 - 25	94 → 66	6 → 34	Linear concentration gradient
25 - 40	66 → 10	34 → 90	Linear concentration gradient
40 - 45	10	90	Constant concentration

Flow rate: 0.2 mL/min

Purity (1) ***High molecular weight proteins***

Test solution: Dissolve Filgrastim concentrated solution (rDNA) in acetic acid buffer solution to a protein concentration of 0.4 mg/mL. The acetic acid buffer solution used to dissolve Filgrastim solution for Injection (rDNA) is prepared by dissolving 4.1 g of sodium acetate in 400 mL of water, adjusting the pH to 4.0 with acetic acid, and then diluting with water to a final volume of 500 mL.

Standard solution: Dissolve the filgrastim RS in acetic acid buffer solution to a concentration of 0.4 mg/mL.

Reference solution for system suitability: Use the standard solution after stirring vigorously for 30 seconds.

Perform the test with 20 μL each of the test solution and the standard solution as directed under Liquid chromatography according to the following conditions; the sum of all peak areas detected before the main peak of the test solution is NMT 2% of the sum of the total peak area. The retention time of the filgrastim monomer peak is similar to that of the monomer peak obtained from the standard solution, and the relative retention times of the individual polymers are as follows: aggregate form about 0.60, filgrastim polymer 1 about 0.75, filgrastim polymer 2 about 0.80, and filgrastim dimer about 0.85.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 215 nm)

Column: A stainless steel column about 7.8 mm in internal diameter and about 30 cm in length, packed with hydrophilic silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 30 °C

Mobile phase: Dissolve 7.9 g of ammonium bicarbonate in 1 L of water, adjust the pH to 7.0 with phosphoric acid, and then add water to make 2 L.

Flow rate: 0.5 mL/min

System suitability

Set the retention time of the filgrastim monomer peak in the reference solution for system suitability to 17 to 20 minutes, and the resolution of the filgrastim dimer and monomer peaks is NLT 3.

(2) ***Molecular-weight variants (reducing and non-reducing gel electrophoresis)***

Non-reducing buffer solution: Dissolve 1.89 g of tris, 5.0 g of sodium dodecyl sulfate, 50 mg of bromophenol blue, and 25.0 mL of glycerin in 100 mL of water. Adjust the pH to 6.8 with hydrochloric acid and add water to make 125 mL. Mix equal parts water before use.

Reducing buffer solution: Prepare the buffer in the same way as the non-reducing buffer, but add 12.5 mL of 2-mercaptoethanol before adjusting the pH. Mix equal parts water before use.

Test solution (a): Dissolve Filgrastim concentrated solution (rDNA) in the buffer solution to a concentration of 100 μg/mL.

Test solution (b): Add 0.20 mL of the buffer solution to 0.20 mL of the test solution (a).

Test solution (c): Add the buffer solution to 0.20 mL of the test solution (b) and dilute to 1 mL.

Test solution (d): Add the buffer solution to 0.20 mL of the test solution (c) and dilute to 1 mL.

Test solution (e): Add 0.20 mL of the buffer solution to 0.20 mL of the test solution (d).

Standard solution (a): Use an appropriate molecular weight standard solution to calibrate sodium dodecyl sulfate-polyacrylamide gel electrophoresis in the molecular weight range of 14.4 to 94 kDa.

Standard solution (b): Dissolve the filgrastim RS in the buffer solution to a concentration of 100 μg/mL.

Heat the test solution and the standard solution on a water bath for 5 minutes. Then, inject standard solution (a), standard solution (b), test solution (a), test solution (b), test solution (c), test solution (d), and test solution (e) in that order into the wells of a 1-mm-thick 13% acrylamide gel, spot 20 μL of each, and then perform electrophoresis. When staining with silver dye, bands other than the main band in the electropherogram of test solution (a) are not more intense (2.0%) than the main band in the electropherogram of test solution (d).

System suitability

The standard solution (a) must be well separated by molecular weight. A band is visible in the electropherogram of the test solution (e). In the electropherogram obtained from the test solution (a) to the test solution (e), the intensity of the staining gradually decreases.

(3) ***Charge variants (gel electrophoresis)***

Test solution: Dilute Filgrastim concentrated solution (rDNA) with water to a concentration of 0.3 mg/mL.

Standard solution (a): Dilute the filgrastim RS with water to a concentration of 0.3 mg/mL.

Standard solution (b): Dilute the filgrastim RS with water to a concentration of 0.03 mg/mL.

Standard solution (c): Use a calibration solution with an isoelectric point of 2.5 to 6.5.

Perform the test with 20 μL each of the test solution and the standard solutions as directed under Gel electrophoresis according to the following conditions; no band other than the main band of the electropherogram obtained from the test solution is more intense (10%) than the main band of the electropherogram obtained from standard solution (b).

Operating conditions

pH gradient: 4.5 to 8.0

Catholyte: 1 mol/L sodium hydroxide solution

Anolyte: An aqueous solution of 0.04 mol/L glutamic acid dissolved in 0.0025% v/v phosphoric acid

Detection: Stain with Coomassie Brilliant Blue stain.

System suitability

Each band of the isoelectric point calibration solution (standard solution (c)) is well separated. The isoelectric point of the main band in the electropherogram obtained from standard solution (a) is 5.7 to 6.3.

(4) ***Related substances***

Test solution: Dilute Filgrastim concentrated solution (rDNA) with water to a concentration of 0.5 mg/mL.

Standard solution (a): Dilute the filgrastim RS with water to a concentration of 0.5 mg/mL.

Standard solution (b): Add 2.5 μL of 4.5 g/L hydrogen peroxide to 250 μL of standard solution (a), mix and allow the mixture to stand at 25 °C for 30 minutes, and then add 1.9 mg of L-methionine.

Standard solution (c): Add 0.25 mg of dithiothreitol to 250 μL of standard solution (a), mix, and allow the mixture to react for 60 minutes at 35 ± 2 °C.

Perform the test with 50 μL each of the test solution and the standard solutions as directed under Liquid chromatography according to the following conditions; the peak area of each related substance is NMT 1.0% of the sum of the total peak area. The sum of the peak areas of all related substances is NMT 2.0% of the sum of the total peak area. The retention time of the main peak of filgrastim is similar to that of the main peak of the standard solution, and the relative retention times of the related substances are as follows: oxidized form 1 about 0.84, oxidized form 2 about 0.98, and deamidated form about 1.04.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 215 nm)

Column: A stainless steel column about 4.6 mm in internal diameter and about 25 cm in length, packed with butylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 60 °C

Mobile phase: Use mobile phases A and B to control a step or concentration gradient as follows.

Mobile phase A: A mixture of water and trifluoroacetic acid (999 : 1)

Mobile phase B: A mixture of water, acetonitrile, and trifluoroacetic acid (99 : 900 : 1)

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)	Chromatographic conditions
0 - 30	60 → 20	40 → 80	Linear concentration gradient
30 - 35	20	80	Linear concentration gradient
35 - 45	20 → 60	80 → 40	Linear concentration gradient

Flow rate: 0.8 mL/min

System suitability (b)

The symmetry factor of the filgrastim peak in the standard solution (b) is NMT 1.8.

The peak-to-valley ratio in the standard solution (b) is NLT 2.0.

System suitability (c)

The resolution between the filgrastim and reduced filgrastim peaks in the standard solution (c) is NLT 1.5.

The symmetry factor of the filgrastim peak in standard solution (c) is NMT 1.8.

Bacterial endotoxins Filgrastim concentrated solution (rDNA) is NMT 2 IU per 1.0 mg of filgrastim.

Assay (1) ***Protein content*—**Determine the amount of filgrastim in the sample based on the labeled amount of the filgrastim RS using the chromatogram results of the test solution and the standard solution under the related substances of the purity test.

(2) ***Potency*—**The activity of Filgrastim solution for Injection (rDNA) is expressed in IU compared to International Filgrastim RS or Filgrastim Standard Solution corrected by International Units (IU).

The potency is determined by the following test method, where test conditions such as dilution concentration of the test solution and standard solution, number of cells, and culture time may vary. If validated, alternative assays such as intracellular ATP measurement with luciferase can be used.

Well-established cell lines that respond to filgrastim (e.g. M-NFS-60 cells (ATCC No. CRL-1838)) should be used. After diluting and culturing the test solution and standard solution appropriately, add tetrazolium salt solution to further incubate. At this time, filgrastim in the test solution and standard solution affects cell proliferation, and as cells proliferate, intracellular dehydrogenase also increases. The tetrazolium salt forms a colored formazan product by the dehydrogenase, and the amount of this formazan is measured spectrophotometrically.

Fill all wells of a 96-well microplate with 50 μL of the dilution medium. Add another 50 μL of the dilution medium to the blank test well. Add 50 μL of the test sample to each of the three wells (the test solution and the standard solution with a concentration of 800 IU/mL, and the twofold serially diluted solutions).

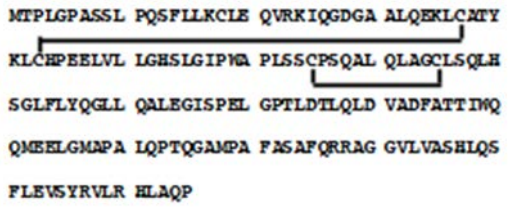
Prepare a suspension of M-NFS-60 cells to 7×10^5 cells/mL. Add 2-mercaptoethanol to a final concentration of 0.1 mmol/L immediately before use. Add 50 μL of the prepared cell suspension to each well. Ensure that the cells maintain a uniform suspension during addition.

Incubate the cell culture plate at 36.0 to 38.0 °C for 44 to 48 hours in an incubator maintained with a CO_2 concentration of 6 ± 1%. After incubation, add 20 μL of 5.0 g/L sterilized tetrazolium salt solution to each well and incubate for another 4 hours. Determine the amount of formazan formed at a wavelength of 490 nm using a microplate reader.

Calculate the potency of the test solution with a common statistical method using the parallel line test. The measured potency is NLT 80% and NMT 125% of the labeled potency. The confidence limit (P=0.95) is NLT 74% and NMT 136% of the measured potency.

Packaging and storage Preserve in hermetic containers (below -20 ℃). It should not be frozen and thawed repeatedly.

Filgrastim Solution for Injection (rDNA)
필그라스팀 주사액 (유전자재조합)



$C_{845}H_{1339}N_{223}O_{243}S_9$: 18799

Filgrastim solution for Injection (rDNA) is an aseptic protein preparation in which methionine is added to the *N*-terminus of the primary structure of human granulocyte colony-stimulating factor (G-CSF).

Filgrastim solution for Injection (rDNA) contains NLT 90.0% and NMT 110.0% of the labeled amount, and the potency is NLT 80% and NMT 125% of the labeled amount.

Description Filgrastim solution for Injection (rDNA) occurs as a colorless, transparent or slightly yellowish clear liquid.

Identification (1) ***Potency*—**Proceed with Filgrastim solution for Injection (rDNA) as directed under the Assay; biological activity should be confirmed.

(2) ***Purity*—**Proceed with Filgrastim solution for Injection (rDNA) as directed under the Related substances; The retention time and the shape of the main peak obtained from the chromatogram of the test solution should be similar to the chromatogram of the standard solution.

Purity (1) ***High molecular weight proteins***

Test solution: Dissolve Filgrastim solution for Injection (rDNA) in acetic acid buffer solution to a protein concentration of 0.2 mg/mL. The acetic acid buffer solution used to dissolve Filgrastim solution for Injection (rDNA) is prepared by dissolving 4.1 g of sodium acetate in 400 mL of water, adjusting the pH to 4.0 with acetic acid, and then diluting with water to a final volume of 500 mL.

Standard solution: Dissolve the filgrastim RS in acetic acid buffer solution to a concentration of 0.2 mg/mL.

Reference solution for system suitability: Use the standard solution after stirring vigorously for 30 seconds. Perform the test with 20 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions; among the peaks detected before the main peak of the test solution, the peaks excluding the dimer peaks are NMT 0.5%, and the sum of all peak areas is NMT 1.0% of the sum of total peak area. The retention time of the filgrastim monomer peak is similar to that of the monomer peak obtained from the standard solution, and the relative retention times of the individual polymers are as follows: aggregate form about 0.60, filgrastim polymer 1 about 0.75, filgrastim polymer 2 about 0.80, and filgrastim dimer about 0.85.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 215 nm)

Column: A stainless steel column about 7.8 mm in internal diameter and about 30 cm in length, packed with hydrophilic silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 30 °C.

Mobile phase: Dissolve 7.9 g of ammonium bicarbonate in 1 L of water, adjust the pH to 7.0 with phosphoric acid, and then add water to make 2 L.

Flow rate: 0.5 mL/min

System suitability

Set the retention time of the filgrastim monomer peak in the reference solution for system suitability to 17 to 20 minutes, and the resolution of the filgrastim dimer and monomer peaks is NLT 3.

(2) ***Charge variants (gel electrophoresis)***

Diluent: Prepare a solution of 10 mM leucine, 10 mM arginine, 7.5% glycerol.

Test solution: Dilute Filgrastim solution for Injection (rDNA) with the diluent to a concentration of 0.15 mg/mL.

Standard solution (a): Dilute the filgrastim RS with the diluent to a concentration of 0.15 mg/mL.

Standard solution (b): Dilute the filgrastim RS with the diluent to a concentration of 0.015 mg/mL.

Perform the test with 20 μL each of the test solution and the standard solutions as directed under the Gel electrophoresis according to the following conditions; no band other than the main band of the electropherogram obtained from the test solution is more intense (10%) than the main band of the electropherogram obtained from the standard solution (b).

Operating conditions

pH gradient: 5.0 - 7.0

Catholyte: A solution of 40 mM leucine, 40 mM arginine

Anolyte: 7 mM Phosphoric acid

Detection: Stain with Coomassie Brilliant Blue stain.

System suitability

The isoelectric point marker must be evenly distributed throughout the gel, and the isoelectric point of the main band in the electropherogram obtained from standard solution (a) is 5.7 to 6.3.

(3) ***Related substances***

Test solution: Dilute Filgrastim solution for Injection (rDNA) with water to a concentration of 0.2 mg/mL.

Standard solution (a): Dilute the filgrastim RS with water to a concentration of 0.2 mg/mL.

Standard solution (b): Add 2.5 μL of 4.5 g/L hydrogen peroxide to 250 μL of standard solution (a), mix and allow the mixture to stand at 25 ± 2 °C for 30 minutes, and then add 1.9 mg of L-methionine.

Standard solution (c): Add 0.25 mg of dithiothreitol to 250 μL of standard solution (a), mix, and allow the mixture to react for 60 minutes at 35 ± 2 °C.

Perform the test with 50 μL each of the test solution and the standard solutions as directed under the Liquid Chromatography according to the following conditions; the peak area of each related substance is NMT 3.0% of the sum of the total peak area. The sum of the peak areas of all related substances is NMT 6.5% of the sum of the total peak area. The retention time of the main peak of filgrastim (about 23 minutes) is similar to that of the main peak of the standard solution, and the relative retention times of the related substances are as follows: oxidized form 1 about 0.84, oxidized form 2 about 0.98, and reduced filgrastim about 1.04.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 215 nm)

Column: A stainless steel column about 4.6 mm in internal diameter and about 15 cm in length, packed with butylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 60 °C.

Mobile phase: Use mobile phases A and B to control stepwise or based on the concentration gradient as follows.

Mobile phase A: A mixture of water and trifluoroacetic acid (999 : 1)

Mobile phase B: A mixture of water, acetonitrile, and trifluoroacetic acid (99 : 900 : 1)

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)	Chromatographic conditions
0 - 30	60 → 20	40 → 80	Linear concentration gradient
30 - 35	20	80	Linear concentration gradient
35 - 45	20 → 60	80 → 40	Linear concentration gradient

Flow rate: 0.8 mL/min

System suitability

In stand Dissolve the filgrastim RS he symmetry factor of the filgrastim peak is NMT 1.8, and the peak-to-valley ratio is NLT 2.0.

In standard solution (c), the symmetry factor of filgrastim is NMT 1.8, and the resolution between the filgrastim and reduced filgrastim peaks is NLT 1.5.

Sterility Meets the requirements.

Bacterial endotoxins Filgrastim solution for Injection (rDNA) is NMT 10 IU per 1.0 mg of filgrastim.

Particulate contamination: Visible particles Meets the requirements.

Insoluble particulate matter in injections Meets the requirements.

Extractable volume of injections Meets the requirements.

Assay (1) ***Protein content***—Determine the amount of filgrastim in the sample based on the labeled amount of the filgrastim RS using the chromatogram results of the test solution and the standard solution under the Related substances of the purity test.

(2) ***Potency***—The activity of Filgrastim solution for Injection (rDNA) is expressed in IU compared to International Filgrastim RS or Filgrastim Standard Solution corrected by International Units (IU).

The potency is determined by the following test method, where test conditions such as dilution concentration of the test solution and standard solution, number of cells, and culture time may vary. If validated, alternative assays such as intracellular ATP measurement with luciferase can be used.

Well-established cell lines that respond to filgrastim (e.g. M-NFS-60 cells (ATCC No. CRL-1838)) should be used. After diluting and culturing the test solution and standard solution appropriately, add tetrazolium salt solution to further incubate. At this time, filgrastim in the test solution and standard solution affects cell proliferation, and as cells proliferate, intracellular dehydrogenase also increases. The tetrazolium salt forms a colored

formazan product by the dehydrogenase, and the amount of this formazan is measured spectrophotometrically.

Fill all wells of a 96-well microplate with 50 μL of the dilution medium. Add another 50 μL of the dilution medium to the blank test well. Add 50 μL of the test sample to each of the three wells (the test solution and the standard solution with a concentration of 800 IU/mL, and the twofold serially diluted solutions). Prepare a suspension of M-NFS-60 cells to 7×10^5 cells/mL. Add 2-mercaptoethanol to a final concentration of 0.1 mmol/L immediately before use. Add 50 μL of the prepared cell suspension to each well. Ensure that the cells maintain a uniform suspension during addition.

Incubate the cell culture plate at 36.0 to 38.0 °C for 44 to 48 hours in an incubator maintained with a CO_2 concentration of 6 ± 1%. After incubation, add 20 μL of 5.0 g/L sterilized tetrazolium salt solution to each well and incubate for another 4 hours. Determine the amount of formazan formed at a wavelength of 490 nm using a microplate reader.

Calculate the potency of the test solution with a common statistical method using the parallel line test. The measured potency is NLT 80% and NMT 125% of the labeled potency. The 95% confidence interval is NLT 74% and NMT 136% of the measured potency.

Packaging and storage Preserve in hermetic containers (2 to 8 °C).

Freeze-dried Agkistrodon (Salmusa) Antivenom (Equine)
건조 살무사 항독소

Freeze-Dried Agkistrodon (Salmusa) Antivenum (Equine) is a freeze-dried preparation that contains Agkistrodon (Salmusa) Antivenum in equine immunoglobulin. It becomes a liquid preparation on the addition of a solvent.

Freeze-dried Agkistrodon (Salmusa) Antivenom (Equine) meets the requirements in the section of the Freeze-dried Agkistrodon (Salmusa) Antivenom (Equine) under the Specifications and Test Methods for Biologicals, specified separately.

Freeze-dried BCG Vaccine for Intradermal Use
피내용 건조 비씨지 백신

Freeze-dried BCG Vaccine for Intradermal Use is a freeze-dried preparation containing live BCG (Bacillus of Calmette and Guerin). It becomes a turbid liquid preparation on the addition of a solvent.

Freeze-dried BCG Vaccine for Intradermal Use meets the requirements in the section of the Freeze-dried BCG Vaccine for Intradermal Use under the Specifications and Test Methods for Biologicals, specified separately.

Freeze-dried BCG Vaccine for Percutaneous Use
경피용 건조 비씨지 백신

Freeze-dried BCG Vaccine for Percutaneous Use is a freeze-dried preparation containing live BCG (Bacillus of Calmette and Guerin). It becomes a turbid liquid preparation on the addition of a solvent.

Freeze-dried BCG Vaccine for Percutaneous Use meets the requirements in the section of the Freeze-dried BCG Vaccine for Percutaneous under the Specifications and Test Methods for Biologicals, specified separately.

Freeze-dried Concentrated Human Antithrombin III
건조 농축 사람 항트롬빈 III

Freeze-dried Concentrated Human Antithrombin III is a freeze-dried preparation containing Human Antithrombin III of human serum. It becomes a liquid preparation on addition of solvent.

Freeze-dried Concentrated Human Antithrombin III meets the requirements in the section of the Freeze-dried Concentrated Human AntithrombinIII under the Specifications and Test Methods for Biologicals, specified separately.

Freeze-dried Concentrated Human Blood Coagulation Factor VIII
건조 농축 사람 혈액응고 제VIII인자

Freeze-dried Concentrated Human Blood Coagulation Factor VIII contains blood coagulation factor VIII of human serum and is a freeze-dried preparation for injection having low protein contents, except for coagulatory proteins. It becomes a liquid preparation on the addition of a solvent.

Freeze-dried Concentrated Human Blood Coagulation Factor VIII meets the requirements in the section of the Freeze-dried Concentrated Human Blood Coagulation Factor VIII under the Specifications and Test Methods for Biologicals, specified separately.

Freeze-dried Human Blood Coagulation Factor IX Complex
건조 사람 혈액응고 제IX인자 복합체

Freeze-dried Human Blood Coagulation Factor IX Complex is a freeze-dried preparation that contains blood coagulation factor IX complex in human plasma. It becomes a liquid preparation on the addition of a solvent.

Freeze-dried Human Blood Coagulation Factor IX Complex meets the requirements in the section of the Freeze-dried Human Blood Coagulation Factor IX complex under the Specifications and Test Methods for Biologicals, specified separately.

Freeze-dried Human Fibrinogen
건조 사람 피브리노겐

Freeze-dried Human Fibrinogen is a freeze-dried preparation that contains fibrinogen of human plasma. It becomes a liquid preparation on the addition of a solvent.

Freeze-dried Human Fibrinogen meets the requirements in the section of the Freeze-dried Human Fibrinogen under the Specifications and Test Methods for Biologicals, specified separately.

Freeze-dried Live Attenuated Measles-Mumps-Rubella Combined Vaccine
홍역, 유행성이하선염 및 풍진 혼합 생바이러스 백신

Freeze-Dried Live Attenuated Measles-Mumps-Rubella Combined Vaccine is a freeze-dried preparation containing live attenuated measles, mumps and rubella virus. It becomes a liquid preparation on the addition of a solvent.

Freeze-dried Live Attenuated Measles-Mumps-Rubella Combined Vaccine meets the requirements in the section of the Freeze-dried Live Attenuated Measles-Mumps-Rubella Combined Vaccine under the Specifications and Test Methods for Biologicals, specified separately.

Freeze-dried Smallpox Vaccine
건조 두창 백신

Freeze-dried Smallpox Vaccine is a freeze-dried preparation containing live vaccinia virus. It becomes a liquid preparation on the addition of a solvent.

Freeze-dried Smallpox Vaccine meets the requirements in the section of the Freeze-dried Smallpox Vaccine under the Specification and Test Methods for Biologicals, specified separately.

Haemophilus Influenzae Type b Conjugated to Diphtheria CRM197 Vaccine (Aluminum Adjuvanted)
헤모필루스 인플루엔자 비형·디프테리아 CRM197단백 접합 백신 (알루미늄 흡착)

Hemophilus influenzae type b Conjugated to Diphtheria CRM197 Vaccine (Aluminum Adjuvanted) is a liquid preparation containing *Haemophilus influenzae type b* oligosaccharides conjugated to a non-toxic variant of diphtheria (CRM197).

Hemophilus influenzae type b Conjugated to Diphtheria CRM197 Vaccine (Aluminum Adjuvanted) meets the requirements of *Hemophilus influenzae type b* Conjugated to Diphtheria CRM197 Vaccine (Aluminum Adjuvanted) in the Specifications and Test Methods for Biologicals, specified separately.

Haemophilus Influenzae Type b Conjugated to Meningococcal Protein Vaccine
헤모필루스 인플루엔자 비형.수막구균 외막단백 접합 백신

Haemophilus influenzae type b Conjugated to Meningococcal outer membrane Protein Vaccine is a liquid preparation containing *Haemophilus influenzae* type b polysaccharides and meningococcal outer membrane protein conjugate.

Haemophilus influenzae type b Conjugated to Meningococcal outer membrane Protein Vaccine meets the requirements in the section of the *Haemophilus influenzae* type b Conjugated to Meningococcal Protein Vaccine under the Specifications and Test Methods for Biologicals, specified separately.

Haemophilus Influenzae Type b Conjugated to Tetanus Toxoid Vaccine
헤모필루스 인플루엔자 비형.파상풍 톡소이드 접합 백신

Haemophilus influenzae type b Conjugated to Tetanus Toxoid Vaccine is a dried preparation containing Haemophilus influenzae type b polysaccharides conju-gated to tetanus toxoids. It becomes a liquid preparation on the addition of a solvent.

Haemophilus influenzae type b Conjugated to Tetanus Toxoid Vaccine meets the requirements of *Haemophilus influenzae* type b Conjugated to Tetanus Toxoid Vaccine in the Specifications and Test Methods for Biologicals, specified separately.

Hepatitis A Vaccine (Adsorbed, Inactivated)
A형 간염 백신

Hepatitis A Vaccine (Adsorbed, Inactivated) is a liquid preparation containing inactivated hepatitis A virus antigen.

Hepatitis A Vaccine (Adsorbed, Inactivated) meets the requirements in the section of the hepatitis A vaccine (adsorbed, inactivated) under the Specifications and Test Methods for Biologicals, specified separately.

Hepatitis A Vaccine (Virosome, Inactivated)
흡착 A형 간염-비로좀 백신

Hepatitis A Vaccine (Virosome, Inactivated) is a liquid preparation obtained by adsorbing inactivated hepatitis A virus (HAV) antigen to a virosome composed of influenza hemagglutinin and phospholipids.

Hepatitis A Vaccine (Virosome, Inactivated) meets the requirements in the section of the Hepatitis A Vaccine (Virosome, Inactivated) under the Specifications and Test Methods for Biologicals, specified separately.

Hepatitis B Vaccine (rDNA)
B형 간염 백신 (유전자재조합)

Hepatitis B Vaccine (rDNA) is a liquid preparation containing surface antigens of recombinant hepatitis B virus.

Hepatitis B Vaccine (rDNA) meets the requirements in the section of the Hepatitis B Vaccine (rDNA) under the Specifications and Test Methods for Biologicals, specified separately.

Human Hepatitis B Immunoglobulin
B형 간염 사람 면역글로불린

Human Hepatitis B Immunoglobulin is a liquid preparation containing hepatitis B antibody among human serum immunoglobulin G.

Human Hepatitis B Immunoglobulin meets the requirements in the section of the Human Hepatitis B Immunoglobulin under the Specifications and Test Methods for Biologicals, specified separately.

Human Hepatitis B Immunoglobulin for Intravenous Administration
정맥주사용 B형 간염 사람 면역글로불린

Human Hepatitis B Immunoglobulin for Intravenous Administration is a liquid preparation containing hepatitis B antibody among human serum immunoglobulin G.

Human Hepatitis B Immunoglobulin for Intravenous Administration meets the requirements in the section of the Human Hepatitis B Immunoglobulin for Intravenous Administration under the Specifications and Test Methods for Biologicals, specified separately.

Human Insulin (rDNA)
사람 인슐린 (유전자재조합)

$C_{257}H_{383}N_{65}O_{77}S_6$: 5808

Human insulin (rDNA) is a recombinant DNA protein of insulin produced in the human pancreas, is comprised of 2 peptides, and has hypoglycemic activity. Human insulin (rDNA) contains NLT 95.0% and NMT 105.0% of human insulin plus A21 desamino human insulin (previously dried), and 0.0347 mg of human insulin is equivalent to 1 IU in biological activity units.

Description Human insulin (rDNA) occurs as a white powder.

Identification (1) ***Assay***—Perform the test with human insulin (rDNA) as directed under the Assay; the retention time of the major peak from the chromatogram for the test solution matches the retention time of the major peak of the standard solution.

(2) ***Peptide mapping***—Take a suitable amount of human insulin (rDNA) and dissolve in 0.01 mol/L hydrochloric acid solution to a concentration of 2.0 mg/mL. Transfer 500 μL of this solution to a tube, and add 2.0 mL of pH 7.5 HEPES buffer solution. Prepare the pH 7.5 HEPES buffer solution by dissolving 2.38 g of 2-[4-(2-hydroxyethyl)piperazin-1-yl]ethanesulfonic acid in 90 mL of water, adjusting pH to 7.5 with sodium hydroxide, then adding water to make 100 mL. Add 400 μl of a 1 mg/ml solution of a proteolytic enzyme solution (e.g. *Staphylococcus aureus* strain V8 proteolytic enzyme XVII-B), then react for 6 hours at 25 ℃. Stop the reaction by adding 2.9 mL of pH 2.0 sulfuric acid buffer solution; use this solution as the test solution. Proceed with human insulin RS in the same manner as the test solution, and use this solution as the standard solution. Perform the test with 50 μL each of the test solution and the standard solution as directed under Liquid chromatography according to the following conditions; the shape of the chromatogram obtained from the test solution matches the chromatogram obtained from the standard solution.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 214 nm)

Column: A stainless steel tube about 4.6 mm in internal diameter and about 10 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (3 μm in particle diameter).

Column temperature: A constant temperature of about 40 °C

Mobile phase: Use mobile phases A and B to control stepwise or concentration gradient as follows.

Mobile phase A: A mixture of water, sulfuric acid buffer solution (pH 2.0) and acetonitrile (7 : 2 : 1)

Mobile phase B: A mixture of water, acetonitrile and sulfuric acid buffer solution (pH 2.0) (4 : 4 : 2)

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)	Chromatographic conditions
0 - 60	90 → 30	10 → 70	Linear concentration gradient
60 - 65	30 → 0	70 → 100	Linear concentration gradient
65 - 70	0	100	Constant concentration

Flow rate: 1 mL/min

Column equilibration: Run for NLT 15 minutes at initial conditions to reach an equilibrium state.

System suitability

The chromatogram of the test solution is qualitatively identical to the chromatogram of the standard solution. Peaks for peptide fragments I, II and III are identified in the chromatogram of the standard solution. The resolution between peptide fragments II and III is NLT 3.4, and the symmetry factor is NMT 1.5.

Purity (1) ***Macromolecular protein***—Dissolve Human Insulin (rDNA) in 0.01 mol/L hydrochloric acid solution to a concentration of 4.0 mg/mL; use this solution as the test solution. Dissolve reference standard comprising NLT 0.4% of macromolecular protein in 0.01 mol/L hydrochloric acid solution to a final concentration of about 4.0 mg/mL; use this solution as the system suitability solution. This solution may be prepared by letting human insulin powder stand for about 10 days at room temperature. Store at 2 to 8 ℃ after preparation, and use within 7 days.

Perform the test with 100 μL each of the test solution and the standard solution as directed under Liquid chromatography according to the following conditions; the sum area of all peaks detected prior to the major peak of the test solution is NMT 1.0%

of the sum of all peak areas. Disregard all peaks detected after the human insulin peak. Human insulin polymer is detected at 13 to 17 minutes, human insulin dimer is detected at about 17.5 minutes, and human insulin monomer is detected at about 20 minutes; salts are detected thereafter.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 276 nm)

Column: A stainless steel tube about 7.5 mm in internal diameter and 30 cm in length, packed with hydrophilic silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Mobile phase: A mixture of 1.0 g/L aqueous solution of L-arginine, acetonitrile and acetic acid (100) (65 : 20 : 15)

Flow rate: 0.5 mL/minute

Column equilibration: To perform analysis using a new column, equilibrate by repeatedly injecting human insulin solution comprising macromolecular protein prior to use. Equilibrate the column by injecting the system suitability solution a minimum of 3 times. Repeatable results are obtained from 2 consecutive injections.

System suitability

With the system suitability solution, verify that that peak to valley ratio of the human insulin dimer peaks and monomer peaks is NLT 2.0.

(2) ***Related substances***

Test solution: Dissolve 7.5 mg of Human Insulin (rDNA) in 2.0 mL of 0.01 mol/L hydrochloric acid solution. This solution must be used within 2 hours at room temperature, and within 12 hours at 2 ℃ to 8 ℃.

Standard solution (a): Dissolve human insulin RS in 0.01 mol/L hydrochloric acid RS to a concentration of 3.75 mg/mL. This standard solution must be used within 12 hours at room temperature, and within 48 hours at 2 ℃ to 8 ℃.

Standard solution (b): Add 0.01 mol/L hydrochloric acid solution to 1.0 mL of standard solution (a) to make 10 mL. This standard solution must be used within 12 hours at room temperature, and within 48 hours at 2 ℃ to 8 ℃.

Standard solution (c): Add 0.01 mol/L hydrochloric acid solution to 1.0 mL of standard solution (b) to make 10 mL. This standard solution must be used within 12 hours at room temperature, and within 48 hours at 2 ℃ to 8 ℃.

System suitability solution: Dissolve 1.5 mg of human insulin RS in 1.0 mL of 0.01 mol/L hydrochloric acid solution and let stand at room temperature for NLT 3 days to allow NLT 5% of A21-desamido human insulin to be formed.

Perform the test with 20 μL each of the test solution and the standard solution according to the following conditions as directed under Liquid chromatography; in the chromatogram from the test solution, the peak area of A21-desamido human insulin is NMT 2.0% of the sum of all peak areas, and the sum of all peak areas excluding the human insulin peak and the A21-desamido human insulin peak is NMT 2.0% of the sum of all peak areas.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 214 nm)

Column: A stainless steel tube about 4.6 mm in internal diameter and about 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 40 °C

Mobile phase: Control the mixing ratio of mobile phase A and mobile phase B stepwise or via the concentration gradient as follows.

Sulfuric acid solution: Dissolve 28.4 g of sodium sulfate in 1L of water, add 2.7 mL of 85% phosphoric acid, then adjust the pH to 2.3 with phosphoric acid or ethanol amine.

Mobile phase A: A mixture of sulfuric acid solution and acetonitrile (82 : 18)

Mobile phase B: A mixture of sulfuric acid solution and acetonitrile (50 : 50)

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)	Chromatographic conditions
0 - 60	81	19	Constant concentration
60 - 85	81 → 36	19 → 64	Linear concentration gradient
85 - 91	36	64	Constant concentration
91 - 92	36 → 81	64 → 19	Linear concentration gradient

Flow rate: 1 mL/minute

Column equilibration: Adjust the concentration of the mobile phase and the time of the initial efflux condition so that human insulin is detected at bout 31 minutes, and A21-desamido human insulin is detected immediately after human insulin.

System suitability

The value of the ratio of standard solution (b) and standard solution (a) multiplied by 10 is between 0.91 and 1.09, and the value of the ratio of standard solution (c) and standard solution (a) multiplied by 100 is between 0.7 and 1.3. With the system suitability solution, verify that the resolution between the human insulin peak and the A21-desamido human insulin peak is NLT 2.0, and that the symmetry factor of the human insulin peak is NMT 1.8.

(3) ***Zinc***—NMT 1.0% on the dried basis when tested as directed under Atomic Absorption Spectroscopy according to the following conditions.

Test solution: Dissolve 50.0 mg of Human Insulin (rDNA) in 0.01 mol/L hydrochloric acid solution to make 25.0 mL. As needed, dilute using 0.01 mol/L hydrochloric acid solution so that the concentration of zinc is 0.4 to 1.6 μg/mL.

Standard solution: Immediately before use, dilute 5 mg/mL zinc standard solution with 0.01 mol/L hydrochloric acid solution to zinc concentrations of 0.40, 0.80, 1.00, 1.20 and 1.60 μg per mL.

Operating conditions

Gas: Dissolved acetylene – Air

Lamp: Zinc hollow-cathode lamp.

Wavelength: 213.9 nm

(4) ***Human insulin precursor***

Human insulin precursor is detected using immunochemical testing. Place anti-human insulin precursor antibody in each well of a microplate and let stand overnight. Wash with phosphoric acid buffer solution, add a blocking solution comprising powdered milk or fetal bovine serum, and shake to mix on a shaker for 1 to 2 hours. Wash with phosphoric acid buffer solution, then place the test solution and the standard solutions at NLT 8 different concentrations in 4 wells each, and shake to mix on a shaker for 2 to 3 hours. Wash with phosphoric acid buffer solution, add anti-human insulin antibody, and shake to mix on a shaker to 1 to 2 hours. React the antibody bound to human insulin precursor with a secondary antibody marked with an enzyme of alkaline phosphatase, then add the substrate for color development. Create a standard calibration curve using the standard solution, then calculate the human insulin precursor in the test

solution using this standard calibration curve; the result is NMT 10 ppm.

Loss on drying NMT 10.0% (0.200 g, 105 ℃, 24 hours).

Residue on ignition NMT 2.5% (0.200 g on the dried basis).

Bacterial endotoxins NMT 10 EU per 1 mg of human insulin.

Assay

Test solution: Dissolve 40.0 mg of Human Insulin (rDNA) in 10 mL of 0.01 mol/L hydrochloric acid solution to a concentration of 4.0 mg/mL. Store this solution at 2 to 8 ℃ and use within 48 hours.

Standard solution (a): Dissolve human insulin RS in 0.01 mol/L hydrochloric acid RS to a concentration of 4.0 mg/mL.
Standard solution (b): Add 0.01 mol/L hydrochloric acid solution to 1.0 mL of standard solution (a) to make 10.0 mL.

Store the standard solutions and test solution at 2 to 10 ℃ and use within 48 hours.

System suitability solution: Dissolve 1.5 mg of human insulin RS in 1.0 mL of 0.01 mol/L hydrochloric acid solution and let stand at room temperature for NLT 3 days to allow NLT 5% of A21-desamido human insulin to be formed.

Perform the test with 20 μL each of the test solution and the standard solution according to the following conditions as directed under Liquid chromatography; use the peak areas of the test solution and the standard solution and the labeled amount of human insulin RS to calculate the total amount of human insulin and A21-desamido human insulin.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 214 nm)

Column: A stainless steel tube about 4.6 mm in internal diameter and about 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 40 °C

Mobile phase: A mixture of mobile phase concentrate B and mobile phase concentrate A (58 : 42)

Mobile phase concentrate A - Dissolve 28.4 g of anhydrous sodium sulfate in 1 L of water, add 2.7 mL of 85% phosphoric acid, and adjust the pH to 2.3 with phosphoric acid or ethanol amine.

Mobile phase concentrate B - Prepare with mobile phase A and acetonitrile at a volumetric ratio of 55 : 45. This reaction is endothermic; let stand until at least 20 ℃ before using.

Flow rate: 1 mL/min

System suitability

The major peak area of the chromatogram obtained from standard solution (a) is 10 ± 0.5 times the major peak area of the chromatogram obtained from standard solution (b). If this requirement is not met, adjust the injection volume to 10 to 20 μL to bring within the linearity range of the detector.

With the system suitability solution, confirm that the resolution between the human insulin peak and the A21-desamido human insulin peak is NLT 2.0, and that the symmetry factor of the human insulin peak is NMT 1.8.

Packaging and storage Preserve in light-resistant, tight containers (below -18 ℃).

Human Insulin Injection (rDNA)
사람 인슐린 주사액 (유전자재조합)

$C_{257}H_{383}N_{65}O_{77}S_6$: 5808

Human insulin injection (rDNA) is an aqueous injections, an aqueous suspension for injection, or a mixture of an aqueous solution and aqueous suspension for injection, and contains NLT 90.0% and NMT 110.0% of the labeled amount.

Description The aqueous injections occurs as a clear, colorless liquid. The aqueous suspension for injection or mixture of an aqueous solution and aqueous suspension for injection are a white suspension which separates into a white sediment and clear, colorless supernatant when let stand; this sediment is readily re-suspended by gently shaking.

pH Between 6.9 and 7.8.

Purity (1) ***Macromolecular protein***

Test solution: Add 4 μL of 6 mol/L hydrochloric acid solution per 1 mL of Human Insulin Injection (rDNA) to render the human insulin injection transparent. When sampling, agitate the material prior to sampling in order to obtain a homogeneous sample. If the human insulin injection does not turn transparent within 5 minutes, additionally add 4 μL of 6 mol/L hydrochloric acid solution per 1 mL. If the concentration of human insulin is 100 IU/mL or higher, dilute with 0.01 M hydrochloric acid to avoid overloading the column.

System suitability solution: Let 4 mg/mL human insulin solution stand for 10 days at room temperature, and add a suitable amount of 6 mol/L hydrochloric acid solution until transparent to contain NLT 0.4% macromolecular protein. Keep at 2 ~ 8 ℃ after preparation; use the aqueous injections within 30 hours, and use other preparations for injection within 7 days.

With 100 μL each of the test solution and the system suitability solution, perform the test as directed under Liquid chromatography according to the following conditions. The sum of all peaks detected prior to the major peak of the test solution is NMT 3.0% of the sum of all peak areas for preparations containing protamine, and NMT 2.0% of the sum of all peak areas for preparations not containing protamine. Human insulin polymer is detected at 13 to 17 minutes, human insulin dimer is detected at about 17.5 minutes, and human insulin monomer is detected at about 20 minutes; salts are detected thereafter.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 276 nm)

Column: A stainless steel tube about 7.5 mm in internal diameter and 30 cm in length, packed with hydrophilic silical gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Mobile phase: A mixture of 1.0 g/L L-arginine aqueous solution, acetonitrile and acetic acid (100) (65 : 20 : 15)

Flow rate: 0.5 mL/minute

Column equilibration: To perform analysis using a new column, equilibrate by repeatedly injecting human insulin solution comprising macromolecular protein prior to use. Equilibrate the column by injecting the system suitability solution a

minimum of 3 times. Repeatable results are obtained from 2 consecutive injections. To analyze a sample comprising protamine, perform column equilibration using a solution comprising protamine.

System suitability

With the system suitability solution, verify that that peak to valley ratio of the human insulin dimer peaks and monomer peaks is NLT 2.0.

(2) ***Related substances***

Test solution: Add 4 μL of 6 mol/L hydrochloric acid solution per 1 mL of Human Insulin Injection (rDNA) to render the human insulin injection transparent. Mix thoroughly to obtain a homogeneous sample. If the human insulin injection does not turn transparent within 5 minutes, additionally add NMT 4 μL of 6 mol/L hydrochloric acid solution per 1 mL. If the concentration of human insulin is 100 IU/mL or higher, dilute with 0.01 M hydrochloric acid to avoid overloading the column.

Standard solution (a): Dissolve human insulin RS in 0.01 mol/L hydrochloric acid RS to a concentration of 3.75 mg/mL. This standard solution must be used within 2 hours at room temperature, and within 48 hours at 2 to 8℃.

Standard solution (b): To 1.0 mL of standard solution (a), add 0.01 mol/L hydrochloric acid solution to make 10 mL. This standard solution must be used within 2 hours at room temperature, and within 48 hours at 2 to 8℃.

Standard solution (c): Add 0.01 mol/L hydrochloric acid solution to 1.0 mL of standard solution (b) to make 10 mL. This standard solution must be used within 2 hours at room temperature, and within 48 hours at 2 to 8℃.

System suitability solution: Dissolve 1.5 mg of human insulin RS in 1.0 mL of 0.01 mol/L hydrochloric acid solution and let stand at room temperature for NLT 3 days to allow NLT 5% of A21-desamido human insulin to be formed.

Perform the test with 20 μL each of the test solution and the standard solution according to the following conditions as directed under Liquid chromatography; in the chromatogram from the test solution, the peak area of A21-desamido human insulin is NMT 5.0% of the sum of all peak areas, and the sum of all peak areas excluding the human insulin peak and the A21-desamido human insulin peak is NMT 6.0% of the sum of all peak areas. Disregard peaks from preservative or protamine.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 214 nm)

Column: A stainless steel tube about 4.6 mm in internal diameter and about 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 40 °C

Mobile phase: Use mobile phases A and B to control a step or concentration gradient as follows.

Sulfuric acid solution: Dissolve 28.4 g of sodium sulfate in 1L of water, add 2.7 mL of 85% phosphoric acid, then adjust the pH to 2.3 with phosphoric acid or ethanol amine.

Mobile Phase A - A mixture of sulfuric acid solution and acetonitrile (82 : 18)

Mobile Phase B - A mixture of sulfuric acid solution and acetonitrile (50 : 50)

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)	Chromatographic conditions
0 - 60	81	19	Constant concentration
60 - 85	81 → 36	19 → 64	Linear concentration gradient
85 - 91	36	64	Constant concentration
91 - 92	36 → 81	64 → 19	Linear concentration gradient

Flow rate: 1 mL/minute

Column equilibration: Adjust the concentration of the mobile phase and the time of the initial efflux condition so that human insulin is detected at bout 31 minutes, and A21-desamido human insulin is detected immediately after human insulin.

System suitability

The value of the ratio of standard solution (b) and standard solution (a) multiplied by 10 is between 0.91 and 1.09, and the value of the ratio of standard solution (c) and standard solution (a) multiplied by 100 is between 0.7 and 1.3. With the system suitability solution, verify that the resolution between the human insulin peak and the A21-desamido human insulin peak is NLT 2.0, and that the symmetry factor of the human insulin peak is NMT 1.8.

(3) *Water soluble human insulin*—Pipet human insulin injection in suspension form, centrifuge for 10 minutes at 1500 g to obtain a clear supernatant, then perform the test as directed under the Assay. Calculated according to the following equation, water soluble human insulin is NMT 2.5% of the total human insulin content.

$$\frac{100S}{T}$$

S: Insulin content of the clear supernatant

T: Total human insulin content

Zinc Test as directed under Atomic absorption spectroscopy according to the following conditions; zinc content is 10 to 40 μg per 100 IU of human insulin.

Test solution: Add 0.01 mol/L hydrochloric acid solution to 200 IU of human insulin injection (rDNA) to make 25.0 mL. As needed, dilute using 0.01 mol/L hydrochloric acid solution so that the concentration of zinc is 0.4 to 1.6 μg/mL.

Standard solution: Immediately before use, dilute 5 mg/mL zinc standard solution with 0.01 mol/L hydrochloric acid solution to zinc concentrations of 0.40, 0.80, 1.00, 1.20 and 1.60 μg per mL.

Operating conditions

Gas: Dissolved acetylene – Air

Lamp: Zinc hollow-cathode lamp.

Wavelength: 213.9 nm

Sterility Meets the requirements.

Bacterial endotoxins NMT 80 EU in each 100 IU of human insulin.

Particulate contamination: Visible particles Meets the requirements.

Insoluble particulate matter in injections Meets the requirements.

Extractable volume of injections Meets the requirements.

Assay

Test solution: Add 4 μL of 6 mol/L hydrochloric acid solution per 1 mL of Human Insulin Injection (rDNA) to render the human insulin injection transparent. When sampling, agitate the

material prior to sampling in order to obtain a homogeneous sample. If the human insulin injection does not turn transparent within 5 minutes, additionally add NMT 4 μL of 6 mol/L hydrochloric acid solution per 1 mL. If the concentration of human insulin is 100 IU/mL or higher, dilute with 0.01 M hydrochloric acid to avoid overloading the column.

Standard solution (a): Dissolve human insulin RS in 0.01 mol/L hydrochloric acid RS to a concentration of 4.0 mg/mL.

Standard solution (b): To 1.0 mL of standard solution (a), add 0.01 mol/L hydrochloric acid solution to make 10.0 mL.

Store the standard solutions and test solution at 2 to 10 ℃ and use within 48 hours.

System suitability solution: Dissolve 1.5 mg of human insulin RS in 0.01 mol/L hydrochloric acid solution to make 1.5 mg/mL, and let stand at room temperature for NLT 3 days to allow NLT 5% of A21-desamido human insulin to be formed.

Perform the test with 20 μL each of the test solution and the standard solution according to the following conditions as directed under Liquid chromatography; use the peak areas of the test solution and the standard solution and the labeled amount of human insulin RS to calculate the total amount of human insulin and A21-desamido human insulin.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 214 nm)

Column: A stainless steel tube about 4.6 mm in internal diameter and about 25 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 40 °C

Mobile phase: A mixture of mobile phase concentrate B and mobile phase concentrate A (58 : 42)

Mobile phase concentration A: Dissolve 28.4 g of anhydrous sodium sulfate in 1L of water, add 2.7 mL of 85% phosphoric acid, then adjust the pH to 2.3 with phosphoric acid or ethanol amine.

Mobile phase concentrate B: Prepare with mobile phase concentrate A and acetonitrile at a volumetric ratio of 55 : 45. This reaction is endothermic; let stand until at least 20 ℃ before using.

Flow rate: 1 mL/minute

System suitability

The major peak area of the chromatogram obtained from standard solution (a) is 10 ± 0.5 times the major peak area of the chromatogram obtained from standard solution (b). If this requirement is not met, adjust the injection volume to 10 to 20 μL to bring within the linearity range of the detector.

With the system suitability solution, confirm that the resolution between the human insulin peak and the A21-desamido human insulin peak is NLT 2.0, and that the symmetry factor of the human insulin peak is NMT 1.8.

Packaging and storage Preserve in light-resistant, hermetic containers (at 2 ℃ to 8 ℃).

Human Normal Immunoglobulin
사람 면역글로불린

Human Normal Immunoglobulin is a liquid preparation containing immunoglobulin G from human serum globulin.

Human Normal Immunoglobulin meets the requirements in the section of the Human Normal Immunoglobulin under the Specifications and Test Methods for Biologicals, specified separately.

Human Normal Immunoglobulin in Maltose (pH 4.25)
말토즈 첨가 사람 면역글로불린 (pH 4.25)

Human Normal Immunoglobulin in Maltose (pH 4.25) is a liquid preparation containing immunoglobulin G of human serum globulin, and maltose.

Human Normal Immunoglobulin in Maltose (pH 4.25) meets the requirements in the section of the Human Normal Immunoglobulin in Maltose (pH 4.25) under the Specifications and Test Methods for Biologicals, specified separately.

Human Papillomavirus Vaccine (rDNA)
인유두종 바이러스 백신 (유전자재조합)

Human Papillomavirus Vaccine (rDNA) is a liquid preparation containing recombinant human papillomavirus capsid (L1).

Human Papillomavirus Vaccine (rDNA) meets the requirements in the section of the Human Papillomavirus Vaccine (rDNA) under the Specifications and Test Methods for Biologicals, specified separately.

Human Serum Albumin
사람 혈청 알부민

Human Serum Albumin is a liquid preparation containing albumin from human serum.

Human Serum Albumin meets the requirements in the section of the Human Serum Albumin under the Specifications and Test Methods for Biologicals, specified separately.

Human Tetanus Immunoglobulin
항파상풍 사람 면역글로불린

Human Tetanus Immunoglobulin is a liquid preparation containing anti-tetanus human immunoglobulin G from human serum globulin.

Human Tetanus Immunoglobulin meets the requirements in the section of the Human Tetanus Immunoglobulin section under the Specifications and Test Methods for Biologicals, specified separately.

Tetanus Antitoxin (Equine)
파상풍 항독소

Tetanus Antitoxin (Equine) is a liquid preparation containing tetanus antitoxin of animal immunoglobulin.

Tetanus Antitoxin (Equine) meets the requirements in the

section of the Tetanus Antitoxin (Equine) under the Specifications and Test Methods for Biologicals, specified separately.

Human Varicella Immunoglobulin
수두 사람 면역글로불린

Human Varicella Immunoglobulin is a liquid preparation containing human varicella antibody from human serum immunoglobulin G.

Human Varicella Immunoglobulin meets the requirements in the section of the Human Varicella Immunoglobulin under the Specifications and Test Methods for Biologicals, specified separately.

Inactivated Oral Cholera Vaccine
경구용 불활화 콜레라 백신

Inactivated Oral Cholera Vaccine is a preparation that contains inactivated cholera bacteria and recombinant cholera toxin B (rCTB-213).

Inactivated Oral Cholera Vaccine meets the requirements in the section of the Inactivated Oral Cholera Vaccine under the Specifications and Test Methods for Biologicals, specified separately.

Influenza HA Vaccine
인플루엔자 에이취 에이 (HA) 백신

Influenza HA Vaccine is a liquid preparation containing hemagglutinin of inactivated influenza virus.

Influenza HA Vaccine meets the requirements in the section of the Influenza HA Vaccine under the Specifications and Test Methods for Biologicals, specified separately.

Influenza Vaccine (Surface Antigen, Inactivated)
인플루엔자 표면항원 백신

Influenza Vaccine (Surface Antigen, Inactivated) is a liquid preparation containing hemagglutinin and neuraminidase of influenza virus, split and inactivated to maintain antigenicity.

Influenza Vaccine (Surface Antigen, Inactivated) meets the requirements in the section of the Influenza Vaccine (Surface Antigen, Inactivated under the Specifications and Test Methods for Biologicals, specified separately.

Influenza Vaccine (Surface Antigen-Virosome, Inactivated)
인플루엔자 표면항원-비로좀 백신

Influenza Vaccine (Surface Antigen-Virosome, Inactivated) is a liquid preparation obtained by mixing hemagglutinin and neuraminidase of influenza virus, split and inactivated to maintain antigenicity, with phospholipid to form virosomes.

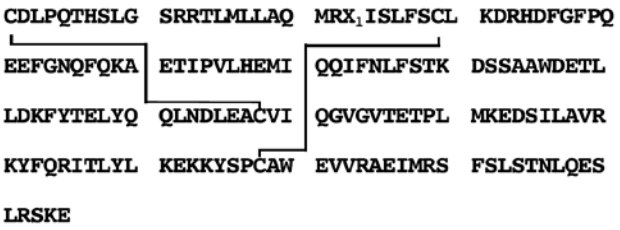
Influenza Vaccine (Surface Antigen-Virosome, Inactivated) meets the requirements in the section of the Influenza Vaccine (Surface Antigen-Virosome, Inactivated) under the Specifications and Test Methods for Biologicals, specified separately.

Influenza Vaccine (Split Virion, Inactivated)
인플루엔자 분할 백신

Influenza Vaccine (Split Virion, Inactivated) is a liquid preparation containing influenza virions that fragmented and inactivated to maintain antigenicity.

Influenza Vaccine (Split Virion, Inactivated) meets the requirements in the section of the Influenza Vaccine (Split Virion, Inactivated) under the Specifications and Test Methods for Biologicals, specified separately.

Interferon Alpha-2 Concentrated Solution (rDNA)
인터페론 알파-2 농축액 (유전자재조합)



Interferon alpha-2 Concentrated Solution (rDNA) is a concentrated solution of a recombinant protein expressed by the interferon alpha-2 gene and has non-specific antiviral activity and antiproliferative activity. Depending on the amino acid at position 23, there are two types as follows.

Designation	Residue at position 23 (X_1)
alpha-2a	Lys
alpha-2b	Arg

Interferon alpha-2 Concentrated Solution (rDNA) contains NLT 1.4×10^8 IU per mg of protein. There are also NLT 2×10^8 IU of interferon alpha-2 per mL of Interferon alpha-2 Concentrated Solution (rDNA).

Description Interferon alpha-2 Concentrated Solution (rDNA) occurs as a clear, colorless to pale yellow liquid.

Identification (1) ***Inhibition of virus proliferation*—**Proceed with Interferon alpha-2 Concentrated Solution (rDNA) as directed under the Potency; an inhibitory effect on the proliferation of vesicular stomatitis virus, etc. is confirmed.

(2) ***Isoelectric focusing*—**Dilute Interferon alpha-2 Concentrated Solution (rDNA) with water to a protein concentration of 1 mg/mL and use this solution as the test solution. Separately, dissolve the interferon alpha-2 RS in water to a concentration of 1 mg/mL and use this solution as the standard solution. Prepare an isoelectric point calibration solution with an isoelectric point in the pH range of 3.0 to 10.0. Perform the test with these solutions according to the following test method. Use a suitable

apparatus connected to a temperature-controlled water bath set at 10 °C and gels for isoelectric focusing (pH 3.5 to 9.5). Use phosphoric acid (98 g/L $_3PO_4$) as anolyte and 1 mol/L sodium hydroxide as catholyte. Spot 15 μL each of the test solution and the standard solution onto a filter paper. Place the filter paper on the gel near the cathode. Start the isoelectric focusing at 1500 V and 50 mA. Turn off the power after 30 minutes, remove the filter paper and reconnect the power for 1 hour. Keep the power constant during the isoelectric focusing process. After isoelectric focusing, immerse the gel in an appropriate volume of a solution containing 115 g of trichloroacetic acid and 34.5 g of sulfosalicylic acid in water and gently shake the container for 60 minutes. Transfer the gel to a mixture of water, ethanol (99.5), and acetic acid (100) (268 : 100 : 32) and soak for 5 minutes. Immerse the gel for 10 minutes in a staining solution preheated to 60 °C with 1.2 g of Coomassie Brilliant Blue R-250 added to the mixture of water, ethanol (99.5), and acetic acid (100) (268 : 100 : 32). Wash the gel several times with the mixture of water, ethanol (99.5), and acetic acid (100) (268 : 100 : 32) and leave the gel in this mixture until it becomes clear (12 to 24 hours). After sufficient destaining, soak the gel for 1 hour in a 10% v/v solution of glycerol in the mixture of water, ethanol (99.5), and acetic acid (100) (268 : 100 : 32). Draw a standard curve from the migration distance of the isoelectric point marker and the corresponding isoelectric point and determine the isoelectric points of the test solution and the standard solution. The position of the main band obtained from the test solution is consistent with that of the standard solution. The isoelectric points of the bands of the test solution should not differ from the isoelectric points of the bands of the standard solution by more than 0.2 pH units. The isoelectric point markers are distributed along the entire length of the gel and the isoelectric points of the main bands in the electropherogram obtained from the standard solution are between 5.8 and 6.3.

(3) ***Electrophoresis*—**Observe the electropherogram pattern obtained under reducing conditions in the test for abnormal peptides of the purity test. The main band obtained from the test solution (a) corresponds in position to the main band of standard solution (a).

(4) ***Peptide map*—**Dilute Interferon alpha-2 Concentrated Solution (rDNA) with water to a protein concentration of 1.5 mg/mL. Transfer 25 μL to a 1.5-mL polypropylene or glass container, add 1.6 μL of 1 mol/L phosphate buffer (pH 8.0), 2.8 μL of freshly prepared 1.0 mg/mL trypsin solution, and 3.6 μL of water, and shake vigorously to mix. Place a stopper and allow to react at 37 °C for 18 hours. Then, add 100 μL of 573 g/L guanidine hydrochloride solution and mix well. Add 7 μL of 154.2 g/L dithiothreitol (DTT) reducing solution, mix well, and allow to stand at 95 to 100 °C for 1 minute. Cool to room temperature and use it as the test solution. Separately, dissolve the interferon alpha-2 RS in water to a concentration of 1.5 mg/mL, process it simultaneously and in the same manner as the test solution, and use it as the standard solution. Perform the test with 100 μL each of the test solution and the standard solutions as directed under Liquid chromatography; the chromatogram pattern obtained from the test solution is consistent with the chromatogram obtained from the standard solution.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 214 nm)

Column: A stainless steel column about 4.6 mm in internal diameter and 10 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 30 °C

Mobile phase: Use mobile phases A and B to control a step or concentration gradient as follows.

Mobile phase A: A mixture of water and trifluoroacetic acid (999 : 1)

Mobile phase B: A mixture of acetonitrile, water, and trifluoroacetic acid (899 : 100 : 1)

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)	Chromatographic conditions
0 - 8	100	0	Constant concentration
8 - 68	100 → 40	0 → 60	Linear concentration gradient
68 - 72	40	60	Constant concentration
72 - 75	40 → 100	60 → 0	Linear concentration gradient
75 - 80	100	0	Constant concentration

Flow rate: 1.0 mL/minute. Equilibrate the column with mobile phase A for at least 15 minutes.

Purity (1) ***Abnormal peptides*—**To identify abnormal peptides that have a different molecular weight from interferon alpha-2, perform the test with Interferon alpha-2 Concentrated Solution (rDNA) under reducing and non-reducing conditions as follows; the electropherogram obtained with the test solution (a) under reducing conditions may show less intense bands with lower molecular masses than the main band in addition to the main band. None of these bands is more intense than the main band of the standard solution (d) (1.0%) and NMT 3 such bands are more intense than the main band of the standard solution (e) (0.2%). In addition to the main band, the electropherogram obtained with the test solution (a) under non-reducing conditions may have less intense bands with higher molecular masses than the main band. None of these bands is more intense than the main band of standard solution (d) (1.0%) and NMT 3 such bands are more intense than the main band of standard solution (e) (0.2%). Prepare test solutions and standard solutions for Interferon alpha-2 Concentrated Solution (rDNA) under non-reducing and reducing conditions according to the following conditions and test them by electrophoresis using a resolving gel containing 14% acrylamide and silver staining.

Concentrated sodium dodecyl sulfate-polyacrylamide gel electrophoresis buffer: Weigh 1.89 g of Tris, 5.0 g of sodium dodecyl sulfate, and 50 mg of bromophenol blue and dissolve them in water. Then, add 25.0 mL of glycerol and add water again to make 100 mL. Titrate this solution with hydrochloric acid VS to pH 6.8 and then add water to make exactly 125 mL.

Dilution buffer (non-reducing conditions): Mix equal volumes of concentrated sodium dodecyl sulfate-polyacrylamide gel electrophoresis buffer and water.

Dilution buffer (reducing conditions): Mix equal volumes of concentrated sodium dodecyl sulfate-polyacrylamide gel electrophoresis buffer containing 2-mercaptoethanol, etc. as a reducing agent and water.

Test solution (a): Dilute Interferon alpha-2 Concentrated Solution (rDNA) with the dilution buffer to a protein concentration of 0.5 mg/mL.

Test solution (b): Add the dilution buffer to 0.20 mL of the test solution (a) to make 1 mL.

Standard solution (a): Dilute the interferon alpha-2 RS with the dilution buffer to a concentration of 0.625 mg/mL.

Standard solution (b): Add the dilution buffer to 0.20 mL

of standard solution (a) to make 1 mL.

Standard solution (c): Add the dilution buffer to 0.20 mL of standard solution (b) to make 1 mL.

Standard solution (d): Add the dilution buffer to 0.20 mL of standard solution (c) to make 1 mL.

Standard solution (e): Add the dilution buffer to 0.20 mL of standard solution (d) to make 1 mL.

Molecular weight standard solution (f): Use an appropriate molecular weight standard solution to calibrate sodium dodecyl sulfate-polyacrylamide gel electrophoresis in the molecular weight range of 15000 to 67000.

Heat the test solutions and standard solutions on a water bath for 2 minutes, then add 10 μL of the molecular weight standard solution (f) and apply 20 to 50 μL each of the test solutions and the standard solutions to the wells of the concentrated gel. Examine the gel with silver staining after electrophoresis is complete; a band is identified in the standard solution (e). In addition, a gradation of the intensity of the staining is visible in the electropherograms obtained with the test solution (a) and the test solution (b) as well as with the standard solutions (a) to (e).

(2) ***Related substances*—**Prepare the test solution by diluting Interferon alpha-2 Concentrated Solution (rDNA) with water to a protein concentration of 1 mg/mL. Add an appropriate amount of 0.25% hydrogen peroxide solution to the test solution so that the final concentration is 0.005%. Then, let the solution stand at room temperature for 1 hour or so that about 5% of oxidized interferon is produced, and then use it as the standard solution. Add 12.5 mg of L-methionine per mL of the standard solution and allow the solution to stand at room temperature for 1 hour. Store the reaction solution in the refrigerator and use it within 24 hours. Perform the test with 50 μL each of the test solution and the standard solution as directed under Liquid chromatography; interferon alpha-2 is separated at a retention time of about 20 minutes, and in the chromatogram obtained from the standard solution, the peak related to oxidized interferon has a retention time of about 0.9 minutes compared to the main peak. The total area of peaks other than the main peak is NMT 5.0% of the total area of all peaks.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 210 nm)

Column: A stainless steel tube about 4.6 mm in internal diameter and about 25 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

Mobile phase: Use mobile phases A and B to control a step or concentration gradient as follows.

Mobile phase A: A mixture of water, acetonitrile, and trifluoroacetic acid (700 : 300 : 2)

Mobile phase B: A mixture of acetonitrile, water, and trifluoroacetic acid (800 : 200 : 2)

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)	Chromatographic conditions
0 - 1	72	28	Constant concentration
1 - 5	72 → 67	28 → 33	Linear concentration gradient
5 - 20	67 → 63	33 → 37	Linear concentration gradient
20 - 30	63 → 57	37 → 43	Linear concentration gradient
30 - 40	57 → 40	43 → 60	Linear concentration gradient
40 - 42	40	60	Constant concentration
42 - 50	40 → 72	60 → 28	Linear concentration gradient
50 - 60	72	28	Constant concentration

Flow rate: 1.0 mL/minute. Equilibrate the column with the mobile phases in the initial gradient ratio for at least 15 minutes.

System suitability

The resolution between the main interferon peak and the oxidized form is NLT 1.0. The retention time of the oxidized interferon peak is 0.7 to 1.4 compared to the main peak. The area of each peak other than the main peak in the chromatogram obtained from the test solution is NMT 3.0% of the sum of all peak areas.

Bacterial endotoxins Interferon alpha-2 Concentrated Solution (rDNA) is less than 100 EU per mg of interferon alpha-2.

Assay (1) ***Protein content*—**Dilute Interferon alpha-2 Concentrated Solution (rDNA) with water so that the concentration of interferon alpha-2 is about 0.5 mg/mL and use this solution as the test solution. Use 0.5 mg/mL bovine albumin solution as the standard stock solution. Take an appropriate amount of the standard stock solution and dilute it to 3 to 30 μg/mL to prepare the standard solutions with 8 different concentrations. Perform the test with the test solution and standard solutions as follows. Prepare 30- and 50-fold dilutions of the test solution. Mix 2.0 mL of 20 g/L copper sulfate solution and 2.0 mL of 40 g/L tartrate solution. Then, mix with 96.0 mL of 40 g/L sodium carbonate solution dissolved in 0.2 mol/L sodium hydroxide. Mix 1.25 mL of the resulting solution with 1.5 mL of water, then add 1.5 mL of the diluted test solution or standard solution and mix. After about 10 minutes, add 0.25 mL of a mixture of the same volume of water and phosphomolybdotungstic acid TS to each container and mix. After about 30 minutes, measure the absorbance of each solution at 750 nm using the blank solution as the calibration solution. Plot a standard curve from the absorbance of 8 standard solutions and the corresponding protein contents and determine the protein content in the test solution using this standard curve.

(2) ***Potency*—**The potency of interferon alpha-2 is evaluated by comparing its effect in protecting cells from a viral apoptotic effect with the effect of the corresponding international standard for recombinant human interferon alpha-2 or a reference standard calibrated in international units. Determine the potency by testing under the following conditions. Incubate a certain number of cells with 3 or more different concentrations of the test solution and standard in each well of a microtiter plate and make sure to include control groups of untreated cells. Calculate the potency based on the results of four or more consecutive tests performed by the same method. Select the range of interferon concentration so that some protection against viral apoptosis occurs at the lowest concentration and at the highest concentration, the protective effect against viral apoptosis is below the maximum value. Add the apoptotic virus to all wells at the appropriate time and make sure to include a control group without virus treatment. Once the progression of the apoptotic effect is confirmed, stain the cells with an appropriate reagent, wash them, and dry them at room temperature. Then, extract the cells with 2-methoxyethanol, etc., and measure the absorbance of each well at 550 nm to quantify the apoptotic effect of the virus. Calculate the potency of the test solution with a common statistical method using

the parallel line test. The measured potency is NLT 80% and NMT 125% of the labeled potency, and the confidence limit (P = 0.95) is NLT 64% and NMT 156% of the labeled potency.

Use a well-established cell line sensitive to the apoptotic effect of each virus in standard culture conditions. The following cell cultures and viruses are suitable: MDBK (Madin-Darby bovine kidney) cells (ATCC No. CCL22) or Mouse L cells (NCTC clone 929; ATCC No. CCL1) as the cell culture and vesicular stomatitis virus, Indiana strain (ATCC No. VR-158) as the viral injection; or A549 cells (ATCC No. CCL-185) responsive to interferon as the cell culture and encephalomyocarditis virus (ATCC No. VR-129B) as the viral injection.

Packaging and storage Preserve in light-resistant, hermetic containers (-20 ℃ or below).

Japanese Encephalitis Vaccine
일본뇌염 백신

Japanese Encephalitis Vaccine is a liquid preparation obtained by cultivating Japanese Encephalitis virus then isolating, purifying and inactivating the antigen.

Japanese Encephalitis Vaccine meets the requirements in the section of the Japanese Encephalitis Vaccine under the Specifications and Test Methods for Biologicals, specified separately.

Live Attenuated Oral Rotavirus Vaccine
경구용 로타 생바이러스 백신

Live Attenuated Oral Rotavirus Vaccine is a freeze- dried preparation or liquid preparation containing live attenuated rotavirus.

Live Attenuated Oral Rotavirus Vaccine meets the requirements in the section of the Live Attenuated Oral Rotavirus Vaccine under the Specifications and Test Methods for Biologicals, specified separately.

Live Attenuated Varicella Vaccine
수두 생바이러스 백신

Live Attenuated Varicella Vaccine is a freeze-dried preparation containing live attenuated varicella virus. It becomes a liquid preparation on the addition of a solvent.

Live Attenuated Varicella Vaccine meets the requirements in the section of the Live Attenuated Varicella Vaccine under the Specifications and Test Methods for Biologicals, specified separately.

Oral Typhoid Vaccine
경구용 장티푸스 백신

Oral Typhoid Vaccine is a freeze-dried, capsule-filled or enteric-coated product of an attenuated strain of typhoid bacillus Ty21a.

Oral Typhoid Vaccine meets the requirements in the section of the Oral Typhoid Vaccine under the Specifications and Test Methods for Biologicals, specified separately.

Pneumococcal Polysaccharide Vaccine
폐렴구균 백신

Pneumococcal Polysaccharide Vaccine is a liquid preparation containing purified capsular polysaccharides extracted from each of pneumococcal capsular types 1, 2, 3, 4, 5, 6B, 7F, 8, 9N, 9V, 10A, l1A, 12F, 14, l5B, 17F, 18C, 19A, 19F, 20, 22F, 23F and 33F (Danish nomenclature).

Pneumococcal Polysaccharide Vaccine meets the requirements in the section of the Pneumococcal Polysaccharide Vaccine under the Specifications and Test Methods for Biologicals, specified separately.

Pneumococcus Conjugated to Diphtheria CRM197 Vaccine
폐렴구균.디프테리아 CRM197단백 접합 백신

Pneumococcus Conjugated to Diphtheria CRM197 Vaccine is a liquid preparation containing purified serotype polysaccharides extracted from each of pneumococcal serotypes 4, 6B, 9V, 14, 18C, 19F and 23F (Danish nomenclature), or pneumococcus to which serotypes 1, 3, 5, 6A, 7F and 19A (Danish nomenclature) have been added, conjugated to the CRM197 protein, which is a non-toxic variant of diphtheria toxin.

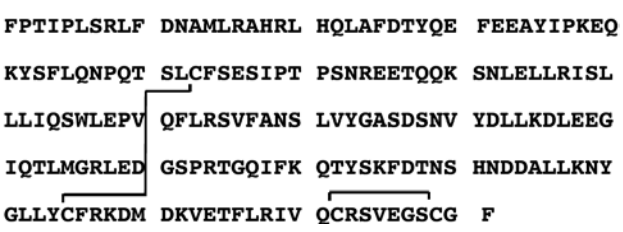
Pneumococcus Conjugated to Diphtheria CRM197 Vaccine meets the requirements in the section of the Pneumococcus Conjugated to Diphtheria CRM197 Vaccine under the Specifications and Test Methods for Biologicals, specified separately.

Purified Vi Polysaccharide Typhoid Vaccine
정제 브이아이 장티푸스 백신

Purified Vi Polysaccharide Typhoid Vaccine is a liquid preparation containing inactivated purified Vi capsular polysaccharide of *Salmonella typhi*.

Purified Vi Polysaccharide Typhoid Vaccine meets the requirements in the section of the Purified Vi Polysaccharide Typhoid Vaccine under the Specifications and Test Methods for Biologicals, specified separately.

Somatropin (rDNA)
소마트로핀 (유전자재조합)



$C_{990}H_{1528}N_{262}O_{300}S_7$: 22125

Somatropin (rDNA) is a recombinant protein of the human growth hormone, which consists of 191 amino acid residues.

Somatropin (rDNA) contains NLT 91.0% and NMT 105.0% of somatropin calculated with RS to the anhydrous basis. 1 mg of anhydrous somatropin ($C_{990}H_{1528}N_{262}O_{300}S_7$) is equivalent to 3.0 IU of the biological activity.

Description Somatropin (rDNA) occurs as a white powder.

Identification (1) Perform the test as directed in (1) or (2) of the Charged variants under the Purity.

(1-1) ***Capillary electrophoresis***—Proceed as directed in the test method described in the Capillary electrophoresis of charged variants under the Purity, after applying the following modification; only a single major peak corresponding to somatropin is observed.

Injection: Inject the test solution (b) for at least 3 seconds under pressure or under vacuum, and then inject the capillary electrophoresis buffer solution for 1 second.

(1-2) ***Isoelectric focusing***—Examine the eletropherograms of isoelectric focusing described in the test method for charged variants under the Purity; the position of the principal band obtained from the test solution (a) is the same as that obtained from the standard solution (a).

(2) ***Reverse-phase liquid chromatography***—Examine the chromatograms of the related substances obtained as directed under the Related substances; the retention time and size of the major peak obtained from the test solution is similar to those obtained from the standard solution.

(3) ***Peptide map***

Test solution: Dilute Somatropin (rDNA) with 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) to adjust the concentration of somatropin to 2.0 mg/mL. Transfer about 1.0 mL of this solution into a tube made of a suitable material, such as polypropylene. Prepare 1 mg/mL trypsin solution diluted with 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) and add 30 μL of this solution to the test sample. Cap the tube and allow to react on a water bath at 37 °C for 4 hours. Remove the tube from the water bath and stop the reaction immediately by an appropriate method, such as freezing. Analyze the sample immediately using an automatic injector, while maintaining the temperature at 2 to 8 °C.

Standard solution: Prepare the standard solution using somatropin RS, concurrently with the test solution in the same manner.

Perform the test with 100 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions; the profiles of the chromatograms obtained from the test solution and the standard solution are the same.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 214 nm)

Column: A stainless steel tube about 4.6 mm in internal diameter and about 25 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (about 5 μm to10 μm in particle diameter).

Column temperature: A constant temperature of about 30 °C.

Mobile phase: Control the mobile phases A and B stepwise as directed in the following table or via the concentration gradient.

Mobile phase A: A mixture of trifluoroacetic acid and water (999 : 1)

Mobile phase B: A mixture of acetonitrile, water and trifluoroacetic acid (899 : 100 : 1)

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)	Chromatographic conditions
0 - 20	100 → 80	0 → 20	Linear concentration gradient
20 - 40	80 → 75	20 → 25	Linear concentration gradient
40 - 65	75 → 50	25 → 50	Linear concentration gradient
65 - 70	50 → 20	50 → 80	Linear concentration gradient

Flow rate: 1 mL/min

(4) ***Size-exclusion liquid chromatography***—Examine the chromatograms obtained from the Assay; the retention time and size of the major peak obtained from the chromatogram of the test solution is similar to those obtained from the chromatogram of the standard solution.

Purity (1) ***Related substances***

Test solution: Dilute Somatropin (rDNA) with 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) to adjust the concentration of somatropin to 2.0 mg/mL. If the concentration of the test solution is lower than this, adjust the injection volume.

Standard solution: Dilute somatropin RS with 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) to adjust the concentration of protein to 2.0 mg/mL.

System suitability test solution: Dilute somatropin/desamido-somatropin mixture RS solution with 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) to adjust the concentration of somatropin to 2 mg/mL.

Perform the test with 20 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions; the amount of related substances is NMT 6.0%.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 220 nm)

Column: A stainless steel tube about 4.6 mm in internal diameter and about 25 cm in length, packed with butylsilyl silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 45 °C.

Mobile phase: A mixture of 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) and propanol (71 : 29)

Column equilibration: Equilibrate the column using 200 to 500 mL of a solution containing 50% acetonitrile and 0.1% trifluoroacetic acid. If necessary, repeat the equilibration process to improve the performance of the column.

Flow rate: 0.5 mL/min

System suitability

System performance: The relative retention time of somatropin in the standard solution to the peak of desamido is about 0.85 (Adjust the retention time of somatropin to about 33 minutes, if necessary, by modifying the concentration of propanol in the mobile phase). Perform the test with the system suitability test solution; the resolution between the peaks of desamido and somatropin should be NLT 1.0 with the symmetry factor of the somatropin peak being 0.9 to 1.8.

(2) ***Dimer and the related substance of higher molecular***

weight

Test solution: Dilute Somatropin (rDNA) with 0.025 mol/L phosphate buffer solution (pH 7.0) to adjust the concentration of somatropin to 1.0 mg/mL.

Standard solution: Dilute somatropin RS with 0.025 mol/L phosphate buffer solution (pH 7.0) to adjust the concentration of somatropin to 1.0 mg/mL.

System suitability test solution: Place somatropin RS in an oven at 50 °C for a sufficient amount of time (usually 12 to 24 hours) to generate 1 to 2% of dimers. Dissolve the resulting content in the phosphate buffer solution (pH 7.0) and adjust the concentration of somatropin to 1.0 mg/mL.

Perform the test with 20 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions; the sum of the peak areas of any peaks detected before the major peak obtained from the test solution is NMT 4.0%.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 214 nm)

Column: A stainless steel column about 7.8 mm in internal diameter and about 30 cm in length, packed with hydrophilic silica gel for liquid chromatography, which is capable of separating proteins having a molecular weight of 5000 to 150000.

Mobile phase: A mixture of 0.063 mol/L phosphate buffer solution (pH 7.0) and 2-propanol (97 : 3)

Flow rate: 0.6 mL/min

System suitability

System performance: The relative retention time of the peak of the related substance with higher molecular weight to the retention time of the peak of somatropin monomer (12 to 17 minutes) from the standard solution is about 0.65. The relative retention time of the peak of somatropin dimer to the retention time of the peak of somatropin monomer (12 to 17 minutes) from the standard solution is about 0.9. The peak-to-valley ratio is NLT 2.5.

(3) ***Charged variants***—Perform the test as directed in (1) or (2).

(1) Capillary electrophoresis

Test solution (a): Dilute Somatropin (rDNA) with water to adjust the concentration of somatropin to 1 mg/mL.

Test solution (b): Mix equal volumes of the test solution (a) and the standard solution.

Standard solution: Dilute somatropin RS with water to adjust the concentration of somatropin to 1 mg/mL.

Capillary electrophoresis buffer solution: 13.2 g/L ammonium phosphate buffer solution. Adjust the pH to 6.0 with phosphoric acid, and then filter through a membrane filter.

Proceed with the test solution and the standard solution as directed under the Capillary electrophoresis according to the following operating conditions; the amount of the deamidated form is NMT 5.0%, the amount of each impurity is NMT 2.0%, and the total amount of charged variants is NMT 10.0%.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 200 nm)

Capillary: An uncoated silica capillary about 50 μm in internal diameter and about 70 cm in effective length

Column temperature: A constant temperature of about 30 °C.

Step	Solution	Time	Condition
Capillary equilibration	1 mol/L sodium hydroxide	20 min	Under pressure or under vacuum
	Water	10 min	
	Capillary electrophoresis buffer solution	20 min	
Between-run washing	0.1 mol/L sodium hydroxide	2 min	Under pressure or under vacuum
	Capillary electrophoresis buffer solution	6 min	
Injection	Injection of the test solution and the standard solution	3 sec	Under pressure or under vacuum
	Capillary electrophoresis buffer solution	1 sec	
Separation	Capillary electrophoresis buffer solution	80 min	217 V/cm

System suitability

System performance: The relative migration distance of the deamidated form to somatropin is 1.02 to 1.11, and the profiles of the electropherograms obtained from the standard solution and the test solution are similar. Two peaks (I1, I2) are detected before the major peak, and more than two peaks (I3, I4) are detected after the major peak. I2 exhibits a fragmented form. As a deamidated form, I4 is detected as two peaks.

(2) Isoelectric focusing

Test solution (a): Dilute Somatropin (rDNA) with 0.025 mol/L phosphate buffer solution (pH 7.0) to adjust the concentration of somatropin to 2.0 mg/mL.

Mobile phase: To 1.9 mL of 0.025 mol/L phosphate buffer solution (pH 7.0), add 0.1 mL of the test solution (a).

Standard solution (a): Dilute somatropin RS with 0.025 mol/L phosphate buffer solution (pH 7.0) to adjust the concentration of somatropin to 2.0 mg/mL.

Standard solution (b): Use an isoelectric point calibration solution in the pH range of 2.5 to 6.

Operating conditions

Perform the isoelectric focusing using a polyacrylamide gel in the pH range of 4.0 to 6.5. Spot 15 μL each of the test solution and the standard solution on the gel. Use a solution, in which 14.7 g of glutamic acid is dissolved in 1L of a phosphoric acid (50 g/L H_3PO_4) solution, as the anode solution and 89.1 g/L β-alanine solution as the cathode solution. Set the operating conditions to 2000 V and 25 mA for isoelectric focusing so that the voltage is maintained constant for 2.5 hours during the focusing process. At this time, the power should not exceed 25 W. After the focusing is completed, immerse the gel in a suitable volume of a solution containing 115 g/L trichloroacetic acid and 34.5 g/L sulfosalicylic acid TS for 30 minutes, and then transfer the gel to a mixture of water, ethanol (99.5) and acetic acid (100) (67 : 25 : 8) to immerse for 5 minutes. Prepare the staining solution by dissolving 1.15 g of Coomassie Brilliant Blue R-250 in 1 L of a mixture of water, ethanol (99.5) and acetic acid (100) (67 : 25 : 8). Stain the gel in the staining solution, previously warmed to 60 °C, for 10 minutes. After staining is completed, place the gel in the destaining solution, a mixture of water, ethanol (99.5) and acetic acid (100) (67 : 25 : 8), until the excess staining reagents are sufficiently removed.

No band other than the principal band obtained from the test solution (a) is more intense than the principal band obtained from the test solution (b) (NMT 5%).

System suitability

System performance: All bands obtained from the standard solution (b), which is the isoelectric point calibration

solution, is well separated from each other. With the standard solution (a), the principal band appears at the isoelectric point of about 5.0, and the minor band at the isoelectric point of about 4.8.

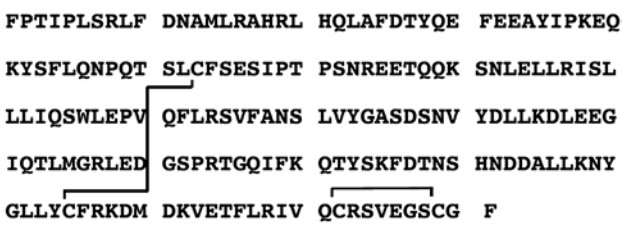
Water NMT 10.0%

Bacterial endotoxins Less than 5 EU/mg of somatropin.

Assay Use the chromatograms obtained from the test for dimer and the related substance of higher molecular weight to calculate the amount of somatropin in the test sample based on the labeled amount of somatropin RS.

Packaging and storage Preserve in hermetic container (2 to 8 °C).

Somatropin Concentrated Solution (rDNA)
소마트로핀 농축액 (유전자재조합)



$C_{990}H_{1528}N_{262}O_{300}S_7$: 22125

Somatropin Concentrated Solution (rDNA) is a solution containing a recombinant protein of the human growth hormone, which consists of 191 amino acid residues.

Somatropin Concentrated Solution (rDNA) contains NLT 91.0% and NMT 105.0% of the labeled amount. 1 mg of anhydrous somatropin ($C_{990}H_{1528}N_{262}O_{300}S_7$) is equivalent to 3.0 IU of the biological activity.

Description Somatropin Concentrated Solution (rDNA) occurs as a clear or slightly turbid, colorless liquid.

Identification (1) Perform the test as directed in (1) or (2) of the Charged variants under the Purity.

(1-1) ***Capillary electrophoresis***—Proceed as directed in the test method described in the Capillary electrophoresis of charged variants under the Purity, after applying the following modification; only a single major peak corresponding to somatropin is observed.

Injection: Inject the test solution (b) for at least 3 seconds under pressure or under vacuum, and then inject the capillary electrophoresis buffer solution for 1 second.

(1-2) ***Isoelectric focusing***—Examine the eletropherograms of isoelectric focusing described in the test method for charged variants under the Purity; the position of the principal band obtained from the test solution (a) is the same as that obtained from the standard solution (a).

(2) ***Reverse-phase liquid chromatography***—Examine the chromatograms of the related substances obtained as directed under the Related substances; the retention time and size of the major peak obtained from the test solution is similar to those obtained from the standard solution.

(3) ***Peptide map***

Test solution: Dilute Somatropin Concentrated Solution (rDNA)with 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) to adjust the concentration of somatropin to 2.0 mg/mL. Transfer about 1.0 mL of this solution into a tube made of a suitable material, such as polypropylene. Prepare 1 mg/mL trypsin solution diluted with 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) and add 30 μL of this solution to the test sample. Cap the tube and allow to react on a water bath at 37 °C for 4 hours. Remove the tube from the water bath and stop the reaction immediately by an appropriate method, such as freezing. Analyze the sample immediately using an automatic injector, while maintaining the temperature at 2 to 8 °C.

Standard solution: Prepare the standard solution using somatropin RS, concurrently with the test solution in the same manner.

Perform the test with 100 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions; the profiles of the chromatograms obtained from the test solution and the standard solution are the same.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 214 nm)

Column: A stainless steel tube about 4.6 mm in internal diameter and about 25 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (about 5 μm to10 μm in particle diameter).

Column temperature: A constant temperature of about 30 °C.

Mobile phase: Control the mobile phases A and B stepwise as directed in the following table or via the concentration gradient.

Mobile phase A: A mixture of trifluoroacetic acid and water (999 : 1)

Mobile phase B: A mixture of acetonitrile, water and trifluoroacetic acid (899 : 100 : 1)

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)	Chromatographic conditions
0 - 20	100 → 80	0 → 20	Linear concentration gradient
20 - 40	80 → 75	20 → 25	Linear concentration gradient
40 - 65	75 → 50	25 → 50	Linear concentration gradient
65 - 70	50 → 20	50 → 80	Linear concentration gradient

Flow rate: 1 mL/min

(4) ***Size-exclusion liquid chromatography***—Examine the chromatograms obtained from the Assay; the retention time and size of the major peak obtained from the chromatogram of the test solution is similar to those obtained from the chromatogram of the standard solution.

Purity (1) ***Related substances***

Test solution: Dilute Somatropin Concentrated Solution (rDNA) with 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) to adjust the concentration of somatropin to 2.0 mg/mL. If the concentration of the test solution is lower than this, adjust the injection volume.

Standard solution: Dilute somatropin RS with 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) to adjust the concentration of protein to 2.0 mg/mL,

System suitability test solution: Dilute somatropin/desamido-somatropin mixture RS solution with 0.05 mol/L

tris-hydrochloric acid buffer solution (pH 7.5) to adjust the concentration of somatropin to 2 mg/mL.

Perform the test with 20 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions; the amount of related substances is NMT 6.0%.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 220 nm)

Column: A stainless steel tube about 4.6 mm in internal diameter and about 25 cm in length, packed with butylsilyl silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 45 °C.

Mobile phase: A mixture of 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) and propanol (71 : 29)

Column equilibration: Equilibrate the column using 200 to 500 mL of a solution containing 50% acetonitrile and 0.1% trifluoroacetic acid. If necessary, repeat the equilibration process to improve the performance of the column.

Flow rate: 0.5 mL/min

System suitability

System performance: The relative retention time of somatropin in the standard solution to the peak of desamido is about 0.85 (Adjust the retention time of somatropin to about 33 minutes, if necessary, by modifying the concentration of propanol in the mobile phase). Perform the test with the system suitability test solution; the resolution between the peaks of desamido and somatropin should be NLT 1.0 with the symmetry factor of the somatropin peak being 0.9 to 1.8.

(2) ***Dimer and the related substance of higher molecular weight***

Test solution: Dilute Somatropin Concentrated Solution (rDNA) with 0.025 mol/L phosphate buffer solution (pH 7.0) to adjust the concentration of somatropin to 1.0 mg/mL.

Standard solution: Dilute somatropin RS with 0.025 mol/L phosphate buffer solution (pH 7.0) to adjust the concentration of somatropin to 1.0 mg/mL.

System suitability test solution: Place somatropin RS in an oven at 50 °C for a sufficient amount of time (usually 12 to 24 hours) to generate 1 to 2% of dimers. Dissolve the resulting content in the phosphate buffer solution (pH 7.0) and adjust the concentration of somatropin to 1.0 mg/mL.

Perform the test with 20 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions; the sum of the peak areas of any peaks detected before the major peak obtained from the test solution is NMT 4.0%.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 214 nm)

Column: A stainless steel column about 7.8 mm in internal diameter and about 30 cm in length, packed with hydrophilic silica gel for liquid chromatography, which is capable of separating proteins having a molecular weight of 5000 to 150000.

Mobile phase: A mixture of 0.063 mol/L phosphate buffer solution (pH 7.0) and 2-propanol (97 : 3)

Flow rate: 0.6 mL/min

System suitability

System performance: The relative retention time of the peak of the related substance with higher molecular weight to the retention time of the peak of somatropin monomer (12 to 17 minutes) from the standard solution is about 0.65. The relative retention time of the peak of somatropin dimer to the retention time of the peak of somatropin monomer (12 to 17 minutes) from the standard solution is about 0.9. The peak-to-valley ratio is NLT 2.5.

(3) ***Charged variants***—Perform the test as directed in (1) or (2).

(1) Capillary electrophoresis

Test solution (a): Dilute Somatropin Concentrated Solution (rDNA) with water to adjust the concentration of somatropin to 1 mg/mL.

Test solution (b): Mix equal volumes of the test solution (a) and the standard solution.

Standard solution: Dilute somatropin RS with water to adjust the concentration of somatropin to 1 mg/mL.

Capillary electrophoresis buffer solution: 13.2 g/L ammonium phosphate buffer solution. Adjust the pH to 6.0 with phosphoric acid, and then filter through a membrane filter.

Proceed with the test solution and the standard solution as directed under the Capillary electrophoresis according to the following operating conditions; the amount of the deamidated form is NMT 5.0%, the amount of each impurity is NMT 2.0%, and the total amount of charged variants is NMT 10.0%.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 200 nm)

Capillary: An uncoated silica capillary about 50 μm in internal diameter and about 70 cm in effective length

Column temperature: A constant temperature of about 30 °C.

Step	Solution	Time	Condition
Capillary equilibration	1 mol/L sodium hydroxide	20 min	Under pressure or under vacuum
	Water	10 min	
	Capillary electrophoresis buffer solution	20 min	
Between-run washing	0.1 mol/L sodium hydroxide	2 min	Under pressure or under vacuum
	Capillary electrophoresis buffer solution	6 min	
Injection	Injection of the test solution and the standard solution	3 sec	Under pressure or vacuum
	Capillary electrophoresis buffer solution	1 sec	
Separation	Capillary electrophoresis buffer solution	80 min	217 V/cm

System suitability

System performance: The relative migration distance of the deamidated form to somatropin is 1.02 to 1.11, and the profiles of the electropherograms obtained from the standard solution and the test solution are similar. Two peaks (I1, I2) are detected before the major peak, and more than two peaks (I3, I4) are detected after the major peak. I2 exhibits a fragmented form. As a deamidated form, I4 is detected as two peaks.

(2) Isoelectric focusing

Test solution (a): Dilute Somatropin Concentrated Solution (rDNA) with 0.025 mol/L phosphate buffer solution (pH 7.0) to adjust the concentration of somatropin to 2.0 mg/mL.

Mobile phase: To 1.9 mL of 0.025 mol/L phosphate buffer (pH 7.0), add 0.1 mL of the test solution (a).

Standard solution (a): Dilute somatropin RS with 0.025

mol/L phosphate buffer solution (pH 7.0) to adjust the concentration of somatropin to 2.0 mg/mL.

Standard solution (b): Use an isoelectric point calibration solution in the pH range of 2.5 to 6.

Operating conditions

Perform the isoelectric focusing using a polyacrylamide gel in the pH range of 4.0 to 6.5. Spot 15 μL each of the test solution and the standard solution on the gel. Use a solution, in which 4.7 g of glutamic acid is dissolved in 1L of a phosphoric acid (50 g/L H_3PO_4) solution, as the anode solution and 89.1 g/L β-alanine solution as the cathode solution. Set the operating conditions to 2000 V and 25 mA for isoelectric focusing so that the voltage is maintained constant for 2.5 hours during the focusing process. At this time, the power should not exceed 25 W. After the focusing is completed, immerse the gel in a suitable volume of a solution containing 115 g/L trichloroacetic acid and 34.5 g/L sulfosalicylic acid TS for 30 minutes, and then transfer the gel to a mixture of water, ethanol (99.5) and acetic acid (100) (67 : 25 : 8) to immerse for 5 minutes. Prepare the staining solution by dissolving 1.15 g of Coomassie Brilliant Blue R-250 in 1 L of a mixture of water, ethanol (99.5) and acetic acid (100) (67 : 25 : 8). Stain the gel in the staining solution, previously warmed to 60 °C, for 10 minutes. After staining is completed, place the gel in the destaining solution, a mixture of water, ethanol (99.5) and acetic acid (100) (67 : 25 : 8), until the excess staining reagents are sufficiently removed.

No band other than the principal band obtained from the test solution (a) is more intense than the principal band obtained from the test solution (b) (NMT 5%).

System suitability

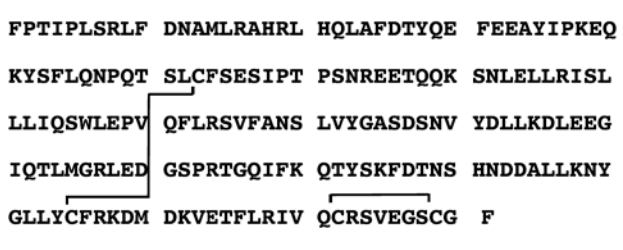
System performance: All bands obtained from the standard solution (b), which is the isoelectric point calibration solution, is well separated from each other. With the standard solution (a), the principal band appears at the isoelectric point of about 5.0, and the minor band at the isoelectric point of about 4.8.

Bacterial endotoxins Less than 5 EU/mg of somatropin.

Assay Use the chromatograms obtained from the test for dimer and the related substance of higher molecular weight to calculate the amount of somatropin in the test sample based on the labeled amount of somatropin RS.

Packaging and storage Preserve in hermetic containers (-20 ℃). Avoid repeated freezing and thawing.

Somatropin for Injection (rDNA)
주사용 소마트로핀 (유전자재조합)



$C_{990}H_{1528}N_{262}O_{300}S_7$: 22125

Somatropin for Injection (rDNA) is a preparation for injection, which is reconstituted before use. Somatropin for Injection (rDNA), calculated on the anhydrous basis, contains NLT 89.0% and NMT 105.0% of the labeled amount of somatropin. 1 mg of anhydrous somatropin ($C_{990}H_{1528}N_{262}O_{300}S_7$) is equivalent to 3.0 IU of the biological activity.

Description Somatropin for Injection (rDNA) occurs as a white powder.

Identification (1) Perform the test as directed in (1) or (2) of the Charged variants under the Purity.

(1-1) ***Capillary electrophoresis***—Proceed as directed in the test method described in the Capillary electrophoresis of charged variants under the Purity, after applying the following modification; only a single major peak corresponding to somatropin is observed.

Injection: Inject the test solution (b) for at least 3 seconds under pressure or under vacuum, and then inject the capillary electrophoresis buffer solution for 1 second.

(1-2) ***Isoelectric focusing***—Examine the eletropherograms of isoelectric focusing described in the test method for charged variants under the Purity; the position of the principal band obtained from the test solution (a) is the same as that obtained from the standard solution (a).

(2) ***Reverse-phase liquid chromatography***—Examine the chromatograms of the related substances obtained as directed under the Related substances; the retention time and size of the major peak obtained from the test solution is similar to those obtained from the standard solution.

(3) ***Peptide map***

Test solution: Dilute Somatropin for Injection (rDNA) with 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) to adjust the concentration of somatropin to 2.0 mg/mL. Transfer about 1.0 mL of this solution into a tube made of a suitable material, such as polypropylene. Prepare 1 mg/mL trypsin solution diluted with 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) and add 30 μL of this solution to the test sample. Cap the tube and allow to react on a water bath at 37 °C for 4 hours. Remove the tube from the water bath and stop the reaction immediately by an appropriate method, such as freezing. Analyze the sample immediately using an automatic injector, while maintaining the temperature at 2 to 8 °C.

Standard solution: Prepare the standard solution using somatropin RS, concurrently with the test solution in the same manner.

Perform the test with 100 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions; the profiles of the chromatograms obtained from the test solution and the standard solution are the same.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 214 nm)

Column: A stainless steel tube about 4.6 mm in internal diameter and about 25 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (about 5 μm to10 μm in particle diameter).

Column temperature: A constant temperature of about 30 °C.

Mobile phase: Control the mobile phases A and B stepwise as directed in the following table or via the concentration gradient.

Mobile phase A: A mixture of trifluoroacetic acid and water (999 : 1)

Mobile phase B: A mixture of acetonitrile, water and trifluoroacetic acid (899 : 100 : 1)

Time (min)	Mobile phase A (vol%)	Mobile phase B (vol%)	Chromatographic conditions
0 - 20	100 → 80	0 → 20	Linear concentration gradient
20 - 40	80 → 75	20 → 25	Linear concentration gradient
40 - 65	75 → 50	25 → 50	Linear concentration gradient
65 - 70	50 → 20	50 → 80	Linear concentration gradient

Flow rate: 1 mL/min

(4) **Size-exclusion liquid chromatography**—Examine the chromatograms obtained from the Assay; the retention time and size of the major peak obtained from the chromatogram of the test solution is similar to those obtained from the chromatogram of the standard solution.

Purity (1) ***Related substances***

Test solution: Dilute Somatropin for Injection (rDNA) with 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) to adjust the concentration of somatropin to 2.0 mg/mL. If the concentration of the test solution is lower than this, adjust the injection volume.

Standard solution: Dilute somatropin RS with 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) to adjust the concentration of protein to 2.0 mg/mL.

System suitability test solution: Dilute somatropin/desamido-somatropin mixture RS solution with 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) to adjust the concentration of somatropin to 2 mg/mL.

Perform the test with 20 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions; the amount of related substances is NMT 13.0%.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 220 nm)

Column: A stainless steel tube about 4.6 mm in internal diameter and about 25 cm in length, packed with butylsilyl silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 45 °C.

Mobile phase: A mixture of 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) and propanol (71 : 29)

Column equilibration: Equilibrate the column using 200 to 500 mL of a solution containing 50% acetonitrile and 0.1% trifluoroacetic acid. If necessary, repeat the equilibration process to improve the performance of the column.

Flow rate: 0.5 mL/min

System suitability

System performance: The relative retention time of somatropin in the standard solution to the peak of desamido is about 0.85 (Adjust the retention time of somatropin to about 33 minutes, if necessary, by modifying the concentration of propanol in the mobile phase). Perform the test with the system suitability test solution; the resolution between the peaks of desamido and somatropin should be NLT 1.0 with the symmetry factor of the somatropin peak being 0.9 to 1.8.

(2) ***Dimer and the related substance of higher molecular weight***

Test solution: Dilute Somatropin for Injection (rDNA) with 0.025 mol/L phosphate buffer solution (pH 7.0) to adjust the concentration of somatropin to 1.0 mg/mL.

Standard solution: Dilute somatropin RS with 0.025 mol/L phosphate buffer solution (pH 7.0) to adjust the concentration of somatropin to 1.0 mg/mL.

System suitability test solution: Place somatropin RS in an oven at 50 °C for a sufficient amount of time (usually 12 to 24 hours) to generate 1 to 2% of dimers. Dissolve the resulting content in the phosphate buffer solution (pH 7.0) and adjust the concentration of somatropin to 1.0 mg/mL.

Perform the test with 20 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions; the sum of the peak areas of any peaks detected before the major peak obtained from the test solution is NMT 6.0%.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 214 nm)

Column: A stainless steel column about 7.8 mm in internal diameter and about 30 cm in length, packed with hydrophilic silica gel for liquid chromatography, which is capable of separating proteins having a molecular weight of 5000 to 150000.

Mobile phase: A mixture of 0.063 mol/L phosphate buffer (pH 7.0) and 2-propanol (97 : 3)

Flow rate: 0.6 mL/min

System suitability

System performance: The relative retention time of the peak of the related substance with higher molecular weight to the retention time of the peak of somatropin monomer (12 to 17 minutes) from the standard solution is about 0.65. The relative retention time of the peak of somatropin dimer to the retention time of the peak of somatropin monomer (12 to 17 minutes) from the standard solution is about 0.9. The peak-to-valley ratio is NLT 2.5.

(3) ***Charged variants***—Perform the test as directed in (1) or (2).

(1) Capillary electrophoresis

Test solution (a): Dilute Somatropin for Injection (rDNA) with water to adjust the concentration of somatropin to 1 mg/mL.

Test solution (b): Mix equal volumes of the test solution (a) and the standard solution.

Standard solution: Dilute somatropin RS with water to adjust the concentration of somatropin to 1 mg/mL.

Capillary electrophoresis buffer solution: 13.2 g/L ammonium phosphate buffer solution. Adjust the pH to 6.0 with phosphoric acid, and then filter through a membrane filter.

Proceed with the test solution and the standard solution as directed under the Capillary electrophoresis according to the following operating conditions; the amount of the deamidated form is NMT 6.5%, the amount of each impurity is NMT 2.0%, and the total amount of charged variants is NMT 11.5%.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 200 nm)

Capillary: An uncoated silica capillary about 50 μm in internal diameter and about 70 cm in effective length

Column temperature: A constant temperature of about 30 °C.

Step	Solution	Time	Condition
Capillary equilibration	1 mol/L sodium hydroxide	20 min	Under pressure or under vacuum
	Purified water	10 min	
	Capillary electrophoresis buffer solution	20 min	
Between-run washing	0.1 mol/L sodium hydroxide	2 min	Under pressure or under vacuum
	Capillary electrophoresis buffer solution	6 min	
Injection	Injection of the test solution and the standard solution	3 sec	Under pressure or under vacuum
	Capillary electrophoresis buffer solution	1 sec	
Separation	Capillary electrophoresis buffer solution	80 min	217 V/cm

System suitability

System performance: The relative migration distance of the deamidated form to somatropin is 1.02 to 1.11, and the profiles of the electropherograms obtained from the standard solution and the test solution are similar. Two peaks (I1, I2) are detected before the major peak, and more than two peaks (I3, I4) are detected after the major peak. I2 exhibits a fragmented form. As a deamidated form, I4 is detected as two peaks.

(2) Isoelectric focusing

Test solution (a): Dilute Somatropin for Injection (rDNA) with 0.025 mol/L phosphate buffer solution (pH 7.0) to adjust the concentration of somatropin to 2.0 mg/mL.

Mobile phase: To 1.9 mL of 0.025 mol/L phosphate buffer (pH 7.0), add 0.1 mL of the test solution (a).

Standard solution (a): Dilute somatropin RS with 0.025 mol/L phosphate buffer solution (pH 7.0) to adjust the concentration of somatropin to 2.0 mg/mL.

Standard solution (b): Use an isoelectric point calibration solution in the pH range of 2.5 to 6.

Operating conditions

Perform the isoelectric focusing using a polyacrylamide gel in the pH range of 4.0 to 6.5. Spot 15 μL each of the test solution and the standard solution on the gel. Use a solution, in which 4.7 g of glutamic acid is dissolved in 1L of a phosphoric acid (50 g/L H_3PO_4) solution, as the anode solution and 89.1 g/L β-alanine solution as the cathode solution. Set the operating conditions to 2000 V and 25 mA for isoelectric focusing so that the voltage is maintained constant for 2.5 hours during the focusing process. At this time, the power should not exceed 25 W. After the focusing is completed, immerse the gel in a suitable volume of a solution containing 115 g/L trichloroacetic acid and 34.5 g/L sulfosalicylic acid TS for 30 minutes, and then transfer the gel to a mixture of water, ethanol (99.5) and acetic acid (100) (67 : 25 : 8) to immerse for 5 minutes. Prepare the staining solution by dissolving 1.15 g of Coomassie Brilliant Blue R-250 in 1 L of a mixture of water, ethanol (99.5) and acetic acid (100) (67 : 25 : 8). Stain the gel in the staining solution, previously warmed to 60 °C, for 10 minutes. After staining is completed, place the gel in the destaining solution, a mixture of water, ethanol (99.5) and acetic acid (100) (67 : 25 : 8), until the excess staining reagents are sufficiently removed.

No band other than the principal band obtained from the test solution (a) is more intense than the principal band obtained from the test solution (b) (NMT 6.25%).

System suitability

System performance: All bands obtained from the standard solution (b), which is the isoelectric point calibration solution, is well separated from each other. With the standard solution (a), the principal band appears at the isoelectric point of about 5.0, and the minor band at the isoelectric point of about 4.8.

Water NMT 3.0%

Sterility Meets the requirements.

Bacterial endotoxins Less than 5 EU/mg of somatropin.

Particulate contamination: Visible particles Meets the requirements.

Insoluble particulate matter in injections Meets the requirements.

Extractable volume of injections Meets the requirements.

Assay Use the chromatograms obtained from the test for dimer and the related substance of higher molecular weight to calculate the amount of somatropin in the test sample based on the labeled amount of somatropin RS.

Packaging and storage Preserve in hermetic container (2 to 8 °C).

3) Radiopharmaceuticals

2-Deoxy-2-fluoro-D-glucose (^{18}F) Injection
2-데옥시-2-플루오로-D-글루코스(^{18}F) 주사액

2-Deoxy-2-fluoro-*D*-glucose (^{18}F) Injection is an aqueous injection containing fluorine-18 in the form of 2-deoxy-2-fluoro-*D*-glucose.

2-Deoxy-2-fluoro-*D*-glucose (^{18}F) Injection contains NLT 90.0% and NMT 110.0% of the fluorine-18 radioactivity labeled at the time of testing.

Method of preparation Obtain fluorine-18 by irradiating oxygen-18 with a proton, or by using a similar method. Then synthesize 1,3,4,6-tetraacetyl-2-deoxy-2-fluoro-β-D-glucose (^{18}F) through nucleophilic substitution reaction with 1,3,4,6-tetraacetyl-2-O-trifluoromethanesulfonyl-β-D-mannopyranose, hydrolyze to remove the acetyl group, purify, and then prepare as directed under Injections.

Description 2-Deoxy-2-fluoro-*D*-glucose (^{18}F) Injection occurs as a clear, colorless liquid.

Identification (1) With 2-Deoxy-2-fluoro-*D*-glucose (^{18}F) Injection, perform the test as directed in the spectroscopy using gamma-ray spectrometer under the Gamma-ray spectrometry; confirm the peak at 0.511 MeV.

(2) 2-Deoxy-2-fluoro-*D*-glucose (^{18}F) Injection has a half-life of 105 to 115 minutes.

(3) Confirm according to the Purity (1).

pH Between 4.5 and 8.5.

Purity (1) ***Radiochemical impurity***—Perform the test with 2-Deoxy-2-fluoro-*D*-glucose (^{18}F) Injection by thin-layer chromatography using a mixture of acetonitrile and water (95 : 5) (as a developing solvent); the radioactivity in parts other than 2-deoxy-2-fluoro-D-glucose (^{18}F) spots (R_f value of about 0.4) is NMT 10% of the total radioactivity. Prepare the thin-layer chromatographic plates made of silica gel for thin-layer chromatography.

(2) ***Heteronuclide***—Perform the test according to Identification (1) with 2-Deoxy-2-fluoro-*D*-glucose (^{18}F) Injection; no peaks other than at 0.511 MeV and at 1.02 MeV appear.

(3) ***Chemical impurity***—Depending on the manufacturing method, it may contain a different chemical impurity. When synthesized using aminopolyether (Cryptopix), perform the test as directed under the Thin Layer Chromatography as follows. Using 2-Deoxy-2-fluoro-*D*-glucose (^{18}F) Injection as the test solution, separately dissolve 4,7,13,16,21,24-hexaoxa-1,10-diazabicyclo[8,8,8]hexacosane RS in normal saline solution to make about 50 μg/mL, and use this solution as the standard solution. Spot the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of methanol and ammonia water (9 : 1) (as a developing solvent), and air-dry the plate. Place the plate in a suitable container containing iodine crystal, cover with a lid, and allow to stand for 3 to 5 minutes; if the spots from the test solution have the same R_f value as those in the standard solution, they are not more intense than those from the standard solution.

Sterility Meets the requirements.

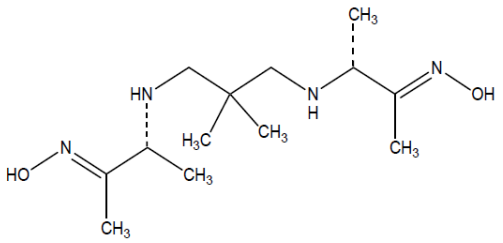
Bacterial endotoxins NMT 175/V EU per each mL of 2-Deoxy-2-fluoro-*D*-glucose (^{18}F) Injection. However, V is the maximum recommended amount per mL during the effective time.

Particulate contamination: Visible particles Meets the requirements.

Assay Perform the test with 2-Deoxy-2-fluoro-*D*-glucose (^{18}F) Injection as directed in Assay under the Gamma-ray spectrometry.

Packaging and storage Preserve in shielded hermetic containers.

Exametazime
엑사메타짐



$C_{13}H_{28}N_4O_2$: 272.39

(2*E*,2'*E*,3*R*,3'*R*)-*rel*-3,3'-[(2,2-Dimethyl-1,3-propanediyl)diimino]bis-2-butanone 2,2'-dioxime, [*105613-48-7*]

Exametazime, when dried, contains NLT 97.0% and NMT 101.0% of exametazime ($C_{13}H_{28}N_4O_2$).

Description Exametazime occurs as a white or milky white crystalline powder.
It is freely soluble in methanol and very slightly soluble in ether. It is slightly soluble in water.

Melting point Between 130 ℃ and 133 ℃

Identification Determine the infrared spectra of Exametazime and exametazime RS as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

Purity (1) ***Heavy metals***—Proceed with 1.0 g of Exametazime according to Method 2, and perform the test. Prepare the control solution with 5.0 mL of lead standard solution (NMT 50 ppm).

(2) ***Arsenic***—Proceed with 0.3 g of Exametazime according to Method 3 and perform the test. Prepare the control solution with 3.0 mL of arsenic standard solution (NMT 10 ppm).

(3) ***Boron***—Weigh accurately about 0.1 g of Exametazime, previously dried, dissolve in 1 mL of 10% sodium hydroxide solution, and evaporate to dryness for 15 hours using an infrared lamp. Heat quickly on a burner until the organic substances are decomposed and the reaction is complete. After cooling, add 0.1 mL of water, and then add 3 mL of curcumin TS. To prevent it

from boiling over, pour it around the plate. Heat it using a low-temperature Bunsen burner, and dissolve the residue (black carbon residue) by stirring well with a plastic rod. Allow to stand at room temperature for about 10 minutes, and make sure that it is completely dissolved. Using two platinum dishes, put 0.1 mL of 50 μg/mL boron standard solution into one of them, and 0.1 mL of water into the other one. Slowly add 3 mL of curcumin TS to each, while continuously stirring. After then, stir for 1 minute. Allow to stand at room temperature for about 15 minutes. Prepare three 100-mL volumetric flasks and a plastic filter, slowly add 10 mL of methanol to the first dish, stir the content to dilute the acid in the methanol layer, and then transfer the upper layer to the first flask. Continue washing the plate with methanol until the washings become clear, and then transfer them to the flask. Repeat the process for the second and third dishes. Add methanol to the content of the flask up to the gauge line, and mix well. Filter the test solution, and determine the absorbance of the test solution and standard solution at 500 nm as directed under the Ultraviolet-visible Spectroscopy; the boron content in the test solution is NMT 50 μg/g.

$$\text{Boron content in the test solution (μg/g Exametazime)} = \frac{Absorbance\ at\ the\ test\ solution\ (A_T)}{Absorbance\ at\ the\ standard\ solution\ (A_S)} \times 50$$

Loss on drying NMT 0.5% (105 ℃, 6 hours).

Residue on ignition NMT 0.5% (1 g).

Assay Weigh accurately about 100 mg of Exametazime, previously dried, dissolve in 50 mL of acetic acid, and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration under the Titrimetry). Perform a blank test in the same way and make necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 13.620 mg of $C_{13}H_{28}N_4O_2$

Packaging and storage Preserve in well-closed containers.

Gallium (^{67}Ga) Citrate Injection
시트르산갈륨(^{67}Ga) 주사액

Gallium (^{67}Ga) Citrate Injection is an aqueous injection containing gallium-67 in the form of gallium citrate.

Gallium (^{67}Ga) Citrate Injection contains NLT 90.0% and NMT 110.0% of the gallium-67 radioactivity labeled at the time of testing.

Method of preparation React gallium chloride (^{67}Ga) with sodium citrate solution to produce gallium citrate (^{67}Ga), and then prepare Gallium (^{67}Ga) Citrate Injection as directed under Injections.

Description Gallium (^{67}Ga) Citrate Injection occurs as a clear and colorless or pale red solution.

Identification (1) With Gallium (^{67}Ga) Citrate Injection, perform the test as directed in the spectroscopy using gamma-ray spectrometer under Gamma-ray spectrometry; confirm the peaks at 0.093, 0.185, 0.300 and 0.394 MeV.

(2) Confirm according to Purity (1).

pH Between 6.0 and 8.0

Purity (1) ***Radiochemical impurity***—With Gallium (^{67}Ga) Citrate Injection, develop the filter paper with a mixture of 0.1 mol/L sodium citrate and ethanol (95) (5 : 3) (as a developing solvent) for about 3 hours as directed under the Paper chromatography; the radioactivity other than the spot of gallium citrate (^{67}Ga) (R_f values of about between 0.7 and 0.9) is NMT 2% of the total radioactivity.

(2) ***Zinc***—Add 0.75 mL of water and 1 drop of thymol blue TS to 0.05 mL of Gallium (^{67}Ga) Citrate Injection, shake well to mix, adjust pH to 8.5 with 0.01 mol/L ammonia, and use this solution as the test solution. Add 0.05 mL of sodium diethyldithiocarbamate TS and 1 mL of dithizone-carbon tetrachloride TS in this order, and shake vigorously to mix each time. Then add 1 mL of sodium sulfide TS, and shake vigorously to mix. Allow to stand for several minutes; the color of the lower layer is not more intense than that of the control solution. Prepare the control solution with 0.05 mL of zinc standard solution in the same manner as in the preparation of the test solution (NMT 5 ppm).

(3) ***Iron***—Take 1.0 mL of Gallium (^{67}Ga) Citrate Injection in a Nessler tube, add 6 mL of dilute nitric acid and water to make 20 mL, add 50 mg of ammonium peroxydisulfate and 5 mL of ammonium thiocyanate TS, shake to mix each time, then add 15 mL of 1-butanol, and shake vigorously to mix for 30 seconds; the color of the 1-butanol layer is not more intense than that of the control solution. Prepare the control solution as follows: to 2.0 mL of the iron standard solution, add 6 mL of dilute nitric acid and water to make 20 mL, and proceed in the same manner as in the preparation of the test solution (NMT 20 ppm).

(4) ***Heavy metals***—Take 2.0 mL of Gallium (^{67}Ga) Citrate Injection, add 0.2 mL of dilute acetic acid and water to make 5 mL, add 1 drop of sodium sulfide TS, mix and allow to stand for 5 minutes; the color of the solution is not more intense than that of the control solution. Prepare the control solution as follows: take 0.1 mL of lead standard solution, add 0.2 mL of dilute acetic acid and water to make 5 mL, and proceed in the same manner as in the preparation of the test solution (NMT 0.5 ppm).

Sterility Meets the requirements.

Bacterial endotoxins NMT 175/V EU per each mL of Gallium (^{67}Ga) Citrate Injection. However, V is the maximum recommended amount per mL during the effective time.

Particulate contamination: Visible particles Meets the requirements.

Assay Perform the test with Gallium (^{67}Ga) Citrate Injection as directed in the Assay under the Gamma-ray spectrometry.

Packaging and storage Preserve in shielded hermetic containers.

Hexakis(2-methoxyisobutylisonitril) Technetium (^{99}mTc) Injection
헥사키스(2-메톡시이소부이소니트릴)테크네튬(^{99m}Tc) 주사액

Hexakis (2-methoxyisobutylisonitril) Technetium (^{99m}Tc) Injection is an aqueous injection, which contains technetium-99m in the form of hexakis (2-methoxyisobutylisonitril) technetium.

Hexakis (2-methoxyisobutylisonitril) Technetium (^{99m}Tc) Injection contains NLT 90.0% and NMT 110.0% of technetium-99m radioactivity labeled at the time of the test.

Method of preparation Mix sodium pertechnetate (^{99m}Tc) injection, tin(II) chloride dihydrate injection which is separately prepared as directed under Injections and tetrakis (2-methoxyisobutylisonitril) technetium and Heat. Then prepare Hexakis (2-methoxyisobutylisonitril) Technetium (^{99m}Tc) Injection as directed under Injections.

Description Hexakis (2-methoxyisobutylisonitril) Technetium (^{99m}Tc) Injection occurs as a clear and colorless liquid.

Identification (1) Perform the test with Hexakis (2-methoxyisobutylisonitril) Technetium (^{99m}Tc) Injection as directed in the spectroscopy using gamma-ray spectrometer under the Gamma-ray spectrometry; confirm the peak at 0.141 MeV.

(2) Perform the test as directed under the Purity.

pH Between 5.0 and 6.0

Purity ***Radiochemical impurity***—Develop the plate with Hexakis (2-methoxyisobutylisonitril) Technetium (^{99m}Tc) Injection as directed under the Thin Layer Chromatography using a mixture of acetonitrile, methanol, 0.5 mol/L ammonium acetate TS and tetrahydrofuran (4 : 3 : 2 : 1) (as a developing solvent) to a distance of about 10 cm; the radioactivity other than the spot of hexakis (2-methoxyisobutylisonitril) technetium (^{99m}Tc) (R_f value: approximately 0.35 to 0.55) is NMT 10% of the total radioactivity. In this case, prepare the thin-layer chromatographic plate using octadecylsilanized silica gel for thin-layer chromatography.

Sterility Meets the requirements.

Particulate contamination: Visible particles Meets the requirements.

Assay Perform the test with Hexakis (2-methoxyisobutylisonitril) Technetium (^{99m}Tc) Injection as directed in the Assay under the Gamma-ray spectrometry.

Packaging and storage Preserve in shielded hermetic containers.

3-Iodobenzylguanidine (^{131}I) Injection
3-요오도벤질구아니딘(^{131}I) 주사액

3-Iodobenzylguanidine (^{131}I) Injection is an aqueous injection containing iodine-131 in the form of 3-iodobenzylguanidine.

3-Iodobenzylguanidine (^{131}I) Injection contains NLT 90.0% and NMT 110.0% of iodine-131 radioactivity labeled at the time of testing.

The specific radioactivity of 3-Iodobenzylguanidine (^{131}I) Injection is between 111 MBq and 185 MBq per mg of 3-iodobenzylguanidine at the time of testing.

Method of preparation Substitute the iodine atom of 3-iodobenzylguanidine with iodine-131, purify by removing unreacted iodine-131 and liberated iodine, and prepare as directed under Injections.

Description 3-Iodobenzylguanidine (^{131}I) Injection occurs as a clear and colorless liquid.

Identification (1) With 3-Iodobenzylguanidine (^{131}I) Injection, perform the test as directed in the spectroscopy using gamma-ray spectrometer under the Gamma-ray spectrometry; confirm the peak at 0.364 MeV.

(2) Confirm according to the Purity.

pH Between 4.0 and 5.0

Purity ***Radiochemical impurity***—Dissolve 0.5 g of sodium iodide, 1.0 g of sodium iodate, and 5.0 g of sodium bicarbonate in water to make 1 L. Develop the plate using an appropriate amount of this solution as a carrier and an 80% methanol solution (as a developing solvent) to a distance of about 10 cm as directed under the Thin Layer Chromatography; the radioactivity other than the 3-iodobenzylguanidine (^{131}I) spot is NMT 5% of the total radioactivity. However, for the 3-iodobenzylguanidine (^{131}I) spot, develop the plate with an appropriate amount of normal saline solution of 3-iodobenzylguanidine sulfate (1 in 200) in the same manner, spray the plate with thymine-1-naphthol TS repeatedly, dry, and spray diluted sodium hypochlorite TS (1 in 5); confirm the color change. However, thin-layer chromatographic plates are made using octadecylsilanized silica gel for thin-layer chromatography.

Sterility Meets the requirements.

Bacterial endotoxins NMT 175/V EU per each mL of 3-Iodobenzylguanidine (^{131}I) Injection. However, V is the maximum recommended amount per mL during the effective time.

Particulate contamination: Visible particles Meets the requirements.

Assay Perform the test with 3-Iodobenzylguanidine (^{131}I) Injection as directed in the Assay under the Gamma-ray spectrometry.

Packaging and storage Preserve in shielded hermetic containers below −20 °C.

N-Isopropyl-4-iodoamphetamine (^{123}I) Hydrochloride Injection
N-이소프로필-4-요오도암페타민(^{123}I)염산염 주사액

N-Isopropyl-4-iodoamphetamine (^{123}I) Hydrochloride

Injection is an aqueous injection containing iodine-123 in the form of *N*-isopropyl-4-iodoamphetamine hydrochloride.

N-Isopropyl-4-iodoamphetamine (^{123}I) Hydrochloride Injection contains NLT 90.0% and NMT 110.0% of the iodine-131 radioactivity labeled at the time of testing.

Method of preparation Produce cesium-123 by irradiating protons to xenon-124, and obtain iodine-123 from the decay of xenon-123. Substitute them with the iodine atom of *N*-isopropyl-4-iodoamphetamine hydrochloride, and then prepare as directed under Injections.

Description *N*-Isopropyl-4-iodoamphetamine (^{123}I) Hydrochloride Injection occurs as a clear and colorless liquid.

Identification (1) With *N*-Isopropyl-4-iodoamphetamine (^{123}I) Hydrochloride Injection as is, or a solution obtained by dissolving it in an appropriate amount of warm water, perform the test as directed in the spectroscopy using gamma-ray spectrometer under the Gamma-ray spectrometry; confirm the peak at 0.159 MeV.

(2) Identify according to the Purity (1).

pH Between 4.0 and 7.0.

Purity (1) ***Radiochemical impurity***—Perform the test with *N*-Isopropyl-4-iodoamphetamine (^{123}I) Hydrochloride Injection as directed under the Thin Layer Chromatography using a mixture of chloroform, methanol, and acetic acid (100) (84 : 15 : 1) (as a developing solvent); the radioactivity other than the spot of *N*-isopropyl-4-iodoamphetamine (^{123}I) hydrochloride is NMT 5% of the total radioactivity. Meanwhile, for the spot of *N*-isopropyl-4-iodoamphetamine (^{123}I) hydrochloride, perform the test with *N*-isopropyl-4-iodoamphetamine hydrochloride solution (1 in 100) in the same manner, and expose the thin-layer chromatographic plate to iodine vapor; check with coloring. However, prepare the thin-layer chromatographic plates using silica gel for thin-layer chromatography.

(2) ***Heteronuclide***—With *N*-Isopropyl-4-iodoamphetamine (^{123}I) Hydrochloride Injection, determine the radioactivity as directed in the spectroscopy using gamma-ray spectrometry under the Gamma-ray spectrometry; the radioactivity other than iodine-123 labeled at the time of test is NMT 0.3% of the total radioactivity.

(3) ***Formic acid***—Take 0.1 mL of *N*-Isopropyl-4-iodoamphetamine (^{123}I) Hydrochloride Injection, and add 0.1 mL of 2 mol/L hydrochloric acid and about 10 mg of magnesium powder. After the completion of hydrogen generation, completely dissolve the magnesium powder in 2.0 mL of diluted sulfuric acid (1 in 2), add 1.0 mL of chromotropic acid TS, and heat; the dark purple color that appears is not more intense than that of the following control solution (NMT 1 mg/mL).

Control solution: Weigh 1.0 g of formic acid and add water for injection to make a concentration of 1 mg/mL. Take 0.1 mL of this solution and proceed in the same manner.

(4) ***Copper***—To 0.5 mL of *N*-Isopropyl-4-iodoamphetamine (^{123}I) Hydrochloride Injection, add 1 drop each of citric acid-sodium edetate TS and thymol blue TS, and then add ammonia TS (3 in 10) until the solution turns blueish green. To this solution, add 1 drop of sodium diethyldithiocarbamate solution (1 in 100) and 0.2 mL of carbon tetrachloride, and shake to mix; the yellowish brown color appearing in the carbon tetrachloride layer is not more intense than the following control solution (NMT 1 μg/mL).

Control solution: Weigh 0.157 g of copper(II) sulfate pentahydrate, add 1 drop of sulfuric acid, and dissolve in water to make 200 mL. Take 1.0 mL of this solution and add water to make 200 mL. Take 0.5 mL of this solution and proceed in the same manner.

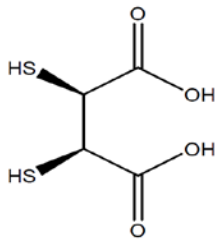
Sterility Meets the requirements.

Particulate contamination: Visible particles Meets the requirements.

Assay Perform the test with *N*-Isopropyl-4-iodoamphetamine (^{123}I) Hydrochloride Injection and determine the radioactivity as directed in Assay using the ionization chamber under the Gamma-ray spectrometry.

Packaging and storage Preserve in shielded hermetic containers.

Meso-2,3-dimercaptosuccinic Acid
디메르캅토숙신산



$C_4H_6O_4S_2$: 182.22

(2R,3S)-*rel*-2,3-Dimercapto-butanedioic acid, [*304-55-2*]

Meso-2,3-dimercaptosuccinic Acid is in the meso form, and contains NLT 99.0 and NMT 101.0% of meso-2,3-dimercaptosuccinic acid ($C_4H_6O_4S_2$), calculated on the dried basis.

Description Meso-2,3-dimercaptosuccinic Acid occurs as a colorless to white crystal and is odorless.
It is practically insoluble in water, ethanol and ether, and freely soluble in alkaline solution.
Melting point: Between 205 and 211 °C

Identification (1) Dissolve 1 g of Meso-2,3-dimercaptosuccinic Acid in 100 mL of ammonia TS, and adjust the pH to about 7 with dilute hydrochloric acid. Then, take 5 mL of this solution and add 2 to 3 drops of iron(III) chloride T; it exhibits a dark brown color, and slowly fades when allowed to stand.

(2) Dissolve 1 g of Meso-2,3-dimercaptosuccinic Acid in 500 mL of 0.1 mol/L sodium hydroxide TS, then take 20 mL of this solution, add 0.2 mL of Sodium pentacyanonitrosylferrate(III) TS, and shake to mix; it immediately exhibits a violet color.

Purity (1) ***Heavy metals***—Proceed with 1.0 g of Meso-2,3-dimercaptosuccinic Acid according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 20 ppm).

(2) ***Arsenic***—Proceed with 0.5 g of Meso-2,3-dimercaptosuccinic Acid according to Method 3 and perform the test (NMT 4 ppm).

Loss on drying NMT 0.10% (1 g, 105 °C, 4 hours).

Residue on ignition NMT 0.10%.

Sterility Meets the requirements.

Assay Weigh accurately about 10 mg of Meso-2,3-dimercaptosuccinic Acid, dissolve in 1 mL of ammonia water, and use this solution as the test solution. Add 0.2 mol/L nitric acid to this solution to adjust the pH to about 5, then add 20 mL of dithizone (0.0005% chloroform solution), and titrate with 0.01 mol/L silver nitrate VS. However, the endpoint of titration is when the color of the dithizone layer changes from dark green to orange. Perform a blank test in the same manner and make any necessary correction.

Amount (%) of meso-2,3-dimercaptosuccinic acid ($C_4H_6O_4S_2$)

$$= \frac{10.846 \times (A - B) \times f}{\text{Amount (mg) of sample taken}} \times 100$$

B: Amount (mL) of 0.01 mol/L silver nitrate VS consumed for this test.

B: Amount (mL) of 0.01 mol/L silver nitrate VS consumed for the blank test.

f: Titrimetric factor of 0.01 mol/L silver nitrate VS.

Packaging and storage Preserve in well-closed containers.

Methylenediphosphonic Acid
메틸렌디포스폰산

$H_2O_3P\text{-}CH_2\text{-}PO_3H_2$

$CH_6O_6P_2$: 176.00

P,*P'*-Methylenebis-phosphonic acid, [*1984-15-2*]

Methylenediphosphonic Acid, when dried, contains NLT 99.0% and NMT 101.0% of methylenediphosphonic acid ($CH_6O_6P_2$).

Description Methylenediphosphonic Acid occurs as a white crystalline powder and is freely soluble in water.

Identification Dissolve 50 mg of Methylenediphosphonic Acid in 10 mL of sulfuric acid, take 1 mL of this solution, and place it in a test tube with a stopper. Install a Dimroth-type cooling tube, heat until white fumes evolve, and boil for 30 minutes. Cool this solution, add water to make 3 mL, and add 8 mol/L sodium hydroxide TS to adjust the pH to between 5 and 7. Take 1 mL of this solution in a test tube, add 1 mL of ammonium molybdate acetic acid, and heat; a yellow precipitate is formed and it disappears when 1 mL of sodium hydroxide TS added.

Melting point Between 200 °C and 203 °C.

Purity (1) ***Clarity and color of solution***—Dissolve 1 g of Methylenediphosphonic Acid in 20 mL of water; the solution is clear and colorless.

(2) ***Heavy metals***—Proceed with 1.0 g of Methylenediphosphonic Acid according to Method 1 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 20 ppm).

(3) ***Arsenic***—Proceed with 0.5 g of Methylenediphosphonic Acid according to Method 3 and perform the test (NMT 4 ppm).

Loss on drying NMT 1.0% (1 g, 105 °C, 4 hours).

Assay Weigh accurately about 0.20 g of Methylenediphosphonic Acid, previously dried, dissolve in 10 mL of water, and titrate with 0.1 mol/L sodium hydroxide VS (indicator: bromocresol green). Perform a blank test in the same way and make necessary correction.

Each mL of 0.1 mol/L sodium hydroxide VS
= 8.800 mg of $CH_6O_6P_2$

Packaging and storage Preserve in well-closed containers.

Sestamibi
세스타미비

$C_{24}H_{44}NBCuF_4N_4O_4$: 602.97

Tetrakis(2-methoxy-isobutyl isonitrile) copper(I) tetrafluoroborate, [*109581-73-9*]

Sestamibi, when dried, contains NLT 99.0% and NMT 101.0% of sestamibi ($C_{24}H_{44}NBCuF_4N_4O_4$).

Description Sestamibi occurs as a white crystal or crystalline powder.
It is soluble in acetone, chloroform, dichloromethane, ethanol or methanol and insoluble in ether, pentane, or cyclohexane.

Identification (1) ***Tetrafluoroborate***—Dissolve 50 mg of Sestamibi in 50 mL of water, take 1 mL of this solution in a test tube, add 0.35 mL of strong ammonia solution, and add 0.25 mL of tetraphenylarsonium chloride; a white crystalline precipitate is formed within 5 minutes.

(2) ***Copper***—Dissolve 0.1 g of Sestamibi in 5 mL of water, and add 5 drops of nitric acid to make it acidic. After 30 minutes, spot 1 drop of this solution on a thin-layer chromatographic plate made of silica gel (with fluorescent indicator) for thin layer chromatography, add 1 drop of 20% malonic acid solution to dry, and then add 1 drop of 1% rubeanic acid solution; it turns dark green.

(3) ***Sestamibi***—Determine the infrared spectra of Sestamibi and sestamibi RS as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

Loss on drying NMT 1.0% (between 1 g and 2 g, in vacuum, 24 hours).

Assay Weigh accurately about 10 mg each of Sestamibi and sestamibi RS, dissolve in the water to make exactly 10 mL, and use these solutions as the test solution and the standard solution, respectively. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas, A_T and A_S, of sestamibi in each solution.

Amount (mg) of sestamibi ($C_{24}H_{44}NBCuF_4N_4O_4$)
= Amount (mg) of sestamibi RS× $\frac{A_T}{A_S}$

Operating conditions

Detector: An ultraviolet absorption photometer

(wavelength: 225 nm)

Column: A stainless steel column about 4 mm in internal diameter and 25 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

Mobile phase: A mixture of methanol and 7.5 mmol/L ammonium sulfate (70 : 30).

Flow rate: 1.5 mL/min

Packaging and storage Preserve in well-closed containers.

Sodium Chromate (^{51}Cr) Injection
크롬산나트륨(^{51}Cr) 주사액

Sodium Chromate (^{51}Cr) Injection contains is an aqueous injection and contains chromium-51 in the form of sodium chromate.

Sodium Chromate (^{51}Cr) Injection contains NLT 90.0% and NMT 110.0% of chromium-51 radioactivity labeled at the time of the test.

The specific radioactivity of Sodium Chromate (^{51}Cr) Injection is NLT 370 MBq per mg of sodium chromate.

For Sodium Chromate (^{51}Cr) Injection, the Extractable volume of Injections and the Insoluble particulate matter in injections are not applicable.

Method of preparation Prepare Sodium Chromate (^{51}Cr) Injection as directed under Injections, with sodium chromate (^{51}Cr), previously purified.

Description Sodium Chromate (^{51}Cr) Injection occurs as a clear and colorless to pale yellow liquid. It has no odor or odor due to preservatives.

Identification (1) Perform the test with Sodium Chromate (^{51}Cr) Injection as directed in the spectroscopy using gamma-ray spectrometer under the Gamma-ray spectrometry; confirm the peak at 0.320 MeV.

(2) Perform the test as directed under the Purity.

pH Between 5.5 and 8.0

Purity ***Radiochemical impurity***—Develop the plate with Sodium Chromate (^{51}Cr) Injection as directed under the Paper chromatography for 6 hours, using 1 drop of sodium chromate solution (1 in 10) (as a carrier) and a mixture of water, methanol, 1-butanol and benzene (50 : 20 : 10 : 1) (as a developing solvent); the radioactivity other than sodium chromate (^{51}Cr) spot is NMT 10% of the total radioactivity. In this case, confirm the sodium chromate (^{51}Cr) spot by its color.

Sterility Meets the requirements.

Bacterial endotoxins NMT 175/V EU per each mL of Sodium Chromate (^{51}Cr) Injection. In this case, V represents the maximum recommended amount per mL during the effective time.

Particulate contamination: Visible particles Meets the requirements.

Assay (1) Perform the test with Sodium Chromate (^{51}Cr) Injection as directed in the Assay under the Gamma-ray spectrometry.

(2) Weigh accurately a certain amount of Sodium Chromate (^{51}Cr) Injection, dissolve in 1.0 mL of 1,5-diphenylcarbohydrazide in 8% sulfuric acid solution (2 in 5), 0.4 mL of dilute sulfuric acid and water to make 10.0 mL, shake well to mix, allow to stand for about 20 minutes, and use this solution as the test solution. Proceed with between 1.0 mL and 6.0 mL of potassium chromate standard solution in the same manner as in the preparation of the test solution, and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Ultraviolet-visible Spectroscopy and determine the absorbances at the wavelength 550 nm. In this case, use water as the blank. Determine the amount of sodium chromate in the test solution using the calibration curve obtained from the standard solution, and calculate the specific radioactivity from the radioactivity determined in (1).

Packaging and storage Preserve in shielded hermetic containers in a cold place.

Sodium Iodide (^{123}I) Injection
요오드화나트륨(^{123}I) 주사액

Sodium Iodide (^{123}I) Injection contains iodine-123 in the form of sodium iodide. Sodium Iodide (^{123}I) Injection contains NLT 90.0% and NMT 110.0% of the iodine-123 radioactivity labeled at the time of testing.

Method of preparation (1) Separate the iodine-123 produced by irradiating tellium-124 with protons, convert into sodium iodide (^{123}I), purify, and prepare Sodium Iodide (^{123}I) Injection as directed under Injections.

(2) Separate cesium-123 produced by irradiating xenon-124 with protons, and separate iodine-123 obtained for the decay of xenon-123, convert into a form of sodium iodide (^{123}I), purify, and prepare Sodium Iodide (^{123}I) Injection as directed under Injections.

Description Sodium Iodide (^{123}I) Injection occurs as a clear colorless and odorless liquid.

Identification (1) With the appropriate amount of Sodium Iodide (^{123}I) Injection, perform the test as directed in the spectroscopy under the Gamma-ray spectrometry; the maximum peak is observed at 0.159 MeV.

(2) Perform the test according to Purity (1); maximum radioactivity appears in iodide spots.

pH Between 7.0 and 9.0

Purity (1) ***Radiochemical impurity*—**Dissolve 0.5 g of sodium iodide, 1.0 g of sodium iodate, and 5.0 g of sodium bicarbonate in water to make 100 mL. Develop the filter paper with one drop of this solution (as a carrier) and 75% methanol (as a developing solvent) for about 1 hour as directed under the Paper chromatography; the radioactivity other than the spot of sodium iodide (^{123}I) is NMT 5% of the total radioactivity. Meanwhile, for the sodium iodide (^{123}I) spot, spray evenly starch TS, dilute acetic acid and potassium nitrite TS, respectively; confirm the coloring.

(2) ***Heteronuclide*—**With Sodium Iodide (^{123}I) Injection, determine the radioactivity as directed in the spectroscopy using gamma-ray spectrometer under the Gamma-ray spectrometry; it is NLT 95% of the iodine-123 radioactivity labeled at the time of testing

Sterility Meets the requirements.

Bacterial endotoxins NMT 175/V EU per each mL of Sodium Iodide (^{123}I) Injection. However, V is the maximum recommended amount per mL during the effective time.

Particulate contamination: Visible particles Meets the requirements.

Assay Weigh accurately a certain amount of Sodium Iodide (^{123}I) Injection, and perform the test as directed in the Assay under the Gamma-ray spectrometry

Packaging and storage Preserve in shielded hermetic containers.

Sodium Iodide (^{131}I) Capsules
요오드화나트륨(^{131}I) 캡슐

Sodium Iodide (^{131}I) Capsules is capsule preparations containing iodine-131 in the form of sodium iodide.

Sodium Iodide (^{131}I) Capsules contain NLT 90.0% and NMT 110.0% of the iodine-131 radioactivity labeled at the time of testing.

Method of preparation Prepare Sodium Iodide (^{131}I) Capsules as directed under Capsules, with Sodium Iodide (^{131}I) Solution.

Identification (1) With 1 capsule of Sodium Iodide (^{131}I) Capsules as is, or a solution dissolving 1 capsule of Sodium Iodide (^{131}I) Capsules in appropriate volume of warm water, perform the test as directed in the spectroscopy using gamma-ray spectrometer under the Gamma-ray spectrometry; confirm the peak at 0.364 MeV.

(2) Identify according to the Purity (2).

Purity (1) ***Carrier*—**Dissolve 1 capsule of Sodium Iodide (^{131}I) Capsules in 6 mL of warm water, add 2 to 3 drops of iron(III) chloride TS and 1 mL of chloroform, shake to mix, and allow to stand; the chloroform layer is colorless.

(2) ***Radiochemical impurity*—**Dissolve 1 capsule of Sodium Iodide (^{131}I) Capsules in an appropriate amount of warm water, and perform the test as follows. Dissolve 0.5 g of sodium iodide, 1.0 g of sodium iodate and 5.0 g of sodium bicarbonate in water to make 100 mL. Develop the filter paper with 1 drop of this solution (as a carrier) and 75% methanol (as a developing solvent) as directed under the Paper chromatography for about 4 hours; the radioactivity of the iodate spot is NMT 5% of the total radioactivity of the iodide spot, and for the parts other than the spots of iodide and iodate, some radioactivity can be recognized at the origin, no radioactivity is observed in other parts. However, for the spots of iodide and iodate, perform the test in the same manner using the above carrier as a sample, and confirm according to the following procedure. Dry the developed filter paper, put it in a glass column, pass through hydrogen sulfide for 1 to 2 minutes, spray fluorescein sodium solution (1 in 1000), and than spray chlorine TS on the surface; iodide and iodic acid exhibit colors. Spray fluorescein sodium solution (1 in 1000) on the developed filter paper without passing through hydrogen sulfide, and then spray chlorine TS on the surface; only the iodide exhibits a color.

Disintegration Meets the requirements.

Uniformity of dosage units Meets the requirements.

Assay With 1 capsule of Sodium Iodide (^{131}I) Capsules as is, or a solution dissolving Sodium Iodide (^{131}I) Capsules in appropriate volume of warm water, and perform the test as directed in the Assay under the Gamma-ray spectrometry.

Packaging and storage Preserve in shielded hermetic containers in a cold place.

Sodium Iodide (^{131}I) Solution
요오드화나트륨(^{131}I) 액

Sodium Iodide (^{131}I) Solution is liquid preparations containing iodine-131 in the form of sodium iodide.

Sodium Iodide (^{131}I) Solution contains NLT 90.0% and NMT 110.0% of the iodine-131 radioactivity labeled at the time of testing.

Method of preparation Purify sodium iodide (^{131}I) and prepare Sodium Iodide (^{131}I) Solution as directed under Liquids.

Description Sodium Iodide (^{131}I) Solution occurs as a clear colorless liquid, and is odorless, but has an odor due to preservatives or stabilizers.

Identification (1) With Sodium Iodide (^{131}I) Solution, perform the test as directed in the spectroscopy using gamma-ray spectrometer under the Gamma-ray spectrometry; confirm the peak at 0.364 MeV.

(2) Confirm according to the Purity (2).

pH Between 7.0 and 10.0.

Purity (1) ***Carrier*—**Add 0.1 mL of Sodium Iodide (^{131}I) Solution to 6 mL of water, add 2 to 3 drops of iron(III) chloride TS and 1 mL of chloroform, shake to mix, and allow to stand; the chloroform layer is colorless.

(2) ***Radiochemical impurity*—**Dissolve 0.5 g of sodium iodide, 1.0 g of sodium iodate, and 5.0 g of sodium bicarbonate in water to make 100 mL. Develop the filter paper with 1 drop of this solution (as a carrier) and 75% methanol (as a developing solvent) as directed under the Paper chromatography for about 4 hours; the radioactivity of the iodate spot is NMT 5% of the total radioactivity of the iodide spot. No radioactivity other than iodide and iodate spots is identified. However, for the spots of iodide and iodate, perform the test in the same manner using the above carrier as a sample, and confirm according to the following procedure. Dry the developed filter paper, put it in a glass column, pass through hydrogen sulfide for 1 to 2 minutes, spray fluorescein sodium solution (1 in 1000), and then spray chlorine TS on the surface; iodide and iodic acid exhibit colors. Spray the fluorescein sodium solution (1 in 1000) on the developed filter paper without passing through hydrogen sulfide, and spray chlorine TS on the surface; only the iodide exhibits a color.

Assay Perform the test with Sodium Iodide (^{131}I) Solution as directed in the Assay under the Gamma-ray spectrometry.

Packaging and storage Preserve in shielded hermetic containers in a cold place.

Sodium Iodohippurate (^{131}I) Injection
요오도히푸르산나트륨(^{131}I) 주사액

Sodium Iodohippurate (^{131}I) Injection is an aqueous injection containing iodine-131 in the form of 2-sodium iodohippurate.

Sodium Iodohippurate (^{131}I) Injection contains NLT 90.0% and NMT 110.0% of the iodine-131 radioactivity labeled at the time of testing.

The Insoluble particulate matter in injections is not applied to Sodium Iodohippurate (^{131}I) Injection.

Method of preparation Substitute the iodine atom of 2-Iodohippurate with iodine-131, remove unreacted iodine-131 and liberated iodine, purify, and prepare Sodium Iodohippurate (^{131}I) Injection as directed under Injections.

Description Sodium Iodohippurate (^{131}I) Injection occurs as a clear and colorless liquid.

Identification (1) With Sodium Iodohippurate (^{131}I) Injection, perform the test as directed in the spectroscopy using gamma-ray spectrometer under the Gamma-ray spectrometry; confirm the peak at 0.364 MeV.

(2) Identify according to the Purity.

pH Between 7.0 and 9.0

Purity ***Radiochemical impurity*—** With Sodium Iodohippurate (131I) Injection, develop the filter paper with the upper layer of a mixture of acetic acid (100), benzene and water (2 : 2 : 1) (as a developing solvent) for about 3 hours as directed under the Paper chromatography; the radioactivity other than the spot of 2-Iodohippurate (131I) is NMT 5% of the total radioactivity. However, for the 2-iodohippurate (131I) spot, develop the filter paper with 2-iodohippurate in chloroform solution (1 in 100) in the same manner, then spray bromocresol green TS; confirm the coloring.

Sterility Meets the requirements.

Bacterial endotoxins NMT 175/V EU per each mL of Sodium Iodohippurate (^{131}I) Injection. However, V is the maximum recommended dose per mL during the effective time.

Particulate contamination: Visible particles Meets the requirements.

Assay Perform the test with Sodium Iodohippurate (^{131}I) Injection as directed in the Assay under the Gamma-ray spectrometry.

Packaging and storage Preserve in shielded hermetic containers in a cold place.

Sodium Pertechnetate (^{99m}Tc) Injection
과테크네튬산나트륨(^{99m}Tc) 주사액

Sodium Pertechnetate (^{99m}Tc) Injection is an aqueous injection containing technetium-99m in the form of sodium pertechnetate.

Sodium Pertechnetate (^{99m}Tc) Injection contains NLT 90.0% and NMT 110.0% of the technetium-99m radioactivity labeled at the time of testing.

Method of preparation Prepare Sodium Pertechnetate (^{99m}Tc) Injection as follows: Elute sodium pertechnetate (^{99m}Tc) from the Sodium Pertechnetate (^{99m}Tc) Injection Generator or a generator prepared accordingly, using normal saline solution, and prepare as directed under Injections.

Description Sodium Pertechnetate (^{99m}Tc) Injection occurs as a clear and colorless liquid.

Identification (1) With Sodium Pertechnetate (^{99m}Tc) Injection, perform the test as directed in the spectroscopy using gamma-ray spectrometer under the Gamma-ray spectrometry; confirm the peak at 0.141 MeV.

(2) Confirm according to Purity (1).

pH Between 4.5 and 7.0.

Purity (1) ***Radiochemical impurity***—With Sodium Pertechnetate (^{99m}Tc) Injection, develop the filter paper with 75% methanol (as a developing solvent) for about 3 hours as directed under the Paper chromatography. The radioactivity in the parts other than sodium pertechnetate (^{99m}Tc) spots (R_f values of about between 0.6 and 0.7) is NMT 5% of the total radioactivity.

(2) ***Molybdenum-99***—Take accurately a certain amount of Sodium Pertechnetate (^{99m}Tc) Injection in vial, put in a lead container of a certain thickness, and determine radioactivity of molybdenum-99 as directed in the Assay under Gamma-ray spectrometry. When measuring radioactivity as directed under Assay by a gamma-ray spectrometer, calculate the radioactivity of molybdenum-99 using the radioactivity peak at 0.739 MeV as the coefficient. The radioactivity of molybdenum-99 is NMT 0.015% of the total radioactivity.

(3) ***Aluminum***— Take 3.0 mL of Sodium Pertechnetate (99mTc) Injection and 1.5 mL of aluminum standard solution, add 2 mL and 3.5 mL of water, respectively, then add 2.4 mL each of L-ascorbic acid solution (1 in 20) (prepare just before use), shake to mix, and allow to stand for 15 minutes. Next, add 5 mL of water and ammonia water (28) to each to adjust the pH to 8, and add dilute hydrochloric acid to adjust the pH to 7. Allow to stand for 20 minutes, and use these solutions as the test solution and the standard solution, respectively. Separately, add 2.4 mL of L-ascorbic acid solution (1 in 20) (prepare just before use) to 5.0 mL of water, and use the resulting solution obtained in the same manner as a reference solution. Determine the absorbance at a layer length of 1 cm and a wavelength of 530 nm according to the Ultraviolet-visible Spectroscopy; the absorbance of the test solution is not greater than that of the standard solution (NMT 10 ppm).

Sterility Meets the requirements.

Bacterial endotoxins NMT 175/V EU per each mL of Sodium Pertechnetate (^{99m}Tc) Injection. However, V is the maximum recommended amount per mL during the effective time.

Particulate contamination: Visible particles Meets the requirements.

Assay Perform the test with Sodium Pertechnetate (^{99m}Tc) Injection as directed in the Assay under Gamma-ray spectrometry.

Packaging and storage Preserve in shielded hermetic containers.

Sodium Pertechnetate (^{99m}Tc) Injection Generator
과테크네튬산나트륨(^{99m}Tc) 주사액 제너레이터

Sodium Pertechnetate (^{99m}Tc) Injection Generator is a generator. To prepare it, adsorb molybdenum-99 in the form of ammonium molybdate or sodium molybdate into alumina packed in a suitable column. Combine the alumina with apparatus required to dissolve sodium pertechnetate (^{99m}Tc) injection along with sufficient shielding to evade unnecessary radiation exposure.

To elute sodium pertechnetate (^{99m}Tc) injection, pass normal saline solution through the column of Sodium Pertechnetate (^{99m}Tc) Injection Generator. When the molybdenum-99 and technetium-99m contained in Sodium Pertechnetate (^{99m}Tc) Injection Generator are arranged radially, the sodium pertechnetate (^{99m}Tc) injection eluted from Sodium Pertechnetate (^{99m}Tc) Injection Generator, as directed under the method of use, contains NLT 60.0% and NMT 110.0% of the molybdenum-99 radioactivity labeled at the time of testing.

Method of preparation To prepare Sodium Pertechnetate (^{99m}Tc) Injection Generator, fill an appropriate amount of alumina into an appropriate column, add molybdate (^{99}Mo) salt solution to the alumina, adsorb, and wash it well with a cleaning solution. After sterilizing, combine with other apparatus as directed under the Generator.

Test of the dissolved solution The solution eluted from Sodium Pertechnetate (^{99m}Tc) Injection Generator as directed under the method of use for Sodium Pertechnetate (^{99m}Tc) Injection Generator meets the requirements for the Description, Identification, pH, and Purity of Sodium Pertechnetate (^{99m}Tc) Injection.

Packaging and storage Preserve in shielded hermetic containers.

Sodium Phosphate (^{32}P) Solution
인산나트륨(^{32}P) 액

Sodium Phosphate (^{32}P) Solution contains phosphorus-32 in the form of sodium phosphate.

Sodium Phosphate (^{32}P) Solution contains NLT 90.0% and NMT 110.0% of phosphorus-32 radioactivity labeled at the time of the test.

Method of preparation Prepare Sodium Phosphate (^{32}P) Solution as directed under Solutions, with phosphoric acid (^{32}P), previously neutralized with sodium hydroxide.

Description Sodium Phosphate (^{32}P) Solution occurs as a clear and colorless liquid.

Identification Take a certain amount of Sodium Phosphate (^{32}P) Solution and phosphorus-32 RS equivalent to an amount presenting between 10,000 and 15,000 counts per minute, place into homogeneous sample dishes, respectively, dry, and count the radioactivity using a Geiger-Mueller counter (GM counter) with NLT 6 aluminum absorption plates of different thicknesses, selected in the range of 10 mg/cm^2 to 200 mg/cm^2. For each aluminum absorption plate, create the graph with the logarithm of the counting values on the vertical axis plotting against the total absorption layer for each aluminum absorption plate, where the total absorption layer is the sum of the thickness of the aluminum absorption plate (mg/cm^2), the thickness of the GM counter's mica window (mg/cm^2) and the thickness of the air layer (a × 1.205 mg/cm^2 where 'a' represents the distance in cm between the sample and the mica window), and determine the best-fit straight line between each set of coordinates. Take 2 points on the horizontal axis with a difference of NLT 100 mg/cm^2, and determine the coordinates on the best-fit straight line corresponding to each point. Calculate the absorption factor using the following equation; the absorption factor is NLT 95% and NMT 105% of the absorption factor of phosphorus-32.

$$\text{Absorption factor} = \frac{1}{t_2 - t_1} \times \ln\frac{Nt_1}{Nt_2} \times \frac{2.303(logNt_1 - logNt_{2)}}{t_2 - t_1}$$

t_1: Thickness of the total absorption layer of the thin-side absorption plate

t_2: Thickness of the total absorption layer of the thick-side absorption plate

Nt_1: Radioactivity on the graph at t_1

Nt_2: Radioactivity on the graph at t_2

pH Between 5.0 and 7.0

Purity ***Radiochemical impurity***—Dilute Sodium Phosphate (^{32}P) Solution with water to make a solution which 1 drop presenting between 10,000 and 15,000 counts in 1 minute, perform the test with this solution as directed under the Descending Paper chromatography using 1 drop of sodium dihydrogen phosphate dihydrate (1 in 100) (as a carrier) and a mixture of t-butanol, water and formic acid (8 : 4 : 1) (as a developing solvent); no radioactivity other than the sodium phosphate (^{32}P) spot are detected. Spray evenly a mixture of ammonium molybdate solution (1 in 20) and diluted nitric acid (1 in 2) (25 : 9); confirm the spot of sodium phosphate (^{32}P) by the coloring.

Assay Weigh accurately a certain amount of Sodium Phosphate (^{32}P) Solution and phosphorus-32 RS, add water so that each 0.1 mL of these solutions present between 10,000 and 15,000 counts in 1 minute, and use these solutions as the test solution and the standard solution, respectively. Add slowly 1 drop each of dioctyl sulfosuccinate sodium solution (1 in 100) in 2 homogeneous sample dishes, allow to spread evenly across the bottom surface of the dishes, and dry. Take 0.1 mL of the test solution using a micropipet, place in one of the sample dishes, wash twice the micropipet with water, transfer the washings into the same dish, and dry at below 50 °C using an infrared lamp. Separately, take 0.1 mL of the test solution using a micropipet, place in another dish, and proceed in the same manner as the test solution. With the dried test solution and dried standard solution, use a GM counter to measure the radioactivity under the same time and conditions, and determine A and A'. Calculate the radioactivity of a certain amount of Sodium Phosphate (^{32}P) Solution using the following equation.

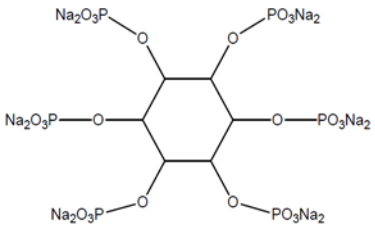
Radioactivity in a certain amount of Sodium Phosphate (^{32}P) Solution

$$= S \times \frac{A}{A'} \times \frac{D}{D'}$$

S: Radioactivity in a certain amount of reference standard
A: Count number in 1 minute of the dried test solution
A': Count number in 1 minute of the dried standard solution
D: Dilution factor of the test solution
D': Dilution factor of the standard solution

Packaging and storage Preserve in shielded hermetic containers.

Sodium Phytate
피트산나트륨



$C_6H_{18}O_{24}P_6 \cdot 12Na$: 935.91
Myo-inositol hexakis(dihydrogen phosphate) dodecasodium salt [*14306-25-3*]

Sodium Phytate contains NLT 95.0% and NMT 101.0% of sodium phytate ($C_6H_{18}O_{24}P_6 \cdot 12Na$).

Description Sodium Phytate occurs as a white powder. It is freely soluble in 1 mol/L hydrochloric acid, slightly soluble in sodium hydroxide TS, and practically insoluble in acetone, ethanol or ether.
It is freely soluble in water.

Melting point Between 58 °C and 60 °C

pH An aqueous solution of Sodium Phytate (1 in 500) is between 10.5 and 11.5.

Identification (1) To an aqueous solution of Sodium Phytate (1 in 400), add 1 to 2 drops of calcium chloride (1 in 1000); a white precipitate is formed.

(2) Determine the infrared spectra of Sodium Phytate and sodium phytate RS, previously dried at 105°C for 3 hours, as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar absorptions at the same wavenumbers.

Purity (1) ***Heavy metals***—Proceed with 1.0 g of Sodium Phytate according to Method 1 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 20 ppm).

(2) ***Arsenic***—Proceed with 0.5 g of Sodium Phytate according to Method 1 and perform the test (NMT 20 ppm).

Loss on drying Between 20% and 25% (1 g, 100 °C, 1 hours).

Assay Weigh accurately about 0.2 g of Sodium Phytate and dissolve in 25 mL of water. Add 100 mL of 0.1% calcium chloride solution, mix well, and precipitate. Filter through a filter paper, previously weighed, and dry the precipitates at between 100℃ and 105℃. Cool to ordinary temperature in a desiccator and measure the mass.

Each mg of anhydrous calcium phytate
= 0.565 mg of $C_6H_{18}O_{24}P_6 \cdot 12Na$

Packaging and storage Preserve in well-closed containers.

Technetium (^{99m}Tc) Dimercaptosuccinic Acid Injection
디메르캅토숙신산테크네튬(^{99m}Tc) 주사액

Technetium (^{99m}Tc) Dimercaptosuccinic Acid Injection is an aqueous injection containing technetium-99m in the form of technetium (^{99m}Tc) dimercaptosuccinic acid.

Technetium (^{99m}Tc) Dimercaptosuccinic Acid Injection contains NLT 90.0% and NMT 110.0% of the technetium-99m radioactivity labeled at the time of testing.

Method of preparation Prepare Technetium (^{99m}Tc) Dimercaptosuccinic Acid Injection as directed under Injections by mixing sodium pertechnetate (^{99m}Tc) injection with tin(II) chloride dihydrate and meso-2,3-dimercaptosuccinic acid for injection, prepared separately as directed under Injections.

Description Technetium (^{99m}Tc) Dimercaptosuccinic Acid Injection occurs as a clear and colorless liquid.

Identification (1) With Technetium (^{99m}Tc) Dimercaptosuccinic Acid Injection, perform the test as directed in the spectroscopy using gamma-ray spectrometer under Gamma-ray spectrometry; confirm the peak at 0.141 MeV.

(2) Confirm as directed under Purity.

pH Between 2.0 and 3.5.

Purity ***Radiochemical impurity***—With Technetium (^{99m}Tc) Dimercaptosuccinic Acid Injection, develop the plate using acetone (as a developing solvent) for about 15 minutes, and perform the test as directed under the Thin Layer Chromatography; the radioactivity other than the spot of Technetium (^{99m}Tc) Dimercaptosuccinic Acid is NMT 5% of the total radioactivity. However, prepare the thin-layer chromatographic plates using aluminum oxide for thin layer chromatography.

Sterility Meets the requirements.

Particulate contamination: Visible particles Meets the requirements.

Assay Perform the test with Technetium (^{99m}Tc) Dimercaptosuccinic Acid Injection as directed in the Assay under the Gamma-ray Spectrometry.

Packaging and storage Preserve in shielded hermetic containers.

Technetium (^{99m}Tc) Human Albumin Macroaggregated Injection
테크네튬대응집인혈청알부민(^{99m}Tc) 주사액

Technetium (^{99m}Tc) Human Albumin Macroaggregated Injection is an aqueous injection and contains technetium-99m in the form of technetium human albumin macroaggregated.

Technetium (^{99m}Tc) Human Albumin Macroaggregated Injection contains NLT 90.0% and NMT 110.0% of technetium-99m radioactivity labeled at the time of the test.

Method of preparation Prepare Technetium (^{99m}Tc) Human Albumin Macroaggregated Injection as directed under Injections, by mixing Sodium Pertechnetate (^{99m}Tc) Injection, tin(II) chloride dihydrate for injection which is separately prepared as directed under Injections, and human albumin macroaggregated suspension.

Description Technetium (^{99m}Tc) Human Albumin Macroaggregated Injection occurs as a white to pale yellow suspension.

Identification (1) Perform the test with Technetium (^{99m}Tc) Human Albumin Macroaggregated Injection as directed in the spectroscopy using gamma-ray spectrometer under the Gamma-ray spectrometry; confirm the peak at 0.141 MeV.

(2) Perform the test as directed under the Purity.

pH Between 4.5 and 6.0

Purity ***Radiochemical impurity***—Develop the plate with Technetium (^{99m}Tc) Human Albumin Macroaggregated Injection as directed under the Paper chromatography for 2 hours, using 75% methanol (as a developing solvent); the radioactivity other than the principal spot is NMT 5% of the total radioactivity.

Particle size distribution estimation by analytical sieving Shake Technetium (^{99m}Tc) Human Albumin Macroaggregated Injection sufficiently to disperse the particles evenly, dye with 1 to 2 drops of 4,5,6,7-tetrachloro-2,4,5,7-tetraiodofluorescein sodium solution (1 in 100), and determine the diameters of some particles using a microscope; NLT 90% of particles measure between 10 μm and 90 μm in diameter, and no particle diameter is greater than 150 μm.

Sterility Meets the requirements.

Bacterial endotoxins NMT 175/V EU of per each mL of Technetium (^{99m}Tc) Human Albumin Macroaggregated Injection. In this case, V represents the maximum recommended amount per mL during the effective time.

Particulate contamination: Visible particles Meets the requirements.

Assay Perform the test with Technetium (^{99m}Tc) Human Albumin Macroaggregated Injection as directed in the Assay under the Gamma-ray spectrometry.

Packaging and storage Preserve in shielded hermetic containers at 2 ℃ to 8 ℃.

Technetium (^{99m}Tc) Phytate Injection
피트산테크네튬(^{99m}Tc) 주사액

Technetium (^{99m}Tc) Phytate Injection is an aqueous injection, which contains technetium-99m in the form of technetium phytate.

Technetium (^{99m}Tc) Phytate Injection contains NLT 90.0% and NMT 110.0% of technetium-99m radioactivity labeled at the time of the test.

Method of preparation Prepare Technetium (^{99m}Tc) Phytate Injection as directed under Injections, by mixing Sodium Pertechnetate (^{99m}Tc) Injection, tin(II) chloride dihydrate injection which is separately prepared as directed under Injections, and Sodium Phytate.

Description Technetium (^{99m}Tc) Phytate Injection occurs as a clear and colorless liquid.

Identification (1) Perform the test with Technetium (^{99m}Tc) Phytate Injection as directed in the spectroscopy using gamma-ray spectrometer under the Gamma-ray spectrometry; confirm the peak at 0.141 MeV.

(2) Perform the test as directed under the Purity.

pH Between 3.0 and 7.0

Purity ***Radiochemical impurity***—Develop the plate with Technetium (^{99m}Tc) Phytate Injection as directed under the Paper chromatography for 2 hours using 85% methanol (as a developing solvent); the radioactivity other than the principal spot is NMT 5% of the total radioactivity.

Sterility Meets the requirements.

Particulate contamination: Visible particles Meets the requirements.

Assay Perform the test with Technetium (^{99m}Tc) Phytate Injection as directed in the Assay under the Gamma-ray spectrometry.

Packaging and storage Preserve in shielded hermetic containers at 2 ℃ to 8 ℃.

Technetium (^{99m}Tc) Hydroxymethylenediphosphonic Acid Injection
히드록시메틸렌디포스폰산테크네튬(^{99m}Tc) 주사액

Technetium (^{99m}Tc) Hydroxymethylenediphosphonic Acid Injection is an aqueous injection, which contains technetium-99m in the form of technetium hydroxymethylenediphosphonic.

Technetium (^{99m}Tc) Hydroxymethylenediphosphonic Acid Injection contains NLT 90.0% and NMT 110.0% of technetium-99m radioactivity labeled at the time of the test.

Method of preparation Prepare Technetium (^{99m}Tc) Hydroxymethylenediphosphonic Acid Injection as directed under

Injections, by mixing Sodium Pertechnetate (^{99m}Tc) Injection, tin(II) chloride dihydrate injection which is separately prepared as directed under the Injection, and hydroxymethylenediphosphonic acid.

Description Technetium (^{99m}Tc) Hydroxymethylenediphosphonic Acid Injection occurs as a clear and colorless liquid.

Identification (1) Perform the test with Technetium (^{99m}Tc) Hydroxymethylenediphosphonic Acid Injection as directed in the spectroscopy using gamma-ray spectrometer under the Gamma-ray spectrometry; confirm the peak at 0.141 MeV.

(2) Perform the test as directed under the Purity.

pH Between 4.0 and 6.0

Purity ***Radiochemical impurity***—Develop the plate with Technetium (^{99m}Tc) Hydroxymethylenediphosphonic Acid Injection as directed under the Thin Layer Chromatography using a mixture of ammonium chloride TS, 10 mol/L urea TS and acetic acid (100) (49 : 49 : 2) (as a developing solvent) to a distance about 10 cm; the radioactivity other than the spot of technetium (^{99m}Tc) hydroxymethylenediphosphonic acid (R_f value: approximately 0.90 to 1.00) is NMT 5% of the total radioactivity. In this case, prepare the thin-layer chromatographic plate using cellulose for thin layer chromatography.

Sterility Meets the requirements.

Particulate contamination: Visible particle Meets the requirements.

Assay Perform the test with Technetium (^{99m}Tc) Hydroxymethylenediphosphonic Acid Injection as directed in the Assay under the Gamma-ray spectrometry.

Packaging and storage Preserve in shielded hermetic containers.

Technetium (^{99m}Tc) Methylenediphosphonic Acid Injection
메틸렌디포스폰산테크네튬(^{99m}Tc) 주사액

Technetium (^{99m}Tc) Methylenediphosphonic Acid Injection is an aqueous injection containing technetium-99m in the form of technetium methylenediphosphonic acid.

Technetium (^{99m}Tc) Methylenediphosphonic Acid Injection contains NLT 90.0% and NMT 110.0% of the technetium-99m radioactivity labeled at the time of testing.

Method of preparation Prepare Technetium (^{99m}Tc) Methylenediphosphonic Acid Injection as directed under Injections by mixing Sodium Pertechnetate (^{99m}Tc) Injection with tin(II) chloride dihydrate and methylenediphosphonic acid for injection, prepared separately as directed under Injections.

Description Technetium (^{99m}Tc) Methylenediphosphonic Acid Injection occurs as a clear and colorless liquid.

Identification (1) With Technetium (^{99m}Tc) Methylenediphosphonic Acid Injection, perform the test as directed in the spectroscopy gamma-ray spectrometer under Gamma-ray spectrometry; confirm the peak at 0.141 MeV.

(2) Confirm as directed in the Purity.

pH Between 5.0 and 7.5

Purity ***Radiochemical impurity***—Develop the plate using 1-butanol (as a developing solvent) for about 40 minutes according to the Thin Layer Chromatography; the radioactivity other than the spot Technetium (^{99m}Tc) Methylenediphosphonic Acid is NMT 5% of the total radioactivity. However, prepare the thin-layer chromatographic plates using silica gel for thin-layer chromatography.

Sterility Meets the requirements.

Particulate contamination: Visible particles Meets the requirements.

Assay Perform the test with Technetium (^{99m}Tc) Methylenediphosphonic Acid Injection as directed in the Assay under the Gamma-ray spectrometry.

Packaging and storage Preserve in shielded hermetic containers.

Technetium (^{99m}Tc) Pyrophosphate Injection
피로인산테크네튬(^{99m}Tc) 주사액

Technetium (^{99m}Tc) Pyrophosphate Injection is an aqueous injection and contains technetium-99m in the form of technetium pyrophosphate.

Technetium (^{99m}Tc) Pyrophosphate Injection contains NLT 90.0% and NMT 110.0% of technetium-99m radioactivity labeled at the time of the test.

Method of preparation Prepare Technetium (^{99m}Tc) Pyrophosphate Injection as directed under Injections, by mixing Sodium Pertechnetate (^{99m}Tc) Injection, tin(II) chloride dihydrate injection which is separately prepared as directed under Injections, and sodium pyrophosphate.

Description Technetium (^{99m}Tc) Pyrophosphate Injection occurs as a clear and colorless liquid.

Identification (1) Perform the test with Technetium (^{99m}Tc) Pyrophosphate Injection as directed in the spectroscopy using gamma-ray spectrometer under the Gamma-ray spectrometry; confirm the peak at 0.141 MeV.

(2) Perform the test as directed under the Purity.

pH Between 4.5 and 5.5

Purity ***Radiochemical impurity***—Develop the plate with Technetium (^{99m}Tc) Pyrophosphate Injection as directed under the Paper chromatography for 2 hours using a mixture of methanol and ammonia TS (7:3) (as a developing solvent); the radioactivity other than the principal spot is NMT 5% of the total radioactivity.

Sterility Meets the requirements.

Bacterial endotoxins NMT 175/V EU per each mL of Technetium (^{99m}Tc) Pyrophosphate Injection. In this case, V represents the maximum recommended amount per mL during the

effective time.

Particulate contamination: Visible particles Meets the requirements.

Assay Perform the test with Technetium (^{99m}Tc) Pyrophosphate Injection as directed in the Assay under the Gamma-ray spectrometry.

Packaging and storage Preserve in shielded hermetic containers at 2 ℃ to 8 ℃.

Technetium (^{99m}Tc) Tetrofosmin Injection
테트로포스민테크네튬(^{99m}Tc) 주사액

Technetium (^{99m}Tc) Tetrofosmin Injection is an aqueous injection and contains technetium-99m in the form of technetium tetrofosmin.
Technetium (^{99m}Tc) Tetrofosmin Injection contains NLT 90.0% and NMT 110.0% of technetium-99m radioactivity labeled at the time of the test.

Method of preparation Prepare Technetium (^{99m}Tc) Tetrofosmin Injection as directed under Injections, by mixing Sodium Pertechnetate (^{99m}Tc) Injection, tin(II) chloride dihydrate injection which is separately prepared as directed under Injections, and Tetrofosmin Sulfosalicylate.

Description Technetium (^{99m}Tc) Tetrofosmin Injection occurs as a clear and colorless liquid.

Identification (1) Perform the test with Technetium (^{99m}Tc) Tetrofosmin Injection as directed in the spectroscopy using gamma-ray spectrometer under the Gamma-ray spectrometry; confirm the peak at 0.141 MeV.
(2) Perform the test as directed under the Purity.

pH Between 7.5 and 9.0

Purity ***Radiochemical impurity***—Develop the plate with Technetium (^{99m}Tc) Tetrofosmin Injection as directed under the Thin Layer Chromatography using a mixture of dichloromethane and acetone (13 : 7) (as a developing solvent); the radioactivity other than the spot of technetium tetrofosmin (^{99m}Tc) (R_f value: approximately 0.3 to 0.7) is NMT 10% of the total radioactivity. In this case, prepare the thin-layer chromatographic plate using silica gel for thin-layer chromatography.

Sterility Meets the requirements.

Bacterial endotoxins NMT 175/V EU per each mL of Technetium (^{99m}Tc) Tetrofosmin Injection. In this case, V represents the maximum recommended amount per mL during the effective time.

Particulate contamination: Visible particles Meets the requirements.

Assay Perform the test with Technetium (^{99m}Tc) Tetrofosmin Injection as directed in the Assay under the Gamma-ray spectrometry.

Packaging and storage Preserve in shielded hermetic containers.

Tetrofosmin Sulfosalicylate
테트로포스민설포살리실산염

$C_{32}H_{52}O_{16}P_2S_2$: 818.82
6,9-Bis(2-ethoxyethyl)-3,12-dioxa-6,9 diphosphatetradecane 2-hydroxy-5- sulfobenzoic acid [*127455-27-0*]
Tetrofosmin Sulfosalicylate, when dried, contains NLT 97.0% and NMT 101.0% of tetrofosmin sulfosalicylate ($C_{32}H_{52}O_{16}P_2S_2$).

Description Tetrofosmin Sulfosalicylate occurs as a white crystalline powder.
It is freely soluble in ethanol, methanol or dimethylformamide, and practically insoluble in chloroform.
It is soluble in water.

Melting point Between 106°C and 109°C.

Identification (1) Determine the infrared spectra of Tetrofosmin Sulfosalicylate and tetrofosmin sulfosalicylate RS as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

Loss on drying NMT 0.5% (1 g, 70 ℃, 6 hours).

Residue on ignition NMT 0.5% (1 g).

Purity (1) ***Heavy metals***—Proceed with 1.0 g of Tetrofosmin Sulfosalicylate according to Method 1 and perform the test. Prepare the control solution with 5.0 mL of lead standard solution (NMT 50 ppm).
(2) ***Arsenic***—Proceed with 0.3 g of Tetrofosmin Sulfosalicylate according to Method 2 and perform the test (NMT 5 ppm).

Assay Weigh accurately about 0.2 g of Tetrofosmin Sulfosalicylate, previously dried, dissolve in 20 mL of water while shaking to mix for 15 minutes, and titrate with 0.02 mol/L sodium hydroxide VS. Perform a blank test in the same way and make necessary correction (indicator: 4 drops of phenolphthalein TS).

Each mL of 0.02 mol/L sodium hydroxide VS
= 16.376 mg of $C_{32}H_{52}O_{16}P_2S_2$

Packaging and storage Preserve in well-closed containers.

Thallium (^{201}TI) Chloride Injection
염화탈륨 (^{201}TI) 주사액

Thallium (^{201}TI) Chloride Injection is an aqueous injection containing thallium-201 in the form of thallium(I) chloride.
Thallium (^{201}TI) Chloride Injection contains NLT 90.0% and NMT 110.0% of the thallium-201 radioactivity labeled at the time of testing.
The Insoluble particulate matter in injections is not applied to Thallium (^{201}TI) Chloride Injection.

Method of preparation Produce lead-201 by irradiating thallium with accelerated particles, obtain thallium-201 from the decay of lead-201, separate thallium-201, purify thallium (^{201}TI) chloride solution, and prepare Thallium (^{201}TI) Chloride Injection as directed under Injections.

Description Thallium (^{201}TI) Chloride Injection occurs as a clear and colorless liquid.

Identification (1) With Thallium (^{201}TI) Chloride Injection, perform the test as directed in the spectroscopy using gamma-ray spectrometer under the Gamma-ray spectrometry; confirm the peaks at 0.135 MeV and 0.167 MeV.

(2) Identify according to the Purity (1).

pH Between 4.0 and 8.0

Purity (1) ***Radiochemical impurity*—**Saturate Thallium (^{201}TI) Chloride Injection with 1 mol/L hydrochloric acid TS, develop the filter paper with 1-butanol (as a developing solvent) for about 2 hours as directed under the Paper chromatography, and perform the test; the radioactivity other than the spot of thallium (^{201}TI) chloride is NMT 5% of the total radioactivity. However, for the thallium chloride spot, develop with thallium chloride solution (1 in 250) in the same manner, spray phosphomolybdic acid TS, dry the paper, spray hydrobromic acid solution (1 in 2); confirm the coloring.

(2) ***Heteronuclide*—**With Thallium (^{201}TI) Chloride Injection, determine the radioactivity as directed in the Assay using the gamma-ray spectrometry under the Gamma-ray spectrometry; the radioactivities of thallium-200, thallium-202 and lead-203 account for NMT 1.0%, NMT 1.0% and NMT 0.01% of the total radioactivity at the time of the test, respectively.

(3) ***Copper*—**Spot 50 μL of Thallium (^{201}TI) Chloride Injection on a white plate, add 50 μL of water and 50 μL of ferric thiocyanate solution (1 in 100), and add 50 μL of sodium thiosulfate pentahydrate solution (1 in 100). Proceed in the same manner with 50 μL of the following control solution; the time for disappearing the color of ferric thiocyanate is not shorter than that in the control solution (NMT 2 ppm).

Control solution: Weigh accurately 0.786 g of copper(II) sulfate pentahydrate, dissolve in 1000 mL of 0.1 mol/L hydrochloric acid TS. Take 1.0 mL of this solution, and add 0.1 mol/L hydrochloric acid TS to make 100 mL.

(4) ***Thallium*—**To 0.5 mL of Thallium (^{201}TI) Chloride Injection, add 1 drop of aqua regia, 0.5 mL of 10 W/V% sulfosalicylic acid TS, 1 drop of hydrochloric acid, 2 drops of rhodamine B TS and 0.5 mL of benzene, and shake to mix; the color of the benzene layer is not more intense than that of the control solution (NMT 2 ppm).

Control solution: Weigh accurately 0.052 g of thallium acetate, and add water to make 200 mL. Take 1.0 mL of this solution, and add water to make 100 mL. Take 0.5 mL of this solution, and proceed in the same manner as in the preparation of the test solution.

(5) ***Heavy metals*—**Place 2.0 mL of Thallium (^{201}TI) Chloride Injection in a Nessler tube, add 0.2 mL of dilute acetic acid and water to make 5 mL, add 1 drop of sodium sulfide TS, and mix. Allow to stand for 5 minutes; the color of the solution is not more intense than that of the control solution (5 ppm).

Control solution: Place 1.0 mL of lead standard solution in a Nessler tube, add 0.2 mL of dilute acetic acid and water to make 5 mL, and proceed in the same manner as in the preparation of the test solution.

Sterility Meets the requirements.

Bacterial endotoxins NMT 175/V EU per each mL of Thallium (^{201}TI) Chloride Injection. However, V is the maximum recommended dose per mL during the effective time.

Particulate contamination: Visible particles Meets the requirements.

Assay Perform the test with Thallium (^{201}TI) Chloride Injection as directed in the Assay under the Gamma-ray spectrometry.

Packaging and storage Preserve in shielded hermetic containers at 2 ℃ to 8 ℃.

Xenon (^{133}Xe) Injection
제논(^{133}Xe) 주사액

Xenon (^{133}Xe) Injection contains is an aqueous injection and contains xenon-133 in the form of solution.

Xenon (^{133}Xe) Injection contains NLT 90.0% and NMT 110.0% of xenon-133 radioactivity labeled at the time of the test.

Method of preparation Prepare Xenon (^{133}Xe) Injection as directed under the Injections, with xenon-133, previously purified.

Description Xenon (^{133}Xe) Injection occurs as a clear and colorless liquid.
When Xenon (^{133}Xe) Injection is packaged in a syringe cartridge, no bubbles are present in the fluid.

Identification Perform the test with Xenon (^{133}Xe) Injection as directed in the spectroscopy using gamma-ray spectrometer under the Gamma-ray spectrometry; confirm the peaks at 0.031 MeV (X-ray of cesium-133) and 0.081 MeV.

Purity ***Heteronuclide***—Measure the radioactivity with Xenon (^{133}Xe) Injection as directed in the Assay under the Gamma-ray spectrometry; the radioactivity other than xenon-133 and xenon-133m is NMT 0.01% of the total radioactivity.

Sterility Meets the requirements.

Bacterial endotoxins NMT 175/V EU per each mL of Xenon (^{133}Xe) Injection. In this case, V represents the maximum recommended amount per mL during the effective time.

Particulate contamination: Visible particles Meets the requirements.

Assay Measure the radioactivity with Xenon (^{133}Xe) Injection as directed in the Assay under the Gamma-ray spectrometry, and label the result as A. Separately, remove the rubber part of the Xenon (^{133}Xe) Injection container, rinse the part with water, seal in an appropriate container, measure the radioactivity with it under the same condition, and label the result as B. Calculate the radioactivity of Xenon (^{133}Xe) Injection using the following equation.

Radioactivity in a certain amount of Xenon (^{133}Xe) Injection
= A — B

Packaging and storage Preserve in shielded hermetic

containers in a cold place.

4) Compound Preparations

Alum Solution
백반수

Aluminium potassium sulfate solution

Alum Solution contains NLT 0.27% and NMT 0.33% of aluminum potassium sulfate [$AlK(SO_4)_2 \cdot 12H_2O$: 474.39].

Method of preparation

Aluminium potassium sulfate	3 g
Peppermint solution	50 mL
Water or Purified Water	A sufficient quantity
To make	1000 mL

Prepare by mixing and dissolving the above.

Description Alum Solution occurs as a clear, colorless liquid with an odor of peppermint oil and an astringent taste.

Identification (1) Take 5 mL of Alum Solution and add 3 mL of ammonium chloride TS and 1 mL of ammonia TS; a white, gelatinous precipitate is formed. Also, it changes to red upon the addition of 5 drops of alizarin S TS (aluminum sulfate).

(2) Alum Solution responds to the Chemical identification reactions (1) and (2) for sulfate.

(3) Put 100 mL of Alum Solution in an evaporating dish, evaporate on a water bath to dryness, and dissolve the residue in 5 mL of water; the resulting solution responds to the Chemical identification reactions for potassium salt.

Assay Pipet 50.0 mL of Alum Solution, add exactly 30.0 mL of 0.02 mol/L ethylenediaminetetraacetic acid disodium salt, add 20 mL of acetic acid-ammonium acetate buffer solution (pH 4.8), and boil for 5 minutes. After cooling, add 55 mL of ethanol (95) and titrate with 0.02 mol/L zinc acetate VS (indicator: 2 mL of dithizone TS). The endpoint of titration is when the color of the solution changes from pale dark green to pale red. Perform a blank test in the same manner.

Each mL of 0.02 mol/L ethylenediaminetetraacetic acid disodium salt VS = 9.488 mg of $AlK(SO_4)_2 \cdot 12H_2O$

Packaging and storage Preserve in tight containers.

Epinephrine Solution
에피네프린 액

Epinephrine Hydrochloride Solution
Adrenaline Hydrochloride Solution

Epinephrine Solution contains NLT 0.085 w/v% and NMT 0.115 w/v% of epinephrine ($C_9H_{13}NO_3$: 183.20).

Method of preparation

Epinephrine	1 g
Sodium chloride	8.5 g
Diluted hydrochloric acid (9 in 100)	10 mL
Preservative	A suitable amount
Stabilizer	A suitable amount
Purified water	A suitable amount
To make	1000 mL

Prepare with the above by mixing.

Description Epinephrine Solution occurs as a colorless to pale red, clear solution.
It slowly turns to pale red and subsequently to brown by the air or light.

pH—Between 2.3 and 5.0

Identification Proceed as directed in the Identification under Epinephrine Injection.

Assay Proceed as directed in the Assay under Epinephrine Injection.

Packaging and storage Preserve in light-resistant, tight containers.

Hydrophilic Petrolatum
친수바셀린

Method of preparation

White beeswax	80 g
Stearyl alcohol or Cetanol	30 g
Cholesterol	30 g
White petrolatum	An appropriate amount
Total amount	1000 g

Melt Stearyl Alcohol or Cetanol, White Beeswax and White Petrolatum by warming on a water bath by stirring to mix, add Cholesterol, and stir to mix until it melts completely. Then, stop warming and stir well to mix until the mixture congeals.

Description Hydrophilic Petrolatum is white and has a slightly characteristic odor.
When mixed with the same amount of water, Hydrophilic Petrolatum has the same consistency as ointments.

Packaging and storage Preserve in tight containers.

Compound Iodine Glycerin
복방요오드·글리세린

Compound Iodine Glycerin contains NLT 1.1 w/v% and NMT 1.3 w/v% of iodine (I: 126.90), NLT 2.2 w/v% and NMT 2.6 w/v% of potassium iodide (KI: 166.00), NLT 2.7 w/v% to 3.3 w/v% of total iodine (as I), and NLT 0.43 w/v% NMT 0.53 w/v% of phenol (C_6H_6O : 94.11).

Method of preparation

Iodine	12 g
Potassium iodide	24 g
Glycerin	900 mL
Mentha water	45 mL
Liquid phenol	5 mL

Purified water	An appropriate amount
	Total volume 1000 mL

Dissolve Potassium Iodide and Iodine in about 25 mL of Purified Water, add Glycerin, then add Mentha Water, Liquefied Phenol, and Purified Water to make 1000 mL in total and mix. However, it may be prepared using an appropriate amount of Concentrated Glycerin and Purified Water instead of Glycerin'.

Description Compound Iodine Glycerin occurs as a reddish brown, viscous liquid and has a characteristic odor.

Specific gravity d_{20}^{20}: About 1.23

Identification (1) The colored solution obtained in the Assay (1) exhibits a red color. Determine the absorption spectrum of this solution as directed under the Ultraviolet-visible Spectroscopy; it exhibits a maximum at the wavelengths of 510 nm to 514 nm (iodine).

(2) The colored solution obtained in the Assay (2) exhibits a red color. Determine the absorption spectrum of this solution as directed under the Ultraviolet-visible Spectroscopy; it exhibits a maximum at the wavelengths of 510 nm to 514 nm (potassium iodide).

(3) The colored solution obtained in the Assay (4) exhibits a yellow color. Determine the absorption spectrum of this solution as directed under the Ultraviolet-visible Spectroscopy; it exhibits a maximum at the wavelengths of 401 nm to 405 nm (phenol).

(4) Take 1 mL of Compound Iodine Glycerin in a test tube with a stopper, add 10 mL of ethanol (95), 2 mL of sodium hydroxide solution and 1 mL of the solution of cupric chloride solution in ethanol (95) (1 in 0), and shake to mix; the solution exhibits a blue color. (glycerin).

Assay (1) ***Iodine*—**Determine the specific gravity of Compound Iodine Glycerin in advance according to Method 2 of the Specific gravity. Weigh accurately the mass equivalent to about 7 mL, add ethanol (95) to make exactly 200 mL, and use this solution as the test solution. Separately, weigh accurately about 80 mg of the iodine RS and about 0.17 g of the potassium iodide RS, previously dried at 105°C for 4 hours, dissolve in ethanol (95) to make exactly 200 mL, and use this solution as the standard solution. Pipet 3 mL each of the test solution and the standard solution, put in a 50-mL separatory funnel, add exactly 10 mL of a mixture of chloroform and hexane (2 : 1) and 15 mL of water to each, shake to mix, and take the chloroform and hexane layers separately [use the water layer for (2)], and filter through cotton wool. Perform the test with the filtrate as directed under the Ultraviolet-visible Spectroscopy, using a mixture of chloroform and hexane (2:1) as the reference solution and determine the absorbances A_T and A_S of each solution obtained from the test solution and the standard solution at 512 nm.

$$\text{Amount (mg) of iodine (I)} = \text{Amount (mg) of iodine RS} \times \frac{A_T}{A_S}$$

(2) ***Potassium iodide*—**Pipet 10 mL each of the aqueous layer obtained from the test solution and the standard solution in (1), add exactly 1 mL of diluted dilute hydrochloric acid (1 in 2), 1 mL of sodium nitrite TS, 10 mL of a mixture of chloroform and hexane (2 : 1), respectively, and shake vigorously to mix. Take the chloroform and hexane layers separately and filter using cotton wool. Perform the test with the filtrate as directed under the Ultraviolet-visible Spectroscopy, using a mixture of chloroform and hexane layer (2 : 1) as the blank and determine the absorbances, A_T and A_S, of each solution obtained from the test solution and the standard solution at 512 nm.

$$\text{Amount (mg) of potassium iodide (KI)} = \text{Amount (mg) of potassium iodide RS} \times \frac{A_T}{A_S}$$

(3) ***Total iodine*—**Determine the specific gravity of Compound Iodine Glycerin according to Method 2 under the Specific gravity and density. Weigh accurately the mass equivalent to about 5 mL and add water to make exactly 50 mL. Pipet 5 mL of this solution into a 50-mL flask, add 0.5 g of zinc powder and 5 mL of acetic acid (100), shake to mix until the color of iodine disappears, attach a reflux condenser, and heat on a water bath for 30 minutes. Add 10 mL of hot water through the condenser, wash the condenser, and filter using a glass filter. Wash the flask twice with 10 mL of warm water, combine the filtrate and the washings, cool, add water to make exactly 50 mL, and use this solution as the test solution. Separately, weigh accurately about 0.2 g of potassium iodide RS, previously dried at 105 °C for 4 hours, and dissolve in water to make exactly 50 mL. Pipet 5 mL of this solution, add 5 mL of acetic acid (100) and water to make exactly 50 mL, and use this solution as the standard solution. Pipet 4 mL each of the test solution and the standard solution into a 30-mL separatory funnel, and add exactly 5 mL of water, 1 mL of diluted dilute hydrochloric acid (1 in 2), 1 mL of sodium nitrite TS, and 10 mL of a mixture of chloroform and hexane (2 : 1) respectively, and shake vigorously to mix. Perform the test as directed in (2).

$$\text{Amount (mg) of total iodine (I)} = \text{Amount (mg) of potassium iodide RS} \times \frac{A_T}{A_S} \times 0.7644$$

(4) ***Phenol*—**Determine the specific gravity of Compound Iodine Glycerin according to Method 2 under Specific Gravity and Density. Weigh accurately the mass equivalent to about 2 mL, add 3 mL of 0.1 mol/L sodium thiosulfate solution, shake to mix, add 2 mL of dilute hydrochloric acid, and extract twice with 10 mL each of chloroform. Combine all chloroform extracts and extract twice with 10 mL each of 0.5 mol/L sodium hydroxide TS. Combine all water layers, add water to make exactly 500 mL, and use this solution as the test solution. Separately, weigh accurately about 0.5 g of phenol RS and dissolve in ethanol (95) to make exactly 100 mL. Pipet 2 mL of this solution, proceed in the same manner as in the preparation of the test solution, and use this solution as the standard solution. Pipet 3 mL each of the test solution and the standard solution, add 2 mL of dilute hydrochloric acid each, and put on a water bath at 30 °C. Add exactly 2 mL of sodium nitrite solution (1 in 100) after allowing to stand for 10 minutes, shake to mix, and allow to stand at 30 °C for 60 minutes. Next, add dilute potassium hydroxide and ethanol TS to make exactly 25 mL. Determine the absorbances, A_T and A_S, of each solution obtained from the test solution and the standard solution, at 403 nm as directed under the Ultraviolet-visible Spectroscopy, using the solution prepared in the same manner with 3 mL of water as the blank.

$$\text{Amount (mg) of phenol } (C_6H_6O) = \text{Amount (mg) of phenol RS} \times \frac{A_T}{A_S} \times \frac{1}{50}$$

Packaging and storage Preserve in light-resistant, tight

containers.

Iodine Tincture
요오드 틴크

Iodine Tincture contains NLT 5.7 w/v% and NMT 6.3 w/v% of Iodine (I: 126.90) and NLT 3.8 w/v% and NMT 4.2 w/v% of potassium iodide (KI: 166.00).

Method of preparation

Iodine	60 g
Potassium iodide	40 g
70% ethanol	A sufficient quantity
	To make 1000 mL

Prepare as directed under Spirits, with the above. A sufficient amount of Ethanol or Ethanol for Disinfection and Purified Water may be used instead of 70 vol% ethanol.

Description Iodine Tincture occurs as dark reddish brown liquid with a characteristic odor.

Specific gravity d_{20}^{20}: About 0.97

Identification (1) Add 1 drop of Iodine Tincture to a mixture of 1 mL of starch TS and 9 mL of water; the solution exhibits a dark bluish purple.

(2) Evaporate 3 mL of Iodine Tincture to dryness on a water bath and heat gently on a direct flame; a white residue is produced, which responds to the Chemical identification reactions for potassium salt and iodide.

Alcohol number NLT 6.6 (Method 2). Perform the procedure (B) in Method 1.

Assay (1) ***Iodine***—Pipet 5 mL of Iodine Tincture, add 0.5 g of potassium iodide, 20 mL of water and 1 mL of dilute hydrochloric acid, and titrate with 0.1 mol/L sodium thiosulfate VS (indicator: 2 mL of starch TS).

Each mL of 0.1 mol/L sodium thiosulfate VS = 12.690 mg of I

(2) ***Potassium iodide***—Take exactly 5 mL of Iodine Tincture to an iodine flask, add 20 mL of water, 50 mL of hydrochloric acid and 5 mL of chloroform, cool to room temperature, and titrate with 0.05 mol/L potassium iodate VS until the reddish purple color disappears from the chloroform layer, with agitating the mixture vigorously. After the chloroform layer has been decolorized, allow the mixture to stand for 5 minutes. If the color reappears, continue titration. Calculate the amount (mg) of potassium iodide (KI) by the following formula, from the volume (a mL) of 0.05 mol/L potassium iodate VS obtained here and the volume (b mL) of 0.1 mol/L sodium thiosulfate VS used in the titration.

$$\text{Amount (mg) of potassium iodide (KI)} = 16.600\times\left(a-\frac{b}{2}\right)$$

Packaging and storage Preserve in tight containers.

Dilute Iodine Tincture
묽은요오드 틴크

Dilute Iodine Tincture contains NLT 2.8 w/v% and NMT 3.2 w/v% of iodine (I: 126.90) and NLT 1.9 w/v% and NMT 2.1 w/v% of potassium iodide (KI: 166.00).

Method of preparation

Iodine	30 g
Potassium iodide	20 g
70 vol% ethanol	An appropriate amount
	Total volume 1000 mL

Prepare as directed under Spirits, with the above ingredients. However, it may be prepared using an appropriate amount of Ethanol or Ethanol for Disinfection and Purified Water instead of 70 vol% ethanol. It may also be prepared by taking 500 mL of "Iodine Tincture and adding 70 vol% ethanol to make it 1000 mL in total.

Description Dilute Iodine Tincture occurs as a dark reddish brown solution and has a characteristic odor.

Specific gravity d_{20}^{20}: About 0.93

Identification (1) Add 1 drop of Dilute Iodine Tincture to a mixture of 1 mL of starch TS and 9 mL of water; the resulting solution exhibits a dark bluish-purple color.

(2) Evaporate 3 mL of Dilute Iodine Tincture to dryness on a water bath and heat gently over a direct flame; a white residue is formed and this residue responds to Chemical identification reactions for potassium salt and iodide.

Alcohol number NLT 6.7 (Method 2). However, perform the test according to the Procedure (B) under Method 1

Assay (1) ***Iodine—***Pipet 10 mL of Dilute Iodine Tincture, add 0.5 g of potassium iodide, 20 mL of water, and 1 mL of dilute hydrochloric acid, and titrate with 0.1 mol/L sodium thiosulfate VS (indicator: 2 mL of starch TS).

Each mL of 0.1 mol/L sodium thiosulfate VS
= 12.690 mg of I

(2) ***Potassium iodide—***Take exactly 10 mL of Dilute Iodine Tincture, put it in an iodine bottle, add 20 mL of water, 50 mL of hydrochloric acid, and 5 mL of chloroform, cool at room temperature, shake vigorously to mix until the reddish purple color of the chloroform layer disappears, and titrate with 0.05 mol/L potassium iodate VS. Allow to stand for 5 minutes after the color of the chloroform layer disappears and continue the titration when the color reappears. Calculate the amount (mg) of potassium iodide (KI) from the volume (a mL) of 0.05 mol/L potassium iodate VS consumed as above and the volume (b mL) of 0.1 mol/L sodium thiosulfate VS consumed in the titration in (1) according to the following equation.

$$\text{Amount (mg) of potassium iodide (KI)} = 16.600\times\left(a-\frac{b}{2}\right)$$

Packaging and storage Preserve in tight containers.

Morphine and Atropine Injection
모르핀·아트로핀 주사액

Morphine and Atropine Injection is an aqueous injections. Morphine and Atropine Injection contains NLT 0.91% and NMT 1.09% of morphine hydrochloride hydrate ($C_{17}H_{19}NO_3 \cdot HCl \cdot 3H_2O$:375.84) and NLT 0.027% and NMT 0.033% of atropine sulfate hydrate [$(C_{17}H_{23}NO_3)_2 \cdot H_2SO_4 \cdot H_2O$: 694.83]

Method of preparation

Morphine hydrochloride hydrate	10 g
Atropine sulfate hydrate	0.3 g
Water for Injection	A sufficient quantity
	To make 1000 mL

Prepare as directed under Injections with the above.

Description Morphine and Atropine Injection occurs as a clear, colorless liquid.
It is gradually colored by light.

pH Between 2.5 and 5.0

Identification To 2 mL of Morphine and Atropine Injection, add 2 mL of ammonia TS, extract with 10 mL of ether, and filter the extract with a filter paper. Evaporate the filtrate to dryness on a water bath, dissolve the residue in 1 mL of anhydrous ethanol, and use this solution as the test solution. Separately, dissolve 0.1 g of morphine hydrochloride hydrate RS and atropine sulfate hydrate RS in each 10 mL of water, proceed with each 2 mL of these solutions in the same manner as in the preparation of the test solution, and use the resulting solutions as the standard solution (1) and the standard solution (2), respectively. Spot each 10 μL of the test solution, the standard solution (1) and the standard solution (2) on a thin-layer chromatographic plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of methanol and strong ammonia water (200 : 3) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly Dragendorff TS for spraying on the plate; the color and R_f values of the two spots obtained from the test solution are the same as those of the spots obtained from the standard solution (1) and the standard solution (2) (morphine and atropine).

Sterility Meets the requirements.

Particulate contamination: Visible particles Meets the requirements.

Insoluble particulate matter in injections Meets the requirements.

Extractable volume of injections Meets the requirements.

Assay (1) ***Morphine Hydrochloride Hydrate***—Pipet 2.0 mL of Morphine and Atropine Injection, add 10.0 mL of the internal standard solution, then add water to make 50 mL, and use this solution as the test solution. Separately, weigh accurately about 25 mg of morphine hydrochloride hydrate RS, dissolve in exactly 10 mL of the internal standard solution, then add water to make 50 mL, and use this solution as the standard solution. Perform the test with each 20 μL of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions. Determine the peak area ratio, Q_T and Q_S, of morphine to the peak area of the internal standard.

Amount (mg) of morphine hydrochloride hydrate ($C_{17}H_{19}Cl_3O_2$) = Amount (mg) of morphine hydrochloride hydrate RS on the anhydrous basis $\times \frac{Q_T}{Q_S} \times 1.1679$

Internal standard solution—An etilefrine hydrochloride solution (1 in 500).

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 285 nm)

Column: A stainless steel column, about 4.6 mm in internal diameter and 15 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 40 °C.

Mobile phase: Dissolve 1.0 g of sodium lauryl sulfate in 500 mL of diluted phosphoric acid (1 in 1000), and adjust the pH to 3.0 with sodium hydroxide TS. To 240 mL of this solution, add 70 mL of tetrahydrofuran, and mix.

Flow rate: Adjust the flow rate so that the retention time of morphine is about 10 minutes.

System suitability

Column performance: Perform the test with 20 μL of the standard solution according to the above operating conditions; morphine and the internal standard are eluted in this order, and the peak resolution is NLT 3.

System repeatability: Proceed with 20 μL of the standard solution under the above operating conditions and repeat the test 6 times; the relative standard deviation of the ratio of the peak area of morphine to that of the internal standard is NMT 1.0%.

(2) ***Atropine sulfate hydrate***—Pipet 2 mL of Morphine and Atropine Injection, add exactly 2 mL of the internal standard solution, and use the solution as the test solution. Separately, weigh accurately about 15 mg of Atropine Sulfate RS (previously determine its loss on drying), and dissolve in water to make exactly 50 mL. Pipet 2 mL of this solution, add exactly 2 mL of the internal standard solution, and use this solution as the standard solution. Perform the test with 20 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions. Determine the peak area ratio, Q_T and Q_S, of morphine to the peak area of the internal standard.

Amount (mg) of atropine sulfate hydrate ($C_{17}H_{23}NO_3 \cdot H_2SO_4 \cdot H_2O$) = Amount (mg) of morphine sulfate hydrate on the dried basis $\times \frac{Q_T}{Q_S} \times \frac{1}{50} \times 1.0266$

Internal standard solution—A solution of etilefrine hydrochloride (1 in 12500).

Operating conditions

For the column, column temperature and mobile phase, proceed as directed in the Assay under Morphine hydrochloride hydrate.

Detector: An ultraviolet absorption photometer (wavelength: 225 nm)

Flow rate: Adjust the flow rate so that the retention time of morphine is about 7 minutes.

System suitability

System performance: Proceed with 20 μL of the test solution under the above conditions; morphine, the internal standard, and atropine are eluted in this order with the resolution between morphine and the internal standard being NLT 3.

System repeatability: Repeat the test 6 times with 20 μL of the standard solution under the above conditions; the relative standard deviation of the peak area ratio of atropine to the peak area of the internal standard is NMT 1.0%.

Packaging and storage Preserve in light-resistant, well-closed containers.

Absorptive Ointment
흡수연고

Method of preparation

White petrolatum	400 g
Cetanol	100 g
White beeswax	50 g
Sorbitan sesquioleate	50 g
Lauromacrogol	5 g
Ethylparaben or Methylparaben	1 g
Butylparaben or Propylparaben	1 g
Purified water	An appropriate amount
	Total amount 1000 g

Melt White Petrolatum, Cetanol, White Beeswax, Sorbitan Sesquioleate, and Lauromacrogol by heating on a water bath, stir to mix, and maintain at about 75 °C. Add Methylparaben or Ethylparaben and Propylparaben or Butylparaben to Purified Water, melt by warming at 80 °C. Combine both solutions, mix to make an emulsion, cool, and stir thoroughly until it congeals.

Description Absorptive Ointment occurs as white and lustrous. It has a slightly characteristic odor.

Packaging and storage Preserve in tight containers.

Hydrophilic Ointment
친수연고

Method of preparation

White petrolatum	250 g
Stearyl alcohol	200 g
Propylene glycol	120 g
Polyoxyethylene hydrogenated castor oil 60	40 g
Glycerin monostearate	10 g
Methyl p-hydroxybenzoate	1 g
Propyl p-hydroxybenzoate	1 g
Purified water	An appropriate amount
	Total amount 1000 g

Melt White Petrolatum, Stearyl Alcohol, polyoxyethylene hydrogenated castor oil 60 and Glycerin Monostearate by warming on a water bath by stirring to mix, and maintain the temperature at about 75 ℃. To Propylene Glycol, add Methyl p-hydroxybenzoate and Propyl p-hydroxybenzoate, and melt the mixture by warming, if necessary. Add Purified Water and warm to about 75 °C. Stir the resulting solution to mix and form an emulsion, cool and stir thoroughly until it congeals.

Description Hydrophilic Ointment occurs as white and has a slightly characteristic odor.

Packaging and storage Preserve in tight containers.

Simple Ointment
단미 연고

Method of preparation

Beeswax	330 g
Fixed oil	A sufficient quantity
	To make 1000 g

Prepare as directed under Ointments, with the above.

Description Simple Ointment occurs as yellow and has a faint characteristic odor.

Packaging and storage Preserve in tight containers.

White Ointment
백색 연고

Method of preparation

White beeswax	50 g
Sorbitan sesquioleate	20 g
White Petrolatum	A sufficient quantity
	To make 1000 g

Prepare as directed under Ointments, with the above.

Description White Ointment occurs as white and has a faint characteristic odor.

Packaging and storage Preserve in tight containers.

Phenolated Water
페놀수

Carbolic acid water

Phenolated Water contains NLT 1.8 w/v% and NMT 2.3 w/v% of phenol (C_6H_6O : 94.11).

Method of preparation

Liquefied phenol	22 mL
Water or purified water	An appropriate amount
	Total amount 1000 mL

Prepare by mixing, with the above ingredients.

Description Phenolated Water occurs as clear and colorless liquid and has the odor of phenol.

Identification (1) Add 1 drop of iron(III) chloride TS to 10 mL of Phenolated Water; it exhibits a bluish-purple color.

(2) Proceed with 5 mL of an aqueous solution of Phenolated Water (1 in 200) as directed in the Identification (2) under Phenol for Disinfection.

Assay Pipet 2 mL of Phenolated Water, put in an iodine bottle, add 25 mL of water, add exactly 40 mL of 0.05 mol/L bromine solution and 5 mL of hydrochloric acid, and perform the test as directed in the Assay under Phenol for Disinfection.

Each mL of 0.05 mol/L bromine VS
= 1.5685 mg of C_6H_6O

Packaging and storage Preserve in tight containers.

Polyethylenglycol Ointment
폴리에틸렌글리콜 연고

Macrogol ointments

Method of preparation

Polyethylene glycol 4000	500 g
Polyethylene glycol 400	500 g
Total amount	1000 g

Melt Polyethylene Glycol 4000 and Polyethylene Glycol 400 by warming on a water bath to 65 °C. Stir well until it congeals. However, amounts of Polyethylene Glycol 4000 and Polyethylene Glycol 400 may be adjusted within 100 g to make the total amount of 1000 g to prepare an ointment with an appropriate viscosity (consistency).

Description Polyethylenglycol Ointment occurs as white and has a slightly characteristic odor.

Identification Dissolve 50 mg of Polyethylenglycol Ointment in 5 mL of dilute hydrochloric acid, add 1 mL of barium chloride TS, shake to mix, filter, if necessary, and add 1 mL of phosphomolybdic acid solution (1 in 10) to the filtrate; yellowish green precipitates are produced.

Packaging and storage Preserve in tight containers.

Ringer's Solution
링거 주사액

Ringer's Solution is an aqueous injections. Ringer's Solution contains NLT 0.53% and NMT 0.58% of chlorine [as (Cl: 35.45)] and NLT 0.030% and NMT 0.036% of calcium chloride (CaCl2• 2H2O: 147.02).

Method of preparation

Sodium chloride	8.6 g
Potassium chloride	0.3 g
Calcium chloride dihydrate	0.33 g
Water for Injection	A sufficient quantity
To make	1000 mL

Prepare as directed under Injections with the above.
No preservative is added.

Description Ringer's Solution occurs as a colorless, clear liquid with a slightly salty taste.

Identification (1) Ringer's Solution responds to the Chemical identification reactions for sodium salt and chloride.

(2) Concentrate about 10 mL of Ringer's Solution to 5 mL. The resulting solution responds to the Chemical identification reactions for potassium salt and calcium salt.

pH Between 5.0 and 7.5.

Purify (1) ***Heavy metals***—Concentrate about 100 mL of Ringer's Solution to 40 mL and add 2 mL of dilute acetic acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution by adding 2 mL of dilute acetic acid and water to 3.0 mL of lead standard solution to make 50 mL (NMT 0.3 ppm).

(2) ***Arsenic***—Take 20 mL of Ringer's Solution and perform the test, using this solution as the test solution (NMT 0.1 ppm).

Sterility Meets the requirements.

Bacterial endotoxins Less than 0.50 EU/mL of Ringer's Solution

Particulate contamination: Visible particles Meets the requirements.

Insoluble particulate matter in injections Meets the requirements.

Extractable volume of injections Meets the requirements.

Assay (1) ***Chlorine*—**Pipet 20.0 mL of Ringer's Solution, add 30 ml of water, and titrate with 0.1 mol/L silver nitrate VS, shaking vigorously to mix (indicator: 3 drops of fluorescein sodium TS).

Each mL of 0.1 mol/L silver nitrate VS
= 3.5453 mg of Cl

(2) ***Calcium Chloride Hydrate***—Pipet 50.0 mL of Ringer's Solution, add 2 mL of 8 mol/L potassium hydroxide TS and 50 mg of NN indicator, and titrate immediately with 0.01 mol/L disodium ethylenediaminetetraacetate VS. The endpoint of titration is when the color of the solution changes from reddish purple to blue.

Each mL of 0.01 mol/L ethylenediaminetetraacetic acid disodium salt VS
= 1.4701 mg of $CaCl_2 \cdot 2H_2O$

Packaging and storage Preserve in hermetic containers. A plastic container for aqueous injection may be used for Ringer's Solution.

Salicylic Acid Adhesive Plaster
살리실산 반창고

Method of preparation

Salicylic Acid, fine powder	500 g
Adhesive plaster base	A sufficient quantity
	To make 1000 g

Prepared with the above by mixing carefully selected rubber, resin, zinc oxide and other substances to obtain an adhesive mixture and spread evenly on a fabric.

Description The application surface of Salicylic Acid Adhesive Plaster occurs as milky white and readily adheres to the skin.

Packaging and storage Preserve in light-resistant, well-closed containers.

Salicylic Acid Spirit
살리실산 주정

Salicylic acid spirit contains NLT 2.7 w/v% and NMT 3.3 w/v% of salicylic acid ($C_7H_6O_3$: 138.12).

Method of preparation

Salicylic acid	30 g
Glycerin	50 mL
Ethanol	A sufficient quantity
	To make 1000 mL

Prepare as directed under Spirits, with the above.

Description Salicylic Acid Spirit occurs as a clear, colorless liquid.

Specific gravity d_{20}^{20}: About 0.86

Identification The colorimetric solution prepared as directed in the Assay exhibits a purple color. Determine the absorption spectrum of the solution as directed under the Ultraviolet-visible Spectroscopy; it exhibits a maximum between 520 nm and 535 nm (salicylic acid).

Alcohol number NLT 8.8 (Method 2).

Assay Pipet 10.0 mL of Salicylic Acid Spirit and add 10 mL of ethanol (95) and water to make exactly 100 mL. Pipet 3.0 mL of this solution, add hydrochloric acid-potassium chloride buffer solution, pH 2.0, to make exactly 100 mL, and use this solution as the test solution. Separately, weigh accurately about 0.3 g of salicylic acid RS, previously dried in a desiccator (silica gel) for 3 hours, and dissolve in 10 mL of ethanol (95) and water to make exactly 100 mL. Pipet 3.0 mL of this solution, add hydrochloric acid-potassium chloride buffer solution, pH 2.0, to make exactly 100 mL, and use this solution as the standard solution. Pipet 10.0 mL each of the test solution and the standard solution, add exactly 5 mL of iron(III) nitrate nonahydrate solution (1 in 200), and then add hydrochloric acid-potassium chloride buffer solution, pH 2.0, to make exactly 25 mL. With these solutions, determine the absorbances, A_T and A_S, of the test solution and the standard solution at 530 nm as directed under the Ultraviolet-visible Spectroscopy, using a reference solution prepared in the same manner with water.

Amount (mg) of salicylic acid ($C_7H_6O_3$)

= Amount (mg) of salicylic acid RS× $\frac{A_T}{A_S}$

Packaging and storage Preserve in tight containers.

Silver Nitrate Ophthalmic Solution
질산은 점안액

Silver Nitrate Ophthalmic Solution is an aqueous ophthalmic solution, which contains NLT 0.95 w/v% and NMT 1.05 w/v% of silver nitrate ($AgNO_3$: 169.87).

Method of preparation

Silver nitrate	10 g
Sterile purified water	An appropriate amount
	Total volume 1000 mL

Prepare as directed under Ophthalmic Solutions, with the above ingredients.

Description Silver Nitrate Ophthalmic Solution occurs as a clear, colorless liquid.

Identification Silver Nitrate Ophthalmic Solution responds to the Chemical identification reactions for silver salt and nitrate.

Sterility Meets the requirements.

Particulate contamination: Visible particles Meets the requirements.

Particulate matter in ophthalmic solutions Meets the requirements.

Assay Pipet 20 mL of Silver Nitrate Ophthalmic Solution, add 30 mL of water and 2 mL of nitric acid, and titrate with 0.1 mol/L ammonium thiocyanate VS (indicator: 2 mL of ammonium iron(III) sulfate TS).

Each mL of 0.1 mol/L ammonium thiocyanate VS
= 16.987 mg of $AgNO_3$

Packaging and storage Preserve in light-resistant, tight containers.

Silver Protein Solution
프로테인은 액

Silver Protein Solution contains NLT 0.22 w/v% and NMT 0.26 w/v% of silver (Ag : 107.87).

Method of preparation

Silver Protein	30 g
Glycerin	100 mL
Mentha water	An appropriate amount
	Total amount 1000 mL

Prepare by dissolving and shaking to mix the above ingredients.

Description Silver Protein Solution occurs as a brown, clear liquid with the odor of mentha oil.

Identification (1) Mix 10 mL of ethanol (95) with 1 mL of Silver Protein Solution, add 2 mL of sodium hydroxide TS, add 1 mL of a solution of cupric chloride in ethanol (95) (1 in10), shake to mix, and filter; the filtrate exhibits a blue color. (glycerin).

(2) Take 3 mL of Silver Protein Solution, add water to make 10 mL, add 2 mL of dilute hydrochloric acid, shake to mix occasionally for 5 minutes, and filter. Add 5 mL of sodium hydroxide solution (1 in 10) to the filtrate, and add 2 mL of diluted cupric sulfate TS (2 in 25); the resulting solution exhibits a violet color (silver protein).

(3) Add 1 drop of Iron(III) chloride TS to 5 mL of the test solution in (2); a brown precipitate is produced (silver protein).

(4) Put 3 mL of Silver Protein Solution in a crucible and heat carefully, almost evaporate to dryness, and then ignite slowly to incinerate. Add 1 mL of nitric acid to the residue, warm to dissolve, and add 10 mL of water; the resulting solution responds to the Chemical identification reactions for silver salt.

Assay Pipet 25 mL of Silver Protein Solution, put in a 250-mL Kjeldahl flask, and heat carefully until a white smoke of glycerin is evolved. Cool, then add 25 mL of sulfuric acid, put a small funnel on the mouth of the flask, and heat gently for 5 minutes. Cool, then add slowly 5 mL of nitric acid dropwise, and heat on a water bath for 45 minutes while shaking to mix occasionally. Cool, then add 2 mL of nitric acid, boil over low heat, and repeat this operation until the resulting solution is colorless when cooled. Wash carefully the contents of the flask with 250 mL of water into a 500-mL Erlenmeyer flask, boil gently for 5 minutes, cool, and titrate with 0.1 mol/L ammonium thiocyanate VS (indicator: 3 mL of ammonium Iron(III) sulfate TS).

Each mL of 0.1 mol/L ammonium thiocyanate VS
= 10.787 mg of Ag

Packaging and storage Preserve in light-resistant, tight containers.

Simple Syrup
단미 시럽

Simple Syrup is an aqueous solution of Sucrose.

Method of preparation

Sucrose	850 g
Purified Water	A sufficient quantity
	To make 1000 mL

Prepare as directed under Syrups, with the above.

Description Simple Syrup occurs as a colorless to pale yellow, viscous liquid and is odorless with a sweet taste.

Identification (1) Evaporate Simple Syrup to dryness and heat 1 g of the residue; it melts to swell, emitting a caramel odor and is charred to a voluminous residue.

(2) Take 0.1 g of the residue obtained in (1), add 2 mL of dilute sulfuric acid, boil, add 4 mL of sodium hydroxide TS and 3 mL of Fehling's TS and heat to boiling: a red to dark red precipitate is produced.

Specific gravity d_{20}^{20}: Between 1.310 and 1.325

Purity (1) ***Artificial sweetening agent***—To 100 mL of Simple Syrup, add 100 mL of water and shake to mix. To each 50 mL of this solution, add dilute sulfuric acid and sodium hydroxide TS to make the solutions acidic and basic, respectively. Add 100 mL of ether to each solution, shake to mix, collect the ether layer, and combine them. Evaporate the ether on a water bath and further evaporate the residue to dryness; the residue has no sweet taste.

(2) ***Salicylic acid***—Take the residue obtained in (1) and add 2 to 3 drops of dilute Iron(III) chloride TS; the solution does not exhibit a purple color.

Packaging and storage Preserve in tight containers.

5) Excipients

Acacia
아라비아고무

Acacia is a secretion obtained from the stem and branches of the *Acacia senegal* Willdenow or other plants of the same genus (Leguminosae).

Description Acacia occurs as a colorless to pale yellowish brown, transparent or slightly turbid round lump or fragment, with many cracked lines on the outer surface and is easily broken. Sometimes, the broken surface has a glass-like sheen.
It is odorless and mucilaginous.
1.0 g of the powder of Acacia almost dissolves in 2.0 mL of water, and the solution exhibits acidity.
It is practically insoluble in ethanol (95).

Identification Add 25 mL of water and 1 mL of sulfuric acid to 1 g of Acacia and heat the mixture in a boiling water bath with a reflux condenser for 60 minutes. After cooling, gently add 2.0 g of anhydrous sodium carbonate. Add 9 mL of methanol to 1 mL of the resulting solution, centrifuge, and use the clear supernatant as the test solution. Separately, dissolve 10 mg of D-galactose in 1 mL of water, add methanol to make 10 mL, and use this solution as the standard solution (1). Proceed with each of L-arabinose and L-rhamnose hydrate in the same manner, and use these solutions as the standard solutions (2) and (3), respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solutions (1), (2) and (3) on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Develop the plate with a mixture of acetone and water (9 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly 1-naphthol-sulfuric acid TS to the plate, heat at 105 ℃ for 5 minutes; the colors and the R_f values of the 3 spots obtained from the test solution are the same as those of the spots obtained from D-galactose, L-arabinose and L-rhamnose in the standard solution.

Purity (1) ***Insoluble substances***—Add 100 mL of water and 10 mL of dilute hydrochloric acid to 5.0 g of the powdered Acacia, and dissolve over low heat for 15 minutes while shaking. Filter this through a pre-measured glass filter while hot, wash the filtrate well with hot water, and dry at 105 ℃ for 5 hours; the amount of the residue is NMT 10.0 mg.

(2) ***Tannin-containing rubber***—Add 3 drops of ferric chloride TS to the aqueous solution of Acasia (1 in 50); the resulting solution does not exhibit a dark green color.

(3) ***Heavy metals—***Weigh 2.0 g of Acasia and perform the test according to Method 2. Prepare the control solution by adding 4.0 mL of the lead standard solution (NMT 40 ppm).

(4) ***Glucose***—Dissolve 10 mg of glucose in 1 mL of water, add methanol to make 10 mL, and use this solution as the standard solution. Perform the test with the test solution prepared in the Identification and the standard solution as directed under the Thin Layer Chromatography; the spot with the R_f value is the same as that of the glucose spot obtained from the standard solution does not appear in the test solution.

(5) ***Mercury***—Spread about 1 g of the excipient (1) evenly on a ceramic boat, and apply 10 to 300 mg of the powdered Acasia on the top. Spread about 0.5 g of the excipient (a) and 1 g of the excipient (b) on top of it in turn to form a layer. However, in the case of an automatic mercury analyzer equipped with a separate catalyst in the combustion section, take only a sample without adding excipients to the nickel boat. Put the boat in a combustion furnace, let air or oxygen flow in the furnace for 0.5 to 1 L/min, discharge mercury by heating the furnace at about 900 ℃, and collect it with a collection tube. Heat the collection tube at about 700 ℃ to send mercury steam to a cold atomic absorption spectrophotometer, and determine the absorbance as A. Separately, determine the absorbance in the same manner using only the excipients in a ceramic boat and designate as Ab. Separately, proceed in the same manner as for the above steps only using the mercury standard solution, and prepare the calibration curve from the absorbance. Substitute A and Ab values to the calibration curve, and calculate the amount of mercury in the sample (NMT 1.0 ppm).

Operating conditions

Apparatus: Use an automated mercury analyzer from sample combustion to collection by gold amalgam and measurement by cold atomic absorption spectrophotometry. However, a mercury analyzer equipped with a separate catalyst in the combustion section can be used.

Standard mercury stock solution—Dissolve 0.135 g of mercury(II) chloride in 0.001% L-cysteine solution to make 1000 mL. 1 mL of this solution contains 100 μg of mercury(II) chloride.

Mercury standard solution—Dilute the standard mercury stock solution with 0.001% L-cysteine solution to make 0 to 200 ng/mL.

Excipients—Activate excipients at 950 ℃ for 30 minutes when using (a) aluminum oxide and (b) calcium hydroxide-sodium carbonate (1 : 1).

(6) ***Cadmium***—Weigh accurately 5.0 g of Acacia, place it into a platinum crucible, dry and carbonize it, and incinerate at 450 to 550 ℃. If not incinerated well, let it cool down, add 2 to 5 mL of nitric acid (1 in 2) or 50% magnesium nitrate solution or aluminum nitrate-calcium nitrate solution (40 g of aluminum nitrate and 20 g of calcium nitrate dissolved in 100 mL of water) as an incinerating agent to wet it. Dry again, and continue incinerating. If incineration is not sufficient, repeat the above operation once and, if necessary, add 2 to 5 mL of nitric acid (1 in 2) for final incineration. After incinerating completely, moisten the residue with water, add 2 to 4 mL of hydrochloric acid, and evaporate to dryness. Add 0.5 mol/L nitric acid, and dissolve by heating. If there are insoluble substances, filter them with filter paper, add 0.5 mol/L nitric acid to make 25 mL unless otherwise specified, and use this solution as the test solution. Separately, pipet 5.0 mL of the standard cadmium solution, proceed in the same manner as the test solution, and use the resulting solution as the standard solution. Perform the test with the test solution and the standard solutions as directed under Atomic absorption spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 1.0 ppm).

Gas: Acetylene or hydrogen-air
Lamp: A cadmium hollow cathode lamp
Wavelength: 228.8 nm

(7) ***Lead***—Weigh accurately 5.0 g of Acacia, place it into a platinum crucible, dry and carbonate it and incinerate at 450 to 550 ℃ If not incinerated well, let it cool down, add 2 to 5 mL of nitric acid (1 in 2) or 50% magnesium nitrate solution or aluminum nitrate-calcium nitrate solution (40 g of aluminum nitrate and 20 g of calcium nitrate dissolved in 100 mL of water) as an incinerating agent to wet it. Dry again, and continue incinerating. If incineration is not sufficient, repeat the above operation once and, if necessary, add 2 to 5 mL of nitric acid (1 in 2) for final incineration. After incineration, moisten the residue with water, add 2 to 4 mL of hydrochloric acid, and evaporate to dryness. Add 0.5 mol/L nitric acid, and dissolve by heating. If there are insoluble substances, filter them with filter paper, add 0.5 mol/L nitric acid to make 25 mL unless otherwise specified, and use this solution as the test solution. Separately, pipet 0.5 mL of the lead standard solution, proceed in the same manner as the test solution, and use the resulting solution as the standard solution. Perform the test with the test solution and the standard solutions as directed under Atomic absorption spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 1.0 ppm).

Gas: Acetylene or hydrogen-air
Lamp: A lead hollow cathode lamp
Wavelength: 283.3 nm

(8) ***Arsenic***—Weigh 0.5 g of Acacia and perform the test according to Method 3 (NMT 4 ppm).

(9) **Starch and *dextrin***—Dissolve 1 g of Acacia in 50 mL of water, boil and cool the solution, and add a few drops of iodine TS; the resulting solution does not exhibit a blue or red color

Loss on drying NMT 17.0% (4 g, 105 ℃, 6 hours).

Ash NMT 4.0%.

Acid-insoluble ash NMT 0.5%.

Microbial limit The total aerobic microbial count is NMT 1000 CFU per 1 g of Acasia, and the total combined yeasts/mold count is NMT 100 CFU per 1 g of Acasia. Also, *Escherichia coli (E.coli), Salmonella, Pseudomonas aeruginosa* and *Staphylococcus aureus* are not detected.

Acetic Acid
아세트산

Acetic Acid contains NLT 30.0 w/v% and NMT 32.0 w/v% of acetic acid ($C_2H_4O_2$: 60.05).

Description Acetic Acid occurs as a clear and colorless liquid with a characteristic odor and sour taste.
It is miscible with water, ethanol or glycerin.
Specific gravity: About 1.04.

Identification Acetic Acid changes the color of blue litmus paper to red and responds to the Chemical identification reactions for acetate.

Purity (1) ***Chloride, sulfate and potassium permanganate-reducing substances***—Add 40 mL of water to 20 mL of Acetic Acid, and use this solution as the test solution. Perform the test as directed under the Purity (1), (2) and (4) of Glacial Acetic Acid.

(2) ***Heavy metals***—Evaporate 10 mL of Acetic Acid on a water bath to dryness, add 2 mL of dilute acetic acid and water to the residue to make 50 mL. Use this solution as the test solution, and perform the test. Prepare the control solution with 1.0 mL of the lead standard solution, 2 mL of dilute acetic acid and water to make 50 mL (NMT 5 ppm).

(3) ***Arsenic***—Weigh 0.5 g of Acetic Acid and perform the test according to Method 3 (NMT 4 ppm).

(4) ***Residue on evaporation***—Pipet 30 mL of Acetic Acid and perform the test as directed under the Identification (8) of Glacial Acetic Acid.

Assay Pipet 5 mL of Acetic Acid, add 30 mL of water, and titrate with 1 mol/L sodium hydroxide VS (indicator: 2 drops of phenolphthalein TS).

Each mL of 1 mol/L sodium hydroxide VS
= 60.05 mg of $C_2H_4O_2$

Packaging and storage Preserve in tight containers.

Glacial Acetic Acid
아세트산무수물

CH_3COOH

$C_2H_4O_2$: 60.05

Acetic acid [*64-19-7*]

Glacial Acetic Acid contains NLT 99.0% and NMT 101.0% of acetic acid ($C_2H_4O_2$).

Description Glacial Acetic Acid occurs as a clear, volatile colorless liquid or a colorless or white, crystalline mass.
It has a characteristic pungent odor.
It is miscible with water, ethanol or ether.

Boiling point—About 118 °C.
Specific gravity—d_{20}^{20}: About l.049.

Identification An aqueous solution of Glacial Acetic Acid (1 in 3) changes the color of blue litmus paper to red and responds to the Chemical identification reactions for acetate.

Congealing temperature NLT 14.5 ℃.

Purity (1) ***Chloride***—Add water to 10 mL of Glacial Acetic Acid to make 100 mL, and use this solution as the test solution. Add 5 drops of silver nitrate TS to 10 mL of the test solution; no turbidity is produced.

(2) ***Sulfate***—Add 1 mL of barium chloride TS to 10 mL of the test solution prepared in (1); no turbidity is produced.

(3) ***Heavy metals***—Evaporate 2.0 mL of Glacial Acetic Acid on a water bath to dryness, dissolve the residue in 2 mL of dilute acetic acid and water to make 50 mL. Use this solution as the test solution, and perform the test. Prepare the control solution by adding 2 mL of dilute acetic acid and water to 1.0 mL of the lead standard solution to make 50 mL (NMT 5 ppm).

(4) ***Mercury***—Spread about 1 g of the excipient (a) on a ceramic boat evenly, and apply 10 to 300 mg of Glacial Acetic Acid on the top. Spread about 0.5 g of the excipient (a) and 1 g of the excipient (b) on top of it in turn to form a layer. However,

in the case of an automatic mercury analyzer equipped with a separate catalyst in the combustion section, take only a sample without adding excipients to the nickel boat. Put the boat in a combustion furnace, let air or oxygen flow in the furnace for 0.5 to 1 L/min, discharge mercury by heating the furnace at about 900 ℃, and collect it with a collection tube. Heat the collection tube at about 700 ℃ to send mercury steam to a cold atomic absorption spectrophotometer, and determine the absorbance as A. Separately, determine the absorbance in the same manner using only the excipients in a ceramic boat and determine as Ab. Separately, proceed in the same manner as for the above steps only using the mercury standard solution, and prepare the calibration curve from the absorbance. Substitute A and Ab values to the calibration curve, and calculate the amount of mercury in the sample (NMT 1.0 ppm).

Operating conditions

Apparatus: Use an automated mercury measuring instrument for sample combustion, trapping with gold amalgam, and measurement using cold vapor atomic absorption spectrophotometry. However, a mercury analyzer equipped with a separate catalyst in the combustion section can be used.

Standard mercury stock solution—Dissolve 0.135 g of mercury(II) chloride in 0.001% L-cysteine solution to make 1000 mL. 1 mL of this solution contains 100 μg of mercury(II) chloride.

Mercury standard solution—Dilute the standard mercury stock solution with 0.001% L-cysteine solution to make 0 to 200 ng/mL.

Excipient—Activate excipients at 950 ℃ for 30 minutes when using (a) aluminum oxide and (b) calcium hydroxide-sodium carbonate (1 : 1).

(5) ***Lead***—Weigh accurately 5.0 g of Glacial Acetic Acid, place it into a platinum crucible, dry and carbonate it, and incinerate at 450 to 550 ℃. If incineration is not complete, cool and moisten with 2 to 5 mL of nitric acid (1 in 2), 50% magnesium nitrate solution or aluminum nitrate-calcium nitrate solution (prepared by dissolving 40 g of aluminum nitrate and 20 g of calcium nitrate in 100 mL of water) as an incineration supplement, dry, and continue with incineration. If incineration is incomplete, repeat the above procedure once and then add a final 2 mL to 5 mL of nitric acid (1 in 2), if necessary, and incinerate. After incineration, moisten the residue with water, add 2 to 4 mL of hydrochloric acid, and evaporate to dryness. Add 0.5 mol/L nitric acid, dissolve by warming, and filter with filter paper if any insoluble matter exists. Unless otherwise specified, add 0.5 mol/L nitric acid to make 25 mL and use this solution as the test solution. Separately, take 0.25 mL of lead standard solution to a platinum crucible, proceed in the same manner as the test solution, and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Atomic Absorption Spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 0.5 ppm).

Gas: Acetylene or hydrogen - Air.
Lamp: A lead hollow cathode lamp
Wavelength: 283.3 nm

(6) ***Arsenic***—Weigh 1.54 g of Glacial Acetic Acid and perform the test according to Method 1 (NMT 1.3 ppm).

(7) ***Potassium permanganate-reducing substances***—Add 0.10 mL of 0.02 mol/L potassium permanganate to 20 mL of the test solution prepared in (1); the red color of the resulting solution does not disappear within 30 minutes.

(8) ***Residue on evaporation***—Evaporate and dry 10 mL of Glacial Acetic Acid on a water bath and dry the residue at 105 ℃ for 1 hour; the amount of the residue is NMT 1.0 mg.

Assay Add 10 mL of water to a stoppered flask, weigh its mass accurately, add about 1.5 g of Glacial Acetic Acid, and weigh accurately the solution again. Add 30 mL of water, and titrate 1 mol/L sodium hydroxide VS (indicator: 2 drops of phenolphthalein TS).

Each mL of 1 mol/L sodium hydroxide VS
= 60.05 mg of $C_2H_4O_2$

Packaging and storage Preserve in tight containers.

Agar
한천(寒天)

Agar is obtained by freeze-drying of a mucilage derived from *Gelidium amansii* Lamouroux, other congeneric plants (*Gelidiaceae*), or various species of red algae (*Rhodophyta*).

Description Agar is white, semi-translucent and comes in rectangular columns, strings or thin pieces of flakes. Agar in the form of a rectangular column is about 26 cm in length, 4 cm^2 in cross section; the string form is about 35 cm in length and about 3 mm in width and the flake forms are thin pieces of about 3 mm in length, externally, with wrinkles and somewhat lustrous on the surface, light and pliable.
It is practically insoluble in organic solvents.
A boiling aqueous solution of Agar (1 in 100) is neutral.
It is odorless, tasteless and mucilagenous.

Identification (1) To a fragmented piece of Agar, drop iodine TS; a dark blue to reddish violet color develops.

(2) Dissolve 1 g of Agar in 65 mL of water by boiling for 10 minutes with constant stirring and add a sufficient amount of hot water to replenish the water lost by evaporation. The solution is clear. Cool the solution to 30 °C to 39 °C; the solution forms an elastic gel, which does not melt below 85 °C when heated.

Purity (1) ***Sulfuric acid***—Dissolve 1.0 g of Agar in 100 mL of water by boiling; the solution does not show acidity.

(2) ***Sulfurous acid and starch***—To 5 mL of the solution obtained in (1), add 2 drops of iodine TS; the solution does not decolorize immediately. The solution does not exhibit a blue color.

(3) ***Insoluble matter***—To 7.5 g of Agar, add 500 mL of water, boil for 15 minutes and add water to make exactly 500 mL. Pipet 100 mL of this solution, add 100 mL of hot water, heat to boiling, filter while hot through a glass filter, previously weighed, wash the residue with a small amount of hot water and dry the residue at 105 °C for 4 hours; the amount of the residue is NMT 15.0 mg.

(4) ***Water absorption***—To 5.0 g of Agar, add water to make 100 mL, shake well to mix, allow to stand at 25 °C for 24 hours and filter through moistened glass fiber in a 100-mL measuring cylinder; the volume of the filtrate is NMT 75 mL.

(5) ***Heavy metals***—Proceed with 1.0 g of Agar according

to Method 2 and perform the test. Prepare the control solution with 4.0 mL of lead standard solution (NMT 40 ppm).

(6) ***Arsenic***—Proceed with 0.67 g of Agar according to Method 2 and perform the test. (NMT 3 ppm).

(7) ***Gelatin***—Dissolve 1 g of Agar in 100 mL of boiling water and cool to 50 °C. To 5 mL of this solution, add 2 to 3 drops of a mixture of 0.2 mol/L dichromic acid TS and 3 mol/L hydrochloric acid TS (4 : 1); no yellow precipitate is produced.

Loss on drying NMT 22.0% (6 hours).

Ash NMT 4.5%.

Acid-insoluble ash NMT 0.5%.

Microbial limit The total aerobic microbial count is NMT 1000 CFU, and the total combined yeasts/moulds count is NMT 100 CFU per 1 g of Agar. Also, *Escherichia coli*, *Salmonella spp.*, *Pseudomonas aeruginosa* and *Staphylococcus aureus* are not detected.

Aluminum Monostearate
알루미늄모노스테아레이트

Aluminum Monostearate is mainly aluminum compound of stearic acid (C18H36O2: 284.48) and palmitic acid (C16H32O2: 256.42). Aluminum Monostearate, when dried, contains NLT 7.2% and NMT 8.9% of aluminum (Al: 26.98).

Description Aluminum Monostearate occurs as a white to pale yellow powder. It is odorless or has a slightly characteristic odor. It is practically insoluble in water, ethanol or ether.

Identification (1) To 3 g of Aluminum Monostearate, add 30 mL of hydrochloric acid, heat the mixture on a water bath for 10 minutes with occasional shaking to mix, and cool. Add 50 mL of water and 30 mL of ether, shake vigorously for 3 minutes to mix, and allow to stand. Separately, take the aqueous layer, add sodium hydroxide TS until the solution becomes slightly turbid, and filter: the filtrate responds to the Chemical identification reactions for aluminum salt.

(2) Wash the ether layer separated in (1) twice with 20 mL of water and evaporate the ether layer on a water bath; the residue melts at above 54 °C (Method 2).

Acid value of fatty acid 193 to 210. Weigh accurately about 1 g of the fatty acid obtained in the Identification (2), place in a 250-mL flask with a stopper, add 100 mL of a mixture of ether and ethanol (2 : 1), and dissolve by warming. Add several drops of phenolphthalein TS and perform the test with the resulting solution as directed in the acid value under the Fats and Fatty Oils.

Purity (1) ***Free fatty acid***—Weigh 1.0 g of Aluminum Monostearate, add to about 50 mL of a mixture of neutralized ethanol and ether (1 : 1), shake to mix, and filter with a dry filter paper. Wash the container and filter paper with a small amount of a mixture of neutralized ethanol and ether (1 : 1), combine the washings with the filtrate, and add 2.1 mL of 0.1 mol/L potassium hydroxide solution; the solution exhibits a red color.

(2) ***Water-soluble salts***—Transfer about 2.0 g of Aluminum Monostearate into an Erlenmeyer flask, add 80 mL of water, loose stopper, and heat on a water bath for 30 minutes with occasional shaking. After cooling, filter through dry filter paper, wash the residue with a small amount of water, combine the washings with the filtrate add water to make 100 mL. Evaporate 50 mL of this solution on a water bath and ignite at 600 °C; the residue is NMT 10.0 mg.

(3) ***Heavy metals***—Heat 1.0 g of Aluminum Monostearate over a small flame with caution at the beginning and continue the heating while gradually raising the temperature to incinerate. After cooling, add 10 mL of diluted hydrochloric acid (1 in 2), evaporate on a water bath, add 20 mL of water to the residue, and boil for 1 minute. After cooling, filter, wash with water, and combine the filtrate and the washings. Add 2 mL of dilute acetic acid and then water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution by evaporating 10 mL of diluted hydrochloric acid (1 in 2) on a water bath to dryness, add 2 mL of dilute acetic acid and 5.0 mL of the lead standard solution to make 50 mL (NMT 50 ppm).

(4) ***Arsenic***—Mix 1.0 g of Aluminum Monostearate with 2 g of magnesium nitrate, incinerate over a small flame, moisten the residue after cooling with 0.5 mL of nitric acid, and heat again. Add 10 mL of dilute sulfuric acid to the residue and heat until white fumes evolve, and add water to make 5 mL. Use this solution as the test solution and perform the test (NMT 2 ppm).

Loss on drying NMT 3.0% (1 g, 105 °C, 3 hours).

Assay Weigh accurately about 1 g of Aluminum Monostearate, previously dried, and incinerate over a weak flame. After cooling, add 0.5 mL of nitric acid dropwise, heat on a water bath to evaporate, ignite at 900 to 1100 °C to constant mass, and cool. Then, immediately measure the mass to determine the amount of aluminum oxide (Al_2O_3: 101.96).

$$\text{Amount (mg) of aluminum (Al)} = \text{Amount (mg) of aluminum oxide } (Al_2O_3) \times 0.5293$$

Packaging and storage Preserve in well-closed containers.

Aluminum Potassium Sulfate Hydrate
황산알루미늄칼륨수화물

Alum

Potassium alum $AlK(SO_4)_2 \cdot 12H_2O$: 474.39

Aluminum Potassium Sulfate Hydrate contains NLT 99.5% and NMT 101.0% of aluminum potassium sulfate hydrate [$AlK(SO_4)_2 \cdot 12H_2O$].

Description Aluminum Potassium Sulfate Hydrate occurs as a colorless to white crystal or powder, is odorless and has a slightly sweet, strong astringent taste.

It is freely soluble in water and practically insoluble in ethanol or ether.

An aqueous solution of Aluminum Potassium Sulfate Hydrate (1 in 20) is acidic.

Identification An aqueous solution of Aluminum Potassium Sulfate Hydrate (1 in 10) responds to the Chemical identification reactions for aluminum salt, the Chemical identification reactions (1), (3) and (4) for potassium salt and the Chemical identification reactions (1) and (3) for sulfate.

Purity (1) ***Clarity and color of solution***—Dissolve 1 g of Aluminum Potassium Sulfate Hydrate in 10 mL of water; the

solution is colorless and almost clear. To 2 g of dried Aluminum Potassium Sulfate Hydrate, add 200 mL of water, boil for 10 minutes and cool. Filter through a glass filter, wash the insoluble residue with 100 mL of water and dry at 105 °C for 2 hours; the amount is NMT 40 mg.

(2) ***Heavy metals***—Proceed with 1.0 g of Aluminum Potassium Sulfate Hydrate according to Method 1 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 20 ppm).

(3) ***Mercury***—Spread evenly about 1 g of excipient (a) into a ceramic boat and place 10 mg to 300 mg of Aluminum Potassium Sulfate Hydrate on top. Next, spread evenly about 0.5 g of excipient (a) and 1 g of excipient (b) successively to form layers. In the case of an automated mercury analyzer equipped with a separate catalyst in the combustion chamber, take only the sample without adding excipients to a nickel boat. Place the boat inside the combustion furnace and heat to about 900 °C with a current of air or oxygen at 0.5 to 1 L/minute. Elute the mercury and collect in a collecting tube. Transfer the mercury vapor to a cold vapor atomic absorption spectrometer by heating the collecting tube to about 700 °C and determine the absorbance as A. Separately, determine the absorbance in the same manner using only the excipients in a ceramic boat and designate as Ab. Separately, proceed with mercury standard solution in the same manner and create a calibration curve from the absorbances. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the sample (NMT 1.0 ppm).

Operating conditions

Apparatus: Use an automated mercury analyzer for specimen combustion, trapping with gold amalgam and measurement using a cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Standard mercury stock solution—Dissolve 0.135 g of mercury (II) chloride in 0.001% L-cysteine to make 1000 mL. Each mL of this solution contains 100 μg of mercury(II) chloride.

Mercury standard solution—Dilute standard mercury stock solution with 0.001% L-cysteine so that each mL contains 0 to 200 ng.

Excipients—Use (a) aluminum oxide and (b) a mixture of calcium hydroxide and sodium carbonate (1 : 1) and activate at 950 °C for 30 minutes before use.

(4) ***Lead***—Weigh accurately 5.0 g of Aluminum Potassium Sulfate Hydrate, transfer to a 150-mL beaker and add 30 mL of water. Add hydrochloric acid in small amounts until the sample is completely dissolved, and then add 1 mL of hydrochloric acid. Boil the solution for about 5 minutes, cool, add water to make about 100 mL and adjust to have the pH of 2 to 4 with sodium hydroxide solution (1 in 4) or hydrochloric acid (1 in 4). Transfer this solution to a 250-mL separatory funnel, add water to make about 200 mL, add 2 mL of 2% ammonium pyrrolidine dithiocarbamate solution (2 in 100) and shake well to mix. Extract this solution twice with 20 mL each of chloroform and evaporate the extract to dryness on a water bath. To the residue, add 3 mL of nitric acid and heat until almost dry. Add 0.5 mL of nitric acid and 10 mL of water, concentrate until the final solution becomes 3 mL to 5 mL, add water to make 10 mL, and use this solution as the test solution. Separately, proceed with 2.5 mL of lead standard solution in a platinum crucible in the same manner as the test solution, and use this solution as the standard solution. Perform the test with the test solution and the standard solutions as directed under Atomic Absorption Spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 5.0 ppm).

Gas: Acetylene or hydrogen-air.
Lamp: Lead hollow-cathode lamp
Wavelength: 283.3 nm

(5) ***Iron***—Prepare the test solution with 1.0 g of Aluminum Potassium Sulfate Hydrate according to Method 1 and perform the test according to Method A. Prepare the control solution with 2.0 mL of the iron standard solution (NMT 20 ppm).

(6) ***Selenium***—Weigh accurately 0.2 g of Aluminum Potassium Sulfate Hydrate, add carefully to a 150-mL beaker containing 25 mL of 4 mol/L hydrochloric acid, mix and heat to boil. Heat on a water bath for 15 minutes, add 25 mL of water, cool, and use this solution as the test solution. Transfer 2 mL of the standard solution to a beaker, dilute with 50 mL of 2 mol/L hydrochloric acid and use this solution as the control solution. Use 50 mL of 2 mol/L hydrochloric acid as the blank test solution. To the test solution, the control solution and the blank test solution, add carefully 5 mL of ammonia water, cool and adjust the pH of each solution to between 1.8 and 2.2 with ammonia water (1 in 2). To each solution, add 0.2 g of hydroxylamine hydrochloride, shake carefully to dissolve, add immediately 5 mL of 2,3-diaminonaphthalene, mix and allow to stand for 100 minutes. Transfer each solution to a separatory funnel, wash with 10 mL of water, combine and extract with 5 mL of cyclohexane. Discard the water layer, centrifuge the cyclohexane layer to remove traces of water and determine the absorbances at 380 nm; the absorbance of the test solution is not greater than that of the control solution (NMT 30 ppm).

Standard solution—Dilute selenium standard solution with water to make the concentration at 3 ppm.

2,3-Diaminonaphthalene—Dissolve 0.1 g of 2,3-diaminonaphthalene and 0.5 g of hydroxylamine hydrochloride in 0.1 mol/L hydrochloric acid to make 100 mL.

(7) ***Arsenic***—Proceed with 0.6 g of Aluminum Potassium Sulfate Hydrate according to Method 1, and perform the test (NMT 3.3 ppm).

(8) ***Fluoride***—Weigh 1 g of Aluminum Potassium Sulfate Hydrate, transfer to a beaker and dissolve in 10 mL of hydrochloric acid (1 in 10). Heat this solution, boil for 1 minute, transfer to a polyethylene beaker and cool immediately. Add 15 mL of sodium citrate solution (1 in 4) and 10 mL of ethylenediaminetetraacetic acid disodium salt solution (1 in 40) and shake to mix. Adjust the pH to between 5.4 and 5.6 with hydrochloric acid (1 in 10) or sodium hydroxide solution (2 in 5), add water to make 100 mL and use this solution as the test solution. Transfer 50 mL of the test solution to a polyethylene beaker, determine the potential using a fluoride electrode and determine the amount of fluoride from the calibration curve; the amount is NMT 30 ppm.

Calibration curve: Weigh accurately 2.210 g of sodium fluoride, previously dried at 200 °C for 4 hours, and transfer to a polyethylene beaker. Dissolve in 200 mL of water, add water to make 1 L and store in a polyethylene container. Pipet 5 mL of this solution, transfer to a volumetric flask and add water to make 1000 mL (each mL of this solution contains 5 μg of fluoride). Take 1 mL, 2 mL, 3 mL, 5 mL, 10 mL and 15 mL of this solution, transfer each to a polyethylene beaker, add 15 mL of sodium

citrate solution (1 in 4) and 10 mL of ethylenediaminetetraacetic acid disodium salt solution (1 in 40), and mix. Adjust the pH to between 5.4 and 5.6 with hydrochloric acid (1 in 10) or sodium hydroxide solution (2 in 5), add water to make 100 mL each and use these solutions as the standard solutions. Pipet 50 mL of each standard solution into polyethylene beakers. Determine the potential using a fluoride electrode and plot a calibration curve with the log values of the fluoride concentrations.

Assay Weigh accurately about 4.5 g of Aluminum Potassium Sulfate Hydrate and dissolve in water to make exactly 200 mL. Pipet 20 mL of this solution, add exactly 30 mL of 0.05 mol/L ethylenediaminetetraacetic acid disodium salt solution, add 20 mL of pH 4.8 acetic acid-ammonium acetate buffer solution, and then boil for 5 minutes. After cooling, add 55 mL of ethanol, and titrate with 0.02 mol/L zinc sulfate VS (indicator: 2 mL of dithizone TS). However, the endpoint of the titration is when the color of the solution changes from pale dark green to pale red. Perform a blank test in the same manner.

Each mL of 0.05 mol/L Ethylenediaminetetraacetic acid disodium salt VS
= 23.719 mg of $AlK(SO_4)_2 \cdot 12H_2O$

Packaging and storage Preserve in tight containers.

Dried Aluminum Potassium Sulfate
건조황산알루미늄칼륨

Burnt Alum
Exsiccated Alum
Anhydrous Aluminum Potassium Sulfate
Aluminum Potassium Sulfate $AlK(SO_4)_2$: 258.21

Dried Aluminum Potassium Sulfate, when dried, contains NLT 98.0% and NMT 101.0% of aluminum potassium sulfate [$AlK(SO_4)_2$].

Description Dried Aluminum Potassium Sulfate occurs as a white mass or powder, is odorless and has a slightly sweet, astringent taste.
It is freely soluble in hot water and practically insoluble in ethanol.
It dissolves slowly in water.

Identification An aqueous solution of Dried Aluminum Potassium Sulfate (1 in 20) responds to the Chemical identification reactions for aluminum salt, the Chemical identification reactions (1), (3) and (4) for potassium salt and the Chemical identification reactions (1) and (3) for sulfate.

Purity (1) ***Clarity and color of solution***—Dissolve 1 g of crystals of Dried Aluminum Potassium Sulfate in 10 mL of water: the solution is colorless. To 2 g of Dried Aluminum Potassium Sulfate, add 200 mL of water, boil for 10 minutes and cool. Filter through a glass filter, wash the insoluble residue with 100 mL of water and dry with the glass filter at 105 °C for 2 hours; the amount is NMT 40 mg.

(2) ***Water-insoluble substances***—Take 2.0 g of Dried Aluminum Potassium Sulfate, add 40 mL of water, shake frequently and allow to stand for 48 hours. Collect the insoluble residue on a glass filter, wash with 50 mL of water and dry at 105 °C for 2 hours; the amount is NMT 50 mg.

(3) ***Mercury***—Spread evenly about 1 g of excipient (a) into a ceramic boat and place 10 mg to 300 mg of Dried Aluminum Potassium Sulfate on top. Next, spread evenly about 0.5 g of excipient (a) and 1 g of excipient (b) successively to form layers. In the case of an automatic mercury analyzer equipped with a separate catalyst in the combustion chamber, place only the sample in a nickel boat without the excipients. Place the boat inside the furnace and heat to about 900 °C with a current of air or oxygen at 0.5 to 1 L/minute. Elute the mercury and collect in a sampling tube. Transfer the mercury vapor to a cold vapor atomic absorption spectrophotometer by heating the sampling tube to about 700 °C and determine the absorbance, A. Separately, place only the excipients in the ceramic boat and determine the absorbance, Ab, in the same manner. Separately, proceed with mercury standard solution in the same manner and plot a calibration curve from the absorbances thus determined. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the sample (NMT 1.0 ppm).

Operating conditions
Apparatus: Use a mercury analyzer automated from sample combustion to collection by gold amalgam and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Standard mercury stock solution—Dissolve 0.135 g of mercury (II) chloride in 0.001% L-cysteine to make 1000 mL. 1 mL of this solution contains 100 μg of mercury(II) chloride.

Mercury standard solution—Dilute standard mercury stock solution with 0.001% L-cysteine so that each mL contains 0 to 200 ng.

Excipients—Use (a) aluminum oxide and (b) a mixture of calcium hydroxide and sodium carbonate (1 : 1) and activate at 950 °C for 30 minutes before use.

(4) ***Lead***—Weigh accurately 5.0 g of Dried Aluminum Potassium Sulfate, transfer to a 150-mL beaker and add 30 mL of water. Add hydrochloric acid in small amounts until the sample is completely dissolved, and then add 1 mL of hydrochloric acid. Boil the solution for about 5 minutes, cool, add water to make about 100 mL and adjust the pH to 2 to 4 with sodium hydroxide solution (1 in 4) or hydrochloric acid (1 in 4). Transfer this solution to a 250-mL separatory funnel, add water to make about 200 mL, 2 mL of 2% ammonium pyrrolidine dithiocarbamate solution (2 in 100) and shake well to mix. Extract this solution twice with 20 mL each of chloroform and evaporate the extract to dryness on a water bath. To the residue, add 3 mL of nitric acid and heat until almost dry. Add 0.5 mL of nitric acid and 10 mL of water, concentrate until the final solution becomes 3 mL to 5 mL, add water to make 10 mL, and use this solution as the test solution. Separately, transfer 2.5 mL of lead standard solution to a platinum crucible, proceed in the same manner as the test solution and use this solution as the standard solution Perform the test with the test solution and the standard solutions as directed under Atomic Absorption Spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 5.0 ppm).

Gas: Acetylene or hydrogen - Air.
Lamp: A lead hollow-cathode lamp
Wavelength: 283.3 nm

(5) ***Heavy metals***—Dissolve 0.5 g of Dried Aluminum Potassium Sulfate in 45 mL of water, and filter the solution, if

necessary. Add 2 mL of dilute acetic acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution by adding 2 mL of dilute acetic acid and water to 2.0 mL of lead standard solution to make 50 mL (NMT 40 ppm).

(6) ***Selenium***—Weigh accurately 0.2 g of Dried Aluminum Potassium Sulfate, add carefully to a 150-mL beaker containing 25 mL of 4 mol/L hydrochloric acid, mix and heat to boil. Heat on a water bath for 15 minutes, add 25 mL of water, cool, and use this solution as the test solution. Transfer 2 mL of the standard solution to a beaker, dilute with 50 mL of 2 mol/L hydrochloric acid and use this solution as the control solution. Use 50 mL of 2 mol/L hydrochloric acid as the blank test solution. To the test solution, the control solution and the blank test solution, add carefully 5 mL of ammonia water, cool and adjust the pH of each solution to between 1.8 and 2.2 with ammonia water (1 in 2). To each solution, add 0.2 g of hydroxylamine hydrochloride, shake carefully to dissolve, add immediately 5 mL of 2,3-diaminonaphthalene, mix and allow to stand for 100 minutes. Transfer each solution to a separatory funnel, wash with 10 mL of water, combine and extract with 5 mL of cyclohexane. Discard the water layer, centrifuge the cyclohexane layer to remove traces of water and determine the absorbances at 380 nm; the absorbance of the test solution is not greater than that of the control solution (NMT 30 ppm).

Standard solution—Dilute selenium standard solution with water to make the concentration at 3 ppm.

2,3-Diaminonaphthalene—Dissolve 0.1 g of 2,3-diaminonaphthalene and 0.5 g of hydroxylamine hydrochloride in 0.1 mol/L hydrochloric acid to make 100 mL.

(7) ***Iron***—Prepare the test solution with 0.54 g of Dried Aluminum Potassium Sulfate according to Method 1 and perform the test according to Method A. Prepare the control solution with 2.0 mL of the iron standard solution (NMT 37 ppm).

(8) ***Arsenic***—Proceed with 0.40 g of Dried Aluminum Potassium Sulfate according to Method 1, and perform the test (NMT 5 ppm).

(9) ***Fluoride***—Weigh 1 g of Dried Aluminum Potassium Sulfate, transfer to a beaker and dissolve in 10 mL of hydrochloric acid (1 in 10). Heat this solution, boil for 1 minute, transfer to a polyethylene beaker and cool immediately. Add 15 mL of sodium citrate solution (1 in 4) and 10 mL of ethylenediaminetetraacetic acid disodium salt solution (1 in 40) and shake to mix. Adjust the pH to between 5.4 and 5.6 with hydrochloric acid (1 in 10) or sodium hydroxide solution (2 in 5), add water to make 100 mL and use this solution as the test solution. Transfer 50 mL of the test solution to a polyethylene beaker, determine the potential using a fluoride electrode and determine the amount of fluoride from the calibration curve; the amount is NMT 30 ppm.

Creating a calibration curve: Weigh accurately 2.210 g of sodium fluoride, previously dried at 200 °C for 4 hours, and transfer to a polyethylene beaker. Dissolve in 200 mL of water, add water to make 1 L and store in a polyethylene container. Pipet 5 mL of this solution, transfer to a volumetric flask and add water to make 1000 mL (each mL of this solution contains 5 μg of fluoride). Take 1 mL, 2 mL, 3 mL, 5 mL, 10 mL and 15 mL of this solution, transfer each to a polyethylene beaker, add 15 mL of sodium citrate solution (1 in 4) and 10 mL of ethylenediaminetetraacetic acid disodium salt solution (1 in 40) and mix. Adjust the pH to between 5.4 and 5.6 with hydrochloric acid (1 in 10) or sodium hydroxide solution (2 in 5), add water to make 100 mL each and use these solutions as the standard solutions. Pipet 50 mL of each standard solution into polyethylene beakers. Determine the potential using a fluoride electrode and create a calibration curve with the log values of the fluoride concentrations.

Loss on drying NMT 15.0% (2 g, 200 °C, 4 hours).

Assay Weigh accurately about 1.2 g of Dried Aluminum Potassium Sulfate, previously dried, add 80 mL of water, and heat on a water bath with occasional shaking for 20 minutes. Cool and add water to make exactly 100 mL. Filter, if necessary. Discard the first 30 mL of the filtrate, take exactly the subsequent 20 mL of the filtrate and proceed as directed in the Assay under Aluminum Potassium Sulfate Hydrate.

Each mL of 0.05 mol/L ethylenediaminetetraacetic acid disodium salt VS
= 12.910 mg of $AlK(SO_4)_2$

Packaging and storage Preserve in tight containers.

Apricot Kernel Water
행인수

Apricot Kernel Water contains NLT 0.09 w/v% and NMT 0.11 w/v% of hydrogen cyanide (HCN: 27.03).

Method of preparation Prepare by one of the following methods.

(1) Take apricot kernels, previously ground and pressed to remove fatty oils as much as possible, add a suitable amount of water or purified water, and carry out steam distillation. Determine the content of hydrogen cyanide in the distillate as directed in the Assay, and carry on the distillation until the content of hydrogen cyanide in the distillate reaches about 0.14%. To the distillate, add ethanol of about 1/3 of the volume of the distillate, and add a mixture of purified water and ethanol (3 : 1) until the content of hydrogen cyanide meets the specification.

(2) Dissolve 7.5 mL of freshly prepared mandelonitrile in 1000 mL of a mixture of purified water and ethanol (3 : 1), mix well, and filter. Determine the content of hydrogen cyanide in this solution as directed in the Assay, and, if the content exceeds the specification, dilute the solution to the specified concentration by adding the mixture of purified water and ethanol (3 : 1).

Description Apricot Kernel Water occurs as a clear, colorless to pale yellow liquid, has an odor of benzaldehyde and has a characteristic taste

pH—Between 3.5 and 5.0.

Identification To 2 mL of Apricot Kernel Water, add 1 mL of ammonia TS, and allow to stand for 10 minutes; a slight turbidity is produced. Allow to stand for 20 minutes; the turbidity becomes more intense.

Specific gravity d_{20}^{20}: Between 0.968 and 0.978

Purity (1) ***Sulfate***—To 5.0 mL of Apricot Kernel Water, add 0.1 mol/L sodium hydroxide to make slightly alkaline, evaporate on a water bath to dryness, and ignite at 450 °C to 550 °C. Dissolve the residue in 1 mL of dilute hydrochloric acid, and add water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution with 0.50 mL of

0.005 mol/L (NMT 0.005%).

(2) ***Heavy metals***—Evaporate 50 mL of Apricot Kernel Water on a water bath to dryness, ignite at 450 ℃ to 550 ℃, dissolve the residue in 5 mL of dilute acetic acid with warming, add water to make exactly 50 mL, and filter. Discard the first 10 mL of the filtrate, take 20 mL of the subsequent filtrate, and add water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution by adding 2 mL of dilute acetic acid and water to 2.0 mL of lead standard solution to make 50 mL (NMT 1 ppm).

(3) ***Free hydrogen cyanide***—To 10 mL of Apricot Kernel Water, add 0.8 mL of 0.1 mol/L silver nitrate solution and 2 to 3 drops of nitric acid at 15 ℃, filter, and add 0.1 mol/L silver nitrate solution to the filtrate; the solution remains unchanged.

(4) ***Residue on evaporation***—Evaporate 5.0 mL of Apricot Kernel water to dryness, and dry the residue at 105 ℃ for 1 hour; the amount of the residue is NMT 1.0 mg.

Assay Pipet 25 mL of Apricot Kernel Water, add 100 mL of water, 2 mL of potassium iodide TS and 1 mL of ammonia TS, and titrate with 0.1 mol/L silver nitrate VS until a persistent, yellow turbidity develops.

Each mL of 0.1 mol/L silver nitrate VS
= 5.405 mg of HCN

Packaging and storage Preserve in light-resistant, tight containers.

Beef Tallow
우지

Beef Tallow is a purified fat obtained by wet rendering from the fresh adipose tissue of *Bos taurus* Linné var. *domesticus* Gmelin (Bovidae).

Description Beef Tallow occurs as a white, uniform mass, has a slight, characteristic odor and a mild taste.
It is freely soluble in ether or petroleum ether, very slightly soluble in ethanol, and practically insoluble in water.
It is breakable at low temperatures but softens at temperatures NLT 30 °C.

Melting point—Between 42 ℃ and 50 ℃ (Method 2).

Saponification value Between 193 and 200.

Acid value NMT 2.0.

Iodine value 33 to 50. (When the sample is not soluble in 20 mL of cyclohexane, dissolve it by shaking a stoppered flask in warm water. If it is still not soluble, increase the volume of solvent.).

Purity (1) ***Water and coloration***—Melt 5.0 g of Beef Tallow by heating on a water bath; the resulting liquid is clear and no water separates from it. Observe this liquid in a 10-mm thick layer; it is colorless to pale yellow.

(2) ***Alkalinity***—To 2.0 g of Beef Tallow, add 10 mL of water, dissolve by heating on a water bath and shake vigorously. After cooling, add 1 drop of phenolphthalein TS to the separated infusion; the solution is colorless.

(3) ***Chloride***—To 1.5 g of Beef Tallow, add 30 mL of ethanol, and boil the mixture under a reflux condenser for 10 minutes. Filter after cooling. To 20 mL of the filtrate, add 5 drops of a solution of silver nitrate in ethanol (1 in 50); the turbidity of the resulting solution is not more intense than the following control solution

Control solution—To 1.0 mL of 0.01 mol/L hydrochloric acid, add ethanol to make 50 mL and add 5 drops of a solution of silver nitrate in ethanol (1 in 50).

Packaging and storage Preserve in well-closed containers.

White Beeswax
백납

White Beeswax is prepared by bleaching Beeswax.

Description White Beeswax occurs as a white to yellow mass with a characteristic odor. It is relatively brittle when cold, and the broken surface is amorphous.
It is slightly soluble in ether and practically insoluble in water or anhydrous ether.

Saponification value Between 80 and 100. Weigh accurately about 3 g of White Beeswax, transfer to a 250-mL flask with a stopper, add exactly 25 mL of 0.5 mol/L potassium hydroxide-ethanol solution and 50 mL of ethanol, and heat the mixture on a water bath for 4 hours with a reflux condenser. Perform the test as directed in the Saponification value under Fats and Fatty Oils.

Acid value Between 5 and 9 or between 17 and 22. Weigh accurately about 6 g of White Beeswax, transfer it to a 250-mL flask with a stopper, add 50 mL of anhydrous ethanol, and warm the mixture to dissolve. Add 1 mL of phenolphthalein TS, and perform the test as directed in the Acid value under Fats and Fatty Oils. However, do not neutralize the solvent in advance. Perform a blank test in the same manner, and make any necessary correction.

Melting point Between 60 ℃ and 67 ℃ (Method 2).

Ester value Between 72 and 79.

Purity (1) ***Paraffin, fat, japan wax or resin, ceresin and other waxes***—Melt White Beeswax at as low a temperature as possible and drop it into ethanol to make grains. Allow it to stand in air for 24 hours, and pour it into a mixture of ethanol and water with a specific gravity of 0.95 and 0.97, respectively; the grains sink or float in a mixture with a specific gravity of 0.95, and float in a mixture with a specific gravity of 0.97.

(2) ***Glycerin and polyol***—Weigh accurately 0.2 g of White Beeswax, add 10 mL of potassium hydroxide-ethanol solution, and heat the mixture on a steam bath of a reflux condenser for 30 minutes. Add 50 mL of dilute sulfuric acid to the resulting solution, cool, and filter. Wash the flask, add the washings to the filtrate, and dilute with dilute sulfuric acid to 100 mL. Use this solution as the test solution. Transfer 1.0 mL of this solution to a test tube, add 0.5 mL of sodium periodate TS (10.7 in 1000), shake to mix, and allow the mixture to stand for 5 minutes. Add 1.0 mL of fuchsin solution decolorized, and shake to mix; all precipitates disappear. Place the test tube in a beaker containing water at 40 °C, and cool for 10 to 15 minutes. Separately, weigh accurately glycerin RS, dissolve in dilute sulfuric acid, and make a solution containing 10 mg per 1000 mL. Proceed with 1.0 mL

of this solution in the same manner as the test solution, and use the resulting solution as the standard solution. At this time, the color of the test solution at the same time is not more intense than that of the standard solution (NMT 0.5% as glycerin).

(3) ***Mercury***—Spread evenly about 1 g of the excipient (a) on a ceramic boat, and apply 10 to 300 mg of White Beeswax on the top. Spread evenly about 0.5 g of the excipient (a) and 1 g of the excipient (b) on the top of it again to form a layer. However, in the case of an automatic mercury analyzer equipped with a separate catalyst in the combustion section, take only a sample without adding excipients to the nickel boat. Put the boat in a combustion furnace, let air or oxygen flow in the furnace for 0.5 to 1 L/min, discharge mercury by heating the furnace at about 900 ℃, and collect it with a collection tube. Heat the collection tube at about 700 ℃ to send mercury steam to a cold atomic absorption spectrophotometer, and determine the absorbance as A. Separately, determine the absorbance in the same manner using only the excipients in a ceramic boat and designate as Ab. Separately, proceed in the same manner as for the above steps only using the mercury standard solution, and prepare the calibration curve from the absorbance. Substitute A and Ab values to the calibration curve, and calculate the amount of mercury in the sample (NMT 1.0 ppm).

Operating conditions

Apparatus: Use an automated mercury analyzer from sample combustion to collection by gold amalgam and measurement by cold atomic absorption spectrophotometry. However, a mercury analyzer equipped with a separate catalyst in the combustion section can be used.

Standard mercury stock solution—Dissolve 0.135 g of mercuric chloride in 0.001% L-cysteine solution to make 1000 mL (1 mL of the standard mercury stock solution = 100 μg of Hg).

Mercury standard solution—Dilute the standard mercury stock solution with 0.001% L-cysteine solution to make 0 to 200 ng/mL.

Excipients—Activate excipients at 950 ℃ for 30 minutes when using (a) aluminum oxide and (b) calcium hydroxide-sodium carbonate (1 : 1).

(4) ***Lead***—Weigh accurately about 5.0 g of White Beeswax and place it in a platinum crucible. Dry, carbonize, and incinerate it at 450 to 550 °C. If not incinerated well, let it cool down, add 2 to 5 mL of nitric acid (1 in 2) or 50% magnesium nitrate solution or aluminum nitrate-calcium nitrate solution (40 g of aluminum nitrate and 20 g of calcium nitrate dissolved in 100 mL of water) as an incinerating agent to wet it. Dry again, and continue incinerating. If incineration is not sufficient, repeat the above operation once and, if necessary, add 2 to 5 mL of nitric acid (1 in 2) for final incineration. After incineration, moisten the residue with water, add 2 to 4 mL of hydrochloric acid, and evaporate to dryness. Add 0.5 mol/L nitric acid, and dissolve by heating. If there are insoluble substances, filter them with filter paper, add 0.5 mol/L nitric acid to make 25 mL unless otherwise specified, and use this solution as the test solution. Separately, pipet 5.0 mL of the lead standard solution, proceed in the same manner as the test solution, and use the resulting solution as the standard solution. Perform the test with the test solution and the standard solution as directed under Atomic absorption spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 2.0 ppm).

Gas: Acetylene or hydrogen - air
Lamp: A lead hollow cathode lamp
Wavelength: 283.3 nm

(5) ***Arsenic***—Weigh 0.5 g of White Beeswax and perform the test according to Method 3 (NMT 4 ppm).

(6) ***Peroxide value***—Weigh accurately 5 g of White Beeswax and place it in a stoppered 250=mL Erlenmeyer flask. Add 35 mL of a mixture of acetic acid and chloroform (3 : 2), and shake gently to dissolve transparently. Replace the air in the flask sufficiently with clean nitrogen. Add exactly 1 mL of potassium iodine TS while passing the nitrogen. Stop the nitrogen and stopper immediately, shake to mix for 1 minute, and allow it to stand in a dark place for 5 minutes. Add 75 mL of water to this solution, stopper again, and shake vigorously to mix. Titrate with 0.01 mol/L sodium thiosulfate VS (indicator: starch TS) and calculate the peroxide value according to the following formula; the value is NMT 5. Perform a blank test in the same manner, and make any necessary correction.

$$\text{Peroxide value} = \frac{Amount\ (mL)\ of\ 0.01\ mol/L\ Sodium\ thiosulfate\ solution\ consumed}{Amount\ (g)\ of\ smaple\ taken} \times 10$$

Packaging and storage Preserve in well-closed containers.

Yellow Beeswax
황납

Beeswax
Cera Flava

Yellow Beeswax is the purified wax obtained from honeycombs such as those of *Apis indica* Radoszkowski or *Apis mellifera* Linné (*Apidae*).

Description Yellow Beeswax occurs as a pale yellow to brownish yellow mass, and has a characteristic odor, which is not rancid.
It is comparatively brittle when cooled and the fractured surface is granular and non-crystalline.

Saponification value Between 80 and 100. Weigh accurately about 3 g of Yellow Beeswax, place in a 250-mL stoppered flask and add exactly 25 mL of 0.5 mol/L potassium hydroxide-ethanol solution and 50 mL of ethanol, heat for 4 hours on a water bath under a reflux condenser, and perform the test as directed in the Saponification value under the Fats and Fatty Oils

Acid value Between 5 and 9 or between 17 and 22. Weigh accurately about 6 g of Yellow Beeswax, place in a stoppered 250-mL flask and add 50 mL of anhydrous ethanol. Warm the mixture to dissolve the wax, add 1 mL of phenolphthalein TS and perform the test as directed in the Acid value under the Fats and Fatty Oils. Perform a blank test in the same manner and make any necessary correction using solvent not previously neutralized.

Ester value Between 72 and 77

Melting point Between 60 ℃ and 67 ℃ (Method 2)

Purity (1) ***Paraffin, fat, Japan wax or resin***—Melt Yellow

Beeswax at the lowest possible temperature, drip the liquid into ethanol to form granules and allow to stand in the air for 24 hours. Drop the granules into two mixtures of ethanol and water, with a specific gravity of 0.95 and 0.97, respectively, the granules sink or are suspended in the mixture with a specific gravity of 0.95 and float or are suspended in the mixture with a specific gravity of 0.97.

(2) ***Glycerin and other polyols***—Weigh accurately 0.2 g of Yellow Beeswax, add 10 mL of a solution of potassium hydroxide in ethanol and heat on a steam bath with a reflux condenser for 30 minutes. Add 50 mL of dilute sulfuric acid, cool, filter and wash the flask. Add the washings to the filtrate, add dilute sulfuric acid to make 100 mL and use this solution as the test solution. Transfer 1.0 mL of this solution to a test tube, add 0.5 mL of sodium periodate TS (10.7 in 1000), shake to mix and allow to stand for 5 minutes. Add 1.0 mL of fuchsin solution decolorized and mix; all precipitates disappear. Place the test tube in a beaker containing water of 40 °C and cool for 10 to 15 minutes. Separately, weigh accurately glycerin RS, dissolve in dilute sulfuric acid to make a solution containing 10 mg per L, proceed with 1.0 mL of this solution in the same manner as the test solution, and use this solution as the standard solution. The color of the test solution is not more intense than that of the standard solution at the same time (NMT 0.5% as glycerin).

(3) ***Mercury***—Spread evenly about 1 g of excipient (a) into a ceramic boat and place 10 mg to 300 mg of Yellow Beeswax on top. Next, spread evenly about 0.5 g of excipient (a) and 1 g of excipient (b) successively to form layers. In the case of an automated mercury analyzer equipped with a separate catalyst in the combustion chamber, take only the sample without adding excipients to a nickel boat. Place the boat inside the combustion furnace and heat to about 900 °C with a current of air or oxygen at 0.5 to 1 L/minute. Elute the mercury and collect in a collecting tube. Transfer the mercury vapor to a cold vapor atomic absorption spectrometer by heating the collecting tube to about 700 °C and determine the absorbance as A. Separately, determine the absorbance in the same manner using only the excipients in a ceramic boat and designate as Ab. Separately, proceed with mercury standard solution in the same manner and plot a calibration curve from the absorbances thus determined. Substitute the value of A–Ab into the calibration curve to calculate the amount of mercury in the sample (NMT 1.0 ppm).

Operating conditions

Apparatus: Use an automated mercury analyzer for specimen combustion, trapping with gold amalgam and measurement using a cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Standard mercury stock solution—Dissolve 0.135 g of mercury (II) chloride in 0.001% L-cysteine to make 1000 mL. (Each mL of standard mercury stock solution = 100 μg Hg)

Mercury standard solution—Dilute standard mercury stock solution with 0.001% L-cysteine so that each mL contains 0 to 200 ng.

Excipients—Use (a) aluminum oxide and (b) a mixture of calcium hydroxide and sodium carbonate (1 : 1) and activate at 950 °C for 30 minutes before use.

(4) ***Lead***—Weigh accurately 5.0 g of Yellow Beeswax and transfer to a platinum crucible. Dry, carbonize and incinerate at 450 °C to 550 °C. If incineration is not complete, cool and moisten with 2 to 5 mL of nitric acid (1 in 2), 50% magnesium nitrate solution or aluminum nitrate-calcium nitrate solution (prepared by dissolving 40 g of aluminum nitrate and 20 g of calcium nitrate in 100 mL of water) as an incineration supplement, dry, and continue with incineration. If incineration is incomplete, repeat the above procedure once and then add a final 2 mL to 5 mL of nitric acid (1 in 2), if necessary, and incinerate. After incineration, moisten the residue with water, add 2 to 4 mL of hydrochloric acid, and evaporate to dryness. Add 0.5 mol/L nitric acid, dissolve by warming, and filter with filter paper if any insoluble matter exists. Unless otherwise specified, add 0.5 mol/L nitric acid to make 25 mL and use this solution as the test solution. Separately, take 5.0 mL of the lead standard solution to a platinum crucible, proceed in the same manner as the test solution, and use the resulting solution as the standard solution. Perform the test with the test solution and the standard solutions as directed under the Atomic Absorption Spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 2.0 ppm).

Gas: Acetylene or hydrogen-air
Lamp: Lead hollow-cathode lamp
Wavelength: 283.3 nm

(5) ***Arsenic***—Proceed with 0.5 g of Yellow Beeswax according to Method 3 and perform the test (NMT 4 ppm).

(6) ***Peroxide value***—Weigh accurately 5 g of Yellow Beeswax, transfer to a 250-mL stoppered Erlenmeyer flask, add 35 mL of a mixture of acetic acid and chloroform (3 : 2) and shake gently to dissolve until clear. Sufficiently displace the air inside the flask by passing a current of clean nitrogen. While passing a current of nitrogen, add exactly 1 mL of potassium iodide TS, stop the nitrogen, stopper immediately, shake for 1 minute and allow to stand for 5 minutes in a dark place. To this solution, add 75 mL of water, stopper, and shake vigorously. Titrate with 0.01 mol/L sodium thiosulfate VS (indicator: starch TS) and calculate the peroxide value by the following equation; the value is NMT 5. Separately, perform a blank test and make any necessary correction.

$$\text{Peroxide value} = \frac{Volume\ (mL)\ of\ 0.01\ mol/L\ sodium\ thiosulfate\ VS\ consumed}{\text{Amount (g) of the sample taken}} \times 10$$

Packaging and storage Preserve in well-closed containers.

Bentonite
벤토나이트

Bentonite is a natural, colloidal, hydrous aluminum silicate.

Description Bentonite occurs as a white to pale yellowish brown, fine powder with no odor and a slightly earthy taste.
It is practically insoluble in water, ethanol or ether.
It swells when added to water.

Identification (1) Add 3 mL of diluted sulfuric acid (1 in 3) to 0.5 g of Bentonite, heat the mixture until white smoke appears, and cool. Add 20 mL of water, filter, and then add 3 mL of ammonia TS to 5 mL of the filtrate; a white, gel-like precipitate is formed. Add 5 drops of alizarin S TS; it turns to a red color.

(2) Wash the residue in (1) with water, add 2 mL of methylene blue solution (1 in 10000), and then wash with water; the residue turns blue.

pH Add 50 mL of water to 1.0 g of Bentonite, and shake to suspend; the pH of the resulting solution is 9.0 to 10.5.

Purity (1) ***Heavy metals***—Add 80 mL of water and 5 mL of hydrochloric acid to 1.5 g of Bentonite, shake well for 20 minutes, heat gently, and cool. Then, centrifuge and collect the clear supernatant, and wash the precipitate twice with 10 mL each of water. After centrifugation each time, combine the clear supernatant and the washings, and add 1 drop each of strong ammonia water. When a slight precipitate is formed, shake vigorously, and dissolve in 1 drop each of diluted hydrochloric acid again. Add 0.45 g of hydroxylamine hydrochloride to this solution, heat, and cool. Then, add 0.45 g of sodium acetate, 6 mL of dilute acetic acid, and water to make 150 mL. Take 50 mL of this solution, and use this solution as the test solution. Prepare the control solution by adding 0.15 g of hydroxylamine hydrochloride, 0.15 g of sodium acetate, 2 mL of dilute acetic acid, and water to 2.5 mL of the lead standard solution to make 50 mL (NMT 50 ppm).

(2) ***Arsenic***—Add 5 mL of dilute hydrochloric acid to 1.0 g of Bentonite, shake well to mix, heat gently until boiling. and centrifuge after cooling quickly. Add 5 mL of dilute hydrochloric acid to the residue, shake well to mix, and centrifuge. Add 10 mL of water again to perform the same procedure, combine all the extracts, and heat to concentrate on a water bath to make 5 mL. Use this solution as the test solution and perform the test (NMT 2 ppm).

(3) ***Foreign matter*—**Place 2.0 g of Bentonite to a mortar, add 20 mL of water to swell it, and suspend evenyl with a pestle, and then add water to make 100 mL. Pass the dispersion through a No. 200 sieve, wash it with water, and rub with a finger over the eyes of the sieve; there is no sand-like substance.

Loss on drying Between 5.0% and 10.0% (2 g, 105 °C, 2 hours).

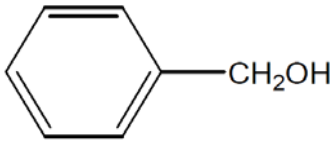
Gel-forming power Mix 6.0 g of Bentonite with 0.30 g of magnesium oxide, add it in several portions to a 500-mL stoppered cylinder containing 200 mL of water, and shake for 1 hour. Transfer 100 mL of the resulting suspension to a 100 mL measuring cylinder, and allow it to stand for 24 hours; the clear liquid separated in the upper layer is NMT 2 mL.

Swelling power Weigh 2.0 g of Bentonite, and add it in 10 portions to a 100 mL measuring cylinder containing 100 mL of water. However, add the sample after the first sample has almost settled. Allow the mixture to stand for 24 hours; the volume of the lump at the bottom of the container is NLT the 20 mL scale.

Microbial limit Perform the microbial limit test; the total aerobic microbial count is NMT 1000 CFU and the total combined yeasts/molds count is NMT 100 CFU per 1 g of Bentonite. Also, *Escherichia coli (E.coli)*, *Salmonella*, *Pseudomonas aeruginosa* and *Staphylococcus aureus* are not detected.

Packaging and storage Preserve in well-closed containers.

Benzyl Alcohol
벤질알코올



C_7H_8O: 108.14

Benzyl alcohol [*100-51-6*]

Benzyl Alcohol contains NLT 98.0% and NMT 100.5% of benzyl alcohol (C_7H_8O).

Indicate which of Benzyl Alcohol is used for injections.

Description Benzyl Alcohol occurs as a colorless, clear, oily liquid. It is miscible with ethanol (95), oils or essential oils.
It is soluble in water.

Specific gravity—d_{20}^{20}: Between 1.043 and 1.049.

Identification Determine the infrared spectra of Benzyl Alcohol and benzyl alcohol RS as directed in the liquid film method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

Refractive index n_D^{20}: Between 1.538 and 1.541.

Purity (1) ***Clarity and color of solution***—Dissolve 2.0 mL of Benzyl Alcohol in 60 mL of water; the resulting solution is colorless and clear.

(2) ***Acidity***—Add 10 mL of ethanol (95) and 2 drops of phenolphthalein TS to 10 mL of Benzyl Alcohol. Add 0.1 mol/L sodium hydroxide solution until the color of the resulting solution turns to a pale red color; the amount is NMT 1.0 mL.

(3) ***Benzaldehyde and other related substances***—Use Benzyl Alcohol as the test solution. Separately, weigh accurately 0.750 g of benzaldehyde and 0.500 g of cyclohexylmethanol, and add benzyl alcohol to make exactly 25 mL. Pipet 1 mL of this solution, add exactly 2 mL of the internal standard solution of ethylbenzene and 3 mL of the internal standard solution of dicyclohexyl, add Benzyl Alcohol to make exactly 20 mL, and use this solution as the standard solution (1). Perform the test with 0.1 μL each of the test solution and the standard solution (1) as directed under the Gas Chromatography according to the following conditions. If a peak in the chromatogram obtained from the test solution appears at the same retention time as ethylbenzene and dicyclohexyl, use the corrected peak area by subtracting the corresponding peak areas from the chromatogram of the standard solution (1) or the standard solution (2). Use all peaks obtained from the chromatogram of the test solution to calculate the sum of peak areas. The peak area of benzaldehyde in the test solution is not greater than the difference between the peak areas of benzaldehyde in the standard solution (1) and the test solution (0.15%). The peak area of cyclohexylmethanol in the test solution is not greater than the difference between the peak areas of cyclohexylmethanol in the standard solution (1) and the test solution (0.10%). The sum of the peak areas excluding those of benzaldehyde and cyclohexylmethanol with a shorter retention time than benzyl alcohol in the test solution is not greater than 4 times the peak area of ethylbenzene in the standard solution (1) (0.04%). The sum of the peak areas with a retention time shorter than that of benzyl alcohol in the test solution is not greater than

the peak area of dicyclohexyl in the standard solution (1) (0.3%). However, the peaks NMT 1/100 of the peak area of ethylbenzene in the standard solution (1) are not calculated (0.0001%).

The procedure and limit values for those marked "Used for injection" are as follows.

Use Benzyl Alcohol as the test solution. Separately, weigh accurately 0.250 g of benzaldehyde and 0.500 g of cyclohexylmethanol, and add Benzyl Alcohol to make exactly 25 mL. Pipet 1 mL of this solution, add exactly 2 mL of the internal standard solution of ethylbenzene and 2 mL of the internal standard solution of dicyclohexyl, add Benzyl Alcohol to make exactly 20 mL, and use this solution as the standard solution (2). Perform the test with 0.1 μL each of the test solution and the standard solution (2) as directed under the Gas Chromatography according to the following conditions. If a peak in the chromatogram obtained from the test solution appears at the same retention time as ethylbenzene and dicyclohexyl, use the corrected peak area by subtracting the corresponding peak areas from the chromatogram of the standard solution (1) or the standard solution (2). Use all peaks obtained from the chromatogram of the test solution to calculate the sum of peak areas. The peak area of benzaldehyde in the test solution is not greater (0.15%) than the difference between the peak areas of benzaldehyde in the standard solution (2) and the test solution (0.05%). The peak area of cyclohexylmethanol in the test solution is not greater than the difference between the peak areas of cyclohexylmethanol in the standard solution (2) and the test solution (0.10%). The sum of the peak areas excluding those of benzaldehyde and cyclohexylmethanol with a shorter retention time than benzyl alcohol in the test solution is not greater than 2 times the peak area of ethylbenzene in the standard solution (2) (0.02%). The sum of the peak areas with a retention time shorter than that of benzyl alcohol in the test solution is not larger than the peak area of dicyclohexyl in the standard solution (2) (0.2%). However, the peaks NMT 1/100 of the peak area of ethylbenzene in the standard solution (2) are not calculated (0.0001%).

Internal standard solution of ethylbenzene—Weigh accurately 0.100 g of ethylbenzene, and dissolve in Benzyl Alcohol to make exactly 10 mL. Pipet 2 mL of this solution, and add Benzyl Alcohol to make exactly 20 mL.

Internal standard solution of dicyclohexyl—Weigh accurately 2.000 g of dicyclohexyl, and dissolve in Benzyl Alcohol to make exactly 10 mL. Pipet 2 mL of this solution, and add Benzyl Alcohol to make exactly 20 mL.

Operating conditions

Detector: A flame ionization detector

Column: A fused silica column about 0.32 mm in internal diameter and about 30 m in length, coated inside with polyethylene glycol 20M for gas chromatography, 0.5 μm in thickness.

Column temperature: Raise the temperature from 50 to 220 °C at 5 °C per minute, and maintain at 220 °C for 35 minutes.

Sample injection port temperature: A constant temperature of about 200 °C.

Detector temperature: A constant temperature of about 310 °C.

Carrier gas: Helium

Flow rate: 25 cm/sec (50 °C)

Split ratio: No Split

System suitability

System performance: Perform the test according to the above conditions with the standard solution (1); the retention time of benzyl alcohol is about 26 minutes, and the relative retention time for benzyl alcohol is about 0.28 for ethylbenzene, about 0.59 for dicyclohexyl, about 0.68 for benzaldehyde, and about 0.71 for cyclohexylmethanol. The resolution between the peaks of benzaldehyde and cyclohexylmethanol is NLT 3.0. However, use the standard solution (2) for those marked "Used for injection".

(4) ***Peroxide value***—Weigh accurately 5 g of Benzyl Alcohol to place in a stoppered Erlenmeyer flask, and dissolve in 30 mL of a mixture of acetic acid (100) and chloroform (3 : 2). Add 0.5 mL of saturated potassium iodide solution to this solution, shake to mix for exactly 1 minute, and then add 30 mL of water. Titrate this solution with 0.01 mol/L sodium thiosulfate VS. The endpoint of the titration is when the color of the solution turns to a pale yellow and the blue color created by adding 10 mL of starch solution disappears. Perform a blank test in the same manner and determine the peroxide value according to the following formula; the value is NMT 5.

$$\text{Peroxide value (mEq/kg)} = \frac{10 \times (V_I - V_O)}{W}$$

V_I: Amount (mL) of 0.01 mol/L sodium thiosulfate VS consumed for the titration of the test solution

V_O: Amount (mL) of 0.01 mol/L sodium thiosulfate VS consumed for the titration of the blank test solution

W: Amount (g) of Benzyl Alcohol taken

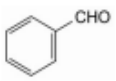
(5) ***Residue on evaporation***—Perform the test after confirming its suitability for the peroxide value test. Place 10.0 g of Benzyl Alcohol in a porcelain or quartz crucible or a platinum dish, and evaporate to dryness on a hot plate, being careful not to boil over 200°C. Dry the residue on a hot plate for 1 hour, and cool it in a desiccator; the resulting amount of the residue is NMT 5 mg.

Assay Weigh accurately about 0.9 g of Benzyl Alcohol, add 15 mL of a mixture of anhydrous pyridine and acetic anhydride (7 : 1), and heat the mixture on a water bath for 30 minutes with a reflux condenser. After cooling, add 25 mL of water, and titrate the excess acetic acid with 1 mol/L sodium hydroxide VS (indicator: 2 drops of phenolphthalein TS). Perform a blank test in the same manner.

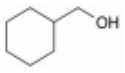
Each mL of 1 mol/L sodium hydroxide VS
= 108.14 mg of C_7H_8O

Packaging and storage Preserve in light-resistant, tight containers.

Note

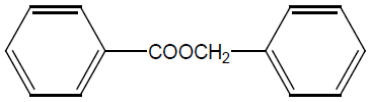


A. Benzaldehyde



B. Cyclohexylmethanol

Benzyl Benzoate
벤조산벤질



$C_{14}H_{12}O_2$: 212.24

Benzyl benzoate [*120-51-4*]

Benzyl Benzoate contains NLT 99.0% and NMT 101.0% of benzyl benzoate ($C_{14}H_{12}O_2$).

Description Benzyl Benzoate occurs as a colorless, clear, viscous liquid with a slightly aromatic, irritating, and burning taste.
It is miscible with ethanol or ether.
It is practically insoluble in water.
Congealing temperature: About 17 °C.

Specific gravity—d_{20}^{20}: About 1.123.

Boiling point: About 323 °C.

Identification (1) Add 5 mL of sodium carbonate TS and 2 mL of potassium permanganate TS to 1 mL of Benzyl Benzoate, and heat over low heat; the mixture smells like benzaldehyde.

(2) In the Assay, heat the titrated solution on a water bath to evaporate the ethanol, and add 0.5 mL of ferric chloride TS; a pale yellowish red precipitate is formed, and this precipitate turns white when adding dilute hydrochloric acid.

Refractive index n_D^{20}: Between 1.568 and 1.570.

Purity (1) ***Acidity***—Dissolve 5.0 mL of Benzyl Benzoate in 25 mL of neutralized ethanol, and add 0.50 mL of 0.1 mol/L sodium hydroxide solution; the resulting solution exhibits a red color.

(2) ***Aldehyde***—Weigh accurately 10.0 g of Benzyl Benzoate into a 125-mL Erlenmeyer flask, add 50 mL of alcohol and 5 mL of hydroxylamine hydrochloride TS (3.5 in 100), mix, and allow to stand for 10 minutes. Add 1 mL of bromophenol blue TS, and titrate with 0.1 mol/L sodium hydroxide VS until the color of the resulting solution turns to a bright green color. Perform a blank test in the same manner and make any necessary correction. The consumption of 0.1 mol/L sodium hydroxide VS is NMT 0.50 mL (NMT 0.05% as benzaldehyde).

Residue on ignition NMT 0.05% (2 g).

Assay Weigh accurately about 2 g of Benzyl Benzoate, add 50 mL of 0.5 mol/L potassium hydroxide-ethanol solution, boil over low heat for 1 hour using a reflux condenser equipped with a carbon dioxide absorption tube (soda lime), and cool. Titrate excess potassium hydroxide with 0.5 mol/L hydrochloric acid VS (indicator: 2 drops of phenolphthalein TS). Perform a blank test in the same manner.

Each mL of 0.5 mol/L potassium hydroxide-ethanol VS
= 106.12 mg of $C_{14}H_{12}O_2$

Packaging and storage Preserve in light-resistant, tight containers.

Black Iron Oxide
흑색산화철

Black Iron Oxide contains NLT 67.1% of Iron(Fe: 55.85).

Description Black Iron Oxide occurs as a black powder and is odorless. It is slightly soluble in water.
It is slightly soluble in organic solvents and soluble in strong acids.

Identification Weigh 1.0 g of Black Iron Oxide, add 5 mL of hydrochloric acid, warm to dissolve, and add water to make 100 mL. To this solution, add 5 drops of nitric acid, boil, and cool. Filter, if necessary, then take 5 mL of this solution, and add 5 mL of 10% potassium ferrocyanide TS; a blue precipitate is formed.

Purity (1) ***Lead***—Prepare the test with 1.0 g of Black Iron Oxide as directed in the section of Ion Oxide under NF and perform the test (NMT 10 ppm).

(2) ***Mercury***—Prepare the test with 1.0 g of Black Iron Oxide as directed in the section of Ion Oxide under NF and perform the test (NMT 3 ppm).

(3) ***Arsenic***—Proceed with 0.67 g of Black Iron Oxide according to Method 3 and perform the test (NMT 3 ppm).

Loss on ignition NMT 2.0% (1.0 g, 800 ℃, 2 hours).

Assay Weigh accurately about 0.125 g of Black Iron Oxide, put it in a 400-mL beaker, add 25 mL of hydrochloric acid and 5 mL of water, and warm gently until it dissolves completely. Boil this solution to concentrate until it becomes about 10 to 20 mL, add 0.5 mol/L tin chloride TS until the color of the solution changes from yellow to clear green, and then add 1 to 2 drops of mol/L tin chloride TS more. After cooling, add 10 mL of hydrochloric acid. Rinse the wall of the beaker with water and add water to make 250.0 mL. To this solution, add 5 mL of saturated mercuric chloride TS, allow to stand for 1 to 2 minutes, then, add 5 drops of *o*-phenanthroline TS as an indicator, and titrate with 0.1 mol/L cerium sulfate VS until the color of the solution changes from yellowish red to clear green. Perform a blank test in the same way and make any necessary correction.

Each mL of 0.1 mol/L cerium sulfate VS
= 5.587 mg of Fe

Packaging and storage Preserve in tight containers.

Cacao Butter
카카오지

Cacao Butter is a fat obtained from the seeds of the *Theobroma cacao* Linné (*Sterculiaceae*).

Description Cacao Butter occurs as a pale yellow, hard but brittle mass, has a slightly chocolate-like odor, and has no order of rancidity.
It is freely soluble in ether or petroleum ether, soluble in boiling hot anhydrous ethanol, and very slightly soluble in ethanol.

Congealing point of fatty acids—Between 45 ℃ and 50 ℃

Melting point—Between 31 ℃ and 35 ℃ (Fill the sample,

not melted, into the capillary tube and then follow the Method 2)

Saponification value Between 188 and 195

Specific gravity d_{20}^{20}: Between 0.895 and 0.904

Acid value NMT 3.0

Iodine value Between 35 and 43

Packaging and storage Preserve in well-closed containers.

Calcium Hydroxide
수산화칼슘

Slaked Lime $Ca(OH)_2$: 74.09

Calcium Hydroxide contains NLT 90.0% and NMT 101.0% of calcium hydroxide [$Ca(OH)_2$].

Description Calcium Hydroxide occurs as a white powder and has a slightly bitter taste.
It is slightly soluble in water, very slightly soluble in boiling water, and practically insoluble in ethanol or ether.
It is soluble in dilute acetic acid, dilute hydrochloric acid and dilute nitric acid.
It absorbs carbon dioxide in the air.

Identification (1) Add water as much as 3 to 4 times the amount of Calcium Hydroxide; the mixture becomes a slush and is alkaline.

(2) Dissolve 1 g of Calcium Hydroxide in 30 mL of dilute acetic acid, boil, cool, and neutralize with ammonia TS; the solution responds to the Chemical identification reaction (2) and (3) for calcium salt.

Purity (1) ***Acid-insoluble substances***—To 5 g of Calcium Hydroxide, add 100 mL of water, add hydrochloric acid dropwise with stirring until the solution turns acidic, and then add another 1 mL of hydrochloric acid. Boil this solution for 5 minutes, cool, and filter through a glass filter previously tared. Wash the residue with hot water until the washing exhibits no turbidity upon the addition of silver nitrate TS. Dry the residue at 105 °C to a constant mass; the amount of the residue is NMT 25 mg.

(2) ***Fluoride***—Weigh 1 g of Calcium Hydroxide, transfer it to a beaker, and dissolve in 10 mL of hydrochloric acid (1 in 10). Heat this solution to boil for 1 minute, transfer the mixture to a polyethylene beaker, and cool immediately. Add 15 mL of sodium citrate solution (1 in 4) and 10 mL of ethylenediaminetetraacetic acid disodium salt solution (1 in 40) and shake to mix. Adjust the pH to between 5.4 and 5.6 with hydrochloric acid (1 in 10) or sodium hydroxide (2 in 5), add water to make 100 mL, and use this solution as the test solution. Transfer 50 mL of the test solution to a polyethylene beaker, measure the potential using a fluoride electrode, and determine the amount of fluoride from the calibration curve; the amount of fluoride is NMT 50 ppm.

Standard solution—Weigh accurately 2.210 g of sodium fluoride, previously dried at 200 °C for 4 hours, transfer it to a polyethylene beaker, dissolve in 200 mL of water, and add water to make 1000 mL. Store the solution in a polyethylene container. Pipet 5 mL of this solution, transfer it to a volumetric flask, and add water to make 1000 mL (the resulting solution contains 5 μg of fluoride per mL).

Creating a calibration curve—Take 1 mL, 2 mL, 3 mL, 5 mL, 10 mL and 15 mL of the standard solution, transfer each to a polyethylene beaker, add 15 mL of sodium citrate solution (1 in 4) and 10 mL of ethylenediaminetetraacetic acid disodium salt solution (1 in 40) and mix. Adjust the pH to between 5.4 and 5.6 with hydrochloric acid (1 in 10) or sodium hydroxide (2 in 5) and add water to make 100 mL each. Take each 50 mL of these solutions and transfer to separate polyethylene containers. Measure the potential using a fluoride electrode and create a calibration curve with the log values of the fluoride concentrations.

(3) ***Heavy metals***—Dissolve 2.0 g of Calcium Hydroxide in 10 mL of dilute hydrochloric acid, evaporate to dryness on a water bath, dissolve the residue in 40 mL of water, and filter. To 20 mL of the filtrate, add 2 mL of dilute nitric acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution by evaporating 5 mL of dilute hydrochloric acid to dryness on a water bath, adding 2.0 mL of the lead standard solution and 2 mL of dilute acetic acid, and adding water to make 50 mL (NMT 20 ppm).

(4) ***Lead***—Weigh accurately 5.0 g of Calcium Hydroxide, transfer it to a 150-mL beaker, add 30 mL of water, and add hydrochloric acid in small portions until the sample is completely dissolved. Add another 1 mL of hydrochloric acid. Boil for about 5 minutes, cool, add water to make about 100 mL, and adjust the pH to between 2 and 4 with sodium hydroxide (1 in 4) or hydrochloric acid (1 in 4). Transfer 250 mL of this solution to a separatory funnel, add water to make about 200 mL, add 2 mL of 2% ammonium pyrrolidine dithiocarbamate (2 in 100), and shake to mix. Extract twice with each 20 mL of chloroform, evaporate the extract to dryness on a water bath, add 3 mL of nitric acid to the residue, and heat until almost dried. Add 0.5 mL of nitric acid and 10 mL of water, concentrate to a final volume of 3 to 5 mL, add water to make 10 mL, and use this solution as the test solution. Separately, pipet 5.0 mL of the lead standard solution, proceed in the same manner as the test solution, and use the resulting solution as the standard solution. Perform the test with the test solution and the standard solutions as directed under the Atomic absorption spectroscopy according to the following conditions; the absorbance of the test solution is not greater than that of the standard solution (NMT 2.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow-cathode lamp
Wavelength: 283.3 nm

(5) ***Barium***—Dissolve 1.5 g of Calcium Hydroxide in 15 mL of dilute hydrochloric acid, add water to make 30 mL, and filter. To 20 mL of the filtrate, add 2 g of sodium acetate, 1 mL of dilute acetic acid and 0.5 mL of potassium chromate TS, and allow the mixture to stand for 15 minutes; the turbidity of this solution is NMT that of the solution prepared by adding water to 0.3 mL of the barium standard solution to make 20 mL and proceeding in the same manner as above (NMT 0.03%).

(6) ***Magnesium and alkali metals***—To 1.0 g of Calcium Hydroxide, add 20 mL of water and 10 mL of dilute hydrochloric acid, and boil the mixture. Add ammonia TS to neutralize, and add ammonium oxalate TS dropwise until the precipitation of calcium oxalate is completed. Heat the mixture on a water bath for 1 hour, cool, add water to make 100 mL, shake well to mix, and filter. To 50 mL of the filtrate, add 0.5 mL of sulfuric acid, evaporate to dryness, and ignite at 600 °C to constant mass; the amount of the residue is NMT 24 mg.

(7) ***Arsenic***—Dissolve 0.5 g of Calcium Hydroxide in 5 mL

of dilute hydrochloric acid. Use this solution as the test solution and perform the test (NMT 4 ppm).

(8) ***Carbonate***—To 2.0 g of Calcium Hydroxide, add 50 mL of water, shake to mix, and add an excess amount of 3 mol/L hydrochloric acid TS; no foam is produced.

Assay Weigh accurately about 1.0 g of Calcium Hydroxide, dissolve in 10 mL of dilute hydrochloric acid, and add water to make exactly 100 mL. Pipet 10.0 mL of this solution, add 90 mL of water and 1.5 mL of 8 mol/L potassium hydroxide TS, shake well to mix, and allow the mixture to stand for 3 to 5 minutes. Add 0.1 g of NN indicator and immediately titrate with 0.05 mol/L ethylenediaminetetraacetic acid disodium salt VS. The endpoint of the titration is when the purple color of the solution turns blue.

Each mL of 0.05 mol/L ethylenediaminetetraacetic acid disodium salt VS
= 3.7046 mg of $Ca(OH)_2$

Packaging and storage Preserve in tight containers.

Calcium Oxide
산화칼슘

CaO : 56.08

Calcium Oxide, when ignited, contains NLT 98.0% and NMT 101.0% of calcium oxide (CaO).

Description Calcium Oxide occurs as a white, solid mass containing a powder with no odor.
It is very slightly soluble in boiling water and practically insoluble in ethanol.
1 g of Calcium Oxide is almost soluble in 2500 mL of water.
It slowly absorbs moisture and carbon dioxide from the air.

Identification (1) Calcium Oxide generates heat, and turns into a white powder when wetted with water; it becomes alkaline when mixed with about 5 times the amount of water.

(2) Mix 1 g of Calcium Oxide with 20 mL of water, and add 1 drop each of acetic acid; the resulting solution responds to the Chemical identification reactions for calcium salt.

Purity (1) ***Acid-insoluble substances***—Add a small amount of water to 5.0 g of Calcium Oxide to disintegrate, add 100 mL of water, and stir to mix. Add 1 drop each of hydrochloric acid until the liquid appears acidic, and then add 1 mL of hydrochloric acid again. Boil this solution for 5 minutes, cool, and filter with a glass filter. Wash the residue with boiling water until it remains clear even after adding silver nitrate solution. Dry at 105°C to a constant mass; the resulting amount is NMT 10.0 mg.

(2) ***Carbonate***—Add a small amount of water to 1.0 g of Calcium Oxide to disintegrate, add 50 mL of water to mix well, and allow to stand for a while. Tilt and discard most of the upper emulsion, and add excess diluted hydrochloric acid to the residue; the resulting solution does not produce excessive foam.

(3) ***Magnesium or alkaline metals***—Mix 1.0 g of Calcium Oxide with 75 mL of water, dissolve in 1 drop each of hydrochloric acid, and add 1 mL of hydrochloric acid more. Boil for 1 to 2 minutes, neutralize with ammonia TS, add 1 drop each of excess hot ammonium oxalate TS, and heat on a water bath for 2 hours. After cooling, add water to make 200 mL, mix well and filter. Take 50 mL of the filtrate, add 0.5 mL of sulfuric acid, evaporate to dryness, and ignite the residue at 600 ℃ to a constant mass; the resulting amount is NMT 15 mg.

Loss on ignition NMT 10.0% (1 g, 900 ℃, constant mass).

Assay Ignite Calcium Oxide at 900 ℃ to a constant mass, cool in a desiccator (silica gel), and weigh accurately about 0.7 g. Add 50 mL of water and 8 mL of diluted hydrochloric acid (1 in 3), heat to dissolve, cool, and then add water to make exactly 250 mL. Pipet 10 mL of this solution, add 50 mL of water and 2 mL of 8 mol/L potassium hydroxide TS, add 0.1 g of NN indicator again, and titrate with 0.02 mol/L disodium ethylenediaminetetraacetate VS. However, the endpoint of titration is when the purple color of the solution turns to the blue color.

Each mL of 0.02 mol/L Ethylenediaminetetraacetic acid disodium salt VS
= 1.1215 mg of CaO

Packaging and storage Preserve in tight containers.

Calcium Stearate
스테아르산칼슘

Calcium Stearate mainly consists of calcium salts of stearic acid ($C_{18}H_{36}O_2$: 284.48) and palmitic acid ($C_{16}H_{36}O_2$: 256.42).

Calcium Stearate, when dried, contains NLT 6.4% and NMT 7.1% of calcium (Ca: 40.08).

Description Calcium Stearate occurs as a white, light and bulky powder with a smooth texture and adheres easily to the skin. It has no odor or a slightly peculiar odor.
It is practically insoluble in water, ethanol or ether.

Identification (1) Add 20 mL of diluted hydrochloric acid (1 in 2) and 30 mL of ether to 3 g of Calcium Stearate, shake vigorously to mix for 3 minutes, and allow the mixture to stand. The separated aqueous layer responds to the Chemical identification reactions (1), (2) and (4) for calcium salt.

(2) Wash the ether layer obtained in (1) with 20 mL and 10 mL of dilute hydrochloric acid and 20 mL of water successively and evaporate the ether on a water bath; the residue melts at a temperature above 54 ℃ (Method 2).

Purity (1) ***Heavy metals*—**Weigh 1.0 g of Calcium Stearate, heat it gently and carefully at first, and ignite it to incinerate. After cooling, add 2 mL of hydrochloric acid, evaporate on a water bath to dryness, warm the residue with 20 mL of water and 2 mL of dilute acetic acid for 2 minutes, cool, filter and wash the residue with 15 mL of water. Combine the filtrate and the washings and add water again to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution by evaporating 2 mL of hydrochloric acid on a water bath to dryness and adding 2 mL of dilute acetic acid, 2.0 mL of lead standard solution and then water to make 50 mL (NMT 20 ppm).

(2) **Arsenic—**Add 5 mL of diluted hydrochloric acid (1 in 2) and 20 mL of chloroform to 1.0 g of Calcium Stearate, shake vigorously for 3 minutes to mix, allow to stand, and take separately the water layer. Perform the test using the water layer as the test solution (NMT 2 ppm).

Loss on drying NMT 4.0% (1 g, 105 ℃, 3 hours).

Microbial limit The total aerobic microbial count is NMT 1000 CFU, and the total combined yeasts/moulds count is NMT 100 CFU per 1 g of Calcium Stearate. Also, *Escherichia coli (E.coli), Salmonella, Pseudomonas aeruginosa* and *Staphylococcus aureus* are not detected.

Assay Weigh accurately about 0.5 g of Calcium Stearate, previously dried, heat gently and carefully at first, and then ignite to incinerate. After cooling, add 10 mL of dilute hydrochloric acid to the residue, warm for 10 minutes on a water bath, and transfer the contents to a flask with the aid of 10 mL, 10 mL and 5 mL volumes of hot water. Add sodium hydroxide TS until the solution becomes slightly turbid and then add 25 mL of 0.05 mol/L disodium ethylenediamine tetraacetate solution, 10 mL of ammonia-ammonium chloride buffer solution, pH 10.7, 4 drops of eriochrome black T TS and 5 drops of methyl yellow TS, and titrate the excess ethylenediaminetetraacetic acid disodium salt with 0.05 mol/L magnesium chloride VS. Then endpoint of titration is when the green color of the solution disappears and a red color develops. Perform a blank test in the same manner.

Each mL of 0.05 mol/L ethylenediaminetetraacetic acid disodium salt VS
= 2.0039 mg of Ca

Packaging and storage Preserve in well-closed containers.

Camellia Oil
동백유

Camellia Oil is the fatty oil obtained from the peeled seeds of *Camellia japonica* Linné or other allied plants in the same genus (*Theaceae*).

Description Camellia Oil occurs as a colorless to pale yellow clear oil. It is almost odorless and tasteless.
It is miscible with ether or petroleum ether.
It is slightly soluble in ethanol.
It partially congeals at -10 ℃ and completely congeals at -15 ℃.

Specific gravity d_{25}^{25}: Between 0.910 and 0.914.

Identification To 2 mL of Camellia Oil, gently add 10 mL of a mixture of fuming nitric acid, sulfuric acid, and water (1 : 1 : 1), previously cooled to room temperature; a bluish green color develops at the zone of contact.

Saponification value Between 188 and 194.

Unsaponifiable matter NMT 1.0%

Acid value NMT 2.8.

Iodine value 78 to 83.

Packaging and storage Preserve in tight containers.

Capsules
캡슐

Capsules are made by using an appropriate capsule formation listed in the Pharmacopoeia, such as gelatin, and are a pair of cylindrical bodies that are closed at one end and can be inserted into each other.

Method of preparation To prepare Capsules, add water to an appropriate capsule formation listed in the Pharmacopoeia, such as Gelatin, and dissolve by warming, add Glycerin, D-Sorbitol, emulsifying agents, dispersing agents, preservatives, coloring agents, etc., if necessary, to form a thick, glue-like liquid, and mold into capsules while it's hot.
Capsules may be coated with a lubricant, if necessary.

Description Capsules occur as odorless and elastic.

Purity ***Odor, clarity and color of solution, and acidity or alkalinity***—Place one piece (pair) of Capsules (without putting the pair together) in a 100-mL Erlenmeyer flask, add 50 mL of water, and shake occasionally while maintaining the temperature at 37 ± 2 ℃. Repeat the test 5 times; they all dissolve within 10 minutes. Also, these solutions are odorless, and are neutral or slightly acidic.

Packaging and storage Preserve in well-closed containers.

Carboxymethylcellulose
카르복시메틸셀룰로오스

CMC
[*9000-11-7*]

Carmellose is partially o-carboxymethylated carboxymethyl ether of cellulose.

Description Carmellose occurs as a white or almost white powder.
It is practically insoluble in ethanol (99.5).
Add water to Carmellose; it swells to form a suspension.
Add sodium hydroxide TS to Carmellose; it becomes a viscous liquid.
It is hygroscopic.

Identification (1) Determine the infrared spectra of Carmellose and carmellose RS as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

(2) Suspend 1 g of Carmellose in 100 mL of freshly boiled and cooled water; the pH of the solution is between 3.5 and 5.0.

Purity (1) ***Chloride***—To 0.8 g of Carmellose, add 50 mL of water, shake well to mix, dissolve in 10 mL of sodium hydroxide TS, and add water to make 100 mL. To 20 mL of this solution, add 10 mL of dilute nitric acid, heat on a water bath until a flocculent precipitate forms, cool, and centrifuge. Take the clear supernatant, wash the precipitate 3 times with 10 mL each of water, and centrifuge each time. Combine the clear supernatant and the washings, and add water to make 100 mL. Take 25 mL of this solution, and add 6 mL of dilute nitric acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution with 0.40 mL of 0.01 mol/L hydrochloric acid (NMT 0.36%).

(2) ***Sulfate***—To 0.40 g of Carmellose, add 25 mL of water, shake well to mix, dissolve in 5 mL of sodium hydroxide TS, and add 20 mL of water. Add 2.5 mL of hydrochloric acid to this solution, heat on a water bath until a flocculent precipitate forms, cool, and centrifuge. Take the clear supernatant, wash the precipitate 3 times with 10 mL each of water, centrifuge each time, combine the clear supernatant and the washings, and add water to make 100 mL. Filter this solution, discard the first 5 mL of the filtrate, take 25 mL of the subsequent filtrate, and add 1 mL of dilute hydrochloric acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution with 1.5 mL of 0.005 mol/L sulfuric acid (NMT 0.72%).

Loss on drying NMT 8.0% (1 g, 105 ℃, 4 hours).

Residue on ignition NMT 1.5% (after drying, 1 g).

Packaging and storage Preserve in tight containers.

Carboxymethylcellulose Calcium
카르복시메틸셀룰로오스칼슘

Carmellose Calcium
CMC Calcium
[9050-04-8]

Carboxymethylcellulose Calcium is the calcium salt of polycarboxymethylether of cellulose.

Description Carboxymethylcellulose Calcium occurs as a white to yellowish white powder.
It is practically insoluble in ethanol (95), ether, acetone or chloroform.
Add water to Carboxymethylcellulose Calcium; it swells to form a suspension.
Add 100 mL of water to 1 g of Carboxymethylcellulose Calcium, and shake to mix; the pH of the resulting suspension is between 4.5 and 6.0.
It is hygroscopic.

Identification (1) To 0.1 g of Carboxymethylcellulose Calcium, add 10 mL of water, shake well to mix, add 2 mL of sodium hydroxide TS, shake to mix, and allow the mixture to stand for 10 minutes, and use the resulting solution as the test solution. To 1 mL of the test solution, add water to make 5 mL. To 1 drop of this solution, add 0.5 mL of concentrated chromotropic acid TS, and heat on a water bath for 10 minutes; the solution exhibits a reddish purple color.

(2) To 5 mL of the test solution in (1), add 10 mL of acetone and shake to mix; a white, flocculent precipitate is formed.

(3) Add 1 mL of ferric chloride TS to 5 mL of the test solution in (1), and shake to mix; a brown, flocculent precipitate is formed.

(4) Ignite 1 g of Carboxymethylcellulose Calcium to incinerate, and dissolve the resulting residue with 10 mL of water and 6 mL of acetic acid. Filter if necessary, boil, cool, and neutralize with ammonia TS; the resulting solution responds to Chemical identification reactions (1) and (3) for calcium salt.

Purity (1) ***Alkali***—To 1.0 g of Carboxymethylcellulose Calcium, add 50 mL of freshly boiled and cooled water, shake well to mix, and add 2 drops of phenolphthalein TS; the resulting solution does not exhibit a red color.

(2) ***Chloride***—Weigh 0.8 g of Carboxymethylcellulose Calcium, add 50 mL of water, and shake well to mix. Dissolve with 10 mL of sodium hydroxide TS, add water to make 100 mL, and use this solution as the test stock solution. To 20 mL of this solution, add 10 mL of 2 mol/L nitric acid TS, heat on a water bath until a flocculent precipitate forms, cool, and centrifuge. Take the clear supernatant, wash the precipitate 3 times with 10 mL each of water, and centrifuge each time. Combine the clear supernatant and the washings, and add water to make 100 mL. Take 25 mL of this solution, and add 1 mL of nitric acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution with 0.40 mL of 0.01 mol/L hydrochloric acid (NMT 0.36%).

2 mol/L nitric acid TS—Add water to 12.9 mL of nitric acid to make 100 mL.

(3) ***Sulfate***—To 10 mL of the test stock solution in (2), add 1 mL of hydrochloric acid, heat on a water bath until a flocculent precipitate forms, cool, and then centrifuge. Take the clear supernatant, wash the precipitate 3 times with 10 mL each of water, and centrifuge each time. Combine the clear supernatant and the washings, and add water to make 100 mL. Use 25 mL of this solution as the test solution and perform the test. Prepare the control solution with 0.42 mL of 0.005 mol/L hydrochloric acid. Add 1 mL of 3 mol/L hydrochloric acid TS and 3 mL of barium chloride TS each to the test solution and the control solution, then add water to make 50 mL, and mix. Allow to stand for 10 minutes, and compare turbidity. The turbidity of the test solution is not thicker than that of the control solution (NMT 1.0%).

Loss on drying NMT 10.0% (1 g, 105 ℃, 4 hours).

Residue on ignition Between 10.0% and 20.0% (after drying, 1 g).

Packaging and storage Preserve in tight containers.

Carboxymethylcellulose Sodium
카르복시메틸셀룰로오스나트륨

Carmellose Sodium
CMC Sodium
[*9004-32-4*]

Carboxymethylcellulose Sodium is the sodium salt of a polycarboxymethylether of cellulose. Carboxymethylcellulose Sodium, when dried, contains NLT 6.5% and NMT 8.5% of sodium (Na: 22.99).

Description Carboxymethylcellulose Sodium occurs as a white to yellow powder or grain, and is tasteless.
It is practically insoluble in methanol, acetic acid (100), ethanol or ether.
Add water or warm water to Carboxymethylcellulose Sodium; it becomes a viscous liquid.
It is hygroscopic.

Identification (1) Dissolve 2.0 g of Carboxymethylcellulose Sodium in 20 mL of warm water while stirring to mix, cool, and use this solution as the test solution. To 1 mL of the test solution, add water to make 5 mL. 1 drop of this solution, add 0.5 mL of concentrated chromotropic acid TS, and heat on a water bath for 10 minutes; the solution exhibits a purple color.

(2) To 10 mL of the test solution in (1), add 1 mL of

copper sulfate TS; a blue flocculent precipitate forms.

(3) To 3 g of Carboxymethylcellulose Sodium, add 20 mL of methanol and 2 mL of dilute hydrochloric acid, boil gently on a water bath for 5 minutes, and filter. Evaporate the filtrate to dryness, and add 20 mL of water to the residue; the solution responds to the Chemical identification reactions for sodium salt.

pH Dissolve 1.0 g of Carboxymethylcellulose Sodium in small portions in 100 mL of warm water while stirring, and cool; the pH of the solution is 6.5 to 8.0.

Viscosity Weigh the amount equivalent to 2.00 g of Carboxymethylcellulose Sodium, calculated on the dried basis, and slowly add it to 50 mL of water while mixing well using a stirrer. If necessary for the preparation of a low-viscosity substance, dilute to the corresponding concentration. Slowly heat this solution while stirring until it reaches approximately 90 °C, then cool to an ordinary temperature, add water to make 100 mL, and stir well until completely dissolved. Perform the test according to Method 2 under Viscosity at 20 ℃; it is 75.0% to 140.0% of the labeled viscosity.

Purity (1) ***Clarity and color of solution*—**Firmly attach a glass plate of good quality, 2 mm in thickness, to the bottom of a glass column 250 mm in height, 25 mm in inner diameter and 2 mm in thickness, and use this tube as an outer tube. Similarly prepare an inner tube by attaching a glass plate of good quality, 2 mm in thickness, to the bottom of a glass column 300 mm in height, 15 mm in inner diameter and 2 mm in thickness. Weigh 1.0 g of Carboxymethylcellulose Sodium, dissolve in 100 mL of water, and transfer to the outer tube. Place the tube on a piece of white paper on which 15 parallel black lines, 1 mm in width and 1 mm in interval, are drawn. Observe the lines from the upper part while moving the inner tube up and down, and determine the height of the solution up to the lower edge of the inner tube when the lines become indistinguishable. Repeat this procedure 3 times; the average value obtained is greater than that obtained by proceeding with the following control solution in the same manner.

Control solution—To 5.50 mL of 0.005 mol/L sulfuric acid, add 1 mL of dilute hydrochloric acid, 5 mL of ethanol and water to make 50 mL. Mix 2 mL of barium chloride TS, allow to stand for 10 minutes, and shake to mix before use.

(2) ***Chloride*—**Dissolve 0.5 g of Carboxymethylcellulose Sodium in 50 mL of water, and use this solution as the test solution. To 10 mL of this solution, add 10 mL of dilute nitric acid, shake to mix, heat on a water bath until a flocculent precipitate forms, cool, and centrifuge. Take the clear supernatant, wash the precipitate 3 times with 10 mL each of water, centrifuge each time, combine the clear supernatant and the washings, and add water again to make 200 mL. Use 50 mL of this solution as the test solution and perform the test. Prepare the control solution with 0.45 mL of 0.01 mol/L hydrochloric acid (NMT 0.640%).

(3) ***Sulfate*—**To 10 mL of the test solution in (2), add 1 mL of hydrochloric acid, shake well to mix, and heat on a water bath until a flocculent precipitate forms. After cooling, centrifuge. Take the clear supernatant, wash the precipitate 3 times with 10 mL each of water, centrifuge each time, combine the clear supernatant and washings, and add water again to make 50 mL. To 10 mL of this solution, add water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution with 0.40 mL of 0.005 mol/L sulfuric acid (NMT 0.960%).

(4) ***Silicate*—**Weigh accurately about 1 g of Carboxymethylcellulose Sodium, transfer to a platinum dish, ignite to incinerate, and add 20 mL of dilute hydrochloric acid. Cover with a watch glass, and boil gently for 30 minutes. Remove the watch glass, heat on a water bath in a current of air, and evaporate to dryness. Continue heating for another hour, then add 10 mL of hot water, stir well to mix, and filter using filter paper for assay. Wash the residue with hot water until no turbidity forms on the addition of silver nitrate TS to the washings, dry the residue together with the filter paper, and ignite again to a constant mass; the amount is NMT 0.5%.

(5) ***Heavy metals*—**Proceed with 1.0 g of Carboxymethylcellulose Sodium according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 20 ppm).

(6) ***Mercury*—**Spread about 1 g of excipient (a) evenly on a ceramic boat, and place 10 mg to 300 mg of Carboxymethylcellulose Sodium on top. Then, evenly spread about 0.5 g of excipient (a) and 1 g of excipient (b) on top it in turn to form a layer. However, in the case of an automatic mercury analyzer equipped with a separate catalyst in the combustion chamber, take only the sample without adding excipients to the nickel boat. Place the boat inside the combustion furnace, and heat to about 900°C with a current of air or oxygen at 0.5 to 1 L/minute. Elute the mercury and collect in a collection tube. Transfer the mercury vapor to a cold vapor atomic absorption spectrometer by heating the collecting tube to about 700 ℃ and determine the absorbance, A. Separately, place only the excipients in a ceramic boat and determine the absorbance, Ab in the same manner. Separately, proceed with mercury standard solution in the same manner and create a calibration curve from the absorbances. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the sample (NMT 1.0 ppm).

Operating conditions

Apparatus: Use an automated mercury analyzer for specimen combustion, trapping with gold amalgam and measurement using a cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Standard mercury stock solution—Dissolve 0.135 g of mercury(II) chloride in 0.001% L-cysteine solution to make 1000 mL. Each mL of this solution contains 100 μg of mercury(II) chloride.

Mercury standard solution—Dilute the standard mercury stock solution with 0.001% L-cysteine solution to make 0 to 200 ng/mL.

Excipients—Activate excipients at 950 ℃ for 30 minutes when using (a) aluminum oxide and (b) calcium hydroxide-sodium carbonate (1 : 1).

(7) ***Cadmium*—**Weigh accurately 5.0 g of Carboxymethylcellulose Sodium, transfer to a platinum crucible, dry, carbonize, and incinerate at 450 °C to 550 °C. If incineration is not complete, cool and moisten with 2 mL to 5 mL of nitric acid (1 in 2), 50% magnesium nitrate solution or aluminum nitrate-calcium nitrate solution (prepared by dissolving 40 g of aluminum nitrate and 20 g of calcium nitrate in 100 mL of water) as an incineration supplement, dry, and continue with incineration. If incineration is incomplete, repeat the above procedure once and then add a final 2 mL to 5 mL of nitric acid (1 in 2), if necessary, and incinerate After incineration, moisten the residue with water, add 2 mL to 4 mL of hydrochloric acid, and evaporate to dryness. Add 0.5 mol/L nitric acid, dissolve by warming, and filter with filter

paper if any insoluble matter exists. Unless otherwise specified, add 0.5 mol/L nitric acid to make 25 mL and use this solution as the test solution. Separately, proceed with 5.0 mL of cadmium standard solution in a platinum crucible in the same manner as the test solution, and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Atomic absorption spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 1.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: A cadmium hollow-cathode lamp
Wavelength: 228.8 nm

(8) ***Lead*—**Weigh 5.0 g of Carboxymethylcellulose Sodium, place it in a platinum crucible, dry, carbonize, and incinerate at 450 °C to 550 °C. If incineration is not complete, cool and moisten with 2 mL to 5 mL of nitric acid (1 in 2), 50% magnesium nitrate solution or aluminum nitrate-calcium nitrate solution (prepared by dissolving 40 g of aluminum nitrate and 20 g of calcium nitrate in 100 mL of water) as an incineration supplement, dry, and continue with incineration. If incineration is incomplete, repeat the above procedure once and then add a final 2 mL to 5 mL of nitric acid (1 in 2), if necessary, and incinerate After incineration, moisten the residue with water, add 2 mL to 4 mL of hydrochloric acid, and evaporate to dryness. Add 0.5 mol/L nitric acid, dissolve by warming, and filter with filter paper if any insoluble matter exists. Unless otherwise specified, add 0.5 mol/L nitric acid to make 25 mL and use this solution as the test solution. Separately, proceed with 1.0 mL of lead standard solution in a platinum crucible in the same manner as the test solution, and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Atomic absorption spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 2.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: A lead hollow-cathode lamp
Wavelength: 283.3 nm

(9) ***Arsenic*—**Weigh 1.0 g of Carboxymethylcellulose Sodium, add 20 mL of nitric acid, and heat gently until it becomes fluid. After cooling, add 5 mL of sulfuric acid, and heat until white fumes are evolved. If necessary, after cooling, add 5 mL of nitric acid and heat again. Repeat this procedure until the solution becomes a colorless to pale yellow. After cooling, add 15 mL of saturated ammonium oxalate solution, and heat until white fumes are evolved again. After cooling, add water to make 25 mL. Perform the test with 5 mL of this solution as the test solution; the color is not more intense than the following standard color.

◦ *Standard color* Prepare a solution by proceeding in the same manner without using Carboxymethylcellulose Sodium. Put 5 mL of this solution into a generator bottle, add exactly 2 mL of arsenic standard solution, and then proceed in the same manner as in the preparation of the test solution (NMT 10 ppm).

(10) ***Starch*—**Take 10 mL of the test solution in (2), and add 2 drops of iodine solution, 1 drop at a time; the solution does not exhibits a blue color.

Loss on drying NMT 10.0% (1 g, 105 ℃, 4 hours).

Assay Weigh accurately about 0.5 g of Carboxymethylcellulose Sodium, previously dried, and add 80 mL of acetic acid (100), attach a reflux condenser, and heat in an oil bath at 130 °C for 2 hours. After cooling, titrate with 0.1 mol/L perchloric acid VS (potentiometric titration under the Titrimetry). Perform a blank test in the same manner and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 2.2990 mg of Na

Packaging and storage Preserve in tight containers.

Carboxymethylcellulose Sodium Tablets
카르복시메틸셀룰로오스나트륨 정

Carmellose Sodium Tablets
CMC Sodium Tablets

Carboxymethylcellulose Sodium Tablets contain sodium (Na: 22.99) equivalent to NLT 6.5% and NMT 9.5% of the labeled amount of carboxymethylcellulose sodium.

Method of preparation Prepare as directed under Tablets, with Carboxymethylcellulose Sodium

Identification Powder Carboxymethylcellulose Sodium Tablets, weigh an amount equivalent to 1 g of carboxymethylcellulose sodium, dissolve in 50 mL of water, and filter. Perform the test with the filtrate as directed below.

(1) Add 3 mL of hydrochloric acid to 30 mL of the filtrate; a white precipitate is formed.

(2) Add the same amount of barium chloride TS to the filtrate; a fine white precipitate is formed.

(3) The filtrate obtained in (1) responds to the Chemical identification reactions for sodium salt.

Disintegration Meets the requirements. However, the test time is 2 hours.

Uniformity of dosage units Meets the requirements.

Assay Take NLT 20 tablets of Carboxymethylcellulose Sodium Tablets, weigh accurately the mass, and powder them. Weigh accurately an amount of the powdered tablets equivalent to about 0.5 g of carboxymethylcellulose sodium, add 80 mL of acetic acid (100), heat on a water bath for 2 hours, cool, and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration under the Titrimetry). Perform a blank test in the same manner and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 2.2990 mg of Na

Packaging and storage Preserve in tight containers.

Carnauba Wax
카르나우바납

Carnauba Wax is a wax obtained from the leaves of *Copernicia cerifera* Mart (Palmae).

Description Carnauba Wax occurs as a pale yellow to pale brown, hard but brittle mass or a white to pale yellow powder. It has a slightly characteristic odor and is nearly tasteless.
It is practically insoluble in water, ethanol, ether or xylene.

Specific gravity d_{20}^{20}: Between 0.990 and 1.002
Melting point: Between 80 ℃ and 86 ℃

Saponification value Between 78 and 95. Weigh accurately about 3 g of Carnauba Wax, transfer to a 300-mL flask, add 25 mL of xylene, and warm to dissolve. Add 50 mL of ethanol and exactly 25 mL of 0.5 mol/L potassium hydroxide-ethanol solution, and perform the saponification value test. However, heat for 2 hours and titrate when it is hot.

Acid value NMT 10.0. Use a mixture of xylene and ethanol (2 : 1) as a solvent.

Iodine value Between 5 and 14 (Put the sample in a flask with a stopper, and shake in a warm bath to dissolve.)

Purity ***Heavy metals*—**Proceed with about 1.0 g of Carnauba Wax according to Method 2 under the Heavy metals, and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 20 ppm).

Residue on ignition Weigh accurately about 2 g of Carnauba Wax, transfer to a porcelain or platinum dish, and heat; the volatile substance has no foul odor, and when ignited to a constant mass, the residue is NMT 5 mg (NMT 0.25%).

Packaging and storage Preserve in well-closed containers.

Casein
카세인

Casein is prepared by reacting fresh skim milk with acid or lactic acid bacteria, followed by purification and drying processes. Casein, when dried, contains NLT 13.8% and NMT 16.0% of nitrogen (N : 14.01).

Description Casein occurs as a white to pale yellow powder, granule or flake. It is odorless and tasteless or has a slight, characteristic odor or taste.
It is practically insoluble in water, in ethanol or in diethyl ether.
It is soluble in sodium hydroxide TS or in dilute hydrochloric acid.

Identification (1) Dissolve 0.1 g of Casein in 10 mL of a solution of sodium hydroxide (1 in 10), and add 8 mL of 6 mol/L acetic acid TS; a white, cotton-like precipitate is formed.

(2) Dissolve 0.1 g of Casein in 10 mL of sodium hydroxide solution (1 in 10), add 1 drop of copper sulfate TS, and shake to mix; a blue precipitate is formed and the solution exhibits a violet color.

(3) Ignite 0.1 g of Casein at 450 to 550 ℃; smoke is generated and a characteristic odor is emitted. When no more smoke is generated, stop heating, cool, add 5 mL of dilute nitric acid to the black residue, warm to dissolve, and filter. Add 1 mL of ammonium molybdate TS to this filtrate and warm; a yellow precipitate is formed.

Purity (1) ***Clarity and color of solution***—After drying Casein in a desiccator (in vacuum, silica gel) for 4 hours, grind it into a fine powder, weigh 0.10 g of this powder, add 30 mL of water, shake to mix, and allow to stand for about 10 minutes. Then, add 2 mL of dilute sodium hydroxide TS, warm for 60 minutes at 60 ℃ to dissolve while shaking occasionally to mix, cool, and add water to make 100 mL; the resulting solution is colorless and not more turbid than the following control solution.

Control solution—To 15.0 mL of chloride standard solution, add 10 mL of water, 1 mL of diluted nitric acid (1 in 3), 0.2 mL of dextrin solution (1 in 50) and 1 mL of silver nitrate TS, then add water to make 50 mL, shake to mix, and allow to stand for 15 minutes, avoiding direct sunlight.

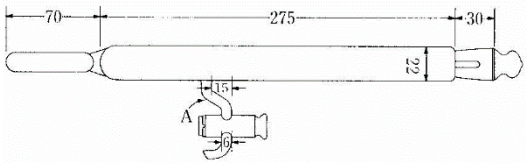
(2) ***Acidity or alkalinity***— Dissolve 1.0 g of Casein in 50 mL of water, shake for 10 minutes to mix, and filter; the pH of the filtrate is 3.7 to 6.5.

(3) ***Water-soluble substances***—Dissolve 1.0 g of Casein in 30 mL of water, shake for 10 minutes to mix, and filter. Evaporate 20 mL of this filtrate to dryness on a water bath and dry it at 100°C to a constant mass, and weigh; the amount of residue is NMT 10.0 mg.

(4) ***Heavy metals***—Proceed with 1.0 g of Casein according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 20 ppm).

(5) ***Arsenic***—Prepare the test with 1.0 g of Casein according to Method 4 and perform the test (NMT 2 ppm).

(6) ***Fat***—Dry a flask in advance at 100°C for 30 minutes, leave it in a desiccator (silica gel) to cool, and then measure its mass. Weigh accurately 2.5 g of Casein in another container, add 15 mL of diluted hydrochloric acid (2 in 3), heat slowly to dissolve, and heat on a water bath for 20 minutes. After cooling, add 10 mL of ethanol, transfer to a cooler (Rörig tube), add 25 mL of diethyl ether, shake vigorously for 1 minute to mix, add 25 mL of petroleum ether, shake vigorously for 30 seconds to mix, and allow to stand. Filter the clear supernatant obtained from the geodesic tube (A) and place the filtrate into a flask, of which mass has been measured previously. Repeat the procedure in the same manner twice each time with 15 mL of diethyl ether and 15 mL of petroleum ether, combine the clear supernatant, and then evaporate the diethyl ether and petroleum ether in the water bath. Dry the residue at 100°C for 4 hours, leave in a desiccator (silica gel) to cool, and then weigh; the amount is NMT 1.5%.



Rörig tube (size: mm)

Loss on drying NMT 12.0% (1 g, 100 ℃, 3 hours).

Residue on ignition NMT 2.5% (1 g, after drying).

Assay Weigh accurately about 15 mg of Casein, previously dried, and perform the test as directed under the Nitrogen Determination (Semimicro-Kjeldahl Method).

Each mL of 0.005 mol/L sulfuric acid VS
= 0.14007 mg of N

Packaging and storage Preserve in tight containers.

Castor Oil
피마자유

leum Ricini

Castor Oil is the fatty oil obtained by expression from the seeds of *Ricinus communis* Linné (*Euphorbiaceae*).

Description Castor Oil occurs as a colorless to pale yellow, clear, viscous oil, has a slight, characteristic odor and tastes bland at first but afterward turns slightly acrid.
It is miscible with anhydrous ethanol or ether.
It is freely soluble in ethanol and practically insoluble in water.
When cooled to 0 °C, Castor Oil becomes more viscous and turbidity gradually develops.

Identification To 3 g of Castor Oil, add 1 g of potassium hydroxide and heat the mixture carefully to fuse; a characteristic odor is perceptible. Dissolve the fused matter in 30 mL of water, add an excess of magnesium oxide and filter. Acidify the filtrate with hydrochloric acid; white crystals are produced.

Saponification value Between 176 and 187

Specific gravity d_{25}^{25}: Between 0.953 and 0.965

Acid value NMT 1.5

Hydroxyl value Between 155 and 177

Iodine value Between 80 and 90

Purity (1) ***Adulteration***—To 1.0 g of Castor Oil, add 4.0 mL of ethanol and shake to mix; it dissolves clearly. Add 15 mL of ethanol; the solution exhibits no turbidity.

(2) ***Heavy metals***—Proceed with 2.0 g of Castor Oil according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 10 ppm).

(3) ***Peroxide value***—Weigh accurately 5 g of Castor Oil, transfer to a stoppered Erlenmeyer flask and dissolve in 50 mL of a mixture of trimethylpentane and acetic acid (100) (2 : 3). To this solution, add 0.5 mL of a saturated potassium iodide solution, stopper the flask, allow to stand for 1 minute, shake continuously, and then add 30 mL of water. Titrate this solution with 0.01 mol/L sodium thiosulfate VS. However, the endpoint of the titration is when the blue color of the solution disappears after addition of 0.5 mL of starch TS when the color of the solution turns to a pale yellow color. Perform a blank test and make any necessary correction (NMT 0.1 mL of 0.01 mol/L sodium thiosulfate VS is consumed by the blank test solution). The peroxide value calculated by the following equation is NMT 10.0.

$$\text{Peroxide value (mEq/kg)} = [10 \times (V_1 - V_0)] / W$$

V_1: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed in the titration of the test solution
V_0: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed in the titration of the blank test solution
W: Amount (g) of Castor Oil taken

Packaging and storage Preserve in tight containers.

Cellacefate
셀라세페이트

Cellulose acetate phthalate

Cellulose acetate phthalate [*9004-38-0*]

Cellacefate is a reaction product of anhydrous phthalic acid and partially acetylated cellulose.

Cellacefate contains NLT 21.5% and NMT 26.0% of acetyl group (-$COCH_3$: 43.04) and NLT 30.0% and NMT 36.0% of phthalyl group (o-carboxybenzoyl) (-COC_6H_4COOH: 149.12), calculated on the anhydrous basis without free acid.

Description Cellacefate occurs as a white powder or granule.
It is freely soluble in acetone, and practically insoluble in water or ethanol (99.5).
It dissolves in dilute alkaline hydroxide solution.

Identification Determine the infrared spectra of Cellacefate and cellacefate RS, without drying, as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

Viscosity Weigh accurately an amount of Cellacefate, equivalent to 15 g calculated on the anhydrous basis, and dissolve in 85 g of a mixture of acetone and water in a mass ratio of 249 : 1. With this solution, determine the kinematic viscosity ν at 25 ± 0.12 ℃ according to Method 1 under the Viscosity and density ρ as directed under the Specific gravity and density, and calculate the viscosity η using the equation η =ρν; the resulting value is 45 to 90 mPa·s.

Purity ***Free acid***—Weigh accurately 3.0 g of Cellacefate, put into an Erlenmeyer flask with a stopper, add 100 mL of diluted methanol (1 in 2), and stopper. Shake to mix for 2 hours, and filter. Wash both the flask and residue twice with 10 mL each of diluted methanol (1 in 2), combine the washings and the filtrate, and titrate with 0.1 mol/L sodium hydroxide VS (indicator: 3 drops of phenolphthalein TS). Perform a blank test with 120 mL of diluted methanol (1 in 2) and make any necessary correction. The quantity of free acid is NMT 3.0% as phthalic acid ($C_8H_6O_4$: 166.13).

$$\text{Content (\%) of free acid} = (0.8306 \times A)/W$$

A: Amount (mL) of 0.1 mol/L sodium hydroxide VS consumed
W: Amount (g) of sample calculated on the anhydrous basis

Water NMT 5.0% (0.5 g, volumetric titration, direct titration. In this case, use a mixture of ethanol (99.5) and dichloromethane (3 : 2), instead of methanol for Karl Fischer titration).

Residue on ignition NMT 0.1% (1 g).

Assay (1) ***Phthalyl group***—Weigh accurately about 1 g of Cellacefate, dissolve in 50 mL of a mixture of ethanol (95) and acetone (3 : 2), and titrate with 0.1 mol/L sodium hydroxide VS (indicator: 2 drops of phenolphthalein TS). Perform a blank test in the same manner and make any necessary correction.

$$\text{Content (\%) of phthalyl group } (C_8H_5O_3) = \frac{\frac{1.491 \times A}{W} - 1.795 \times B}{100 - B} \times 100$$

A: Amount (mL) of 0.1 mol/L sodium hydroxide VS consumed
B: Content (%) of free acid obtained from the free acid test

W: Amount (g) of sample calculated on the anhydrous basis

(2) ***Acetyl group***—Weigh accurately about 0.1 g of Cellacefate, put into an Erlenmeyer flask with a stopper, add exactly 25 mL of 0.1 mol/L sodium hydroxide, and boil for 30 minutes under a reflux condenser. After cooling, add 2 to 3 drops of phenolphthalein TS, and titrate the excess sodium hydroxide with 0.1 mol/L sulfuric acid VS. Perform a blank test in the same manner.

$$\text{Content (\%) of acetyl group } (C_2H_3O) \text{ in free acid and combined acid} = 0.4305 \times A / W$$

A: Volume (mL) of 0.1 mol/L sodium hydroxide consumed, corrected by a blank test
W: Amount (g) of sample calculated on the anhydrous basis

$$\text{Content (\%) of acetyl group } (C_2H_3O) = 100 \times [P - (0.5182 \times B)]/(100 - B) - (0.5772 \times C)$$

B: Content (%) of free acid obtained from the free acid test
C: Content (%) of phthalyl group obtained from the phthalyl group test
P: Content (%) of acetyl group (C_2H_3O) in free acid and combined acid

Packaging and storage Preserve in tight containers.

Microcrystalline Cellulose
미결정셀룰로오스

[*9004-34-6*, cellulose]

Microcrystalline Cellulose is purified, partially depolymerized α-cellulose, obtained as a pulp from fibrous plant material, with mineral acids.

The label indicates the degree of polymerization, loss on drying and bulk density values with the range.

Description Microcrystalline Cellulose occurs as a white or nearly white crystalline fine powder, fluid and non-fibrous.
It is practically insoluble in sodium hydroxide solution (1 in 20), and insoluble in water, dilute acid and most organic solvents.

Identification (1) Dissolve 20 g of zinc chloride and 6.5 g of potassium iodide in 10.5 mL of water, add 0.5 g of iodine, and shake to mix for 15 minutes. Disperse 10 mg of Microcrystalline Cellulose in 2 mL of this solution on a watch glass; the substances exhibit a bluish purple color.

(2) Determine the infrared spectra of Microcrystalline Cellulose and microcrystalline cellulose RS as directed in the ATR method or in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers. In this case, exclude the absorptions in the range of 800 cm^{-1} to 825 cm^{-1} and 950 cm^{-1} to 1000 cm^{-1}.

(3) Transfer about 1.3 g of Microcrystalline Cellulose, accurately weighed, into a 125-mL Erlenmeyer flask and add 25 mL each of water and 1 mol/L cupriethylenediamine TS. Pass through nitrogen, seal it tightly, and shake to dissolve using a shaker. Determine the kinematic viscosity ν of this solution at 25 ± 0.1 ℃ according to Method 1 under the Viscosity, using a capillary viscometer with an approximate viscometer constant (K) of 0.03. Separately, proceed with a mixture of 25 mL each of water and 1 mol/L copper ethylenediamine TS, and determine the kinematic viscosity ν_0 of this solution in the same manner as the above, using a capillary viscometer with an approximate viscometer constant (K) of 0.01. Determine the relative viscosity η_{re1} of Microcrystalline Cellulose with the following equation.

$$\eta_{rel} = \nu / \nu_o$$

Obtain the value [η]•C of multiplying the limiting viscosity [η] (mL/g) and concentration C (g/100 mL) from the relative viscosity η_{re1} according to the following table, and calculate the average degree of polymerization P with the following equation; P is NMT 350 or within the labeled range.

$$P = 95[\eta] \cdot C / W$$

W: = Amount (g) of sample calculated on the dried basis

pH Dissolve 5.0 g of Microcrystalline Cellulose in 40 mL of freshly boiled and cooled water, shake for 20 minutes to mix, and centrifuge; the pH of the clear supernatant is 5.0 to 7.5.

Purity (1) ***Water-soluble substances***—To 5.0 g of Microcrystalline Cellulose, add 80 mL of water, shake for 10 minute to mix, and filter under suction with a filter paper. Evaporate the filtrate to dryness in a previously weighed beaker, preventing the contents from scorching, dry at 105 ℃ for 1 hour, allow to cool in a desiccator (silica gel), and weigh accurately the mass; the residue is NMT 12.5 mg (0.25%). Perform a blank test in the same manner and make any necessary correction.

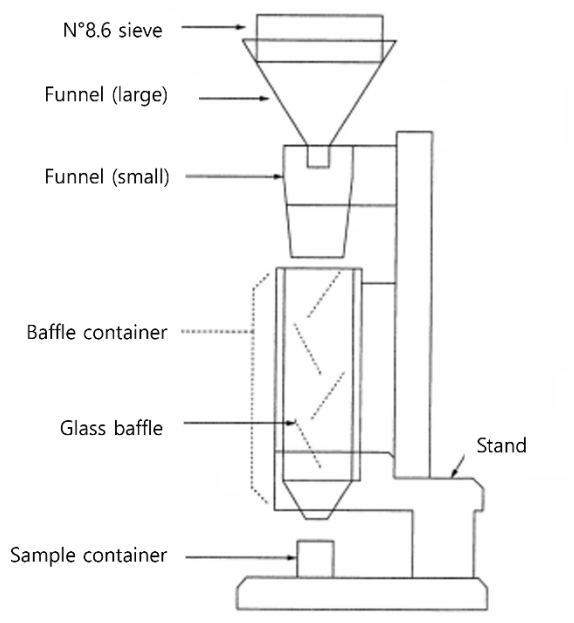
(2) ***Ether solubles***—Put 10.0 g of Microcrystalline Cellulose in a column for chromatography with an internal diameter of about 20 mm, and add slowly 50 mL of peroxide-free ether into this column. Evaporate the eluate to dryness in a previously dried and tared evaporation dish. Dry the residue at 105 ℃ for 30 minutes, allow to cool in a desiccator (silica gel), and weigh accurately the mass; it is NMT 5.0 mg (0.05%). Perform a blank test in the same manner and make any necessary correction.

Conductivity Use the clear supernatant from the pH section as the test solution. Measure and compare the conductivities of the test solution and the water used for the test at 25 ± 0.1 ℃; the difference is NMT 75 $\mu S \cdot cm^{-1}$.

Loss on drying NMT 7.0% or within the labeled range (1.0 g, 105 ℃, 3 hours).

Residue on ignition NMT 0.1% (1.0 g).

Bulk density (1) ***Equipment***—Use a volumetric measuring instrument as shown in the figure.



<Figure>

On top of the volumetric measuring instrument, place a N°8.6 (2000 μm) sieve. Place the funnel on top of the baffled container with 4 glass baffle plates attached. Drop the sample onto the 4 glass baffle plates while allowing to flow. Collect the dropped sample in the sample container through the sliding device attached to the bottom of the baffle container.

(2) ***Procedure*—**Weigh accurately the mass of the sample container made of copper or stainless steel with an internal diameter of 30.0 ± 2.0 mm and an inner volume of 25.0 ± 0.05 mL, and place under the sliding device of the volumetric measuring instrument. Pour slowly Microcrystalline Cellulose from a height of 5.1 cm above the upper boarder of the funnel on the volumetric measuring instrument, ensuring that the sieve is not blocked, and allow the sample to flow through the sieve until it overflows from the container. When the sample overflows, immediately use a slide glass to scrub away the excess, and measure accurately the mass. From this value, calculate the mass of the content, and determine the bulk density with the following equation; the value is within the labeled range.

$$\text{Bulk density (g/cm}^3\text{)} = A / 25$$

A: Mass (g) of the sample measured

Microbial limit Perform the test; the total number of aerobic microorganisms is NMT 10^3 CFU and the total number of fungi is NMT 10^2 CFU per g of Microcrystalline Cellulose. Also, *Escherichia coli*, *Salmonella*, *Pseudomonas aeruginosa* and *Staphylococcus aureus* are not detected.

Packaging and storage Preserve in tight containers.

Table for calculating the value of multiplying the limiting viscosity and the concentration from the relative viscosity ηre1, [η]·C

η_{rel}	[η]C									
	0.00	0.01	0.02	0.03	0.04	0.05	0.06	0.07	0.08	0.09
1.1	0.098	0.106	0.115	0.125	0.134	0.143	0.152	0.161	0.170	0.180
1.2	0.189	0.198	0.207	0.216	0.225	0.233	0.242	0.250	0.259	0.268
1.3	0.276	0.285	0.293	0.302	0.310	0.318	0.326	0.334	0.342	0.350
1.4	0.358	0.367	0.375	0.383	0.391	0.399	0.407	0.414	0.422	0.430
1.5	0.437	0.445	0.453	0.460	0.468	0.476	0.484	0.491	0.499	0.507
1.6	0.515	0.522	0.529	0.536	0.544	0.551	0.558	0.566	0.573	0.580
1.7	0.587	0.595	0.602	0.608	0.615	0.622	0.629	0.636	0.642	0.649
1.8	0.656	0.663	0.670	0.677	0.683	0.690	0.697	0.704	0.710	0.717
1.9	0.723	0.730	0.736	0.743	0.749	0.756	0.762	0.769	0.775	0.782
2.0	0.788	0.795	0.802	0.809	0.815	0.821	0.827	0.833	0.840	0.846
2.1	0.852	0.858	0.864	0.870	0.876	0.882	0.888	0.894	0.900	0.906
2.2	0.912	0.918	0.924	0.929	0.935	0.941	0.948	0.953	0.959	0.965
2.3	0.971	0.976	0.983	0.988	0.994	1.000	1.006	1.011	1.017	1.022
2.4	1.028	1.033	1.039	1.044	1.050	1.056	1.061	1.067	1.072	1.078
2.5	1.083	1.089	1.094	1.100	1.105	1.111	1.116	1.121	1.126	1.131
2.6	1.137	1.142	1.147	1.153	1.158	1.163	1.169	1.174	1.179	1.184
2.7	1.190	1.195	1.200	1.205	1.210	1.215	1.220	1.225	1.230	1.235
2.8	1.240	1.245	1.250	1.255	1.260	1.265	1.270	1.275	1.280	1.285
2.9	1.290	1.295	1.300	1.305	1.310	1.314	1.319	1.324	1.329	1.333
3.0	1.338	1.343	1.348	1.352	1.357	1.362	1.367	1.371	1.376	1.381
3.1	1.386	1.390	1.395	1.400	1.405	1.409	1.414	1.418	1.423	1.427
3.2	1.432	1.436	1.441	1.446	1.450	1.455	1.459	1.464	1.468	1.473
3.3	1.477	1.482	1.486	1.491	1.496	1.500	1.504	1.508	1.513	1.517
3.4	1.521	1.525	1.529	1.533	1.537	1.542	1.546	1.550	1.554	1.558
3.5	1.562	1.566	1.570	1.575	1.579	1.583	1.587	1.591	1.595	1.600
3.6	1.604	1.608	1.612	1.617	1.621	1.625	1.629	1.633	1.637	1.642
3.7	1.646	1.650	1.654	1.658	1.662	1.666	1.671	1.675	1.679	1.683
3.8	1.687	1.691	1.695	1.700	1.704	1.708	1.712	1.715	1.719	1.723
3.9	1.727	1.731	1.735	1.739	1.742	1.746	1.750	1.754	1.758	1.762
4.0	1.765	1.769	1.773	1.777	1.781	1.785	1.789	1.792	1.796	1.800
4.1	1.804	1.808	1.811	1.815	1.819	1.822	1.826	1.830	1.833	1.837
4.2	1.841	1.845	1.848	1.852	1.856	1.859	1.863	1.867	1.870	1.874
4.3	1.878	1.882	1.885	1.889	1.893	1.896	1.900	1.904	1.907	1.911
4.4	1.914	1.918	1.921	1.925	1.929	1.932	1.936	1.939	1.943	1.946
4.5	1.950	1.954	1.957	1.961	1.964	1.968	1.971	1.975	1.979	1.982
4.6	1.986	1.989	1.993	1.996	2.000	2.003	2.007	2.010	2.013	2.017
4.7	2.020	2.023	2.027	2.030	2.033	2.037	2.040	2.043	2.047	2.050
4.8	2.053	2.057	2.060	2.063	2.067	2.070	2.073	2.077	2.080	2.083
4.9	2.087	2.090	2.093	2.097	2.100	2.103	2.107	2.110	2.113	2.116
5.0	2.119	2.122	2.125	2.129	2.132	2.135	2.139	2.142	2.145	2.148
5.1	2.151	2.154	2.158	2.160	2.164	2.167	2.170	2.173	2.176	2.180
5.2	2.183	2.186	2.190	2.192	2.195	2.197	2.200	2.203	2.206	2.209
5.3	2.212	2.215	2.218	2.221	2.224	2.227	2.230	2.233	2.236	2.240
5.4	2.243	2.246	2.249	2.252	2.255	2.258	2.261	2.264	2.267	2.270
5.5	2.273	2.276	2.279	2.282	2.285	2.288	2.291	2.294	2.297	2.300
5.6	2.303	2.306	2.309	2.312	2.315	2.318	2.320	2.324	2.326	2.329
5.7	2.332	2.335	2.338	2.341	2.344	2.347	2.350	2.353	2.355	2.358
5.8	2.361	2.364	2.367	2.370	2.373	2.376	2.379	2.382	2.384	2.387
5.9	2.390	2.393	2.396	2.400	2.403	2.405	2.408	2.411	2.414	2.417
6.0	2.419	2.422	2.425	2.428	2.431	2.433	2.436	2.439	2.442	2.444
6.1	2.447	2.450	2.453	2.456	2.458	2.461	2.464	2.467	2.470	2.472
6.2	2.475	2.478	2.481	2.483	2.486	2.489	2.492	2.494	2.497	2.500
6.3	2.503	2.505	2.508	2.511	2.513	2.516	2.518	2.521	2.524	2.526
6.4	2.529	2.532	2.534	2.537	2.540	2.542	2.545	2.547	2.550	2.553
6.5	2.555	2.558	2.561	2.563	2.566	2.568	2.571	2.574	2.576	2.579

6.6	2.581	2.584	2.587	2.590	2.592	2.595	2.597	2.600	2.603	2.605
6.7	2.608	2.610	2.613	2.615	2.618	2.620	2.623	2.625	2.627	2.630
6.8	2.633	2.635	2.637	2.640	2.643	2.645	2.648	2.650	2.653	2.655
6.9	2.658	2.660	2.663	2.665	2.668	2.670	2.673	2.675	2.678	2.680
7.0	2.683	2.685	2.687	2.690	2.693	2.695	2.698	2.700	2.702	2.705
7.1	2.707	2.710	2.712	2.714	2.717	2.719	2.721	2.724	2.726	2.729
7.2	2.731	2.733	2.736	2.738	2.740	2.743	2.746	2.748	2.750	2.752
7.3	2.755	2.757	2.760	2.762	2.764	2.767	2.769	2.771	2.774	2.776
7.4	2.779	2.781	2.783	2.786	2.788	2.790	2.793	2.795	2.798	2.800
7.5	2.802	2.805	2.807	2.809	2.812	2.814	2.816	2.819	2.821	2.823
7.6	2.826	2.828	2.830	2.833	2.835	2.837	2.840	2.842	2.844	2.847
7.7	2.849	2.851	2.854	2.856	2.858	2.860	2.863	2.865	2.868	2.870
7.8	2.873	2.875	2.877	2.879	2.881	2.884	2.887	2.889	2.891	2.893
7.9	2.895	2.898	2.900	2.902	2.905	2.907	2.909	2.911	2.913	2.915
8.0	2.918	2.920	2.922	2.924	2.926	2.928	2.931	2.933	2.935	2.937
8.1	2.939	2.942	2.944	2.946	2.948	2.950	2.952	2.955	2.957	2.959
8.2	2.961	2.963	2.966	2.968	2.970	2.972	2.974	2.976	2.979	2.981
8.3	2.983	2.985	2.987	2.990	2.992	2.994	2.996	2.998	3.000	3.002
8.4	3.004	3.006	3.008	3.010	3.012	3.015	3.017	3.019	3.021	3.023
8.5	3.025	3.027	3.029	3.031	3.033	3.035	3.037	3.040	3.042	3.044
8.6	3.046	3.048	3.050	3.052	3.054	3.056	3.058	3.060	3.062	3.064
8.7	3.067	3.069	3.071	3.073	3.075	3.077	3.079	3.081	3.083	3.085
8.8	3.087	3.089	3.092	3.094	3.096	3.098	3.100	3.102	3.104	3.106
8.9	3.108	3.110	3.112	3.114	3.116	3.118	3.120	3.122	3.124	3.106
9.0	3.128	3.130	3.132	3.134	3.136	3.138	3.140	3.142	3.144	3.146
9.1	3.148	3.150	3.152	3.154	3.156	3.158	3.160	3.162	3.164	3.166
9.2	3.168	3.170	3.172	3.174	3.176	3.178	3.180	3.182	3.184	3.186
9.3	3.188	3.190	3.192	3.194	3.196	3.198	3.200	3.202	3.204	3.206
9.4	3.208	3.210	3.212	3.214	3.215	3.217	3.219	3.221	3.223	3.225
9.5	3.227	3.229	3.231	3.233	3.235	3.237	3.239	3.241	3.242	3.244
9.6	3.246	3.248	3.250	3.252	3.254	3.256	3.258	3.260	3.262	3.264
9.7	3.266	3.268	3.269	3.271	3.273	3.275	3.277	3.279	3.281	3.283
9.8	3.285	3.287	3.289	3.291	3.293	3.295	3.297	3.298	3.300	3.302
9.9	3.304	3.305	3.307	3.309	3.311	3.313	3.316	3.318	3.320	3.321
10	3.32	3.34	3.36	3.37	3.39	3.41	3.43	3.45	3.46	3.48
11	3.50	3.52	3.53	3.55	3.56	3.58	3.60	3.61	3.63	3.64
12	3.66	3.68	3.69	3.71	3.72	3.74	3.76	3.77	3.79	3.80
13	3.80	3.83	3.85	3.86	3.88	3.89	3.90	3.92	3.93	3.95
14	3.96	3.97	3.99	4.00	4.02	4.03	4.04	4.06	4.07	4.09
15	4.10	4.11	4.13	4.14	4.15	4.17	4.18	4.19	4.20	4.22
16	4.23	4.24	4.25	4.27	4.28	4.29	4.30	4.31	4.33	4.34
17	4.35	4.36	4.37	4.38	4.39	4.41	4.42	4.43	4.44	4.45
18	4.46	4.47	4.48	4.49	4.50	4.52	4.53	4.54	4.55	4.56
19	4.57	4.58	4.59	4.60	4.61	4.62	4.63	4.64	4.65	4.66

Powdered Cellulose
분말셀룰로오스

[*9004-34-6*, cellulose]

Powdered Cellulose is purified, mechanically pulverized α-cellulose obtained as a pulp from fibrous plant materials after processing, such as partial hydrolysis, if necessary.

The label indicates the mean degree of polymerization value with the range.

Description Powdered Cellulose occurs as a white or almost white powder.
It is slightly soluble in sodium hydroxide solution (1 in 20), and insoluble in water, dilute acid or almost all organic solvents.

Identification (1) Dissolve 20 g of zinc chloride and 6.5 g of potassium iodide in 10.5 mL of water, add 0.5 g of iodine, and shake for 15 minutes to mix. Disperse about 10 mg of Powdered Cellulose in 2 mL of this solution on a watch glass; the substances exhibit a bluish purple color.

(2) Weigh accurately about 0.25 g of Powdered Cellulose, put in a 125-mL Erlenmeyer flask, and add exactly 25 mL of water and 25 mL of 1 mol/L copper ethylenediamine TS. Pass through nitrogen, seal it tightly, and shake to dissolve using a shaker. Determine the kinematic viscosity ν of this solution at 25 ± 0.1 ℃ according to Method 1 under the Viscosity, using a capillary viscometer with an approximate viscometer constant (K) of 0.03. Separately, prepare a mixture of 25 mL of water and 25 mL of 1 mol/L copper ethylenediamine TS, and determine the kinematic viscosity ν_0 of this solution in the same manner as the above, using a capillary viscometer with an approximate viscometer constant (K) of 0.01. Determine the relative viscosity η_{re1} of Powdered Cellulose with the following equation.

$$\eta_{rel} = \nu / \nu_0$$

Obtain the value [η]•C of multiplying the limiting viscosity [η] (mL/g) and concentration C (g/100 mL) from the relative viscosity η_{re1} according to the following table, and calculate the average degree of polymerization P by the following equation; P is NLT 440 or within the labeled range.

$$P = 95[\eta] \cdot C / W$$

W: = Amount (g) of sample calculated on the dried basis

pH Dissolve 10 g of Powdered Cellulose in 90 mL of freshly boiled and cooled water, and allow to stand for 1 hour with occasional shaking; the pH of the clear supernatant is 5.0 to 7.5.

Purity (1) ***Water-soluble substances***—Add 90 mL of freshly boiled and cooled water to 6.0 g of Powdered Cellulose, shake occasionally to mix for 10 minutes, and then filter under suction. Discard the first 10 mL of the filtrate, filter under suction again with the same filter, if necessary, and take 15 mL of the clear filtrate in an evaporating dish, previously weighed. Evaporate the filtrate to dryness, while preventing the contents from scorching, dry the residue at 105 ℃ for 1 hour, allow to cool in a desiccator (silica gel), and weigh the mass; the amount is NMT 15.0 mg (1.5%). Perform a blank test in the same manner and make any necessary correction.

(2) ***Ether solubles***—Put 10.0 g of Powdered Cellulose in a column for chromatography with an internal diameter of about 20 mm, and add 50 mL of peroxide-free ether into this column. Evaporate the eluate to dryness in an evaporation dish, previously weighed. Dry the residue at 105 ℃ for 30 minutes, allow to cool in a desiccator, and weigh the mass; it is NMT 15.0 mg (0.15%). Perform a blank test in the same manner and make any necessary correction.

Loss on drying NMT 6.5% (1 g, 105 ℃, 3 hours).

Residue on ignition NMT 0.3% (1 g, calculated on the dried basis).

Microbial limit Perform the test; the total number of aerobic microorganisms is NMT 10^3 CFU and the total number of fungi is NMT 10^2 CFU per g of Powdered Cellulose. Also, *Escherichia coli*, *Salmonella*, *Pseudomonas aeruginosa* and *Staphylococcus aureus* are not detected.

Packaging and storage Preserve in tight containers.

Cetanol
세탄올

Cetanol is a mixture of solid alcohols and consists chiefly of cetanol ($C_{16}H_{34}O$: 242.44).

Description Cetanol occurs as a white lead-like substance in the form of flakes, granules or masses, has a slight characteristic odor and is tasteless.
It is very soluble in pyridine, freely soluble in ethanol, anhydrous ethanol or ether, very slightly soluble in acetic anhydride and practically insoluble in water.

Melting point Between 47 ℃ and 53 ℃. Perform the test as directed under the Melting temperature of Stearyl Alcohol.

Acid Value NMT 1.0

Hydroxyl Value Between 210 and 232

Ester Value NMT 2.0.

Iodine value NMT 2.0.

Purity Perform the test as directed in the Purity under Stearyl Alcohol.

Residue on ignition NMT 0.05% (2 g).

Packaging and storage Preserve in well-closed containers.

Chlorinated Lime
표백분

Chlorinated Lime contains NLT 30.0% of available chlorine (Cl: 35.45).

Description Chlorinated Lime occurs as a white powder and has a chlorine-like odor.
It dissolves partially in water. The solution changes red litmus paper to blue, then gradually decolorizes.

Identification (1) To Chlorinated Lime, add dilute hydrochloric acid: a gas having the odor of chlorine evolves and the gas turns the color of a moistened starch potassium iodide paper to blue.

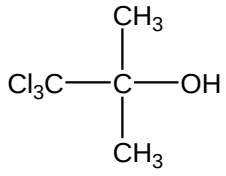
(2) To 1 g of Chlorinated Lime, add 10 mL of water, shake well to mix, and filter: the filtrate responds to the Chemical identification reactions for calcium salt.

Assay Weigh accurately about 5.0 g of Chlorinated Lime, transfer to a mortar and triturate thoroughly with 50 mL of water. Transfer to a 500-mL volumetric flask with the aid of water, and add water to make 500 mL. Shake well to mix, add 10 mL of dilute hydrochloric acid and titrate the liberated iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 3 mL of starch TS). Perform a blank test in the same manner and make any necessary correction.

Each mL of 0.1 mol/L sodium thiosulfate VS
= 3.5453 mg of Cl

Packaging and storage Preserve in light-resistant, tight containers in a cold place.

Chlorobutanol
클로로부탄올



$C_4H_7Cl_3O$: 177.46

1,1,1-Trichloro-2-methylpropan-2-ol [*57-15-8*]

Chlorobutanol contains NLT 98.0% and NMT 101.0% of chlorobutanol ($C_4H_7Cl_3O$), calculated on the anhydrous basis.

Description Chlorobutanol occurs as a colorless or white crystal and has a camphor-like odor.
It is very soluble in methanol, ethanol or ether, and slightly soluble in water.
It slowly volatilizes in the air.
Melting point: NLT about 76 ℃

Identification (1) To 5 mL of aqueous solution (1 in 200) of Chlorobutanol, add 1 mL of sodium hydroxide TS, and slowly add 3 mL of iodine TS; a yellow precipitate forms and it smells like iodoform.

(2) To 0.1 g of Chlorobutanol, add 5 mL of sodium hydroxide TS, shake well to mix, add 3 to 4 drops of aniline, and warm gently; an unpleasant odor of toxic phenyl isocyanide is perceptible.

Purity (1) ***Acid***—To 0.10 g of Chlorobutanol, previously powdered, add 5 mL of water, and shake well to mix; the solution is neutral.

(2) ***Chloride***—Dissolve 0.5 g of Chlorobutanol in 25 mL of dilute ethanol, and add 6 mL of dilute nitric acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution with 1.0 mL of 0.01 mol/L hydrochloric acid by adding 25 mL of dilute ethanol, 6 mL of dilute nitric acid and water to make 50 mL (NMT 0.071%).

Water NMT 6.0% (0.2 g, volumetric titration, direct titration).

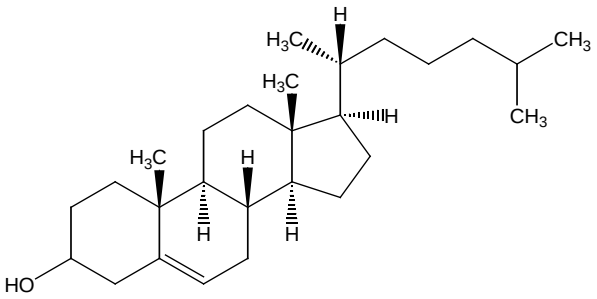
Residue on ignition NMT 0.1% (1 g).

Assay Weigh accurately about 0.1 g of Chlorobutanol, transfer to a 200-mL Erlenmeyer flask, and dissolve in 10 mL of ethanol. Then add 10 mL of sodium hydroxide TS, attach a reflux condenser, and boil for 10 minutes. After cooling, add 40 mL of dilute nitric acid and exactly 25 mL of 0.1 mol/L silver nitrate solution, and shake well to mix. Then add 3 mL of nitrobenzene, shake vigorously to mix until the precipitate hardens, and then titrate the excess silver nitrate with 0.1 mol/L ammonium thiocyanate VS (indicator: 2 mL of ammonium ferric sulfate TS). Perform a blank test in the same manner.

Each mL of 0.1 mol/L silver nitrate VS
= 5.915 mg of $C_4H_7Cl_3O$

Packaging and storage Preserve in tight containers.

Cholesterol
콜레스테롤



$C_{27}H_{46}O$: 386.65

(1*R*,2*S*,5*R*,10*S*,11*S*,14*R*,15*R*)-2,15-Dimethyl-14-[(2*R*)-6-methylheptan-2-yl]tetracyclo[8.7.0.0^{2,7}.0^{11,15}]heptadec-7-en-5-ol [*57-88-5*]

Description Cholesterol occurs as a white to pale yellow crystal or grain. It has no or a slight odor, and is tasteless.
It is freely soluble in ether or chloroform, soluble in dioxane, sparingly soluble in anhydrous ethanol, and practically insoluble in water.
It is gradually colored to yellow to pale yellowish brown by light.

Identification (1) Dissolve 10 mg of Cholesterol in 1 mL of chloroform, add 1 mL of sulfuric acid, and shake to mix; the chloroform layer exhibits a red color and the sulfuric acid layer exhibits a green fluorescence.

(2) Dissolve 5 mg of Cholesterol in 2 mL of chloroform, and add 1 mL of acetic anhydride and 1 drop of sulfuric acid, and shake to mix; the solution exhibits a red color and turns blue and then green.

Specific optical rotation Between -30° and -34° (after drying, 0.2 g, acetone, 10 mL, 100 mm).

Melting point Between 147 °C and 150 °C

Purity (1) ***Clarity and color of solution***—Weigh 0.5 g of

Cholesterol, transfer to a stoppered flask, dissolve in 50 mL of warm ethanol, and allow to stand at room temperature for 2 hours; neither turbidity nor precipitate is produced.

(2) ***Acid***—Weigh 1.0 g of Cholesterol, transfer to a flask, and dissolve in 10 mL of ether. Then add 10.0 mL of 0.1 mol/L sodium hydroxide solution, shake to mix for 1 minute, evaporate the ether, and boil again for 5 minutes. After cooling, add 10 mL of water, and titrate with 0.05 mol/L sulfuric acid VS (indicator: 2 drops of phenolphthalein TS). Perform a blank test in the same manner. The consumed volume of 0.1 mol/L sodium hydroxide solution is NMT 0.30 mL.

Loss on drying NMT 0.3% (l g, in vacuum, 60 ℃, 4 hours).

Residue on ignition NMT 0.1% (1 g).

Packaging and storage Preserve in light-resistant, tight containers.

Cinnamon Oil
육계유

Cinnamon Oil is an essential oil distilled with steam from the leaves and small branches or stem bark of *Cinnamomum cassia* Blume or the stem bark of *Cinnamomum zeylanicum* Nees (*Lauraceae*). Cinnamon Oil contains NLT 60 vol% of total aldehydes and NMT 4.0% of coumarin ($C_9H_6O_2$: 146.14).

Description Cinnamon Oil occurs as a yellow to brown liquid, and has a peculiar odor and a pungent, sweet taste.
It is miscible with ethanol or ether.
It is practically insoluble in water.
It is slightly acidic, and if stored for a long time or left in the air for a long time, its color gets more intense and its viscosity increases.

Specific gravity d_{20}^{20}: Between 1.010 and 1.065

Identification Add 4 drops of nitric acid to 4 drops of Cinnamon Oil, and shake to mix; white to pale yellow crystals form at 5℃ or lower.

Purity (1) ***Rosin***—Mix 1.0 mL of Cinnamon Oil with 5 mL of ethanol, and then add 3 mL of freshly prepared saturated ethanol solution of lead acetate; no precipitate is formed.

(2) ***Heavy metals***—Proceed with 1.0 g of Mentha Oil according to Method 2 and perform the test. Prepare the control solution with 4.0 mL of lead standard solution (NMT 40 ppm).

Assay (1) ***Total aldehydes***—Place 5.0 mL of Cinnamon Oil in a Cassia flask, dissolve in 70 mL of sodium bisulfite TS by heating on a water bath, shaking occasionally to mix, add sodium bisulfite TS up to the gauge line, allow to stand for 2 hours, and measure the amount (mL) of precipitated oil.

Total aldehydes (vol%)
= [5.0 - (Amount of precipitated oil)]×20

(2) ***Coumarin***—Take a suitable volume of Cinnamon Oil and use as the test solution. Separately, take accurately 20 mg of coumarin RS, dissolve in 1 mL of acetone, and use this solution as the standard solution. Perform the test with 0.2 μL each of the test solution and the standard solution as directed under Gas Chromatography according to the following operating conditions and determine the peak areas, A_T and A_S, of the test solution and the standard solution.

$$\text{Amount (mg) of coumarin } (C_9H_6O_2) = \text{Amount (mg) of coumarin RS} \times \frac{A_T}{A_S}$$

Operating conditions
Detector: A flame ionization detector
Column: A capillary column about 0.25 mm in internal diameter and 60 m in length, coated with polyethylene glycol 20 M for gas chromatography.
Column temperature: Maintain the temperature at 60 ℃ for 10 minutes, then raise to 190 ℃ at the rate of 2 ℃ per minute for 65 minutes, and maintain at 190 ℃ for 85 minutes.
Sample injection port temperature: A constant temperature of about 200 ℃
Detector temperature: A constant temperature of about 240 ℃
Carrier gas: Helium
Flow rate: 1.5 mL/min

Packaging and storage Preserve in light-resistant, tight containers.

Clove Oil
정향유

Clove Oil is an essential oil distilled with steam from the flower buds or leaves of *Syzygium aromaticum* Merrill et Perry (Myrtaceace). Clove Oil contains NLT 80.0 vol% of total eugenol.

Description Clove Oil occurs as a colorless to pale yellowish brown, clear liquid, and has a characteristic aroma and burning taste.
It is miscible with ethanol or ether.
It is slightly soluble in water.
It changes to brown if stored for a long time or allowed to stand in the air.

Identification (1) To 5 drops of Clove Oil, add 10 mL of calcium hydroxide TS and shake vigorously to mix; a flocculent precipitate is produced, and the solution exhibits a white to pale yellow color.

(2) Dissolve 2 drops of Clove Oil in 4 mL of ethanol, and add 1 to 2 drops of ferric chloride TS; the solution exhibits a green color.

Refractive index n_D^{20}: Between 1.527 and 1.537

Specific optical rotation $[\alpha]_D^{20}$: Between 0° and -1.5° (100 mm).

Specific gravity d_{20}^{20}: Between 1.040 and 1.068

Purity (1) ***Clarity and color of solution*—**Dissolve 1.0 mL of Clove Oil in 2.0 mL of diluted ethanol (7 in 10); the solution is clear.

(2) ***Water-soluble phenols*—**To 1.0 mL of Clove Oil, add 20 mL of hot water, shake vigorously to mix, cool, filter the water

layer, and add 1 to 2 drops of ferric chloride TS to the filtrate; the solution exhibits a yellowish green color, but no blue to violet color is observed.

(3) ***Heavy metals***—Proceed with 1.0 g of Clove Oil according to Method 2, and perform the test. Prepare the control solution with 4.0 mL of lead standard solution (NMT 40 ppm).

Assay Transfer 10.0 mL of Clove Oil into a Cassia flask, add 70 mL of sodium hydroxide TS, shake to mix for 5 minutes, and warm on a water bath for 10 minutes while shaking occasionally. After cooling, add sodium hydroxide TS up to the gauge line, allow to stand for 18 hours, and determine the amount (mL) of precipitated oil.

Amount (vol%) of total eugenol
= [10-(amount of precipitated oil)] × 10

Packaging and storage Preserve in light-resistant, tight containers.

Coconut Oil
야자유

Coconut Oil is a fatty oil obtained from the seed of *Cocos nucifera* Linné (Palmae).

Description Coconut Oil occurs as a white to pale yellow mass or a colorless to pale yellow, clear oil and has a characteristic odor and soft taste.
It is freely soluble in ether or petroleum ether and practically insoluble in water.
At a temperature below 15 ℃, Coconut Oil congeals to a hard and brittle solid.
Melting point: Between 20 ℃ and 28 ℃ (Method 2).

Saponification value Between 246 and 264

Unsaponifiable matter NMT 1.0%.

Acid value NMT 0.2.

Iodine value Between 7 and 11.

Purity (1) ***Peroxide value***—Weigh accurately 5 g of Coconut Oil, place in a 250-mL Erlenmeyer flask with a stopper, and dissolve in 30 mL of a mixture of acetic acid (100) and chloroform (3 : 2). To this solution, add 0.5 mL of a saturated solution of potassium iodide, shake for exactly 1 minute to mix, and add 30 mL of water. Titrate this solution with 0.01 mol/L sodium thiosulfate VS. The endpoint of titration is when the blue color of the solution disappears after the addition of 5 mL of starch TS at a point when the solution turns to a pale yellow color. Perform a blank test and make any necessary correction (NMT 0.1 mL of 0.01 mol/L sodium thiosulfate VS is consumed by the blank test solution). Determine the peroxide value according to the following formula; the value is NMT 5.

Peroxide value (mEq/kg)
= [10 × (V_1 - V_0)] / W

V1: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed for titration of the test solution
V0: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed for titration of the blank test solution
W: Amount (g) of Coconut Oil taken

(2) ***Alkaline impurities***—To a mixture of 10 mL of freshly distilled acetone and 0.3 mL of purified water, add 0.04 mL of bromophenol blue TS. If necessary, neutralize with 0.01 mol/L hydrochloric acid TS or 0.01 mol/L sodium hydroxide. Add 10 mL of Coconut Oil, shake to mix, allow to stand, and titrate until the clear supernatant becomes yellow in color; NMT 0.1 mL of 0.01 mol/L hydrochloric acid TS is consumed.

Packaging and storage Preserve in tight containers.

Hydrogenated Coconut Palm Oil
야자경화유

Hydrogenated Coconut Palm Oil is a fat obtained by adding hydrogen to the coconut oil.

Description Hydrogenated Coconut Palm Oil occurs as a white to pale yellow mass. It is slightly soluble in ether, chloroform, or ethanol and practically insoluble in water.

Melting point Between 38 ℃ and 40 ℃.

Congealing temperature Between 18 ℃ and 30 ℃.

Iodine value About 1.5.

Acid value NMT 0.3.

Saponification value About 260.

Loss on drying NMT 0.3% (2 g to 3 g, 105 ℃, 2 hours).

Peroxide NMT 3 mEq/kg.

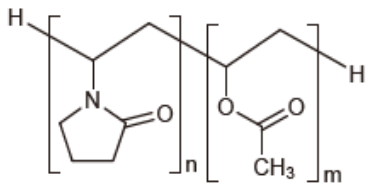
Assay Weigh accurately about 1 g of Hydrogenated Coconut Palm Oil, transfer into a 250-mL stoppered Erlenmeyer flask, and dissolve in 10 mL of chloroform. Add 15 mL of acetic acid (100) and 1 mL of potassium iodide TS, close the stopper, shake gently, and then allow to stand in a dark place for 10 minutes. Add 30 mL of water, close the stopper, and then shake vigorously. Titrate free iodine with 0.01 mol/L sodium thiosulfate VS using starch TS as the indicator. Separately, perform a blank test and make any necessary correction.

$$\text{Peroxide (mEq/kg)} = \frac{(S - B) \times F}{Amount\ (g)\ of\ sample} \times 10$$

S: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed in the test solution.
B: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed in the blank test.
F: Factor of 0.01 mol/L sodium thiosulfate VS

Packaging and storage Preserve in tight containers.

Copovidone
코포비돈



$(C_6H_9NO)_n(C_4H_6O_2)_m$

Poly[(2-oxopyrrolidin-1-yl)ethylene-co-(1-acetoxyethylene)] [*25086-89-9*]

Copovidone is a copolymer of 1-vinyl-2-pyrrolidone and vinyl acetate, in which the mass ratio is 3 : 2.

Copovidone contains NLT 35.3% and NMT 42.0% of vinyl acetate ($C_4H_6O_2$: 86.09) and NLT 7.0% and NMT 8.0% of nitrogen (N: 14.01), calculated on the dried basis.

Copovidone states its K value on the label.

Description Copovidone occurs as a white to yellowish white powder and has no odor or a slight characteristic odor.
It is very soluble in water or ethanol (95) and dichloromethane.
It is hygroscopic.

Identification Determine the infrared spectra of Copovidone and copovidone RS as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

pH Dissolve 1 g of Copovidone in 10 mL of water; the pH of this solution is 3.0 to 7.0.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Copovidone in 10 mL of water; the resulting solution is colorless to pale yellow or pale red in color, clear or slightly turbid.

(2) ***Aldehydes***—Weigh accurately about 1 g of Copovidone and dissolve in 0.05 mol/L potassium dihydrogen phosphate buffer solution (pH 9.0) to make exactly 100 mL. Seal and warm the mixture at 60 °C for 60 minutes, then allow it to cool down to room temperature, and use this solution as the test solution. Separately, weigh 0.140 g of acetaldehyde ammonia trimer trihydrate and dissolve in water to make exactly 200 mL. Pipet 1 mL of this solution, add 0.05 mol/L potassium dihydrogen phosphate buffer solution (pH 9.0) to make exactly 100 mL, and use this solution as the standard solution. Pipet 0.5 mL each of the test solution, the standard solution, and water into 1-cm cells, mix, cover the cells, and allow to stand for 2 to 3 minutes at 22 ± 2 ℃. With these solutions, perform the test as directed under the Ultraviolet-visible Spectroscopy with water as a reference solution, and determine absorbances A_{T1}, A_{S1}, and A_{B1} at the wavelength of 340 nm. Then, add 0.05 mL of aldehyde dehydegenase TS to each solution, stir, cover the cells, and allow to stand at 22 ± 2 ℃ for 5 minutes. Proceed in the same manner to determine the absorbances A_{T2}, A_{S2}, and A_{B2} (NMT 500 ppm).

Potassium dihydrogen phosphate buffer solution, 0.05 mol/L, pH 9.0—Weigh 17.4 g of potassium dihydrogen phosphate and dissolve it in 80 mL of water. Add 1 mol/L potassium hydroxide to adjust pH to 9.0. Add water again to make 100 mL.

Amount (ppm) of aldehyde [as acetaldehyde (CH_3CHO)

$$= (C / M) \times \{(A_{T2} - A_{T1}) - (A_{B2} - A_{B1})\} / \{(A_{S2} - A_{S1}) - (A_{B2} - A_{B1})\} \times 100000$$

M: Amount (g) of Copovidone taken, calculated on the dried basis

C: Concentration (mg/mL) of acetaldehyde in the standard solution. The conversion factor from acetaldehyde ammonia trimer trihydrate to acetaldehyde is 0.72.

(3) ***1-Vinyl-2-pyrrolidone and free vinyl acetate***—Store the test solution and the standard solution below 10 ℃ and use them within 8 hours. Weigh accurately about 0.25 g of Copovidone, dissolve it in a mixture of water and acetonitrile (23 : 2) to make exactly 10 mL, and use this solution as the test solution. Separately, weigh 50 mg of 1-vinyl-2-pyrrolidone and 50 mg of vinyl acetate and dissolve in methanol to make exactly 100 mL. Pipet 1 mL of this solution and add methanol to make exactly 100 mL. Pipet 5 mL of the resulting solution, add a mixture of water and acetonitrile (23 : 2) to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 20 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas A_{Ta}, A_{Tb}, A_{Sa}, and A_{Sb} of 1-vinyl-2-pyrrolidone and free vinyl acetate (NMT 10 ppm, respectively)

Amount (ppm) of 1-vinyl-2-pyrrolidone

$$= (A_{Ta} / A_{Sa}) \times (C_{Sa} / C_T) \times 1000$$

Amount (ppm) of free vinyl acetate

$$= (A_{Tb} / A_{Sb}) \times (C_{Sb} / C_T) \times 1000$$

C_{ST}: Concentration (μg/mL) of 1-vinyl-2-pyrrolidone in the standard solution

C_{Sb}: Concentration (μg/mL) of vinyl acetate in the standard solution

C_T: Concentration (mg/mL) of Copovidone in the test solution, calculated on the dried basis

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 235 nm for 1-vinyl-2-pyrrolidone and 205 nm for vinyl acetate)

Column: A stainless steel column about 4 mm in internal diameter and 250 mm in length, packed with octylsilane silica gel for liquid chromatography (5 μm in particle diameter). For a guard column, use a stainless steel column about 4 mm in internal diameter and 33 mm in length, packed with octylsilane silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 40 °C.

Mobile phase: A mixture of water and acetonitrile (23 : 2)

Flow rate: 1.0 mL/min

Time span of measurement: 40 minutes

Column washing: After performing the test with the test solution, wash the column or the guard column by allowing the mobile phase to flow through at the above flow rate in the opposite direction to the test procedure for about 30 minutes to elute the test solution

System suitability

System performance: Proceed with 20 μL of the standard solution according to the above conditions; 1-vinyl-2-pyrrolidone and vinyl acetate are eluted in this order with the resolution between these peaks being NLT 2.0.

System repeatability: Repeat the test 6 times with 20 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak areas of 1-vinyl-2-pyrrolidone and vinyl acetate is NMT 2.0%, respectively.

(4) ***Peroxides***—Weigh accurately an amount of Copovidone equivalent to 4.0 g calculated on the dried basis, dissolve it in water to make exactly 100 mL, and use this solution as the test solution. Add 2 mL of titanium(III) chloride-sulfuric acid TS to 25 mL of this solution, stir, and allow the mixture to stand for 30 minutes. Separately, add 2 mL of dilute sulfuric acid (13 in 100) to 25 mL of the test solution. Perform the test with this solution as a reference solution as directed under the Ultraviolet-visible Spectroscopy; the absorbance of the test solution at the wavelength of 405 nm is NMT 0.35 (NMT 400 ppm as hydrogen peroxide),

(5) ***Hydrazine***—Weigh accurately an amount of Copovidone equivalent to 2.5 g calculated on the dried basis, place it in a 50-mL centrifuge tube, add 25 mL of water, and stir to dissolve. Add 500 μL of a solution of salicylaldehyde in methanol (1 in 20) and warm on a water bath at 60 °C for 15 minutes while slowly shaking. After cooling, add 2.0 mL of toluene, stopper the tube, shake vigorously for 2 minutes to mix, centrifuge, and use the supernatant as the test solution. Separately, dissolve 90 mg of salicylaldazine in toluene to make exactly 100 mL. Pipet 1 mL of this solution, add toluene to make exactly 100 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel (with fluorescent indicator) for thin layer chromatography. Develop the plate with a mixture of methanol and water (2 : 1) as the developing solvent to a distance of about 3/4 of the length of the plate, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 365 nm); the spots obtained from the test solution are not more intense than the spots obtained from the standard solution (NMT 1 ppm).

(6) ***2-Pyrrolidone***—Weigh accurately about 1 g of Copovidone, add 5 mL of methanol, dissolve by sonication, and add water to make exactly 100 mL. Use this solution as the test solution. Separately, weigh 0.150 g of 2-pyrrolidone and dissolve it in a mixture of water and methanol (19 : 1) to make exactly 100 mL. Pipet 3 mL of this solution, add a mixture of water and methanol (19 : 1) to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 20 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas, A_T and A_S, of 2-pyrrolidone (NMT 0.5%).

$$\text{Amount (\%) of 2-pyrrolidone} = (A_T / A_S) \times (C_S / C_T) \times 100$$

C_S: Concentration (mg/mL) of 2-pyrrolidone in the standard solution

C_T: Concentration (mg/mL) of Copovidone in the test solution, calculated on the dried basis

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 205 nm)

Column: A stainless steel column about 4 mm in internal diameter and about 150 mm in length, packed with octylsilane silica gel for liquid chromatography (5 μm in particle diameter). For a guard column, use a stainless steel column about 4 mm in internal diameter and about 10 mm in length, packed with octylsilane silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 40°C.

Mobile phase: A mixture of water and methanol (19 : 1)

Flow rate: 0.8 mL/min

Time span of measurement: 30 minutes

Column washing: After performing the test with the test solution, wash the column or the guard column by allowing the mobile phase to flow through at the above flow rate in the opposite direction to the test procedure for about 30 minutes to elute the test solution

System suitability

System performance: Proceed with 20 μL of the standard solution according to the above conditions; the symmetry factor of the 2-pyrrolidone peak is NMT 1.5.

System repeatability: Repeat the test 6 times with 20 μL each of the standard solution according to the above operating conditions; the relative standard deviation of the 2-pyrrolidone peak areas is NMT 2.0%.

Loss on drying NMT 5.0% (0.5 g, 105℃, 3 hours).

Residue on ignition NMT 0.1% (1 g)

K-value Weigh accurately an amount of Copovidone equivalent to 1.00 g calculated on the dried basis, dissolve it in water to make exactly 10 mL, allow the solution to stand for 60 minutes, and use this solution as the test solution. Perform the test with the test solution and water at 25 °C according to Method 1 under the Viscosity. Determine the K value using the following formula; it meets the requirements when it is 90.0% to 110.0% of the nominal K value stated on the label.

$$K = \frac{1.5 \log \upsilon_{rel} - 1}{0.15 + 0.003_c} + \frac{\sqrt{300c \log \upsilon_{rel} + (c + 1.5c \log \upsilon_{rel})^2}}{0.15_c + 0.003c^2}$$

c: Mass (g) of Copovidone calculated on the dried basis in 100 mL of the solution

$\upsilon_{rel.}$: Ratio of the kinematic viscosity of the test solution to the kinematic viscosity of water

Assay (1) ***Vinyl acetate***—Weigh accurately about 2 g of Copovidone, and add exactly 25 mL of a mixture of 0.5 mol/L potassium hydroxide and ethanol. Add a few boiling stones, reflux the mixture for 30 minutes, and immediately titrate with 0.5 mol/L hydrochloric acid VS (indicator: 1 mL of phenolphthalein TS). Perform a blank test in the same manner and make any necessary correction.

$$\text{Amount (\%) of vinyl acetate} = 0.1 \times (86.09 / 56.11) \times [28.05 (n_2 - n_1) / M]$$

M: Amount (g) of Copovidone taken, calculated on the dried basis

n_1: Volume (mL) of 0.5 mol/L hydrochloric acid VS consumed in the titration of Copovidone

n_2: Volume (mL) of 0.5 mol/L hydrochloric acid VS consumed in the blank test

(2) ***Nitrogen***—Weigh accurately about 0.1 g of Copovidone, place it in a Kjeldahl flask, and add 5 g of reaction catalyst (a powdered mixture of 33 g of potassium sulfate, 1 g of Copper (II) sulfate pentahydrate, and 1 g of titanium(IV) oxide). Rinse down any adhering material on the neck of the flask with a

small amount of water and add 7 mL of sulfuric acid along the inner wall of the flask. Heat the flask slowly, and when the liquid turns a clear, yellowish green color and no carbonaceous material is visible on the inner wall of the flask, heat it further for 45 minutes. After cooling, carefully add 20 mL of water. Connect the flask to a distillation apparatus previously washed with steam. Add 30 mL of boric acid solution (1 in 25) and 3 drops of bromocresol green-methyl red TS to the receiver J, and add an appropriate amount of water so that the end of the condenser I is covered in the liquid. Add 30 mL of sodium hydroxide solution (2 in 5) into the funnel F, rinse carefully with 10 mL of water, then immediately close the pinch cock of the rubber tube G and distill until 80 mL to 100 mL of distillate is obtained through vapor. Lift the lower end of the condenser I from the liquid surface, rinse the immersed part with a small amount of water, and titrate with 0.025 mol/L sulfuric acid VS. However, the endpoint of the titration is when the color of the solution changes from green through pale grayish blue to pale grayish reddish purple. Perform a blank test in the same manner and make any necessary correction.

Each mL of 0.025 mol/L sulfuric acid VS
= 0.700 mg of N

Packaging and storage Preserve in tight containers.

Corn Oil
옥수수기름

Corn Oil is the fatty oil obtained from the embryo of *Zea mays* Linné (Gramineae).

Description Corn Oil occurs as a clear, pale yellow oil, odorless with a faint odor and a mild taste.
It is miscible with ether or petroleum ether.
It is slightly soluble in ethanol (95) and practically insoluble in water.
At -7 °C, Corn Oil solidifies into an ointment-like mass.

Specific gravity d_{25}^{25}: Between 0.915 and 0.921.

Saponification value Between 187 and 195.

Unsaponifiable matter NMT 1.5%.

Acid Value NMT 0.2.

Iodine Value 103 to 130.

Purity ***Heavy Metals***—Weigh 1.0 g of Corn Oil and perform the test according to Method 2. Prepare the control solution by adding 1.0 mL of the lead standard solution (NMT 10 ppm).

Packaging and storage Preserve in tight containers.

Corn Starch
옥수수전분

Corn Starch is a starch obtained from grains of *Zea mays* Linné (Gramineae).

Description Corn Starch occurs as a white to pale yellow mass or powder.
It is practically insoluble in water or ethanol (95).

Identification (1) Add a mixture of water and glycerin (1 : 1) to Corn Starch and observe the mixture under the microscope; the mixture is mainly irregular polygonal grains of about 2 to 23 μm in diameter, or irregular round or spherical grains of about 25 to 35 μm in diameter. The hilum has a clear cavity or 2 to 5 radial clefts and no concentric fibers. Corn Starch shows a clear black cross crossing at the hilum between crossed polarizing prisms.

(2) To 1 g of Corn Starch add 50 mL of water, boil for 1 minute, and allow to cool; a pale white, turbid, viscous liquid is formed.

(3) To 1 mL of the liquid obtained in (2), add 0.05 mL of diluted iodine TS (1 in 10); an orange to dark bluish purple color develops and the color disappears when heated.

pH Place 5.0 g of Corn Starch in a non-metal vessel, add 25.0 mL of freshly boiled and cooled water, mix gently for 1 minute to make in the form of suspension, and allow it to stand for 15 minutes; the pH of the solution is between 4.0 and 7.0.

Purity (1) ***Iron***—To 1.5 g of Corn Starch, add 15 mL of 2 mol/L hydrochloric acid TS, shake, filter and use the filtrate as the test solution. To 2.0 mL of the iron standard solution, add water to make 20 mL, and use this solution as the control solution. Place 10 mL each of the test solution and the control solution in test tubes, add 2 mL of citric acid (1 in 5) and 0.1 mL of mercaptoacetic acid, and mix. Add strong ammonia water to this solution until the litmus paper clearly indicates alkalinity, and add 20 mL of water to mix. Transfer 10 mL each of these solutions into test tubes, allow them to stand for 5 minutes and compare the color of the solutions against a white background; the color of the test solution is not more intense than that of the control solution (NMT 10 ppm).

(2) ***Oxidizing substances—***To 4.0 g of Corn Starch, add 50.0 mL of water, shake for 5 minutes and centrifuge. To 30.0 mL of the clear supernatant, add 1 mL of acetic acid (100) and 0.5 g to 1.0 g of potassium iodide, shake to mix and allow the mixture to stand for 25 to 30 minutes in a dark place. Add 1 mL of starch TS and titrate with 0.002 mol/L sodium thiosulfate VS until the solution becomes colorless. Perform a blank test in the same manner and make any necessary correction. NMT 1.4 mL of 0.002 mol/L sodium thiosulfate VS is consumed (NMT 20 ppm, calculated as hydrogen peroxide).

(3) ***Sulfur dioxide***—NMT 50 ppm under the test.

(4) ***Foreign matter***—Under the microscope, Corn Starch does not contain starch granules of any other origin. It may contain a minute quantity, if any, of fragments of the tissue of the original plant.

Loss on drying NMT 15.0% (1 g, 130 ℃, 90 minutes).

Residue on ignition NMT 0.6% (1 g).

Microbial limit The total aerobic microbial count is NMT 10^3 CFU/g, and the total combined yeasts/molds count is NMT 10^2 CFU/g. No escherichia coli (E.coli) or salmonella is detected.

Packaging and storage Preserve in well-closed containers.

Creosote
크레오소트

Creosote is a mixture of phenols, obtained from wood tar derived by dry distillation of trunks and branches of the Pinus genus (*Pinaceae*), Cryptomeria genus (*Taxodiaceae*), *Fagus* genus (*Fagaceae*), *Afzelia* genus (*Intsia*) (*Leguminosae*), *Shorea* genus (*Dipterocarpaceae*) or *Tectona* genus (*Verbenaceae*), followed by distillation and collection of oil at 180 ℃ to 230 ℃, then purification and re-distillation. Creosote contains NLT 23% and NMT 35% of guaiacol ($C_7H_8O_2$: 124.14).

Description Creosote occurs as a clear, colorless to pale yellow liquid, and has a characteristic odor.
It is slightly soluble in water. Creosote is miscible with methanol or ethanol.
A saturated solution of Creosote is acidic.
It strongly refracts light.
It gradually changes in color by light or by air.

Identification Use the test solution in the Assay as the test solution. Separately, dissolve 0.1 g each of phenol, *p*-cresol, guaiacol and 2-methoxy-4-methylphenol in methanol to make 100 mL. Dissolve 10 mL each of these solutions in methanol to make 50 mL and use these solutions as the phenol standard solution, the *p*-cresol standard solution, the guaiacol standard solution, and the 2-methoxy-4-methylphenol standard solution, respectively. Perform the test with 10 μL each of the test solution and the standard solutions as directed under the liquid chromatography according to the following conditions; the retention time of the major peak obtained from the test solution is the same as that of the phenol standard solution, *p*-cresol standard solution, guaiacol standard solution, and 2-methoxy-4-methylphenol standard solution.

Operating conditions

Proceed as directed in the operating conditions under the Assay.

Specific gravity d_{20}^{20}: NLT 1.076.

Purity (1) ***Coal creosote*—**Take exactly 10.0 mL of Creosote, dissolve in methanol to make exactly 20 mL, and use this solution as the test solution. Separately, weigh 1 mg each of benzo[α]pyrene, benz[α]anthracene and dibenz[α,h]anthracene. If necessary, dissolve in a small amount of ethyl acetate, add methanol to make 100 mL, and use these solutions as the standard solutions. Perform the test with 1 μL each of the test solution and the standard solutions as directed under the Gas Chromatography according to the following conditions; the peaks having the retention times corresponding to benzo[α]pyrene, benz[α]anthracene, and dibenz[α,h]anthracene in the standard solution are not detected form the test solution. If any peaks having retention times corresponding to benzo[α]pyrene, benz[α]anthracene, and dibenz[α,h]anthracene are detected, change the conditions and analyze again to confirm that these peaks do not belong to benzo[α]pyrene, benz[α]anthracene or dibenz[α,h]anthracene.

Operating conditions

Detector: A high-performance mass spectrometer (electron impact ionization)

Monitored ion:

Benz[α]anthracene: Molecular ion *m*/*z* 228, fragment ion *m*/*z* 114, about 14 to 20 minutes

Benzo[α]pyrene: Molecular ion *m*/*z* 252, fragment ion *m*/*z* 125 about 20 to 25 minutes

Dibenz[α,h]anthracene: Molecular ion *m*/*z* 278, fragment ion *m*/*z* 139, about 25 to 30 minutes.

Sample injection port temperature: A constant temperature around 250 ℃

Column: A quartz column about 0.25 mm in internal diameter and about 30 m in length, coated with 5% diphenyl-95% dimethylpolysiloxane for gas chromatography in 0.25 μm to 0.5 μm in thickness.

Column temperature: Inject the sample at a constant temperature of about 45 °C, raise the temperature to 240 °C at a rate of 40 °C per minute, and maintain at 240 ℃ for 5 minutes. Then, raise to 300 °C at a rate of 4 °C per minute, raise to 320 °C at a rate of 10 °C per minute, and maintain at 320 °C for 3 minutes.

Interface temperature: A constant temperature of about 300 °C.

Carrier gas: Helium

Flow rate: Adjust the flow rate so that the retention time of benzo[α]pyrene is about 22 minutes.

System suitability

Confirmation of detection: Pipet 1 mL of the standard solution, add the methanol to make exactly 10 mL, and use this solution as the system suitability solution. Proceed with 1 μL of this solution according to the above operating conditions; the signal-to-noise ratio of each peak is NLT 3.

System performance: Proceed with 1 μL of the system suitability solution according to the above operating conditions; benz[α]anthracene, benzo[α]pyrene and dibenz[α,h]anthracene are eluted in this order.

System repeatability: Repeat the test 6 times with 1 μL of the system suitability solution under the above operating conditions; the relative standard deviations of the peak areas of benzo[α]pyrene, benz[α]anthracene and dibenz[α,h]anthracene are NMT 10.0%, respectively.

(2) ***Acenaphthene*—**Dissolve 0.12 g of Creosote in methanol to make exactly 50 mL, and use this solution as the test solution. Separately, dissolve 25 mg of acenaphthene in methanol to make 50 mL. Take 5 mL of this solution, and add methanol to make 20 mL. Take 2 mL of this solution again, add methanol to make 100 mL, and use this solution as the standard solution. Perform the test with 1 μL each of the test solution and the standard solution as directed under the Gas Chromatography according to the following conditions; no peak having the retention time corresponding to acenaphthene in the standard solution in detected from the test solution. If any peak is detected for the retention time corresponding to acenaphthene, change these conditions and repeat the analysis to verify that such a peak does not belong to acenaphthene.

Operating conditions

Detector: A flame ionization detector

Column: A fused silica column about 0.25 mm in internal diameter and about 60 m in length, coated with polymethylsiloxane for gas chromatography (0.25 μm to 0.5 μm in thickness).

Column temperature: Inject the sample at a constant temperature of about 45 °C, raise the temperature to 160 °C at a rate of 11.5 °C per minute, and raise to 180 °C at a rate of 4 °C per minute. Then raise to 270 °C at a rate of 8 °C per minute, and maintain at 270 °C for 3 minutes.

Sample injection port temperature: About 250 ℃

Detector temperature: 250 °C

Carrier gas: Helium

Flow rate: Adjust the flow rate so that the retention time of acenaphthene is 18 minutes.

System suitability

Confirmation of detection: Pipet 1 mL of the standard solution, add the methanol to make exactly 10 mL, and use this solution as the system suitability solution. Proceed with 1 μL of this solution according to the above operating conditions; the signal-to-noise ratio of acenaphthene peak is NLT 3.

System repeatability: Repeat the test 6 times with 1 μL of the system suitability solution according to the above conditions; the relative standard deviation of the peak area of acenaphthene is NMT 6.0%.

(3) ***Other related substances***—To 1.0 mL of Creosote, add 2 mL of petroleum benzine, add 2 mL of barium hydroxide TS, shake to mix, and allow to stand; the upper layer does not exhibit a blue or muddy brown color, and the lower layer does not exhibit a red color.

Distilling range Between 200 ℃ and 220 ℃, NLT 85 vol%

Assay Weigh accurately 0.1 g of Creosote, and dissolve in methanol to make exactly 50 mL. Pipet 10 mL of this solution, add methanol to make exactly 50 mL, and use this solution as the test solution. Separately, weigh accurately about 30 mg of the guaiacol RS, dissolve in methanol to make exactly 50 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions, and determine the peak areas, A_T and A_S, of guaiacol in each solution.

$$\text{Amount (mg) of guaiacol } (C_7H_8O_2) = \text{Amount (mg) of the guaiacol RS taken} \times \frac{A_T}{A_S}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 275 nm)

Column: A stainless steel column about 4.6 mm in internal diameter and about 15 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 40°C.

Mobile phase: A mixture of water and acetonitrile (4 : 1)

Flow rate: Adjust the flow rate so that the retention time of guaiacol is 9 minutes.

System suitability

System performance: Dissolve 2 mg each of guaiacol and phenol in methanol to make 10 mL. Proceed with 10 μL of this solution under the above operating conditions; phenol and guaiacol are eluted in this order with the resolution being NLT 2.5.

System repeatability: Repeat the test 6 times with 10 μL of the standard solution according to the above operating conditions; the relative standard deviation of the peak areas of guaiacol is NMT 1.5%

Packaging and storage Preserve in light-resistant, tight containers.

Croscarmellose Sodium
크로스카르멜로오스나트륨

[*74811-65-7*]

Croscarmellose Sodium is a sodium salt of a partly *O*-carboxymethylated and cross-linked cellulose.

Description Croscarmellose Sodium occurs as a white to grayish white powder.
It is practically insoluble in ethanol (99.5) or diethyl ether.
It swells when water is added and becomes a suspension.
It is hygroscopic.

Identification (1) Determine the infrared spectra of Croscarmellose Sodium and croscarmellose sodium RS as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers. However, disregard the absorption around a wavenumber of 1750 cm^{-1} in the spectrum of Croscarmellose Sodium.

(2) Add about 1 g of Croscarmellose Sodium to 100 mL of a methylene blue solution (1 in 250000), stir to mix, and allow the mixture to stand; a blue, fibrous precipitate is formed.

(3) Dissolve 0.1 g of the residue from Residue on ignition in 2 mL of water, add 2 mL of potassium carbonate solution (3 in 20), and heat the mixture to boiling; no precipitate is formed. Add 4 mL of potassium hexahydroxoantimonate(V) TS to this solution and heat to boiling. Then, cool the solution in ice water while scratching the inner wall of the test tube with a glass rod if necessary; a white crystalline precipitate is formed.

pH Add 1.0 g of Croscarmellose Sodium to 100 mL of water, stir to mix for 5 minutes; the pH of the clear supernatant is 5.0 to 7.0.

Purity (1) ***Sodium chloride and sodium glycolate***—The sum of the amount of sodium chloride and sodium glycolate in Croscarmellose Sodium is NMT 0.5%, calculated on the dried basis.

(i) Sodium chloride: Weigh accurately 5 g of Croscarmellose Sodium, add 50 mL of water and 5 mL of hydrogen peroxide (30), and heat the mixture on a water bath for 20 minutes with occasional stirring. Allow this solution to stand to cool down, add 100 mL of water and 10 mL of nitric acid, and titrate with 0.1 mol/L silver nitrate VS (potentiometric titration under the Titrimetry).

Each mL of 0.1 mol/L silver nitrate VS
= 5.844 mg of NaCl

(ii) Sodium glycolate: Weigh accurately 5 g of Croscarmellose Sodium, add 2 mL of acetic acid (100) and 5 mL of water, and stir the mixture for 15 minutes to mix. While stirring this solution, add 50 mL of acetone dropwise, then add 1 g of sodium chloride and stir for 3 minutes. Filter this solution using a filter paper previously moistened with acetone, wash the residue with 30 mL of acetone, combine the filtrate and the washings, add acetone to make exactly 100 mL, and use it as the stock solution of the test solution. Separately, weigh exactly 0.100 g of glycolic acid and add water to make 200 mL. Pipet 0.5 mL, 1 mL, 2 mL, 3 mL, and 4 mL of this solution and add water to make 5 mL, respectively. Then, add 5 mL of acetic acid (100), and then acetone to make 100 mL and use these solutions as the standard stock solution (1), standard stock solution (2), standard stock solution (3), standard stock solution (4), and standard stock solution (5), respectively. Pipet 2 mL of the stock solution of the test solution and the standard stock solutions (1) to (5), heat on a water bath for 20 minutes to evaporate acetone, and allow to cool down. To these solutions, add exactly 5 mL of 2,7-dihydroxynaphthalene TS, stir to mix, and add 15 mL of 2,7-dihydroxynaphthalene

again to mix. Cover the opening of the container containing these solutions with aluminum foil and heat it on a water bath for 20 minutes. After cooling these solutions, add sulfuric acid to make exactly 25 mL, and use these solutions as the test solution, standard solution (1), standard solution (2), standard solution (3), standard solution (4), and standard solution (5). Separately, add 10 mL of a mixture of water and acetic acid (100) (1 : 1) to acetone to make 100 mL. Pipet 2 mL of this solution, proceed in the same manner as the stock solution for the test solution. Use this solution as the blank test solution. For the test solution and the standard solutions (1) to (5), determine the absorbances, A_T, A_{S1}, A_{S2}, A_{S3}, A_{S4}, and A_{S5}, at a wavelength of 540 nm as directed under the Ultraviolet-visible Spectroscopy, using the blank test solution as the blank. Using the calibration curve obtained from the standard solution, determine the amount (g) of glycolic acid, X, in 100 mL of the stock solution for the test solution, and calculate the amount of sodium glycolate according to the following formula.

$$\text{Amount (\%) of sodium glycolate} = (X / M) \times 100 \times 1.289$$

M: Amount (g) of Croscarmellose Sodium taken, calculated on the dried basis

(2) ***Water-soluble substances***—Weigh accurately about 10 g of Croscarmellose Sodium, disperse in 800 mL of water, and stir the mixture for 1 minute every 10 minutes for the first 30 minutes. If settlement is slow, allow the mixture to stand for another hour. Suction filter or centrifuge this solution, and accurately weigh about 150 mL of the filtrate or clear supernatant. Heat and concentrate this solution to the point where it does not solidify, dry at 105°C for 4 hours, and accurately weigh the mass of the residue. Determine the amount of water-soluble substances according to the following formula; it is NMT 10.0%.

$$\text{Amount (\%) of water-soluble substances} = 100M_3 \times (800 + M_1) / (M_1 \times M_2)$$

M_1: Amount (g) of Croscarmellose Sodium taken, calculated on the dried basis
M_2: Amount (g) of 150 mL of the filtrate or the supernatant
M_3: Amount (g) of the residue

Setting volume Add 75 mL of water to a 100-mL stoppered measuring cylinder and add 0.5 g of Croscarmellose Sodium at a time to a total of 1.5 g while shaking vigorously. Add water to make 100 mL, shake well until homogeneously dispersed, and allow the mixture to stand for 4 hours; the volume of the sediment is between 10.0 mL and 30.0 mL.

Degree of substitution Weigh accurately about 1 g of Croscarmellose Sodium into a 500-mL stoppered Erlenmeyer flask, add 300 mL of sodium chloride TS, and add exactly 25.0 mL of 0.1 mol/L sodium hydroxide solution, and stopper the flask. Allow the mixture to stand for 5 minutes with occasional shaking. Add 5 drops of metacresol purple TS, add 15 mL of 0.1 mol/L hydrochloric acid, stopper the flask and mix. If the solution exhibits a purple color, pipet 1 mL of 0.1 mol/L hydrochloric acid at a time and add it while shaking each time until the solution turns yellow. Titrate this solution with 0.1 mol/L sodium hydroxide VS. The endpoint of the titration is when the yellow color of the solution changes to violet. Perform a blank test in the same manner. Determine the degree of substitution A of the acid/carboxymethyl group and the degree of substitution S of the sodium/carboxymethyl group; the sum of A and S is 0.60 to 0.85.

$$A = 1150M / (7102 - 412M - 80C)$$

$$S = (162 + 58A) C / (7102 – 80C)$$

M: Amount (mmol) of sodium hydroxide VS required to neutralize 1 g of Croscarmellose Sodium calculated on the dried basis.

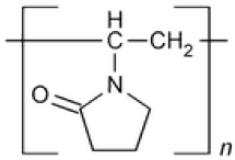
C: Result (%) obtained from Residue on ignition

Loss on drying NMT 10.0% (1 g, 105 °C, 6 hours).

Residue on ignition 14.0% to 28.0% (1 g, calculated on the dried basis).

Packaging and storage Preserve in tight containers.

Crospovidone
크로스포비돈



$(C_6H_9N_2O)_n$

1-Ethenyl-2-pyrrolidinone homopolymer;
1-Vinyl-2-pyrrolidinone homopolymer [*9003-39-8*]

Crospovidone is a cross-linked polymer of 1-vinyl-2-pyrrolidone.

Crospovidone contains NLT 11.0% and NMT 12.8% of nitrogen (N: 14.01), calculated on the dried basis.

Two types of Crospovidone are available, depending on the particle size: Type A and Type B

The label of Crospovidone states the type.

Description Crospovidone occurs as a white to pale yellow powder.
It is practically insoluble in water, methanol, or ethanol (99.5).
It is hygroscopic.

Identification (1) Suspend 1 g of Crospovidone in 10 mL of water, add 0.1 mL of iodine TS, and mix for 30 seconds. Add 1 mL of starch TS and shake to mix; the resulting solution does not exhibit a blue color within 30 seconds.

(2) Add 0.1 g of Crospovidone to 10 mL of water, and shake to mix; a suspension is formed. Allow it to stand; no clear liquid is formed within 15 minutes.

(3) Determine the infrared spectrum of Crospovidone and crospovidone RS, previously dried for 1 hour at 105 °C, as directed in the potassium bromide disk method under the Ultraviolet-visible Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

Particle size distribution Weigh accurately about 20 g of Crospovidone, place it in a 1000-mL Erlenmeyer flask, and add 500 mL of water. After shaking for 30 minutes, pour the mixture into a No. 235 (63 μm) sieve, previously washed with hot water, dried overnight at 105 °C and accurately weighed, and rinse the sieve

with water until the liquid passing through becomes clear. Place the sieve with the residue in an oven, dry at 105°C for 5 hours without circulating air, cool in a desiccator for 30 minutes , and weigh the mass. Determine percentage sieving residue fraction of Crospovidone on the No. 235 (63 μm) sieve according to the following formula; type A exceeds 15% and type B is NMT 15%.

$$\text{Amount (\%) of the residue of Crospovidone on No. 235 (63 μm) sieve} = \{(M_1 - M_3) / M_2\} \times 100$$

M_1: Amount (g) of the residue of Crospovidone on the sieve after 5 hours of drying

M_2: Amount (g) of Crospovidone taken, calculated on the dried basis

M_3: Mass (g) of the sieve

Purity (1) ***Water soluble substances*—**Weigh 25.0 g of Crospovidone, add 200 mL of water, and stir for 1 hour. Add water to the resulting suspension to make exactly 250 mL. After allowing the solids to settle, filter about 100 mL of the clear supernatant by superimposing a membrane filter with a pore diameter of 0.45 μm on top of a membrane filter with a pore diameter of 3 μm. Pipet 50 mL of the clear filtrate, evaporate to dryness, and dry the residue at 105 to 110 ℃ for 3 hours; the amount of the residue is NMT 75 mg.

(2) ***1-Vinyl-2-pyrrolidone***—Add exactly 50 mL of methanol to 1.250 g of Crospovidone, shake for 60 minutes to mix, allow the mixture to stand so that solids settle, then filter through a membrane filter with a pore size of 0.2 μm and use the filtrate as the test solution. Separately, weigh 50 mg of 1-vinyl-2-pyrrolidone and dissolve in methanol to make exactly 100 mL. Pipet 1 mL of this solution and add methanol to make exactly 100 mL. Weigh accurately 5 mL of this solution, add the mobile phase to make exactly 100 mL, and use the resulting solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions; the peak area of 1-vinyl-2-pyrrolidone in the test solution is not greater than that of 1-vinyl-2-pyrrolidone in the standard solution (NMT 10 ppm).

Operating conditions

Detector: An ultraviolet absorption spectrophotometer (wavelength: 235 nm)

Column: A stainless steel column about 4 mm in internal diameter and about 250 mm in length, packed with octylsilanized silica gel for liquid chromatography (5 μm in particle diameter). Use a guard column made of a stainless steel column about 4 mm in internal diameter and about 25 mm in length, packed with octylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 40 ℃

Mobile phase: A mixture of water and acetonitrile (9 : 1)

Flow rate: 1.0 mL/min

Guard column washing: After performing the test with the test solution, wash the guard column by allowing the mobile phase to flow through at the above flow rate in the opposite direction to the test operation for approximately 30 minutes.

System suitability

System performance: Dissolve 10 mg of 1-vinyl-2-pyrrolidone and 0.5 g of vinyl acetate in 100 mL of methanol. Pipet 1 mL of this solution, and add the mobile phase to make 100 mL. Proceed with 50 μL of this solution under the above operating conditions; 1-vinyl-2-pyrrolidone and vinyl acetate are eluted in this order with the resolution being NLT 2.0.

System repeatability: Repeat the test 6 times with 50 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak area of 1-vinyl-2-pyrrolidone is NMT 2.0%.

(3) ***Peroxides Method 1***—This method applies to Type A of Crospovidone. Suspend 4.0 g of Crospovidone in 100 mL of water to make a suspension. Take 25 mL of the suspension, add 2 mL of titanium(III) chloride-sulfuric acid TS, allow the mixture to stand for 30 minutes, filter, and use the filtrate as the test solution. Separately, filter the suspension and add 2 mL of dilute sulfuric acid (13 in 100) to 25 mL of the filtrate. Perform the test with the test solution as directed under the Ultraviolet-visible Spectroscopy, using the mixture as the blank; the absorbance of the test solution at the wavelength of 405 nm is NMT 0.35 (NMT 400 ppm as hydrogen peroxide),

Method 2—This method applies to Type B of Crospovidone. Suspend 2.0 g of Crospovidone in 50 mL of water to make a suspension. Take 10 mL of the suspension and add water to make 25 mL. Add 2 mL of titanium(III) chloride-sulfuric acid TS, allow the mixture to stand for 30 minutes, filter, and use the filtrate as the test solution. Separately, filter the suspension, add water to 10 mL of the suspension to make 25 mL, and add 2 mL of dilute sulfuric acid (13 in 100). Perform the test with the test solution as directed under the Ultraviolet-visible Spectroscopy, using the mixture as the blank; the absorbance of the test solution at the wavelength of 405 nm is NMT 0.35 (NMT 1000 ppm as hydrogen peroxide),

Loss on drying NMT 5.0% (0.5 g, 105 ℃, constant mass).

Residue on ignition NMT 0.1% (1.0 g)

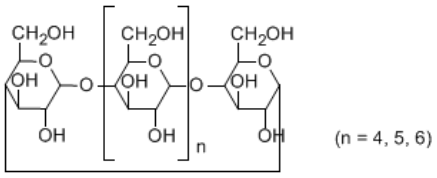
Assay Weigh accurately about 0.1 g of Crospovidone, place it in a Kjeldahl flask, and add 5 g of reaction catalyst (a powdered mixture of 33 g of potassium sulfate, 1 g of copper(II) sulfate pentahydrate, and 1 g of titanium(IV) oxide) and 3 glass beads. Rinse down any adhering material on the wall of the flask with a small amount of water and add 7 mL of sulfuric acid along the inner wall of the flask. Heat the flask slowly until the solution becomes yellowish green and transparent and the inner wall of the flask is free of carbonaceous material, and then heat for an additional 45 minutes. After cooling, carefully add 20 mL of water. Connect the flask to a distillation apparatus previously washed with steam. Add 30 mL of boric acid solution (1 in 25) and 3 drops of bromocresol green-methyl red TS to the receiver, and add an appropriate amount of water so that the lower end of the condenser is submerged in the liquid. Add 30 mL of sodium hydroxide solution (21 in 50) into the funnel, rinse carefully with 10 mL of water, then immediately close the pinch cock of the rubber tube and distill until 80 mL to 100 mL of distillate is collected through vapor. Remove the lower end of the condenser from the liquid surface, wash with a small amount of water, and titrate with 0.025 mol/L sulfuric acid VS. The endpoint of the titration is when the color of the solution changes from green through blue to reddish purple. Perform a blank test in the same manner and make any necessary correction.

$$\text{Each mL of 0.025 mol/L sulfuric acid VS} = 0.7003 \text{ mg of N}$$

Packaging and storage Preserve in tight containers.

Cyclodextrin
시클로덱스트린

Cyclodextrin consists of α-cyclodextrin, β--cyclodextrin, and γ-cyclodextrin. It is obtained by treating cyclodextrin synthase to starch, resulting in circular oligosaccharides where 6, 7, and 8 glucose units are linked by α-1,4 glycosidic bonds. They are respectively referred to as α-cyclodextrin, β-cyclodextrin, and γ-cyclodextrin.



α-Cyclodextrin $(C_6H_{10}O_5)_6$ Molecular weight 972.85
β-Cyclodextrin $(C_6H_{10}O_5)_7$ Molecular weight 1134.99
γ-Cyclodextrin $(C_6H_{10}O_5)_8$ Molecular weight 1297.14

Cyclodextrin, when dried, contains 98.0% to 101.0% of α-cyclodextrin $[(C_6H_{10}O_5)_6]$, 98.0% to 101.0% of β-cyclodextrin $[(C_6H_{10}O_5)_7]$, and 98.0% to 101.0% of γ-cyclodextrin $[(C_6H_{10}O_5)_8]$.

Description Cyclodextrin occurs as a white crystal or crystalline powder.
It is odorless and has a slightly sweet taste.

Identification To 0.2 g of Cyclodextrin, add 1 mL of 0.1 mol/L iodine TS, heat on a water bath to dissolve, and allow to stand at room temperature; α-cyclodextrin, β-cyclodextrin, and γ-cyclodextrin form bluish purple, yellowish brown, and reddish brown precipitate, respectively.

Purity (1) ***Clarity and color of solution***—Dissolve 0.5 mg of Cyclodextrin in 50 mL of water; the resulting solution is clear and colorless.

(2) ***Specific optical rotation***

α-Cyclodextrin $[\alpha]_D^{20}$ = +147.0° to +152.0°
β-Cyclodextrin $[\alpha]_D^{20}$ = +160.0° to +164.4°
γ-Cyclodextrin $[\alpha]_D^{20}$ = +173.0° to +178.0°
(1 g after drying, 100 mL of water, 100 mm)

(3) ***Chloride***—Perform the test with about 0.5 g of Cyclodextrin. Prepare the control solution with 0.25 mL of 0.01 mol/L hydrochloric acid VS (NMT 0.018%).

(4) ***Lead***—Transfer 5.0 g of Cyclodextrin into a crucible, heat weakly to carbonize, and then ignite at 450 ℃ to 550 ℃. After cooling, moisten the residue with a small amount of water, add 4 mL of hydrochloric acid to evaporate to dryness, then add 0.5 mol/L nitric acid, and warm to dissolve. Filter through a filter paper if there are insoluble, then add 0.5 mol/L nitric acid to make exactly 25 mL, and use this solution as the test solution. Separately, transfer 0.5 mL of lead standard solution into a crucible, proceed in the same manner as in the preparation of the test solution, and use this solution as the standard solution. Perform the test with the blank test solution, the test solution, and the standard solution as directed under the Atomic Absorption Spectroscopy according to the following conditions, using 0.5 mol/L nitric acid as the blank test solution; the absorbance of the test solution is NMT the absorbance of the standard solution (NMT 1.0 ppm).

Gas: Dissolved acetylene – Air
Lamp: A lead hollow cathode lamp
Wavelength: 283.3 nm

(5) ***Arsenic***—Prepare the test solution with 2.0 g of Cyclodextrin according to Method 2 and perform the test (NMT 1 ppm).

(6) ***Residual solvents***—Perform the test with 0.25 g of Cyclodextrin as directed under the Residual Solvents; the amount of toluene and 1,1,2-trichloroethene is NMT 1.0 ppm, respectively.

Loss on drying NMT 12% (NMT 5 mm Hg in vacuum, 105 ℃, 4 h).

Residue on ignition NMT 0.1% (1 g).

Assay Weigh accurately about 0.1 g of Cyclodextrin, add water to make exactly 10 mL, and use this solution as the test solution. Separately, weigh accurately 0.1 g each of α-cyclodextrin RS, β-cyclodextrin RS, and γ-cyclodextrin RS, previously dried, add water to make exactly 10 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas, A_T and A_S, of cyclodextrin in each solution.

$$\text{Amount (mg) of cyclodextrin} = \text{Amount (mg) of cyclodextrin RS} \times \frac{A_T}{A_S}$$

Operating conditions

Detector: A differential refractometer
Column: A stainless steel column about 7.8 mm in internal diameter and about 30 cm in length, packed with styrene-divinylbenzene copolymer sulfonic acid resin for liquid chromatography (25 μm in particle diameter).
Column temperature: A constant temperature of about 60 ℃.
Mobile phase: Water
Flow rate: 0.6 mL/min to 1.0 mL/min

Cyclodextrin Syrup
시클로덱스트린 시럽

Cyclodextrin Syrup is a purified and concentrated starch hydrolysate of an aqueous solution containing cyclodextrin, obtained by treating cyclodextrin synthase to the starch solution. It contains α-cyclodextrin, β-cyclodextrin, γ-cyclodextrin, and various sugars like glucose and maltose, where 6, 7, and 8 glucose units are linked in a ring by α-1,4 glycosidic bonds. However, it includes dried cyclodextrin syrup. Cyclodextrin Syrup, when dried, contains NLT the labeled amount of cyclodextrin.

Description Cyclodextrin Syrup occurs as a colorless, clear, viscous liquid or white powder. It is odorless and has a sweet taste. It precipitates a crystal in a cold place and sometimes turns cloudy in white color.

Identification (1) To 0.5 g of Cyclodextrin Syrup, add 1 mL of 0.1 mol/L iodine TS, heat on a water bath to dissolve, and then allow to stand at room temperature; a yellowish brown precipitate forms.

(2) To 0.5 g of Cyclodextrin Syrup, add 3 mL of water, heat

on a water bath to dissolve, add 1 mL of 1,1,2-trichloroethene, and stir vigorously; a white cloudy precipitate forms.

Purity (1) ***Clarity and color of solution***—To 2 g of Cyclodextrin Syrup, add 50 mL of water, and warm at 50 ℃ to dissolve; the resulting solution is clear and colorless.

(2) ***Chloride***—Perform the test with 0.5 g of Cyclodextrin Syrup. Prepare the control solution with 0.4 mL of 0.01 mol/L hydrochloric acid (NMT 0.028%).

(3) ***Heavy metals***—Proceed with 5.0 g of Cyclodextrin Syrup according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 4 ppm).

(4) ***Arsenic***—Prepare the test solution with 2.0 g of Cyclodextrin Syrup according to Method 3 and perform the test (NMT 1 ppm).

Loss on drying NMT 25% (in vacuum, NMT 5 mm Hg, 105 ℃, 4 h).

Residue on ignition NMT 0.05% (1 g).

Assay Weigh accurately an amount of Cyclodextrin Syrup, equivalent to 0.5 g of cyclodextrin according to the labeled amount, add water to make 50 mL, then take 20 mL of this solution, and heat on a water bath for 10 minutes. After cooling, add 2 mL of glucoamylase solution (10 IU/mL), and allow to react at 40 ℃ on a water bath for 1 hour. Heat on a water bath for 10 minutes, filter, then cool at room temperature, and add water to make exactly 25 mL. Use this solution as the test solution. Separately, weigh accurately 0.1 g each of α-cyclodextrin RS, β-cyclodextrin RS, and γ-cyclodextrin RS, previously dried, add water to make exactly 20 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas of α-cyclodextrin, β-cyclodextrin, and γ-cyclodextrin. Determine the content of cyclodextrin as the sum of the contents of α-cyclodextrin, β-cyclodextrin, and γ-cyclodextrin.

$$\text{Content (\%) of cyclodextrin (CD)} = \text{Content (\%) of } \alpha\text{-CD} + \beta\text{-CD} + \gamma\text{-CD}$$

$$\text{Content (\%) of α-CD} = \frac{Concentration\ (ppm)\ of\ the\ \alpha-CD\ standard\ solution \times 50 \times 25}{Amount\ (g)\ of\ sample\ taken \times 20} \times \frac{\alpha-CD\ peak\ area\ of\ the\ test\ solution}{\alpha-CD\ peak\ area\ of\ the\ mixed\ standard\ solution} \times \frac{100}{10^6}$$

$$\text{Content (\%) of β-CD} = \frac{Concentration\ (ppm)\ of\ the\ \beta-CD\ standard\ solution \times 50 \times 25}{Amount\ (g)\ of\ sample\ taken \times 20} \times \frac{\beta-CD\ peak\ area\ of\ the\ test\ solution}{\beta-CD\ peak\ area\ of\ the\ mixed\ standard\ solution} \times \frac{100}{10^6}$$

$$\text{Content (\%) of γ-CD} = \frac{Concentration\ (ppm)\ of\ the\ \gamma-CD\ standard\ solution \times 50 \times 25}{Amount\ (g)\ of\ sample\ taken \times 20} \times \frac{\gamma-CD\ peak\ area\ of\ the\ test\ solution}{\gamma-CD\ peak\ area\ of\ the\ mixed\ standard\ solution} \times \frac{100}{10^6}$$

Operating conditions

Detector: A differential refractometer

Column: A stainless steel column about 7.8 mm in internal diameter and about 30 cm in length, packed with styrene-divinylbenzene copolymer sulfonic acid resin for liquid chromatography (25 μm in particle diameter).

Column temperature: A constant temperature of about 80 ℃.

Mobile phase: Water

Flow rate: 0.6 mL/min to 1.0 mL/min

Dextrin
덱스트린

Description Dextrin occurs as a white to pale yellow amorphous powder or grain. It has a characteristic odor and sweet taste and has no irritation when placed on the tongue.
It is freely soluble in hot water, soluble in water, and practically insoluble in ethanol or ether.

Identification To 0.1 g of Dextrin, add 100 mL of water, shake to mix, filter if necessary, and add 1 drop of iodine TS into 5 mL of the filtrate; the resulting solution exhibits a pale reddish brown or pale purple color.

Purity (1) ***Clarity and color of solution***—Transfer 2.0 g of Dextrin into a Nessler tube, add 40 mL of water, heat to dissolve, and cool. Then, add water to make 50 mL; the resulting solution is colorless to pale yellow. It may be clear or often turbid, but its turbidity is not more intense than the following control solution.

Control solution—To 1.0 mL of 0.005 mol/L sulfuric acid, add 1 mL of dilute hydrochloric acid, 46 mL of water, and 2 mL of barium chloride TS, allow to stand for 10 minutes, and shake to mix.

(2) ***Acid***—Take 1.0 g of Dextrin, add 5 mL of water, dissolve by heating, cool and add 1 drop of phenolphthalein TS and 0.50 mL of 0.1 mol/L sodium hydroxide TS; the solution exhibits a red color.

(3) ***Chloride***—To 2.0 g of Dextrin, add 80 mL of water, and heat to dissolve. After cooling, add water to make 100 mL, and filter. To 40 mL of the filtrate, add 6 mL of dilute nitric acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution with 0.30 mL of 0.01 mol/L hydrochloric acid (NMT 0.013%).

(4) ***Sulfate***—To 45 mL of the filtrate obtained in (3), add 1 mL of dilute hydrochloric acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution with 0.35 mL of 0.005 mol/L sulfuric acid (NMT 0.019%).

(5) ***Oxalate***—To 1.0 g of Dextrin, add 20 mL of water, heat to dissolve, cool, and then add 1 mL of acetic acid to filter. To 5 mL of the filtrate, add 5 drops of calcium chloride TS; the resulting solution does not immediately become turbid.

(6) ***Reducing sugars***—To 2.0 g of Dextrin, add 100 mL of water, mix for 30 minutes, add water to make 200 mL, and filter. Use the filtrate as the test solution (A). To 10 mL of Fehling's TS, add 20 mL of the test solution, shake to mix, heat until the solution can boil within 3 minutes, and cool after heating for 2 minutes. Add 5 mL of potassium iodide solution (3 in 10) and 10 mL of 2 mol/L sulfuric acid, mix, and titrate with 0.1 mol/L sodium thiosulfate VS (indicator: starch). Separately, proceed with 20 mL of anhydrous dextrose solution (1 in 1000) as the test solution (B) in the same manner and perform a blank test; it meets the following requirements (corresponds to 10% dextrose).

$$(V_B - Vu) \leqq (V_B - Vs)$$

V_B: Volume (mL) of 0.1 mol/L sodium thiosulfate VS

consumed in the blank test.

Vu: Volume (mL) of 0.1 mol/L sodium thiosulfate VS consumed when tested with the test solution (A).

Vs: Volume (mL) of 0.1 mol/L sodium thiosulfate VS consumed when tested with the test solution (B).

(7) ***Calcium***—To 5 mL of the filtrate from (5), add 5 drops of ammonium oxalate TS; the resulting solution does not immediately become turbid.

(8) ***Heavy metals***—Proceed with 1.0 g of Dextrin according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 20 ppm).

(9) ***Lead***—Weigh accurately 5.0 g of Dextrin, transfer to a platinum crucible, dry, carbonize, and then incinerate at 450 °C to 550 °C. If incineration is not complete, cool and moisten with 2 to 5 mL of nitric acid (1 in 2), 50% magnesium nitrate solution or aluminum nitrate-calcium nitrate solution (prepared by dissolving 40 g of aluminum nitrate and 20 g of calcium nitrate in 100 mL of water) as an incineration supplement, dry, and continue with incineration. If incineration is incomplete, repeat the above procedure once and then add a final 2 mL to 5 mL of nitric acid (1 in 2), if necessary, and incinerate. After incineration, moisten the residue with water, add 2 to 4 mL of hydrochloric acid, and evaporate to dryness. Add 0.5 mol/L nitric acid, dissolve by warming, and filter with filter paper if any insoluble matter exists. Unless otherwise specified, add 0.5 mol/L nitric acid to make 25 mL and use this solution as the test solution. Separately, transfer 0.5 mL of lead standard solution into a platinum crucible, proceed in the same manner as in the preparation of the test solution, and use this solution as the standard solution. Perform the test with the test solution and the standard solutions as directed under the Atomic Absorption Spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 1.0 ppm).

Gas: Acetylene or Hydrogen - Air
Lamp: lead hollow-cathode lamp
Wavelength: 283.3 nm

Loss on drying NMT 10% (0.5 g, 105 ℃, 4 hours).

Residue on ignition NMT 0.5% (0.5 g).

Packaging and storage Preserve in well-closed containers.

Diacetylated Monoglycerides
디아세틸레이티드모노글리세리드

Diacetylated Monoglycerides is glycerin estrified with edible fatty acids and acetic acid. Depending on the molecular weights, it may be prepared by interesterification of edible oils with triacetin in the presence of catalytic agents or by the direct acetylation of edible monoglycerides with acetic acid (100) without the use of catalyst.

Description Diacetylated Monoglycerides is transparent liquids.
IT is very soluble in 80%(w/w) ethanol, vegetable oil, and mineral oil and sparingly soluble in 70%(w/w) ethanol.

Identification Determine the infrared spectra of Diacetylated Monoglycerides and diacetylated monoglycerides RS as directed in the Liquid film method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

Saponification value Between 365 and 395.

Acid value NMT 3.

Hydroxyl value NMT 15.

Purity ***Heavy metals***—Proceed with 1.0 g of Diacetylated Monoglycerides and perform the test according to Method 2. Prepare the control solution with 1.0 mL of lead standard solution (NMT 10 ppm).

Residue on ignition NMT 0.1% (1.0 g)

Packaging and storage Preserve in tight containers.

Anhydrous Dibasic Calcium Phosphate
무수인산수소칼슘

$CaHPO_4$: 136.06

[*7757-93-9*]

Anhydrous Dibasic Calcium Phosphate contains NLT 97.5% and NMT 102.5% of dibasic calcium phosphate ($CaHPO_4$).

Description Anhydrous Dibasic Calcium Phosphate occurs as a white, crystalline powder or grain.
It is practically insoluble in water or ethanol (99.5).
It dissolves in dilute hydrochloric acid or dilute nitric acid.

Identification (1) Dissolve 0.1 g of Anhydrous Dibasic Calcium Phosphate in 10 mL of 2 mol/L hydrochloric acid TS by warming, add 2.5 mL of ammonia TS while shaking to mix, and add 5 mL of ammonium oxalate TS; a white precipitate is formed.

(2) Dissolve 0.1 g of Anhydrous Dibasic Calcium Phosphate in 5 mL of dilute nitric acid, warm at 70 ℃ for 1 to 2 minutes, and add 2 mL of ammonium molybdate TS; a yellow precipitate is formed.

Purity (1) ***Acid-insoluble substance***—Add 40 mL of water and 10 mL of hydrochloric acid to 5.0 g of Anhydrous Dibasic Calcium Phosphate, boil for 5 minutes, and cool. Collect the insoluble substance by filtration using a filter paper for qualitative analysis, wash the residue with water until no more turbidity of the washing is produced by adding silver nitrate TS, and ignite to incinerate the residue and the filter paper at 600 ± 50 °C; the amount is NMT 10 mg (NMT 0.2%).

(2) ***Chloride***—Dissolve 0.20 g of Anhydrous Dibasic Calcium Phosphate Hydrate in 20 mL of water and 13 mL of dilute nitric acid, add water to make 100 mL, and filter if necessary. Perform the test with 50 mL of this solution as the test solution. Separately, take 0.70 mL of 0.01 mol/L hydrochloric acid, add 6 mL of dilute nitric acid and water to make 50 mL, and use this solution as the control solution. Add 1 mL of silver nitrate TS each to the test solution and the control solution, mix well, and allow to stand for 5 minutes protected from light. Compare the turbidity of the test solution and the control solution against a black background; the test solution is not more intense than the control solution (NMT 0.25%).

(3) ***Sulfate***—Dissolve 0.5 g of Anhydrous Dibasic Calcium Phosphate in 5 mL of water and 5 mL of dilute hydrochloric acid

by warming, add water to make 100 mL, and filter if necessary To 20 mL of the filtrate, add 1 mL of dilute hydrochloric acid and water to make 50 mL, and use this solution as the test solution. Separately, take 1.0 mL of 0.005 mol/L sulfuric acid, and add 1 mL of dilute hydrochloric acid and water to make 50 mL, and use this solution as the control solution. Add 2 mL of barium chloride TS each to the test solution and the control solution, mix well, and allow to stand for 10 minutes. Compare the turbidity of the test solution and the control solution against a black background; the test solution is not more intense than the control solution (NMT 0.5%).

(4) ***Carbonate***—To 1.0 g of Anhydrous Dibasic Calcium Phosphate, add 5 mL of water, shake to mix, and immediately add 2 mL of hydrochloric acid; no foam is produced.

(5) ***Barium***—Dissolve 0.5 g of Anhydrous Dibasic Calcium Phosphate in 10 mL of water by adding 1 mL of hydrochloric acid dropwise while heating and stirring to mix, filter if necessary, add 2 mL of potassium sulfate TS, and allow to stand for 10 minutes; no turbidity is produced.

(6) ***Arsenic***—Dissolve 1.0 g of Anhydrous Dibasic Calcium Phosphate in 5 mL of dilute hydrochloric acid, and perform the test using this solution as the test solution (NMT 2 ppm).

(7) ***Fluoride***—Place 1 g of Anhydrous Dibasic Calcium Phosphate in a beaker and dissolve with 10 mL of hydrochloric acid (1 in 10). Heat this solution, boil for 1 minute, then transfer it into a polyethylene beaker, and cool immediately. Add 15 mL of sodium citrate solution (1 in 4) and 10 mL of ethylenediaminetetraacetic acid disodium salt solution (1 in 40), and shake to mix. Adjust the pH to between 5.4 and 5.6 with hydrochloric acid (1 in 10) or sodium hydroxide solution (2 in 5), add water to make 100 mL, and use this solution as the test solution. Put 50 mL of the test solution in a polyethylene beaker, measure the potential using a fluoride electrode, and determine the amount of fluorine from the calibration curve; the amount should be NMT 50 ppm.

Creation of calibration curve: Weigh accurately 2.210 g of sodium fluoride, previously dried at 200 °C for 4 hours, put it in a polyethylene beaker, and dissolve with 200 mL of water. Then add water to make 1000 mL and store in a polyethylene container. Take exactly 5 mL of this solution, place it in a mess flask, and add water to make 1000 mL (each mL of this solution contains 5 μg of fluorine). Take 1 mL, 2 mL, 3 mL, 5 mL, 10 mL and 15 mL of this solution, place each in a polyethylene beaker, add 15 mL of sodium citrate solution (1 in 4) and 10 mL of ethylenediaminetetrisodium (1 in 40) to each of the beakers, and mix. Adjust the pH to between 5.4 and 5.6 with hydrochloric acid (1 in 10) or sodium hydroxide solution (2 in 5), then add water to make 100 mL, and use these solutions as the standard solutions. Take 50 mL from each standard solution, place it in a polyethylene beaker, measure the potential with a fluoride electrode, and plot a calibration curve using the log values of the fluorine concentrations.

Loss on ignition 6.6% to 8.7% (1 g, 800 to 825 °C, constant mass)

Assay Weigh accurately about 0.4 g of Anhydrous Dibasic Calcium Phosphate, dissolve in 12 mL of dilute hydrochloric acid by heating on a water bath if necessary, and add water to make exactly 200 mL. Take exactly 20 mL of this solution, add exactly 25 mL of 0.02 mol/L ethylenediaminetetraacetic acid disodium salt solution, add 50 mL of water and 5 mL of ammonia-ammonium chloride buffer solution, pH 10.7, and titrate the excess ethylenediaminetetraacetic acid disodium salt with 0.02 moL/L zinc sulfate VS. (indicator: 25 mg of eryochrome black T-sodium chloride indicator). Perform a blank test in the same manner.

Each mL of 0.02 mol/L ethylenediaminetetraacetic acid disodium salt VS
= 2.721 mg of $CaHPO_4$

Packaging and storage Preserve in well-closed containers.

Dibasic Calcium Phosphate Hydrate
인산수소칼슘수화물

Dibasic calcium phosphate $CaHPO_4 \cdot 2H_2O$: 172.09
[*7789-77-7*]

Dibasic Calcium Phosphate Hydrate contains NLT 98.0% and NMT 105.0% of dibasic calcium phosphate hydrate ($CaHPO_4 \cdot 2H_2O$).

Description Dibasic Calcium Phosphate Hydrate occurs as a white crystalline powder.
It is practically insoluble in water or ethanol (99.5).
It dissolves in dilute hydrochloric acid or dilute nitric acid.

Identification (1) Dissolve 0.1 g of Dibasic Calcium Phosphate Hydrate in 10 mL of 2 mol/L hydrochloric acid TS by warming, add 2.5 mL of ammonia TS dropwise while shaking to mix, and add 5 mL of ammonium oxalate TS; a white precipitate is formed.

(2) Dissolve 0.1 g of Dibasic Calcium Phosphate Hydrate in 5 mL of dilute nitric acid, warm at 70 °C for 1 to 2 minutes, and add 2 mL of ammonium molybdate TS; a yellow precipitate is formed.

Purity (1) ***Acid-insoluble substance***—Add 40 mL of water and 10 mL of hydrochloric acid to 5.0 g of Dibasic Calcium Phosphate Hydrate, boil for 5 minutes, and cool. Collect the insoluble substance by filtration using a filter paper for qualitative analysis, wash the residue with water until no more turbidity of the washing is produced by adding silver nitrate TS, and ignite to incinerate the residue and the filter paper at 600 ± 50; the amount is NMT 10 mg (NMT 0.2%).

(2) ***Chloride***—Dissolve 0.20 g of Dibasic Calcium Phosphate Hydrate in 20 mL of water and 13 mL of dilute nitric acid, add water to make 100 mL; and filter if necessary. Perform the test with 50 mL of this solution as the test solution. Separately, take 0.70 mL of 0.01 mol/L hydrochloric acid, add 6 mL of dilute nitric acid and water to make 50 mL, and use this solution as the control solution. Add 1 mL of silver nitrate TS each to the test solution and the control solution, mix well, and allow to stand for 5 minutes protected from light. Compare the turbidity of the test solution and the control solution against a black background; the test solution is not more intense than the control solution (NMT 0.25%).

(3) ***Sulfate***—Dissolve 0.5 g of Dibasic Calcium Phosphate Hydrate in 5 mL of water and 5 mL of dilute hydrochloric acid by warming, add water to make 100 mL, and filter if necessary. To 20 mL of the filtrate, add 1 mL of dilute hydrochloric acid and water to make 50 mL, and use this solution as the test solution. Separately, take 1.0 mL of 0.005 mol/L sulfuric acid, and add 1 mL of dilute hydrochloric acid and water to make 50 mL, and use this solution as the control solution. Add 2 mL of barium chloride TS each to the test solution and the control solution, mix well, and allow to stand for 10 minutes. Compare the turbidity of the

test solution and the control solution against a black background; the test solution is not more intense than the control solution (NMT 0.5%).

(4) ***Carbonate*—**To 1.0 g of Dibasic Calcium Phosphate Hydrate, add 5 mL of water, shake to mix, and immediately add 2 mL of hydrochloric acid; no foam is produced.

(5) ***Barium*—**Dissolve 0.5 g of Dibasic Calcium Phosphate Hydrate in 10 mL of water by adding 1 mL of hydrochloric acid dropwise while heating and stirring to mix, filter if necessary, add 2 mL of potassium sulfate TS, and allow to stand for 10 minutes; no turbidity is produced.

(6) ***Arsenic*—**Dissolve 1.0 g of Dibasic Calcium Phosphate Hydrate in 5 mL of dilute hydrochloric acid, and perform the test with this solution as the test solution (NMT 2 ppm).

(7) ***Fluoride*—**Place 1 g of Dibasic Calcium Phosphate Hydrate in a beaker, and dissolve with 10 mL of hydrochloric acid (1 in 10). Heat this solution, boil for 1 minute, then transfer to a polyethylene beaker, and cool immediately. Add 15 mL of sodium citrate solution (1 in 4) and 10 mL of ethylenediaminetetraacetic acid disodium salt solution (1 in 40), and shake to mix. Adjust the pH to between 5.4 and 5.6 with hydrochloric acid (1 in 10) or sodium hydroxide solution (2 in 5), add water to make 100 mL, and use this solution as the test solution. Put 50 mL of the test solution in a polyethylene beaker, measure the potential using a fluoride electrode, and determine the amount of fluorine from the calibration curve; the amount should be NMT 50 ppm.

Creation of calibration curve: Weigh accurately 2.210 g of sodium fluoride, previously dried at 200 °C for 4 hours, put it in a polyethylene beaker, and dissolve with 200 mL of water. Then add water to make 1000 mL and store in a polyethylene container. Take exactly 5 mL of this solution, place it in a mess flask, and add water to make 1000 mL (each mL of this solution contains 5 μg of fluorine). Take 1 mL, 2 mL, 3 mL, 5 mL, 10 mL and 15 mL of this solution, transfer each to a polyethylene beaker, add 15 mL of sodium citrate (1 in 4) and 10 mL of disodium ethylenediaminetetra (1 in 40) and mix. Adjust the pH to between 5.4 and 5.6 with hydrochloric acid (1 in 10) or sodium hydroxide solution (2 in 5), then add water to make 100 mL, and use these solutions as the standard solutions. Take 50 mL from each standard solution into a polyethylene beaker, measure the potential with a fluoride electrode, and create a calibration curve using the log values of the fluorine concentrations.

Loss on ignition Between 24.5% and 26.5% (1 g, 800 to 825 ℃, constant mass).

Assay Weigh accurately about 0.4 g of Dibasic Calcium Phosphate Hydrate, dissolve in 12 mL of dilute hydrochloric acid by heating on a water bath if necessary, and add water to make exactly 200 mL. Take exactly 20 mL of this solution, add exactly 25 mL of 0.02 mol/L ethylenediaminetetraacetic acid disodium salt solution, add 50 mL of water and 5 mL of ammonia-ammonium chloride buffer solution, pH 10.7, and titrate the excess ethylenediaminetetraacetic acid disodium salt with 0.02 moL/L zinc sulfate VS. (indicator: 25 mg of eryochrome black T-sodium chloride indicator). Perform a blank test in the same manner.

Each mL of 0.02 mol/L ethylenediaminetetraacetic acid disodium salt VS
= 3.442 mg of $CaHPO_4 \cdot 2H_2O$

Packaging and storage Preserve in well-closed containers.

Dibasic Sodium Phosphate Hydrate
인산수소나트륨수화물

Dibasic Sodium Phosphate
Sodium phosphate $Na_2HPO_4 \cdot 12H_2O$: 358.14

Dibasic Sodium Phosphate Hydrate, when dried, contains NLT 98.0% and NMT 101.0% of dibasic sodium phosphate (Na_2HPO_4 : l41.96).

Description Dibasic Sodium Phosphate Hydrate occurs as a colorless or white crystal and is odorless.
It is freely soluble in water and practically insoluble in ethanol or ether.
It effloresces in hot, dry air.

Identification An aqueous solution of Dibasic Sodium Phosphate Hydrate (1 in 10) responds to Chemical identification reactions (1) and (2) for sodium salt and the Chemical identification reactions for phosphate.

pH Dissolve 1.0 g of Dibasic Sodium Phosphate Hydrate in 50 mL of water; the pH of this solution is 9.0 to 9.4.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Dibasic Sodium Phosphate Hydrate in 20 mL of water; the resulting solution is clear and colorless.

(2) ***Water-insoluble substances***—Weigh accurately 10 g of Dibasic Sodium Phosphate Hydrate, add 100 mL of hot water, and filter through a glass filter (1G4). Wash the insoluble matter with 30 mL of hot water and dry with a glass filter at 105 °C for 2 hours; the amount is NMT 0.2%.

(3) ***Chloride***—Dissolve 1.0 g of Dibasic Sodium Phosphate Hydrate in 7 mL of dilute nitric acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution with 0.40 mL of 0.01 mol/L hydrochloric acid (NMT 0.014%).

(4) ***Sulfate***—Dissolve 0.5 g of Dibasic Sodium Phosphate Hydrate in 2 mL of dilute hydrochloric acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution with 0.40 mL of 0.005 mol/L sulfuric acid (NMT 0.038%).

(5) ***Carbonate***—Add 5 mL of water to 2.0 g of Dibasic Sodium Phosphate Hydrate, boil, cool, and add 2 mL of hydrochloric acid; the solution doesn't produce foams.

(6) ***Heavy metals***—Dissolve 2.0 g of Dibasic Sodium Phosphate Hydrate in 4 mL of acetic acid and add water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution by adding 2 mL of dilute acetic acid and water to 2.0 mL of lead standard solution to make 50 mL (NMT 10 ppm).

(7) ***Mercury***—Spread evenly about 1 g of excipient (a) into a ceramic boat and place 10 to 300 mg of Dibasic Sodium Phosphate Hydrate on top. Spread about 0.5 g of the excipient (a) and 1 g of the excipient (b) on top of it in turn to form a layer. In the case of an automated mercury analyzer equipped with a separate catalyst in the combustion chamber, take only the sample without adding excipients to a nickel boat. Place the boat inside the combustion furnace and heat to about 900 °C with a current of air or oxygen at 0.5 to 1 L/minute. Elute the mercury and collect in a collecting tube. Transfer the mercury vapor to a cold vapor atomic absorption spectrometer by heating the collecting tube to about 700 °C and determine the absorbance, A. Separately, place only the excipients in a ceramic boat and determine the absorbance, Ab, in the same manner. Separately, proceed with mercury

standard solution in the same manner and create a calibration curve from the absorbances. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the sample (NMT 1.0 ppm).

Operating conditions

Apparatus: Use an automated mercury analyzer for specimen combustion, trapping with gold amalgam and measurement using a cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Standard mercury stock solution—Dissolve 0.135 g of mercury(II) chloride in 0.001% L-cysteine solution to make 1000 mL. Each mL of this solution contains 100 μg of mercury(II) chloride.

Mercury standard solution—Dilute the standard mercury stock solution with 0.001% L-cysteine solution to make 0 to 200 ng/mL.

Excipients—Activate excipients at 950 ℃ for 30 minutes when using (a) aluminum oxide and (b) calcium hydroxide-sodium carbonate (1 : 1).

(8) ***Cadmium***—Weigh accurately 5.0 g of Dibasic Sodium Phosphate Hydrate, transfer to a platinum crucible, dry, carbonize, and incinerate at 450 to 550 °C. If incineration is not complete, cool and moisten with 2 to 5 mL of nitric acid (1 in 2), 50% magnesium nitrate solution or aluminum nitrate-calcium nitrate solution (prepared by dissolving 40 g of aluminum nitrate and 20 g of calcium nitrate in 100 mL of water) as an incineration supplement, dry, and continue with incineration. If incineration is incomplete, repeat the above procedure once and then add a final 2 to 5 mL of nitric acid (1 in 2), if necessary, and incinerate. After incineration, moisten the residue with water, add 2 to 4 mL of hydrochloric acid, and evaporate to dryness. Add 0.5 mol/L nitric acid, dissolve by warming, and filter with filter paper if any insoluble matter exists. Unless otherwise specified, add 0.5 mol/L nitric acid to make 25 mL and use this solution as the test solution. Separately, proceed with 5.0 mL of cadmium standard solution in a platinum crucible in the same manner as the test solution, and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Atomic absorption spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 1.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: A cadmium hollow-cathode lamp
Wavelength: 228.8 nm

(9) ***Lead***—Weigh accurately 5.0 g of Dibasic Sodium Phosphate Hydrate, transfer into a 150-mL beaker, and add 30 mL of water. Add hydrochloric acid in small amounts until the sample is completely dissolved, and then add 1 mL of hydrochloric acid. Boil for about 5 minutes, cool, add water to make about 100 mL, and adjust the pH to 2 to 4 with sodium hydroxide (1 in 4) or hydrochloric acid (1 in 4). Transfer this solution to a 250-mL separatory funnel, add water to make about 200 mL, add 2 mL of 2% ammonium pyrrolidine dithiocarbamate solution (2 in 100), and shake to mix. Extract this solution twice with 20 mL of chloroform and evaporate the extract to dryness on a water bath. To the residue, add 3 mL of nitric acid and heat until almost dry. Add 0.5 mL of nitric acid and 10 mL of water, concentrate until the final solution becomes 3 to 5 mL, add water to make 10 mL, and use this solution as the test solution. Separately, proceed with 2.0 mL of the lead standard solution in the same manner as the test solution and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Atomic absorption spectroscopy according to the following operating conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 4.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: A lead hollow-cathode lamp
Wavelength: 283.3 nm

(10) ***Arsenic***—Weigh 1.0 g of Dibasic Sodium Phosphate Hydrate according to Method 1 and perform the test using the solution as the test solution (NMT 2 ppm).

(11) ***Fluoride***—Weigh 1 g of Dibasic Sodium Phosphate Hydrate, transfer to a beaker, and dissolve in 10 mL of hydrochloric acid (1 in 10). Heat this solution, boil for 1 minute, transfer to a polyethylene beaker, and cool immediately. Add 15 mL of sodium citrate solution (1 in 4) and 10 mL of ethylenediaminetetraacetic acid disodium salt solution (1 in 40), and shake to mix. Adjust the pH to 5.4 to 5.6 with hydrochloric acid (1 in 10) or sodium hydroxide (2 in 5), add water to make 100 mL, and use this solution as the test solution. Transfer 50 mL of the test solution to a polyethylene beaker, determine the potential using a fluoride electrode, and determine the amount of fluoride from the calibration curve; the amount is NMT 10 ppm.

Creating a calibration curve: Weigh accurately 2.210 g of sodium fluoride, previously dried at 200 °C for 4 hours, and transfer to a polyethylene beaker. Dissolve in 200 mL of water, add water to make 1000 mL, and preserve in a polyethylene container. Pipet 5 mL of this solution, transfer to a volumetric flask, and add water to make 1000 mL (each mL of this solution contains 5 μg of fluoride). Take 1 mL, 2 mL, 3 mL, 5 mL, 10 mL and 15 mL of this solution, transfer each to a polyethylene beaker, add 15 mL of sodium citrate (1 in 4) and 10 mL of disodium ethylenediaminetetraacetate (1 in 40) to mix. Adjust the pH to 5.4 to 5.6 with hydrochloric acid (1 in 10) or sodium hydroxide (2 in 5), add water to make 100 mL each, respectively, and use these solutions as the standard solutions. Pipet 50 mL of each standard solution to polyethylene containers. Determine the potential using a fluoride electrode and create a calibration curve with the log values of the fluoride concentrations.

Loss on drying Between 57.0% and 61.0% (10 g, at 40 ℃ for 3 hours at first, then at 105 ℃ for 5 hours).

Assay Weigh accurately about 3 g of Dibasic Sodium Phosphate Hydrate, previously dried, dissolve in 50 mL of water, and titrate with 0.5 mol/L sulfuric acid VS while maintaining the temperature at 15°C (indicator: 3 to 4 drops of methyl orange-xylene cyanol FF TS). However, the endpoint of titration is when the color of the solution changes from green to dark greenish purple.

Each mL of 0.5 mol/L sulfuric acid VS
= 141.96 mg of Na_2HPO_4

Packaging and storage Preserve in tight containers.

Dimethylaminoethyl Methacrylat·Methyl Methacrylate Copolymer
메타아크릴산디메틸아미노에틸메타아크릴산 메틸공중합체

Eudragit E

Dimethylaminoethyl Methacrylate·Methyl Methacrylate Copolymer in a copolymer of dimethylaminoethyl methacrylate and methyl methacrylate.

Dimethylaminoethyl Methacrylate·Methyl Methacrylate Copolymer, when dried, contains 4.0% to 6.0% of nitrogen (N: 14.01).

Description Dimethylaminoethyl Methacrylate·Methyl Methacrylate Copolymer is a pale yellow resin-like particle.
It is odorless or has a slight characteristic odor and is tasteless.
It is freely soluble in methanol, ethanol, acetone, and ether and soluble in dilute hydrochloric acid.
It is practically insoluble in water.

Identification (1) Dissolve 0.1 g of Dimethylaminoethyl Methacrylate·Methyl Methacrylate Copolymer, previously powdered, in 10 mL of 1 mol/L hydrochloric acid TS, and add sodium hydroxide TS to make alkaline; a white resin-like substance forms.

(2) Weigh 1.0 mg of Dimethylaminoethyl Methacrylate·Methyl Methacrylate Copolymer, previously dried at 105 ℃ for 4 hours, and determine the absorption as directed under the potassium bromide disk method of the Mid-infrared Spectroscopy; it exhibits absorption at the wavenumbers of about 3400 cm^{-1}, 2950 cm^{-1}, 1720 cm^{-1}, 1450 cm^{-1}, 1150 cm^{-1}, 960 cm^{-1}, 1156 cm^{-1}, and 750 cm^{-1}.

Viscosity Between 2.5 mm^2/s and 5.5 mm^2/s (Method 1, 20 ℃)

Purity (1) ***Clarity and color of solution***—Dissolve 0.5 g of Dimethylaminoethyl Methacrylate·Methyl Methacrylate Copolymer, previously powdered, in 20 mL of 1 mol/L hydrochloric acid; the resulting solution is colorless and clear.

(2) ***Heavy metals***—Proceed with 1.0 g of Dimethylaminoethyl Methacrylate·Methyl Methacrylate Copolymer, previously powdered, according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 20 ppm).

(3) ***Arsenic***—Prepare the test solution with 1.0 g of Dimethylaminoethyl Methacrylate·Methyl Methacrylate Copolymer, previously powdered, according to Method 3 and perform the test (NMT 2 ppm).

(4) ***Methyl methacrylate and dimethylaminoethyl methacrylate***—Weigh accurately about 1.0 g of Dimethylaminoethyl Methacrylate·Methyl Methacrylate Copolymer, transfer into 8 mL of acetone, shake to mix, and then add acetone to make 10 mL. Use this solution as the test solution. Separately, weigh accurately about 10 mg of methyl methacrylate RS and about 20 mg of dimethylaminoethyl methacrylate, dissolve in acetone to make 100 mL, and use this solution as the standard solution. Perform the test with 1 μL each of the test solution and the standard solution as directed under the Gas Chromatography according to the following conditions; the peak heights of methyl methacrylate and dimethylaminoethyl methacrylate obtained from the test solution are less than those obtained from the standard solution.

Operating conditions

Detector: A flame ionization detector

Column: A stainless steel column about 3 mm in internal diameter and about 2 m in length, packed with diatomaceous earth for gas chromatography, coated with polyethylene glycol 20 M for gas chromatography at the ratio of 20% (177 μm to 297 μm in particle diameter).

Column temperature: A constant temperature of about 90 ℃.

Carrier gas: Nitrogen

Flow rate: Adjust the flow rate so that the retention time of methyl methacrylate is about 3 minutes, and the retention time of dimethylaminoethyl methacrylate is about 22 minutes.

Detection sensitivity: Adjust the sensitivity so that the peak height of methyl methacrylate obtained from the standard solution is about 2 cm and dimethylaminoethyl methacrylate is about 1 cm.

Loss on drying NMT 2.0% (1 g, 105 ℃, 4 hours).

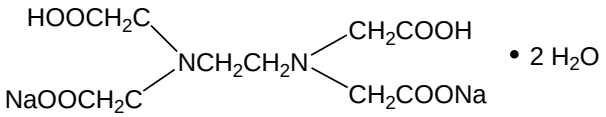
Residue on ignition NMT 0.2% (1 g).

Assay Weigh accurately about 0.2 g of Dimethylaminoethyl Methacrylate·Methyl Methacrylate Copolymer, previously dried, and perform the test as directed under the Nitrogen Determination (Semimicro-Kjeldahl Method).

Each mL of 0.005 mol/L sulfuric acid VS
= 0.1401 mg of N

Packaging and storage Preserve in tight containers.

Disodium Edetate Hydrate
에데트산나트륨수화물



Ethylenediaminetetraacetic acid disodium salt
Disodium Edetate
EDTA Sodium $C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O$: 372.24
Disodium 2-({2-[*bis*(carboxymethyl)amino]ethyl} (carboxymethyl)amino)acetate [*6381-92-6*]

Disodium Edetate Hydrate contains NLT 99.0% and NMT 101.0% of disodium edetate hydrate ($C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O$).

Description Disodium Edetate Hydrate occurs as a white crystal or crystalline powder, is odorless and has a slightly sour taste. It is soluble in water and practically insoluble in ethanol or ether.

Identification (1) Dissolve 10 mg of Disodium Edetate Hydrate in 5 mL of water, add 2 mL of potassium chromate solution (1 in 200) and 2 mL of arsenic trioxide TS and heat the mixture on a water bath for 2 minutes; the resulting solution exhibits a violet color.

(2) Dissolve 0.5 g of Disodium Edetate Hydrate in 20 mL of water and add 1 mL of dilute hydrochloric acid; a white precipitate is produced. Filter and collect the precipitate, wash with 50 mL of water and dry at 105 ℃ for 1 hour; the melting point of the precipitate is between 240 ℃ and 244 ℃ (with decomposition).

(3) An aqueous solution of Disodium Edetate Hydrate (1 in

20) responds to the Chemical identification reactions (1) for sodium salt.

pH Dissolve 1.0 g of Disodium Edetate Hydrate in 100 mL of water; the pH of the resulting solution is 4.3 to 4.7.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Disodium Edetate Hydrate in 50 mL of water; the resulting solution is clear and colorless.

(2) ***Cyanide***—Weigh 1.0 g of Disodium Edetate Hydrate and place it into a round-bottom flask, dissolve in 100 mL of water, add 10 mL of phosphoric acid and distill. In the receiver, add 15 mL of 0.5 mol/L sodium hydroxide in a 100-mL measuring cylinder, immerse the top end of the condenser into the solution. Distill the solution until the total amount of distillate is 100 mL and use this solution as the test solution. Transfer 20 mL of the test solution to a stoppered test tube, add 1 drop of phenolphthalein TS, neutralize with dilute acetic acid, and add 5 mL of phosphate buffer solution (pH 6.8) and 1.0 mL of diluted chloramine TS (1 in 5). Immediately stopper the tube, mix gently and allow it to stand for 2 to 3 minutes. Add 5 mL of pyridine-pyrazolone TS, mix well, and allow the mixture to stand between 20 °C and 30 °C for 50 minutes; the color of the solution is not more intense than the following control solution.

Control solution—Pipet 1.0 mL of the cyanide standard solution, add 15 mL of 0.5 mol/L sodium hydroxide solution, and add water to make exactly 1000 mL. Transfer 20 mL of this solution to a stoppered test tube and proceed in the same manner as the test solution.

(3) ***Heavy metals***—Weigh 2.0 g of Disodium Edetate Hydrate and perform the test according to Method 2. Prepare the control solution by adding 2.0 mL of the lead standard solution (NMT 10 ppm).

(4) ***Lead***—Weigh accurately 5.0 g of Disodium Edetate Hydrate and transfer it to a platinum crucible. Dry, carbonize and incinerate at 450 °C to 550 °C. If incineration is not complete, cool and moisten with 2 to 5 mL of nitric acid (1 in 2), 50% magnesium nitrate solution or aluminum nitrate-calcium nitrate solution (prepared by dissolving 40 g of aluminum nitrate and 20 g of calcium nitrate in 100 mL of water) as an incineration supplement, dry, and continue with incineration. If incineration is incomplete, repeat the above procedure once and then add a final 2 mL to 5 mL of nitric acid (1 in 2), if necessary, and incinerate. After incineration, moisten the residue with water, add 2 to 4 mL of hydrochloric acid, and evaporate to dryness. Add 0.5 mol/L nitric acid, dissolve by warming, and filter with filter paper if any insoluble matter exists. Unless otherwise specified, add 0.5 mol/L nitric acid to make 25 mL and use this solution as the test solution. Separately, take 1.0 mL of lead standard solution to a platinum crucible, proceed in the same manner as the test solution, and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Atomic absorption spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 2.0 ppm).

Gas: Acetylene or hydrogen - air
Lamp: Lead hollow-cathode lamp
Wavelength: 283.3 nm

(5) ***Arsenic***—Weigh 1.0 g of Disodium Edetate Hydrate and perform the test according to Method 1 (NMT 2 ppm).

(6) ***Nitrilotriacetic acid***—Weigh accurately 1 g of Disodium Edetate Hydrate, dissolve in copper(II) nitrate solution to make exactly 100 mL, and use this solution as the test solution. Separately, weigh accurately 0.1 g of nitrilotriacetic acid RS, add 0.5 mL of ammonia solution, add water to make exactly 10 mL, and use this solution as the standard stock solution. Weigh accurately 1.0 g of ethylenediaminetetraacetic acid disodium salt, add 100 μL of the standard stock solution, add copper(II) nitrate solution to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 50 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak area of each solution by the automatic integration method; the peak area of nitrilotriacetic acid in the test solution is not greater than that of nitrilotriacetic acid in the standard solution (NMT 0.1%).

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 254 nm)

Column: A stainless steel column about 4 mm in internal diameter and about 15 cm in length, packed with octasillane silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about room temperature.

Mobile phase: Add 10 mL of 1 mol/L tetrabutylammonium hydroxide-methanol solution to 200 mL of water, adjust the pH to 7.5 ± 0.1 with 1 mol/L phosphoric acid, add 90 mL of methanol, and add water to make exactly 1000 mL.

Flow rate: 2.0 mL/min

System suitability

System performance: Weigh 10 mg of ethylenediaminetetraacetic acid disodium salt, add 100 μL of the standard stock solution and add copper(II) nitrate solution to make exactly 100 mL. Proceed with 50 μL of this solution according to the above operating conditions; the resolution between the peaks of nitrilotriacetic acid and copper is NLT 3. The relative retention times of nitrilotriacetic acid and copper with respect to the retention time of ethylenediaminetetraacetic acid disodium salt are 0.35 and 0.65, respectively.

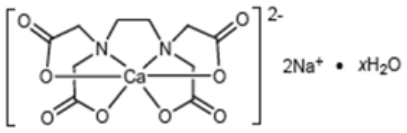
Residue on ignition Between 37.0% and 39.0% (1 g).

Assay Weigh accurately about 1 g of Disodium Edatate Hydrate, dissolve in 50 mL of water, add 2 mL of ammonia-ammonium chloride buffer solution (pH 10.7), and titrate with 0.1 mol/L zinc VS (Indicator: 40 mg of eryochrome black T-sodium chloride indicator). The endpoint of the titration is when the color of the solution changes from blue to red.

Each mL of 0.1mol/L zinc VS
= 37.224 mg of $C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O$

Packaging and storage Preserve in well-closed containers.

Edetate Calcium Sodium Hydrate
에데트산칼슘나트륨수화물



$C_{10}H_{12}CaN_2Na_2O_8 \cdot xH_2O$: 374.27
Disodium [{*N,N* ′-ethane-1,2-diylbis[*N*-(carboxymethyl)glycinato]}(4-)-*N,N′,O,O′,ON,ON* ′]calciate(2-)hydrate [*23411-34-9*]

Edetate Calcium Sodium Hydrate contains NLT 98.0% and NMT 102.0% of sodium calcium edetate ($C_{10}H_{12}CaN_2Na_2O_8$), calculated on the anhydrous basis.

Description Edetate Calcium Sodium Hydrate occurs as a white crystal or crystalline powder.
It is freely soluble in water, sparingly soluble in methanol and practically insoluble in ethanol (99.5).
It is hygroscopic.

Identification (1) Dissolve 2 g of Edetate Calcium Sodium Hydrate in 10 mL of water completely, add 6 mL of lead (II) nitrate solution (33 in 1000), shake to mix, and add 3 mL of potassium iodide TS; no precipitate is formed. To this solution, add dilute ammonia solution (7 in 50) to render the solution alkaline, and add 3 mL of ammonium oxalate TS; a white precipitate is formed.

(2) Determine the infrared spectra of Edetate Calcium Sodium Hydrate and sodium calcium edetate RS as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

(3) An aqueous solution of Edetate Calcium Sodium Hydrate (1 in 20) responds to the Chemical identification reactions (2) for sodium salt.

pH Dissolve 2.0 g of Edetate Calcium Sodium Hydrate in 10 mL of water; the pH of this solution is 6.5 to 8.0.

Purity (1) ***Clarity and color of solution***—Dissolve 0.25 g of Edetate Calcium Sodium Hydrate in 10 mL of water; the solution is clear and colorless.

(2) ***Chloride***—Dissolve 0.7 g of Edetate Calcium Sodium Hydrate in 5 mL of water, add 30 mL of dilute nitric acid, allow the mixture to stand for 30 minutes, and filter. Take 10 mL of the filtrate and add water to make 50 mL. Use this solution as the test solution. Prepare the control solution with 0.40 mL of 0.01 mol/L hydrochloric acid (NMT 0.10%).

(3) ***Edetate sodium***—Dissolve 100 g of Edetate Calcium Sodium Hydrate in 50 mL of water, add 5 mL of ammonia-ammonium chloride buffer solution (pH 10.7), and titrate with 0.01 mol/L magnesium chloride VS (indicator: 40 mg of eryochrome black T-sodium chloride indicator). The endpoint of the titration is when the color of the solution changes from blue to reddish purple (NMT 1.0%).

(4) ***Nitrilotriacetic acid***—Dissolve 0.100 g of Edetate Calcium Sodium Hydrate in the solvent to make exactly 25 mL, and use this solution as the test solution. Separately, weigh 40.0 mg of nitrilotriacetic acid RS and dissolve in the solvent to make exactly 100 mL. Pipet 1 mL of this solution, add 0.1 mL of the test solution and the solvent to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 20 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak area in each solution according to the automatic integration method; the peak area of nitrilotriacetic acid in the test solution is not greater than that in the standard solution (NMT 0.1%).

Solvent—Dissolve 10.0 g of Iron(III) sulfate *n*-hydrate in 20 mL of 0.5 mol/L sulfuric acid TS and 780 mL of water. Adjust the pH to 2.0 with sodium hydroxide TS and add water to make 1000 mL.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 273 nm)

Column: A stainless steel column about 4.6 mm in internal diameter and about 10 cm in length, packed with graphitic carbon for liquid chromatography (5 μm in average particle diameter, 120 m^2/g).

Column temperature: A constant temperature of about 40 °C.

Mobile phase: Dissolve 50.0 mg of Iron(III) sulfate *n*-hydrate in 50 mL of 0.5 mol/L sulfuric acid TS, add 750 mL of water, and adjust the pH to 1.5 with 0.5 mol/L sulfuric acid TS or sodium hydroxide TS. Add 20 mL of ethylene glycol and water to make exactly 1000 mL.

Flow rate: 1.0 mL/min

System suitability

System performance: Proceed with 20 μL of the standard solution according to the above conditions; nitrilotriacetic acid and edetic acid are eluted in this order with the resolution being NLT 7. The signal-to-noise ratio of nitrilotriacetic acid is NLT 50.

System repeatability: Repeat the test 6 times with 20 μ L of the standard solution according to the above conditions; the relative standard deviation of the peak area of nitrilotriacetic acid is NMT 1.0%.

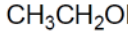
Water Between 5.0% and 13.0% (0.2 g, volumetric titration, direct titration).

Assay Weigh accurately about 0.5 g of Edetate Calcium Sodium Hydrate and add water to make 200 mL. Pipet 20 mL of this solution, add 80 mL of water and adjust the pH to 2 to 3 with dilute nitric acid, and titrate with 0.01 mol/L bismuth nitrate VS (indicator: 2 drops of xylenol orange TS). The endpoint of titration is when the yellow color of the solution turns into a red color.

Each mL of 0.01 mol/L bismuth nitrate VS
= 3.743 mg of $C_{10}H_{12}CaN_2Na_2O_8$

Packaging and storage Preserve in tight containers.

Ethanol
에탄올(95)



Alcohol C_2H_6O: 46.07
Ethanol [*64-17-5*]

Ethanol (95) contains NLT 95.1 vol% and NMT 96.9% (by specific gravity) of ethanol (C_2H_6O) at 20 °C.

Description Ethanol (95) occurs as a clear, colorless liquid.
It is miscible with water and dichloromethane.
It is flammable and burns with a pale blue flame on ignition.
It is volatile.
Boiling point: About 78 °C.

Identification (1) Determine the infrared spectra of Ethanol (95) and ethanol RS as directed in the liquid film method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

Specific gravity d_{20}^{20}: Between 0.805 and 0.812.

Purity (1) ***Clarity and color of solution***—Ethanol (95) is clear and colorless. To 1.0 mL of Ethanol (95), add water to make 20 mL, and allow the mixture to stand for 5 minutes: the resulting solution is clear.

(2) ***Acidity or alkalinity***—To 20 mL of Ethanol (95), add 20 mL of freshly boiled and cooled water and 0.1 mL of a solution prepared by adding 7.0 mL of ethanol (95) and 2.0 mL of water to 1.0 mL of phenolphthalein TS; the resulting solution is colorless. Add 1.0 mL of 0.01 mol/L sodium hydroxide solution; the resulting solution exhibits a pale red color (30 ppm as acetic acid).

(3) ***Volatile impurities***—Pipet 500 mL of Ethanol (95), add 150 μL of 4-methylpentan-2-ol, and use this solution as the test solution. Separately, to 100 μL of anhydrous methanol, add Ethanol (95) to make exactly 50 mL. Pipet 5 mL of this solution, add Ethanol (95) to make exactly 50 mL, and use this solution as the standard solution (1). Separately, pipet 50 μL each of anhydrous methanol and acetaldehyde and add Ethanol (95) to make exactly 50 mL. To 100 μL of this solution, add Ethanol (95) to make exactly 10 mL, and use this solution as the standard solution (2). Then, to 150 μL of acetal, add Ethanol (95) to make exactly 50 mL. To 100 μL of this solution, add Ethanol (95) to make exactly 10 mL, and use this solution as the standard solution (3). Then, to 100 μL of benzene, add Ethanol (95) to make exactly 100 mL. To 100 μL of this solution, add Ethanol (95) to make exactly 50 mL, and use this solution as the standard solution (4). Pipet 1 μL each of Ethanol (95), the test solution, the standard solution (1), the standard solution (2), the standard solution (3), and the standard solution (4), and perform the test as directed under the Gas Chromatography according to the following conditions. Determine respective peak areas of Ethanol (95) and each solution according to the automatic integration method and calculate the peak areas, A_E, B_E, and C_E, of acetaldehyde, benzene, and acetal, respectively, obtained from Ethanol (95), the peak area of methanol from the standard solution (1), peak area of acetaldehyde, A_T, from the standard solution (2), peak area of acetal, C_T, from the standard solution (3), and the peak area of benzene, B_T, from the standard solution (4); the peak area of methanol from Ethanol (95) is NMT 1/2 times the peak area of methanol from the standard solution (1) (200 vol ppm). Calculate the amount of the impurity by the following equation: the sum of the amounts of acetaldehyde and acetal as acetaldehyde is NMT 10 vol ppm, and the amount of benzene is NMT 2 vol ppm. The total area of the peaks of other impurities in the test solution is NMT the peak area of 4-methylpentan-2-ol (300 ppm). However, ignore the peaks NMT 3% of the peak area of 4-methylpentan-2-ol (9 ppm).

$$\text{Total amount (vol ppm) of acetaldehyde and acetal} = (10 \times A_E) / (A_T - A_E) + (30 \times C_E \times 44.05) / [(C_T - C_E) \times 118.2]$$

$$\text{Amount (vol ppm) of benzene} = 2B_E / (B_T - B_E)$$

If necessary, identify benzene according to different suitable chromatography conditions of a stationary phase (liquid) with different polarity.

Operating conditions

Detector: A flame ionization detector

Column: A fused silica column about 0.32 mm in internal diameter and about 30 m in length, coated inside with 6% cyanopropyl phenyl-94% dimethyl silicone polymer for gas chromatography in 1.8 μm thickness.

Column temperature: Inject the sample at a constant temperature of about 40 °C, maintain the temperature for 12 minutes, and raise the temperature to 240 °C at a rate of 10 °C per minute, and maintain at a constant temperature of about 240 °C for 10 minutes.

Sample injection port temperature: A constant temperature of about 200 °C.

Detector temperature: A constant temperature of about 280 °C.

Carrier gas: Helium

Flow rate: 35 cm/sec

Split ratio: About 1 : 20

System suitability

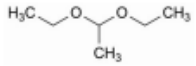
System performance: Proceed with 1 μL of the standard solution (2) according to the above conditions; acetaldehyde and methanol are eluted in this order with the resolution being NLT 1.5.

(4) ***Other impurities (absorbance)*** Perform the test with Ethanol (95) as directed under the Ultraviolet-visible Spectroscopy using a 5-cm cell with water as the blank and determine the absorption spectrum between 235 nm and 340 nm; the absorbances at 240 nm, 250 nm to 260 nm and 270 nm to 340nm are NMT 0.40, 0.30 and 0.10 and the slope of absorption curve is gentle.

(5) ***Residue on evaporation***—Pipet 100 mL of Ethanol (95), evaporate to dryness on a water bath, and dry the residue at 105 ºC for 1 hour; the amount (mg) of the residue is NMT 2.5 mg (25 w/v ppm).

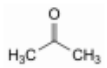
Packaging and storage Preserve in light-resistant, tight containers.

Note



A. 1,1-diethoxyethane (acetal)

B. Acetaldehyde

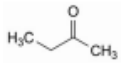


C. Propan-2-one (acetone)

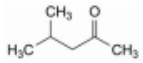
D. Benzene

E. Cyclohexane

F. Methanol

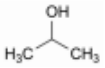


G. Butan-2-one (methyl ethyl ketone)



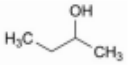
H. 4-methylpentan-2-one (methyl isobutyl ketone)

I. Propan-1-ol (propanol)

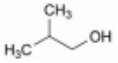


J. Propan-2-one (acetone)

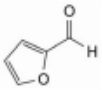
K. Butan-1-ol (butanol)



L. Butan-2-ol

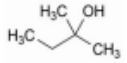


M 2-methylpropan-1-ol (isobutanol)

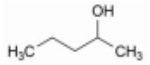


N. Furan-2-carbaldehyde (furfural)

O. 2-methylpropan-2-ol (1,1-dimethylethyl alcohol)



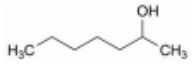
P. 2-methylbutan-2-ol



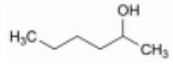
Q. Pentan-2-ol

R. Pentan-1-ol (Pentanol)

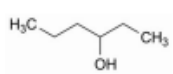
S. Hexan-1-ol (hexanol)



T. Heptan-2-ol



U. Hexan-2-ol



V. Hexan-3-ol

Anhydrous Ethanol
에탄올(99.5)

CH_3CH_2OH

Anhydrous Alcohol C_2H_6O: 46.07
Ethanol [*64-17-5*]

Ethanol (99.5) contains NLT 99.5 vol% (by specific gravity) of ethanol (C_2H_6O) at 20 °C.

Description Ethanol (99.5) occurs as a clear, colorless liquid.
It is miscible with water and dichloromethane.
It is flammable and burns with a pale blue flame on ignition.
It is volatile.
Boiling point: About 78℃.

Identification (1) Determine the infrared spectra of Ethanol (99.5) and ethanol (99.5) RS as directed in the liquid film method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

Specific gravity d_{20}^{20}: Between 0.790 and 0.793.

Purity (1) ***Clarity and color of solution***—Ethanol (99.5) is clear and colorless. To 1.0 mL of Ethanol (99.5), add water to make 20 mL, and allow the mixture to stand for 5 minutes; the resulting solution is clear.

(2) ***Acidity or alkalinity***—To 20 mL of Ethanol (99.5), add 20 mL of freshly boiled and cooled water and 0.1 mL of a solution prepared by adding 7.0 mL of ethanol (95) and 2.0 mL of water to 1.0 mL of phenolphthalein TS; the resulting solution is colorless. Add 1.0 mL of 0.01 mol/L sodium hydroxide solution; the resulting solution exhibits a pale red color (30 ppm as acetic acid).

(3) ***Volatile impurities***—Pipet 500 mL of Ethanol (99.5), add 150 μL of 4-methylpentan-2-ol, and use this solution as the test solution. Separately, to 100 μL of anhydrous methanol, add Ethanol (99.5) to make exactly 50 mL. Pipet 5 mL of this solution, add Ethanol (99.5) to make exactly 50 mL, and use this solution as the standard solution (1). Separately, pipet 50 μL each of anhydrous methanol and acetaldehyde, add Ethanol (99.5) to make exactly 50 mL. To 100 μL of this solution, add Ethanol (99.5) to make exactly 10 mL, and use this solution as the standard solution (2). Then, to 150 μL of acetal, add Ethanol (99.5) to make exactly 50 mL. To 100 μL of this solution, add Ethanol (99.5) to make exactly 10 mL, and use this solution as the standard solution (3). Then, to 100 μL of benzene, add Ethanol (99.5) to make exactly 100 mL. To 100 μL of this solution, add Ethanol (99.5) to make exactly 50 mL, and use this solution as the standard solution (4). Pipet 1 μL each of Ethanol (99.5), the test solution, the standard solution (1), the standard solution (2), the standard solution (3), and the standard solution (4), and perform the test as directed under the Gas Chromatography according to the following conditions. Determine respective peak areas of Ethanol (99.5) and each solution according to the automatic integration method and calculate the peak areas, A_E, B_E, and C_E, of acetaldehyde, benzene, and acetal, respectively, obtained from Ethanol (99.5), the peak area of methanol from the standard solution (1), peak area of acetaldehyde, A_T, from the standard solution (2), peak area of acetal, C_T, from the standard solution (3), and the peak area of benzene, B_T, from the standard solution (4); the peak area of methanol from Ethanol (99.5) is NMT 1/2 times the peak area of methanol from the standard solution (1) (200 vol ppm). Calculate the amount of the impurities by the following equation;

the sum of the amount of acetaldehyde and acetal is NMT 10 vol ppm as acetaldehyde, and the amount of benzene is NMT 2 vol ppm. The total area of the peaks of other impurities in the test solution is NMT the peak area of 4-methylpentan-2-ol (300 ppm). However, disregard the peaks NMT 3% of the peak area of 4-methylpentan-2-ol (9 ppm).

$$\text{Total amount (vol ppm) of acetaldehyde and acetal} = (10 \times A_E) / (A_T - A_E) + (30 \times C_E \times 44.05)/[(C_T - C_E) \times 118.2]$$

$$\text{Amount (vol ppm) of benzene} = 2B_E / (B_T - B_E)$$

If necessary, identify benzene according to different suitable chromatography conditions of a stationary phase (liquid) with different polarity.

Operating conditions

Detector: A flame ionization detector

Column: A fused silica column about 0.32 mm in internal diameter and about 30 m in length, coated inside with 6% cyanopropyl phenyl-94% dimethyl silicone polymer for gas chromatography in 1.8 μm thickness.

Column temperature: Inject the sample at a constant temperature of about 40 °C, maintain the temperature for 12 minutes, and raise the temperature to 240 °C at a rate of 10 °C per minute, and maintain at a constant temperature of about 240 °C for 10 minutes.

Sample injection port temperature: A constant temperature of about 200 °C.

Detector temperature: A constant temperature of about 280 °C.

Carrier gas: Helium

Flow rate: 35 cm/sec

Split ratio: About 1 : 20

System suitability

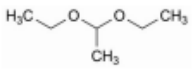
System performance: Proceed with 1 μL of the standard solution (2) according to the above conditions; acetaldehyde and methanol are eluted in this order with the resolution being NLT 1.5.

(4) ***Other impurities (absorbance)***—Perform the test with Ethanol (99.5) as directed under the Ultraviolet-visible Spectroscopy using a 5-cm cell with water as the blank, and determine the absorption spectrum between 235 nm and 340 nm; the absorbances at 240 nm, 250 nm to 260 nm, and 270 nm to 340 nm are not greater than 0.40, 0.30 and 0.10, respectively, and the slope of absorption curve is gentle.

(5) ***Residue on evaporation***—Pipet 100 mL of Ethanol (99.5), evaporate to dryness on a water bath, and dry the residue at 105 ºC for 1 hour; the amount (mg) of the residue is NMT 2.5 mg (25 w/v ppm).

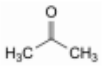
Packaging and storage Preserve in light-resistant, tight containers.

Note



A. 1,1-diethoxyethane (acetal)

B. Acetaldehyde

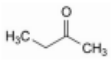


C. Propan-2-one (acetone)

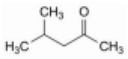
D Benzene

E. Cyclohexane

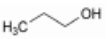
F. Methanol



G. Butan-2-one (methyl ethyl ketone)



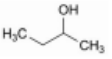
H. 4-methylpentan-2-one (methyl isobutyl ketone)



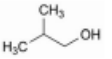
I. Propan-1-ol (propanol)

J. Propan-2-one (isopropyl alcohol)

K. Butan-1-ol (butanol)



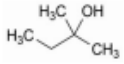
L. Butan-2-ol



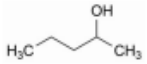
M 2-methylpropan-1-ol (isobutanol)

N. Furan-2-carbaldehyde (furfural)

O. 2-methylpropan-2-ol (1,1-dimethylethyl alcohol)



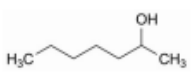
P. 2-methylbutan-2-ol



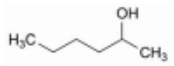
Q. Pentan-2-ol

R. Pentan-1-ol (pentanol)

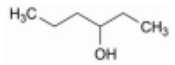
S. Hexan-1-ol (hexanol)



T. Heptan-2-ol



U. Hexan-2-ol



V. Hexan-3-ol

Ether
에테르

$CH_3CH_2OCH_2CH_3$

$C_4H_{10}O$: 74.12

Ethoxyethane [*60-29-7*]

Ether contains NLT 96.0% and NMT 98.0% (by specific gravity) of ether ($C_4H_{10}O$). Ether contains a small quantity of ethanol and water. Ether cannot be used for anesthesia.

Description Ether occurs as a colorless, clear, mobile liquid and has a characteristic odor.
It is miscible with ethanol.
It is soluble in water.
It is highly volatile and flammable.
It is slowly oxidized by air and light, producing peroxides.
The vapors of Ether, when mixed with air and ignited, explode violently.
Boiling point: Between 35 ℃ and 37 ℃.

Specific gravity d_{20}^{20}: Between 0.718 and 0.721.

Purity (1) ***Other odor***—Place 10 mL of Ether in an evaporating dish, and allow to evaporate spontaneously to about 1 mL; no other odor is perceptible. Dispense the remaining liquid dropwise onto an odorless filter paper to evaporate the ether; no other odor is perceptible.

(2) ***Acidity***—Place 10 mL of diluted ethanol (4 in 5) and 0.5 mL of phenolphthalein TS into a stoppered flask, and add 0.02 mol/L sodium hydroxide solution dropwise; the resulting solution exhibits a red color. Shake the flask to mix; the red color remains for 30 seconds. To this solution, add 25 mL of Ether, stopper the flask, shake gently, and add 0.40 mL of 0.02 mol/L sodium hydroxide solution while mixing by shaking; the resulting solution exhibits a red color.

(3) ***Aldehyde***—Place 10 mL of Ether in a Nessler tube, add 1 mL of potassium hydroxide TS, and allow the mixture to stand for 2 hours, protected from light, with occasional shaking; no color develops in the ether layer or the water layer.

(4) ***Peroxide***—Place 10 mL of Ether in a Nessler tube, add 1 mL of freshly prepared solution of potassium iodide solution (1 in 10), shake for 1 minute, then add 1 mL of starch TS, and shake well; no color develops in the ether layer or the water layer.

(5) ***Residue on evaporation***—Evaporate 140 mL of Ether and dry the residue at 105 ℃ for 1 hour; the weight of the residue is NMT 1.0 mg.

Packaging and storage Preserve in light-resistant, tight containers below 25 ℃, protected from fire, without filling up.

Ethyl Methacrylate·Trimethylammoniumethyl Methacrylate Chloride Copolymer
메타아크릴산에틸·메타아크릴산트리메틸암모늄에틸염화물공중합체

Eudragit RS

Ethyl Methacrylate·Trimethylammoniumethyl Methacrylate Chloride Copolymer is a copolymer of ethyl methacrylate and trimethylammoniumethyl methacrylate chloride.

Ethyl Methacrylate·Trimethylammoniumethyl Methacrylate Chloride Copolymer, when dried, contains 0.27% to 0.80% of nitrogen (N: 14.01).

Description Ethyl Methacrylate·Trimethylammoniumethyl Methacrylate Chloride Copolymer occurs a white resin-like mass or powder. It is odorless or has a slight characteristic odor and is tasteless.
It is freely soluble in ethanol of acetone and practically insoluble in ether.
It is practically insoluble in water.

Identification Weigh 1 mg of Ethyl Methacrylate·Trimethylammoniumethyl Methacrylate Chloride Copolymer, previously dried at 105 ℃ for 4 hours, and determine the absorption as directed under the potassium bromide disk method of the Mid-infrared Spectroscopy; it exhibits absorption at the wavenumbers of about 2940 cm^{-1}, 1720 cm^{-1}, 1450 cm^{-1}, 1385 cm^{-1}, 1235 cm^{-1}, 1150 cm^{-1}, and 1020 cm^{-1}.

Viscosity Between 1.0 mm^2/s and 3.0 mm^2/s (Method 1, 20 ℃) However, perform the test by dissolving in acetone.

Purity (1) ***Water-solubles***—Transfer 2.0 g of Ethyl Methacrylate·Trimethylammoniumethyl Methacrylate Chloride Copolymer, previously powdered, in 100 mL of water, shake and mix until it becomes turbid like liquid, then filter. Take 25 mL of the filtrate, evaporate to dryness on a water bath, and dry the residue at 105 ℃ for 4 hours; the mass of the residue is NMT 2.0 mg.

(2) ***Heavy metals***—Proceed with 1.0 g of Ethyl Methacrylate·Trimethylammoniumethyl Methacrylate Chloride Copolymer according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 20 ppm).

(3) ***Arsenic***—Proceed with 1.0 g of Ethyl Methacrylate·Trimethylammoniumethyl Methacrylate Chloride Copolymer according to Method 3, and perform the test (NMT 2 ppm).

(4) ***Ethyl methacrylate***—Weigh accurately about 1.0 g of Ethyl Methacrylate·Trimethylammoniumethyl Methacrylate Chloride Copolymer, transfer into 8 mL of acetone, shake to dissolve, then add acetone to make 10.0 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of ethyl methacrylate RS, dissolve in acetone to make 100.0 mL, and use this solution as the standard solution. Perform the test with 1 μL each of the test solution and the standard solution as directed under the Gas Chromatography according to the following conditions; the peak height of ethyl methacrylate obtained from the test solution is less than the peak height of ethyl methacrylate obtained from the standard solution.

Operating conditions

Detector: A flame ionization detector

Column: A stainless steel column about 3 mm in internal diameter and about 2 m in length, packed with diatomaceous earth for gas chromatography, coated with polyethylene glycol 20 M for gas chromatography at the ratio of 20% (177 μm to 297 μm in particle diameter).

Column temperature: A constant temperature of about 90 ℃.

Carrier gas: Nitrogen

Flow rate: Adjust the flow rate so that the retention time of ethyl methacrylate is about 4 minutes.

Detection sensitivity: Adjust the sensitivity so that the peak height of ethyl methacrylate obtained from the standard solution is 2 cm.

Loss on drying NMT 5.0% (1 g, 105 ℃, 4 hours).

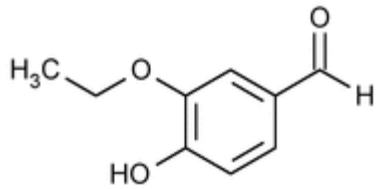
Residue on ignition NMT 0.2% (1 g).

Assay Weigh accurately about 0.2 g of Ethyl Methacrylate·Trimethylammoniumethyl Methacrylate Chloride Copolymer, previously dried, and perform the test as directed under the Nitrogen Determination (Semimicro-Kjeldahl Method).

Each mL of 0.005 mol/L sulfuric acid VS
= 0.1401 mg of N

Packaging and storage Preserve in tight containers.

Ethyl Vanillin
에틸바닐린



$C_9H_{10}O_3$: 166.17

3-Ethoxy-4-hydroxybenzaldehyde [*121-32-4*]

Ethyl Vanillin, when dried, contains NLT 98.0% and NMT 101.0% of ethyl vanillin ($C_9H_{10}O_3$).

Description Ethyl Vanillin occurs as a white to pale yellow crystal and has an odor and taste similar to vanillin.
It is sensitive to light.
An aqueous solution of Ethyl Vanillin is acidic.
It is freely soluble in ethanol, chloroform, ether or alkaline solution and sparingly soluble in 50 ℃ water.

Identification (1) Determine the absorption spectra of solutions of Ethyl Vanillin and ethyl vanillin RS in methanol (1 in 125000) as directed under the Ultraviolet-visible Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavelengths.

(2) Determine the infrared spectra of Ethyl Vanillin and ethyl vanillin RS, previously dried, as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

Melting point Between 76 ℃ and 78 ℃.

Loss on drying NMT 1.0% (phosphorus pentoxide, 4 hours).

Residue on ignition NMT 0.1% (1 g).

Assay Weigh accurately 300 mg of Ethyl Vanillin, previously dried, dissolve in 50 mL of dimethylformamide, add thymol blue TS, and titrate with 0.1 mol/L sodium methoxide VS (Titrimetry). Perform a blank test in the same manner and make any necessary correction.

Each mL of 0.1 mol/L sodium methoxide VS
= 16.62 mg of $C_9H_{10}O_3$

Packaging and storage Preserve in light-resistant, tight containers.

Ethylcellulose
에틸셀룰로오스

[*9004-57-3*]

Ethylcellulose is partly *O*-ethylated cellulose.

Ethylcellulose contains NLT 44.0% and NMT 51.0% of ethoxy group ($-OC_2H_5$: 45.06), calculated on the dried basis.

A suitable antioxidant may be added to Ethylcellulose.

The label of Ethylcellulose indicates its nominal viscosity in millipascal seconds (mPa·s).

Description Ethylcellulose occurs as a white to yellowish white powder or grains.
It is soluble in dichloromethane.
Add 100 mL of warm water to 1 g of Ethylcellulose, shake to make it turbid, cool down to room temperature, and add freshly boiled and cooled water to make 100 mL; the resulting solution is neutral.

Identification Dispense 2 drops each of a dichloromethane solution (1 in 25) of Ethylcellulose and ethylcellulose RS between a sodium chloride plates. Then, remove one plate, evaporate the solvent, and determine the absorption spectra of these solutions as directed under the Liquid film method in the Mid-infrared Spectroscopy; both exhibit similar intensities of absorption at the same wavenumbers.

Viscosity Weigh accurately an amount of Ethylcellulose equivalent to 5.00 g calculated on the dried basis, add it to 95 g of a mixture of 80 g of toluene and 20 g of ethanol (95), and shake to mix. Perform the test with the resulting solution according to Method 1 at 25 ℃; those with a labeled viscosity more than 6 mPa·s are 80.0% to 120.0% of the labeled viscosity, and those with a labeled viscosity of NMT 6 m Pa·s are 75.0% to 140.0% of the labeled viscosity.

Purity (1) ***Acidity or alkalinity***—Add 25 mL of freshly boiled and cooled water to 0.5 g of Ethylcellulose, shake for 15 minutes to mix, and filter the mixture through a 16 - 40 μm glass filter. Use the filtrate as the test solution. Add 0.1 mL of phenolphthalein TS (1 in 10) and 0.5 mL of 0.01 mol/L sodium hydroxide solution to 10 mL of the test solution; the resulting solution exhibits a pale red color. Add 0.1 mL of methyl red-sodium hydroxide TS and 0.5 mL of 0.01 mol/L hydrochloric acid to 10 mL of the test solution; the resulting solution exhibits a red color.

Methyl red-sodium hydroxide TS—Dissolve 50 mg of methyl red in a mixture of 1.86 mL of 0.1 mol/L sodium hydroxide solution and 50 mL of ethanol (95) and add water to make 100 mL.

(2) ***Chloride***—Add 0.250 g of Ethylcellulose to 50 mL of water and boil with occasional shaking. Allow the mixture to stand to cool down and filter. Discard the first 10 mL of the filtrate, take 10 mL of the subsequent filtrate, add water to make 15 mL, and use this solution as the test solution. Separately, pipet 10 mL of standard chloride solution, add 5 mL of water, and use this solution as the control solution. Add 1 mL of 2 mol/L nitric acid solution to 15 mL of the test solution and the control solution, then place each in a test tube containing 1 mL of silver nitrate solution (17 in 1000), and allow it to stand for 5 minutes, protected from light. Compare the turbidity by observing from the side of the test tube against a black background; the turbidity of the test solution is not more intense than that of the control solution (NMT 0.1%).

(3) ***Acetaldehyde***—Add 3.0 g of Ethylcellulose into a 250-mL stoppered Erlenmeyer flask, add 10 mL of water, and mix for 1 hour. Allow the mixture to stand for 24 hours, and then filter. Add water to the filtrate to make 100 mL and use this solution as the test solution. Separately, weigh 1.0 g of acetaldehyde RS and dissolve in water to make 100 mL. To 5 mL of this solution, add water to make 500 mL. To 3 mL of this solution, add water to make 100 mL, and use this solution as the control solution. Pipet 5 mL each of the test solution and the control solution, add 5 mL of an aqueous solution of 3-methyl-2-benzothiazolone hydrazone hydrochloride monohydrate (1 in 2000), and heat the mixture on a water bath at 60°C for 5 minutes. Add 2 mL of Iron(III) chloride-ammonium sulfate TS, heat again at 60°C for 5 minutes, cool, then add water to make 25 mL, and compare the colors of the test solution and the control solution; the color of the test solution is not more intense than the control solution (NMT 100 ppm).

Loss on drying NMT 3.0% (1 g, 105 ℃, 2 hours).

Residue on ignition NMT 0.5% (1.0 g).

Assay Weigh accurately 30 mg of Ethylcellulose and add it to a 5-mL pressure-resistant serum vial. Add exactly 60 mg of adipic acid, 2 mL of the internal standard solution, and 1 mL of hydroiodic acid, then immediately fix the vial with a septum stopper coated with fluorine resin and an aluminum cap, or use a stopper with equivalent air-tightness, and weigh accurately the mass of the vial. Be careful not to mix the contents of the vial before heating. Heat the block until the internal temperature of the vial is 115 ± 2 °C, and stir for 70 minutes using a magnetic stirrer or shaker provided with the heater. After cooling, weigh accurately the mass of the vial. If the difference in mass before and after heating exceeds 10 mg, this solution should not be used in the test. If the difference in mass before and after heating is NMT 10 mg, the phase is separated. Then, using a cooled syringe, draw a sufficient amount of the upper layer through the septum stopper of the vial and use it as the test solution. Separately, pipet 60 mg of adipic acid, 2 mL of the internal standard solution, and 1 mL of hydroiodic acid into a pressure-resistant serum vial, seal it immediately, and weigh accurately the mass of the vial. Then, using a syringe, inject 25 μL of iodoethane RS through the septum stopper, and weigh accurately the mass of the vial. After shaking well to separate the phases, use a cooled syringe to draw a sufficient amount of the upper layer through the septum stopper of the vial to use it as the standard solution. Perform the test with 1 μL each of the test solution and standard solution as directed under the Gas Chromatography according to the following conditions, and determine the ratios, Q_T and Q_S, of the peak area of the iodoethane to that of the internal standard.

$$\text{Amount (\%) of ethoxy group } (C_2H_5O) = (W_S/W_T) \times (Q_T/Q_S) \times 28.89$$

W_S: Amount (mg) of iodoethane taken for assay
W_T: Amount (mg) of Ethylcellulose taken, calculated on the dried basis

Internal standard solution—A solution of *n*-octane in o-xylene (1 in 200).

Operating conditions
Detector: A flame ionization detector
Column: A fused silica capillary column about 0.53 mm in internal diameter and about 30 m in length, coated inside with poly(dimethyl)siloxane for gas chromatography 3 μm in thickness.
Column temperature: Maintain the temperature at 50 ℃ for the first 3 minutes, then raise the temperature at the rate of 10 ℃ per minute up to 100 ℃, then raise at the rate of 35 ℃ per minute up to 250 ℃, and maintain at 250 ℃ for 8 minutes.
Sample injection port temperature: A constant temperature of about 250 ℃.
Detector temperature: A constant temperature of about 280 ℃.
Carrier gas: Helium
Flow rate: 4.2 mL/min
Split ratio: About 1 : 40
System suitability
System performance: Proceed with 1 μL of the standard solution according to the above conditions; iodoethane and the internal standard are eluted in this order, and the relative retention time of iodoethane to the internal standard is about 0.6 with the resolution between these peaks being NLT 5.0.
System repeatability: Repeat the test 6 times according to the above conditions with 1 μL each of the standard solution; the relative standard deviation of the peak area ratio of iodoethane to that of the internal standard is NMT 2.0%.

Packaging and storage Preserve in well-closed containers.

Ethylenediamine
에틸렌디아민

$H_2NCH_2CH_2NH_2$

$C_2H_8N_2$: 60.10

Ethane-1,2-diamine [*107-15-3*]

Ethylenediamine contains NLT 97.0% and NMT 101.0% of ethylenediamine ($C_2H_8N_2$).

Description Ethylenediamine occurs as a clear, colorless to pale yellow liquid and has a characteristic odor like ammonia.
It is miscible with water, ethanol or ether.
It is corrosive and has an irritant property.
It changes slowly when exposed to air.

Specific gravity d_{20}^{20}: About 0.898.

Identification (1) The aqueous solution of Ethylenediamine (1 in 500) is alkaline.

(2) Add 2 drops of Ethylenediamine to 2 mL of cupric sulfate TS and shake to mix; the resulting solution exhibits a bluish purple color.

(3) To 40 mg of Ethylenediamine, add 6 drops of benzoyl chloride and 2 mL of sodium hydroxide solution (1 in 10), and warm for 2 to 3 minutes with occasional shaking. Filter the white precipitate formed and wash with water. Dissolve the precipitate in 8 mL of ethanol by warming. Promptly, add 8 mL of water, cool, filter the crystals, wash with water, and dry at 105 °C for 1 hour; the melting point of the crystals is between 247 °C and 251 °C.

Purity (1) ***Heavy metals***—Weigh 1.0 g of Ethylenediamine and place into a crucible, evaporate to dryness on a water bath, loosely cover it with a lid, and heat gently to carbonize. Proceed according to Method 2 and perform the test. Prepare the control solution by adding 2.0 mL of the lead standard solution (NMT 20 ppm).

(2) ***Residue on evaporation***—Pipet 5 mL of Ethylenediamine, evaporate to dryness on a water bath, and dry the residue at 105 ºC to a constant mass; the amount is NMT 3.0 mg.

Distilling range Between 81 °C and 119 °C, NLT 95 vol%.

Assay Weigh accurately about 0.7 g of Ethylenediamine in a stoppered Erlenmeyer flask containing 25 mL of water, add 50 mL of water and titrate with 1 mol/L hydrochloric acid VS (indicator: 3 drops of bromphenol blue TS).

Each mL of 1 mol/L hydrochloric acid VS
= 30.049 mg of $C_2H_8N_2$

Packaging and storage Preserve in light-resistant, tight containers, almost filled to the full.

Eucalyptus Oil
유칼리유

Eucalyptus Oil is the essential oil obtained by steam distillation of the leaves of *Eucalyptus globulus* Labillardiere or related plants (Myrtaceae). Eucalyptus Oil contains NLT 70.0% of cineole ($C_{10}H_{18}O$: 154.25).

Description Eucalyptus Oil occurs as a clear, colorless or pale yellow liquid and has a characteristic, aromatic odor and pungent taste.
It is neutral.

Identification To 1 mL of Eucalyptus Oil, add 1 mL of phosphoric acid, shake vigorously, and allow the mixture to stand; the solution congeals within 30 minutes.

Refractive index n_D^{20}: Between 1.458 and 1.470.

Specific gravity d_{20}^{20}: Between 0.907 and 0.927.

Purity (1) ***Clarity and color of solution***—Mix 1.0 mL of Eucalyptus Oil with 5 mL of diluted ethanol (7 in 10); the solution is clear.

(2) ***Heavy metals***—Weigh 1.0 g of Eucalyptus Oil and perform the test according to Method 2. Prepare the control solution by adding 4.0 mL of the lead standard solution (NMT 40 ppm).

(3) ***Phenol***—To 5 mL of Eucalyptus Oil, add 5 mL of sodium hydroxide TS and shake to mix; the volume of Eucalyptus Oil does not decrease.
To 1 mL of Eucalyptus Oil, add 20 mL of water, shake well and allow the mixture to stand to separate the layers. Collect 10 mL of the aqueous layer and add 1 drop of ferric chloride TS; the resulting solution does not exhibit a violet color.

(4) ***Aldehyde***—Place 10 mL of Eucalyptus Oil into a glass-stoppered test tube (25 mm × 150 mm). Add 5 mL of toluene and 4 mL of alcoholic hydroxylamine solution, shake vigorously and titrate with a 0.5 mol/L solution of potassium hydroxide in ethanol VS (60 v/v% ethanol) until the color of the solution changes from red to yellow. The endpoint of the titration is when the pale yellow color of the indicator persists in the lower layer even after 2 minutes of vigorous shaking. The reaction is completed within about 15 minutes. Add another 10 mL of Eucalyptus Oil and repeat the titration. To the solution from the first titration, add 0.5 moL of a 0.5 mol/L solution of potassium hydroxide in ethanol (60 v/v% ethanol), use this solution as the control solution with respect to the endpoint, and repeat the titration. In the second titration, NMT 2.0 mL of a 0.5 mol/L solution of potassium hydroxide in ethanol (60 v/v% ethanol) VS is consumed.

Assay Weigh accurately about 0.1 g of Eucalyptus Oil and dissolve in hexane to make exactly 25 mL. Pipet 5 mL of this solution, add exactly 5 mL of the internal standard solution, add hexane again to make exactly 100 mL, and use this solution as the test solution. Separately, weigh accurately about 0.1 g of cineole RS and proceed in the same manner as in the preparation of the test solution, and use this solution as the standard solution. Perform the test with 2 μL of the test solution and the standard solution as directed under the Gas Chromatography according to the following conditions, and calculate the peak area ratios, Q_T and Q_S, of cineole to that of the internal standard in each solution.

$$\text{Amount (mg) of cineole } (C_{10}H_{18}O) = \text{Amount (mg) of cineole RS} \times \frac{Q_T}{Q_S}$$

Internal standard solution—A solution of anisole in hexane (1 in 250).

Operating conditions

Detector: A flame ionization detector

Column: A column about 3 mm in internal diameter and about 5 m in length, packed with alkylene glycol phthalic acid ester for gas chromatography coated at a ratio of 10% on silanized diatomaceous earth for gas chromatography for gas chromatography (150 μm to 180 μm in particle diameter).

Column temperature: A constant temperature of about 120 °C.

Carrier gas: Nitrogen

Flow rate: Adjust the flow rate so that the retention time of cineole is about 11 minutes.

System suitability

System performance: Dissolve 0.1 g each of cineole and limonene in 25 mL of hexane. Pipet 1 mL of this solution, and add hexane to make 20 mL. Proceed with about 2 μL of this solution according to the above operating conditions; limonene and cineole are eluted in this order with the resolution being NLT 1.5.

Packaging and storage Preserve in light-resistant, tight containers.

Eudragit
유드라짓

Eudragit is an ethyl methacrylate-trimethylammoniumethyl methacrylate chloride copolymer, of which ratio (X : Y) is about (35 : 1) and of which weight ratio is about (19 : 1) with the average molecular weight of 30,000 to 50,000.
Eudragit contains NLT 0.27% and NMT 0.40% of nitrogen (N:14.01), calculated on the dried basis.

Description Eudragit occurs as a white solid. It is odorless or has a slight, characteristic odor and is tasteless.
It is freely soluble in acetone or in chloroform and practically insoluble in ethanol or in ether.
It is practically insoluble in water.

Identification To 0.3 g of Eudragit, add 10 mL of carbon tetrachloride, shake well to mix, allow to stand for about 20 minutes, and use this solution as the test solution. Determine the infrared spectrum of the test solution as directed in the solution method under the Mid-infrared Spectroscopy; it exhibits absorption at the wavenumbers of about 1145 cm^{-1}, 1170 cm^{-1}, 1235 cm^{-1} and 1380 cm^{-1} and between 1435 cm^{-1} and 1480 cm^{-1}, between 1725 cm^{-1} and 1735 cm^{-1}, and between 2925 cm^{-1} and 2980 cm^{-1}.

Purity (1) ***Soluble substances***—Dissolve 2.0 g of pulverized Eudragit in 100 mL of water, shake well to mix until it becomes a homogeneous suspension, and filter. Take 25 mL of the filtrate, evaporate to dryness on a water bath, and then dry at 105°C for 16 hours; the mass of the residue does not exceed 2.5 mg (NMT 0.5%).

(2) ***Heavy metals***—Proceed with 2.0 g of Eudragit according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 10 ppm).

(3) ***Arsenic***—Proceed with 1.0 g of Eudragit according to Method 3 and perform the test (NMT 2 ppm).

Loss on drying NMT 2.0% (2 g, 105 ℃, 16 hours).

Residue on ignition NMT 0.15% (1 g)

Assay Weigh accurately about 0.2 g of Eudragit and perform the test as directed under the Nitrogen Determination (Semimicro-Kjeldahl Method).

Each mL of 0.005 mol/L sulfuric acid VS
= 0.1401 mg of N

Packaging and storage Preserve in well-closed containers.

Exsiccated Gypsum
소석고

Exsiccated Gypsum has a composition roughly corresponding to $CaSO_4 \cdot ½H_2O$.

Description Exsiccated Gypsum occurs as a white to grayish white powder, which is odorless and tasteless.
It is slightly soluble in water and practically insoluble in ethanol.
It absorbs moisture slowly on standing in air to lose its solidifying property.
When Exsiccated Gypsum is heated to yield an anhydrous compound at a temperature above 200 °C; it loses its solidifying property.

Identification To 1 g of Exsiccated Gypsum, add 20 mL of water, shake for 5 minutes to mix, and filter. The filtrate responds to the Chemical identification reactions (2) and (3) for calcium salt and to the Chemical identification reactions for sulfate.

Purity ***Alkali—***Transfer 3.0 g of Exsiccated Gypsum into a stoppered test tube, add 10 mL of water and 1 drop of phenolphthalein TS, and shake vigorously to mix; the solution does not exhibit a red color.

Solidification Add 10 mL of water to 10.0 g of Exsiccated Gypsum, immediately stir for 3 minutes, and allow to stand; the time taken until no water comes out upon pressing with a finger is within 10 minutes from the time when water was first added.

Packaging and storage Preserve in tight containers.

Fennel Oil
회향유

Oleum Foeniculi

Funnel Oil is the essential oil distilled with steam from the fruit of *Foeniculum vulgare* Miller (*Umbelliferae*) or *Illicium verum* Hooker fil. (*Illiciaceae*).

Description Fennel Oil is colorless to pale yellow liquid and has a characteristic odor and sweet initial taste with slightly bitter aftertaste.
It is miscible with ethanol or ether.
It is practically insoluble in water.
When cold, white crystals or crystalline masses may often separate from Fennel Oil.

Identification Dissolve 0.30 g of Fennel Oil in 20 mL of hexane. Pipet 1 mL of this solution, add hexane to make exactly 10 mL, and use this solution as the test solution. With this solution, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL of the test solution on a thin-layer chromatographic plate made of silica gel for thin-layer chromatography (with fluorescent indicator). Develop the plate with a mixture of hexane and ethyl acetate (20 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength : 254 nm); a spot with a dark purple color appears at the R_f value of about 0.4

Refractive index n_D^{20}: Between 1.528 and 1.560.

Specific gravity d_{20}^{20}: Between 0.955 and 0.995.

Purity (1) ***Clarity and color of solution***—To 1.0 mL of Fennel Oil, add 3 mL of ethanol; the solution is clear. To this solution, add 7 mL of ethanol; the solution remains unchanged.

(2) ***Heavy metals***—Proceed with 1.0 mL of Fennel Oil according to Method 2 and perform the test. Prepare the control solution with 4.0 mL of lead standard solution (NMT 40 ppm).

Packaging and storage Preserve in light-resistant, tight containers.

Formalin
포르말린

Formalin contains NLT 35.0% and NMT 38.0% of formaldehyde (CH_2O : 30.03). Formalin contains 5% to 13% of methanol to prevent polymerization.

Description Formalin occurs as a colorless, clear liquid and its vapor is irritating to the mucous membrane.
It is miscible with water or ethanol (95).
When stored for a long time, especially in a cold place, Formalin may become cloudy.

Identification (1) To 2 mL of Formalin, add 10 mL of water and 1 mL of silver nitrate-ammonia TS: a gray precipitate is produced, or a silver mirror is formed on the wall of the test tube.

(2) Add 2 drops of Formalin to a solution obtained from dissolving 0.1 g of salicylic acid in 5 mL of sulfuric acid and warm the mixture; the solution exhibits a persistent, dark red color.

Purity (1) ***Acid***—To 20 mL of Formalin, add 20 mL of water and add 5.0 mL of 0.1 mol/L sodium hydroxide and 2 drops of bromothymol blue TS: the solution exhibits a blue color.

(2) ***Methanol***—Pipet 10.0 mL of Formalin, add exactly 10.0 mL of the internal standard solution, add water to make exactly 100 mL, and use this solution as the test solution. Separately, pipet 1.0 mL of methanol, add exactly 10.0 mL of the internal standard solution, add water to make exactly 100 mL and use this solution as the standard solution. Perform the test with 1 μL each of the test solution and the standard solution as directed under the Gas Chromatography according to the following operating conditions and determine the peak area ratios, Q_T and Q_S, of methanol to the peak area of the internal standard (5% to 13%).

$$\text{Content (\%) of methanol} = \frac{Amount\ of\ methanol \times \frac{Q_T}{Q_S}}{Amount\ of\ sample} \times 100$$

Internal standard solution—Dilute 10 mL of anhydrous ethanol with water to make 100 mL.

Operating conditions

Detector: A flame ionization detector

Column: A column 2 mm to 4 mm in internal diameter and 1.5 m to 2.0 m in length, packed with ethylbenzene-divinylbenzene copolymer for gas chromatography (150 μm to 180 μm in particle diameter).

Column temperature: 120 °C

Sample injection port temperature: 150 °C

Carrier gas: Nitrogen

Flow rate: 30 to 40 mL/min

System suitability

System performance: Proceed with 1 μL of the standard solution according to the above operating conditions; the resolution between the peaks of methanol and ethanol is NLT 2.0.

Residue on ignition NMT 0.06 w/v% (5 mL, after evaporation).

Assay Weigh accurately the mass of a weighing bottle containing 5 mL of water, add about 1 g of Formalin and weigh accurately again. Add water to make exactly 100 mL. Pipet 10 mL of this solution, add exactly 50 mL of 0.05 mol/L iodine VS and 20 mL of potassium hydroxide TS and allow to stand for 15 minutes at an ordinary temperature. To this mixture, add 15 mL of dilute sulfuric acid and titrate the excess iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 1 mL of starch TS). Perform a blank test in the same manner.

Each mL of 0.05 mol/L iodine VS
= 1.5013 mg of CH_2O

Packaging and storage Preserve in light-resistant, tight containers.

High Fructose Corn Syrup
이성화당

High Fructose Corn Syrup is a starch sugar prepared by liquid-saccharification of starch using enzymatic or chemical methods and then isomerization of part of the glucose into fructose, followed by a purification step. High Fructose Corn Syrup contains NLT 55% of fructose ($C_6H_{12}O_6$: 180.16) and NLT 94.0% and NMT 101.0% in total of fructose ($C_6H_{12}O_6$: 180.16) and glucose ($C_6H_{12}O_6$: 180.16), calculated on the dried basis.

Description High Fructose Corn Syrup occurs as a colorless to pale yellow liquid. It is odorless and has a sweet taste.

Identification (1) To 5 drops of an aqueous solution (1 in 20) of High Fructose Corn Syrup, add 5 mL of hot Ferring's TS; a red precipitate of cuprous oxide is formed.

(2) To 1 mL of an aqueous solution of High Fructose Corn Syrup (1 in 100), add 1 mL of 0.1% resorcin-ethanol solution and 6 mL of 30% hydrochloric acid and heat on a water bath for 5 to 15 minutes; the resulting solution exhibits a red color.

(3) The retention time of the major peaks obtained from the test solution and the standard solution obtained in the Assay are the same.

pH Between 4.5 and 6.5 (30% aqueous solution)

Purity (1) ***Oligosaccharides***— Subtract the amount of glucose and fructose from the total amount of sugars and determine the remaining amount as the amount of oligosaccharides; the amount of oligosaccharides is NMT 6.0%.

(2) ***Heavy metals***—Proceed with 5.0 g of High Fructose Corn Syrup according to Method 2 and perform the test. Prepare the control solution with 2.5 mL of lead standard solution (NMT 5 ppm).

(3) ***Arsenic***—Prepare the test by dissolving 6.0 g of High Fructose Corn Syrup in 35 mL of water and perform the test according to Method 1 (NMT 0.33 ppm).

(4) ***Artificial sweeteners***—Weigh 20 g of High Fructose Corn Syrup, add 50 mL of water, transfer into an Erlenmeyer flask, and heat in a boiling water bath for 5 minutes. After cooling, transfer the solution into a separatory funnel, add 10% hydrochloric acid to acidify, and extract twice each time with 50 mL of ethyl acetate. Combine the extracts, wash twice each time with 10 mL of saturated sodium chloride solution, add anhydrous sodium sulfate to dehydrate, and vacuum-concentrate. Dissolve the residue in 0.5 mL of ethanol and use this solution as the test solution. Separately, weigh 100 mg of sodium saccharin RS,

dissolve in 25 mL of water, then add ethanol to make 50 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 1 to 30 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel (with a fluorescent indicator) for thin layer chromatography. Next, develop the plate with a mixture of 1-butanol, ammonia water (9 : 1) as the developing solvent, and dry the plate at 100 °C for 20 minutes. Examine the plate under ultraviolet light (main wavelength: 254 nm); the color and the R_f value of the spot obtained from the test solution are not the same as those of the spot obtained from the standard solution.

(5) ***Tar pigment***—Weigh 20 g of High Fructose Corn Syrup and dissolve in 50 mL of water. Take 5 mL of this solution, add 1 mL of 1% acetic acid and 0.1 g of cotton wool, shake well to mix, warm on a water bath for 30 minutes, and take out the cotton wool. If the cotton wool is not dyed, it is considered undetectable. If the cotton wool is dyed, put this dyed cotton wool in 5 mL of 1% ammonia solution, warm for 30 minutes, add acetic acid to neutralize, and adjust he concentration to about 1%, and use this solution as the test solution. Prepare 0.1% aqueous solutions of each tar pigment (Red No. 2, Red No. 3, Red No. 40, Red No. 102, Red No. 104, Red No. 105, Red No. 106, Yellow No. 4, Yellow No. 5, Green No. 3, Blue No. 1, and Blue No. 2) and use these solutions as the standard solutions. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot the test solution and the standard solutions on the thin-layer chromatographic plate made of silica gel (with fluorescent indicator) for thin layer chromatography. Next, develop the plate with a mixture of ethyl acetate, methanol and ammonia water (28) (3 : 1 : 1) as the developing solvent to a distance of about 13 to 25 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 365 nm); the color and the R_f value of the spot obtained from the test solution are not the same as those of the spot obtained from the standard solution.

Loss on drying Place about 25 g of purified sea sand in an evaporation dish, insert a glass rod, dry at 105 °C to a constant mass, cool, and weigh. Weigh accurately 1 g of High Fructose Corn Syrup, add to this dried sea sand, add 10 mL of water, stir with the glass rod to mix thoroughly, and dry at 105°C for 30 minutes. Then, dry further to a constant mass in a vacuum desiccator (80°C, not exceeding 0.67 kPa, NLT 6 hours). After cooling, weigh the mass and calculate the difference in mass before and after drying (NMT 25.0%).

Residue on ignition NMT 0.1% (5 g, 550 °C, 5 hours).

Assay Weigh accurately about 3.0 g of High Fructose Corn Syrup, add water to make exactly 1000 mL, and use this solution as the test solution. Separately, weigh accurately about 1.0 g each of fructose RS and glucose RS, add water to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 5 μL each of the test solution and the standard solution as directed under the Liquid Chromatography, and determine the peak areas, A_{T1}, A_{T2}, A_{S1} and A_{S2} of fructose and glucose from each solution.

$$\text{Amount (g) of fructose } (C_6H_{12}O_6) = \text{Amount (g) of fructose RS} \times \frac{A_{T1}}{A_{S1}}$$

$$\text{Amount (g) of glucose } (C_6H_{12}O_6) = \text{Amount (g) of glucose RS} \times \frac{A_{T2}}{A_{S2}}$$

Operating conditions

Detector: A differential refractometer

Column: A stainless steel column about 8 mm in internal diameter and about 50 cm in length, packed with gel-typed strongly acidic ion-exchange resin (cross-linking rate: 6%) for liquid chromatography (11 μm in particle diameter).

Column temperature: A constant temperature of about 85 °C.

Mobile phase: Water

Flow rate: 0.5 mL/min

Packaging and storage Preserve in tight containers.

Gelatin
젤라틴

Gelatin is a purified protein obtained from collagen of animals by partial hydrolysis with acid or alkali, enzymatic hydrolysis or thermal hydrolysis. Depending on the hydrolysis conditions, gelling gelatin and non-gelling gelatin can be obtained. The label states a gel strength for the gelling grade and a non-gelling grade for the non-gelling grade.

Description Gelatin is colorless or white to pale yellowish brown sheets, shreds, grains or powder.
It is freely soluble in hot water, and is practically insoluble in ethanol (95).
The gelling grade does not dissolve in water, but when water is added, it slowly swells and softens, absorbing water 5 to 10 times its own mass. The isoelectric point of the gelling type obtained by acid treatment is pH 7.0 to 9.0, and that of the gelling type obtained by alkali treatment is pH 4.5 to 5.0.
Non-gelling grade is freely soluble in water.

Identification (1) Dissolve 1.00 g of Gelatin in water, freshly boiled and cooled to 55 °C, to make 100 mL, and use this solution as the test solution. To 2 mL of the test solution, maintained at 55 °C, add 0.05 mL of copper(II) sulfate TS, shake to mix, and add 0.5 mL of 2 mol/L sodium hydroxide TS; the resulting solution exhibits a violet color.

(2) Place 0.5 g of Gelatin in a test tube with an internal diameter of about 15 mm, add 10 mL of water, and allow to stand for 10 minutes. Heat at 60°C for 15 minutes, and allow to stand the test tube upright at 2 °C to 8 °C for 6 hours. Invert the test tube; the contents do not flow out immediately for the gelling grade, but the contents flow out immediately for the non-gelling grade.

(3) Apply to the non-gelling grade. Transfer 0.5 g of Gelatin into a 250-mL flask, and add 10 mL of water and 5 mL of sulfuric acid. Cover with a watch glass or other means, avoiding complete closure, and heat at 105°C for 4 hours. After cooling, add 200 mL of water, and adjust the pH to 6.0 to 8.0 with sodium hydroxide solution (1 in 5). Transfer 2 mL of this solution to a test tube, add 2 mL of oxidizing agent, shake to mix, and allow to stand for 20 minutes. Add 2 mL of coloring TS, shake to mix, and warm in a 60°C water bath for about 15 minutes; the solution exhibits a red to violet color.

Oxidizing agent—Dissolve 1.4 g of sodium toluensulfonchloramide trihydrate in a solution prepared by dissolving 5.53 g of dibasic sodium phosphate dodecahydrate and 0.48 g of citric acid monohydrate in water to make 100 mL. Prepare before use.

Coloring TS—Dissolve 1.0 g of 4-dimethylaminobenzaldehyde in 3.5 mL of perchloric acid solution (1 in 2), and slowly add 6.5 mL of 2-propanol. Prepare before use.

Gell strength (Bloom value) Apply to the gelling grade. Determine the mass (g) necessary to produce the force which, applied to a plunger 12.7 mm in diameter, makes a depression 4 mm deep at 10 °C in a gel prepared with a 6.67% solution of Gelatin.

(1) ***Apparatus and device*** Use a physical property tester such as a texture analyzer or gelometer with a cylindrical piston 12.7 ± 0.1 mm in diameter with a very flat bottom As a container, use a bottle (gel cup) with an internal diameter of 59 ± 1 mm and a height of 85 mm.

(2) ***Procedure*** Transfer 7.5 g of Gelatin into a gel cup, add 105 mL of water, stopper the cup, and allow to stand for 4 hours. Then, warm in a 65 ± 2 ℃ water bath while mixing slowly with a glass rod for 15 minutes. Incorporate any water condensed at the top of the bottle to the solution to make the solution uniform, and allow to stand at room temperature for 15 minutes. Place the bottle on a completely horizontal test platform in a thermostatic bath at 10.0 ± 0.1°C, cover the bottle with a stopper, and allow to stand for 17 ± 1 hours. Take the bottle out of the thermostatic bath, immediately wipe off the water on the outside of the bottle, and place the bottle on the physical property tester (gelometer). Adjust the position of the bottle so that the tip of the plunger touches the center of the gel surface as much as possible. Then perform the test with an entry distance of 4 mm and an entry speed of 0.5 mm/sec; the gel strength is 80% to 120% of the labeled value.

pH The pH of the test solution in the Identification (1) measured at 55 °C is between 3.8 and 7.6.

Purity (1) ***Iron***—Transfer 5.00 g of Gelatin to a stoppered flask, add 10 mL of hydrochloric acid, stopper the flask, and heat on a water bath at 75 °C to 80 °C for 2 hours. If necessary, to ensure proper dissolution, add hydrochloric acid and allow to stand until the gelatin swells to extend the heating time or increase the heating temperature. After cooling, add water to make the content of the flask to 100.0 g, and use this solution as the test solution. Separately, transfer 5.00 g each of Gelatin into three stoppered flasks, proceed in the same manner as in the preparation of the test solution. Add accurately 10 mL, 20 mL and 30 mL of iron standard solution for Atomic absorption spectroscopy, add water to make the content of each flask to 100.0 g, and use these solutions as the standard solutions. The amount of the standard solution can be adjusted appropriately depending on the sensitivity of the apparatus. Perform the test with the test solution and the standard solutions as directed in the standard addition method under the Atomic absorption spectroscopy (NMT 30 ppm).

Gas: Acetylene- Air
Lamp: Iron hollow cathode lamp
Wavelength: 248.3 nm

Iron standard solution for Atomic absorption spectroscopy—Pipet 2 mL of standard iron stock solution, and add water to make 250 mL. Add water to 10 mL of this solution to make exactly 100 mL. Prepare before use. Each mL of this solution contains 8 μg of iron.

Standard iron stock solution—Weigh accurately 4.840 g of iron(III) chloride hexahydrate, and dissolve in diluted hydrochloric acid (9 in 25) to make exactly 100 mL.

(2) ***Chromium***—Use the test solution of (1) as the test solution. Separately, transfer 5.00 g each of Gelatin in three stoppered Erlenmeyer flasks and proceed in the same manner as in the preparation of the test solution. Add exactly 0.25 mL, 0.50 mL and 0.75 mL of standard chromium solution for Atomic absorption spectroscopy, respectively, and add water to make the content exactly 100.0 g, and use these solutions as the standard solutions. The amount of the standard solution can be adjusted appropriately depending on the sensitivity of the apparatus. Perform the test using the test solution and the standard solutions as directed in the standard addition method under the Atomic absorption spectroscopy according to the following conditions (NMT 10 ppm).

Gas: Acetylene- Air
Lamp: Chromium hollow cathode lamp
Wavelength: 357.9 nm

Standard chromium solution for Atomic absorption spectroscopy—Weigh accurately 0.283 g of potassium dichromate, and add water to make exactly 1000 mL. Each mL of this solution contains 0.1 mg of chromium (Cr).

(3) ***Zinc***—Use the test solution of (1) as the test solution. Separately, transfer 5.00 g each of Gelatin to three stoppered Erlenmeyer flasks and proceed in the same manner as in the preparation of the test solution. Add exactly 7.5 mL, 15 mL and 22.5 mL of zinc standard solution for Atomic absorption spectroscopy, respectively, and add water to make the content to exactly 100.0 g and use these solutions as the standard solutions. The amount of the standard solution can be adjusted appropriately depending on the sensitivity of the apparatus. Perform the test using the test solution and the standard solutions as directed in the standard addition method under the Atomic absorption spectroscopy according to the following conditions (NMT 30 ppm).

Gas: Acetylene- Air
Lamp: A zinc hollow cathode lamp
Wavelength: 213.9 nm

(4) ***Peroxide***—Peroxidase acts on peroxide to transfer the oxygen atom to the reduced organic redox indicator, changing the indicator to the blue oxidized form. The intensity of the color produced is proportional to the amount of peroxide. Compare the color that appears on the peroxide test strip from this reaction with the peroxide colorimetric table to determine the peroxide concentration of the test solution.

Procedure Transfer 20.0 ± 0.1 g of Gelatin into a beaker, add 80.0 ± 0.2 mL of water, shake to moisten, and allow to stand at room temperature for 1 to 3 hours. Cover the beaker with a watch glass, dissolve by warming on a water bath at 65 ± 2 °C for 20 ± 5 minutes. Then, mix with a glass rod to obtain a homogeneous solution, and use this solution as the test solution. Dip the peroxide test strip into this solution for 1 second to properly wet the reaction zone ofthe strip. Take out the strip, shake off excess liquid, and after 15 seconds, compare the color shown in the reaction zone of the strip with that of the peroxide colorimetric table. Read the concentration corresponding to the best matching color in the peroxide colorimetric table. Multiply the concentration by the correction factor 5 to determine the peroxide concentration in the test substance (NMT 10 ppm).

Peroxide test strip—A strip which can measure peroxide

from 0 ppm to 25 ppm and is good for the following suitability test.

Suitability test—Take exactly 10 mL of standard hydrogen peroxide solution, and add water to make exactly 300 mL. Take exactly 2 mL of this solution, and add water to make exactly 1000 mL (2 ppm). Dip the peroxide test strip into this solution for 1 second to properly wet the reaction zone ofthe strip. Take out the strip, shake off excess liquid, and after 15 seconds, compare the color shown in the reaction zone of the test strip with that of the peroxide colorimetric table; it is equivalent to the color in the colorimetric table where the concentration of peroxide is 2 ppm.

Standard hydrogen peroxide stock solution—Take an appropriate amount of hydrogen peroxide (30), and add water to make a solution containing 0.30 g of hydrogen peroxide per 1 mL. Pipet 1 mL of this solution, and add water to make exactly 10 mL. Take exactly 1 mL of this solution and 10 mL of dilute sulfuric acid, transfer them to a flask containing 10 mL of water, and titrate with 0.02 mol/L potassium permanganate VS. However, the endpoint of titration is when the color of the liquid appears pale red. Perform a blank test in the same manner and make any necessary correction.

Each mL of potassium permanganate TS
= 1.701 mg of H_2O_2

Standard hydrogen peroxide solution—Take exactly 10 mL of standard stock solution of hydrogen peroxide, and add water to make exactly 100 mL. Prepare before use. Each mL of this solution contains 30 mg of hydrogen peroxide.

(5) ***Sulfur dioxide*—**It is NMT 50 ppm

Conductivity Perform the test at 30 ± 1.0 ℃ with the test solution in the Identification (1); it is NMT 1 $mS \cdot cm^{-1}$. However, temperature calibration is not performed.

Loss on drying NMT 15.0% (5.0 g, 105 °C, 16 hours)

Microbial limit Perform the microbial limit test; acceptance criteria of the total aerobic microbial count is NMT 1000 CFU and the total number of fungi is NMT 100 CFU per 1 g of Gelatin. No escherichia coli (E.coli) or salmonella is detected.

Packaging and storage Preserve in tight containers.
Store away from heat and moisture.

Purified Gelatin
정제젤라틴

Purified Gelatin is a purified protein obtained from collagen of animals by partial hydrolysis with acid or alkali, enzymatic hydrolysis or thermal hydrolysis. Gelling gelatin or non-gelling gelatin can be produced. The label states a gel strength for the gelling gelatin and a non-gelling gelatin for the non-gelling gelatin.

Description Purified Gelatin is colorless or white to pale yellowish brown sheets, shreds, grains or powder, and is odorless and tasteless.
It is freely soluble in hot water and is practically insoluble in ethanol (95).
The gelling gelatin does not dissolve in water, but when water is added, it slowly swells and softens, absorbing water 5 to 10 times its own mass.
Non-gelling gelatin is freely soluble in water.

Identification (1) To 5 mL of an aqueous solution of Purified Gelatin (1 in 100), spot chromium trioxide TS or picric acid TS; a precipitate is formed.

(2) To 5 mL of an aqueous solution of Purified Gelatin (1 in 5000), add tannic acid TS; the solution turns turbid.

(3) Weigh 0.5 g of Purified Gelatin, transfer into a test tube with an internal diameter of about 15 mm, add 10 mL of water, and allow to stand for 10 minutes. Then heat at 60 °C for 15 minutes, and allow to stand at 0 °C for 6 hours. Invert the test tube; the gelling gelatin does not flow out immediately, but the non-gelling gelatin flows out immediately.

pH Dissolve 1.0 g of Purified Gelatin in freshly boiled and cooled water at 55 °C, add freshly boiled and cooled water to make 100 mL; the pH of the solution measured at 55 °C is 3.8 to 9.0.

Purity (1) ***Heavy metals*—**Proceed with 1.0 g of Purified Gelatin according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 20 ppm).

(2) ***Iron*—**Weigh accurately about 5.0 g of Purified Gelatin, transfer into an Erlenmeyer flask, and add 10 mL of hydrochloric acid. Stopper the flask, and heat on a water bath at 75 °C to 80 °C for 2 hours. If necessary, extend the heating time or heat at a higher temperature to allow Purified Gelatin to swell. After cooling the solution, add water to make exactly 100.0 g, and use this solution as the test solution. Separately, take about 5.0 g each of Purified Gelatin, transfer into three Erlenmeyer flasks, respectively, proceed in the same manner as in the preparation of the test solution. Then add accurately 10 mL, 20 mL and 30 mL of iron standard solution for Atomic absorption spectroscopy, respectively, add water to make 100.0 g each, and use these solutions as the standard solutions. The amount of the standard solution can be adjusted appropriately depending on the sensitivity of the apparatus. Perform the test using the test solution and the standard solutions as directed in the standard addition method under the Atomic absorption spectroscopy according to the following conditions (NMT 30 ppm).

Gas: Dissolved acetylene – Air
Lamp: A Iron hollow cathode lamp
Wavelength: 248.3 nm

Iron standard solution for Atomic absorption spectroscopy—Take 2 mL of standard iron stock solution, and add water to make 250 mL. Add water to 10 mL of this solution to make exactly 100 mL. Each mL of this solution contains 8 μg of iron.

Standard iron stock solution—Weigh accurately 4.840 g of iron(III) chloride hexahydrate, and dissolve in diluted hydrochloric acid (9 in 25) to make exactly 100 mL.

(3) ***Chromium*—**Weigh accurately about 5.0 g of Purified Gelatin, transfer into an Erlenmeyer flask, and add 10 mL of hydrochloric acid. Stopper the flask, and heat on a water bath at 75 °C to 80 °C for 2 hours. If necessary, extend the heating time or heat at a higher temperature to allow Purified Gelatin to swell. After cooling the solution, add water to make exactly 100.0 g, and use this solution as the test solution. Separately, take about

5.0 g each of Purified Gelatin, transfer into three Erlenmeyer flasks, respectively, proceed in the same manner as in the preparation of the test solution. Then add accurately 0.25 mL, 0.50 mL and 0.75 mL of standard chromium solution for Atomic absorption spectroscopy, respectively, add water to make 100.0 g each, and use these solutions as the standard solutions. The amount of the standard solution can be adjusted appropriately depending on the sensitivity of the apparatus. Perform the test using the test solution and the standard solutions as directed in the standard addition method under the Atomic absorption spectroscopy according to the following conditions (NMT 10 ppm).

Gas: Dissolved acetylene – Air
Lamp: A chromium hollow cathode lamp
Wavelength: 357.9 nm

Standard chromium solution for Atomic absorption spectroscopy—Weigh accurately 0.283 g of potassium dichromate, and add water to make exactly 1000 mL. Each mL of this solution contains 0.1 mg of chromium.

(4) ***Zinc***—Weigh accurately about 5.0 g of Purified Gelatin, transfer into an Erlenmeyer flask, and add 10 mL of hydrochloric acid. Stopper the flask, and heat on a water bath at 75 °C to 80 °C for 2 hours. If necessary, extend the heating time or heat at a higher temperature to allow Purified Gelatin to swell. After cooling the solution, add water to make exactly 100.0 g, and use this solution as the test solution. Separately, take 5.0 g each of Purified Gelatin, transfer into three Erlenmeyer flasks, respectively, and proceed in the same manner as in the preparation of the test solution. Then add accurately 7.5 mL, 15 mL and 22.5 mL of zinc standard solution for Atomic absorption spectroscopy, respectively, add water to make 100.0 g each, and use these solutions as the standard solutions. The amount of the standard solution can be adjusted appropriately depending on the sensitivity of the apparatus. Perform the test using the test solution and the standard solutions as directed in the standard addition method under the Atomic absorption spectroscopy according to the following conditions (NMT 30 ppm).

Gas: Dissolved acetylene – Air
Lamp: A zinc hollow cathode lamp
Wavelength: 213.9 nm

(5) ***Arsenic***—Put 15.0 g of Purified Gelatin in a flask, add 60 mL of diluted hydrochloric acid (1 in 5), and heat to dissolve. Add 15 mL of bromine TS, heat, evaporate excess bromine, add ammonia TS to make neutral, and add 1.5 g of dibasic sodium phosphate. After cooling, add 30 mL of magnesia TS, and allow to stand for 1 hour. Collect the precipitate by filtration, wash the residue 5 times with 10 mL of diluted ammonia TS (1 in 4) each time, and dissolve in diluted hydrochloric acid (1 in 4) to make exactly 50 mL. Perform the test with 5 mL of this solution; the color is not more intense than the following standard color.

Standard color—Proceed with 12 mL of arsenic standard solution, instead of Purified Gelatin, in the same manner (NMT 0.8 ppm).

(6) ***Peroxide***—Transfer 20.0 g of Purified Gelatin to a beaker, add 80.0 mL of water, stir until all of the gelatin is wet, and allow to stand at room temperature for 1 to 3 hours. Cover the beaker with a watch glass. If the gelatin is not completely dissolved, heat on a water bath at 63 °C to 67 °C for 15 to 25 minutes. Mix the content of the beaker well with a glass rod to obtain a homogeneous solution. Wet the reaction zone of the peroxide test paper appropriately for 1 second, shake off excess liquid, and compare the color indicator with the reaction zone after 15 seconds. Multiplying the concentration obtained from the color indicator by the correction factor of 5 to determine the peroxide concentration in the test substance (NMT 10 ppm).

Peroxide test paper—A strip which can measure peroxide from 0 ppm to 25 ppm and is good for the following suitability test.

Suitability test—Take exactly 10 mL of standard hydrogen peroxide solution and add water to make exactly 300 mL. Take exactly 2 mL of this solution, and add water to make exactly 1000 mL, and use this solution as the standard hydrogen peroxide solution. Wet the reaction area of the peroxide test paper appropriately with the standard hydrogen peroxide solution for 1 second, shake off the excess liquid, and compare the color indicator with the reaction area after 15 seconds. If the color matches, the test paper is appropriate.

Standard hydrogen peroxide stock solution—Take an appropriate amount of hydrogen peroxide (30), and add water to make a solution containing 0.30 g of hydrogen peroxide per 1 mL. Pipet 1 mL of this solution, and add water to make exactly 10 mL. Take exactly 1 mL of this solution and 10 mL of dilute sulfuric acid, place them in a flask containing 10 mL of water, and titrate with 0.02 mol/L potassium permanganate VS. However, the endpoint of titration is when the color of the liquid appears pale red. Perform a blank test in the same manner and make any necessary correction. Each mL of potassium permanganate TS = 1.701 mg of H_2O_2

Standard hydrogen peroxide solution—Take exactly 10 mL of standard stock solution of hydrogen peroxide, and add water to make exactly 100 mL. Prepare before use. Each mL of this solution contains 30 mg of hydrogen peroxide.

(7) ***Sulfur dioxide***—NMT 20 ppm

Loss on drying NMT 15.0% (5.0 g, 105 °C, 16 hours)

Microbial limit Perform the microbial limit test; the total aerobic microbial count is NMT 1000 CFU and the total number of fungi is NMT 100 CFU per 1 g of Purified Gelatin. Also, *Escherichia coli* and *Salmonella* are not detected.

Gel strength Perform the test with the gelling gelatin. Weigh 7.5 g of Purified Gelatin, put it in a bottle (jelly cup), and add 105 mL of water. Cover it with a stopper, and allow to stand for 1 to 4 hours. Heat on a water bath at 63 °C to 67 °C for 15 minutes while stirring with a glass rod. After cooling at room temperature for 15 minutes, transfer to a thermostat, and maintain 10.0 ± 0.1 °C. Close the stopper, and allow to stand for 17 ± 1 hours. After taking out the bottle, quickly wipe the outer wall of the bottle with water. Place the bottle in the center of the apparatus so that the plunger is centered on the sample, and measure with 4 mm depression depth and 0.5 mm/sec speed; the value is 80% to 120% of the labeled amount.

Apparatus—A physical property tester or gelometer with a cylinder piston with a diameter of 12.7 ± 0.1 mm. The internal diameter of the bottle (jelly cup) is 59 ± 1 mm and the height is 85 mm. Gell strength refers to the amount (g) required to depress a gel solidified at a concentration of 6.67% at 10 °C to a depth of

4 mm using a plunger with a diameter of 12.7 mm.

Conductivity Dissolve 1.0 g of Purified Gelatin in freshly boiled and cooled water at 55 ℃, and add freshly boiled and cooled water to make 100 mL. With this solution, perform the test under the Conductivity; the conductivity of Purified Gelatin at 30 ± 1.0 ℃ is NMT 1 $mS \cdot cm^{-1}$. Temperature correction is not performed.

Packaging and storage Preserve in tight containers.

Glycerin Esters of Fatty Acids
글리세린지방산에스테르

Glycerin Esters of Fatty Acids are esters and derivatives of fatty acid, glycerin, or polyglycerin, and there are glycerin esters of fatty acid, glycerin esters of acetic acid fatty acid, glycerin esters of lactic acid fatty acid, glycerin esters of citric acid fatty acid, glycerin esters of succinic acid fatty acid, glycerin esters of diacetyl tartaric acid fatty acid, glycerin esters of acetic acid, polyglycerin esters of fatty acid, and polyglycerine polyricinoleate esters.

Description Glycerin Esters of Fatty Acids occurs a colorless to brown powder, piece, coarse powder, granule, mass, semi-fluid, or liquid. It is odorless or has a characteristic odor.

Identification (1) To 5 g (1.5 g for glycerin esters of acetic acid) of Glycerin Esters of Fatty Acids, add 50 mL of 0.5 mol/L potassi-um hydroxide-ethanol TS, heat on a water bath for 1 hour under a reflux condenser, and evaporate alcohol until the residue becomes semi-solid. Then, add 50 mL of hydrochloric acid (1 in 9), shake well to mix, extract the produced fatty acid 3 times with 40 mL each of a mixture of petroleum ether and methyl ethyl ketone (7 : 1), and combine all the extracts. Use this as the petroleum ether-methyl ethyl ketone layer. Shake the water layer well to mix, then add sodium hydroxide solution (1 in 9) to make a neutral solution, and concentrate on a water bath in vacuum. Then, add 20 mL of 40 ℃ methanol, shake well to mix, then cool, and filter. Re-move the methanol in the filtrate on a water bath. Use a solution of residue in methanol (1 in 10) as the test solution. Separately, use a solution of glycerin in methanol (1 in 10) as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Dry the thin-layer chromatographic plate made of silica gel for thin layer chromatography at 110 ℃ for 1 hour, and then spot 5 μL each of the test solution and the standard solution on the plate. Develop the plate with a mixture of 1-butanol, methanol, and chloroform (5 : 3 : 2) as the developing solvent to a distance of about 15 cm, air-dry the plate, then dry by heating at 110 ℃ for 10 minutes, and cool. Then, spray evenly a thymol-sulfuric acid TS, and heat at 110 ℃ for 20 minutes to exhibit a color. Glycerin esters exhibit a white spot at the same position as the standard solution, and polyglycerine esters exhibit a white spot or band-shaped white spot below the same position as the standard solution.

Thymol-sulfuric acid TS—Dissolve 0.5 g of thymol in 5 mL of sulfuric acid to make 100 mL.

(2) ***Other than glycerin esters of acetic acid***—Evaporate the solvent in the petroleum ether-methyl ethyl ketone layer obtained from (1); a yellowish white oil or solid remains. To 0.1 g of this residue, add 5 mL of ether, and shake to mix; it dissolves.

(3) ***Other than glycerin esters of fatty acids and polyglycerine esters***—To 5 mL of the test solution from (1), add 50 mL of water, and shake to mix. With this solution, glycerin esters of acetic acid fatty acids and glycerin esters of acetic acid respond to the Chemical identification reactions for acetate, glycerin esters of lactic acid fatty acid respond to the Chemical identification reactions for lactate, glycerin esters of citric acid fatty acids respond to the Chemical identification reactions (2) for citrate, glycerin esters of succinic acid fatty acids respond to the Chemical identification reactions for succinate, and glycerin esters of diacetyl tartaric acid fatty acids respond to the Chemical identification reactions for acetate and tartrate.

Chemical identification reactions for succinate—Adjust the pH of the succinate solution to 6 to 7, and add 1 mL of iron(III) chloride TS to 5 mL of this solution; a pale yellow to red precipitate forms.

(4) ***Polyglycerine polyricinoleate esters***—Wash the petroleum ether-methyl ethyl ketone layer 2 times each with 50 mL of water, and filter with anhydrous sodium sulfate to dehydrate. Evaporate the solvent by heating in vacuum, weigh accurately about 1 g of the obtained residue, transfer into a 200-mL round-bottom flask, and perform the test as directed under the Hydroxyl value of Fats and Fatty Oils; the value is 150 to 170.

Purity (1) ***Acid value***—Weigh accurately 6 g (0.5 g of the residue from Identification (4) for polyglycerine polyricinoleate esters) of Glycerin Esters of Fatty Acids, dissolve in 120 mL of a mixture of ether and ethanol (1 : 1), use this solution as the test solution, and perform the test as directed under the Acid value of Fats and Fatty Oils; the value is NMT 6.0 for glycerin esters of fatty acids, glycerin esters of acetic acid fatty acids, glycerin esters of lactic acid fatty acids, and glycerin esters of acetic acid, NMT 12 for polyglycerine ethers of fatty acids and polyglycerine polyricinoleate esters, NMT 100 for glycerin esters of citric acid fatty acids, 60 to 120 for glycerin esters of succinic acid fatty acids and glycerin esters of diacetyl tartaric acid fatty acids.

(2) ***Arsenic***—Prepare the test solution with 0.5 g of Glycerin Esters of Fatty Acids according to Method 3 and perform the test (NMT 4 ppm).

(3) ***Lead***—Transfer 5.0 g of Glycerin Esters of Fatty Acids into a crucible, gently heat to carbonize, and then ignite at 450 ℃ to 550 ℃. After cooling, moisten the residue with a small amount of water, add 4 mL of hydrochloric acid to evaporate to dryness, then add 0.5 mol/L nitric acid, and warm to dissolve. Filter through a filter paper if there are insolubles, then add 0.5 mol/L nitric acid to make exactly 25 mL, and use this solution as the test solution. Separately, transfer 1 mL of lead standard solution into a crucible proceed in the same manner as in the preparation of the test solution, and use this solution as the standard solution. Perform the test with the blank test solution, the test solution, and the standard solution as directed under the Atomic Absorption Spectroscopy according to the following conditions, using 0.5 mol/L nitric acid as the blank test solution; the absorbance of the test solution is NMT the absorbance of the standard solution (NMT 2.0 ppm).

Used gas: Dissolved acetylene – Air
Lamp: A lead hollow cathode lamp
Wavelength: 283.3 nm

(4) ***Cadmium***—Use the test solution from (3) as the test solution. Separately, transfer 5.0 mL of standard cadmium solution into a crucible proceed in the same manner as in the

preparation of the test solution, and use this solution as the standard solution. Perform the test with the blank test solution, the test solution, and the standard solution as directed under the Atomic Absorption Spectroscopy according to the following conditions, using 0.5 mol/L nitric acid as the blank test solution; the absorbance of the test solution is NMT the absorbance of the standard solution (NMT 1.0 ppm).

Gas: Dissolved acetylene – Air
Lamp: A cadmium hollow cathode lamp
Wavelength: 228.8 nm

(5) ***Mercury***—It is determined by either of the following methods.

(i) Weigh accurately 5 g to 10 g of the sample, transfer into a decomposition flask, add 10 mL of water and 20 mL of nitric acid, and slowly shake. Then, slowly add 20 mL of sulfuric acid. Heat the decomposition flask under a reflux condenser until the brown smoke does not form. When the decomposed solution does not become transparent, cool the solution, then add 5 mL of the nitric acid, and repeat the above procedure. After cooling, add 50 mL of water and 10 mL of 10% iodine solution, boil for 10 minutes, and cool. Then, add 1 g of potassium permanganate, and allow to stand for about 10 minutes while shaking occasionally. Repeat the procedure until a purplish red color remains, boil for 20 minutes until a purplish red disappears, cool, and then add 1 g of potassium permanganate. Then, heat for 20 minutes. When the purplish red color of the solution disappears, repeat the procedure of adding potassium permanganate and heating 2 times, cool, and carefully add 20% hydroxylammonium chloride solution until the solution becomes colorless and clear. After cooling, transfer the decomposed solution into another decomposition flask. Wash a reflux condenser and the interior and joint of the decomposition flask, combine the washing and add water to make a certain amount of solution, and use this solution as the test solution. Take 100 mL each of the test solution and the blank test solution, previously adjusted to have a concentration of 20% (v/v) sulfuric acid, transfer into a test bottle, connect to the reducing vaporizer, and then add 10 mL of tin(II) chloride solution. Then, immediately close the stopper, use the diaphragm pump to circulate the air within the absorption cell, and determine the absorbance at the wavelength of 253.7 nm. Separately, pipet 1, 5, 10, 15, and 20 mL of the mercury standard solution, add water to make 100 mL, respectively, then proceed in the same manner as in the preparation of the test solution, and determine the absorbance according to the conditions below to prepare a calibration curve. Find the amount of mercury by plugging the absorbance of the test solution to the calibration curve (NMT 1.0 ppm).

Atomic absorption spectrometer: With a quartz absorption cell attached.
Lamp: A mercury hollow cathode lamp
Mercury reducing vaporizer

(ii) Evenly distribute about 1 g of aluminum oxide, previously activated in a ceramic boat at 950 ℃ for 30 minutes. When the sample is solid, finely cut to make it homogeneous, and then weigh accurately 10 mg to 300 mg, and evenly distribute on the aluminum oxide. With liquid sample, pipet 0.1 mL to 0.5 mL, and completely let it permeate into aluminum oxide. Then, on top of it, evenly distribute about 0.5 g of aluminum oxide, previously activated at 950 ℃ for 30 minutes, and 1 g of a mixture of calcium hydroxide and sodium carbonate (1 : 1), previously activated at 950 ℃ for 30 minutes, in this order to form layers. However, with an automated mercury analyzer with a separate catalyst in the combustion unit, do not add additives to the nickel boat, and take only the sample. Place the boat inside the combustion chamber, heat at about 900 ℃ while passing air or oxygen at a rate of 0.5 L/min to 1 L/min to efflux mercury, then collect in the collecting tube. Heat the collecting tube at about 700 ℃, send the mercury vapor to the cold atomic absorption spectrophotometer, and determine the absorbance, A. Separately, add only additive in a ceramic boat, and determine the absorbance, A_b, in the same manner. Separately, use mercury standard solution to prepare the calibration curve from the absorbance obtained by proceeding in the same manner. A - Find the amount of mercury in the sample by plugging A_b to the calibration curve (NMT 1.0 ppm).

Operating conditions

Use an automated mercury measuring instrument for sample combustion, trapping with gold amalgam, and measurement using cold vapor atomic absorption spectrophotometry. However, a mercury measuring instrument with a separate catalyst in the combustion unit can be used.

(6) ***Polyoxyethylene***—Transfer about 1 g of Glycerin Esters of Fatty Acids into a 200-mL flask, add 25 mL of 0.5 mol/L potassium hydroxide-ethanol TS, and allow to stand on a water bath under a reflux condenser for 1 hour while occasionally shaking. Evaporate alcohol to dryness on a water bath or in vacuum, add 20 mL of sulfuric acid solution (3 in 100), shake well to dissolve by warming, and then add 15 mL of ammonium thiocyanate-cobalt nitrate TS. Shake well to mix, then add 10 mL of chloroform, shake well to mix, and then allow to stand; the chloroform layer does not exhibit a blue color.

Residue on ignition Weigh accurately about 1 g of Glycerin Esters of Fatty Acids, and ignite at 800 ± 25 ℃; the amount is NMT 0.5%.

Glyceryl Monostearate
글리세린모노스테아레이트

Glycerol monostearate

Glyceryl Monostearate is a mixture of α- and β- glyceryl monostearate and other fatty acid esters of glycerin.

Description Glyceryl Monostearate occurs as a white to pale yellow waxy mass, thin flakes, or grain. It has a slightly characteristic odor and taste.
It is very soluble in warm ethanol, soluble in chloroform, sparingly soluble in ether, and practically insoluble in water or ethanol.
It is gradually affected by light.

Identification (1) To 0.2 g of Glyceryl Monostearate, add 0.5 g of potassium hydrogen sulfate, and heat until almost carbonized; the irritative odor of acrolein is perceptible.

(2) Dissolve 0.1 g of Glyceryl Monostearate in 2 mL of ethanol by warming, heat with 5 mL of dilute sulfuric acid on a water bath for 30 minutes and cool: a white to yellow solid is produced. Separate this solid, add 3 mL of ether, and shake to mix; it dissolves.

Saponification value Between 157 and 170.

Acid Value NMT 15.

Iodine value NMT 3.0. However, use chloroform instead of cyclohexane.

Melting point NLT 55 ℃ (Method 2).

Purity (1) ***Acidity or alkalinity***—Weigh 1.0 g of Glyceryl Monostearate, add 20 mL of hot water, and cool by shaking to mix; the resulting solution is neutral.

(2) ***Heavy metals***—Proceed with 2.0 g of Glyceryl Monostearate according to Method 2 under the Heavy metals, and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 10 ppm).

Residue on ignition NMT 0.1% (1 g).

Packaging and storage Preserve in light-resistant, tight containers.

Glycine
글리신

H_2NCH_2COOH

$C_2H_5NO_2$: 75.07

Aminoacetic acid [*56-40-6*]

Glycine, when dried, contains NLT 98.5% and NMT 101.0% of glycine ($C_2H_5NO_2$).

Description Glycine occurs as a white crystal or crystalline powder. It is odorless and has a sweet taste.
It is freely soluble in water or formic acid and practically insoluble in ethanol.

Identification Determine the infrared spectra of Glycine and glycine RS, previously dried, as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers. If any difference appears between the two spectra, dissolve Glycine in water, evaporate the water to dryness, and repeat the test with the residue.

pH Dissolve 1.0 g of Glycine in 20 mL of water; the pH of this solution is 5.6 to 6.6.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Glycine in 20 mL of water; the resulting solution is clear and colorless.

(2) ***Chloride***—Perform the test with 0.5 g of Glycine. Prepare the control solution with 0.30 mL of 0.01 mol/L hydrochloric acid (NMT 0.021%).

(3) ***Sulfate***—Perform the test with 0.6 g of Glycine. Prepare the control solution with 0.35 mL of 0.005 mol/L (NMT 0.028%).

(4) ***Ammonium***—Perform the test with 0.25 g of Glycine. Prepare the control solution with 5.0 mL of ammonium standard solution (NMT 0.02%).

(5) ***Heavy metals***—Proceed with 1.0 g of Glycine according to Method 1 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 20 ppm).

(6) ***Mercury***—Spread evenly about 1 g of excipient (a) in a ceramic boat, and add 10 mg to 300 mg of Glycine on top. On top of it, evenly distribute about 0.5 g of excipient (a) and 1 g of excipient (b) in this order to form layers. In the case of an automated mercury analyzer equipped with a separate catalyst in the combustion chamber, take only the sample without adding excipients to a nickel boat. Place the boat inside the combustion furnace and heat to about 900 ℃ with a current of air or oxygen at 0.5 to 1 L/minute. Elute the mercury and collect in a collecting tube. Transfer the mercury vapor to a cold vapor atomic absorption spectrometer by heating the collecting tube to about 700 ℃ and determine the absorbance as A. Separately, determine the absorbance in the same manner using only the excipients in a ceramic boat and designate as Ab. Separately, proceed with mercury standard solution in the same manner and create a calibration curve from the absorbances. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the sample (NMT 1.0 ppm)

Operating conditions

Apparatus: Use an automated mercury analyzer for specimen combustion, trapping with gold amalgam and measurement using a cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Standard mercury stock solution—Dissolve 0.135 g of mercury (II) chloride in 0.001% L-cysteine solution to make 1000 mL. Each mL of this solution contains 100 μg of mercury (II) chloride.

Mercury standard solution—Dilute standard mercury stock solution with 0.001% L-cysteine solution to make 0 ng/mL to 200 ng/mL.

Excipients—Use (a) aluminum oxide and (b) a mixture of calcium hydroxide and sodium carbonate (1 : 1), and activate at 950 ℃ for 30 minutes before use.

(7) ***Lead***—Weigh accurately 5.0 g of Glycine, transfer into a platinum crucible, dry and carbonize, and then incinerate at 450 to 550 ℃. If incineration is not complete, cool and moisten with 2 to 5 mL of nitric acid (1 in 2), 50% magnesium nitrate solution or aluminum nitrate-calcium nitrate solution (prepared by dissolving 40 g of aluminum nitrate and 20 g of calcium nitrate in 100 mL of water) as an incineration supplement, dry, and continue with incineration.

If incineration is incomplete, repeat the above procedure once and then add a final 2 to 5 mL of nitric acid (1 in 2), if necessary, and incinerate. After incineration, moisten the residue with water, add 2 to 4 mL of hydrochloric acid, and evaporate to dryness. Add 0.5 mol/L nitric acid, dissolve by warming, and filter with filter paper if any insoluble matter exists. Unless otherwise specified, add 0.5 mol/L nitric acid to make 25 mL and use this solution as the test solution. Separately, transfer 2.5 mL of lead standard solution into a platinum crucible, proceed in the same manner as in the preparation of the test solution, and use this solution as the standard solution. Perform the test with the test solution and the standard solutions as directed under Atomic Absorption Spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 5.0 ppm).

Gas: Acetylene or Hydrogen - Air
Lamp: A lead hollow-cathode lamp
Wavelength: 283.3 nm

(8) ***Arsenic***—Prepare the test solution with 1.0 g of Glycine according to Method 1, and perform the test (NMT 2 ppm).

(9) ***Related substances***—Dissolve 0.10 g of Glycine in 25 mL of water, and use this solution as the test solution. Pipet 1 mL of this solution, and add water to make exactly 50 mL. Pipet 5 mL of this solution, add water to make exactly 20 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 5 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with a mixture of *n*-butanol, water, and acetic acid (100) (3 : 1 : 1) as the developing solvent to a distance of about 10 cm, and dry the plate at 80 ℃ for 30 minutes. Spray a solution of ninhydrin in acetone (1 in 50) evenly on the plate, and heat at 80 ℃ for 5 minutes; the spots other than the principal spot from the test solution are not more intense than the spots from the standard solution.

Loss on drying NMT 0.3% (1 g, 105 ℃, 3 hours).

Residue on ignition NMT 0.1% (1 g).

Assay Weigh accurately about 0.15 g of Glycine, previously dried, and dissolve in 3 mL of formic acid, add 50 mL of acetic acid (100), and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration under the Titrimetry). Perform a blank test in the same manner and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 7.507 mg of $C_2H_5NO_2$

Packaging and storage Preserve in well-closed containers.

Honey
꿀(蜂蜜)

Mel

Honey is obtained from the saccharine substances collected in the honeycomb by honeybees, *Apis mellifera* Linné or *Apis indica* Radoszkowski (Apidae).

Description Honey occurs as a pale yellow to pale yellowish brown syrup-like liquid. It is mostly transparent but can sometimes become opaque due to the formation of crystals.
It has a characteristic odor and sweet taste.

Specific gravity The specific gravity, d_{20}^{20}, of a solution of 50.0 g of Honey mixed in 100 mL of water is NLT 1.111.

Purity (1) ***Acid***—Dissolve 10 g of Honey in 50 mL of water and neutralize with 0.1 mol/L sodium hydroxide TS (indicator: 2 drops of phenolphthalein TS); the consumed amount is NMT 0.5 mL.

(2) ***Chloride*—**Perform the test with 1.0 g of Honey. Prepare the control solution with 0.5 mL of 0.02 mol/L hydrochloric acid (NMT 0.035%).

(3) ***Sulfate*—**Perform the test with 1.0 g of Honey. Prepare the control solution with 0.5 mL of 0.005 mol/L sulfuric acid (NMT 0.024%).

(4) ***Ammonia-coloring substances*—**Weigh 1 g of Honey, add 2.0 mL of water, mix, filter, and then add 2 mL of ammonia TS to the filtrate; the resulting solution does not immediately change.

(5) ***Resorcin-coloring substances***—Weigh 5 g of Honey, add 15 mL of ether, shake to mix, and then filter. Evaporate the filtered ether solution at ordinary temperature, and add 1 to 2 drops of resorcin TS into the residue; the residue and the solution may exhibit a yellowish red color, but does exhibit a red to purple color lasting for more than 1 hour.

(6) ***Starch and dextrin***—(i) To 7.5 g of Honey, add 15 mL of water, shake to mix, then warm on a water bath, and then add 0.5 mL of tannic acid TS. After cooling, add 1.0 mL of anhydrous ethanol containing 2 drops of hydrochloric acid into 1.0 mL of the filtrate; the resulting solution is not turbid.

(ii) Weigh 2 g of Honey and add 10 mL of water, warm on a water bath, then mix, and cool. To 1.0 mL of this solution, add 1 drop of iodine TS, and shake to mix; the resulting solution does not exhibit a blue, green, or reddish brown color.

(7) ***Foreign matter***—Weigh 1 g of Honey, add 2.0 mL of water to mix, and examine the precipitate obtained by centrifuging microscopically; there is no foreign matter observable other than pollen.

(8) ***5-Hydroxymethylfururals***—Weigh accurately about 5 g of sample, dissolve in 25 mL of water, and transfer into a 50-mL volumetric flask. Add 0.5 mL of 15% potassium ferrocyanide solution and 0.5 mL of 30% zinc acetate solution, mix, add water to the gauge line (if foams develop, add one drop of ethanol), and filter. Discard the first 10 mL of filtrate and use the remaining filtrate as the test solution. Transfer 5 mL of the test solution into two test tubes, respectively, and use one as the test solution and the other one as the blank test solution. To the test solution tube, add 5 mL of water, and to the blank test solution tube, add 5 mL of 0.2% sodium hydrosulfite solution. Mix well, then determine the absorbance at 284 nm and 336 nm using water as the blank for the test solution and 0.1% sodium bisulfite solution for the blank test solution (NMT 80 ppm).

$$\text{Amount (ppm) of hydroxymethylfurfural} = \frac{(A_{234}-A_{335})\times 149.7\times 5}{S}$$

A_{284} and A_{336}: Absorbance at 284 nm and 336 nm (test solution - blank test solution)

S: Amount (g) of sample taken

Ash NMT 0.4%.

Microbial limit Perform the microbial limit test; the total aerobic microbial count is NMT 1000 CFU, and the total combined yeasts/mould count is NMT 100 CFU per 1 g of Honey. Also, *Escherichia coli, Salmonella, Pseudomonas aeruginosa,* and *Staphylococcus aureus* are not detected.

Packaging and storage Preserve in tight containers.

Hydrogenated Oil
경화유

Hydrogenated Oil is a fat obtained by adding hydrogen to fish oil or other animal or vegetable oils.

Description Hydrogenated Oil occurs as a white mass or powder. It has a characteristic odor and a mild taste.
It is freely soluble in ether, very slightly soluble in ethanol and practically insoluble in water. However, the oil obtained by adding hydrogenation of castor oil is slightly soluble in ether, very

slightly soluble in ethanol and practically insoluble in water.

Acid Value NMT 2.0.

Purity (1) ***Water content and coloration*—**Perform the test as directed in Purity (1) under Beef Tallow.

(2) ***Alkali*—**Perform the test as directed in the Purity (2) under Beef Tallow.

(3) ***Chloride*—**Perform the test as directed in the Purity (3) under Beef Tallow.

(4) ***Heavy metals***—Add 5 mL of dilute hydrochloric acid and 10 ml of water to 2.0 g of Hydrogenated Oil, heat on a water bath for 5 minutes with occasional shaking, filter after cooling, and make slightly alkaline by adding 5 ml of ammonia TS to the filtrate, and add 3 drops of sodium sulfide TS; the solution does not change.

(5) ***Nickel*—**Weigh 5.0 g of Hydrogenated Oil in a quartz or ceramic crucible, carefully and gently heat to carbonize, and then ignite to incinerate (500 ± 20 ℃). After cooling, add 1 mL of hydrochloric acid, evaporate on a water bath to dryness, dissolve the residue in 3 mL of dilute hydrochloric acid, and add 7 mL of water. Add 1 mL of bromine TS and 1 mL of citric acid solution (1 in 5), make alkaline with 5 mL of ammonia TS, and cool in running water. To this solution, add 1 mL of dimethylglyoxime TS, then add water to make 20 mL, and use this solution as the test solution. Allow the test solution to stand for 5 minutes; the color of the resulting solution is not more intense than that of the following control solution.

Control solution—Evaporate 1 mL of hydrochloric acid to dryness on a water bath, add 1 mL of nickel standard solution and 3 mL of dilute hydrochloric acid, and then add 6 mL of water. Proceed in the same manner as in the preparation of the test solution, add water to make 20 mL, and allow to stand for 5 minutes.

(6) ***Peroxide value*—**Weigh accurately 5 g of Hydrogenated Oil, transfer into a stoppered 250-mL Erlenmeyer flask, and dissolve in 30 mL of a mixture of acetic acid (100) and chloroform (3 : 2). To this solution, add 0.5 mL of a saturated solution of potassium iodide, shake for exactly 1 minute to mix, and add 30 mL of water. Titrate this solution with 0.01 mol/L sodium thiosulfate VS. The endpoint of titration is when the blue color of the solution disappears after the addition of 5 mL of starch TS at a point when the solution turns to a pale yellow color. Perform the blank test in the same manner (consumed amount of 0.01 mol/L sodium thiosulfate VS in the blank test solution should be NMT 0.1 mL), and calculate the peroxide value according to the following equation; the value is NMT 3.

$$\text{Peroxide value (mEq/kg)} = [10 \times (V_1 - V_0)] / W$$

V_1: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed in the titration of the test solution.

V_0: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed in the titration of the blank test solution.

W: Amount (g) of Hydrogenated Oil taken

Residue on ignition NMT 0.1% (5 g).

Packaging and storage Preserve in well-closed containers.

Hydroxyethylcellulose
히드록시에틸셀룰로오스

Cellulose, 2-hydroxyethyl ether [*9004-62-0*]

Hydroxyethylcellulose is partly O-(2-hydroxyethylated) cellulose.

Hydroxyethylcellulose contains NLT 30.0% and NMT 70.0% of hydroxyethoxy group ($-OC_2H_4OH$: 61.06), calculated on the dried basis.

An appropriate pH adjusting agent such as phosphate may be added to Hydroxyethylcellulose.

Description Hydroxyethylcellulose occurs as a white to yellowish white powder or grains.
It is practically insoluble in ethanol (95).
It forms a viscous liquid when water is added.
It is hygroscopic.

Identification (1) Determine the infrared spectra of Hydroxyethylcellulose and hydroxyethylcellulose RS as directed in the ATR method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

(2) Dissolve 1.0 g of Hydroxyethylcellulose, calculated on the dried basis, in 50 mL of freshly boiled and cooled water. After 10 minutes, add freshly boiled and cooled water to make 100 mL, dissolve completely with thorough stirring, and use this solution as the test solution. Heat 10 mL of this solution to boiling; the liquid remains clear.

Viscosity Weigh accurately an amount of Hydroxyethylcellulose equivalent to 10.00 g calculated on the dried basis, add 400 mL of water, stir to dissolve, and add water to make exactly 500.0 g. Remove air bubbles and use this solution as the test solution. Perform the test at 20 ± 0.1 ℃ using a beaker with an internal diameter of NLT 70 mm, according to Method 2 using a single cylinder type rotational viscometer according to the following conditions; the viscosity is between 75% and 140% of the labeled viscosity.

Operating conditions

Operation of the apparatus: Start the apparatus and rotate it for 2 minutes. Then, read the viscometer and stop for 2 minutes. Repeat the same operation twice and average the three readings.

pH The pH of the test solution from Identification (2) is 5.5 to 8.5.

Purity (1) ***Chloride***—To 1 mL of the test solution from Identification (2), add water to make 30 mL, and use this solution as the test solution. Separately, pipet 10 mL of the standard chloride solution, add 5 mL of water, and use this solution as the control solution. Add 1 mL of dilute nitric acid (1 in 5) to 15 mL of the test solution and the control solution, then place each in a test tube containing 1 mL of silver nitrate solution (17 in 1000), and allow it to stand for 5 minutes, protected from light. Compare the turbidity by observing from the side of the test tube against a black background; the turbidity of the test solution is not more intense than that of the control solution (NMT 1.0%).

(2) ***Nitrate***—Prepare the solutions before use. Dissolve 0.50 g of Hydroxyethylcellulose in the solvent to make exactly 100 mL, and use this solution as the test solution. Separately, dissolve 0.8154 g of potassium nitrate, dissolve in the solvent to make 1,000 mL, and use this solution as the standard potassium

nitrate stock solution. If the viscosity of Hydroxyethylcellulose is NMT 1,000 mPa·s, pipet 10 mL, 20 mL, and 40 mL of the standard nitrate stock solution and add the solvent to make exactly 100 mL, respectively. Use these solutions as the standard solutions. If the viscosity of Hydroxyethylcellulose is more than 1,000 mPa·s, pipet 1 mL, 2 mL, and 4 mL of the standard nitrate stock solution and add the solvent to make exactly 100 mL, respectively. Use these solutions as the standard solutions. For the test solution and the standard solutions, perform the test using a nitrate ion-selective electrode as an indicator electrode, a silver/silver chloride electrode as a reference electrode, and dilute ammonium sulfate TS (1 in 30) as a reference electrolyte. Determine the concentration of nitrate in the test solution using the calibration curve obtained from the potential difference of the standard solutions; the amount of nitrate is NMT 3.0% (calculated on the dried basis) when the viscosity of Hydroxyethylcellulose is NMT 1,000 mPa·s, and NMT 0.2% (calculated on the dried basis) when the viscosity is more than 1,000 mPa·s.

Solvent: Add 135 g of potassium dihydrogen phosphate to a mixture of 50 mL of 1 mol/L sulfuric acid TS and 800 mL of water, and add water to make 1000 mL. To the resulting solution, add water to make an exact 25-fold diluted solution.

To determine the viscosity, use the following method: Add 50 g of water to the amount equivalent to 2.00 g of Hydroxyethylcellulose calculated on the dried basis and shake gently. Add water again to make 100 g and dissolve completely with thorough stirring. Determine the viscosity at 25°C using a rotational viscometer. For those with a viscosity of less than 100 mPa·s, set the shear rate to 100 s^{-1}. For those with a viscosity of NLT 100 mPa·s and NMT 20,000 mPa·s, set the shear rate to 10 s^{-1}. For those with a viscosity greater than 20,000 mPa·s, set the shear rate to NLT 1 s^{-1}. If the shear rate cannot be set accurately to 10 s^{-1} or 100 s^{-1}, set it to a slightly higher or lower rate and correct by interpolation.

(3) ***Aldehyde***—Place 1.0 g of Hydroxyethylcellulose in a glass-stopped centrifuge tube, add 10 mL of ethanol (99.5), stopper the tube, stir for 30 minutes, centrifuge, and use the clear supernatant as the test solution. Use the standard glyoxal solution as the control solution. Pipet 2 mL each of the test solution and the control solution and add 5 mL of a solution prepared by dissolving 4 g of 3-methyl-2-benzothiazolone hydrazone hydrochloride monohydrate in dilute acetic acid (100) (4 in 5) to make 1000 mL. Shake until homogeneous, allow the mixture to stand for 2 hours, and compare the color of the resulting solutions; the color of the test solution is not more intense than the control solution (NMT 20 ppm).

Glyoxal TS—$C_2H_2O_2$ 58.04 [107-22-2] Content: 38 to 42%. Assay: Weigh 1.000 g of Hydroxyethylcellulose, place it in a stoppered flask and add 20 mL of hydroxylamine hydrochloride solution (7 in 100) and 50 mL of water. Stopper the flask, allow it to stand for 30 minutes, and then titrate with 1 mol/L sodium hydroxide VS (indicator: 1.0 mL of methyl red-methylene blue TS). Perform a blank test in the same manner and make any necessary correction.

Each mL of 1 mol/L sodium hydroxide VS
= 29.02 mg of $C_2H_2O_2$

Standard glyoxal stock solution—Take an amount of the glyoxal TS equivalent to 0.200 g as glyoxal and add ethanol (99.5) to make 100 mL. Pipet 1 mL of this solution and add ethanol (99.5) to make 100 mL before use. This solution contains 20 μg of glyoxal ($C_2H_2O_2$) per mL.

Standard glyoxal solution—Pipet 1 mL of the standard glyoxal stock solution and add ethanol (99.5) to make 10 mL. Prepare before use. This solution contains 2 μg of glyoxal ($C_2H_2O_2$) per mL.

Loss on drying NMT 10.0% (1 g, 105 ℃, 3 hours).

Residue on ignition Measure the viscosity according to (2) under the Purity, the residue on ignition is NMT 4.0% when the viscosity is NMT 1000 mPa·s and NMT 1.0% when the viscosity is more than 1000 mPa·s (1 g).

Assay Weigh accurately about 30 mg of Hydroxyethylcellulose and place into a 5-mL pressure-resistant serum vial. Add exactly 60 mg of adipic acid, 2 mL of the internal standard solution, and 1 mL of hydroiodic acid, then immediately fix the vial with a septum stopper coated with fluorine resin and an aluminum cap, or use a stopper with equivalent air-tightness, and weigh accurately the mass of the vial. Be careful not to mix the contents of the vial before heating. Heat the block until the internal temperature of the vial is 165 ± 2 ℃, and stir for 2.5 hours using a magnetic stirrer or shaker provided with the heater. After cooling, weigh accurately the mass of the vial. If the difference in mass before and after heating exceeds 10 mg, this solution should not be used in the test. If the difference in mass before and after heating is NMT 10 mg, the phase is separated. Then, using a cooled syringe, draw a sufficient amount of the upper layer through the septum stopper of the vial and use it as the test solution. Separately, pipet 60 mg of adipic acid, 2 mL of the internal standard solution, and 1 mL of hydroiodic acid into a pressure-resistant serum vial, seal it immediately, and weigh accurately the mass of the vial. Then, using a syringe, inject 55 μL of iodoethane RS through the septum stopper, and weigh accurately the mass of the vial. After shaking well to separate the phases, use a cooled syringe to draw a sufficient amount of the upper layer through the septum stopper of the vial, and use it as the standard solution. Perform the test with 1 μL each of the test solution and standard solution as directed under the Gas Chromatography according to the following conditions, and determine the ratios, Q_T and Q_S, of the peak area of the iodoethane to that of the internal standard.

Amount (%) of hydroxyethoxy group ($C_2H_5O_2$)
$= (W_S / W_T) \times (Q_T / Q_S) \times 39.15$

W_S: Amount (mg) of iodoethane taken for assay
W_T: Amount(mg) of Hydroxyethylcellulose taken, calculated on the dried basis

Internal standard solution—A solution of n-octane in o-xylene (1 in 200).

Operating conditions

Detector: A flame ionization detector

Column: A fused silica capillary column about 0.53 mm in internal diameter and about 30 m in length, coated inside with poly(dimethyl)siloxane for gas chromatography 3 μm in thickness.

Column temperature: Maintain the temperature at 50 ℃ for the first 3 minutes, then raise the temperature at the rate of 10 ℃ per minute up to 100 ℃, then raise at the rate of 35 ℃ per minute up to 250 ℃, and maintain at 250 ℃ for 8 minutes.

Sample injection port temperature: A constant temperature

of about 250 ℃.

Detector temperature: A constant temperature of about 280 ℃.

Carrier gas: Helium

Flow rate: 4.2 mL/min

Split ratio: About 1 : 40

System suitability

System performance: Proceed with 1 μL of the standard solution according to the above conditions; iodoethane and the internal standard are eluted in this order, and the relative retention time of iodoethane to the internal standard is about 0.6 with the resolution between these peaks being NLT 5.0.

System repeatability: Repeat the test 6 times according to the above conditions with 1 μL each of the standard solution; the relative standard deviation of the peak area ratio of iodoethane to that of the internal standard is NMT 2.0%.

Hydroiodic acid TS—HI 127.9 [10034-85-2] Distill hydroiodic acid and red phosphorus to obtain an azeotrope. During distillation, pass carbon dioxide or nitrogen through the apparatus. Use a colorless or almost colorless azeotrope (55% to 58%) with a boiling point of 126 to 127 ℃. Storage: Place in a small brown glass-stoppered bottle, previously washed with carbon dioxide or nitrogen, and seal with paraffin. Store in a dark place.

Packaging and storage Preserve in well-closed containers.

Hydroxypropylcellulose
히드록시프로필셀룰로오스

Cellulose, 2-hydroxypropyl ether
[9004-64-2]

Hydroxypropylcellulose is partly O-2-(hydroxypropylated) cellulose. Hydroxypropylcellulose, contains NLT 53.4% and NMT 80.5% of hydroxypropoxyl group (-OC_3H_6OH: 75.09), calculated on the dried basis.

Hydroxypropylcellulose may contain anti-caking agents such as silica. Anti-caking agents such as silica should be stated on the label. Label it to indicate the average viscosity measured in an aqueous solution of the stated concentration and temperature in the range of 50% to 150% of the average value.

Description Hydroxypropylcellulose occurs as a white to yellowish white powder or grain and is almost tasteless and odorless.
It is soluble in cold water, ethanol (95), chloroform or propylene glycol; the solution becomes a viscous liquid.
It is insoluble in hot water.
It is hygroscopic after drying.

Identification (1) Dissolve 1 g of Hydroxypropylcellulose in 100 mL of water. Transfer 1mL of this solution to a glass plate, and evaporate; a thin film is formed.

(2) Determine the infrared spectra of Hydroxypropylcellulose and hydroxypropylcellulose RS as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers. However, disregard any absorption at wavenumbers of about 1719 cm^{-1}.

pH Evenly distribute 1.0 g of Hydroxypropylcellulose in 100 mL of freshly boiled water, stir with a magnetic stirrer and cool; the pH of the solution is 5.0 to 8.0.

Viscosity Perform the test with Hydroxypropylcellulose according to Method 2 under the Viscosity at the stated values of temperature and concentration.

Purity Silica Perform the test if the addition of silica is stated on the label and the residue on ignition is NLT 0.2%. Weigh accurately the weight of the crucible containing the residue obtained from the Residue on ignition and set this value as a (g). Moisten the residue with water and add 5 mL of hydrofluoric acid. Evaporate the mixture to dryness on a steam bath, add 5 mL of hydrofluoric acid and 0.5 mL of sulfuric acid, and evaporate to dryness again. Slowly increase the temperature until all of the acids have been volatilized, and ignite at 1000 ± 25 ℃. Cool in a desiccator, and measure the weight and set this value as b (g); the amount (%) of silica calculated using the following equation is NMT 0.6%.

$$\text{Amount (\%) of silica } (SiO_2) = (a - b) / W \times 100$$

W: Amount (g) of Hydroxypropylcellulose taken for use in the Residue on ignition

Loss on drying NMT 5.0% (1 g, 105℃, 4 hours)

Residue on ignition NMT 0.8% (1 g, platinum crucible)

Assay Weigh accurately about 30 mg of Hydroxypropylcellulose, place into a reaction vial, add exactly 60 mg of adipic acid, 2 mL of the internal standard solution, and 1 mL of hydroiodic acid, stopper tightly and weigh accurately the mass of the mixture. Place the vial in a drying oven at 115 ± 2 ℃, continue heating for 70 minutes while continuously shaking, cool, and weigh the mass accurately. If the difference of the mass before heating from after heating is more than 10 mg, prepare a new test solution. If the difference is NMT 10 mg, allow to stand to separate the upper layer. After separation, pierce through the septum of the vial with a cooled syringe and take a sufficient amount of the upper layer, and use this solution as the test solution. Place 60 mg of adipic acid, 2 mL of the internal standard solution and 1 mL of hydroiodic acid in a reaction vial. Stopper tightly and weigh the mass accurately. Inject 25 μL of isopropyl iodide RS through the septum, and weigh the mass accurately again. Shake well the reaction vial to mix, allow to stand to separate the upper layer. After phase separation, pierce through the septum of the vial with a cooled syringe and take a sufficient amount of the upper layer, and use this solution as the standard solution. Perform the test with 2 μL each of the test solution and standard solution as directed under the Gas Chromatography according to the following conditions, and determine the peak area ratios, Q_T and Q_S, of isopropyl iodide to that of the internal standard.

$$\text{Amount (\%) of hydroxypropoxy group } (C_3H_7O_2) = (Q_T \times F \times 1.15 \times 75.1) / (W_T \times 170.0) \times 100$$

F: Response factor = $(W_S \times C) / (Q_S \times 100)$

W_T: Amount (mg) of the sample, calculated on the dried basis

W_S: Amount (mg) of isopropyl iodide RS

C: Content (%) of isopropyl iodide R

75.1: Molar mass of hydroxypropoxy group

170.0: Molar mass of isopropyl iodide

1.15: Correction factor

Internal standard solution—A solution of

methylcyclohexane in o-xylene (1 in 50)

Operating conditions

Detector: A flame ionization detector

Column: A fused silica capillary column for gas chromatography, about 0.53 mm in internal diameter and about 30 m in length, coated with poly-(dimethyl)siloxane for gas chromatography in 3-μm thickness.

Column temperature: Maintain the temperature at 40 ℃ for 3 minutes, then raise the temperature to 100 °C at the rate of 10 °C per minute, then raise the temperature to 250 °C at the rate of 50 °C per minute, and maintain the temperature at 250 °C for 3 minutes.

Sample injection port temperature: A constant temperature of about 180 ℃

Detector temperature: A constant temperature of about 280 ℃

Carrier gas: Helium

Flow rate: 52 cm/sec

Split ratio: About 1 : 50

Time span of measurement: 15 minutes

System suitability

System performance: Proceed with 2 μL of the standard solution according to the above operating conditions; isopropyl iodide and the internal standard are eluted in this order; the relative retention time of isopropyl iodide to the internal standard is about 0.8 and the resolution is NLT 2.0. The retention time of internal is about 8 minutes.

System repeatability: Repeat the test 6 times with 2 μL each of the standard solution according to the above conditions; the relative standard deviation of response factor F is NMT 2.0%.

Packaging and storage Preserve in well-closed containers.

Low Substituted Hydroxypropylcellulose
저치환도히드록시프로필셀룰로오스

[*9004-64-2*, Hydroxypropylcellulose]

Low Substituted Hydorxypropylcellulose is a low substituted hydroxypropylether of cellulose. Low Substituted Hydroxypropylcellulose contains NLT 5.0% and NMT 16.0% of hydroxypropoxyl group ($-OC_3H_6OH$: 75.09), calculated on the dried basis.

Description Low Substituted Hydroxypropylcellulose occurs as a white to yellowish white fibrous or granular powder and is almost tasteless and odorless.
It is hygroscopic.
It is practically insoluble in ethanol (95) or ether.
It is soluble in a solution of sodium hydroxide (1 in 10); the solution becomes a viscous liquid.
It swells in water, sodium carbonate TS or 2 mol/L hydrochloric acid TS.

Identification (1) To 0.1 g of Low Substituted Hydroxypropylcellulose, add 10 mL of water, and shake vigorously to mix; it does not dissolve.

(2) To the solution obtained in (1), add 1 g of sodium hydroxide, mix until it becomes homogeneous, and use this solution as the test solution. Take 5 mL of the test solution, add 10 mL of a mixture of acetone and methanol (4 : 1), shake to mix; a white, flocculent precipitate is formed.

(3) Determine the infrared spectra of Low Substituted Hydroxypropylcellulose and low substituted hydroxypropylcellulose RS, previously dried, as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

pH To 1.0 g of Low Substituted Hydroxypropylcellulose, add 100 mL of freshly boiled and cooled water, and shake to mix; the pH of this solution is between 5.0 and 7.5.

Purity (1) ***Chloride***—To 0.5 g of Low Substituted Hydroxypropylcellulose, add 30 mL of hot water, stir well to mix, heat on a water bath for 10 minutes, and filter the clear supernatant by decantation while the solution is hot. Then, wash the residue thoroughly with 50 mL of hot water, and combine the washings with the filtrate. After cooling, add water to make 100 mL. Take 5 mL of the water layer, and add 6 mL of dilute nitric acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution with 0.25 mL of 0.01 mol/L hydrochloric acid (NMT 0.355%).

Loss on drying NMT 5.0% (1 g, 105 ℃, 1 hour).

Residue on ignition NMT 0.8% (1.0 g)

Assay (1) ***Apparatus—Reaction vial*** Use a 5-mL pressure-tight glass vial, about 20 mm in outside diameter, about 50 mm in height, having 20 mm in outside diameter and 13 mm in internal diameter at the neck, equipped with a pressure-tight septum of butyl-rubber with surface processed with fluoroplastics, which can be fixed tightly to vial with aluminum cap, or another structure providing an equivalent air-tightness.

Heater: Use a heating module that has a square-shape aluminum block with holes 20 mm in diameter and 32 mm in depth, into which the reaction vial fits. The heating module is also equipped with a magnetic stirrer capable of mixing the contents of the reaction vial, or use a reciprocal shaker that performs a reciprocating motion of approximately 100 times per minute.

(2) ***Procedure***—Weigh accurately about 65 mg of Low Substituted Hydroxypropylcellulose, place into a reaction vial, add 60 to 100 mg of adipic acid, 2.0 mL of the internal standard solution, and 2.0 mL of hydroiodic acid, immediately stopper tightly and weigh accurately the mass of the mixture. Using a magnetic stirrer or a shaker, mix the contents of the reaction vial continuously for 60 minutes, while heating and maintaining the temperature of the contents of the vial at 130 ± 2°. If magnetic stirrer or shaker cannot be used, shake the vial well by hand every 5 minutes during the initial 30 minutes of the heating time. Allow the vial to cool, and weigh accurately the mass of the mixture, and if the weight loss is NMT 26 mg or there is no leak of the contents, use the upper layer of the mixture test solution. Separately, add 60 to 100 mg of adipic acid, 2.0 mL of the internal standard solution, and 2.0 mL of hydroiodic acid into a reaction vial, stopper tightly and weigh accurately the mass of the mixture. Inject 15 μL of isopropyl iodide RS through the stopper using a micro-syringe, and weigh the mass accurately. Shake the reaction vial to mix, take the upper layer of the contents, and use this solution as the standard solution. Perform the test with 1 to 2 μL of the test solution and the standard solution as directed under the gas chromatography according to the following conditions to obtain the peak area ratios, Q_T and Q_S, of isopropyl iodide to that of the internal standard.

Amount (%) of hydroxypropoxy group ($C_3H_7O_2$)
$= (Q_T / Q_S) \times (W_S / W_T) \times 44.17$

W_S: Amount (mg) of isopropyl iodide RS
W_T: Amount (mg) of the sample, calculated on the dried basis

Internal standard solution—A solution of n-octane in o-xylene (3 in 100).

Operating conditions
Detector: A thermal conductivity detector or a flame ionization detector
Column: A fused silica column about 0.53 mm in internal diameter and about 30 m in length, coated inside with dimethylpolysiloxane for gas chromatography, in 3-μm thickness.
Column temperature: Maintain the temperature at 50 ℃ for 3 minutes, then raise the temperature to 100 °C at the rate of 10 °C per minute, then raise the temperature to 250 °C at the rate of 35 °C per minute, and maintain the temperature at 250 °C for 8 minutes.
Sample injection port temperature: A constant temperature of about 250 ℃
Detector temperature: A constant temperature of about 280 ℃
Carrier gas: Helium
Flow rate: 4.3 mL/min (adjust the flow rate so that the retention time of the internal standard is about 10 minutes. The relative retention time of isopropyl iodide to n-octane is about 0.8.)
Split ratio: 1 : 40
Time span of measurement: 20.3 minutes after injecting the test solution
System suitability
System performance: Perform the test with 1 to 2 μL of the standard solution according to the above operating conditions; isopropyl iodide and the internal standard are eluted in this order with the resolution of their peaks being NLT 5.
System repeatability: Repeat the test 6 times with 1 to 2 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak area ratios of isopropyl iodide to the internal standard is NMT 2.0%.

Packaging and storage Preserve in tight containers.

Hydroxypropyl Starch
히드록시프로필전분

Hydroxypropyl Starch is a hydroxypropyl ether of corn starch.

Hydroxypropyl Starch, when dried, contains NLT 2.0% and NMT 7.0% of hydroxypropoxyl group ($-OC_3H_6OH$: 75.09).

Description Hydroxypropyl Starch occurs as a white to yellowish white powder and is odorless and tasteless.
Under a microscope, Hydroxypropyl Starch appears as spherical or polygonal granules, varying in size.
It is practically insoluble in water.
It is practically insoluble in ethanol or in ether.

Identification (1) Weigh 1 g of Hydroxypropyl Starch, add 50 mL of water, boil, and cool; the resulting solution becomes a cloudy paste-like liquid.

(2) To 5 mL of the paste-like solution obtained in (1), add 1~2 drops of iodine TS; the resulting solution exhibits a deep blue color.

(3) Dry Hydroxypropyl Starch at 105°C for 6 hours, dissolve 0.1 g of dried Hydroxypropyl Starch in 80 mL of water by heating, and then add water to make 100 mL. Add 1 mL of this solution to 8 mL of sulfuric acid while cooling and heat on a water bath for 3 minutes. After cooling, add 0.6 mL of ninhydrin sodium bisulfite solution while cooling, shake to mix, and allow to stand at 25°C; the resulting solution exhibits a bluish purple to violet color within 100 minutes.

pH Dissolve 1.0 g of Hydroxypropyl Starch in 50 mL of freshly boiled and cooled water, heat on a water bath for 15 minutes, and cool down to the room temperature; the pH of this solution is 5.0 to 7.5.

Purity (1) ***Chloride***—Weigh 1.0 g of Hydroxypropyl Starch, add 80 mL of water and 4 mL of nitric acid, heat on a water bath for 20 minutes, cool, add water to make 100 mL, and filter. To 10 mL of the filtrate, add 6 mL of dilute nitric acid and water to make 50 mL. Use this solution as the test solution and perform the test as directed under the Chloride. Prepare the control solution with 0.4 mL of 0.01 mol/L hydrochloric acid (NMT 0.142%).

(2) ***Heavy metals***—Proceed with 1.0 g of Hydroxypropyl Starch according to Method 3 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 20 ppm).

(3) ***Arsenic***—Prepare the test solution with 0.4 g of Hydroxypropyl Starch according to Method 3 and perform the test (NMT 5 ppm).

Loss on drying NMT 15.0% (1 g, 105°C, 6 hours).

Residue on ignition NMT 0.5% (1 g).

Assay (1) ***Apparatus***—(i) Decomposition bottle: A pressure-resistant, 5-mL glass bottle with a screw stopper and a cone-shaped bottom, about 20 mm in outer diameter, about 50 mm in height up to the head and about 2 mL in volume up to the height of about 30 mm. The stopper is made of heat-resistant resin, and the inner stopper or the seal is made of fluorine resin.

(ii) Heater: A square metal aluminum block, 60 to 80 mm in thickness, which contains a hole with a diameter of 20.6 mm and a depth of 32 mm. It has a structure that can control the temperature inside the block within the range of ± 1 ℃.

(2) ***Procedure***—Weigh accurately 0.5 g of Hydroxypropyl Starch, previously dried, place in the decomposition bottle, add 0.1 g of adipic acid, 0.2 mL of the internal standard solution, and 2.0 mL of hydroiodic acid, stopper the bottle, and weigh the mass accurately. Shake the decomposition bottle for 30 seconds, then heat at 150℃ for 30 minutes using the heater, while shaking to mix every 5 minutes, and continue heating for another 30 minutes. After cooling, weigh the mass accurately. If the loss on heating is NMT 10 mg, use the upper layer as the test solution. Separately, add 0.5 g of corn starch, previously dried at 105°C for 6 hours, 0.1 g of adipic acid, 0.2 mL of the internal standard solution, and 2.0 mL of hydroiodic acid into the decomposition bottle, stopper the bottle, weigh the mass accurately, and add 30 μL of isopropyl iodide for assay with a microsyringe, and weigh the mass accurately. Shake the decomposition bottle for 30 seconds, then proceed in the same manner as in the preparation of the test solution, and use the resulting solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Liquid Chromatography according

to the operating conditions, and determine the peak areas of isopropyl iodide and the internal standard.

$$\text{Amount (\%) of hydroxypropoxyl group } (C_3H_7O_2) = \frac{Q_I}{Q_{SI}} \times \frac{Q_{ST}}{Q_T} \times \frac{W_{SI}}{Amount\ (mg)\ of\ sample} \times 44.17$$

Q_I :Peak area of isopropyl iodide from the test solution

Q_T: Peak area of *n*-octane from the test solution

Q_{SI}: Peak area of isopropyl iodide from the standard solution

Q_{ST}: Peak area of *n*-octane from the standard solution

W_{SI}: Amount (mg) of isopropyl iodide from the standard solution

Internal standard solution—Take 10.0 mL of *n*-octane in a 50-mL volumetric flask and add *o*-xylene to make 50 mL.

Operating conditions

Detector: A thermal conductivity detector

Column: A glass column about 3 mm in internal diameter and about 3 m in length, packed with diatomaceous earth for gas chromatography, which is coated with 20% methyl silicon polymer (177 to 250 μm in thickness).

Column temperature: A constant temperature of about 100 °C.

Carrier gas: Helium

Flow rate: Adjust the flow rate with a constant volume of 20 to 40 mL/min, so that the retention time of *n*-octane is 7 to 10 minutes.

Packaging and storage Preserve in tight containers.

Hypromellose
히프로멜로오스

Hydroxypropylmethylcellulose
Cellulose, 2-hydroxypropyl methyl ether
[9004-65-3]

Hypromellose is a methyl and hydroxypropyl mixed ether of cellulose. There are four substitution types of Hypromellose, which are 1828, 2208, 2906, and 2910. Hypromellose contains methoxy group (-OCH_3 : 31.03) and hydroxypropoxy group (-OC_3H_6OH : 75.09) as shown in the following table, calculated on the dried basis.

The viscosity of Hypromellose is shown in millipascal second (mPa·s) on the label, along with its substitution type.

Substitution Type	Methoxy Group (%)		Hydroxypropoxy Group (%)	
	Min.	Max.	Min.	Max.
1828	16.5	20.0	23.0	32.0
2208	19.0	24.0	4.0	12.0
2906	27.0	30.0	4.0	7.5
2910	28.0	30.0	7.0	12.0

Description Hypromellose is white to yellowish white, powder or granules.
It is practically insoluble in ethanol (99.5).
It swells with water and becomes a clear or slightly turbid, viscous solution.

Identification (1) Disperse evenly 1.0 g of Hypromellose over the surface of 100 mL of water in a beaker, while gently tapping the top of the beaker, if necessary, and allow to stand for 1 to 2 minutes; it aggregates on the surface of water.

(2) Add 1.0 g of Hypromellose to 100 mL of hot water and stir; it becomes a suspension. Cool the suspension to 10 ℃, and stir to mix; the resulting solution is a clear or turbid, viscous fluid

(3) Take 0.1 mL of the final solution obtained in (2), add 9 mL of diluted sulfuric acid (9 in 10), shake to mix, heat on a water bath for exactly 3 minutes, immediately cool in an ice bath, add carefully 0.6 mL of ninhydrin TS, shake to mix and allow to stand at 25 ℃; the solution exhibits a red color at first and the color changes to violet within 100 minutes

(4) Pour and spread out 2 to 3 mL of the solution obtained in (2) on to a glass plate and allow the water to evaporate ; a transparent film is formed.

(5) Pipet 50 mL of water, add exactly 50 mL of the final solution obtained in (2) and warm to rise the temperature at a rate of 2 to 5°C per minute while stirring. The temperature of congealing, when a white turbidity of the solution starts to increase, is NLT 50 °C.

Viscosity **Method 1:** Apply to Hypromellose having a labeled viscosity of less than 600 mPa·s. Place an amount of Hypromellose, accurately weighed, equivalent to 4.000 g, calculated on the dried basis, in a wide-mouth bottle, add hot water to make 200.0 g, stopper the bottle, stir by mechanical mixer at 350 to 450 revolutions per minute for 10 to 20 minutes to obtain a homogeneous dispersion. If necessary, take off the sample attached on the walls of the bottle, put them in the dispersion, and dissolve on a water bath not exceeding 10 °C for 20 to 40 minutes while stirring. If necessary, add cold water, to make 200.0 g, centrifuge the solution to remove any air bubbles entrapped inside or on the surface, and use this solution as the test solution. Perform the test with the test solution at 20 ± 1 °C as directed in Method 1 under the Viscosity; the value is 80% to 120% of the labeled viscosity.

Method 2: Apply to Hypromellose having a labeled viscosity of NLT 600 mPa·s. Place an amount of Hypromellose, accurately weighed, equivalent to 10.00 g, calculated on the dried basis, in a wide-mouth bottle, add hot water to make 500.0 g, proceed in the same manner as Method 1, and use this solution as the test solution. Perform the test with the test solution at 20 ± 1 °C as directed in Method 2 under the Viscosity using a single cylinder-type rotational viscometer according to the following operating conditions; the value is 75% to 140% of the labeled viscosity.

Operating conditions

Apparatus: Brookfield type viscometer LV model or equivalent

Rotor No., rotation frequency, and conversion factor: use as shown in the following table, depending on the labeled viscosity

Labeled viscosity (mPa·s)	Rotor No.	Rotation frequency /min	Conversion factor
≥ 600 and < 1400	3	60	20
≥ 1400 and < 3500	3	12	100
≥ 3500 and < 9500	4	60	100
≥ 9500 and < 99500	4	6	1000
≥ 99500	4	3	2000

Operation of apparatus: Read value after 2 minutes of rotation by operating the apparatus, and stop the rotation for 2

minutes. Repeat the procedure twice more, and average three measurements.

pH Dip the detection port in the test solution obtained in the Viscosity and allow to stand for 5 minutes; the pH of the solution is between 5.0 and 8.0.

Loss on drying NMT 5.0% (1 g, 105 °C, 1 hour).

Residue on ignition NMT 1.5% (1 g).

Assay (1) ***Apparatus—Reaction vial*** Use a 5-mL pressure-tight vial, about 20 mm in outside diameter, about 50 mm in height, having the 20 mm in outside diameter and 13 mm in internal diameter at the neck, equipped with a pressure-tight septum of butyl-rubber with surface processed with fluoroplastics, which can be fixed tightly to vial with aluminum cap, or another structure providing an equivalent air-tightness.

Heater: Use a heating module that has a square-shape aluminum block with holes about 20 mm in diameter and 32 mm in depth, into which the reaction vial fits. The heating module is also equipped with a magnetic stirrer capable of mixing the contents of the reaction vial, or use a reciprocal shaker that performs a reciprocating motion of approximately 100 times per minute.

(2) ***Procedure***—Weigh accurately about 65 mg of Hypromellose, place into a reaction bottle, add 60 to 100 mg of adipic acid, 2.0 mL of the internal standard solution, and 2.0 mL of hydroiodic acid, immediately stopper tightly and weigh accurately the mass of the mixture. Using a magnetic stirrer or a shaker, mix the contents of the reaction vial continuously for 60 minutes, while heating the plate and maintaining the temperature of the contents of the vial at 130 ± 2°. If magnetic stirrer or shaker cannot be used, shake the vial well by hand every 5 minutes during the initial 30 minutes of the heating time. Allow the vial to cool, and weigh accurately the mass of the mixture, and if the weight loss is NLT 26 mg or there is no leak of the contents, use the upper layer of the mixture as the test solution. Separately, add 60 to 100 mg of adipic acid, 2.0 mL of the internal standard solution, and 2.0 mL of hydroiodic acid into a reaction bottle, stopper tightly and weigh accurately the mass of the mixture. Add 45 μL of methyl iodide RS and 15 to 22 μL of isopropyl iodide RS through the septum using micro-syringe with weighing accurately each time. Shake the reaction bottle thoroughly and use the upper layer of the mixture as the standard solution. Perform the test with 1 to 2 μL each of the test solution and the standard solution as directed under the Gas Chromatography according to the following operating conditions and calculate the peak area ratios, Q_{Ta} and Q_{Sa}, of methyl iodide to that of the internal standard and Q_{Tb} and Q_{Sb}, of isopropyl iodide to that of the internal standard, respectively, in each solution.

$$\text{Content (\%) of methoxy group} = (Q_{Ta} / Q_{Sa}) \times (W_{Sa} / W) \times 21.86$$

$$\text{Content (\%) of hydroxypropoxy group} = (Q_{Tb} / Q_{Sb}) \times (W_{Sb} / W) \times 44.17$$

W_{Sa}: Amount (mg) of methyl iodide RS
W_{Sb}: Amount (mg) of isopropyl iodide RS
W: Amount (mg) of the sample, calculated on the dried basis

Internal standard solution—A solution of n-octane in o-xylene (3 in 100).

Operating conditions

Detector: A thermal conductivity detector or a flame ionization detector

Column: A fused silica column 0.53 mm in internal diameter and 30 m in length coated inside with dimethylpolysiloxane for gas chromatography in 3-μm thickness.

Column temperature: Maintain the temperature at 50 °C for 3 minutes, then raise the temperature to 100 °C at the rate of 10 °C per minute, then raise the temperature to 250 °C at the rate of 35 °C per minute, and maintain the temperature at 250 °C for 8 minutes.

Sample injection port temperature: A constant temperature of about 250 °C

Detector temperature: A constant temperature of about 280 °C

Carrier gas: Helium

Flow rate: 4.3 mL/min (adjust the flow rate so that the retention time of the internal standard is about 10 minutes.)

Split ratio: 1 : 40

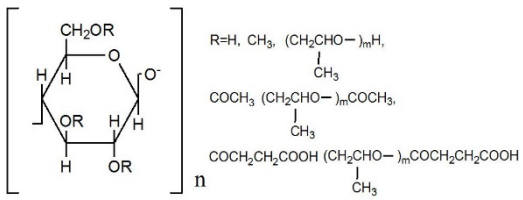
System suitability

System performance: Perform the test with 1 to 2 μL of the standard solution according to the above operating conditions; methyl iodide, isopropyl iodide and the internal standard are eluted in this order with the resolution of their peaks being NLT 5.

System repeatability: Repeat the test 6 times with 1 to 2 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak area ratios of methyl iodide and isopropyl iodide to the internal standard is NMT 2.0%, respectively.

Packaging and storage Preserve in well-closed containers.

Hypromellose Acetate Succinate
히프로멜로오스아세테이트숙시네이트



Hypromellose Acetate Succinate is a mixed ester of acetic acid and monosuccinic acid of hypromellose.

Hypromellose Acetate Succinate, when dried, contains NLT 12.0% and NMT 28.0% of the methoxyl group (-OCH_3: 31.03), NLT 4.0% and NMT 23.0% of the hydroxypropoxyl group (-OC_3H_6OH: 75.09), NLT 2.0% and NMT 16.0% of the acetyl group (-$COCH_3$: 43.04) and NLT 4.0% and NMT 28.0% of succinic acid group (-$COC_2H_4CO_2H$: 101.08).

The viscosity of Hypromellose Acetate Succinate is expressed in mm^2/s.

Description Hypromellose Acetate Succinate occurs as a white to pale yellowish white powder or granule. It is odorless or has a slight, acetic acid-like odor.

It is soluble in sodium hydroxide TS and practically insoluble in anhydrous ethanol, in *o*-xylene, in hexane or in water.

It becomes a colorless or turbid viscous liquid when a mixture of

anhydrous ethanol and dichloromethane (1:1) or acetone is added.
It is slightly hygroscopic.

Identification (1) To 10 mg of Hypromellose Acetate Succinate, add 1 mL of water or 2 mL of anthrone TS and shake to mix; the resulting solution exhibits a green color, which slowly changes to a dark blue color.

(2) Dissolve 10 mg of Hypromellose Acetate Succinate in 40 mL of sodium hydroxide TS and add water to make 100 mL. To 0.1 mL of this solution, add 9 mL of diluted sulfuric acid (9 in 10), shake well to mix, heat on a water bath for exactly 3 minutes, and immediately cool in ice water. Carefully add 0.6 mL of ninhydrin solution, shake to mix, and allow to stand at 25°C; the resulting solution exhibits a pale red color at first, which then changes to a reddish purple color within 100 minutes.

(3) Put 5 mg of Hypromellose Acetate Succinate in a small test tube, add 2 drops of a solution of acetone in benzoyl peroxide (1 in 10), evaporate to dryness on a water bath, fix a glass rod with chromotropic acid TS on the tip inside the test tube with a cork stopper, and heat at 125°C in an oil bath for 5 to 6 minutes; the chromotropic acid TS exhibits a purple color.

(4) To 50 mg of Hypromellose Acetate Succinate, add 2 mL of potassium hydroxide-ethanol TS and heat on a water bath for 5 minutes. After cooling, add 2 mL of diluted sulfuric acid (2 in 7) and boil slowly for 1 minute; a smell of ethyl acetate is emitted.

(5) Add 0.1 g of Hypromellose Acetate Succinate to 2 mL of sodium hydroxide TS and heat on a water bath for 5 minutes. After cooling, add 1 mL of dilute hydrochloric acid and diluted ammonia TS (1 in 10) to adjust the pH to about 6, and then add 2 to 3 drops of ferric chloride TS; the resulting solution becomes turbid in brown.

Viscosity Weigh 2.0 g of Hypromellose Acetate Succinate, previously dried, add dilute sodium hydroxide TS to make 100.0 g, stopper the container, and shake continuously for 30 minutes to dissolve. Proceed with this solution according to Method 1 under the Viscosity at 20 ± 0.1 ℃; the viscosity of Hypromellose Acetate Succinate is NLT 80% and NMT 120% of the labeled unit.

Purity (1) ***Heavy metals***—Proceed with 2.0 g of Hypromellose Acetate Succinate according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 10 ppm).

(2) ***Arsenic***—Prepare the test solution with 1.0 g of Hypromellose Acetate Succinate according to Method 3 and perform the test (NMT 2 ppm).

(3) ***Succinic acid***—Weigh accurately 1.5 g of Hypromellose Acetate Succinate, previously dried, transfer into a separatory funnel, dissolve in 50 mL of a mixture of anhydrous ethanol and dichloromethane mixture (3 : 2), and add 75 mL of water, while mixing by shaking. Then, add 50 mL of hexane and 1 g of sodium chloride, shake well to mix, and take the aqueous layer separately. Wash the filtrate with 50 mL of water, combine the solution used for washing and the aqueous layer, and titrate the combined solution with 0.1 mol/L sodium hydroxide VS (indicator: 3 drops of phenolphthalein TS). Perform a blank test in the same manner and make any necessary correction [succinic acid ($C_4H_6O_4$: 118.09): NMT 1.0%].

Each mL of 0.1 mol/L sodium hydroxide VS
= 5.904 mg of C4H6O4

Loss on drying NMT 5.0% (1 g, 105 ℃, 1 hours).

Residue on ignition NMT 0.2% (1 g)

Assay (1) ***Methoxyl group and hydroxypropoxyl group***—(i) Decomposition bottle: A pressure-resistant, 5-mL glass bottle with a screw stopper and a rounded inner side at the bottom, about 20 mm in outer diameter, about 50 mm in height from the bottom to the head part and about 2 mL in volume up to the height of about 30 mm. The stopper is made of heat-resistant resin, and the inner stopper is made of fluorine resin.

Heater: A square metal aluminum block, 60 to 80 mm in thickness, which contains a hole with a diameter of 20.6 mm and a depth of 32 mm. It has a structure that can control the temperature inside the block within the range of ± 1 ℃.

(ii) Procedure: Weigh accurately about 65 mg of Hypromellose Acetate Succinate, previously dried, place in a decomposition bottle, add 65 mg of adipic acid, 2.0 mL of the internal standard solution and 2.0 mL of hydroiodic acid, stopper the bottle, and weigh accurately the mass. Shake the decomposition bottle for 30 seconds to mix, then heat at 150℃ for 30 minutes using the heater, while shaking and mixing every 5 minutes, and continue heating for another 30 minutes. After cooling, weigh accurately the mass. If the loss on heating is NMT 10 mg, use the upper layer as the test solution. Separately, take 65 mg of adipic acid, 2.0 mL of the internal standard solution and 2.0 mL of hydroiodic acid in a decomposition bottle, seal it, weigh accurately the mass, add 15 μL of isopropyl iodide for assay, and weigh accurately the mass. Then, add 45 μL of methyl iodide for assay and weigh accurately the mass. Shake the decomposition bottle for 30 seconds to mix and use the supernatant as the standard solution. Perform the test with 2 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions. Calculate the peak area ratios, Q_{Ta} and Q_{Tb}, of methyl iodide and isopropyl iodide, respectively, to the internal standard from the test solution and the peak area ratios, Q_{Sa} and Q_{Sb}, of methyl iodide and isopropyl iodide, respectively, to the internal standard from the standard solution.

$$\text{Amount (mg) of methoxyl group } (CH_3O) = \frac{Q_{Ta}}{Q_{Sa}} \times \frac{W_{Sa}}{Amount\ (mg)\ of\ sample} \times 21.864$$

$$\text{Amount (mg) of hydroxypropoxyl group } (C_3H_7O_2) = \frac{Q_{Tb}}{Q_{Sb}} \times \frac{W_{Sb}}{Amount\ (mg)\ of\ sample} \times 44.17$$

W_{Sa}: Amount (mg) of methyl iodide in the standard solution
W_{Sb}: Amount (mg) of isopropyl iodide in the standard solution

Internal standard solution—A solution of toluene in o-xylene (3 in 100).

Operating conditions

Detector: A thermal conductivity detector or flame ionization detector

Column: A glass column about 3 mm in internal diameter and about 3 m in length, packed with diatomaceous earth for gas chromatography, which is coated with methyl silicon polymer (180 to 250 μm in thickness) at a rate of 20%.

Column temperature: A constant temperature of about 100 °C.

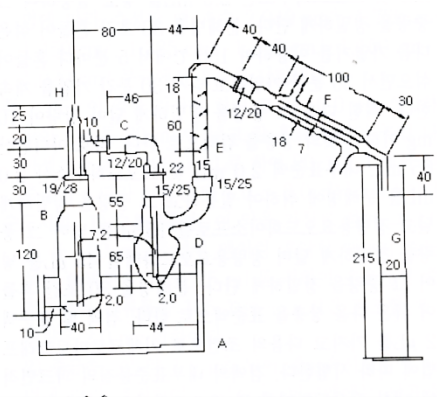
Carrier gas: Helium

Flow rate: Adjust the flow rate so that the retention time of

the internal standard is about 8 minutes.

Column selection: Proceed with 2 μL of the standard solution according to the above conditions. At this time select the column from which methyl iodide, isopropyl iodide and internal standards are eluted in this order with each peak being fully separated.

(2) ***Acetyl group***—(i) Apparatus: Use those displayed in the picture.



A: Oil bath
B: 100-mL steam generator
C: Steam and nitrogen injection port
D: 25-mL decomposition flask
E: Thermal insulation column made of aluminum foil
F: Cooler
G: 100-mL measuring cylinder
H: Nitrogen injection port

(ii)) Procedure: Weigh accurately about 0.15 g of Hypromellose Acetate Succinate, previously dried, place in the decomposition flask D, add 5 mL of sodium hydroxide TS, shake to dissolve, and allow to decompose on a water bath at 60°C for 2 hours. After cooling, add 5 mL of diluted phosphoric acid (1 in 6), immediately assemble the apparatus, and immerse the decomposition flask and the steam generator in the oil bath A at 155°C, while passing nitrogen through the nitrogen injection port H at a rate of 1 to 2 bubbles per second. Continue the distillation at the same temperature and collect the remaining solution in the measuring cylinder G. Take 60 mL of the remaining solution, rinse the inside of the cooling tube F with 10 mL of water, and combine the solution used for washing with the rest of the solution. To this solution, add 5.0 mL of the internal standard solution, add water to make 100.0 mL, and use this solution as the test solution. Separately, weigh accurately about 0.1 g of acetic acid RS and add diluted phosphoric acid (1 in 5000) to make exactly 100.0 mL. Pipet 15.0 mL of this solution, add 5.0 mL of the internal standard solution, add diluted phosphoric acid (1 in 5000) to make exactly 100.0 mL, and use this solution as the standard solution. Perform the test with 2 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and calculate the peak area ratios, Q_T and Q_S, of acetic acid to the internal standard, respectively.

$$\text{Amount (mg) of acetyl group } (C_2H_3O) = \frac{Q_T}{Q_S} \times \frac{W_S}{Amount\ (mg)\ of\ sample} \times \frac{15}{100} \times 71.68$$

W_S: Amount (mg) of acetic acid RS

Internal standard solution—Take 1.0 mL of propionic acid for gas chromatography and add diluted phosphoric acid (1 in 5000) to make exactly 250 mL.

Operating conditions

Detector: A flame ionization detector

Column: A glass column about 3 mm in internal diameter and about 2 m in length, packed with polymer beads (180 to 250 μm) impregnated with phosphoric acid at a rate of 10%.

Column temperature: A constant temperature of about 180 °C.

Carrier gas: Helium

Flow rate: Adjust the flow rate so that the retention time of the internal standard is about 3 minutes.

Selection of column: Proceed with 2 μL of the standard solution according to the above conditions. At this time, use a column from which acetic acid and the internal standard are eluted in this order with each peak being fully separated.

(3) ***Succinic acid group***—(i) Weigh accurately 1 g of Hypromellose Acetate Succinate, previously dried, dissolve in 50 mL of a mixture of ethanol, acetone and water (2 : 2 : 1), titrate with 0.1 mol/L sodium hydroxide VS to determine the volume of 0.1 mol/L sodium hydroxide VS consumed as a mL (Indicator: 2 drops of phenolphthalein solution). Perform a blank test in the same manner, make any necessary correction, and determine the volume of 0.1 mol/L sodium hydroxide VS consumed as b mL.

$$\text{Amount (\%) of succinic acid group } (C_4H_5O_3) = \frac{(a-b)\times 1.0108}{Amount\ (mg)\ of\ sample} - \text{Amount (\%) of succinic acid } (C_4H_6O_4) \times 1.7120$$

Packaging and storage Preserve in tight containers.

Hypromellose Phthalate
히프로멜로오스프탈레이트

Hydroxypropylmethylcellulose Phthalate
Hydroxypropylmethylcellulose Phthalate
[9050-31-1]

Hypromellulose Phthalate is a monophthalic acid ester of hydpomellulose.

Hypromellulose Phthalate contains methoxy group (-OCH_3: 31.03), hydroxypropoxy group (-$OCH_2CHOHCH_3$: 75.09) and phthalyl group (carboxybenzoyl group) (-COC_6H_4COOH: 149.12).

Hypromellose Phthalate contains NLT 21.0% and NMT 35.0% of phthalyl group (carboxybenzoyl group), calculated on the anhydrous basis.

Substitution type	Carboxybenzoyl group (%)	
	Min.	Max.
200731	27.0	35.0
220824	21.0	27.0

The viscosity of Hypromellose Phthalate is shown in millipascal second (mPa·s) on the label, along with its substitution type.

Description Hypromellulose Phthalate is white powder or granules, and is odorless and tasteless.
It is practically insoluble in water, acetonitrile, ethanol (99.5),

and hexane.
It becomes a viscous liquid when a mixture of methanol and dichloromethane (1 : 1) or a mixture of ethanol (99.5) and acetone (1 : 1) is added.

Identification Determine the absorption spectra of Hypromellose Phthalate and hypromellose phthalate RS as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers. The sample is not dried.

Viscosity Dissolve 10 g of Hypromellose Phthalate, previously dried at 105 °C for 1 hour, in 90 g of a mixture of methanol and dichloromethane at a mass ratio of 50% each by mixing and shaking. Determine the viscosity at 20 ± 0.1 °C according to Method 1 under the Viscosity; the value is NLT 80% and NMT 120% of the labeled viscosity.

Purity (1) ***Chloride***—Dissolve 1.0 g of Hypromellose Phthalate in 40 mL of 0.2 mol/L sodium hydroxide TS, add 1 drop of phenolphthalein TS, and shake vigorously to mix until the red color of the solution disappears while adding dilute nitric acid dropwise. Add an additional 20 mL of dilute nitric acid with stirring. Heat on a water bath, with stirring mixing, until the gel-like precipitate formed becomes granular. Cool, and centrifuge the mixture. Take the clear supernatant and wash the precipitate three times with 20 mL each of water by centrifuging each time, combine the clear supernatant and the washings, add water to make 200 mL, and filter. Use 50 mL of the filtrate as the test solution and perform the test. Prepare the control solution by adding 10 mL of 0.2 mol/L sodium hydroxide TS, 7 mL of dilute nitric acid, and water to 0.50 mL of 0.01 mol/L hydrochloric acid to make 50 mL (NMT 0.07%).

(2) ***Free phthalic acid***—Weigh accurately about 2.0 g of Hypromellose Phthalate, add about 50 mL of acetonitrile, sonicate to dissolve partially, add 10 mL of water, sonicate again to dissolve further, cool, add acetonitrile to make exactly 100 mL, and use this solution as the test solution. Separately, weigh accurately about 12.5 mg of phthalic acid, add about 125 mL of acetonitrile, then add 25 mL of water, add acetonitrile to make exactly 250 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions and determine the peak areas, A_T and A_S, of phthalic acid in each solution, respectively; the amount of phthalic acid ($C_8H_6O_4$: 166.13) is NMT 1.0%.

$$\text{Content (\%) of free phthalic acid} = (W_S, W_T) \times (A_T / A_S) \times 40$$

W_S: Amount (mg) of phthalic acid
W_T: Amount (mg) of the sample, calculated on the anhydrous basis.

Operating conditions
Detector: An ultraviolet absorption photometer (wavelength: 235 nm)
Column: A stainless steel column with an internal diameter of about 4.6 mm and a length of about 25 cm, packed with octadecylsilanized silica gel for liquid chromatography (3 μm to 10 μm in particle diameter).
Column temperature: A constant temperature of room temperature
Mobile phase: A mixture of 0.1% trifluoroacetic acid and acetonitrile (9 : 1)
Flow rate: 2.0 mL/min
System suitability
System performance: Proceed with 10 μL of the standard solution according to the above operating conditions; the number of theoretical plates and the symmetry factor of the peak of phthalic acid are NLT 2500 and 1.5 - 2.0, respectively.
System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak areas of phthalic acid is NMT 1.0%.

Water NMT 5.0% (1 g, volumetric titration, direct titration. Use a mixture of ethanol (99.5) and dichloromethane (3 : 2) instead of methanol for Karl Fischer titration).

Residue on ignition NMT 0.2% (1.0 g).

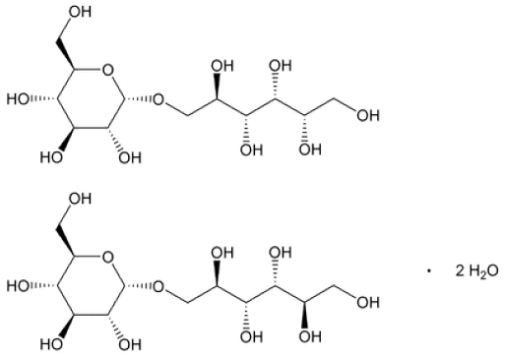
Assay Weigh accurately about 1 g of Hypromellose Phthalate, dissolve in 50 mL of a mixture of ethanol (95), acetone, and water (2 : 2 : 1), and titrate with 0.1 mol/L of sodium hydroxide VS (indicator: 2 drops of phenolphthalein TS). Perform a blank test in the same manner and make any necessary correction.

$$\text{Content (\%) of phthalyl group } (C_8H_5O_3) = [0.01 \times 149.1 \times (V / W)] - [2 \times (149.1 / 166.1) \times P]$$

P: Content (%) of phthalic acid obtained from Phthalic acid
V: Volume (mL) of 0.1 mol/L sodium hydroxide VS used in titration
W: Amount (g) of the sample, calculated on the anhydrous basis

Packaging and storage Preserve in tight containers.

Isomalt Hydrate
이소말트수화물



6-*O*-*α*-*D*-Glucopyranosyl-*D*-glucitol $C_{12}H_{24}O_{11}$: 344.31
1-*O*-*α*-*D*-Glucopyranosyl-*D*-mannitol dihydrate $C_{12}H_{24}O_{11}\cdot 2H_2O$: 380.34

6-*O*-*α*-*D*-Glucopyranosyl-*D*-sorbitol - 1-*O*-*α*-*D*-glucopyranosyl-*D*-mannitol dihydrate [*64519-82-0*]

Isomalt Hydrate is a mixture of 6-*O*-*α*-*D*-glucopyranosyl-*D*-sorbitol and 1-*O*-*α*-*D*-glucopyranosyl-*D*-mannitol.

Isomalt Hydrate contains NLT 98.0% and NMT 102.0% of a mixture of 6-*O*-*α*-*D*-glucopyranosyl-*D*-sorbitol ($C_{12}H_{24}O_{11}$) and 1-*O*-*α*-*D*-glucopyranosyl-*D*-mannitol ($C_{12}H_{24}O_{11}$), and the amount of each component is NLT 3.0%.

The label of Isomalt Hydrate indicates the content (%) of 6-*O*-*α*-*D*-glucopyranosyl-*D*-sorbitol and 1-*O*-*α*-*D*-glucopyranosyl-*D*-mannitol.

Description Isomalt Hydrate occurs as a white powder or grains.
It is very soluble in water and practically insoluble in ethanol (95).

Identification (1) Weigh about 0.5 g each of Isomalt Hydrate and isomalt hydrate RS, dissolve in 100 mL of water, shake well to mix, filter, and use the clear supernatant as the test solution and the standard solution, respectively. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 1 μL each of the test solution and the standard solution on a thin-layer chromatographic plate made of silica gel (with fluorescent indicator) for thin layer chromatography with a thickness of 0.25 mm. Develop the plate with a mixture of ethyl acetate, pyridine, water, acetic acid, and propionic acid (10 : 10 : 2 : 1 : 1) as the developing solvent to a distance of about 10 cm, and air-dry the plate. Immerse the plate in sodium periodate solution (1 in 1000) for 3 seconds and then in a mixture of anhydrous ethanol, sulfuric acid, acetic acid, and anisaldehyde (90 : 5 : 1 : 1) for 3 seconds. Then, air-dry the plate until the spots are visible and expose it to warm vapor; the R_f values of the spots obtained from the test solution and the standard solution are the same.

(2) Proceed with Isomalt Hydrate as directed in the Assay; the retention times of the major peak obtained from the test solution and the standard solution are the same.

Purity (1) Nickel—Weigh accurately an amount of Isomalt Hydrate equivalent to 10.0 g calculated on the anhydrous basis, dissolve it in 30 mL of 2 mol/L acetic acid TS, and add water to make exactly 100 mL. Add exactly 2 mL of ammonium pyrrolidinedithiocarbamate solution (1 in 100) and 10 mL of water-saturated 4-methyl-2-pentanone, and shake for 30 seconds to mix, protected from light. Allow the mixture to stand and use the 4-methyl-2-pentanone layer as the test solution. Separately, weigh accurately an amount of Isomalt Hydrate equivalent to 10.0 g calculated on the anhydrous basis, and place each in three containers, dissolve it in 30 mL of 2 mol/L acetic acid TS, respectively. Add exactly 0.5 mL, 1.0 mL, and 1.5 mL of the nickel standard solution for atomic absorption spectroscopy and add water to make exactly 100 mL, respectively. Proceed in the same manner as the test solution and use these solutions as the standard solutions. Separately, proceed in the same manner as the test solution without using Isomalt Hydrate and use the 4-methyl-2-pentanone layer thus obtained as the blank test solution. Determine the atomic absorption spectra of the test solution, the standard solutions and the blank test solution, as directed in the standard addition method under the Atomic absorption spectroscopy according to the following conditions; the concentration of nickel in the test solution is NMT1 ppm. The blank test solution is used for setting the zero of the instrument. Also, between each measurement of the sample, rinse the sample injection port with water, and use the blank test solution to ascertain that the readings of the absorbance return to zero.

Gas: Acetylene - Air
Lamp : Nickel hollow cathode lamp
Wavelength: 232.0 nm

(2) ***Related substances***—Weigh accurately 0.20 g of Isomalt Hydrate, dissolve it in water to make exactly 10 mL, and use this solution as the test solution. Separately, weigh accurately 10.0 mg of *D*-sorbitol and 10.0 mg of *D*-mannitol, dissolve in water to make to make exactly 100 mL, and use the resulting solutions as the standard solutions. Perform the test with 20 μL each of the test solution and the standard solutions as directed under the Liquid Chromatography according to the following conditions. Determine the peak area of each solution according to the automatic integration method; the peak areas of D-mannitol with a relative retention time of about 1.6 and D-sorbitol with a relative retention time of about 2.0 with respect to 1-*O*-*α*-*D*-glucopyranosyl-*D*-mannitol in the test solution are not greater than the respective peak areas of the standard solution (NMT 0.5%), and the peak area of 6-*O*-*α*-*D*-glucopyranosyl-*D*-sorbitol with a relative retention time of about 1.2 with respect to 1-*O*-*α*-*D*-glucopyranosyl-*D*-mannitol and the peak areas other than the above related substances are not greater than the peak area of *D*-sorbitol in the standard solution (NMT 0.5%). In addition, the sum of the peak areas other than 1-*O*-*α*-*D*-glucopyranosyl-*D*-mannitol and 6-*O*-*α*-*D*-glucopyranosyl-*D*-sorbitol in the test solution is not greater than 4 times the peak area of *D*-sorbitol in the standard solution (NMT 2.0%). However, disregard the peaks whose area is not larger than 1/5 of the peak area of *D*-sorbitol in the standard solution (NMT 0.1%).

Operating conditions

For the detector, column, column temperature, mobile phase and flow rate, proceed as directed in the operating conditions under the Assay.
Time span of measurement: About 2.5 times the retention time of 1-*O*-*α*-*D*-glucopyranosyl-*D*-mannitol

System suitability

Confirmation of detection: Pipet 2 mL of the standard solution and add water to make exactly 10 mL. Confirm that the peak area of *D*-sorbitol obtained from 20 μL of the resulting solution is equivalent to 14% to 26% of the peak area of *D*-sorbitol obtained from the standard solution.

System performance: Proceed as directed in the system suitability under the Assay.

System repeatability: Repeat the test 6 times with 20 μL each of the standard solution according to the above operating conditions; the relative standard deviation of the peak areas of *D*-mannitol and *D*-sorbitol is NMT 2.0%, respectively.

(3) ***Reducing sugars***—Add 10 mL of water to 3.3 g of Isomalt Hydrate and slowly heat the mixture to dissolve. After cooling, add 20 mL of copper (II) citrate TS. Add a few boiling stones and heat the mixture so that it starts to boil after 4 minutes, maintain boiling for 3 minutes, then cool immediately. Add 100 mL of acetic acid (100) solution (3 in 125) and add exactly 20 mL of 0.025 mol/L iodine solution. Add 25 mL of a mixture of water and hydrochloric acid (47 : 3) while stirring. After the precipitate is dissolved, titrate the excess iodine with 0.05 mol/L sodium thiosulfate VS. The endpoint of the titration is when the blue color obtained by adding 1 mL of soluble starch TS is decolorized when the endpoint is nearly reached. The consumed volume of 0.05 mol/L sodium thiosulfate VS is NLT 12.8 mL (NMT 0.3% as glucose).

Copper (II) citrate TS—Dissolve 25 g of copper (II) sulfate pentahydrate, 50 g of citric acid monohydrate, and 144 g of anhydrous sodium carbonate in water to make 1000 mL.

Conductivity Add an appropriate amount of freshly boiled and cooled water to 20 g of Isomalt Hydrate, dissolve by slowly warm at 40 to 50 °C, and cool. Then, add freshly boiled and cooled water to make exactly 100 mL and use this solution as the test

solution. Perform the test at 25 ± 0.1 ℃ while slowly stirring the test solution and determine the conductivity; NMT 20 $\mu S \cdot cm^{-1}$.

Water NMT 7.0% (0.3 g, volumetric titration, direct titration). Use a mixture of methanol for Karl Fischer titration and formamide for Karl Fischer titration (1 : 1) after heating it to 50 ± 5 ℃, instead of methanol for Karl Fischer titration.

Assay Weigh accurately 0.2 g of Isomalt Hydrate, dissolve it in water to make exactly 10 mL, and use this solution as the test solution. Separately, weigh accurately about 0.2 g of isomalt hydrate RS (previously determine the water content in the same manner as for Isomalt Hydrate), dissolve it in water to make exactly 10 mL, and use this solution as the standard solution. Perform the test with 20 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas, A_{Ta}, A_{Tb}, A_{Sa}, and A_{Sb}, of 1-*O*-*α*-*D*-glucopyranosyl-*D*-mannitol and 6-*O*-*α*-*D*-glucopyranosyl-*D*-sorbitol from each of the solutions.

Amount (g) of 1-*O*-*α*-*D*-glucopyranosyl-*D*-mannitol ($C_{12}H_{24}O_{11}$)
$= M_S \times (K_a / 100) \times (A_{Ta} / A_{Sa})$

Amount (g) of 6-*O*-*α*-*D*-glucopyranosyl-*D*-sorbitol ($C_{12}H_{24}O_{11}$)
$= M_S \times (K_b / 100) \times (A_{Tb} / A_{Sb})$

M_S: Mass (g) of isomalt RS, calculated on the anhydrous basis

K_a: Content (%) of 1-*O*-*α*-*D*-glucopyranosyl-*D*-mannitol ($C_{12}H_{24}O_{11}$) in isomalt RS

K_b: Content (%) of 6-*O*-*α*-*D*-glucopyranosyl-*D*-sorbitol ($C_{12}H_{24}O_{11}$) in isomalt RS

Operating conditions

Detector: A differential refractometer at a constant temperature

Column: A stainless steel column about 7.8 mm in internal diameter and about 30 cm in length, packed with strongly acidic ion exchange resin (9 μm in particle diameter) for liquid chromatography (8% cross-linked) (Ca type) consisting of sulfonated polystyrene cross-linked with divinylbenzene. Use a guard column of a stainless steel column about 4.6 mm in internal diameter and about 3 cm in length, with strongly acidic ion exchange resin (9 μm in particle diameter) for liquid chromatography (8% cross-linked) (Ca type) consisting of sulfonated polystyrene cross-linked with divinylbenzene

Column temperature: 80 ± 3 ℃

Mobile phase: Water

Flow rate: Adjust the flow rate so that the retention time of 1-*O*-*α*-*D*-glucopyranosyl-*D*-mannitol is about 12 minutes.

System suitability

System performance: Proceed with 20 μL of the standard solution according to the above conditions; 1-*O*-*α*-*D*-glucopyranosyl-*D*-mannitol and 6-*O*-*α*-*D*-glucopyranosyl-*D*-sorbitol are eluted in this order with the resolution between these peaks being NLT 2.0.

System repeatability: Repeat the test 6 times with 20 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak areas of 1-*O*-*α*-*D*-glucopyranosyl-*D*-mannitol and 6-*O*-*α*-*D*-glucopyranosyl-*D*-sorbitol is NMT 2.0%, respectively.

Packaging and storage Preserve in well-closed containers.

Kaolin
카올린

Kaolin is a native hydrous aluminum silicate.

Description Kaolin occurs as a white to almost white brittle mass or powder, and has a slightly clay-like odor.
It is practically insoluble in water, anhydrous ethanol or ether.
It does not dissolve in dilute hydrochloric acid or sodium hydroxide TS.
Moisten Kaolin with water; it exhibits a dark color and becomes plastic.

Identification (1) Weigh 1 g of Kaolin in a porcelain dish, add 10 mL of water and 5 mL of sulfuric acid, and heat until it is evaporated nearly to dryness. After cooling, add 20 mL of water, boil for 2 to 3 minutes, and filter; the residue is gray.

(2) The filtrate in (1) responds to Chemical identification reactions (1), (2) and (4) for aluminum salt.

Purity (1) ***Acidity or alkalinity***—To 1.0 g of Kaolin, add 25 mL of water, shake well to mix, and filter; the pH of the filtrate is between 4.0 and 7.5.

(2) ***Acid soluble substances***—To 1.0 g of Kaolin, add 20 mL of dilute hydrochloric acid, shake to mix for 15 minutes, and then filter. Evaporate 10 mL of the filtrate to dryness, and ignite at 450 ℃ to 550 ℃ to a constant mass; the residue is NMT 10 mg.

(3) ***Carbonate***—To 1.0 g of Kaolin, add 5 mL of water, stir to mix, and add 10 mL of diluted sulfuric acid (1 in 2); no foam is produced.

(4) ***Heavy metals***—To 1.5 g of Kaolin, add 50 mL of water and 5 mL of hydrochloric acid, boil gently for 20 minutes while shaking well to mix, cool, and centrifuge. Take the clear supernatant, wash the residue twice with 10 mL each of water, and centrifuge each time. Combine the washings with the clear supernatant, and add strong ammonia water dropwise until precipitate is formed slightly, and dissolve the precipitate by adding dilute hydrochloric acid dropwise while vigorous shaking. Add 0.45 g of hydroxylamine hydrochloride to this solution and heat. After cooling, add 0.45 g of sodium acetate and 6 mL of dilute acetic acid, filter if necessary, wash with 10 mL of water, combine the filtrate and the washings, and add water to make 150 mL. Perform the test using 50 mL of this solution as the test solution. For the control solution, to 2.5 mL of lead standard solution, add 0.15 g of hydroxylamine hydrochloride, 0.15 g of sodium acetate, 2 mL of dilute acetic acid, and water to make 50 mL (NMT 50 ppm).

(5) ***Iron***—To 40 mg of Kaolin, add 10 mL of dilute hydrochloric acid, and heat while shaking on a water bath for 10 minutes. After cooling, add 0.5 g of tartaric acid and shake to dissolve. Prepare the test solution with this solution according to Method 2, and then perform the test according to Method B. Prepare the control solution with 2.0 mL of the iron standard solution (NMT 500 ppm).

(6) ***Arsenic***—To 1.0 g of Kaolin, add 5 mL of water and 1 mL of sulfuric acid, heat in a bath until white fumes are evolved, cool, and add water to make 5 mL. Use this solution as the test solution and perform the test (NMT 2 ppm).

(7) ***Foreign matter***—Put 5 g of Kaolin in a beaker, add 100 mL of water, stir to mix, and decant to leave sand. Repeat this procedure several times, using 100 mL of water each time; no sand-like residue remains.

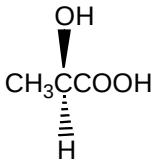
Loss on ignition NMT 15.0% (1 g, 600 ℃, 5 hours).

Plasticity Add 7.5 mL of water to 5 g of Kaolin, and shake well to mix; the resulting mass has no apparent fluidity.

Microbial limit The total aerobic microbial count is NMT 1000 CFU, and the total combined yeasts/moulds count is NMT 100 CFU per 1 g of Kaolin. Also, *Escherichia coli*, *Salmonella*, *Pseudomonas aeruginosa* and *Staphylococcus aureus* are not detected.

Packaging and storage Preserve in well-closed containers.

Lactic Acid
락트산



and enantiomer

$C_3H_6O_3$: 90.08

(2*RS*)-2-Hydroxypropanoic acid [*50-21-5*]

Lactic Acid contains NLT 85.0% and NMT 92.0% of lactic acid ($C_3H_6O_3$), calculated as a mixture of lactic acid and lacatic anhydride.

Description Lactic Acid occurs as a colorless to pale yellow clear and viscous liquid. It is odorless or has a faint, non-repellent odor.
It is miscible with water, ethanol or ether.
It is hygroscopic.

Specific gravity d_{20}^{20}: About 1.20.

Identification An aqueous solution of Lactic Acid (1 in 50) changes blue litmus paper to red, and this solution responds to the Chemical identification reactions for lactate.

Purity (1) ***Chloride***—Perform the test with 1.0 g of Lactic Acid. Prepare the control solution with 1.0 mL of 0.01 mol/L hydrochloric acid (NMT 0.036%).

(2) ***Sulfate***—Perform the test with 2.0 g of Lactic Acid. Prepare the control solution with 0.40 mL of 0.005 mol/L (NMT 0.010%).

(3) ***Heavy metals***—To 2.0 g of Lactic Acid, add 10 mL of water and 1 drop of phenolphthalein TS, add ammonia TS dropwise until the solution exhibits a pale red color, and then add 2 mL of dilute acetic acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution by adding 2 mL of dilute acetic acid and water to 2.0 mL of lead standard solution to make 50 mL (NMT 10 ppm).

(4) ***Mercury***—Spread evenly about 1 g of excipient (a) in a ceramic boat, and add 10 mg to 300 mg of Lactic Acid on top. On top of it, spread evenly about 0.5 g of excipient (a) and 1 g of excipient (b) in this order to form layers. In the case of an automated mercury analyzer equipped with a separate catalyst in the combustion chamber, take only the sample without adding excipients to a nickel boat. Place the boat inside the combustion furnace and heat to about 900 °C with a current of air or oxygen at 0.5 to 1 L/minute. Elute the mercury and collect in a collecting tube. Transfer the mercury vapor to a cold vapor atomic absorption spectrometer by heating the collecting tube to about 700 °C and determine the absorbance as A. Separately, determine the absorbance in the same manner using only the excipients in a ceramic boat and designate as Ab. Separately, proceed with mercury standard solution in the same manner and create a calibration curve from the absorbances. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the sample (NMT 1.0 ppm).

Operating conditions

Apparatus: Use an automated mercury analyzer for specimen combustion, trapping with gold amalgam and measurement using a cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Standard mercury stock solution—Dissolve 0.135 g of mercury (II) chloride in 0.001% L-cysteine solution to make 1000 mL. 1 mL of this solution contains 100 μg of mercury (II) chloride.

Mercury standard solution—Dilute standard mercury stock solution with 0.001% L-cysteine solution to make 0 ng/mL to 200 ng/mL.

Excipients—Use (a) aluminum oxide and (b) a mixture of calcium hydroxide and sodium carbonate (1 : 1), and activate at 950 ℃ for 30 minutes before use.

(5) ***Lead***—Weigh accurately 5.0 g of Lactic Acid, transfer into a platinum crucible, dry and carbonize, and then incinerate at 450 ℃ to 550 ℃. If incineration is not complete, cool and moisten with 2 to 5 mL of nitric acid (1 in 2), 50% magnesium nitrate solution or aluminum nitrate-calcium nitrate solution (prepared by dissolving 40 g of aluminum nitrate and 20 g of calcium nitrate in 100 mL of water) as an incineration supplement, dry, and continue with incineration. If incineration is incomplete, repeat the above procedure once and then add a final 2 mL to 5 mL of nitric acid (1 in 2), if necessary, and incinerate. After incineration, moisten the residue with water, add 2 to 4 mL of hydrochloric acid, and evaporate to dryness. Add 0.5 mol/L nitric acid, dissolve by warming, and filter with filter paper if any insoluble matter exists. Unless otherwise specified, add 0.5 mol/L nitric acid to make 25 mL and use this solution as the test solution. Separately, transfer 1.0 mL of lead standard solution into a platinum crucible, proceed in the same manner as in the preparation of the test solution, and use this solution as the standard solution. Perform the test with the test solution and the standard solutions as directed under Atomic Absorption Spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 2.0 ppm).

Gas: Acetylene or Hydrogen - Air
Lamp: A lead hollow-cathode lamp
Wavelength: 283.3 nm

(6) ***Iron***—Proceed with 4.0 g of Lactic Acid according to Method 1 and perform the test as directed under Method A. Prepare the control solution with 2.0 mL of the iron standard solution (NMT 5 ppm).

(7) ***Arsenic***—Take an amount of Lactic Acid equivalent to 0.8 g of lactic acid, add 5 mL of water, mix, and then add water

to make 10 mL. Take 5 mL of this solution, use this solution as the test solution, and perform the test (NMT 4 ppm).

(8) ***Sugars***—To 1.0 g of Lactic Acid, add 10 mL of water, neutralize with sodium hydroxide TS, add 10 mL of Fehling's TS, and boil for 5 minutes; no red precipitate is produced.

(9) ***Citric acid, oxalic acid, phosphoric acid, and tartaric acid***—To 1.0 g of Lactic Acid, add 1.0 mL of water, then add 40 mL of calcium hydroxide TS, and boil for 2 minutes; no change occurs in the resulting solution does.

(10) ***Glycerin or mannitol***—To 10 mL of Lactic Acid, add 12 mL of ether, and shake to mix; the resulting solution is not turbid.

(11) ***Volatile fatty acid***—Warm Lactic Acid; the solution does not produce the same odor as acetic acid nor butyric acid.

(12) ***Cyanide***—Take 1.0 g of Lactic Acid in a Nessler tube, add 10 mL of water and 1 drop of phenolphthalein TS, add sodium hydroxide (1 in 10) dropwise until the solution exhibits a pale red color while shaking to mix, and add 1.5 mL of sodium hydroxide solution (1 in 10) and water to make 20 mL. Then, heat on a water bath for 10 minutes. After cooling, add dilute acetic acid dropwise until the red color of the solution disappears, then add 1 drop of dilute acetic acid, continuously add 10 mL of pH 6.8 phosphate buffer solution and 0.25 mL of chloramine TS, and stopper immediately. Gently mix, and allow to stand for 5 minutes. To this solution, add 15 mL of pyridine-pyrazolone TS and water to make 50 mL, and allow to stand at 25 ℃ for 30 minutes; the color of the solution is not more intense than the following control solution.

Control solution—Pipet 1.0 mL of cyanide standard solution, and add water to make exactly 20 mL. Transfer 1.0 mL of this solution into a Nessler tube, add 10 mL of water and 1 drop of phenolphthalein TS, and proceed in the same manner.

(13) ***Readily carbonizable substances***—. Superimpose slowly 5 mL of Lactic Acid, previously kept at 15 °C, onto 5 mL of sulfuric acid for Readily carbonizable substances, previously kept at 15 °C, and allow to stand at 15 °C for 15 minutes: no band of dark color develops at the zone of contact.

(14) ***Methyl alcohol***—Take an amount of Lactic Acid equivalent to 4 g of lactic acid, add 8 mL of water and 5 g of calcium carbonate, distill, and take 5 mL of the first distillate. Add water to make 100 mL, and use this solution as the test solution. Take 1 mL of the test solution, add 0.1 mL of phosphoric acid (1 in 20) and 0.2 mL of potassium permanganate solution (1 in 300), allow to stand for 10 minutes, and then add 0.4 mL of anhydrous sodium sulfite solution (1 in 5) and 3 mL of sulfuric acid. Then, add 0.2 mL of chromotropic acid TS; the color of the resulting solution is not more intense than the color of the following solution. : to 1 mL of methanol, add water to make 100 mL, to 1 mL of this solution, add water to make 100 mL and proceed with 1 mL of this solution in the same manner as the test solution

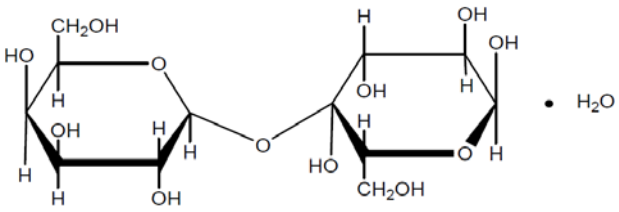
Residue on ignition NMT 0.10% (1 g).

Assay Weigh accurately about 3 g of Lactic Acid in an Erlenmeyer flask, add exactly 40 mL of 1 mol/L sodium hydroxide, cover with a watch glass over the flask, and heat on a water bath for 10 minutes. Immediately titrate excess sodium hydroxide with 0.5 mol/L sulfuric acid VS (indicator: 2 drops of phenolphthalein TS). Perform a blank test in the same manner.

Each mL of 1 mol/L sodium hydroxide VS
= 90.08 mg of $C_3H_6O_3$

Packaging and storage Preserve in tight containers.

Lactose Hydrate
유당수화물



Lactose $C_{12}H_{22}O_{11}\cdot H_2O$: 360.31
(2*R*,3*R*,4*S*,5*R*,6*S*)-2-(Hydroxymethyl)-6-{[(2*R*,3*S*,4*R*,5*R*)-4,5,6-trihydroxy-2-(hydroxymethyl)oxan-3-yl]oxy}oxane-3,4,5-triol monohydrate [*64044-51-5*, Mixture of α- and β-lactose monohydrate]

Lactose Hydrate is a natural disaccharide obtained from milk, consisting of one unit of glucose and one unit of galactose. Lactose Hydrate may contain partially amorphous lactose.

The label indicates the granulated powder form of Lactose Hydrate.

Description Lactose Hydrate occurs as a white crystal, powder or granulated powder and is odorless.
It is freely soluble in water and practically insoluble in ethanol (99.5).

Identification (1) Determine the infrared spectra of Lactose Hydrate and lactose RS as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

Optical rotation $[\alpha]_D^{20}$: Between + 54.4 and + 55.9°.
Weigh accurately about 10 g of Lactose Hydrate, calculated on the anhydrous basis, dissolve in 80 mL of water warmed to 50 °C and allow the mixture to stand and cool. After cooling, add 0.2 mL of ammonia TS, allow the mixture to stand for 30 minutes, and add water to make exactly 100 mL.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Lactose Hydrate in 10 mL of hot water; the solution is clear and colorless or nearly colorless and not more intense than the following control solution. Perform the test with this solution as directed under the Ultraviolet-visible Spectroscopy, using water as the blank; the absorbance at 400 nm is NMT 0.04.

Control solution—To a mixture of 6.0 mL of iron(III) chloride hexahydrate colorimetric stock solution, 2.5 mL of cobalt(II) chloride hexahydrate colorimetric stock solution and 1.0 mL of copper(II) sulfate pentahydrate colorimetric stock solution, add hydrochloric acid solution (1 in 100) to make 10 mL. Pipet 1 mL of this solution and add hydrochloric acid solution (1 in 100) to make 100 mL before use.

(2) ***Acidity or alkalinity***—Dissolve 6 g of Lactose Hydrate in 25 mL of freshly boiled and cooled water by heating, cool, and add 0.3 mL of phenolphthalein TS; the resulting solution is colorless. To this solution, add 0.1 mol/L sodium hydroxide TS until the color of the solution changes from colorless to red; NMT 0.4

mL of 0.1 mol/L sodium hydroxide TS is consumed.

(3) ***Proteins and light-absorbing impurities***—Weigh 1.0 g of Lactose Hydrate, add water to make 100 mL, and use this solution as the test solution. Perform the test with the test solution as directed under the Ultraviolet-visible Spectroscopy, using water as the blank; the absorbance between 210 and 220 nm is NMT 0.25, the absorbance between 270 and 300 nm is NMT 0.07.

Loss on drying NMT 0.5% (1 g, 80 °C, 2 hours, NMT 1.0% for the granulated powder).

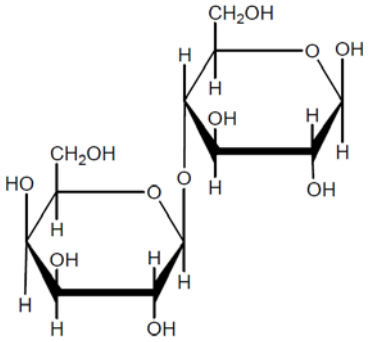
Residue on ignition NMT 0.10% (1.0 g).

Water Between 4.5% and 5.5% (1 g, volumetric titration, direct titration, use a mixture of methanol for Karl Fischer titration and formamide for Karl Fischer titration (2 : 1) instead of methanol for Karl Fischer titration).

Microbial limit The total aerobic microbial count is NMT 10^2 CFU/g and the total combined yeasts/molds count is NMT 5×10^1 CFU/g. *Salmonella and Escherichia coli* are not detected.

Packaging and storage Preserve in tight containers.

Anhydrous Lactose
무수유당



α-lactose: R^1 = H, R^2 = OH
β-lactose: R^1 = OH, R^2 = H

$C_{12}H_{22}O_{11}$: 342.30
(2*R*,3*R*,4*S*,5*R*,6*S*)-2-(Hydroxymethyl)-6-{[(2*R*,3*S*,4*R*,5*R*)-4,5,6-trihydroxy-2-(hydroxymethyl)oxan-3-yl]oxy}oxane-3,4,5-triol
[*63-42-3*, Anhydrous Lactose]

Anhydrous Lactose is β-lactose or a mixture of β-lactose and α-lactose.

The labeling of Anhydrous Lactose indicates the percentage content of α-lactose and β-lactose determined according to the measured values of the percentage content of α- and β-isomers.

If the labeling includes particle size distribution, indicate the particle sizes corresponding to 10%, 50% and 90% of the cumulative particle size distribution.

Description Anhydrous Lactose occurs as a white or almost white crystal or powder.
It is freely soluble in water and practically insoluble in ethanol (95).

Identification Determine the infrared spectra of Anhydrous Lactose and anhydrous lactose RS, previously dried, as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

Optical rotation $[\alpha]_D^{20}$: Between + 54.4° and + 55.9°
Weigh accurately an amount corresponding to about 10 g of Anhydrous Lactose, calculated on the anhydrous basis, dissolve in 80 mL of water warmed to 50 °C and allow the mixture to stand and cool. After cooling, add 0.2 mL of ammonia TS and allow the mixture to stand for 30 minutes. Add water to make exactly 100 mL and proceed with this solution to measure the optical rotation in a cell, 100-mm in path length.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Anhydrous Lactose in 10 mL of hot water; the solution is clear and colorless or nearly colorless and not more intense than the following control solution. Perform the test with this solution as directed under the Ultraviolet-visible Spectroscopy, using water as the blank; the absorbance at 400 nm is NMT 0.04.

Control solution—To a mixture of 6.0 mL of the colorimetric stock solution of iron(III) chloride hexahydrate, 2.5 mL of the colorimetric stock solution of cobalt(II) chloride hexahydrate, and 1.0 mL of the colorimetric stock solution of copper (II) sulfate pentahydrate, add hydrochloric acid solution (1 in 100) to make 10 mL. Pipet 1 mL of this solution and add hydrochloric acid solution (1 in 100) to make 100 mL before use.

(2) ***Acidity or alkalinity***—Dissolve 6 g of Anhydrous Lactose in 25 mL of freshly boiled and cooled water by heating, cool, and add 2 drops of phenolphthalein TS; the resulting solution is colorless. To this solution, add 0.1 mol/L sodium hydroxide TS until the color of the solution changes from colorless to red; NMT 0.4 mL of 0.1 mol/L sodium hydroxide TS is consumed.

(3) ***Proteins and light-absorbing impurities***—Weigh 1.0 g of Anhydrous Lactose, add water to make 100 mL, and use this solution as the test solution. Perform the test with the test solution as directed under the Ultraviolet-visible Spectroscopy, using water as the blank; the absorbance between 210 and 220 nm is NMT 0.25, and the absorbance between 270 and 300 nm is NMT 0.07.

Isomer content Place 10 mg of Anhydrous Lactose in a stoppered reaction vial for gas chromatography, add 4 mL of a mixture of pyridine, trimethylsilylimidazole, and dimethylsulfoxide (117 : 44 : 39), stopper, sonicate for 20 minutes and cool. Transfer 400 μL of this solution into a vial, add 1 mL of pyridine, stopper tightly, shake to mix, and use this solution as the test solution. Perform the test with 0.5 μL of the test solution as directed under the Gas Chromatography according to the following conditions. Determine the peak areas A_a and A_b, of α-lactose and β-lactose, respectively, in the test solution and calculate the content (%) of α-lactose and the content (%) of β-lactose in Anhydrous Lactose by the following equations.

$$\text{Percentage content (\%) of α-lactose} = \{A_a/(A_a + A_b)\} \times 100$$

$$\text{Percentage content (\%) of β-lactose} = \{A_b/(A_a + A_b)\} \times 100$$

Operating conditions
Detector: A flame ionization detector

Column: A column about 0.25 mm in internal diameter and about 15 m in length, coated the inside with 5% diphenyl-95% dimethylpolysiloxane for gas chromatography, 0.25 μm in thickness. Use a fused silica column about 0.53 mm in internal diameter and 2 m in length, packed with deactivated intermediate polarity as a guard column.

Column temperature: Maintain the temperature at 40 °C for 1 minute, raise the temperature to 150 °C at a rate of 35 °C per minute, raise the temperature further to 300 °C at a rate of 12 °C per minute, and then maintain the temperature for 2 minutes.

Sample injection port: A constant temperature of about 275 °C or cold column direct injection method

Carrier gas: Helium

Flow rate: 2.8 mL/min (adjust the flow rate so that the retention time of β-lactose is about 12 minutes).

Split ratio: No split

System suitability

System performance: Prepare a solution with 10 mg of the mixture of α-lactose and β-lactose (1 : 1) in the same manner as the test solution. Proceed with 0.5 μL of this solution according to the above operating conditions; the relative retention time of α-lactose to β-lactose is about 0.9 with the resolution between the peaks being NLT 3.0.

Loss on drying NMT 0.5% (1 g, 80 ℃, 2 hours).

Water NMT 1.0% (1 g, volumetric titration, direct titration, use a mixture of methanol for Karl Fischer titration and formamide for Karl Fischer titration (2 : 1) instead of methanol for Karl Fischer titration).

Residue on ignition NMT 0.1% (1 g).

Microbial limit The total aerobic microbial count is NMT 10^2 CFU/g and the total combined yeasts/molds count is NMT 5×10^1 CFU/g. *Salmonella* species and *Escherichia coli* are not observed.

Packaging and storage Preserve in well-closed containers.

Hydrous Lanolin
가수라놀린

Lanolin

Hydrous Lanolin is Purified Lanolin with water added, and contains NLT 70.0 and NMT 75.0% of Purified Lanolin (as the evaporation residue).

Description Hydrous Lanolin is a pale yellow ointment-like material. It has a non-rancid and slightly characteristic odor.
It is soluble in ether or cyclohexane, with separation of water.
Heat Hydrous Lanolin on a water bath to dissolve; it separates into a clear oil layer and a water layer.

Melting point About 39 ℃

Identification Dissolve 1 g of Hydrous Lanolin in 50 mL of cyclohexane and discard water. Proceed with 1 mL of the cyclohexane solution according to the Identification under Purified Lanolin.

Acid Value NMT 1.0.

Iodine value Between 18 and 36. Heat Hydrous Lanolin on a water bath to evaporate almost all of the moisture, then weigh accurately about 0.8 g in a 500-mL stoppered flask, and perform the test as directed in the Iodine value under Purified Lanolin.

Purity (1) ***Acidity or alkalinity, chloride, ammonia, and water-soluble organics***—Perform the test as directed under the Purity (1), (2), (3), and (4) of Purified Lanolin.

(2) ***Vaseline***—Perform the test with dried evaporation residue as directed in the Purity (5) under Purified Lanolin.

(3) ***Butylated hydroxytoluene***—Perform the test with dried evaporation residue as directed in the Purity (6) under Purified Lanolin (NMT 200 ppm).

Residue on evaporation Weigh accurately about 12.5 g of Hydrous Lanolin, dissolve in 50 mL of ether, and transfer into a separatory funnel. Transfer the separated water layer into another separatory funnel, add 10 mL of ether, shake to mix, and combine the ether layer with the previous separatory funnel. To the ether layer, add 3 g of anhydrous sodium sulfate, shake to mix, then filter through a dried filter paper, and wash the separatory funnel and the filter paper twice each with 20 mL of ether. Combine the washings and the filtrate, evaporate on a water bath until the odor of ether is no longer perceptible, then dry the residue in a desiccator (in vacuum, silica gel) for 24 hours, and weigh the mass.

Packaging and storage Preserve in tight containers and store at below 30 ℃.

Purified Lanolin
정제라놀린

Purified Lanolin is a purified substance similar to fat obtained from the wool of *Ovis aries* Linné (Bovidae).

Description Purified Lanolin is a pale yellow to yellowish brown, viscous, and ointment-like material. It has a non-rancid and slightly characteristic odor.
It is very soluble in ether or cyclohexane, freely soluble in tetrahydrofuran and toluene, and very slightly soluble in ethanol.
It is practically insoluble in water, and miscible without separation with about double the volume of water. It has an ointment-like viscosity.
Melting point: Between 45 ℃ and 43 ℃.

Identification Carefully superimpose 1 mL of a solution of Purified Lanolin in cyclohexane (1 in 50) on 2 mL of sulfuric acid; a reddish brown color develops at the zone of contact, and the sulfuric acid layer exhibits a green fluorescence.

Acid Value NMT 1.0.

Iodine Value Between 18 and 36. Weigh accurately about 0.8 g of Purified Lanolin in a 500-mL stoppered flask, dissolve in 10 mL of cyclohexane, add exactly 25 mL of hanus TS, and shake well to mix. When the solution does not become clear, add cyclohexane again to clarify, then stopper the flask, and allow to stand at 20 ℃ to 30 ℃ for 1 hour while occasionally shaking to mix, protected from light. Then, add 20 mL of potassium iodide solution (1 in 10) and 100 mL of water, and shake to mix. Then, titrate the liberated iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 1mL of starch TS). Perform a blank test in the same

manner.

$$\text{Iodine Value} = \frac{(a-b)\times 1.269}{Amount\ (g)\ of\ sample}$$

a: Volume of 0.1 mol/ L sodium thiosulfate VS consumed in the blank test (mL)

b: Volume of 0.1 mol/L sodium thiosulfate VS consumed in the titration of the main test (mL)

Purity (1) ***Acidity or alkalinity***—Weigh 5 g of Purified Lanolin, add 25 mL of water, boil for 10 minutes, and cool. Then, add water to restore the initial mass, and take the separated water layer; the water layer is neutral.

(2) ***Chloride***—Weigh 2.0 g of Purified Lanolin, add 40 mL of water, boil for 10 minutes, and cool. Add water to restore the initial mass, and filter. To 20 mL of the filtrate, add 6 mL of dilute nitric acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution with 1.0 mL of 0.01 mol/L hydrochloric acid (NMT 0.036%).

(3) ***Ammonium***—To 10 mL of the water layer from (1), add 1 mL of sodium hydroxide TS, and boil; the gas evolved does not change moistened red litmus paper to blue.

(4) ***Water-soluble organic substances***—To 5 mL of the water layer from (1), add 0.25 mL of 0.002 mol/L potassium permanganate, and allow to stand for 5 minutes; the red color of the solution does not disappear.

(5) ***Vaseline—***Weigh 1.0 g of Purified Lanolin, add exactly 10 mL of a mixture of tetrahydrofuran and isooctane (1 : 1), and use this solution as the test solution. With this solution, perform the test as directed under the Thin Layer Chromatography. Dissolve 20 mg of petrolatum in 10 mL of a mixture of tetrahydrofuran and isooctane (1 : 1), and use this solution as the standard solution. Spot 25 μL each of the test solution and the standard solution on the thin-layer chromatographic plate made of silica gel for thin-layer chromatography. Next, develop the plate with isooctane as the developing solvent to a distance of about 10 cm, and air-dry the plate. Spray evenly diluted sulfuric acid (1 in 2) on the plate, and heat at 80 ℃ for 5 minutes. After cooling, examine the plate under ultraviolet light (main wavelength: 365 nm); there are no spots with the same fluorescence as petrolatum at the same position as the spots of petrolatum. However, use the plate, previously developed using isooctane, air-dried, and then heated at 110 ℃ for 60 minutes, to perform this test.

(6) ***Butylated hydroxytoluene***—Weigh accurately 1.0 g of Purified Lanolin, dissolve in carbon disulfide, add exactly 1.0 mL of internal standard solution, and add more carbon disulfide to make exactly 10 mL. Use this solution as the test solution. Separately, weigh accurately about 0.2 g of butylated hydroxytoluene RS, and dissolve in carbon disulfide to make exactly 100 mL. Pipet 1.0 mL of this solution and dissolve in carbon disulfide to make exactly 10 mL. Add exactly 1.0 mL of this solution and 1.0 mL of the internal standard solution, respectively, dissolve in carbon disulfide to make exactly 10 mL, and use this solution as the standard solution. Perform the test with 1 μL each of the test solution and the standard solution as directed under the Gas Chromatography according to the following conditions, and calculate the peak area ratios, Q_T and Q_S, of the peak area of butylated hydroxytoluene to the peak area of the internal standard in the test solution and the standard solution, respectively (NMT 200 ppm).

Operating conditions

Detector: A flame ionization detector

Column: A column 4 mm in internal diameter and 1.5 m in length, packed with silylated diatomaceous earth for gas chromatography, coated with a 10% mass ratio of the poly (dimethyl) siloxane for gas chromatography.

Column temperature: A constant temperature of about 150 ℃.

Sample injection port temperature: 180 ℃

Detector temperature: 300 ℃

Carrier gas: Nitrogen

Flow rate: 40 mL/min

Internal standard solution—Dissolve 0.2 g of methyl decanoate in carbon disulfide to make exactly 100 mL. Pipet 1.0 mL of this solution, dissolve in carbon disulfide to make exactly 10 mL, and use this solution as the internal standard solution.

Loss on drying NMT 0.5% (1 g, 105 ℃, 2 hours).

Ash NMT 0.1% (according to Ash under the Crude Drugs Test).

Packaging and storage Preserve in tight containers and store at below 30 ℃.

Lard
돈지

Lard is a fat of *Sus scrofa* Linné *var. domesticus* Gray (*Suidae*).

Description Lard occurs as a white, soft, and smooth mass. It has a slightly characteristic odor and a bland taste.
It is freely soluble in ether or petroleum ether, very slightly soluble in ethanol, and practically insoluble in water.

Melting point Between 36 ℃ and 42 ℃ (Method 2).

Congealing point of fatty acid Between 36 ℃ and 42 ℃.

Saponification value Between 195 and 203.

Acid Value NMT 2.0.

Iodine value Between 46 and 70.

Purity (1) ***Water and coloration***—Heat 5 g of Lard on a water bath to dissolve; it forms a clear liquid, and no water is separated. Observe this liquid in a layer, 10 mm in thickness: the liquid is colorless to slightly yellow.

(2) ***Alkali***—To 2.0 g of Lard, add 10 mL of water, warm on a water bath to dissolve, and shake vigorously to mix. After cooling, add 1 drop of phenolphthalein TS to the separated water; the water layer is colorless.

(3) ***Chloride***—To 1.5 g of Lard, add 30 mL of ethanol, and boil for 10 minutes under a reflux condenser. After cooling, add 5 drops of a solution of silver nitrate in ethanol (1 in 50) to 20 mL of the filtrate; the turbidity of the resulting solution is not more intense than that of the following control solution.

Control solution—To 1.0 mL of 0.01 mol/L hydrochloric acid, add ethanol to make 20 mL, and 5 drops of a solution of silver nitrate in ethanol (1 in 50).

(4) ***Beef tallow***—Dissolve 5 g of Lard in 20 mL of ether,

seal lightly with absorbent cotton, allow to stand at 20 ℃ for 18 hours, and collect the separated crystal. Immerse in ethanol, and examine under a microscope at 200x magnification; the crystals are in the form of rhomboidal plates grouped irregularly and do not contain prisms or needles grouped in fan-shaped clusters.

Packaging and storage Preserve in tight containers below 30 ℃.

Lauromacrogol
라우로마크로골

Polyoxyethylene Lauryl Alcohol Ether

Lauromacrogol is a polyoxyethylene ether prepared by additional polymerization of ethylene oxide with lauryl alcohol.

Description Lauromacrogol occurs as a colorless to pale yellow and clear liquid, white petrolatum-like, or waxy solid. It has a characteristic odor and has a slightly irritating taste.
It is very soluble in ethanol, ether, or carbon tetrachloride.
It is freely soluble in water or forms fine oil droplets.

Identification (1) To 0.5 g of Lauromacrogol, add 10 mL of water and 5 mL of ammonium thiocyanate-cobalt nitrate TS, shake well to mix, and then add 5 mL of chloroform. Shake to mix, and allow to stand; the chloroform layer exhibits a blue color.

(2) Determine the infrared absorption spectrum of a solution of 0.35 g of Lauromacrogol dissolved in 10 mL of methanol as directed in the Solution method under the Mid-infrared Spectroscopy using a 0.1 mm fixed cell; it exhibits absorption at the wave numbers of about 1347 cm^{-1}, 1246 cm^{-1}, and 1110 cm^{-1}.

Purity (1) ***Acid***—Weigh 10.0 g of Lauromacrogol, transfer into a flask, add 50 mL of neutralized ethanol, heat on a water bath to almost boiling while shaking 1 to 2 times. After cooling, add 5.3 mL of 0.1 mol/L sodium hydroxide TS and 5 drops of phenolphthalein TS; the resulting solution exhibits a red color.

(2) ***Unsaturated compounds***—Weigh 0.5 g of Lauromacrogol, add 10 mL of water, shake to mix, and add 5 drops of bromine TS; the color of the test solution does not disappear.

(3) ***Ethylene oxide and dioxane***—Weigh accurately 1.00 g (M_T) of Lauromacrogol, transfer into a 10-mL vial, add 1.0 mL of water, and mix evenly. Then, allow to stand at 70 ℃ for 45 minutes, and use this solution as the test solution. (3) Weigh accurately 1.00 g of dioxane RS, add water to make 100 mL, then pipet 5.0 mL of this solution, and add water to make 100 mL. Pipet 10.0 mL of this solution, add water to make 50 mL, and use this solution as the standard dioxane solution. Dilute 0.5 mL of 50 mg/mL ethylene oxide solution, previously prepared by dissolving ethylene oxide in methylene chloride, with water to make 50.0 mL (this solution is stable when sealed with a Teflon-coated silicone membrane and a crimp stopper at -20 ℃ for up to 3 months) of a solution containing 50 μg of ethylene oxide per mL. Transfer 10.0 mL of this solution into a flask containing 30 mL of water, shake well, and add water to make 50 mL. Pipet 10.0 mL of this solution, add water to make 50 mL, and use this solution as the standard ethylene oxide solution. Prepare this solution just before use. Separately, transfer 1.00 g (M_R) of Lauromacrogol into a 10-mL vial, add 0.5 mL of standard ethylene oxide solution and 0.5 mL of standard dioxane solution, mix evenly, and allow to stand at 70 ℃ for 45 minutes. Use this solution as the standard solution (1). Transfer 0.5 mL of ethylene oxide RS into a 10-mL vial, add 0.1 mL of 10 mg/L acetaldehyde standard solution and 0.1 mL of dioxane RS, prepared before use, allow to stand at 70 ℃ for 45 minutes, and use this solution as the standard solution (2). Perform the test with 1 mL each of the test solution and the standard solution (1) according to the following conditions, as directed under the Gas Chromatography. Determine the peak areas of ethylene oxide and dioxane in each solution, and calculate the amount; the amount of ethylene oxide is NMT 1 ppm, and the amount of dioxane is NMT 10 ppm.

$$\text{Amount (ppm) of ethylene oxide} = \frac{A_T \times C}{(A_R \times M_T) - (A_T \times M_R)}$$

A_T = Peak area of ethylene oxide in the test solution
A_S = Peak area of ethylene oxide in the standard solution (1)
M_T: Amount (g) of sample in the test solution
M_R: Amount (g) of sample in the standard solution (1)
C = Amount (μg) of ethylene oxide added in the standard solution (1)

$$\text{Amount (ppm) of dioxane} = \frac{D_T \times C}{(D_R \times M_T) - (D_T \times M_R)}$$

D_T = Peak area of dioxane in the test solution
D_R: Peak area of dioxane in the standard solution (1)
C = Amount (μg) of dioxane added in the standard solution (1)

Operating conditions

Detector: A flame ionization detector

Column: A capillary glass or quartz column 0.53 mm in inside diameter and about 30 m in length, coated with a 1.0 μm thickness of poly(dimethyl)siloxane on the inside.

Column temperature: Initially maintain at 50 ℃ for 5 minutes, increase the temperature by 5 ℃ per minute to reach 180 ℃, and finally increase by 30 ℃ per minute to 230 ℃ and maintain for 5 minutes.

Sample injection port temperature: A constant temperature of about 150 ℃

Headspace sampler temperature: 70 ℃

Detector temperature: A constant temperature of about 250 ℃

Carrier gas: Helium

Split ratio: About 1 : 20

System suitability

System performance: Proceed with 1.0 mL of the standard solution (2) according to the above conditions; the resolution between acetaldehyde and ethylene oxide is NLT 2.0 with the signal-to-noise ratio of the dioxane being NLT 10.

Residue on ignition NMT 0.2% (1 g).

Packaging and storage Preserve in tight containers.

Magnesium Stearate
스테아르산마그네슘

Magnesium Stearate is a magnesium salt of solid mixed fatty acids of plant or animal origin, which is a mixture of

magnesium stearate and magnesium palmitate.

When the specific surface area ofMagnesium Stearate is indicated, the specific surface area measurement is indicated.

The label indicates that it is derived from edible fatty acids.

Magnesium Stearate contains NLT 4.0% and NMT 5.0% of magnesium (Mg: 24.31), calculated on the dried basis.

Description Magnesium Stearate occurs as a white, light, fine powder with a slippery feel. It is sticky to the skin, and has no odor or a faint, characteristic odor.
It is practically insoluble in water or ethanol (99.5).

Identification (1) Take 5.0 g of Magnesium Stearate in a round-bottomed flask, add 50 mL of peroxide-free ether, 20 mL of dilute nitric acid and 20 mL of water, and heat the mixture under a reflux condenser until completely dissolved. After cooling, transfer the contents of the flask to a separatory funnel, shake to mix, allow the layers to separate, and transfer the aqueous layer to a flask. Extract the ether layer twice with 4 mL each of water, and add the extract to the aqueous layer. Wash this extract with 15 mL of peroxide-free ether, transfer to a 50-mL volumetric flask, add water to make exactly 50 mL, shake to mix, and use this solution as the test solution. Add 1 mL of ammonia TS to 1 mL of the test solution; a white precipitate are produced. Add 1 mL of ammonium chloride TS; the precipitate dissolves. Add dibasic sodium phosphate solution (4 in 25) again; a white crystalline precipitate is produced.

(2) Perform the test with Magnesium Stearate as directed in the Purity (7) under Stearic Acid and Palmitic Acid, the retention time of the stearic acid peak and palmitic acid peak of the test solution and the retention time of the major peak of the system suitability solution are the same.

Purity (1) ***Acidity or Alkalinity***—Add 20 mL of freshly boiled and cooled water to 1.0 g of Magnesium Stearate, shake to mix, heat on a water bath for 1 minute, cool, and filter. Add 0.05 mL of bromothymol blue TS to 10 mL of this filtrate. Add exactly 0.05 mL of 0.1 mol/L hydrochloric acid or 0.1 mol/L sodium hydroxide solution to this solution; the color of the solution changes.

(2) ***Chloride***—Perform the test with 10.0 mL of the test solution obtained in the Identification. Prepare the control solution with 1.4 mL of 0.02 mol/L hydrochloric acid (NMT 0.10%).

(3) ***Sulfate***—Perform the test with 6.0 mL of the test solution obtained in the Identification. Prepare the control solution with 3.0 mL of 0.02 mol/L sulfuric acid (NMT 1.0%).

(4) ***Cadmium***—Weigh accurately about 0.1 g of Magnesium Stearate, put in a suitable polytetrafluoroethylene-treated acid decomposition device, and add 2.5 mL of nitric acid. Close the acid decomposition device, heat to 170°C for 3 hours, allow to stand, cool slowly to room temperature. Open the acid decomposition device within the fume hood while being careful to discharge corrosive gases, transfer the residue into a volumetric flask, and add water to make 10 mL. Pipet 1 mL of this solution, and add dilute nitric acid (1 in 4) to make exactly 10 mL. Add exactly 0, 0.5, and 1.0 mL of the standard solution, respectively, to 1 mL of this solution, and add exactly 1.0, 0.5, and 0 mL of dilute nitric acid (1 in 4), respectively. Add 50 μL of the matrix conditioning solution to each of the mixed solutions and perform the test with these solutions as directed under the Atomic absorption spectroscopy according to the following conditions using nitric acid (1 in 4) diluted with the prepared solutions as the blank. Determine the absorbances A_1, A_2, and A_3. These solutions contain cadmium of the standard solution at concentrations of 0, 0.00075, and 0.0015 μg/mL. With absorbance A_1, A_2, and A_3 on the vertical axis and 0, 0.00075, 0.0015 μg/mL on the horizontal axis, draw a calibration curve (correlation coefficient of NMT 0.99) with the straight line that best fits the three points using the least squares method and extrapolate to determine the concentration C that intersects the horizontal axis. Calculate the amount of cadmium in Magnesium Stearate (NMT 3 ppm).

Matrix conditioning solution—Dissolve 20 g of ammonium dihydrogen phosphate and 1 g of magnesium nitrate in 100 mL of water.

Standard solution—Pipet 3 mL of the standard cadmium solution and add diluted nitric acid (1 in 4) to make 1000 mL (1 mL of this solution contains 0.0030 μg of cadmium).

$$\text{Amount (ppm) of cadmium} = (C / W) \times 200$$

W: Amount (g) of Magnesium Stearate taken

Operating conditions

Detector: Suitable GFAA spectrophotometer with pyrolysis tube mounted on platform

Lamp: A cadmium hollow-cathode lamp

Wavelength: 228.8 nm

	Dry stage	Ash stage	Atomized stage
Temperature (°C)	110	600	1800
Lamp time (seconds)	10	10	0
Retention time (seconds)	20	30	5

(5) ***Lead***—Weigh accurately about 0.1 g of Magnesium Stearate, put it in a suitable polytetrafluoroethylene-treated acid decomposition device, and add 2.5 mL of nitric acid. Close the acid decomposition device, heat to 170°C for 3 hours, allow to stand, cool slowly to room temperature. Open the acid decomposition device within the fume hood while being careful to discharge corrosive gases, transfer the residue into a volumetric flask, and add water to make 10 mL. Pipet 1 mL of this solution, and add dilute nitric acid (1 in 4) to make exactly 10 mL. Add exactly 0, 0.5, and 1.0 mL of the standard solution, respectively, to 1 mL of this solution, and add exactly 1.0, 0.5, and 0 mL of dilute nitric acid (1 in 4), respectively. Add 50 μL of the matrix conditioning solution to each of the mixed solutions and perform the test with these solutions as directed under the Atomic absorption spectroscopy according to the following conditions using nitric acid (1 in 4) diluted with the prepared solutions as the blank. Determine the absorbances A_1, A_2, A_3. These solutions contain cadmium of the standard solution at concentrations of 0, 0.025, and 0.05 μg/mL. With absorbance A_1, A_2, and A_3 on the vertical axis and 0, 0.025, 0.05 μg/mL on the horizontal axis, draw a calibration curve (correlation coefficient of NMT 0.99) with the straight line that best fits the three points using the least squares method and extrapolate to determine the concentration C that intersects the horizontal axis. Calculate the amount of cadmium in Magnesium Stearate (NMT 10 ppm).

Matrix conditioning solution—Dissolve 20 g of ammonium dihydrogen phosphate and 1 g of magnesium nitrate in 100 mL of water.

Standard solution—Pipet 1 mL of the lead standard solution and add diluted nitric acid (1 in 4) to make 100 mL (1 mL of

this solution contains 0.100 μg of cadmium).

$$\text{Amount (ppm) of lead} = (C / W) \times 20$$

W: Amount (g) of Magnesium Stearate taken

Operating conditions

Detector: Suitable GFAA spectrophotometer with pyrolysis tube mounted on platform

Lamp: A lead hollow-cathode lamp

Wavelength: 283.3 nm

	Dry stage	Ash stage	Atomized stage
Temperature (℃)	110	450	2000
Lamp time (seconds)	10	10	0
Retention time (seconds)	20	30	5

(6) ***Nickel*—**Weigh accurately about 0.1 g of Magnesium Stearate, put it in a suitable polytetrafluoroethylene-treated acid decomposition device, and add 2.5 mL of nitric acid. Close the acid decomposition device, heat to 170°C for 3 hours, allow it to stand, cool slowly to room temperature. Open the acid decomposition device within the fume hood while being careful to discharge corrosive gases, transfer the residue into a volumetric flask, and add water to make 10 mL. Pipet 1 mL of this solution, and add dilute nitric acid (1 in 4) to make exactly 10 mL. Add exactly 0, 0.5, and 1.0 mL of the standard solution, respectively, to 1 mL of this solution, and add exactly 1.0, 0.5, and 0 mL of dilute nitric acid (1 in 4), respectively. Add 50 μL of the matrix conditioning solution to each of the mixed solutions and perform the test with these solutions as directed under the Atomic absorption spectroscopy according to the following conditions using nitric acid (1 in 4) diluted with the prepared solutions as the blank. Determine the absorbances A_1, A_2, A_3. These solutions contain nickel of the standard solution at concentrations of 0, 0.0125, 0.025 μg/mL. With absorbance A_1, A_2, and A_3 on the vertical axis and 0, 0.0125, and 0.025 μg/mL on the horizontal axis, draw a calibration curve (correlation coefficient of NMT 0.99) with the straight line that best fits the three points using the least squares method and extrapolate to determine the concentration *C* that intersects the horizontal axis. Calculate the amount of cadmium in Magnesium Stearate (NMT 5 ppm).

Matrix conditioning solution—Dissolve 20 g of ammonium dihydrogen phosphate and 1 g of magnesium nitrate in 100 mL of water.

Standard solution—Pipet 1 mL of the nickel standard solution and add diluted nitric acid (1 in 4) to make 100 mL (1 mL of this solution contains 0.050 μg of cadmium).

$$\text{Amount (ppm) of nickel} = (C / W) \times 20$$

W: Amount (g) of Magnesium Stearate taken

Operating conditions

Detector: Suitable GFAA spectrophotometer with pyrolysis tube mounted on platform

Lamp: Nickel hollow cathode lamp

Wavelength: 232.0 nm

	Dry stage	Ash stage	Atomized stage
Temperature (℃)	110	450	2000
Lamp time (seconds)	10	10	0
Retention time (seconds)	20	30	5

(7) ***Stearic acid and palmtic acid*—**Take exactly 0.1 g of Magnesium Stearate and put it in a small Erlenmeyer flask equipped with a reflux condenser. Add 5 mL of boron trifluoride-methanol TS, shake to mix, and heat the mixture for about 10 minutes until dissolved. Add 4.0 mL of heptane through the cooler and heat for about 10 minutes. After cooling, add 20 mL of saturated sodium chloride solution, shake to mix, allow it to stand, and separate it into two layers. Take 2 mL of the separated heptane layer, pass about 0.1 g of anhydrous sodium sulfate previously washed with heptane, and put it in a separate flask. Put 1.0 mL of this solution into a 10-mL volumetric flask, add heptane to make exactly 10 mL, shake to mix, and use this solution as the test solution. Perform the test with 1 μL of the test solution as directed under the Liquid Chromatography according to the following conditions. Determine the peak area A of methyl stearate in the test solution and the peak area B (area of all detected peaks) of all obtained fatty acid esters, and calculate the stearic acid content (%) in the fatty acid fraction of Magnesium Stearate according to the following formula.

$$\text{Stearic acid content (\%)} = (A / B) \times 100$$

Calculate the content (%) of palmitic acid contained in Magnesium Stearate in the same manner. The peak area of methyl stearate is NLT 40.0% of the sum of the peak areas of all fatty acid esters obtained from the chromatogram, and the sum of the peak areas of methyl stearate and methyl palmitate is NLT 90.0% of the sum of the peak areas of all fatty acid esters obtained from the chromatogram.

Operating conditions

Detector: A flame ionization detector

Column: A capillary column about 0.32 mm in internal diameter and about 30 m in length, coated with polyethylene glycol 20000 for gas chromatography in 0.5 μm thickness.

Column temperature: After injecting the sample, maintain at 70 °C for about 2 minutes, and increase to 240 °C at a rate of 5 °C per minute, and maintain this temperature for 5 minutes.

Sample injection port temperature: A constant temperature of about 220 ℃.

Detector temperature: A constant temperature of about 260 ℃.

Carrier gas: Helium

Flow rate: 2.4 mL/min

System suitability

Test for required detectability: Weigh accurately about 50 mg each of Stearic Acid RS and Palmitic Acid RS, each previously dried in a desiccator (silica gel) for 4 hours, and place in a small Erlenmeyer flask fitted with a reflux condenser. Add 5.0 mL of boron trifluoride-methanol TS and shake to mix. Proceed in the test manner as directed in the preparation of the test solution, and use the solution as the system suitability solution. Pipet 1.0 mL of the system suitability solution and dilute to 10.0 mL with heptane. Confirm that the peak area of methyl stearate obtained from 1 μL of this solution is NLT 5% and NMT 15% of the peak area of methyl stearate obtained from the system

suitability solution.

System performance: Proceed with 1 μL of the system suitability solution according to the above conditions; methyl palmitate and methyl stearate are eluted in this order, the relative retention time ratio of methyl palmitate to methyl stearate is about 0.9 with the resolution being NLT less 5.0.

System repeatability: Repeat the test 6 times with 1 μL of the system suitability solution according to the above conditions; the relative standard deviation of the peak areas of methyl palmitate and methyl stearate is NMT 3.0% and the relative standard deviation of the peak area ratio of methyl palmitate to the peak area of methyl stearate is NMT 1.0%.

Loss on drying NMT 6.0% (2 g, 105 ℃, constant mass).

Microbial limit Perform the test; the total aerobic microbial count per 1 g of Magnesium Stearate is NMT 103 CFU, and the total combined yeasts/mold count is NMT 5×10^2 CFU. Additionally, salmonella and E. coli are not detected.

Assay Weigh accurately about 0.5 g of Magnesium Stearate, put it in a 250-mL flask, and add 50 mL of a mixture of anhydrous ethanol and n-butanol (1 : 1), 5 mL of strong ammonia water, and 3 mL of ammonium chloride buffer solution (pH 10). Add 30.0 mL of 0.1 mol/L ethylenediaminetetraacetate acid disodium salt solution and 1 to 2 drops of Eriochrome Black T TS and shake to mix. Heat this solution to 45 to 50 ℃ until it becomes clear, cool it, and titrate the excess ethylenediaminetetraacetate acid disodium salt solution with 0.1 mol/L zinc sulfate VS until the color of the solution changes from blue to purple. Perform a blank test in the same manner and make any necessary correction.

Each mL of 0.1 mol/L ethylenediaminetetraacetate acid disodium salt solution
= 2.431 mg of Mg

Packaging and storage Preserve in tight containers.

Medicinal Soap
약용비누

Medicinal Soap is the sodium salts of fatty acids.

Description Medicinal Soap occurs as a white to pale yellow powder or grains and has a non-rancid, characteristic odor.
It is sparingly soluble in water and slightly soluble in ethanol.
A solution of Medicinal Soap in water (1 in 100) is alkaline.

Fatty acid Dissolve 25 g of Medicinal Soap in 300 mL of hot water, slowly add 60 mL of dilute sulfuric acid, and heat the mixture on a water bath for 20 minutes. After cooling, filter the precipitate and wash with warm water until the washings no longer show acidity to methyl orange TS. Transfer the precipitate to a small beaker and heat on a water bath to complete separation of water, resulting in transparent fatty acids. Filter the fatty acid into a small beaker while warm, dry at 100 °C for 20 minutes, and perform the test with this material as directed under the Fats and Fatty Oils; the congealing point of the fatty acid is 18 to 28 ℃, the acid value is 185 to 205, and the iodine value is 82 to 92.

Purity (1) ***Acidity or Alkalinity—***To 5.0 g of Medicinal Soap, add 85 mL of neutralized ethanol, heat the mixture on a water bath to dissolve, and filter through a cotton wool while hot. Wash the container and the residue 3 times with 5 mL of hot neutralized ethanol each time, combine the filtrate with the washings, and add hot neutralized ethanol to make 100 mL. Use this solution as the test solution and immediately perform the test as follows at 70 ℃.

(i) To 40 mL of the test solution, add 3 drops of phenolphthalein TS and 0.20 mL of 0.1 mol/L sodium hydroxide solution; the solution exhibits a red color.

(ii) To 40 mL of the test solution, add 3 drops of phenolphthalein TS and 0.20 mL of 0.05 mol/L sulfuric acid; the solution does not exhibit a red color.

(2) ***Heavy metals—***Weigh 1.0 g of Medicinal Soap and perform the test according to Method 2. Prepare the control solution with 2.0 mL of the lead standard solution (NMT 20 ppm).

(3) ***Ethanol-insoluble substances***—Weigh accurately about 2 g of Medicinal Soap, dissolve by warming in 100 mL of neutralized ethanol and filter the solution through a glass filter. Wash the residue with 100 mL of hot neutralized ethanol and dry at 105 ℃ for 4 hours; the residue is NMT 1.0%.

(4) ***Water-insoluble substances***—Wash thoroughly the dried substances obtained in (3) with 200 mL of water and dry at 105 ℃ for 4 hours; the residue is NMT 0.15%.

(5) ***Alkali carbonates—***To the washings obtained in (4), add 3 drops of methyl orange TS and 2 mL of 0.05 mol/L sulfuric acid; the solution exhibits a red color.

Loss on drying NMT 5.0% as powder, NMT 10.0% as grains. Weigh accurately about 0.5 g of Medicinal Soap in a tared beaker, add 10 g of sea sand, previously dried at 105 ℃ for 1 hour, and again weigh the mass. Add 10 mL of ethanol, evaporate on a water bath to dryness with thorough stirring, and dry at 105 ℃ for 3 hours.

Packaging and storage Preserve in well-closed containers.

Mentha Oil
박하유

Mentha Oil is an essential oil obtained by steam distillation of the aerial parts of *Mentha arvensis* Linné var. *piperascens* Malinvaud (Labiatae), cooling the oil and removing the solids.

Mentha Oil contains NLT 30.0% of menthol ($C_{10}H_{20}O$: 156.27).

Description Mentha Oil occurs as a colorless to pale yellow, clear liquid with a unique and refreshing aroma. The taste is burning at first and then becomes refreshing.
It is miscible with ethanol, anhydrous ethanol, warm ethanol or ether.
It is practically insoluble in water.

Refractive index n_D^{20}: Between 1.455 and 1.467.

Specific optical rotation $[\alpha]_D^{20}$: Between - 17.0° and - 36.0° (100 mm).

Specific gravity d_{25}^{25}: Between 0.885 and 0.910.

Acid Value NMT 1.0.

Purity (1) ***Clarity and color of solution***—Add 3.5 mL of diluted ethanol (7 in 10) to 1.0 mL of Mentha Oil, and shake to

mix; it dissolves clearly. Add 10 mL more of ethanol again; the turbidity of the solution, clear or turbid, is not more intense than that of the following control solution.

Control solution—Add 6 mL of dilute nitric acid and water to 0.70 mL of 0.01 mol/L hydrochloric acid to make 50 mL, add 1 mL of silver nitrate TS, and allow the mixture to stand for 5 minutes.

(2) ***Heavy Metals***—Take 1.0 mL of Mentha Oil and perform as directed under Method 2. Prepare the control solution by adding 4.0 mL of the lead standard solution (NMT 40 ppm).

(3) ***Dimethyl sulfide***—Add carefully 5 mL of mercuric chloride TS to 1 mL of distillate obtained by distilling 25 mL of Mentha Oil; a white film does not form within 1 minute on the area where the two liquids come into contact.

Assay Weigh accurately about 5 g of Mentha Oil, and dissolve in ethanol to make exactly 20 mL. Pipet 10 mL of this solution, add exactly 10 mL of the internal standard solution, and use this solution as the test solution. Separately, weigh accurately about 10 g of *l*-menthol RS, and dissolve in ethanol to make exactly 100 mL. Pipet 10 mL of this solution, add exactly 10 mL of the internal standard solution, and use this solution as the standard solution. Perform the test with 1 μL each of the test solution and the standard solution as directed under the Gas Chromatography according to the following conditions, and determine the ratios, Q_T and Q_S, of the peak area of menthol to that of the internal standard.

$$\text{Amount (mg) of menthol } (C_{10}H_{20}O) = \text{Amount (mg) of } l\text{-menthol RS} \times \frac{Q_T}{Q_S} \times \frac{1}{5}$$

Internal standard solution—Ethanol solution of n-ethyl caprylate (1 in 25).

Operating conditions

Detector: A flame ionization detector

Column: A column, about 3 mm in internal diameter and about 2 m in length, packed with acid-treated diatomaceous earth for gas chromatography (180 to 250 μm in particle diameter), coated with polyethylene glycol 6000 for gas chromatography at a ratio of 25%.

Column temperature: A constant temperature of about 150 °C.

Carrier gas: Nitrogen

Flow rate: Adjust the flow rate so that the retention time of the internal standard is about 10 minutes.

Selection of column: Proceed with 1 μL of the standard solution according to the above conditions; the internal standard and l-menthol are eluted in this order with the resolution being NLT 5.

Packaging and storage Preserve in light-resistant, tight containers.

Methacrylic Acid·Ethyl Acrylate Copolymer
메타아크릴산·아크릴산에틸공중합체

Eudragit L30D-55

Methacrylic Acid·Ethyl Acrylate Copolymer is a suspension of copolymer obtained from an aqueous solution of methacrylic acid and ethyl acrylate in polysorbate 80.

Methacrylic Acid·Ethyl Acrylate Copolymer contains 11.5% to 15.5% of methacrylic acid ($C_4H_6O_2$ 86.09).

Description Methacrylic Acid·Ethyl Acrylate Copolymer occurs a white suspension. It has a characteristic odor and a slightly sour taste.
It is freely soluble in ethanol or acetone and practically insoluble in ether.
slightly sour taste.
It is soluble in dilute sodium hydroxide TS. slightly sour taste.
It evenly disperse in water.

Identification (1) Add 0.5 mL of Methacrylic Acid·Ethyl Acrylate Copolymer to 5 mL of dilute sodium hydroxide TS, and shake to mix; the solution is clear and viscous. Next, add 1 mL of dilute hydrochloric acid; a white resin-like precipitate forms.

(2) To 5 mL of Methacrylic Acid·Ethyl Acrylate Copolymer, add 3 mL of ammonium thiocyanate-cobalt nitrate TS, shake well to mix, and then add 10 mL of chloroform. Shake to mix, and allow to stand; the chloroform layer exhibits a blue color.

(3) Determine the infrared spectrum of 1 mg of the residue obtained from the evaporation residue as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; it exhibits absorption at the wavenumbers of about 2980 cm^{-1}, 1735 cm^{-1}, 1700 cm^{-1}, 1470 cm^{-1}, 1448 cm^{-1}, 1385 cm^{-1}, and 1180 cm^{-1}.

Viscosity Between 3 mm^2/s and 15 mm^2/s (Method 1, 20 °C)

pH Between 2.1 and 3.1

Specific Gravity d_{20}^{20}: Between 1.055 and 1.080.

Purity (1) ***Heavy metals***—Proceed with 2.0 g of Methacrylic Acid·Ethyl Acrylate Copolymer according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 10 ppm).

(2) ***Arsenic***—Proceed with 2.0 g of Methacrylic Acid·Ethyl Acrylate Copolymer according to Method 3 and perform the test (NMT 1 ppm).

(3) ***Ethyl acrylate***—Weigh accurately about 1.0 g of Methacrylic Acid·Ethyl Acrylate Copolymer, transfer into 8 mL of acetone, shake to dissolve, then add acetone to make 10.0 mL, and use this solution as the test solution. Separately, weigh accurately about 10 mg of ethyl acrylate RS, dissolve in acetone to make 100.0 mL, and use this solution as the standard solution. Perform the test with 1 μL each of the test solution and the standard solution as directed under the Gas Chromatography according to the following conditions; the peak height of ethyl acrylate obtained from the test solution is less than the peak height of ethyl acrylate obtained from the standard solution.

Operating conditions

Detector: A flame ionization detector

Column: A stainless steel column about 3 mm in internal diameter and about 2 m in length, packed with diatomaceous earth for gas chromatography, coated with polyethylene glycol 20 M for gas chromatography at the ratio of 20% (177 μm to 297 μm in particle diameter).

Column temperature: A constant temperature of about 70 °C.

Carrier gas: Nitrogen

Flow rate: Adjust the flow rate so that the retention time of

ethyl acrylate is about 3.5 minutes.

Detection sensitivity: Adjust the sensitivity so that the peak height of ethyl acrylate obtained from the standard solution is about 2 cm.

Residue on evaporation Weigh accurately about 1 g of Methacrylic Acid·Ethyl Acrylate, and dry at 105 ℃ for 4 hours; the amount of residue is 27.0% to 33.0%.

Residue on ignition NMT 0.1% (2 g)

Assay Take accurately about 1.0 g of Methacrylic Acid·Ethyl Acrylate Copolymer, transfer into 20 mL of ethanol, heat to dissolve, and cool. Then, titrate with 0.1 mol/L sodium hydroxide VS (indicator: 3 drops of phenolphthalein TS). Perform a blank test in the same way and make necessary correction.

Each mL of 0.1 mol/L sodium hydroxide VS
= 8.609 mg of $C_4H_6O_2$

Packaging and storage Preserve in tight containers.

Methacrylic Acid·Methyl Methacrylate Copolymer
메타아크릴산·메타아크릴산메틸공중합체

Eudragit L

Methacrylic Acid·Methyl Methacrylate Copolymer is a copolymer of methacrylic acid and methyl methacrylate.

Methacrylic Acid·Methyl Methacrylate Copolymer, when dried, contains 97.0% to 52.0% of methacrylic acid ($C_4H_6O_2$86.09).

Description Methacrylic Acid·Methyl Methacrylate Copolymer occurs as a white powder. It is odorless.
It is freely soluble in methanol, ethanol, or dimethylformamide and practically insoluble in ether. It is soluble in sodium hydroxide TS.
It is practically insoluble in water.

Identification (1) Add 1.0 g of Methacrylic Acid·Methyl Methacrylate Copolymer to 10 mL of sodium hydroxide TS, warm to dissolve, and then add 5 mL of dilute hydrochloric acid; a white resin-like substance forms.

(2) Weigh 1 mg of Methacrylic Acid·Methyl Methacrylate Copolymer, previously dried at 105 ℃ for 4 hours, and determine the absorption as directed under the potassium bromide disk method of the Mid-infrared Spectroscopy; it exhibits absorption at the wavenumbers of about 2950 cm^{-1}, 1735 cm^{-1}, 1485 cm^{-1}, 1450 cm^{-1}, 1390 cm^{-1}, 1260 cm^{-1}, 1150 cm^{-1}, and 960 cm^{-1}.

Viscosity Between 10 mm^2/s and 24 mm^2/s (Method 1, 20 ℃)

Purity (1) ***Clarity and color of solution***—Dissolve 0.5 g of Methacrylic Acid·Methyl Methacrylate Copolymer in 15 mL of ethanol; the resulting solution is colorless and clear.

(2) ***Heavy metals***—Proceed with 1.0 g of Methacrylic Acid·Methyl Methacrylate Copolymer according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 20 ppm).

(3) ***Arsenic***—Proceed with 1.0 g of Methacrylic Acid·Methyl Methacrylate Copolymer according to Method 3, and perform the test (NMT 2 ppm).

(4) ***Methyl methacrylate***—Weigh accurately about 0.5 g of Methacrylic Acid·Methyl Methacrylate Copolymer, transfer into 8 mL of dimethylformamide, shake to mix, and then add dimethylformamide to make 10.0 mL. Use this solution as the test solution. Separately, weigh accurately 10 mg of methyl methacrylate RS, dissolve in dimethylformamide to make 10.0 mL, and use this solution as the standard solution. Perform the test with 1 μL each of the test solution and the standard solution as directed under the Gas Chromatography according to the following conditions; the peak height of methyl methacrylate obtained from the test solution is less than the peak height of methyl methacrylate obtained from the standard solution.

Operating conditions

Detector: A flame ionization detector

Column: A stainless steel column about 3 mm in internal diameter and about 2 m in length, packed with diatomaceous earth for gas chromatography, coated with polyethylene glycol 20 M for gas chromatography at the ratio of 20% (177 μm to 297 μm in particle diameter).

Column temperature: A constant temperature of about 150 ℃.

Carrier gas: Nitrogen

Flow rate: Adjust the flow rate so that the retention time of methyl methacrylate is about 1.5 minutes.

Detection sensitivity: Adjust the sensitivity so that the peak height of methyl methacrylate obtained from the standard solution is about 2 cm.

Loss on drying NMT 10.0% (1 g, 105 ℃, 4 hours).

Residue on ignition NMT 0.2% (1 g)

Assay Weigh accurately about 0.3 g of Methacrylic Acid·Methyl Methacrylate Copolymer, previously dried, add 20 mL of ethanol, and warm to dissolve. After cooling, titrate with 0.1 mol/L sodium hydroxide VS (indicator: 3 drops of phenolphthalein TS). Perform a blank test in the same way and make necessary correction.

Each mL of 0.1 mol/L sodium hydroxide VS
= 8.609 mg of $C_4H_6O_2$

Packaging and storage Preserve in tight containers.

Methylcellulose
메틸셀룰로오스

[*9004-67-5*]

Methylcellulose is a methyl ether of cellulose.

Methylcellulose contains NLT 26.0% and NMT 33.0% of the methoxy group (- OCH_3: 31.03), calculated on the dried basis.

The viscosity of Methylcellulose is indicated in millipascal seconds (mPa·s) on the label.

Description Methylcellulose occurs as a white to yellowish white powder or grains.
It is practically insoluble in ethanol (99.5).
To Methylcellulose, add water; it swells and becomes a clear or slightly turbid, viscous liquid.

Identification (1) Disperse 1 g of Methylcellulose evenly on the surface of 100 mL of water in a beaker, gently tap the upper edge of the beaker if necessary, and allow to stand; it aggregates on the surface of water.

(2) Add 1.0 g of Methylcellulose in 100 mL of hot water, and shake to mix; it becomes a suspension. Cool this suspension to 5 ℃, and shake; it becomes a clear or slightly turbid viscous liquid.

(3) To 0.1 mL of the solution obtained from (2), add 9 mL of diluted sulfuric acid (9 in 10), and shake to mix. Heat on a water bath for 3 minutes exactly, then immediately cool in iced-water, carefully add 0.6 mL of ninhydrin TS, and shake to mix. Allow to stand at 25 ℃; the resulting solution exhibits a red color, and after standing for another 100 minutes, the color does not change to purple.

(4) Take 2 mL to 3 mL of the solution obtained from (2), spread out on a slide glass, and evaporate water; a transparent film forms.

(5) Pipet 50 mL of water, add exactly 50 mL of the solution obtained from (2), and warm at the rate of 2 ℃ to 5 ℃ per minute for 1 minute while stirring to mix. Determine the coagulation temperature when white turbidity begins to increase; it is NLT 50 ℃.

Viscosity ***Method 1***—Apply to Methylcellulose with a labeled viscosity of less than 600 mPa·s. Weigh an amount equivalent to 4.000 g of Methylcellulose, calculated on the dried basis, transfer into a wide-mouth bottle, add hot water to make 200.0 g, and stopper the bottle. Use a stirrer to mix at 350 to 450 revolutions per minute for 10 to 20 minutes until the solution becomes a homogeneous dispersion. If necessary, take off the sample adhering to the wall of the bottle, transfer into the dispersion, and then stir on a water bath at NMT 5 ℃ for 20 to 40 minutes to dissolve. If necessary, add cold water to make 200.0 g, and centrifuge if there are air bubbles in the solution or on the surface to remove the bubbles, and use this solution as the test solution. Perform the test with the test solution according to Method 1 under the Viscosity at 20 ± 0.1 ℃; it is 80% to 120% of the labeled viscosity.

Method 2—Apply to Methylcellulose with a labeled viscosity of NLT 600 mPa·s. Weigh exactly an amount equivalent to 10.00 g of Methylcellulose, calculated on the dried basis, transfer into a wide-neck bottle, add hot water to make 500.0 g, and proceed in the same manner as Method 1, and use this solution as the test solution. Perform the test with the test solution using a single cylinder type rotational viscometer used in Method 2 under the Viscosity at 20 ± 0.1 ℃ according to the following conditions; it is 75% to 140% of the labeled viscosity.

Operating conditions

Apparatus model: Brookfield viscometer, LV model or an equivalent viscometer

Cylinder number. Rotation speed and conversion factor: Use the conditions as directed in the following table, according to the labeled viscosity.

Labeled viscosity (mPa·s)	Cylinder number	Rotation speed (rev/min)	Conversion factor
≥ 600 and < 1400	3	60	20
≥ 1400 and < 3500	3	12	100
≥ 3500 and < 9500	4	60	100
≥ 9500 and < 99500	4	6	1000
≥ 99500	4	3	2000

Operation of the apparatus: Turn on the apparatus and rotate for 2 minutes, then read the viscometer reading, and stop for 2 minutes. Repeat the same operation twice, and average the results from three readings.

pH Allow the test solution from the Viscosity to stand at 20 ± 0.2 ℃ for 5 minutes; the pH of the solution is 5.0 to 8.0.

Loss on drying NMT 5.0% (1 g, 105 ℃, 1 hour).

Residue on ignition NMT 1.5% (1 g).

Assay ***Apparatus***—Reaction bottle: Use a 5-mL pressure-resistant serum vial of 20 mm in external diameter, 50 mm in height, 20 mm in the external diameter of the head portion, 13 mm in internal diameter, and a stopper made of butyl–rubber having the surface processed with polytetrafluoroethylene, which can be fixed to the vial with air-tightness using an aluminum seal or a sealing system with an equivalent structure.

Heater: A square aluminum heat plate with holes of 20 mm in diameter and 32 mm in depth, suitable for fitting the reaction bottle. Use a heater with a structure of stirring the content of the reaction bottle with a magnetic stirrer or one fitted with a shaker capable of reciprocal shaking at about 100 times per minute.

Procedure—Weigh accurately about 650 mg of Methylcellulose, transfer into a reaction bottle, add 60 mg to 100 mg of adipic acid, 2.0 mL of internal standard solution, 2.0 mL of hydroiodic acid, immediately seal tightly, and then weigh the mass accurately. Shake to mix using a magnetic stirrer or shaker for 60 minutes while heating the heat plate so that the temperature of contents in the reaction bottle becomes 130 ± 2 ℃. If a magnetic stirrer or shaker is not used, manually shake every 5 minutes during the initial 30 minutes of heating. After cooling, weigh the mass accurately, and if the mass loss is less than 26 mg or if the contents do not leak, use the upper layer of the mixture as the test solution. Separately, transfer 60 mg to 100 mg of adipic acid, 2.0 mL of the internal standard solution, and 2.0 mL of hydroiodic acid into a reaction bottle, seal tightly, and then weigh the mass accurately. Using a micro-syringe, inject 45 μL of iodomethane RS through the stopper, and weigh the mass accurately. Shake the reaction bottle to mix, and use the upper layer of the contents as the standard solution. Perform the test with 1 to 2 μL each of the test solution and the standard solution as directed under the Gas Chromatography according to the following conditions to obtain the peak area ratios, Q_T and Q_S, of iodomethane to the internal standard, respectively.

$$\text{Amount (\%) of methoxy group } (-OCH_3\text{: } 31.03) = (W_S / W) \times (Q_T / Q_S) \times 21.86$$

Internal standard solution—A solution of n-octane in o-xylene (3 in 100).

Operating conditions

Detector: A thermal conductivity detector or flame ionization detector.

Column: A fused silica column 0.53 mm in internal diameter and 30 m in length, coated inside with dimethylpolysiloxane for gas chromatography with 3 μm in thickness. Use a guard column if necessary.

Column temperature: Heat to 50 ℃ and maintain for 3 minutes, then increase the temperature by 10 ℃ per minute to 100 ℃, ramp to 250 ℃ at a rate of 35 ℃ per minute, and maintain at 250 ℃ for 8 minutes.

Temperature of the sample injection port: 250 ℃

Temperature of the detector: 280 ℃

Carrier gas: Helium

Flow rate: 4.3 mL/minute (adjust the flow rate so that the retention time of the internal standard is about 10 minutes).

Split ratio: 1 : 40

System suitability

System performance: Proceed with 1 to 2 μL of the standard solution according to the above operating conditions; iodomethane and the internal standard are eluted in this order with the resolution of each peak being NLT 5.

System repeatability: Repeat the test 6 times with 1 μL to 2 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak area ratio of iodomethane to the internal standard is NMT 2.0%.

Packaging and storage Preserve in well-closed containers.

Monobasic Calcium Phosphate Hydrate
인산이수소칼슘수화물

$Ca(H_2PO_4)_2 \cdot H_2O$: 252.07

Monobasic Calcium Phosphate Hydrate, when dried, contains NLT 90.0% and NMT 101.0% of monobasic calcium phosphate hydrate [$Ca(H_2PO_4)_2 \cdot H_2O$].

Description Monobasic Calcium Phosphate Hydrate occurs as a white crystal or crystalline powder. It is odorless and has a sour taste.

It is sparingly soluble in water and practically insoluble in ethanol or ether.

It dissolves in dilute hydrochloric acid or dilute nitric acid.

It is slightly deliquescent.

Identification Perform the test as directed under the Identification of Dibasic Calcium Phosphate Hydrate.

Purity (1) ***Clarity and color of solution***—Add 19 mL of water and 2 mL of diluted hydrochloric acid (3 in 4) to 1.0 g of Monobasic Calcium Phosphate Hydrate, and heat on a water bath for 5 minutes while shaking occasionally; the solution is colorless and clear.

(2) ***Hydrogen phosphate and acids***—Triturate 1.0 g of Monobasic Calcium Phosphate Hydrate with 3 mL of water, and add 100 mL of water and 1 drop of methyl orange TS; the resulting solution exhibits a red color. Add 1.0 mL of 1 mol/L sodium hydroxide; the resulting solution exhibits a yellow color

(3) ***Chloride***—Dissolve 1.0 g of Monobasic Calcium Phosphate Hydrate in 20 mL of water and 12 mL of dilute nitric acid, and add water to make 100 mL; filter if necessary. Use 50 mL of this solution as the test solution and perform the test. Prepare the control solution with 0.25 mL of 0.01 mol/L hydrochloric acid (NMT 0.018%).

(4) ***Sulfate***—Dissolve 1.0 g of Monobasic Calcium Phosphate Hydrate in 20 mL of water and 1 mL of hydrochloric acid, and add water to make 100 mL; filter if necessary. Use 50 mL of this solution as the test solution and perform the test. Prepare the control solution with 0.5 mL of 0.005 mol/L sulfuric acid (NMT 0.048%).

(5) ***Heavy metals***—Perform the test as directed in the Purity (5) under Dibasic Calcium Phosphate Hydrate.

(6) ***Mercury***—Spread about 1 g of excipients (a) evenly on a porcelain boat, and place 10 mg to 300 mg of Monobasic Calcium Phosphate Hydrate on top. Then, evenly spread about 0.5 g of excipients (a) and 1 g of excipients (b) successively on top to form a layer. In the case of an automated mercury analyzer equipped with a separate catalyst in the combustion chamber, take only the sample without adding excipients to a nickel boat. Place the boat inside the combustion furnace and heat to about 900 °C with a current of air or oxygen at 0.5 to 1 L/minute. Elute the mercury and collect in a collecting tube. Transfer the mercury vapor to a cold vapor atomic absorption spectrometer by heating the collecting tube to about 700 °C and determine the absorbance as A. Separately, determine the absorbance in the same manner using only the excipients in a ceramic boat and designate as Ab. Separately, proceed with mercury standard solution in the same manner and create a calibration curve from the absorbances. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the sample (NMT 1.0 ppm).

Operating conditions

Apparatus: Use an automated mercury analyzer for specimen combustion, trapping with gold amalgam and measurement using a cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Standard mercury stock solution—Dissolve 0.135 g of mercury(II) chloride in 0.001% L-cysteinesolution to make 1000 mL. Each mL of this solution contains 100 μg of mercury(II) chloride.

Mercury standard solution—Dilute the standard mercury stock solution with 0.001% L-cysteine solution 0 ng to 200 ng/mL.

Excipients—Use (a) aluminum oxide and (b) a mixture of calcium hydroxide and sodium carbonate (1 : 1), and activate at 950 °C for 30 minutes before use.

(7) ***Cadmium***—Use the test solution in the Purity (8) as the test solution. Separately proceed with 5.0 mL of lead standard solution in the same manner as the test solution, and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Atomic absorption spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 1.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Cadmium hollow-cathode lamp
Wavelength: 228.8 nm

(8) ***Lead***—Weigh accurately 5.0 g of Dibasic Sodium Phosphate Hydrate, put it in a 150-mL beaker, and add 30 mL of water. Add hydrochloric acid in small amounts until the sample is sufficiently dissolved, and add 1 mL of hydrochloric acid again. Boil for about 5 minutes, cool, add water to make about 100 mL, and adjust the pH to between 2 and 4 with sodium hydroxide solution (1 in 4) or hydrochloric acid (1 in 4). Transfer this solution to a 250-mL separatory funnel, add water to make about 200 mL, add 2 mL of 2% ammonium pyrolidine dithiocarbamate solution (2 in 100), and shake to mix. Then, extract this solution with two 20 mL volumes of chloroform, evaporate the extract to dryness on a water bath, add 3 mL of nitric acid to the residue, and heat until almost dry. Add 0.5 mL of nitric acid and 10 mL of water, concentrate until the final solution becomes 3 mL to 5 mL, add water to make 10 mL, and use this solution as the test solution. Separately, proceed with 2.0 mL of lead standard solution in the

same manner as the test solution, and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Atomic absorption spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 4.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: A lead hollow-cathode lamp
Wavelength: 283.3 nm

(9) ***Arsenic***—Dissolve 1.0 g of Monobasic Calcium Phosphate Hydrate in 5 mL of dilute hydrochloric acid and perform the test using this solution as the test solution (NMT 2 ppm).

(10) ***Fluoride***—Weigh 1 g of Monobasic Calcium Phosphate Hydrate, place in a beaker, and dissolve with 10 mL of hydrochloric acid (1 in 10). Heat this solution, boil for 1 minute, then transfer it into a polyethylene beaker, and cool immediately. Add 15 mL of sodium citrate solution (1 in 4) and 10 mL of ethylenediaminetetraacetic acid disodium salt solution (1 in 40), and shake to mix. Adjust the pH to between 5.4 and 5.6 with hydrochloric acid (1 in 10) or sodium hydroxide solution (2 in 5), add water to make 100 mL, and use this solution as the test solution. Put 50 mL of the test solution in a polyethylene beaker, measure the potential using a fluoride electrode, and determine the amount of fluorine from the calibration curve; the amount should be NMT 10 ppm.

Creation of calibration curve: Weigh accurately 2.210 g of sodium fluoride, previously dried at 200 °C for 4 hours, put it in a polyethylene beaker, and dissolve with 200 mL of water. Then add water to make 1000 mL and store in a polyethylene container. Take exactly 5 mL of this solution, place it in a mess flask, and add water to make 1000 mL (each mL of this solution contains 5 μg of fluorine). Take 1 mL, 2 mL, 3 mL, 5 mL, 10 mL and 15 mL of this solution, place each in a polyethylene beaker, add 15 mL of sodium citrate solution (1 in 4) and 10 mL of ethylenediaminetetrisodium (1 in 40) to each of the beakers, and mix. Adjust the pH to between 5.4 and 5.6 with hydrochloric acid (1 in 10) or sodium hydroxide solution (2 in 5), then add water to make 100 mL, and use these solutions as the standard solutions. Take 50 mL from each standard solution, place each in a polyethylene beaker, measure the potential with a fluoride electrode, and plot a calibration curve using the log values of the fluorine concentrations.

Loss on drying NMT 3.0% (1 g, silica gel, 24 hours).

Loss on ignition Ignite the anhydride of Monobasic Calcium Phosphate Hydrate at 800 °C for 30 minutes; the loss should be 14.0% to 15.5%.

Assay Weigh accurately about 0.4 g of Monobasic Calcium Phosphate Hydrate, previously dried, dissolve in 3 mL of dilute hydrochloric acid, and add water to make exactly 100 mL. Take accurately 20 mL of this solution, and perform the test as directed in the Assay under Dibasic Calcium Phosphate Hydrate.

Each mL of 0.02 mol/L ethylenediaminetetraacetic acid disodium salt VS
= 5.041 mg of $Ca(H_2PO_4)_2 \cdot H2O$

Packaging and storage Preserve in tight containers.

Nitrogen
질소

N_2 : 28.01

Nitrogen contains NLT 99.5 vol% and NLT 101.0 vol% of nitrogen (N_2).

Description Nitrogen occurs as a colorless gas, and is odorless.
1 mL of Nitrogen dissolves in 65 mL of water or 9 mL of ethanol at a temperature of 20°C and a pressure of 101.3 kPa.
1000 mL of Nitrogen is about 1.251 g at a temperature of 0 ℃ and at a pressure of 101.3 kPa.
Nitrogen is inert, and does not burn in air.

Identification Put a burning piece of wood to Nitrogen; it is soon extinguished.

Purity (1) ***Carbon Dioxide***—An amount of Nitrogen is collected after maintaining the container at 18 ℃ to 22 ℃ for NLT 6 hours before performing the test. Convert the amount to a volume at a pressure of 101.3 kPa at 20 ℃, and use the volume as the collected amount of Nitrogen. Put 50 mL of barium hydroxide TS in a Nessler tube, place the end of the gas injection tube (about 1 mm in internal diameter) 2 mm away from the bottom of the Nessler tube, and pass 1000 mL of Nitrogen through the Nessler tube for 15 minutes; the turbidity of the solution is not more intense than the following control solution.

Control solution—Put 50 mL of barium hydroxide TS in a Nessler tube, and add 1 mL of a solution prepared by dissolving 0.1 g of sodium bicarbonate in 100 mL of freshly boiled and cooled water.

(2) ***Oxygen***—Determine the peak areas, A_T and A_S, of oxygen from the test solution and the standard solution chromatograms obtained from the Assay; the amount of oxygen is NMT 0.5%.

$$\text{Amount (vol\%) of oxygen} = \frac{A_T}{A_S}$$

Assay Take Nitrogen as directed under the Purity. Take 1.0 mL of Nitrogen into a gas measuring tube or syringe for gas chromatography directly from a pressure-resistant metal hermetic container with a pressure-reducing valve using a polyvinyl chloride-based injection tube, and perform the test with this gas as directed under the Gas Chromatography according to the following conditions, and determine the peak area, A_T, of oxygen. Separately, transfer 1.0 mL of oxygen into a gas mixer, add carrier gas to make the total volume exactly 100 mL, mix well, and use this gas as the standard gas mixture. Proceed with 1.0 mL of this mixture in the same manner as in the preparation of Nitrogen, and determine the peak area of oxygen, A_S.

$$\text{Amount (vol\%) of nitrogen } (N_2) = 100 - \frac{A_T}{A_S}$$

Operating conditions

Detector: A thermal conductivity detector

Column: A column about 3 mm in internal diameter and about 3 m in length, packed with 250 μm to 350 μm zeolite for gas chromatography (pore diameter 0.5 nm).

Column temperature: A constant temperature of about 50 °C.

Carrier gas: Hydrogen or helium

Flow rate: Adjust the flow rate so that the retention time of oxygen is about 3 minutes.

System suitability

System performance: Take 1.0 mL of Oxygen into a gas mixer, add Nitrogen to make 100 mL, and mix well. Proceed with 1.0 mL of the mixture according to the above conditions; oxygen and nitrogen are eluted in this order, and the individual peaks are completely separated.

System repeatability: Repeat the test 5 times with the standard gas mixture according to the above conditions; the relative standard deviation of the peak areas of oxygen is NMT 2.0%.

Packaging and storage Preserve in pressure-resistant metal hermetic containers at below 40 ℃.

Olive Oil
올리브유

Olive Oil is the fatty oil obtained by pressing the ripe fruit of *Olea europaea* Linné (Oleaceae).

Description Olive Oil is a pale yellow oil, has a faint odor that is not rancid and has a mild taste.
It is miscible with ether or petroleum ether.
It is sparingly soluble in ethanol.
All or part of Olive Oil congeals at between 0 °C and 6 °C.
Congealing point of the fatty acids: Between 17 ℃ and 26 ℃.

Saponification value Between 186 and 194.

Unsaponifiable matter NMT 1.5%.

Specific gravity d_{25}^{25}: Between 0.908 and 0.914.

Acid Value NMT 1.0.

Iodine value Between 79 and 88.

Purity (1) ***Drying oil***—To 2 mL of Olive Oil, add 10 mL of diluted nitric acid (1 in 4), carefully add 1 g of powdered sodium nitrite while shaking and allow the mixture to stand in a cold place for 4 to 10 hours; the mixture congeals to a white solid.

(2) ***Peanut oil***—Weigh accurately about 1.0 g of Olive Oil, dissolve in 60 mL of sulfuric acid-hexane-methanol TS, boil the mixture for 2.5 hours on a water bath under a reflux condenser, cool, transfer it to a separatory funnel, and add 100 mL of water. Wash the flask with 50 mL of petroleum ether, add the washings to the separatory funnel, shake to mix, allow it to stand and collect the petroleum ether layer. Extract the water layer with another 50 mL of petroleum ether and combine the petroleum ether layer with the former petroleum ether solution. Wash the petroleum ether solution repeatedly with 20 mL each of water until the washings show no more acidity to methyl orange TS. Then, add 5 g of anhydrous sodium sulfate, shake to mix, filter, and wash the anhydrous sodium sulfate twice with 10 mL each of petroleum ether. Filter the washings using the funnel described above, combine the filtrates, and evaporate the petroleum ether on a water bath under nitrogen. Dissolve the residue in acetone to make exactly 20 mL and use this solution as the test solution. Separately, dissolve 67 mg of methyl behenate in acetone to make exactly 50 mL. Pipet 2 mL of this solution, add acetone to make exactly 20 mL, and use this solution as the standard solution. Pipet 2 μL each of the test solution and the standard solution, and perform the test as directed under the Gas Chromatography according to the following conditions, and determine the peak heights of methyl behenate in each solution, H_T and H_S; H_T is not greater than H_S.

Operating conditions

Detector: A flame ionization detector

Column: A glass column about 3 mm in internal diameter and about 2 m in length, packed with silane-treated diatomaceous earth for gas chromatography (150 μm to 180 μm in particle diameter), coated with polyethylene glycol 20 M for gas chromatography in a ratio of 5%.

Column temperature: A constant temperature of about 220 ℃.

Carrier gas: Nitrogen

Flow rate: Adjust the flow rate so that the retention time of methyl behenate is about 18 minutes.

Detection sensitivity: Adjust the sensitivity so that the peak height of methyl behenate obtained from 2 μL of the standard solution is between 5 and 10 mm.

(3) ***Heavy metals***—Weigh 1.0 g of Olive Oil and perform the test according to Method 2. Prepare the control solution by adding 1.0 mL of the lead standard solution (NMT 10 ppm).

Packaging and storage Preserve in tight containers.

Orange Oil
오렌지유

Orange Oil is the essential oil obtained by pressing the pericarp of the edible fruit of Citrus species (Rutaceae).

Description Orange Oil occurs as a yellow to yellowish brown liquid and has a characteristic, aromatic odor and a slightly bitter taste.
It is miscible with an equal volume of ethanol with turbidity.

Refractive index n_D^{20}: Between 1.472 and 1.474

Specific optical rotation $[\alpha]_D^{20}$: Between $+85^\circ$ and $+99^\circ$ (100 mm).

Specific gravity d_{20}^{20}: Between 0.842 and 0.848

Purity ***Heavy metals***—Pipet 1.0 mL of Orange Oil and perform the test according to Method 2. Prepare the control solution by adding 4.0 mL of the lead standard solution (NMT 40 ppm).

Packaging and storage Preserve in light-resistant, tight containers.

Palm Oil
팜유

Palm Oil is an oil collected from the pulp of *Elaeis guineensis* L. palm tree.

Description Palm Oil is colorless and transparent at 50℃. It has its own characteristic color and flavor without any other odor.

Specific gravity d_{25}^{40}: Between 0.900 and 0.907

Refractive index n_D^{20}: Between 1.453 and 1.459

Water NMT 0.1%.

Acid value NMT 0.2.

Saponification value Between 190 and 209

Unsaponifiable substances NMT 1.0%

Iodine value Between 44 and 60

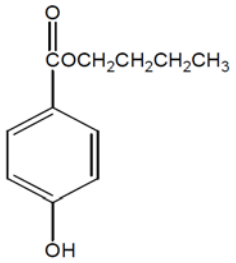
Antioxidants (g/kg) Antioxidants other than those specified below are not detected.

(1) ***Butylated hydroxyanisole and butylated hydroxytoluene***—NMT 0.2 (when used together, the total amount of butylated hydroxyanisole and butylated hydroxytoluene used is NMT 0.2).

(2) ***Propyl gallate***—NMT 0.1.

Packaging and storage Preserve in tight containers.

Butylparaben
파라옥시벤조산부틸



Butyl Parahydroxybenzoate $C_{11}H_{14}O_3$: 194.23
Butyl 4-hydroxybenzoate [94-26-8]

Butylparaben contains NLT 98.0% and NMT 102.0% of butylparaben ($C_{11}H_{14}O_3$).

Description Butylparaben occurs as a colorless crystal or white crystalline powder.
It is freely soluble in methanol, ethanol (95) or acetone and practically insoluble in water.

Identification (1) Determine the absorption spectra of Butylparaben and butylparaben RS as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

Melting point Between 68 °C and 71 °C.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Butylparaben in 10 mL of ethanol (95); the solution is clear and the color is not more intense than that of the following control solution.

Control solution—To 5.0 mL of cobalt chloride colorimetric stock solution, 12.0 mL of ferric chloride colorimetric stock solution and 2.0 mL of copper(II) sulfate pentahydrate colorimetric stock solution, add diluted hydrochloric acid (1 in 10) to make 1000 mL.

(2) ***Acidity***—Dissolve 0.20 g of Butylparaben in 3 mL of ethanol (95), and add 5 mL of freshly boiled and cooled water and 0.1 mL of bromocresol green-sodium hydroxide-ethanol TS. Then, add 0.1 mol/L sodium hydroxide solution until the solution exhibits a blue color: the amount consumed is NMT 0.1 mL.

(3) ***Related substances—***Dissolve 50.0 mg of Butylparaben in 2.5 mL of methanol, and add the mobile phase to make exactly 50 mL. Pipet 10 mL of this solution, add the mobile phase to make exactly 100 mL, and use this solution as the test solution. Separately, dissolve 5mg each of parahydroxybenzoate, propylparaben, and Butylparaben in the mobile phase to make exactly 100 mL. Pipet 1 mL of this solution. Add the mobile phase to make exactly 10 mL and use this solution as the standard solution (1). Separately, weigh accurately 50.0 mg of butylparaben RS, dissolve in 2.5 mL of methanol, and add the mobile phase to make 50.0 mL. Pipet 10 mL of this solution, add the mobile phase to make exactly 100.0 mL, and use this solution as the standard solution (2). Pipet 1 mL of the test solution, and add the mobile phase to make exactly 20 mL. Pipet 1 mL of this solution, add the mobile phase to make exactly 10.0 mL, and use this solution as the standard solution (3). Separately, weigh 5 mg of isobutylparaben RS, add the mobile phase to make exactly 100 mL, and use this solution as the standard solution (4). Pipet 0.5 mL of the standard solution (4), add the standard solution (2) to make exactly 50 mL, and use this solution as the standard solution (5). Perform the test with 10 μL each of the test solution and the standard solution (3) as directed under the Liquid Chromatography according to the following operating conditions and determine the peak area of each solution by the automatic integration method: the peak area of parahydroxybenzoate, which has a relative retention time of about 0.1 to that of butylparaben from the test solution, is not larger than the peak area of butylparaben from the standard solution (3) (0.5%). However, calculate the peak area of parahydroxybenzoate by multiplying by 1.4, the correction factor, to the area obtained by the automatic integration method. The area of the peaks other than butylparaben and parahydroxybenzoate in the test solution is not larger than the peak area of butylparaben in the standard solution (3) (0.5%). The sum of peak areas other than butylparaben in the test solution is not greater than 2 times the peak area of butylparaben in the standard solution (3) (1.0%). Disregard the peaks having areas smaller than 1/5 of the peak area of butylparaben in the standard solution (3) (0.1%).

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 272 nm)

Column: A stainless steel column about 4.6 mm in internal diameter and 15 cm in length, packed with octylsilanized porous silica gel for liquid chromatography of 5 μm.

Mobile phase: A mixture of methanol and buffer solution (1 : 1).

Buffer solution—Dissolve 17 g of potassium dihydrogen phosphate in 2500 mL of water.

Column temperature: A constant temperature of about 35 °C.

Flow rate: 1.3 mL/minute.

Time span of measurement: About 1.5 times the retention

time of butylparaben.

System suitability

Confirmation of detection: Proceed with 10 μL each of the standard solution (1) and the standard solution (5) according to the above conditions; the peaks of parahydroxybenzoate appear in the chromatogram obtained from the standard solution (1), and the peak of isobutylparaben appears in the chromatogram obtained from the standard solution (5). Butylparaben is eluted in about 22 minutes and the relative retention times of parahydroxybenzoate, propylparaben and isobutylparaben are 0.1, 0.5 and 0.9, respectively.

System performance: Proceed with 10 μL each of the standard solution (1) and the standard solution (5) according to the above conditions; the resolution between the peaks of propylparaben and butylparaben obtained from the standard solution (1) is NLT 5.0, and the resolution between the peaks of isobutylparaben and butylparaben obtained from the standard solution (5) is NLT 1.5.

System repeatability: Perform the test 6 times with each 10 μL of the standard solution (3) according to the above operating conditions; the relative standard deviation of the peak areas of butylparaben is NMT 2.0%.

Residue on ignition NMT 0.10% (1 g).

Assay Weigh accurately about 50.0 mg of Butylparaben, dissolve in 5 mL of methanol and add the mobile phase make exactly 50 mL. Pipet 10 mL of this solution, add the mobile phase to make exactly 100 mL, and use this solution as the test solution. Separately, weigh accurately about 50.0 mg of butylparaben RS and dissolve in 2.5 mL of methanol to make exactly 50 mL. Then, pipet 10 mL of this solution, add the mobile phase to make exactly 100.0 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas, A_T and A_S, of butylparaben from each solution.

$$\text{Amount (mg) of butylparaben } (C_{11}H_{14}O_3) = \text{Amount (mg) of butylparaben RS} \times (A_T / A_S)$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 272 nm)

Column: A stainless steel column about 4.6 mm in internal diameter and 15 cm in length, packed with octylsilanized porous silica gel for liquid chromatography of 5 μm.

Mobile phase: A mixture of methanol and buffer solution (1 : 1).

Buffer solution—Dissolve 17 g of potassium dihydrogen phosphate in 2500 mL of water.

Column temperature: A constant temperature of about 35 °C.

Flow rate: 1.3 mL/minute.

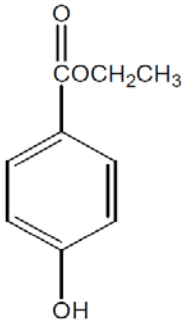
System suitability

For confirmation of detection and system performance, perform the test according to system suitability under the Purity (4) Related substances.

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak areas of butylparaben is NMT 0.85%.

Packaging and storage Preserve in well-closed containers.

Ethylparaben
파라옥시벤조산에틸



Ethyl Parahydroxybenzoate $C_9H_{10}O_3$: 166.17
Ethyl 4-hydroxybenzoate [*120-47-8*]

Ethylparaben contains NLT 98.0% and NMT 102.0% of ethylparaben ($C_9H_{10}O_3$).

Description Ethylparaben occurs as a colorless crystal or white crystalline powder.
It is freely soluble in methanol, ethanol (95) or acetone and very slightly soluble in water.

Identification (1) Determine the absorption spectra of Ethylparaben and ethylparaben RS as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

Melting point Between 115 °C and 118 °C

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Ethylparaben in 10 mL of ethanol (95); the solution is clear and the color is not more intense than that of the following control solution.

Control solution—To 5.0 mL of cobalt chloride colorimetric stock solution, 12.0 mL of ferric chloride colorimetric stock solution and 2.0 mL of copper(II) sulfate pentahydrate colorimetric stock solution, add diluted hydrochloric acid (1 in 10) to make 1000 mL.

(2) ***Acidity***—Dissolve 0.20 g of Ethylparaben in 3 mL of ethanol (95), and add 5 mL of freshly boiled and cooled water and 0.1 mL of bromocresol green-sodium hydroxide-ethanol TS. Then, add 0.1 mol/L sodium hydroxide solution until the solution exhibits a blue color: the amount consumed is NMT 0.1 mL.

(3) ***Related substances***—Dissolve 50.0 mg of Ethylparaben in 2.5 mL of methanol, and add the mobile phase to make exactly 50 mL. Pipet 10 mL of this solution, add the mobile phase to make exactly 100 mL, and use this solution as the test solution. Pipet 1 mL of the test solution, and add the mobile phase to make exactly 20 mL. Pipet 1 mL of this solution, add the mobile phase to make exactly 10 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions and determine the peak area of each solution by the automatic integration

method: the peak area of parahydroxybenzoate, which has a relative retention time of about 0.6 to that of ethylparaben from the test solution, is not larger than the peak area of ethylparaben from the standard solution (0.5%). However, make corrections for the peak area of parahydroxybenzoate by multiplying by 1.4, the correction factor. The area of the peaks other than ethylparaben and parahydroxybenzoate in the test solution is not larger than the peak area of ethylparaben in the standard solution (0.5%). The total area of peaks other than the peak of ethylparaben is not larger than 2 times the peak area of ethylparaben in the standard solution (1.0%). Disregard the peaks having areas smaller than 0.2 times the peak area of ethylparaben in the standard solution (NMT 0.1%).

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 272 nm)

Column: A stainless steel column about 4.6 mm in internal diameter and about 15 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

Mobile phase: A mixture of methanol and buffer solution (13 : 7)

Buffer solution—Dissolve 17 g of potassium dihydrogen phosphate in 2500 mL of water.

Flow rate: 1.3 mL/min (adjust the flow rate so that the retention time of methylparaben is about 3.0 minutes).

System suitability

Confirmation of detection: Take exactly 2 mL of the standard solution, and add the mobile phase to make exactly 10 mL. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and the area of this solution is 14 to 16% of the area of the standard solution.

System performance: Dissolve 5 mg each of Ethylparaben, parahydroxybenzoate and methylparaben in the mobile phase to make exactly 100 mL. Pipet 1 mL of this solution, and add the mobile phase to make exactly 10 mL. Proceed with 10 μL of this solution according to the above conditions; parahydroxybenzoate, methylparaben, and ethylparaben are eluted in this order and the relative retention times of parahydroxybenzoate and methylparaben to ethylparaben are 0.5 and 0.8, respectively, and the resolution between their peaks is NLT 2.

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak areas of methylparaben is NMT 2.0%.

Residue on ignition NMT 0.10% (1 g).

Assay Weigh accurately about 50.0 mg of Ethylparaben, dissolve in 2.5 mL of methanol, and add the mobile phase to make exactly 50 mL. Pipet 10 mL of this solution, add the mobile phase to make exactly 100 mL, and use this solution as the test solution. Pipet 1 mL of the test solution, add the mobile phase to make exactly 20 mL. Pipet 1 mL of this solution, add the mobile phase to make exactly 10 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas, A_T and A_S, of ethylparaben from each solution.

Amount (mg) of ethylparaben ($C_9H_{10}O_3$)
= Amount (mg) of ethylparaben RS × (A_T / A_S)

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 272 nm)

Column: A stainless steel column about 4.6 mm in internal diameter and about 15 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

Mobile phase: A mixture of methanol and buffer solution (13 : 7)

Buffer solution—Dissolve 17 g of potassium dihydrogen phosphate in 2500 mL of water.

Flow rate: 1.3 mL/min (adjust the flow rate so that the retention time of methylparaben is about 3.0 minutes).

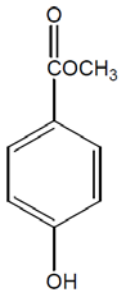
System suitability

System performance: Dissolve 5 mg each of Ethylparaben, parahydroxybenzoate and methylparaben in the mobile phase to make exactly 100 mL. Pipet 1 mL of this solution, and add the mobile phase to make exactly 10 mL. Proceed with 10 μL of this solution according to the above conditions; parahydroxybenzoate, methylparaben, and ethylparaben are eluted in this order and the relative retention times of parahydroxybenzoate and methylparaben to ethylparaben are 0.5 and 0.8, respectively, and the resolution between their peaks is NLT 2.

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak areas of ethylparaben is NMT 0.85%.

Packaging and storage Preserve in well-closed containers.

Methylparaben
파라옥시벤조산메틸



Methyl Parahydroxybenzoate $C_8H_8O_3$: 152.15
Methyl 4-hydroxybenzoate [99-76-3]

Methylparaben contains NLT 98.0% and NMT 102.0% of methylparaben ($C_8H_8O_3$).

Description Methylparaben occurs as a colorless crystal or white crystalline powder.
It is freely soluble in methanol, ethanol (95) or acetone and slightly soluble in water.

Identification Determine the absorption spectra of Methylparaben and methyl p-hydroxybenzoate RS as directed in the potassium bromide disk method under the Mid-infrared

Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

Melting point Between 125 ℃ and 128 ℃

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Methylparaben in 10 mL of ethanol (95); the solution is clear and the color is not more intense than that of the following *control solution.*

Control solution—To 5.0 mL of cobalt chloride colorimetric stock solution, 12.0 mL of ferric chloride colorimetric stock solution and 2.0 mL of copper(II) sulfate pentahydrate colorimetric stock solution, add diluted hydrochloric acid (1 in 10) to make 1000 mL.

(2) ***Acidity***—Dissolve 0.20 g of Methylparaben in 3 mL of ethanol (95), and add 5 mL of freshly boiled and cooled water and 0.1 mL of bromocresol green-sodium hydroxide-ethanol TS. Then, add 0.1 mol/L sodium hydroxide solution until the solution exhibits a blue color: the amount consumed is NMT 0.1 mL.

(3) ***Related substances—***Dissolve 50.0 mg of Methylparaben in 2.5 mL of methanol, and add the mobile phase to make exactly 50 mL. Pipet 10 mL of this solution, add the mobile phase to make exactly 100 mL, and use this solution as the test solution. Pipet 1 mL of the test solution, and add the mobile phase to make exactly 20 mL. Pipet 1 mL of this solution, add the mobile phase to make exactly 10 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions and determine the peak area of each solution by the automatic integration method: the peak area of parahydroxybenzoate, which has a relative retention time of about 0.6 to that of methylparaben from the test solution, is not larger than the peak area of methylparaben from the standard solution (0.5%). However, make corrections for the peak area of parahydroxybenzoate by multiplying by 1.4, the correction factor. In addition, the area of the peaks other than methylparaben and parahydroxybenzoate in the test solution is not larger than the peak area of methylparaben in the standard solution (0.5%). The sum of peak areas other than methylparaben in the test solution is not greater than 2 times the peak area of methylparaben in the standard solution (1.0%). Disregard the peaks having areas smaller than 0.2 times the peak area of methylparaben in the standard solution (NMT 0.1%).

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 272 nm)

Column: A stainless steel column about 4.6 mm in internal diameter and about 15 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

Mobile phase: A mixture of methanol and buffer solution (13 : 7)

Buffer solution—Dissolve 17 g of potassium dihydrogen phosphate in 2500 mL of water.

Flow rate: 1.3 mL/min (adjust the flow rate so that the retention time of methylparaben is about 2.3 minutes).

System suitability

System performance: Dissolve 5 mg each of Methylparaben and parahydroxybenzoate in the mobile phase to make exactly 100 mL. Pipet 1 mL of this solution, and add the mobile phase to make exactly 10 mL. Proceed with 10 μL of this solution according to the above operating conditions; parahydroxybenzoate and methylparaben are eluted in this order with relative retention time of parahydroxybenzoate to methylparaben of 0.6 and the resolution between their peaks being NLT 2.

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak area of methylparaben is NMT 2.0%.

Residue on ignition NMT 0.10% (1 g).

Assay Weigh accurately about 50.0 mg of Methylparaben, dissolve in 2.5 mL of methanol, and add the mobile phase to make exactly 50 mL. Pipet 10 mL of this solution, add the mobile phase to make exactly 100 mL, and use this solution as the test solution. Pipet 1 mL of the test solution, and add the mobile phase to make exactly 20 mL. Pipet 1 mL of this solution, add the mobile phase to make exactly 10 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas, A_T and A_S, of methylparaben in each solution.

Amount (mg) of methylparaben ($C_8H_8O_3$)
= Amount (mg) of methylparaben RS × (A_T / A_S)

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 272 nm)

Column: A stainless steel column about 4.6 mm in internal diameter and about 15 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

Mobile phase: A mixture of methanol and buffer solution (13 : 7)

Buffer solution—Dissolve 17 g of potassium dihydrogen phosphate in 2500 mL of water.

Flow rate: 1.3 mL/min (adjust the flow rate so that the retention time of methylparaben is about 2.3 minutes).

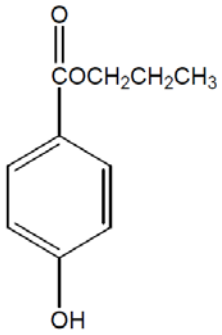
System suitability

System performance: Dissolve 5 mg each of Methylparaben and parahydroxybenzoate in the mobile phase to make exactly 100 mL. Pipet 1 mL of this solution, and add the mobile phase to make exactly 10 mL. Proceed with 10 μL of this solution according to the above operating conditions; parahydroxybenzoate and methylparaben are eluted in this order with relative retention time of parahydroxybenzoate to methylparaben of 0.6 and the resolution between their peaks being NLT 2.

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak area of methylparaben is NMT 0.85%.

Packaging and storage Preserve in well-closed containers.

Propylparaben
파라옥시벤조산프로필



Propyl Parahydroxybenzoate $C_{10}H_{12}O_3$: 180.20
Propyl 4-hydroxybenzoate [*94-13-3*]

Propylparaben contains NLT 98.0% and NMT 102.0% of propylparaben ($C_{10}H_{12}O_3$).

Description Propylparaben occurs as a colorless crystal or white crystalline powder.
It is freely soluble in methanol, ethanol (95) or acetone and very slightly soluble in water.

Identification Determine the absorption spectra of Propylparaben and propylparaben RS as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

Melting point Between 96 °C and 99 °C.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Propylparaben in 10 mL of ethanol (95); the solution is clear and the color is not more intense than that of the following control solution.

Control solution—To 5.0 mL of cobalt chloride colorimetric stock solution, 12.0 mL of ferric chloride colorimetric stock solution and 2.0 mL of copper(II) sulfate pentahydrate colorimetric stock solution, add diluted hydrochloric acid (1 in 10) to make 1000 mL.

(2) ***Acidity***—Dissolve 0.20 g of Propylparaben in 3 mL of ethanol (95), and add 5 mL of freshly boiled and cooled water and 0.1 mL of bromocresol green-sodium hydroxide-ethanol TS. Then, add 0.1 mol/L sodium hydroxide solution until the solution exhibits a blue color: the amount consumed is NMT 0.1 mL.

(3) ***Related substances***—Dissolve 50.0 mg of Propylparaben in 2.5 mL of methanol, and add the mobile phase to make exactly 50 mL. Pipet 10 mL of this solution, add the mobile phase to make exactly 100 mL, and use this solution as the test solution. Pipet 1 mL of the test solution, and add the mobile phase to make exactly 20 mL. Pipet 1 mL of this solution, add the mobile phase to make exactly 10 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions and determine the peak area of each solution by the automatic integration method: the peak area of parahydroxybenzoate, which has a relative retention time of about 0.6 to that of propylparaben from the test solution, is not larger than the peak area of propylparaben from the standard solution (0.5%). However, make corrections for the peak area of parahydroxybenzoate by multiplying by 1.4, the correction factor. The area of the peaks other than propylparaben and parahydroxybenzoate in the test solution is not larger than the peak area of propylparaben in the standard solution (0.5%). The total area of peaks other than the peak of propylparaben is not larger than 2 times the peak area of propylparaben in the standard solution (1.0%). Disregard the peaks having areas smaller than 0.2 times the peak area of propylparaben in the standard solution (NMT 0.1%).

Operating conditions
Detector: An ultraviolet absorption photometer (wavelength: 272 nm)
Column: A stainless steel column about 4.6 mm in internal diameter and about 15 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter).
Mobile phase: A mixture of methanol and buffer solution (13 : 7)

Buffer solution—Dissolve 17 g of potassium dihydrogen phosphate in 2500 mL of water.

Flow rate: 1.3 mL/min (adjust the flow rate so that the retention time of propylparaben is about 4.5 minutes).
System suitability
Confirmation of detection: Take exactly 2 mL of the standard solution, and add the mobile phase to make exactly 10 mL. Perform the test as directed under the Liquid Chromatography; the area of this solution is 14% to 26% of the area of the standard solution.
System performance: Dissolve 5 mg each of Propylparaben, ethyl parahydroxybenzoate and parahydroxybenzoate in the mobile phase to make exactly 100 mL. Pipet 1 mL of this solution, and add the mobile phase to make exactly 10 mL. Proceed with 10 μL of this solution according to the above conditions; the relative retention time of parahydroxybenzoate and ethylparaben to propylparaben is about 0.3 and 0.7, respectively, and the resolution between the peaks of ethylparaben and propylparaben is NLT 3.0.
System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak areas of propylparaben is NMT 2.0%.

Residue on ignition NMT 0.10% (1 g).

Assay Weigh accurately about 50 mg of Propylparaben, dissolve in 2.5 mL of methanol and add the mobile phase make exactly 50 mL. Pipet 10 mL of this solution, add the mobile phase to make exactly 100 mL, and use this solution as the test solution. Perform the test with 10 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas, A_T and A_S, of propylparaben from each solution.

$$\text{Amount (mg) of propylparaben } (C_{10}H_{12}O_3) = \text{Amount (mg) of propylparaben RS} \times (A_T / A_S)$$

Operating conditions
Detector: An ultraviolet absorption photometer (wavelength: 272 nm)
Column: A stainless steel column about 4.6 mm in internal

diameter and about 15 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

Mobile phase: A mixture of methanol and buffer solution (13 : 7)

Buffer solution—Dissolve 17 g of potassium dihydrogen phosphate in 2500 mL of water.

Flow rate: 1.3 mL/min (adjust the flow rate so that the retention time of propylparaben is about 4.5 minutes).

System suitability

System performance: Dissolve 5 mg each of propylparaben, ethylparaben and parahydroxybenzoate in the mobile phase to make exactly 100 mL. Pipet 1 mL of this solution, and add the mobile phase to make exactly 10 mL. Proceed with 10 μL of this solution according to the above conditions; parahydroxybenzoate, ethylparaben and propylparaben are eluted in this order and the relative retention times of parahydroxybenzoate and ethylparaben to propylparaben are about 0.3 and 0.7, respectively, and the resolution between the peaks of ethylparaben and propylparaben is NLT 3.0.

System repeatability: Repeat the test 6 times with 10 μL each of the standard solution according to the above conditions; the relative standard deviation of the peak areas of propylparaben is NMT 0.85%.

Packaging and storage Preserve in well-closed containers.

Paraffin
파라핀

Paraffin is a mixture of solid hydrocarbons obtained from petroleum.

Description Paraffin is colorless or white, slightly clear, crystalline mass, and is odorless and tasteless.
It is sparingly soluble in ether and practically insoluble in water, ethanol or anhydrous ethanol.
Specific gravity d_{20}^{20}: About 0.92 (proceed as directed in the Specific gravity (2) under the Fats and Fatty Oils)

Identification (1) Heat Paraffin strongly in a porcelain crucible and ignite: it burns with a bright flame and the odor of paraffin vapor is perceptible.

(2) Heat 0.5 g of Paraffin with 0.5 g of sulfur while shaking carefully: the odor of hydrogen sulfide is perceptible.

Melting point Between 50 ℃ and 75 ℃ (Method 2)

Purity (1) ***Acidity or alkalinity***—Boil 10.0 g of Paraffin with 10 mL of hot water and 1 drop of phenolphthalein TS on a water bath for 5 minutes and shake vigorously to mix; no red color develops. Add 0.20 mL of 0.02mol/L sodium hydroxide solution to this solution and shake to mix; red color develops.

(2) ***Heavy metals***—Ignite 2.0 g of Paraffin in a crucible, heat slowly until charred, then at 450 °C to 550 °C to ash. Cool, add 2 mL of hydrochloric acid and evaporate on a water bath to dryness. To the residue, add 2 mL of dilute acetic acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution by adding 2 mL of dilute acetic acid and water to 2.0 mL of lead standard solution to make 50 mL (NMT 10 ppm).

(3) ***Arsenic***—Prepare the test solution with 1.0 g of Paraffin according to Method 3 and perform the test (NMT 2 ppm).

(4) ***Sulfur compounds***—To 4.0 g of Paraffin, add 2 mL of anhydrous ethanol, further add 2 drops of a clear saturated solution of lead monoxide in a solution of sodium hydroxide (1 in 5) and heat for 10 minutes at 70 °C with occasional shaking: no dark brown color develops in the water layer.

(5) ***Polycyclic aromatic hydrocarbons***—Weigh accurately 0.50 g of Paraffin, transfer to a stoppered 125-mL separatory funnel, add 25 mL of n-heptane, and shake to mix well. Add 5.0 mL of dimethylsulfoxide, shake vigorously for 1 minute and allow to stand until two layers are formed. Transfer the lower layer to a second separatory funnel, add 2 mL of n-heptane, shake vigorously to mix, and allow to stand until two layers are formed. Separate the lower layer and use as the test solution. Determine the absorbance between 265 nm and 420 nm as directed under the Ultraviolet-visible Spectroscopy, using the clear lower liquid prepared by shaking vigorously for 1 minute 25 mL of n-heptane and 5.0 mL of dimethylsulfoxide as a reference solution. Separately, dissolve naphthalene RS in dimethylsulfoxide to obtain a solution containing 7.0 mg per 1000 mL, and use this solution as the standard solution. Determine the absorbance at 278 nm as directed under the Ultraviolet-visible Spectroscopy, using dimethylsulfoxide as the blank. The absorbance of the test solution between 265 nm and 420 nm is NMT one-third of the absorbance of the standard solution at 278 nm.

(6) ***Readily carbonizable substances***—Melt 5.0 g of Paraffin placed in a Nessler tube at a temperature near the melting point. Add 5 mL of sulfuric acid for the Readily carbonizable substances, warm at 70 °C for 5 minutes on a water bath, and then remove the tube from the water bath. Next, immediately shake the tube vigorously and vertically for 3 seconds and warm for 1 minutes on a water bath at 70 °C. Repeat this procedure 5 times: the color of the sulfuric acid layer is not more intense than that of the following control solution.

Control solution—Add 3.0 mL of ferric chloride colorimetric stock solution, 1.5 mL of cobalt chloride colorimetric stock solution, 0.50 mL of copper(II) sulfate pentahydrate colorimetric stock solution, and 5 mL of liquid paraffin and shake vigorously to mix.

Packaging and storage Preserve in well-closed containers.

Liquid Paraffin
유동파라핀

Liquid Paraffin is a mixture of liquid hydrocarbons obtained from petrolatum. Tocopherol of a suitable form may be added at a concentration not exceeding 0.001% as a stabilizer.

Description Liquid Paraffin is colorless, clear, oily liquid, almost free from fluorescence, and is odorless and tasteless.
It is freely soluble in ether, very slightly soluble in anhydrous ethanol and practically insoluble in water or ethanol.
Boiling point: NLT 300 °C.

Identification Perform the test as directed under the Identification of Paraffin.

Specific gravity d_{20}^{20}: Between 0.860 and 0.890

Viscosity NLT 37 mm^2/s (Method 1, 37.8 ℃)

Purity (1) ***Odor***—Transfer Liquid Paraffin to a small beaker and heat on a water bath: a foreign odor is not perceptible.

(2) ***Acidity or alkalinity***—Shake vigorously 10 mL of Liquid Paraffin with 10 mL of hot water and 1 drop of phenolphthalein TS: no red color develops. Add 0.20 mL of 0.02 mol/L sodium hydroxide solution to this solution and shake to mix; red color develops.

(3) ***Heavy metals—***Perform the test as directed in Purity (2) under Paraffin.

(4) ***Lead—***Weigh accurately 5.0 g of Liquid Paraffin and transfer to a platinum crucible. Dry, carbonize and incinerate at 450 °C to 550 °C. If incineration is not achieved, cool first and moisten with 2 to 5 mL of nitric acid (1 in 2), 50% magnesium nitrate solution or aluminum nitrate-calcium nitrate solution (a solution prepared by dissolving 40 g of aluminum nitrate and 20 g of calcium nitrate in 100 mL of water) as an incineration agent, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 to 5 mL of nitric acid (1 in 2) and incinerate. When incineration is complete, moisten the residue with water, add 2 to 4 mL of hydrochloric acid and evaporate to dryness. Add 0.5 mol/L nitric acid, dissolve by warming, and filter any insoluble matter with filter paper. Unless otherwise specified, add 0.5 mol/L nitric acid to make 25 mL and use this solution as the test solution. Separately, proceed with 1.0 mL of lead standard solution in a platinum crucible in the same manner as the test solution, and use this solution as the standard solution. Perform the test with the test solution and the standard solutions as directed under Atomic Absorption Spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 1.0 ppm).

Gas: Acetylene or hydrogen- Air.
Lamp: A lead hollow-cathode lamp
Wavelength: 283.3 nm

(5) ***Arsenic***—Prepare the test solution with 1.0 g of Liquid Paraffin according to Method 3 and perform the test. Add 10 mL of a solution of magnesium nitrate in ethanol (1 in 50), add 1.5 mL of strong hydrogen peroxide water and ignite to burn. (NMT 2 ppm).

(6) ***Solid paraffin***—Transfer 50 mL of Liquid Paraffin, previously dried at 105 °C for 2 hours, to a Nessler tube and cool in iced water for 4 hours: the turbidity produced, if any, is not more intense than that of the following control solution.

Control solution—To 1.5 mL of 0.01 mol/L hydrochloric acid, add 6 mL of dilute nitric acid and water to make 50 mL, add 1 mL of silver nitrate TS, and allow to stand for 5 minutes.

(7) ***Sulfur compounds***—To 4.0 mL of Liquid Paraffin, add 2 mL of anhydrous ethanol, further add 2 drops of a clear saturated solution of lead monoxide in a solution of sodium hydroxide (1 in 5) and heat for 10 minutes at 70 °C with occasional shaking: the solution exhibits no dark brown color.

(8) ***Polycyclic aromatic hydrocarbons***—Take 25 mL of Liquid Paraffin into a 25-mL volumetric cylinder, transfer to a 100-mL separatory funnel and wash out the cylinder with 25 mL of n-hexane. Combine the washings in the separatory funnel, and shake to mix well. Add 5.0 mL of dimethylsulfoxide, shake vigorously for 2 minutes to mix well and allow to stand for 15 minutes. Transfer the lower layer to a second separatory funnel, add 2 mL of n-hexane, shake vigorously for 2 minutes to mix well, and allow to stand for w minutes. Transfer the lower layer to a 10-mL stoppered centrifuge tube, centrifuge at a rate between 2500 and 3000 revolutions per minute for about 10 minutes, and use the clear liquid thus obtained as the test solution. Transfer 25 mL of n-hexane to another 50-mL separatory funnel, shake vigorously for 2 minutes with 5.0 mL of dimethylsulfoxide, and allow to stand for 2 minutes. Transfer the lower layer to a 10-mL stoppered centrifuge tube, centrifuge at a rate between 2500 and 3000 revolutions per minute for about 10 minutes and use the clear liquid thus obtained as a reference solution to immediately perform the test as directed under the Ultraviolet-visible Spectroscopy; the absorbance of the test solution at wavelengths between 260 and 350 nm is NMT 0.10.

(9) ***Readily carbonizable substances***—Transfer 5 mL of Liquid Paraffin to a Nessler tube and add 5 mL of sulfuric acid for Readily carbonizable substances. After heating on a water bath for 2 minutes, remove the tube from the water bath and immediately shake vigorously and vertically for 5 seconds. Repeat this procedure four times: the liquid paraffin layer remains unchanged in color. Also, the color of the sulfuric acid layer is not more intense than the following control solution.

Control solution—Add 3.0 mL of ferric chloride colorimetric stock solution, 1.5 mL of cobalt chloride colorimetric stock solution and 0.50 mL of copper(II) sulfate pentahydrate colorimetric stock solution, and shake to mix well.

Packaging and storage Preserve in tight containers.

Light Liquid Paraffin
경질유동파라핀

Light Liquid Paraffin is a mixture of liquid hydrocarbons obtained from petrolatum. Tocopherol of a suitable form may be added at a concentration not exceeding 0.001% as a stabilizer.

Description Light Liquid Paraffin is colorless, clear, oily liquid, almost free from fluorescence, and is odorless and tasteless. It is freely soluble in ether, and practically insoluble in water or ethanol.
Boiling point: NLT 300 °C.

Identification Perform the test as directed under the Identification of Paraffin.

Specific gravity d_{20}^{20}: Between 0.830 and 0.870.

Viscosity Less than 37 mm^2/s (Method 1, 37.8 °C).

Purity (1) ***Odor, Acidity or alkalinity, Solid paraffin, Sulfur compounds, Polycyclic aromatic hydrocarbons and Readily carbonizable substances***—Proceed as directed in the Purity (1), (2), (5), (6), (7) and (8) under Liquid Paraffin.

(2) ***Heavy metals and Arsenic***—Proceed as directed in the Purity (2) and (3) under Paraffin.

Packaging and storage Preserve in tight containers.

Peanut Oil
낙화생유

Peanut Oil is a fatty oil obtained from the seeds of *Arachis*

hypogaea Linné (*Leguminosae*).

Description Peanut Oil is a pale yellow, clear oil. It is odorless or has a slight odor and has a mild taste.
It is miscible with ether or petroleum ether.
It is slightly soluble in ethanol.
Specific gravity d_{25}^{25}: Between 0.909 and 0.916.
Congealing point of fatty acid: Between 22 ℃ and 33 ℃.

Identification To 5 g of Peanut Oil, add 2.5 mL of sodium hydroxide solution (3 in 10) and 12.5 mL of ethanol, boil to saponify, then evaporate to remove ethanol, and dissolve the residue in 50 mL of warm water. Add excess dilute hydrochloric acid to separate free fatty acids. Cool this solution, take the separated fatty acids, dissolve in 75 mL of ether, and add a solution of 1 g of lead acetate dissolved in 40 mL of ethanol. Allow to stand for 18 hours, then decant the solution through the filter, transfer the precipitate to the filter with the aid of ether and filter by suction. Transfer the precipitate into a beaker, add 40 mL of dilute hydrochloric acid and 20 mL of water, heat until the oil layer becomes entirely clear, and then cool. Decant and discard the water layer. To the fatty acid, add 50 mL of diluted hydrochloric acid (1 in 100), boil, then allow to stand, and remove the water layer. Repeat this procedure once using 50 mL of diluted hydrochloric acid (1 in 50), then take 0.1 g of fatty acid, dissolve in 10 mL of ethanol, and congeal the fatty acid when the solution does not exhibit dark color after adding 2 drops of sodium sulfide TS. Place it between the filter papers and press to remove moisture, then add 25 mL of diluted ethanol (9 in 10), slightly warm to dissolve, and cool to 15 ℃ to crystallize the fatty acids. Then, collect the crystallized fatty acid and wash with 20 mL of diluted ethanol (9 in 10). Repeat this procedure once using 25 mL and 20 mL of diluted ethanol (9 in 10), and then dry in a desiccator (phosphorus pentoxide, in vacuum) for 4 hours; the melting point is 73 ℃ to 76 ℃.

Saponification value Between 188 and 196.

Unsaponifiable matter NMT 1.5%.

Acid value NMT 0.2

Iodine value Between 84 and 103.

Packaging and storage Preserve in tight containers.

White Petrolatum
백색바셀린

White Petrolatum is purified by decolorizing a mixture of hydrocarbons obtained from petroleum.

White Petrolatum may contain suitable stabilizers. To add a stabilizer, specify its name and quantity.

White Petrolatum is not suitable for oral use.

Description White Petrolatum occurs as a white to pale yellow, homogeneous ointment-like substance. It is odorless and tasteless.
It is practically insoluble in water, ethanol (95) or ethanol (99.5). It is freely soluble in benzene, carbon disulfide or chloroform and soluble in ether, hexane and volatile and non-volatile oils. It is practically insoluble in cold or hot ethanol (95), cold ethanol (99.5) and water.
Warm White Petrolatum; it becomes a clear liquid.

Specific gravity d_{60}^{60}: Between 0.815 and 0.880.

Melting point Between 143 ℃ and 60 ℃ (Method 3).

Consistency ***Apparatus***—Use a penetrometer equipped with a 150-g metal cone and a detachable steel needle. The sharp end of the needle has a diameter of 0.381 ± 0.025 mm, the wide side has a diameter of 8.38 ± 0.05 mm, the height is 14.94 ± 0.05 mm, and the interior angle of the cone is 30°. The angle of the cone excluding the needle is 90°, the height is 28 mm, and the diameter of the wide side is NMT 65 mm. The sample container has a diameter of 100 ± 6 mm, a height of NLT 65 mm, a flat metal cylindrical shape, and a thickness of the container wall is NLT 1.6 mm.

Procedure—Store White Petrolatum and the sample container at a temperature of 82 ± 2.5 ℃. Then, pour White Petrolatum into the sample container until the surface of White Petrolatum is within 6 mm of the upper edge of the sample container, and then cool to 25 ± 2.5 ℃ for NLT 16 hours. Place it on a water bath at 25 ± 0.5 ℃ two hours before testing. Place a penetrometer in the water bath to adjust to 25 ± 0.5 ℃ when the room temperature is below 23.5 ℃ or exceeds 26.5 ℃. Place the sample container at the measuring position of the penetrometer, lower the needle from 25 to 38 mm above the surface of White Petrolatum until it touches the surface of White Petrolatum, set the zero point, and drop it. After dropping, measure the moving distance of the needle that entered the sample for 5 seconds. Test 3 times by changing the test location where the needle of White Petrolatum was dropped. Use a separate sample container with White Petrolatum for each test with the moving distance of the needle exceeding 20 mm. Calculate the average of the needle displacement, and perform a total of 10 tests when each result is different from the average by NLT 3%.

Determination The average of needle displacement is NLT 10.0 mm and NMT 30.0 mm.

Purity (1) ***Color***—Warm 10 g of White Petrolatum to dissolve, take 5 mL into a test tube, and maintain in a liquid state; the color of the solution is not more intense than that of the following control solution. Use a white background to compare the color of the solution from the side using reflected light.

Control solution—Add 3.4 mL of water to 1.6 mL of the colorimetric stock solution of ferric chloride.

(2) ***Acidity or alkalinity***—Add 100 mL of boiled water to 35.0 g of White Petrolatum, shake vigorously for 5 minutes to mix, collect the aqueous layer, and operate the vaseline layer twice in the same manner with 50 mL of boiled water. Combine the aqueous layers, add 1 drop of phenolphthalein TS, and boil; the resulting solution does not exhibit a red color. Add again 2 drops of methyl orange TS; the resulting solution does not exhibit a red color.

(3) ***Organic acids***—Weigh 20.0 g of White Petrolatum, and add 100 mL of dilute ethanol containing 1 drop of phenolphthalein TS, in which 0.01 mol/L sodium hydroxide solution is added until a pale red color appears. Then, boil the mixture for 10 minutes with a reflux condenser, add 2 to 3 drops of phenolphthalein TS, shake vigorously to mix, and add 0.40 mL of 0.1 mol/L sodium hydroxide VS dropwise; the resulting solution exhibits a red color.

(4) ***Fat or resin***—Add 50 mL of sodium hydroxide solution

(1 in 5) to 10.0 g of White Petrolatum, boil for 30 minutes with a reflux condenser, cool, and collect the aqueous layer. Filter, if necessary, and add 200 mL of dilute sulfuric acid; no oily substance or precipitate forms.

Residue on ignition NMT 0.05% (2 g).

Packaging and storage Preserve in well-closed containers.

Yellow Petrolatum
황색바셀린

Yellow Petrolatum is obtained by purifying a mixture of hydrocarbons obtained from petroleum.

Yellow Petrolatum may contain suitable stabilizers. To add a stabilizer, specify its name and quantity.

Yellow Petrolatum is not suitable for oral use.

Description Yellow Petrolatum occurs as a yellow to bright brown, homogeneous ointment-like substance. It is odorless and tasteless.
It is slightly soluble in ethanol (95) and practically insoluble in water.
It is freely soluble in benzene, carbon disulfide, chloroform or turpentine oil and soluble in ether, hexane and volatile and non-volatile oils.
It is practically insoluble in cold or hot ethanol (95), cold ethanol (99.5) and water.
Warm Yellow Petrolatum; the resulting solution turns to a clear, yellow liquid with almost no fluorescence.

Specific gravity d_{60}^{60}: Between 0.815 and 0.880

Melting point Between 38 ℃ and 60 ℃ (Method 3).

Consistency ***Apparatus***—Use a penetrometer equipped with a 150-g metal cone and a detachable steel needle. The sharp end of the needle has a diameter of 0.381 ± 0.025 mm, the wide side has a diameter of 8.38 ± 0.05 mm, the height is 14.94 ± 0.05 mm, and the interior angle of the cone is 30°. The angle of the cone excluding the needle is 90°, the height is 28 mm, and the diameter of the wide side is NMT 65 mm. The sample container has a diameter of 100 ± 6 mm, a height of NLT 65 mm, a flat metal cylindrical shape, and a thickness of the container wall is NLT 1.6 mm.

Procedure Store Yellow Petrolatum and the sample container at a temperature of 82 ± 2.5 ℃. Then, pour Yellow Petrolatum into the sample container until the surface of Yellow Petrolatum is within 6 mm of the upper edge of the sample container, and then cool to 25 ± 2.5 ℃ for NLT 16 hours. Place it on a water bath at 25 ± 0.5 ℃ two hours before testing. Place a penetrometer in the water bath to adjust to 25 ± 0.5 ℃ when the room temperature is below 23.5 ℃ or exceeds 26.5 ℃. Place the sample container at the measuring position of the penetrometer, lower the needle from 25 to 38 mm above the surface of Yellow Petrolatum until it touches the surface of Yellow Petrolatum, set the zero point, and drop it. After dropping, measure the moving distance of the needle that entered the sample for 5 seconds. Test 3 times by changing the test location where the needle of Yellow Petrolatum was dropped. Use a separate sample container with Yellow Petrolatum for each test with the moving distance of the needle exceeding 20 mm. Calculate the average of the needle displacement, and perform a total of 10 tests when each result is different from the average by NLT 3%.

Determination The average of needle displacement is NLT 10.0 mm and NMT 30.0 mm.

Purity (1) ***Color***—Warm 10 g of Yellow Petrolatum to dissolve, take 5 mL into a test tube, and maintain in a liquid state; the color of the solution is not more intense than that of the following control solution. Use a white background for colorimetry from the side using reflected light.

Control solution—Add 1.2 mL of the colorimetric stock solution of cobalt chloride to 3.8 mL of the colorimetric stock solution of ferric chloride.

(2) ***Acidity or alkalinity***—Add 100 mL of boiled water to 35.0 g of Yellow Petrolatum, shake vigorously for 5 minutes to mix, collect the aqueous layer, and operate the vaseline layer twice in the same manner with 50 mL of boiled water. Combine the aqueous layers, add 1 drop of phenolphthalein TS, and boil; the resulting solution does not exhibit a red color. Add again 2 drops of methyl orange TS; the resulting solution does not exhibit a red color.

(3) ***Organic acids***—Weigh 20.0 g of Yellow Petrolatum, and add 100 mL of dilute ethanol containing 1 drop of phenolphthalein TS, in which 0.01 mol/L sodium hydroxide solution is added until a pale red color appears. Then, boil the mixture for 10 minutes with a reflux condenser, add 2 to 3 drops of phenolphthalein TS, shake vigorously to mix, and add 0.40 mL of 0.1 mol/L sodium hydroxide VS dropwise; the resulting solution exhibits a red color.

(4) ***Fat or resin***—Add 50 mL of sodium hydroxide solution (1 in 5) to 10.0 g of Yellow Petrolatum, boil for 30 minutes with a reflux condenser, cool, and collect the aqueous layer. Filter, if necessary, and add 200 mL of diluted sulfuric acid; no oily substance or precipitate forms.

Residue on ignition Not more 0.1% (2 g).

Packaging and storage Preserve in tight containers.

Petroleum Benzin
석유벤진

Petroleum Benzin is a mixture of low boiling point hydrocarbons from petroleum.

Description Petroleum Benzin occurs as a clear and colorless volatile liquid with no fluorescence and a characteristic odor.
It is miscible with anhydrous ethanol or ether.
It is practically insoluble in water.
It is very flammable.

Specific gravity d_{20}^{20}: Between 0.65 and 0.71

Purity (1) ***Acid***—To 10 mL of Petroleum Benzin, add 5 mL of water, shake vigorously for 2 minutes to mix, and allow to stand. The separated aqueous layer does not change moistened blue litmus paper to red.

(2) ***Sulfur compounds and reducing substances***—Add 2.5 mL of ammonia-ethanol TS and 2 to 3 drops of silver nitrate TS to 10 mL of Petroleum Benzin, and warm the mixture at about

50 °C for 5 minutes, protected from light; the solution does not exhibit a brown color.

(3) ***Fatty oil and sulfur compounds***—Put an odorless filter paper spread on a previously warmed glass plate, and drop 10 mL of Petroleum Benzin dropwise in small volumes to evaporate; no spot or no foreign odor is perceptible.

(4) ***Benzene***—Warm 5 drops of Petroleum Benzin with 2 mL of sulfuric acid and 0.5 mL of nitric acid for about 10 minutes, allow to stand for 30 minutes, transfer the mixture to a porcelain dish, and dilute with water; no odor of nitrobenzene is perceptible.

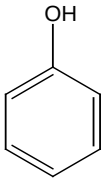
(5) ***Residue on evaporation***—Evaporate 140 mL of Petroleum Benzin on a water bath to dryness, and dry the residue at 105 °C to constant weight; the amount is NMT 1 mg.

(6) ***Readily carbonizable substances***—Transfer 5 mL of Petroleum Benzin into a Nessler tube, add 5 mL of sulfuric acid for readily carbonizable substances test, shake vigorously for 5 minutes, and allow to stand; the sulfuric acid layer is not more intense than Matching fluids A.

Distilling range Between 50 °C and 80 °C, NLT 90 vol%.

Packaging add storage Preserve in tight containers below 30 °C, protected from fire.

Phenol
페놀



Carbolic acid C_6H_6O : 94.11
Phenol [*108-95-2*]

Phenol contains NLT 98.0% and NMT 101.0% of phenol (C_6H_6O).

Description Phenol occurs as a colorless to slightly red crystal or crystalline mass, and has a characteristic odor.
Phenol is very soluble in ethanol or ether and soluble in water.
To 10 g of Phenol, add 1 mL of water; it is liquefied.
It changes color gradually through red to dark red by light or air.
It cauterizes the skin, turning it white.
Congealing point: About 40 °C.

Identification (1) To 10 mL of an aqueous solution of Phenol (1 in 100), add 1 drop of ferric chloride TS; the solution exhibits a blueish purple color.

(2) To 5 mL of an aqueous solution of Phenol (1 in 10000), add bromine TS dropwise: a white precipitate is produced, which at first dissolves with shaking, but does not dissolve after adding the excess of the bromine TS.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Phenol in 15 mL of water: the solution is clear and neutral or only weakly acidic. Add 2 drops of methyl orange TS: the solution exhibits no red color.

(2) ***Residue on evaporation***—Weigh accurately about 5 g of Phenol, evaporate on a water bath and dry the residue at 105 °C for 1 hour: the amount of the residue is NMT 0.05%.

Assay Weigh accurately about 1.5 g of Phenol and dissolve in water to make 1000 mL exactly. Pipet 25 mL of this solution and place into an iodine flask, add exactly 30 mL of 0.05 mol/L bromine solution, then 5 mL of hydrochloric acid and stopper the flask immediately. Shake the flask occasionally for 30 minutes, and allow to stand for 15 minutes. Then, add 7 mL of potassium iodide TS, stopper the flask immediately and shake well to mix. Add 1 mL of chloroform, stopper the flask and shake thoroughly to mix. Titrate the liberated iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 1 mL of starch TS). Perform a blank test in the same manner.

Each mL of 0.05 mol/L bromine VS
= 1.5685 mg of C_6H_6O

Packaging and storage Preserve in light-resistant, tight containers.

Phenol for Disinfection
소독용 페놀

Carbolic acid for disinfection

Phenol for Disinfection contains NLT 95.0% and NMT 101.0% of phenol (C_6H_6O : 94.11).

Description Phenol for Disinfection occurs as a colorless to slightly red crystal or crystalline mass, or liquid containing these crystals or masses and has a characteristic odor.
It is very soluble in ethanol or ether and freely soluble in water.
To 10 g of Phenol for Disinfection, add 1 mL of water; it is liquefied.
It cauterizes the skin, turning it white.
Congealing point: About 30 °C.

Identification (1) To 10 mL of an aqueous solution of Phenol for Disinfection (1 in 100), add 1 drop of ferric chloride TS; the solution exhibits a blueish purple color.

(2) To 5 mL of an aqueous solution of Phenol for Disinfection (1 in 10000), add bromine TS dropwise: a white precipitate is produced, which at first dissolves with shaking, but does not dissolve after adding the excess of the bromine TS.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Phenol for Disinfection in 15 mL of water; the solution is clear.

(2) ***Residue on evaporation***—Weigh accurately about 5 g of Phenol for Disinfection, evaporate on a water bath and dry the residue at 105 °C for 1 hour: the amount of the residue is NMT 0.10%.

Assay Weigh accurately about 1 g of Phenol for Disinfection and dissolve in water to make 1000 mL exactly. Pipet 25 mL of this solution and place into an iodine flask, add exactly 30 mL of 0.05 mol/L bromine solution, then 5 mL of hydrochloric acid and stopper the flask immediately. Shake the flask for 30 minutes, and allow to stand for 15 minutes. Then, add 7 mL of potassium iodide TS, stopper the flask immediately and shake well to mix. Titrate the liberated iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 1 mL of starch TS). Perform a blank test in the same manner.

Each mL of 0.05 mol/L bromine VS

= 1.5685 mg of C_6H_6O

Packaging and storage Preserve in light-resistant, tight containers.

Liquefied Phenol
액상 페놀

Liquefied carbolic acid

Liquefied Phenol is Phenol maintained in a liquid condition by adding Water or Purified Water equivalent to 10%.

Liquefied Phenol contains NLT 88.0% of phenol (C_6H_6O : 94.11).

Description Liquefied Phenol is colorless or slightly red liquid, and has a characteristic odor.
It is miscible with ethanol, ether or glycerin.
A mixture of equal volumes of Liquefied Phenol and glycerin is miscible with water.
It changes color gradually to dark red by light or air.
It cauterizes the skin, turning it white.

Specific gravity d_{20}^{20}: About 1.065.

Identification Perform the test as directed under the Identification of Phenol.

Boiling Point NMT 182 °C.

Purity Perform the test as directed under the Purity of Phenol.

Assay Weigh accurately about 1.7 g of Liquefied Phenol and perform the test as directed under the Assay of Phenol.

Each mL of 0.05 mol/L bromine VS
= 1.5685 mg of C_6H_6O

Packaging and storage Preserve in light-resistant, tight containers.

Polyethylene Glycol 400
폴리에틸렌글리콜 400

Macrogol 400

Polyethylene Glycol 400 is a polymer of ethylene oxide and water, represented by the formula $HOCH_2(CH_2OCH_2)nCH_2OH$, in which the value of n ranges from 7 to 9.

Description Polyethylene Glycol 400 occurs as a clear, colorless and viscous liquid, and has a slight, characteristic odor.
It is miscible with water, methanol, ethanol, or pyridine.
It is soluble in ether.
It is slightly hygroscopic.
Congealing point: Between 4 °C and 8 °C

Specific gravity d_{20}^{20}: Between 1.110 and 1.140

Identification Dissolve 50 mg of Polyethylene Glycol 400 in 5 mL of dilute hydrochloric acid, add 1 mL of barium chloride TS, shake to mix and filter, if necessary. To the filtrate, add 1 mL of a solution of phosphomolybdic acid (1 in 10): a yellowish green precipitate is formed.

pH Dissolve 1.0 g of Polyethylene Glycol 400 in 20 mL of water; the pH of this solution is 4.0 to 7.0.

Purity (1) ***Acid***—Dissolve 5.0 g of Polyethylene Glycol 400 in 20 mL of neutralized ethanol, and add 2 drops of phenolphthalein TS and 0.20 mL of 0.1 mol/L sodium hydroxide solution; the resulting solution exhibits a red color.

(2) ***Heavy metals***—Proceed with 1.0 g of Polyethylene Glycol 400 according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 20 ppm).

(3) ***Ethylene glycol and diethylene glycol***—Dissolve 4.0 g of Polyethylene Glycol 400 in water to make exactly 10 mL, and use this solution as the test solution. Separately, weigh accurately about 50 mg each ethylene glycol RS and diethylene glycol RS, dissolve in water to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 2 μL each of the test solution and standard solution as directed under the Liquid Chromatography according to the following conditions. Determine the peak heights, H_{Ta} and H_{Sa}, of ethylene glycol in each solution, respectively, and the peak heights, H_{Tb} and H_{Sb}, of diethylene glycol in each solution, respectively, and calculate the amount of ethylene glycol and diethylene glycol: the sum of the contents of ethylene glycol and diethylene glycol is NMT 0.25%.

$$\text{Amount (mg) of ethylene glycol} = \text{Amount (mg) of ethylene glycol RS} \times \frac{H_{Ta}}{H_{Sa}} \times \frac{1}{10}$$

$$\text{Amount (mg) of diethylene glycol} = \text{Amount (mg) of diethylene glycol RS} \times \frac{H_{Tb}}{H_{Sb}} \times \frac{1}{10}$$

Operating conditions

Detector: A flame ionization detector

Column: A column, about 3 mm in internal diameter and about 1.5 m in length, packed with diatomaceous earth for gas chromatography (150 μm to 180 μm in particle diameter), coated with D-Sorbitol for gas chromatography at a ratio of 12%.

Column temperature: A constant temperature of about 165 °C.

Carrier gas: Nitrogen or helium

Flow rate: Adjust the flow rate so that the retention time of diethylene glycol is about 3 minutes.

System suitability

System performance: Proceed with 2 μL of the standard solution according to the above conditions; ethylene glycol and diethylene glycol are eluted in this order with each peak showing a complete separation.

Detection sensitivity: Adjust the sensitivity so that the peak height of diethylene glycol from 2 μL of the standard solution is about 80% of the full scale.

(4) ***Ethylene oxide and dioxane***—Weigh accurately 1.00 g (M_T) of Polyethylene Glycol 400, transfer to a 10-mL vial, add 1.0 mL of water and mix well. Allow to stand at 70 °C for 45 minutes and use this solution as the test solution. Weigh accurately 1.00 g of dioxane RS, add water to make 100 mL, pipet 5.0 mL of this solution, and add water to make 100 mL. Pipet 10.0 mL of this solution, add water to make 50 mL, and use this solution as the standard dioxane solution. Dilute 50 mg/mL ethylene oxide solution, prepared by dissolving ethylene oxide in methylene chloride, with water to make a solution containing 50 μg of

ethylene oxide per mL (this solution is stable for 3 months if sealed with a Teflon-coated silicon membrane and a crimped cap and stored at -20 °C). Transfer 10.0 mL of this solution to a flask containing 30 mL of water, mix well and add water to make 50 mL. Pipet 10.0 mL of this solution, add water to make 50 mL, and use this solution as the standard ethylene oxide solution. This preparation is prepared just before use. Separately, put 1.00 g (M_R) of Polyethylene Glycol 400 into an identical 10-mL vial, add 0.5 mL of the standard ethylene oxide solution and 0.5 mL of the standard dioxane solution, mix well, allow to stand at 70 °C for 45 minutes and use this solution as the standard solution (1). Put 0.5 mL of the standard ethylene oxide solution into a 10 mL-vial, add 0.1 mL of a freshly prepared 10 mg/L standard acetaldehyde solution and 0.1 mL of the standard dioxane solution, mix well, allow to stand at 70 °C for 45 minutes and use this solution as standard solution (2). Perform the test with 1 mL each of the test solution and the standard solution (1) according to the following conditions, as directed under the Gas Chromatography. Determine the peak areas of ethylene oxide and dioxane in each solution and calculate their amounts: ethylene oxide is NMT 1 ppm and dioxane is NMT 10 ppm.

$$\text{Amount (ppm) of ethylene oxide} = \frac{A_T \times C}{(A_R \times M_T) - (A_T \times M_R)}$$

A_T: Peak area of ethylene oxide in the test solution
A_R: Peak area of ethylene oxide in the standard solution (1)
M_T: Amount (g) of the sample in the test solution
M_R: Amount (g) of the sample in the standard solution (1)
C: Amount (μg) of ethylene oxide added in the standard solution (1)

$$\text{Amount (ppm) of dioxane} = \frac{D_T \times C}{(D_R \times M_T) - (D_T \times M_R)}$$

D_T: Peak area of dioxane in the test solution
D_R: Peak area of dioxane in the standard solution (1)
C: Amount (μg) of dioxane added in the standard solution (1)

Operating conditions

Detector: A flame ionization detector

Column: A glass or quartz capillary tube 0.32 mm in internal diameter and about 30 m in length, coated inside with poly(dimethyl) siloxane in 1.0-μm thickness.

Column temperature: Initially, maintain at 50 °C for 5 minutes, raise the temperature to 180 °C at the rate of 5 °C per minute, then raise the temperature to 230 °C at the rate of 30 °C per minute, and maintain the temperature for 5 minutes.

Sample injection port temperature: A constant temperature of about 150 °C

Headspace sampler temperature: 70 °C

Detector temperature: A constant temperature of about 250 °C

Carrier gas: Helium

Split ratio: About 1 : 20

System suitability

System performance: Proceed with 1.0 mL of the standard solution (2) according to the above conditions; the resolution between the peaks of acetaldehyde and ethylene oxide is NLT 2.0 and the signal-to-noise ratio of dioxane is NLT 5.

Average ***molecular weight***—Weigh 42 g of anhydrous phthalic acid and place into a 1000-mL light-resistant stoppered bottle containing 300 mL of freshly distilled pyridine, exactly measured. Shake the bottle vigorously to mix and dissolve, and allow to stand for NLT 16 hours. Pipet 25 mL of this solution into an about 200-mL stoppered pressure bottle, add about 1.5 g of Polyethylene Glycol 400, accurately weigh, stopper the bottle, wrap it with strong cloth and immerse on a water bath, previously heated at 98 ± 2 °C. At this time, the solution in the bottle is immersed completely in the water bath. Maintain the temperature of the bath at 98 ± 2 °C for 30 minutes. Remove the bottle from the water bath and allow to cool in the air to room temperature. Add exactly 50 mL of 0.5 mol/L sodium hydroxide VS and 5 drops of a solution of phenolphthalein in pyridine (1 in 100), and then titrate with 0.5 mol/L sodium hydroxide VS. However, the endpoint of the titration is when the solution exhibits a pale red color which persists for about 15 seconds. Perform a blank test in the same manner. The average molecular weight is 380 to 420.

$$\text{Average molecular weight} = \frac{Amount\ (g)\ of\ sample \times 4000}{a - b}$$

a: Volume (mL) of 0.5 mol/L sodium hydroxide VS consumed in the blank test
b: Volume (mL) of 0.5 mol/L sodium hydroxide VS consumed in the test

Water NMT 1.0% (2 g, volumetric titration, direct titration).

Residue on ignition NMT 0.10% (1 g).

Packaging and storage Preserve in tight containers.

Polyethylene Glycol 1500
폴리에틸렌글리콜 1500

Macrogol 1500

Polyethylene Glycol 1500 is a polymer of ethylene oxide and water, represented by the formula $HOCH_2(CH_2OCH_2)nCH_2OH$, a mixture containing equal amounts of lower and higher polymers in which the value of n is 5 to 6 for the lower polymer, and 28 to 36 for the higher polymer.

Description Polyethylene Glycol 1500 occurs as a white, smooth petrolatum-like solid and has a slightly characteristic odor.
It is very soluble in water, pyridine or diphenylether, freely soluble in methanol, sparingly soluble in ethanol, very slightly soluble in anhydrous ethanol and practically insoluble in ether.
Congealing point: Between 37 °C and 41 °C

Identification Dissolve 50 mg of Polyethylene Glycol 1500 in 5 mL of dilute hydrochloric acid, add 1 mL of barium chloride TS, shake to mix and filter, if necessary. To the filtrate, add 1 mL of a solution of phosphomolybdic acid (1 in 10): a yellowish green precipitate is formed.

pH Dissolve 1.0 g of Polyethylene Glycol 1500 in 20 mL of water; the pH of this solution is 4.0 to 7.0.

Purity (1) ***Clarity and color of solution***—Dissolve 5.0 g of Polyethylene Glycol 1500 in 50 mL of water; the resulting solution is clear and colorless.

(2) ***Acid***—Dissolve 5.0 g of Polyethylene Glycol 1500 in

20 mL of neutralized ethanol, and add 2 drops of phenolphthalein TS and 0.20 mL of 0.1 mol/L sodium hydroxide solution; the resulting solution exhibits a red color.

(3) ***Heavy metals***—Proceed with 1.0 g of Polyethylene Glycol 1500 according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 20 ppm).

(4) ***Ethylene glycol and diethylene glycol***—Place 50.0 g of Polyethylene Glycol 1500 in distilling flask, add 75 mL of diphenylether, warm to dissolve, if necessary, distill slowly in a vacuum of 0.13 to 0.27 kPa and collect 25 mL of the distillate in a 100-mL container with 1-mL graduation. To the distillate, add exactly 20 mL of water, shake vigorously to mix, cool in iced water, congeal the diphenylether and filter into a 25-mL volumetric flask. Wash the residue with 5 mL of ice cold water, combine the washings with the filtrate, warm to room temperature and add water to make exactly 25 mL. Transfer this solution to a stoppered flask, shake with 25.0 mL of freshly distilled acetonitrile, and use this solution as the test solution. Separately, to 62.5 mg of diethylene glycol RS, add a mixture of water and freshly distilled acetonitrile (1 : 1) to make exactly 25 mL and use this solution as the standard solution. Pipet 10.0 mL each of the test solution and the standard solution and add exactly 15.0 mL of cerium (IV) ammonium nitrate TS, respectively. Perform the test with these solutions as directed under the Ultraviolet-visible Spectroscopy within 2 to 5 minutes: the absorbance of the solution obtained from the test solution at the wavelength of maximum absorption at about 450 nm is not larger than the absorbance from the standard solution

(5) ***Ethylene oxide and dioxane***—Weigh accurately 1.00 g (M_T) of Polyethylene Glycol 1500, transfer to a 10-mL vial, add 1.0 mL of water and mix well. Allow to stand at 70 °C for 45 minutes and use this solution as the test solution. Weigh accurately 1.00 g of dioxane RS, add water to make 100 mL, pipet 5.0 mL of this solution, and add water to make 100 mL. Pipet 10.0 mL of this solution, add water to make 50 mL, and use this solution as the standard dioxane solution. Dilute 50 mg/mL ethylene oxide solution, prepared by dissolving ethylene oxide in methylene chloride, with water to make a solution containing 50 μg of ethylene oxide per mL (this solution is stable for 3 months if sealed with a Teflon-coated silicon membrane and a crimped cap and stored at -20 °C). Pipet 10.0 mL of this solution, add water to make 50 mL, and use this solution as the standard ethylene oxide solution. This preparation is prepared just before use. Separately, put 1.00 g (M_R) of Polyethylene Glycol 1500 into an identical 10-mL vial, add 0.5 mL of the standard ethylene oxide solution and 0.5 mL of the standard dioxane solution, mix well, allow to stand at 70 °C for 45 minutes, and use this solution as the standard solution (1). Put 0.5 mL of the standard ethylene oxide solution into a 10-mL vial, add 0.1 mL of a freshly prepared 10 mg/L standard acetaldehyde solution and 0.1 mL of the standard dioxane solution, mix well, allow to stand at 70 °C for 45 minutes and use this solution as the standard solution (2). Perform the test with 1 mL each of the test solution and the standard solution (1) according to the following conditions, as directed under the Gas Chromatography. Determine the peak areas of ethylene oxide and dioxane in each solution and calculate their amounts: ethylene oxide is NMT 1 ppm and dioxane is NMT 10 ppm.

$$\text{Amount (ppm) of ethylene oxide} = \frac{A_T \times C}{(A_R \times M_T) - (A_T \times M_R)}$$

A_T: Peak area of ethylene oxide in the test solution

A_R: Peak area of ethylene oxide in the standard solution (1)

M_T: Amount (g) of the sample in the test solution

M_R: Amount (g) of the sample in the standard solution (1)

C: Amount (μg) of ethylene oxide added in the standard solution (1)

$$\text{Amount (ppm) of dioxane} = \frac{D_T \times C}{(D_R \times M_T) - (D_T \times M_R)}$$

D_T: Peak area of dioxane in the test solution

D_R: Peak area of dioxane in the standard solution (1)

C: Amount (μg) of dioxane added in the standard solution (1)

Operating conditions

Detector: A flame ionization detector

Column: A glass or quartz capillary tube 0.32 mm in internal diameter and about 30 m in length, coated inside with poly(dimethyl) siloxane in 1.0-μm thickness.

Column temperature: Initially, maintain at 50 °C for 5 minutes, raise the temperature to 180 °C at the rate of 5 °C per minute, then raise the temperature to 230 °C at the rate of 30 °C per minute, and maintain the temperature for 5 minutes.

Sample injection port temperature: A constant temperature of about 150 ℃

Headspace sampler temperature: 70 ℃

Detector temperature: A constant temperature of about 250 ℃

Carrier gas: Helium

Split ratio: About 1 : 20

System suitability

System performance: Proceed with 1.0 mL of the standard solution (2) according to the above conditions; the resolution between the peaks of acetaldehyde and ethylene oxide is NLT 2.0 and the signal-to-noise ratio of dioxane is NLT 5.

Water NMT 1.0% (2 g, volumetric titration, direct titration).

Residue on ignition NMT 0.10% (1 g).

Packaging and storage Preserve in tight containers.

Polyethylene Glycol 4000
폴리에틸렌글리콜 4000

Macrogol 4000

Polyethylene Glycol 4000 is a polymer of ethylene oxide and water and is represented by the formula $HOCH_2(CH_2OCH_2)nCH_2OH$, in which the value of n ranges from 59 to 84.

Description Polyethylene Glycol 4000 occurs as a white, paraffin-like mass, flakes or powder, and has a faint, characteristic odor.

It is very soluble in water, freely soluble in methanol, ethanol or pyridine and practically insoluble in anhydrous ethanol or ether.

Congealing point: Between 53 °C and 57 °C

Identification Dissolve 50 mg of Polyethylene Glycol 4000 in 5 mL of dilute hydrochloric acid, add 1 mL of barium chloride TS, shake to mix and filter, if necessary. To the filtrate, add 1 mL of a solution of phosphomolybdic acid (1 in 10): a yellowish

green precipitate is formed.

pH Dissolve 1.0 g of Polyethylene Glycol 4000 in 20 mL of water; the pH of this solution is 4.0 to 7.5.

Purity (1) ***Clarity and color of solution***—Dissolve 5.0 g of Polyethylene Glycol 4000 in 50 mL of water; the resulting solution is clear and colorless.

(2) ***Acid***—Dissolve 5.0 g of Polyethylene Glycol 4000 in 20 mL of neutralized ethanol, and add 2 drops of phenolphthalein TS and 0.20 mL of 0.1 mol/L sodium hydroxide; the resulting solution exhibits a red color.

(3) ***Heavy metals***—Proceed with 1.0 g of Polyethylene Glycol 4000 according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 20 ppm).

(4) ***Ethylene oxide and dioxane***—Weigh accurately 1.00 g (M_T) of Polyethylene Glycol 4000, transfer to a 10-mL vial, add 1.0 mL of water and mix well. Allow to stand at 70 °C for 45 minutes and use this solution as the test solution. Weigh accurately 1.00 g of dioxane RS, add water to make 100 mL, pipet 5.0 mL of this solution, and add water to make 100 mL. Pipet 10.0 mL of this solution, add water to make 50 mL, and use this solution as the standard dioxane solution. Dilute 50 mg/mL ethylene oxide solution, prepared by dissolving ethylene oxide in methylene chloride, with water to make a solution containing 50 μg of ethylene oxide per mL (this solution is stable for 3 months if sealed with a Teflon-coated silicon membrane and a crimped cap and stored at -20 °C). Transfer 10.0 mL of this solution to a flask containing 30 mL of water, mix well and add water to make 50 mL. Pipet 10.0 mL of this solution, add water to make 50 mL, and use this solution as the standard ethylene oxide solution. This preparation is prepared just before use. Separately, put 1.00 g (M_R) of Polyethylene Glycol 4000 into an identical 10-mL vial, add 0.5 mL of the standard ethylene oxide solution and 0.5 mL of the standard dioxane solution, mix well, allow to stand at 70 °C for 45 minutes and use this solution as the standard solution (1). Put 0.5 mL of the standard ethylene oxide solution into a 10-mL vial, add 0.1 mL of a freshly prepared 10 mg/L standard acetaldehyde solution and 0.1 mL of the standard dioxane solution, mix well, allow to stand at 70 °C for 45 minutes and use this solution as standard solution (2). Perform the test with 1 mL each of the test solution and the standard solution (1) according to the following conditions, as directed under the Gas Chromatography. Determine the peak areas of ethylene oxide and dioxane in each solution and calculate their amounts: ethylene oxide is NMT 1 ppm and dioxane is NMT 10 ppm.

$$\text{Amount (ppm) of ethylene oxide} = \frac{A_T \times C}{(A_R \times M_T) - (A_T \times M_R)}$$

A_T: Peak area of ethylene oxide in the test solution
A_R: Peak area of ethylene oxide in the standard solution (1)
M_T: Amount (g) of the sample in the test solution
M_R: Amount (g) of the sample in the standard solution (1)
C: Amount (μg) of ethylene oxide added in the standard solution (1)

$$\text{Amount (ppm) of dioxane} = \frac{D_T \times C}{(D_R \times M_T) - (D_T \times M_R)}$$

D_T: Peak area of dioxane in the test solution
D_R: Peak area of dioxane in the standard solution (1)
C: Amount (μg) of dioxane added in the standard solution (1)

Operating conditions

Detector: A flame ionization detector

Column: A glass or quartz capillary tube 0.32 mm in internal diameter and about 30 m in length, coated inside with poly(dimethyl) siloxane in 1.0-μm thickness.

Column temperature: Initially, maintain at 50 °C for 5 minutes, raise the temperature to 180 °C at the rate of 5 °C per minute, then raise the temperature to 230 °C at the rate of 30 °C per minute, and maintain the temperature for 5 minutes.

Sample injection port temperature: A constant temperature of about 150 °C

Headspace sampler temperature: 70 °C

Detector temperature: A constant temperature of about 250 °C

Carrier gas: Helium

Split ratio: About 1 : 20

System suitability

System performance: Proceed with 1.0 mL of the standard solution (2) according to the above conditions; the resolution between the peaks of acetaldehyde and ethylene oxide is NLT 2.0 and the signal-to-noise ratio of dioxane is NLT 5.

Average molecular weight Weigh accurately about 12.5 g of Polyethylene Glycol 4000, transfer to an about 200-mL stoppered pressure bottle, add about 25 mL of pyridine, dissolve by warming, and allow to cool. Separately, weigh 42 g of anhydrous phthalic acid and place into a 1000-mL light-resistant stoppered bottle containing 300 mL of freshly distilled pyridine, exactly measured. Shake the bottle vigorously to mix and dissolve, and allow to stand for NLT 16 hours. Pipet 25 mL of this solution, transfer to the former stoppered pressure bottle, stopper the bottle, wrap it with strong cloth and perform the test as directed in the Average molecular weight under Polyethylene Glycol 400. However, the average molecular weight is 2600 to 3800.

Water NMT 1.0% (2 g, volumetric titration, direct titration).

Residue on ignition NMT 0.2% (1 g).

Packaging and storage Preserve in well-closed containers.

Polyethylene Glycol 6000
폴리에틸렌글리콜 6000

Macrogol 6000

Polyethylene Glycol 6000 is a polymer of ethylene oxide and water, represented by the formula $HOCH_2(CH_2OCH_2)nCH_2OH$, in which the value of n ranges from 165 to 210.

Description Polyethylene Glycol 6000 occurs as a white, paraffin-like mass, flakes or powder, and has a faint, characteristic odor.
It is very soluble in water, freely soluble in pyridine and practically insoluble in methanol, ethanol, anhydrous ethanol or ether.
Congealing point: Between 56 °C and 61 °C

Identification Dissolve 50 mg of Polyethylene Glycol 6000 in 5 mL of dilute hydrochloric acid, add 1 mL of barium chloride TS, shake to mix and filter, if necessary. To the filtrate, add 1 mL

of a solution of phosphomolybdic acid (1 in 10): a yellowish green precipitate is formed.

pH Dissolve 1.0 g of Polyethylene Glycol 6000 in 20 mL of water; the pH of this solution is between 4.5 and 7.5.

Purity (1) ***Clarity and color of solution***—Dissolve 5.0 g of Polyethylene Glycol 6000 in 50 mL of water; the resulting solution is clear and colorless.

(2) ***Acid***—Dissolve 5.0 g of Polyethylene Glycol 6000 in 20 mL of neutralized ethanol, and add 0.20 mL of 0.1 mol/L sodium hydroxide and 1 drop of phenolphthalein TS: the color of the solution is red.

(3) ***Heavy metals***—Proceed with 1.0 g of Polyethylene Glycol 6000 according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 20 ppm).

(4) ***Ethylene oxide and dioxane***—Weigh accurately 1.00 g (M_T) of Polyethylene Glycol 6000, transfer to a 10-mL vial, add 1.0 mL of water and mix well. Allow to stand at 70 °C for 45 minutes and use this solution as the test solution. Weigh accurately 1.00 g of dioxane RS, add water to make 100 mL, pipet 5.0 mL of this solution, and add water to make 100 mL. Pipet 10.0 mL of this solution, add water to make 50 mL, and use this solution as the standard dioxane solution. Dilute 50 mg/mL ethylene oxide solution, prepared by dissolving ethylene oxide in methylene chloride, with water to make a solution containing 50 μg of ethylene oxide per mL (this solution is stable for 3 months if sealed with a Teflon-coated silicon membrane and a crimped cap and stored at -20 °C). Transfer 10.0 mL of this solution to a flask containing 30 mL of water, mix well and add water to make 50 mL. Pipet 10.0 mL of this solution, add water to make 50 mL, and use this solution as the standard ethylene oxide solution. This preparation is prepared just before use. Separately, put 1.00 g (M_R) of Polyethylene Glycol 6000 into an identical 10-mL vial, add 0.5 mL of the standard ethylene oxide solution and 0.5 mL of the standard dioxane solution, mix well, allow to stand at 70 °C for 45 minutes, and use this solution as standard solution (1). Put 0.5 mL of the standard ethylene oxide solution into a 10-mL vial, add 0.1 mL of a freshly prepared 10 mg/L standard acetaldehyde solution and 0.1 mL of the standard dioxane solution, mix well, allow to stand at 70 °C for 45 minutes and use this solution as standard solution (2). Perform the test with 1 mL each of the test solution and the standard solution (1) according to the following conditions, as directed under the Gas Chromatography. Determine the peak areas of ethylene oxide and dioxane in each solution and calculate their amounts: ethylene oxide is NMT 1 ppm and dioxane is NMT 10 ppm.

$$\text{Amount (ppm) of ethylene oxide} = \frac{A_T \times C}{(A_R \times M_T) - (A_T \times M_R)}$$

A_T: Peak area of ethylene oxide in the test solution
A_R: Peak area of ethylene oxide in the standard solution (1)
M_T: Amount (g) of the sample in the test solution
M_R: Amount (g) of the sample in the standard solution (1)
C: Amount (μg) of ethylene oxide added in the standard solution (1)

$$\text{Amount (ppm) of dioxane} = \frac{D_T \times C}{(D_R \times M_T) - (D_T \times M_R)}$$

D_T: Peak area of dioxane in the test solution
D_R: Peak area of dioxane in the standard solution (1)
C: Amount (μg) of dioxane added in the standard solution (1)

Operating conditions
Detector: A flame ionization detector
Column: A glass or quartz capillary tube 0.32 mm in internal diameter and about 30 m in length, coated inside with poly(dimethyl) siloxane in 1.0-μm thickness.
Column temperature: Initially, maintain at 50 °C for 5 minutes, raise the temperature to 180 °C at the rate of 5 °C per minute, then raise the temperature to 230 °C at the rate of 30 °C per minute, and maintain the temperature for 5 minutes.
Sample injection port temperature: A constant temperature of about 150 ℃
Headspace sampler temperature: 70 ℃
Detector temperature: A constant temperature of about 250 ℃
Carrier gas: Helium
Split ratio: About 1 : 20
System suitability
System performance: Proceed with 1.0 mL of the standard solution (2) according to the above conditions; the resolution between the peaks of acetaldehyde and ethylene oxide is NLT 2.0 and the signal-to-noise ratio of dioxane is NLT 5.

Average molecular weight Weigh accurately about 12.5 g of Polyethylene Glycol 6000, transfer to an about 200-mL stoppered pressure bottle, add about 25 mL of pyridine, dissolve by warming, and allow to cool. Separately, weigh 42 g of anhydrous phthalic acid and place into a 1000-mL light-resistant stoppered bottle containing 300 mL of freshly distilled pyridine, exactly measured. Shake the bottle vigorously to mix and dissolve, and allow to stand for NLT 16 hours. Pipet 25 mL of this solution, transfer to the former stoppered pressure bottle, stopper the bottle, wrap it with strong cloth and perform the test as directed in the Average molecular weight under Polyethylene Glycol 400. However, the average molecular weight is 7300 to 9300.

Water NMT 1.0% (2 g, volumetric titration, direct titration).

Residue on ignition NMT 0.2% (1 g).

Packaging and storage Preserve in well-closed containers.

Polyethylene Glycol 20000
폴리에틸렌글리콜 20000

Macrogol 20000

Polyethylene Glycol 20000 is a polymer of ethylene oxide and water, represented by the formula $HOCH_2(CH_2OCH_2)nCH_2OH$, in which the value of n ranges from 340 to 570.

Description Polyethylene Glycol 20000 is white, paraffin-like flakes or powder, and is odorless or has a faint, characteristic odor.
It is freely soluble in water or pyridine and practically insoluble in methanol, ethanol, anhydrous ether, petroleum benzine and polyethylene glycol 400.
Congealing point: Between 56 °C and 64 °C

Identification Dissolve 50 mg of Polyethylene Glycol 20000

in 5 mL of dilute hydrochloric acid, add 1 mL of barium chloride TS, shake to mix and filter, if necessary. To the filtrate, add 1 mL of a solution of phosphomolybdic acid (1 in 10): a yellowish green precipitate is formed.

pH Dissolve 1.0 g of Polyethylene Glycol 20000 in 20 mL of water; the pH of this solution is between 4.5 and 7.5.

Purity (1) ***Clarity and color of solution***—Dissolve 5.0 g of Polyethylene Glycol 20000 in 50 mL of water; the resulting solution is clear and colorless.

(2) ***Acid***—Dissolve 5.0 g of Polyethylene Glycol 20000 in 20 mL of water, add 0.20 mL of 0.1 mol/L sodium hydroxide TS and 1 drop of TS; the color of the solution is green.

(3) ***Heavy metals***—Proceed with 1.0 g of Polyethylene Glycol 20000 according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 20 ppm).

(4) ***Ethylene oxide and dioxane***—Weigh accurately 1.00 g (M_T) of Polyethylene Glycol 20000, transfer to a 10-mL vial, add 1.0 mL of water and mix well. Allow to stand at 70 °C for 45 minutes and use this solution as the test solution. Weigh accurately 1.00 g of dioxane RS, add water to make 100 mL, pipet 5.0 mL of this solution, and add water to make 100 mL. Pipet 10.0 mL of this solution, add water to make 50 mL, and use this solution as the standard dioxane solution. Dilute 50 mg/mL ethylene oxide solution, prepared by dissolving ethylene oxide in methylene chloride, with water to make a solution containing 50 μg of ethylene oxide per mL (this solution is stable for 3 months if sealed with a Teflon-coated silicon membrane and a crimped cap and stored at -20 °C). Transfer 10.0 mL of this solution to a flask containing 30 mL of water, mix well and add water to make 50 mL. Pipet 10.0 mL of this solution, add water to make 50 mL, and use this solution as the standard ethylene oxide solution. Prepared the solution immediately prior to use. Separately, put 1.00 g (M_R) of Polyethylene Glycol 20000 into an identical 10-mL vial, add 0.5 mL of the standard ethylene oxide solution and 0.5 mL of the standard dioxide solution, mix well, allow to stand at 70 °C for 45 minutes and use this solution as the standard solution (1). Put 0.5 mL of the standard ethylene oxide solution into a 10-mL vial, add 0.1 mL of a freshly prepared 10 mg/L standard acetaldehyde solution and 0.1 mL of the standard dioxane solution, mix well, allow to stand at 70 °C for 45 minutes and use this solution as the standard solution (2). Perform the test with 1 mL each of the test solution and the standard solution (1) according to the following conditions, as directed under the Gas Chromatography. Determine the peak areas of ethylene oxide and dioxane in each solution and calculate their amounts: ethylene oxide is NMT 1 ppm and dioxane is NMT 10 ppm.

$$\text{Amount (ppm) of ethylene oxide} = \frac{A_T \times C}{(A_R \times M_T) - (A_T \times M_R)}$$

A_T: Peak area of ethylene oxide in the test solution
A_R: Peak area of ethylene oxide in the standard solution (1)
M_T: Amount (g) of the sample in the test solution
M_R: Amount (g) of the sample in the standard solution (1)
C: Amount (μg) of ethylene oxide added in the standard solution (1)

$$\text{Amount (ppm) of dioxane} = \frac{D_T \times C}{(D_R \times M_T) - (D_T \times M_R)}$$

D_T: Peak area of dioxane in the test solution
D_R: Peak area of dioxane in the standard solution (1)
C: Amount (μg) of dioxane added in the standard solution (1)

Operating conditions

Detector: A flame ionization detector

Column: A glass or quartz capillary tube 0.32 mm in internal diameter and about 30 m in length, coated inside with poly(dimethyl) siloxane in 1.0-μm thickness.

Column temperature: Initially, maintain at 50 °C for 5 minutes, raise the temperature to 180 °C at the rate of 5 °C per minute, then raise the temperature to 230 °C at the rate of 30 °C per minute, and maintain the temperature for 5 minutes.

Sample injection port temperature: A constant temperature of about 150 ℃

Headspace sampler temperature: 70 ℃

Detector temperature: A constant temperature of about 250 ℃

Carrier gas: Helium

Split ratio: About 1 : 20

System suitability

System performance: Proceed with 1.0 mL of the standard solution (2) according to the above conditions; the resolution between the peaks of acetaldehyde and ethylene oxide is NLT 2.0 and the signal-to-noise ratio of dioxane is NLT 5.

Average molecular weight Weigh accurately about 15 g of Polyethylene Glycol 20000, transfer to an about 200-mL stoppered pressure bottle, add about 25 mL of pyridine, dissolve by warming, and allow to cool. Separately, weigh 42 g of anhydrous phthalic acid and place into a 1000-mL light-resistant stoppered bottle containing 300 mL of freshly distilled pyridine, exactly measured. Shake the bottle vigorously to mix and dissolve, and allow to stand for NLT 16 hours. Pipet 25 mL of this solution, transfer to the former stoppered pressure bottle, stopper the bottle, wrap it with strong cloth and place on a water bath, previously heated at 98 ± 2 ℃. At this time, the solution in the bottle is immersed completely in the water bath. Maintain the temperature of the bath at 98 ± 2 °C for 60 minutes. Remove the bottle from the water bath and allow to cool in the air to room temperature. Add exactly 50 mL of 0.5 mol/L sodium hydroxide VS and 5 drops of a solution of phenolphthalein in pyridine (1 in 100), and then titrate with 0.5 mol/L sodium hydroxide VS. However, the endpoint of the titration is when the solution exhibits a pale red color which persists for 15 seconds. Perform a blank test in the same manner. Average molecular mass is 15000 to 25000.

$$\text{Average molecular weight} = \frac{Amount\ (g)\ of\ sample \times 4000}{a - b}$$

a: Volume (mL) of 0.5 mol/L sodium hydroxide VS consumed in the blank test
b: Volume (mL) of 0.5 mol/L sodium hydroxide VS consumed in the test

Water NMT 1.0% (2 g, volumetric titration, direct titration).

Residue on ignition NMT 0.2% (1 g).

Packaging and storage Preserve in well-closed containers.

Polyoxyl 40 Stearate
폴리옥실40스테아레이트

Stearate Polyoxyl 40

Polyoxyl 40 Stearate is a monostearate acid ester of a condensation polymer of ethylene oxide, expressed as $H(OCH_2CH_2)nOOCC_{17}H_{35}$, and n is about 40.

Description Polyoxyl 40 Stearate occurs as a white to pale yellow lead-like lump or powder. It is odorless and has a slightly fat-like odor.
It is soluble in water, ethanol or ether.

Saponification value Between 25 and 35.

Acid value NMT 1.

Congealing temperature Between 39.0 ℃ and 44.0 ℃.

Congealing point of fatty acid NLT 53 ℃.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Polyoxyl 40 Stearate in 20 mL of water; the resulting solution is clear and colorless.

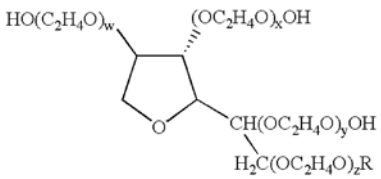
(2) ***Heavy metals*—**Weigh 2.0 g of Polyoxyl 40 Stearate and perform the test according to Method 2. Prepare the control solution by adding 2.0 mL of the lead standard solution (NMT 10 ppm).

(3) ***Arsenic***—Weigh 0.67 g of Polyoxyl 40 Stearate and perform the test according to Method 3 (NMT 3 ppm).

Residue on ignition NMT 0.10% (1 g).

Packaging and storage Preserve in tight containers.

Polysorbate 80
폴리소르베이트 80



Polyoxyethylene 20 sorbitan monooleate [*9005-65-6*]

Polysorbate 80 is a mixture of partial esters of fatty acids, mainly oleic acid, and sorbitol and its anhydrides ethoxylated with 20 moles of ethylene oxide on average for each mole of sorbitol and sorbitol anhydrides.

Description Polysorbate 80 is oily yellow to brownish yellow, clear or slightly opalescent liquid, having a faint, characteristic odor and a warm, slightly bitter taste.
It is miscible with water, ethanol (95), and ethyl acetate, and practically insoluble in liquid paraffin.

Viscosity: About 400 mPa·s (25 ℃)

Specific gravity d_{20}^{20}: About 1.10

Identification (1) Meets the requirements of the test for composition of fatty acids

(2) Determine the infrared spectra of Polysorbate 80 and polysorbate 80 RS as directed in the liquid film method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

Acid value NMT 2.0. However, weigh accurately 5 g of Polysorbate 80 and dissolve in a mixture of ethanol (95) and petroleum ether (1 : 1).

Saponification value Weigh accurately about 4 g of Polysorbate 80 into a 250-mL borosilicate glass flask, add exactly 30.0 mL of 0.5 mol/L potassium hydroxide-ethanol solution and a few glass beads. Attach a small reflux condenser and heat under reflux for 1 hour. To this solution, add 1 mL of phenolphthalein TS and 50 mL of ethanol (99.5), and titrate immediately with 0.5 mol/L hydrochloric acid VS. Perform a blank test in the same manner. Calculate the saponification value according to the following equation; the value ranges between 45 and 55.

Saponification value
= (a - b) × 28.05 / amount (g) of the sample

a: Volume (mL) of 0.5 mol/L hydrochloric acid VS consumed in the blank test solution

b: Volume (mL) of 0.5 mol/L hydrochloric acid VS consumed in the test solution.

Hydroxyl value Weigh accurately about 2 g of Polysorbate 80 into about 150-mL round-bottomed flask, add exactly 5 mL of acetic anhydride-pyridine TS, and heat on a water bath under a reflux condenser. Adjust the level of the water layer to about 2.5 cm above the level of the contents in the flask. After 1 hour, take withdraw the flask, and cool it down. Add 5 mL of water to the flask through the reflux condenser. If cloudiness appears, add sufficient pyridine to clear it, noting the volume of pyridine consumed in this process. Again, shake the flask to mix, and heat on a water bath for 10 minutes. After cooling, rinse the condenser and the inner wall of the flask with 5 mL of neutralized ethanol down into the flask, and titrate with 0.5 mol/L potassium hydroxide-ethanol VS (indicator: 0.2 mL of phenolphthalein TS). Perform a blank test in the same manner. Calculate the hydroxyl value according to the following equation; the value ranges between 65 and 80.

Hydroxyl value
= [(a - b) × 28.05 / Amount (g) of the sample] + Acid value

a: Volume (mL) of 0.5 mol/L potassium hydroxide-ethanol VS consumed in the blank test solution

b: Volume (mL) of 0.5 mol/L potassium hydroxide-ethanol VS consumed in the test solution.

Purity (1) ***Ethylene oxide and 1,4-dioxane***—Place 1.00 g of Polysorbate 80 into a 10-mL headspace vial, dissolve in exactly 2.0 mL of water, seal immediately with a Teflon-coated silicon membrane and an aluminum cap, shake gently and use this solution as test solution (1). Separately, place 1.0 g of Polysorbate 80 into a 10-mL headspace vial, dissolve in 2.0 mL of the standard solution, proceed in the same manner as the test solution (1) and use this solution as the test solution (2). Dilute 0.5 mL of 50 mg/mL ethylene oxide solution, prepared by dissolving ethylene oxide in methylene chloride, with water to make 50.0 mL (this solution is stable for 3 months if sealed with a Teflon-coated silicon membrane and a crimped cap and stored at -20 °C). Allow

to stand at room temperature, pipet 1.0 mL of this solution, add water to make 250.0 mL, and use this solution as the standard ethylene oxide solution. Pipet 1.0 mL of dioxane RS, dilute 20000-fold with water, and use this solution as the standard dioxane solution. Mix 6.0 mL of the standard ethylene oxide solution and 2.5 mL of the standard dioxane solution, add water to make 25.0 mL and use this solution as the standard solution. Prepare a 0.01 g/L solution of acetaldehyde in water and use this solution as the standard acetaldehyde solution. Place 2.0 mL of the standard acetaldehyde solution and 2.0 mL of the standard ethylene oxide solution into a 10-mL headspace vial, seal immediately with a Teflon-coated silicon membrane and an aluminum cap, shake gently to mix, and use this solution as the system suitability solution. Perform the test with exactly 1 μL each of the test solutions (1) and (2) as directed under the Liquid Chromatography according to the following conditions. Determine the peak areas of ethylene oxide and dioxane in each solution and calculate their amounts: the amount of ethylene oxide is NMT 1 ppm and that of dioxane is NMT 10 ppm.

$$\text{Amount (ppm) of ethylene oxide} = 2 \times C_{EO} \times A_a / (A_b - A_a)$$

C_{T2}: Concentration (μg/mL) of ethylene oxide in the test solution (2)

A_{T1}: Peak area of ethylene oxide in the test solution (1)

A_B: Peak area of ethylene oxide in the test solution (2)

$$\text{Amount (ppm) of dioxane} = 2 \times 1.03 \times C_D \times A_a' \times 1000 / (A_b' - Aa')$$

C_D: Concentration (μL/mL) of dioxane in the test solution (2)

1.03: Density of (g/mL) dioxane

A_a': Peak area of dioxane in the test solution (1)

A_b': Peak area of dioxane in the test solution (2)

Operating conditions

Detector: A flame ionization detector

Column: A fused silica column, 0.53 mm in internal diameter and about 50 m in length, coated inside with poly(dimethyl)(diphenyl) siloxane in 5-μm thickness.

Column temperature: From a constant temperature of about 70 °C, raise the temperature to 250 °C at the rate of 10 °C per minute and maintain at a constant temperature of about 250 °C for 5 minutes.

Sample injection port temperature: A constant temperature of about 85 ℃

Headspace sampler temperature: 80 ℃

Detector temperature: A constant temperature of about 250 ℃

Carrier gas: Helium

Flow rate: 4.0 mL/minute (The retention time of ethylene oxide is about 6.5 minutes and the relative retention times of acetaldehyde and dioxane to ethylene oxide are 0.9 and 1.9, respectively.)

Split ratio: About 1 : 3.5

System suitability

System performance: Proceed with 1.0 mL of the system suitability solution according to the above operating conditions; the resolution between the peaks of acetaldehyde and ethylene oxide is NLT 2.0.

(3) ***Peroxide value***—Weigh accurately about 10 g of Polysorbate 80, place in a 100-mL beaker and dissolve in 20 mL of acetic acid (100). Add 1 mL of saturated potassium iodide solution, and allow to stand for 1 minute. Add 50 mL of freshly boiled and cooled water, place a magnetic stirring bar and stir with a magnetic stirrer. Titrate with 0.01 mol/L sodium thiosulfate VS (potentiometric titration under the Titrimetry). Perform a blank test in the same manner (at this time, the consumed amount of 0.01 mol/L sodium thiosulfate VS of the blank test solution is NMT 0.1 mL.). The peroxide value calculated according to the following equation is NMT 10.

$$\text{Peroxide value} = 10 \times (V_1 - V_0) / W$$

V_1: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed for titration of the test solution

V_0: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed for titration of the blank test solution

W: Amount (g) of Polysorbate 80 taken

Water NMT 3.0% (1 g, volumetric titration, direct titration).

Residue on ignition Heat a silica or platinum crucible in advance for 30 minutes, allow to cool in a desiccator, and weigh the mass accurately. Weigh accurately about 2 g of Polysorbate 80, and evenly distribute in the crucible. Dry at 100 ℃ to 105 ℃ for 1 hour and ignite to a constant mass in a muffle furnace at 600 ± 50 ℃, allowing the crucible to cool in a desiccator. Ensure that flames are not generated at any point during the combustion. If the ash still contains black particles after prolonged ignition, add hot water, pass through an ashless filter paper. Ignite the residue and the filter paper and combine the ash with the filtrate, carefully evaporate to dryness, and ignite to a constant mass (NMT 0.25%)

Fatty acids composition Weigh accurately about 0.1 g of Polysorbate 80, put in a 25-mL Erlenmeyer flask, and dissolve in 2 mL of sodium hydroxide solution in methanol (1 in 50). Install a reflux condenser and heat for 30 minutes. Add 2.0 mL of boron trifluoride-methanol TS and heat for 30 minutes. Add 4 mL of n-heptane through a top of the condenser and heat for 5 minutes. After cooling, add 10 mL of saturated sodium chloride solution prepared by mixing water and sodium chloride at a ratio of 2:1 and then filter to contain no remaining undissolved substances. Shake the flask for 15 minutes to mix, and add a sufficient amount of saturated sodium chloride solution so that the clear supernatant is brought to the neck of the flask. Collect 2 mL of the clear supernatant, wash three times with 2 mL each of water, and dry over anhydrous sodium sulfate. Use this solution as the test solution. Separately, dissolve 0.50 g of the mixture with the composition in Table 1 in n-heptane to make exactly 50 mL, and use this solution as the control solution (1). To the control solution (1), add n-heptane to dilute 10-fold, and use this solution as the control solution (2). Dissolve 0.50 g of a mixture of fatty acid methyl esters with known composition of fatty acids in heptane, make exactly 50 mL, and use this solution as the control solution (3). (Commercially available mixtures of fatty acid methyl esters may also be used.)

Mixture of the following components	Composition (%)
Methyl myristate	5
Methyl palmitate	10
Methyl stearate	15
Methyl arachidate	20
Methyl oleate	20
Methyl eicosenoate	10

Methyl behenate	10
Methyl lignocerate	10

Perform the test with 1 μL each of the test solution and the control solution (3) as directed under the Liquid Chromatography according to the following conditions. Confirm the peak of each fatty acid ester with the control solution (3) and determine A_C, the peak area of each fatty acid ester obtained from the test solution and A_T, the total peak area of all fatty acid esters obtained from the test solution. Calculate the content (%) of each fatty acid of Polysorbate 80 according to the following equation; the values are shown in Table 2 below.

Content (%) of fatty acid component
$= A_C / A_T \times 100$

Components	NMT (%)	NLT (%)
Myristic acid	5.0	-
Palmitic acid	16.0	-
Palmitoleic acid	8.0	-
Stearic acid	6.0	-
Oleic acid	-	58.0
Linoleic acid	18.0	-
Linolenic acid	4.0	-

Operating conditions

Detector: A flame ionization detector

Column: A fused silica column for gas chromatography, about 0.32 mm in internal diameter and about 30 m in length, coated inside with polyethylene glycol 20000 for gas chromatography in 5-μm thickness.

Sample injection port temperature: A constant temperature around 250 ℃

Column temperature: Maintain a constant temperature of about 80 °C until injection, raise the temperature to 220 °C at a rate of 10 °C per minute and maintain at a constant temperature of 220 °C for NLT 40 minutes.

Detector temperature: A constant temperature around 250 ℃

Carrier gas: Helium

Flow rate: 50 cm/sec

System suitability

Confirmation of detection: Proceed with 1 μL of the control solution (2) according to the above operating conditions and confirm that the signal-to-noise ratio of the peak of methyl myristate is NLT 5.

System performance: Proceed with 1 μL of the control solution (1) according to the above conditions; the resolution between peaks of methyl stearate and methyl oleate is NLT 1.8, and the number of theoretical plates of methyl stearate is NLT 30000.

Packaging and storage Preserve in light-resistant, tight containers.

Potassium Carbonate
탄산칼륨

K_2CO_3: 138.21

Potassium Carbonate, when dried, contains NLT 99.0% and NMT 101.0% of potassium carbonate (K_2CO_3).

Description Potassium Carbonate occurs as a white grain or powder, and is odorless.
It is very soluble in water and practically insoluble in ethanol.
Dissolve 1 g of Potassium Carbonate in 10 mL of water; the solution is alkaline.
It is hygroscopic.

Identification An aqueous solution of Potassium Carbonate (1 in 10) responds to the Chemical identification reactions for potassium salt and carbonate.

Purity (1) ***Clarity and color of solution*—**Dissolve 1.0 g of Potassium Carbonate in 20 mL of water; the resulting solution is clear and colorless.

(2) ***Chloride*—**Weigh 0.2 g of Potassium Carbonate, add 6 mL of dilute nitric acid, and heat. After cooling, add 6 mL of dilute nitric acid, use this solution as the test solution, and perform the test as directed under the Chloride limit test; the amount is NMT the amount corresponding to 0.3 mL of 0.01 mol/L hydrochloric acid.

(3) ***Mercury*—**Spread about 1 g of excipient (a) evenly on a ceramic boat, and place 10 mg to 300 mg of Potassium Carbonate on top. Spread about 0.5 g of the excipient (a) and 1 g of the excipient (b) on top of it in turn to form a layer. In the case of an automated mercury analyzer equipped with a separate catalyst in the combustion chamber, take only the sample without adding excipients to a nickel boat. Place the boat inside the combustion furnace and heat to about 900 °C with a current of air or oxygen at 0.5 to 1 L/minute. Elute the mercury and collect in a collecting tube. Transfer the mercury vapor to a cold vapor atomic absorption spectrometer by heating the collecting tube to about 700 °C and determine the absorbance, A. Separately, place only the excipients in a ceramic boat and determine the absorbance, Ab in the same manner. Separately, proceed with mercury standard solution in the same manner and create a calibration curve from the absorbances. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the sample (NMT 1.0 ppm).

Operating conditions

Apparatus: Use an automated mercury analyzer for specimen combustion, trapping with gold amalgam and measurement using a cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Standard mercury stock solution—Dissolve 0.135 g of mercury(II) chloride in 0.001% L-cysteine solution to make 1000 mL. Each mL of this solution contains 100 μg of mercury(II) chloride.

Mercury standard solution—Dilute the standard mercury stock solution with 0.001% L-cysteine solution to make 0 to 200 ng/mL.

Excipients—Activate excipients at 950 ℃ for 30 minutes when using (a) aluminum oxide and (b) calcium hydroxide-sodium carbonate (1 : 1).

(4) ***Lead*—**Weigh accurately 5.0 g of Potassium Carbonate, transfer to a 150-mL beaker, add 30 mL of water, and add hydrochloric acid in small amounts until the sample is sufficiently dissolved. Then add 1 mL of hydrochloric acid again. Boil for about 5 minutes, cool, add water to make about 100 mL, and adjust the pH to 2 to 4 with sodium hydroxide (1 in 4) or hydrochloric acid

(1 in 4). Transfer this solution to a 250-mL separatory funnel, add water to make about 200 mL, add 2 mL of 2% ammonium pyrrolidine dithiocarbamate solution (2 in 100), and shake to mix. Extract this solution twice with 20 mL of chloroform and evaporate the extract to dryness on a water bath. To the residue, add 3 mL of nitric acid and heat until almost dry. Add 0.5 mL of nitric acid and 10 mL of water, concentrate until the final solution becomes 3 mL to 5 mL, add water to make 10 mL, and use this solution as the test solution. Separately, put 1.0 mL of lead standard solution in the platinum crucible, proceed in the same manner as in the preparation of the test solution, and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Atomic absorption spectroscopy according to the following operating conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 2.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: A lead hollow-cathode lamp
Wavelength: 283.3 nm

(5) ***Heavy metals*—**Dissolve 1.0 g of Potassium Carbonate in 2 mL of water and 6 mL of dilute hydrochloric acid, evaporate to dryness on a water bath. Dissolve the residue in 35 mL of water and 2 mL of dilute acetic acid, and add water again to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution by evaporating 6 mL of dilute hydrochloric acid to dryness on a water bath and adding 2 mL of dilute acetic acid, 2.0 mL of lead standard solution and water to make 50 mL (NMT 20 ppm).

(6) ***Sodium*—**Dissolve 1.0 g of Potassium Carbonate in 20 mL of water, and perform the test as directed under the Flame Coloration (1); the solution does not exhibit a persistent yellow color.

(7) ***Arsenic*—**Prepare the test solution with 0.5 g of Potassium Carbonate according to Method 1 and perform the test (NMT 4 ppm).

Loss on drying NMT 1.0% (3 g, 180 ℃, 4 hours).

Assay Weigh accurately about 1.5 g of Potassium Carbonate, previously dried, dissolve in 25 mL of water, and titrate with 0.5 mol/L sulfuric acid VS. When the blue color of the solution changes to yellowish green, boil it carefully, cool, and titrate until a greenish yellow color appears (indicator: 2 drops of Bromocresol green TS).

Each mL of 0.5 mol/L sulfuric acid VS
= 69.10 mg of K_2CO_3

Packaging and storage Preserve in tight containers.

Potassium Hydroxide
수산화칼륨

KOH: 56.11

Potassium Hydroxide contains NLT 85.0% and NMT 101.0% of potassium hydroxide (KOH).

Description Potassium Hydroxide occurs as a white mass in small globules, flakes, rods, or other forms. It is hard and brittle with a crystalline fracture.
It is freely soluble in water or ethanol and practically insoluble in ether.
It rapidly absorbs carbon dioxide in the air.
It deliquesces in the presence of moisture.

Identification (1) An aqueous solution of Potassium Hydroxide (1 in 500) is alkaline.

(2) An aqueous solution of Potassium Hydroxide (1 in 25) responds to the Chemical identification reactions for potassium salt.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Potassium Hydroxide in 20 mL of water; the resulting solution is clear and colorless.

(2) **C*hloride***—Dissolve 2.0 g of Potassium Hydroxide in 100 mL of water. To 25 mL of this solution, add 8 mL of dilute nitric acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution by adding 0.7 mL of 0.01 mol/L hydrochloric acid (NMT 0.050%).

(3) ***Heavy metals***—Dissolve 1.0 g of Potassium Hydroxide in 5 mL of water, add 7 mL of dilute hydrochloric acid, and evaporate on a water bath to dryness. Add 35 mL of water, 2 mL of dilute acetic acid and 1 drop of ammonia TS to dissolve, and add water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution by evaporating 7 mL of dilute hydrochloric acid to dryness on a water bath and adding 2 mL of dilute acetic acid, 3.0 mL of the lead standard solution and water to make 50 mL (NMT 30 ppm).

(4) ***Mercury***—Dissolve 2 g of Potassium Hydroxide in 10 mL of water, add 1 mL of potassium permanganate solution (3 in 50) and about 30 mL of water, and shake to mix. Neutralize by slowly adding purified hydrochloric acid, add 5 mL of sulfuric acid (1 in 2), cool, and use this solution as the test solution. Add hydroxylamine hydrochloride solution (1 in 5) until the purple color of potassium permanganate disappears from the test solution, and the manganese dioxide precipitate dissolves. Add water to make 100 mL and transfer it to the sample bottle for an atomic absorption spectrophotometer. Add 10 mL of tin(II) chloride TS, immediately connect the bottle to the atomic absorption spectrophotometer, and circulate air by operating the diaphragm pump. Read the absorbance of the test solution when the reading of the data collection device rises rapidly to a constant; the absorbance is not greater than that of the following solution: to 2 mL of the mercury standard solution, add 1 mL of potassium permanganate (3 in 50), about 30 mL of water and the amount of purified hydrochloric acid used to prepare the test solution and proceed in the same manner as for the preparation of the test solution (NMT 0.1 ppm).

(5) ***Lead***—Weigh accurately 5.0 g of Potassium Hydroxide, transfer it to a 150-mL beaker, and add 30 mL of water. Add hydrochloric acid in small portions until the sample completely dissolves, and then add an additional 1 mL of hydrochloric acid. Boil the mixture for about 5 minutes, cool, add water to make about 100 mL, and adjust the pH to between 2 and 4 with sodium hydroxide (1 in 4) or hydrochloric acid (1 in 4). Transfer the solution to a 250-mL separatory funnel, add water to make about 200 mL, add 2 mL of 2% ammonium pyrrolidine dithiocarbamate solution (2 in 100), and shake to mix. Extract this solution twice with 20 mL of chloroform and evaporate the extract to dryness on a water bath. To the residue, add 3 mL of nitric acid and heat until the mixture becomes almost dry. To the residue, add 0.5 mL of nitric acid and 10 mL of water, concentrate to the final volume of 3 to 5 mL, add water to make 10 mL, and use the solution as the test solution. Separately, transfer 1.0 mL of the lead standard solution to a platinum crucible, proceed in the same manner as for the preparation of the test solution, and use this solution as

the standard solution. Perform the test with the test solution and the standard solution as directed under the Atomic absorption spectroscopy according to the following conditions; the absorbance of the test solution is not greater than that of the standard solution (NMT 2.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: A lead hollow-cathode lamp
Wavelength: 283.3 nm

(6) ***Arsenic***—Dissolve 0.5 g of Potassium Hydroxide in 5 mL of water, neutralize by slowly adding hydrochloric acid, and use this solution as the test solution. Perform the test with the test solution as directed under the Arsenic (NMT 4 ppm).

(7) ***Sodium***—Dissolve 0.10 g of Potassium Hydroxide in 10 mL of dilute hydrochloric acid and perform the test as directed under the Flame Coloration (1); no persistent yellow color develops.

(8) ***Potassium carbonate***—Prepare B (mL) of the solution as directed under the Assay and determine the amount of potassium carbonate (K_2CO_3: 138.21) using the following formula; the amount of potassium carbonate is NMT 2.0%.

$$\text{Amount (mg) of potassium carbonate} = 138.21 \times B$$

Assay Weigh accurately about 1.5 g of Potassium Hydroxide and dissolve in 40 mL of freshly boiled and cooled water. Cool to 15 °C, add 2 drops of phenolphthalein TS, and titrate with 0.5 mol/L sulfuric acid VS until the red color of the solution disappears. Record the amount, A (mL), of 0.5 mol/L sulfuric acid VS consumed. Add 2 drops of methyl orange TS and titrate again with 0.5 mol/L sulfuric acid VS until the solution exhibits a persistent pale red color. Record the amount, B (mL), of sulfuric acid VS consumed. Calculate the amount of Potassium Hydroxide (KOH) by subtracting B (mL) from A (mL).

$$\text{Each mL of 0.5 mol/L sulfuric acid VS} = 56.11 \text{ mg of KOH}$$

Packaging and storage Preserve in tight containers.

Potassium Sulfate
황산칼륨

K_2SO_4 : 174.26

Potassium Sulfate, when dried, contains NLT 99.0% and NMT 101.0% of potassium sulfate (K_2SO_4).

Description Potassium Sulfate occurs as a colorless crystal or a white, crystalline powder, and has a slightly saline and bitter taste.
It is soluble in water and practically insoluble in ethanol.

Identification An aqueous solution of Potassium Sulfate (1 in 20) responds to the Chemical identification reactions for potassium salt and sulfate.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Potassium Sulfate in 20 mL of water; the solution is colorless, clear and neutral.

(2) ***Chloride***—Perform the test with 0.5 g of Potassium Sulfate. Prepare the control solution with 0.40 mL of 0.01 mol/L hydrochloric acid (NMT 0.028%).

(3) ***Heavy metals***—Proceed with 2.0 g of Potassium Sulfate according to Method 1 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 10 ppm).

(4) ***Mercury***—Spread evenly about 1 g of excipient (a) into a ceramic boat and place 10 mg to 300 mg of Potassium Sulfate on top. Next, spread evenly about 0.5 g of excipient (a) and 1 g of excipient (b) successively to form layers. In the case of an automated mercury analyzer equipped with a separate catalyst in the combustion chamber, take only the sample without adding excipients to a nickel boat. Place the boat inside the combustion furnace and heat to about 900 °C with a current of air or oxygen at 0.5 to 1 L/minute. Elute the mercury and collect in a collecting tube. Transfer the mercury vapor to a cold vapor atomic absorption spectrometer by heating the collecting tube to about 700 °C and determine the absorbance as A. Separately, determine the absorbance in the same manner using only the excipients in a ceramic boat and designate as Ab. Separately, proceed with mercury standard solution in the same manner and create a calibration curve from the absorbances. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the sample (NMT 1.0 ppm).

Operating conditions

Apparatus: Use an automated mercury analyzer for specimen combustion, trapping with gold amalgam and measurement using a cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Standard mercury stock solution—Dissolve 0.135 g of mercury (II) chloride in 0.001% L-cysteine to make 1000 mL. Each mL of this solution contains 100 μg of mercury(II) chloride.

Mercury standard solution—Dilute standard mercury stock solution with 0.001% L-cysteine so that each mL contains 0 to 200 ng.

Excipients—Use (a) aluminum oxide and (b) a mixture of calcium hydroxide and sodium carbonate (1 : 1) and activate at 950 °C for 30 minutes before use.

(5) ***Lead***—Weigh accurately 5.0 g of Potassium Sulfate, transfer to a 150-mL beaker and add 30 mL of water. Add hydrochloric acid in small amounts until the sample is completely dissolved, and then add 1 mL of hydrochloric acid. Boil the solution for about 5 minutes, cool, add water to make about 100 mL and adjust the pH to 2 - 4 with sodium hydroxide solution (1 in 4) or hydrochloric acid (1 in 4). Transfer this solution to a 250-mL separatory funnel, add water to make about 200 mL, add 2 mL of 2% ammonium pyrrolidine dithiocarbamate solution (2 in 100) and shake well to mix. Extract this solution twice with 20 mL each of chloroform and evaporate the extract to dryness on a water bath. To the residue, add 3 mL of nitric acid and heat until almost dry. Add 0.5 mL of nitric acid and 10 mL of water, concentrate until the final solution becomes 3 mL to 5 mL, add water to make 10 mL, and use this solution as the test solution. Separately, proceed with 1.0 mL of lead standard solution in a platinum crucible in the same manner as the test solution, and use this solution as the standard solution. Perform the test with the test solution and the standard solutions as directed under Atomic Absorption Spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 2.0 ppm).

Gas: Acetylene or hydrogen- Air.
Lamp: Lead hollow-cathode lamp
Wavelength: 283.3 nm

(6) ***Selenium***—Dissolve 1 g of Potassium Sulfate in 100 mL of water and use this solution as the test solution. Determine the absorbance of the test solution as directed under the electrothermal-type Atomic Absorption Spectroscopy. The absorbance of the test solution is not greater than that of the solution prepared as follows: to 3 mL of selenium standard solution, add water to make 100 mL and proceed in the same manner as the test solution (NMT 30 ppm).

Selenium standard solution—To 1 g of selenium, add 10 mL of sulfuric acid (1 in 2), dissolve on a water bath by heating, evaporate to dryness, and dissolve the residue in water to make 1000 mL. To 10 mL of this solution, add water to make 1000 mL. 1 mL of this solution contains 0.01 mg of Se.

(7) ***Sodium***—Dissolve 1.0 g of Potassium Sulfate in 20 mL of water and perform the test as directed under the Flame Coloration (1): no persistent yellow color develops.

(8) ***Arsenic***—Proceed with 4.0 g of Potassium Sulfate according to Method 1 and perform the test (NMT 5 ppm).

Loss on drying NMT 1.0% (1 g, 110 ℃, 4 hours).

Assay Weigh accurately about 0.5 g of Potassium Sulfate, previously dried, boil with 200 mL of water and 1.0 mL of hydrochloric acid and add gradually 8 mL of boiling barium chloride TS. Heat the mixture on a water bath for 1 hour, collect the precipitate and wash the precipitate until the last washing shows no turbidity even with the addition of silver nitrate TS. Dry, ignite to a constant mass to between 500 and 600 °C by raising the temperature gradually and weigh the mass to determine the amount of barium sulfate ($BaSO_4$: 233.39).

Amount (mg) of potassium sulfate (K_2SO_4)
= Amount (mg) of barium sulfate ($BaSO_4$) × 0.7466

Packaging and storage Preserve in well-closed containers.

Potato Starch
감자전분

Potato Starch is a starch obtained from the tuber of *Solanum tuberosum* Linné (Solanaceae) potato.

Description Potato Starch is a very fine white or almost white powder that makes a crunching sound when pressed with a finger. It is practically insoluble in cold water or in ethanol (95).
It does not contain starch granules of other origin. Sometimes, it may contain a small amount of tissue fragments deriving from the original plant.

Identification (1) Under a microscope, Potato Starch, preserved in a mixture of water and glycerin (1 : 1), appears as unevenly ovoid or pyriform simple grains usually 30–100 μm, often more than 100 μm in diameter, or spherical simple grains 10-35 μm in diameter. Rarely, it consists of 2 to 4 compound grains. Spherical simple grains have non-centric or slightly eccentric hila. All grains exhibit a distinct pattern of striation. Under crossed polarizing prisms, Potato Starch exhibits a clear black cross, with its intersection point at the hilum.

(2) To 1 g of Potato Starch, add 50 mL of water, boil for 1 minute, and allow to cool; a subtle turbid, viscous liquid is formed.

(3) To 1 mL of the liquid obtained in (2), add 0.05 mL of diluted iodine TS (1 in 10); the resulting solution exhibits an orange to dark bluish purple color, which disappears upon heating.

pH Place 0.5 g of Potato Starch in a non-metal container, add 25.0 mL of freshly boiled and cooled water, gently shake for 1 minute to make a suspension, and allow to stand for 15 minutes; the pH of the resulting suspension is 5.0 to 8.0.

Purity (1) ***Iron***—To 1.5 g of Potato Starch, add 15 mL of 2 mol/L hydrochloric acid TS, shake to mix, filter, and use the filtrate as the test solution. To 2.0 mL of iron standard solution, add 20 mL of water and use this solution as the reference solution. Transfer 10 mL each of the test solution and the reference solution into test tubes, add 2 mL of a solution of citric acid (1 in 5) and 0.1 mL of mercaptoacetic acid to each tube, and mix. To each solution, add strong ammonia water until the solutions clearly show an alkaline reaction on a litmus paper, then, add water to make 20 mL, and mix. Transfer 10 mL each of these solutions into test tubes, allow to stand for 5 minutes, and compare the color of these solutions against a white background; the color of the test solution is not more intense than that of the reference solution (NMT 10 ppm).

(2) **Oxidizing substances—**To 4.0 g of Potato Starch, add 50.0 mL of water, shake for 5 minutes to mix, and centrifuge. To 30.0 mL of the clear supernatant, add 1 mL of acetic acid (100) and 0.5 to 1.0 g of potassium iodide, shake to mix, and allow to stand in the dark for 20 to 25 minutes. Add 1 mL of starch TS to the resulting solution and titrate with 0.002 mol/L sodium thiosulfate VS until the color of the solution disappears. Perform a blank test in the same manner and make any necessary correction. The volume of 0.002 mol/L sodium thiosulfate VS consumed is NMT 1.4 mL (NMT 20 ppm, calculated on the hydrogen peroxide basis).

(3) ***Sulfur dioxide***—NMT 50 ppm.

(4) ***Foreign matter***—Under a microscope, no other starch is identified. Also, even if tissue fragments of the original plant are contained in Potato Starch, the amount of such fragments is extremely small.

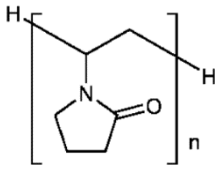
Loss on drying NMT 20.0% (1 g, 130 ℃, 90 hours).

Residue on ignition NMT 0.6% (1 g).

Microbiological examination of non-sterile products Perform the test as directed under the Microbiological Examination of Non-sterile Products; the total number of aerobic microorganisms is NMT 10^3 CFU, and the total number of fungi is NMT 10^2 CFU per g of Potato Starch. Also, neither *Escherichia coli* nor *Salmonella* are detected.

Packaging and storage Preserve in well-closed containers.

Povidone
포비돈



Polyvinylpyrrolidone $(C_6H_9NO)_n$
Poly(1-ethenylpyrrolidin-2-one) [*9003-39-8*]

Povidone is a chain polymer of 1-vinyl-2-pyrrolidone.

Povidone contains NLT 11.5% and NMT 12.8% of nitrogen (N: 14.01), calculated on the anhydrous basis.

Povidone has a nominal K-value of NLT 10 and NMT 120 The nominal K-value is shown on the label of Povidone.

Description Povidone occurs as a white to pale yellow powder. It is freely soluble in water, methanol or ethanol (95), slightly soluble in acetone, and practically insoluble in ether.
It is hygroscopic.

Identification (1) Dissolve 0.05 g of Povidone in water to make exactly 10 mL and add a few drops of iodine TS; the solution exhibits a deep red color.

(2) Dissolve 0.2 g of Povidone in water to make exactly 10 mL, add 20 mL of 1 mol/L hydrochloric acid TS, mix, and then add potassium dichromate TS; a yellow to orange precipitate is formed.

(3) Dissolve 0.2 g of Povidone in water to make exactly 10 mL, and use this solution as the test solution. Separately, dissolve 75 mg of cobalt (II) nitrate hexahydrate and 300 mg of ammonium thiocyanate in 2 mL of water, and use this solution as the solution A. To 5 mL of the test solution, add the solution A, mix, and then add 3 mol/L hydrochloric acid TS to render the solution acidic; a pale blue precipitate is formed.

(4) Dissolve 0.5 g of Povidone in 10 mL of water and shake to mix; it dissolves well.

(5) Determine the infrared spectra of Povidone and povidone RS, previously dried at 105 °C for 6 hours, as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

pH Dissolve 1.0 g of Povidone in 20 mL of water: the pH of this solution is 3.0 to 5.0 for Povidone having the nominal K-value of NMT 30 and 4.0 to 7.0 for Povidone having the nominal K-value exceeding 30.

Purity (1) ***Peroxides***—Weigh accurately an amount equivalent to 4.0 g of Povidone, calculated on the anhydrous basis, dissolve in water to make exactly 100 mL, and use this solution as the test solution. To 25 mL of this solution, add 2 mL of titanium(III) chloride-sulfuric acid TS and allow to stand for 30 minutes. Transfer this solution into a 1-cm cell, using a solution prepared by adding 13% sulfuric acid to 52 mL of the test solution as a reference solution and perform the test as directed under the Ultraviolet-visible Spectroscopy; the absorbance at 405 nm is NMT 0.35 (NMT 400 ppm as peroxide)

(2) ***1-vinyl-2-pyrrolidone***—Weigh accurately about 0.25 g of Povidone, dissolve in the mobile phase to make exactly 10 mL, and use this solution as the test solution. Separately, weigh accurately 50 mg of 1-vinyl-2-pyrrolidone, and dissolve in the mobile phase to make exactly 100 mL. Pipet 1 mL of this solution, and add the mobile phase to make exactly 100 mL. Pipet 5 mL of this solution, add the mobile phase to it make exactly 100 mL, and use this solution as the standard solution. Perform the test with 20 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine the peak areas, A_T and A_S, of 1-vinyl-2-pyrrolidone in each solution; the amount of 1-vinyl-2-pyrrolidone is NMT 0.001%.

$$\text{Amount (\%) of 1-vinyl-2-pyrrolidone} = (A_T / A_S) \times (C_S / C_T) \times 100$$

C_S: Concentration (mg/mL) of 1-vinyl-2-pyrrolidone in the standard solution.

C_T: Concentration (mg/mL) of Povidone in the test solution, calculated on the anhydrous basis.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 235 nm)

Column: A stainless steel column about 4.6 mm in internal diameter and 15 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter). Use a stainless steel column about 4 mm in internal diameter and 1.0 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter) as a guard column.

Column temperature: A constant temperature around 40 °C

Mobile phase: A mixture of water and acetonitrile (9 : 1)

Flow rate: 1.0 mL/min

System suitability

System performance: Dissolve 10 mg of 1-vinyl-2-pyrrolidone and 0.5 g of vinyl acetate in 100 mL of methanol. Pipet 1 mL of this solution, and add the mobile phase to make 100 mL. Proceed with 20 μL of this solution according to the above operating conditions; 1-vinyl-2-pyrrolidone and vinyl acetate are eluted in this order with the resolution being NLT 2.0.

System repeatability: Repeat the test 6 times with 20 μL each of the standard solution according to the above operating conditions; the relative standard deviation of the peak areas of 1-vinyl-2-pyrrolidone is NMT 2.0%.

(3) ***Aldehydes***—Weigh accurately about 1.0 g of Povidone and dissolve in 0.05 mol/L pyrophosphate buffer solution (pH 9.0) to make exactly 100 mL. Stopper, warm the solution at 60 °C for 60 minutes, allow to stand until it cools to room temperature, and use this solution as the test solution. Separately, weigh accurately 140 mg of acetaldehyde ammonia trimer trihydrate and dissolve in water to make exactly 200 mL. Pipet 1 mL of this solution, dissolve in 0.05 mol/L pyrophosphate buffer solution (pH 9.0) to make exactly 100 mL, and use this solution as the standard solution. Transfer 0.5 mL each of the test solution, the standard solution and water to separate cells, add 2.5 mL of 0.05 mol/L pyrophosphate buffer solution (pH 9.0) and 0.2 mL of β-nicotinamide adenine dinucleotide TS to each of these cells, stir to mix and stopper tightly. Allow to stand for 2 to 3 minutes at 22 ± 2 °C. With these solutions, perform the test as directed under the Ultraviolet-visible Spectroscopy with water as a reference solution and determine the absorbance at 340 nm. The absorbances of each solution are A_{T1}, A_{S1} and A_{B1}, respectively. Add 0.05 mL of aldehyde dehydrogenase TS to each solution, stir to mix and stopper. Allow to stand for 5 minutes at 22 ± 2 °C. Determine the absorbances, A_{T2}, A_{S2} and A_{B2}, of these solutions in the same manner as above: the content of aldehydes is NMT 500 ppm

expressed as acetaldehyde.

$$\text{Content (ppm) of aldehyde} = (C / W) \times [(A_{T2} - A_{T1}) - (A_{B2} - A_{B1})]/[(A_{S2} - A_{S1}) - (A_{B2} - A_{B1})] \times 100{,}000$$

C: Concentration (μg/mL) of aldehyde in the standard solution. The conversion factor from acetaldehyde ammonia trimer trihydrate to acetaldehyde is 0.72.

W: Amount (g) of the sample taken, calculated on the anhydrous basis

β-nicotinamide adenine dinucleotide TS—Dissolve 40 mg of β-nicotinamide adenine dinucleotide in 0.05 mol/L pyrophosphate buffer solution (pH 9.0) to make 10.0 mL (this solution is stable at 4 ℃ for 4 weeks.).

(4) ***Formic acid***—Weigh accurately about 2.0 g of Povidone, dissolve in water to make exactly 100 mL, and use this solution as the test stock solution. Pack a chromatography column with internal diameter of about 8mm with strongly acidic ion exchange resin for column chromatography (H type) so that the strongly acidic ion-exchange resin layer is immersed in water. To this column, add 5 mL of water and adjust the flow rate so that water is eluted at a rate of about 1 mL per minute. When the water level reaches the top of the strongly acidic ion-exchange resin layer, add 100 mL of the test stock solution. Discard the first 2 mL of the eluate and take 1.5 mL of the subsequent eluate as the test solution. Separately, weigh accurately about 0.1 g of formic acid, and add water to make exactly 100 mL. Pipet 1 mL of this solution, add water to make 100 mL, and use this solution as the standard solution. Perform the test with 50 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and calculate the peak areas, A_T and A_S, of formic acid from the test solution and the standard solution, respectively; the amount (%) of formic acid is NMT 0.5%.

$$\text{Content (\%) of formic acid} = (A_T / A_S) \times (C_S / C_T) \times 100$$

A_T: Peak area of formic acid in the test solution

A_S: Peak area of formic acid in the standard solution

C_S: Concentration (mg/mL) of formic acid in the standard solution

C_T: Concentration (mg/mL) of povidone in the test solution, calculated on the anhydrous basis.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 210 nm)

Column: A stainless steel column about 7.8 mm in internal diameter and about 30 cm in length, packed with strongly acidic ion exchange resin (hydrogen type) where styrenedivinylbenzene copolymer (9 μm in particle diameter) is bonded to a sulfonic acid group.

Mobile phase: A diluted perchloric acid (1 in 700)

Flow rate: 1.0 mL/min (Retention time of formic acid: about 8 minutes)

System suitability

Proceed with 50 μL of the standard solution according to the above operating conditions and perform the test; the number of theoretical plates and symmetry factor of the peaks of formic acid are NLT 1000 and between 0.5 and 1.5, respectively.

System repeatability: Repeat the test 6 times with 50 μL each of the standard solution according to the above operating conditions; the relative standard deviation of the peak areas of formic acid is NMT 2.0%.

(5) ***2-pyrrolidone***—Weigh accurately about 0.5 g of Povidone, dissolve in the mobile phase to make exactly 100 mL, and use this solution as the test solution. Separately, weigh accurately about 0.15 g of 2-pyrrolidone, and dissolve in the mobile phase to make exactly 100 mL. Pipet 2 mL of this solution, add the mobile phase to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 50 μL each of the test solution and the standard solution as directed under the Liquid Chromatography according to the following conditions. Determine the peak area of 2-pyrrolidone in each solution as directed under the automatic integration method: the amount of 2-pyrrolidone is NMT 3.0%.

$$\text{Amount (\%) of 2-pyrrolidone} = (A_T / A_S) \times (C_S / C_T) \times 100$$

A_T: Peak area of 2-pyrrolidone in the test solution

A_S: Peak area of 2-pyrrolidone in the standard solution

C_S: Concentration (mg/mL) of 2-pyrrolidone in the standard solution

C_T: Concentration (mg/mL) of povidone in the test solution, calculated on the anhydrous basis.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 205 nm)

Column: A stainless steel column about 4.6 mm in internal diameter and 15 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter). Use a stainless steel column about 4 mm in internal diameter and 1.0 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter) as a guard column.

Column temperature: A constant temperature of about 40 ℃

Mobile phase: A mixture of water and methanol (19 : 1)

Flow rate: 0.8 mL/min (Retention time of 2-pyrrolidone: about 7 minutes)

System suitability

System performance: Proceed with 50 μL of the standard solution under the above operating conditions; the number of theoretical plates and symmetry factor of the peak of 2-pyrrolidone are NLT 5000 and NMT 1.5, respectively.

System repeatability: Perform the test 6 times with 50 μL each of the standard solution according to the above operating conditions; the relative standard deviation of the peak areas of 2-pyrrolidone is NMT 2.0%.

(6) ***Hydrazine***—Place 2.5 g of Povidone in a centrifuge tube, and add 25 mL of water to dissolve. Add 500 μL of a solution of salicylaldehyde in methanol (1 in 20), shake to mix, and allow to stand for 15 minutes on a water bath at 60 ℃. Allow to cool, add 2.0 mL of toluene, stopper, and shake vigorously for 2 minutes. Centrifuge, take the upper toluene layer, and use this solution as the test solution Separately, dissolve 90 mg of salicylaldazine in toluene to make exactly 100 mL. Pipet 1 mL of this solution, add toluene to make exactly 100 mL, and use this solution as the standard solution. With these solutions, perform the test as directed under the Thin Layer Chromatography. Spot 10 μL each of the test solution and the standard solution on a thin-

layer chromatographic plate of 0.25 mm in thickness made of silica gel for thin-layer chromatography (with fluorescent indicator). Develop the plate with diluted methanol (2 in 3) as the developing solvent to about 3/4, and air dry the plate. Examine under ultraviolet light (main wavelength: 365 nm): the R_f value of the fluorescent spot obtained from the standard solution is about 0.3 and the fluorescence of the spot obtained from the test solution corresponding to the location of the spot from the standard solution is not more intense than that of the spot from the standard solution (NMT 1 ppm).

Water NMT 5.0% (0.5 g, volumetric titration, direct titration)

Residue on ignition NMT 0.1% (1 g).

K-Value Weigh accurately an amount of Povidone, equivalent to the amount specified in the following table calculated on the anhydrous basis according to the nominal K-value and dissolve in water to make exactly 100 mL, allow to stand for 60 minutes and use this solution as the test solution. Perform the test with the test solution and water at 25 °C according to Method 1 under the Viscosity. Calculate the K-value by the following formula: in the case of the nominal K-value NMT 15, 85.0 to 115.0% of the labeled value is acceptable, and in the case of the nominal K-value more than 15, 90.0 to 108.0% of the labeled value is acceptable.

Nominal K-value	Mass (g) of Povidone, calculated on the anhydrous basis
NMT 18	5.00
> 18 and NMT 95	1.00
> 95	0.1.

$$K = \frac{1.5 \log \upsilon_{re1} - 1}{0.15 + 0.003_c} + \frac{\sqrt{300c \log \upsilon_{re1} + (c + 1.5c \log \upsilon_{re1})^2}}{0.15_c + 0.003c^2}$$

c: Mass (g) of Povidone in 100 mL of the solution, calculated on the anhydrous basis

v_{rel}: Ratio of the kinematic viscosity of the test solution to that of water

Assay Weigh accurately about 0.1 g of Povidone and place in a Kjeldahl flask. Add 5 g of a mixture of potassium sulfate, copper sulfuric acid (II) pentahydrate and titanium(IV) oxide (33 : 1 : 1), wash down any adhering sample from the neck of the flask with a small amount of water, and add 7 mL of sulfuric acid allowing to flow down the inside wall of the flask. Heat the flask gradually until the solution has a clear, yellowish green color and the inside wall of the flask is free from a carbonaceous material and then heat continuously for further 45 minutes. After cooling, add cautiously 20 mL of water, cool the solution and connect the flask to the distillation apparatus, and perform the test as directed under the Nitrogen Determination (Semimicro-Kjeldahl Method). However, in the procedure, add 30 mL of boric acid solution (1 in 25) and use 0.025 mol/L sulfuric acid VS for titration.

Each mL of 0.025 mol/L sulfuric acid VS
= 0.700 mg of N

Packaging and storage Preserve in tight containers.

Pregelatinized Starch
전호화전분

Pregelatinized Starch is a starch prepared by gelatinizing natural starch mass while treating all or part of the starch mass with water chemically or mechanically.

Description Pregelatinized Starch occurs as a white or almost white fine powder.
It is odorless and has a characteristic taste.
It is sparingly soluble in cold water, and practically insoluble in ethanol.

Identification Add 50 mL of water to 1 g of Pregelatinized Starch, boil for 1 minute and cool; a pale-white, turbid, viscous solution is formed. Take 1 mL of this solution, and add 0.05 mL of diluted iodine TS (1 in 10); the solution exhibits an orange to dark bluish purple color, and the color disappears when heated.

pH Dissolve about 10 g of Pregelatinized Starch in 10 mL of ethanol, and add water to make 100 mL. After stirring for 5 minutes, and immediately measure; the pH is 4.5 to 7.0.

Loss on drying NMT 14.0% (1 g, 120 °C, 4 hours).

Residue on ignition NMT 0.5% (2 g)

Purity (1) ***Iron*—**After the Residue on ignition test, add 8 mL of hydrochloric acid to the entire sample, dissolve it by slowly heating, and add water to make 100 mL. Pipet 25 mL of this solution, add water to make 47 mL and use this solution as the test solution. Separately, add water to 1 mL of iron standard solution to make 45 mL, and mix with 2 mL of hydrochloric acid, and use this solution as the control solution. To the test solution and the control solution, add 50 mg of sodium peroxydisulfate and 3 mL of ammonium thiocyanate TS, and shake to mix; the color of the test solution is not more intense than that of the control solution (NMT 20 ppm).

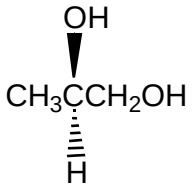
(2) ***Sulfur dioxide*—**Add 20 g of Pregelatinized Starch to 200 mL of anhydrous sodium sulfate solution (1 in 5), shake to mix, and filter. Add 3.0 mL of starch TS as an indicator to 100 mL of the filtrate, and titrate with 0.01 mol/L iodine VS until a blue color persists. Perform a blank test in the same manner and make any necessary correction. The amount of 0.01 mol/L iodine solution consumed is NMT 2.7 mL (NMT 80 ppm).

(3) ***Oxidizing material*—**Weigh 5 g of Pregelatinized Starch and add it to 20 mL of a mixture of methanol and water (1 : 1), add 1 mL of 6 mol/L acetic acid TS, and stir until the suspension becomes homogeneous. Add 0.5 mL of saturated potassium iodide solution, newly prepared, and allow it to stand for 5 minutes; the resulting solution does not exhibit a blue, brown or violet color.

Microbial limit The total number of aerobic microorganisms is NMT 1000 CFU, and the total number of fungi is NMT 100 CFU per 1 g of Pregelatinized Starch. Also, *Salmonella* and *Escherichia coli (E.coli)* are not detected.

Packaging and storage Preserve in well-closed containers.

Propylene Glycol
프로필렌글리콜



and enantiomer

$C_3H_8O_2$: 76.09

(2*RS*)-Propane-1,2-diol [*57-55-6*]

Description Propylene Glycol occurs as a clear, colorless, viscous liquid, is odorless and has a slightly bitter taste.
It is miscible with water, methanol, ethanol, or pyridine.
It is freely soluble in ether.
It is hygroscopic.

Identification (1) Mix 2 to 3 drops of Propylene Glycol with 0.7 g of triphenylchloromethane, add 1 mL of pyridine and heat on a water bath under a reflux condenser for 1 hour. After cooling, dissolve the mixture in 20 mL of acetone by warming, shake with 20 mg of activated charcoal and filter. Concentrate the filtrate to about 10 mL and cool. Collect the separated crystals by filtering and dry in a desiccator (silica gel) for 4 hours: the melting point is 174 °C to 178 °C.

(2) Heat gently 1 mL of Propylene Glycol with 0.5 g of potassium hydrogen sulfate: a characteristic odor is perceptible.

(3) The retention time of the major peak obtained from the test solution in the Purity (8) is the same as that of the major peak from the standard solution.

Specific gravity d_{20}^{20}: Between 1.035 and 1.040.

Purity (1) ***Acid*—**Mix 10.0 mL of Propylene Glycol with 50 mL of freshly boiled and cooled water and add 5 drops of phenolphthalein TS and 0.30 mL of 0.1 mol/L sodium hydroxide: the solution exhibits a red color.

(2) ***Chloride*—**Perform the test with 2.0 g of Propylene Glycol. Prepare the control solution with 0.40 mL of 0.01 mol/L hydrochloric acid (NMT 0.007%).

(3) ***Sulfate*—**Perform the test with 10.0 g of Propylene Glycol. Prepare the control solution with 0.40 mL of 0.005 mol/L (NMT 0.002%).

(4) ***Heavy metals***—Proceed with 5.0 g of Propylene Glycol according to Method 1 and perform the test. Prepare the control solution with 2.5 mL of lead standard solution (NMT 5 ppm).

(5) ***Lead***—Weigh accurately 5.0 g of Propylene Glycol and transfer to a platinum crucible. Dry, carbonize and incinerate at 450 °C to 550 °C. If incineration is not achieved, cool and moisten with 2 to 5 mL of nitric acid (1 in 2), 50% magnesium nitrate solution or aluminum nitrate-calcium nitrate solution (dissolve 40 g of aluminum nitrate and 20 g of calcium nitrate in 100 mL of water) as an incineration agent, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 to 5 mL of nitric acid (1 in 2) and incinerate. After incineration is complete, moisten the residue with water, add 2 to 4 mL of hydrochloric acid and evaporate to dryness. Add 0.5 mol/L nitric acid, dissolve by warming and filter any insoluble matter with filter paper. Unless otherwise specified, add 0.5 mol/L nitric acid to make 25 mL and use this solution as the test solution. Separately, proceed with 1.0 mL of lead standard solution in a platinum crucible in the same manner as the test solution, and use this solution as the standard solution. Perform the test with the test solution and the standard solutions as directed under the Atomic Absorption Spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 2.0 ppm).

Gas: Acetylene or hydrogen - Air.
Lamp: Lead hollow-cathode lamp
Wavelength: 283.3 nm

(6) ***Arsenic***—Proceed with 1.0 g of Propylene Glycol according to Method 1, and perform the test (NMT 2 ppm).

(7) ***Glycerin***—Heat 1.0 g of Propylene Glycol with 0.5 g of potassium hydrogen sulfate and evaporate to dryness: no odor of acrolein is perceptible.

(8) ***Ethylene glycol and diethylene glycol***—Weigh accurately suitable amounts of Propylene Glycol and the internal standard, dissolve by adding methanol, and prepare a solution containing 50 mg of propylene glycol and 0.10 mg of the internal standard per mL, and use this solution as the test solution. Separately, weigh suitable amounts of propylene glycol RS, ethylene glycol RS, diethylene glycol RS and the internal standard, dissolve in methanol to make a solution containing 2.0 mg, 0.050 mg, 0.050 mg and 0.10 mg per mL, respectively, and use this solution as the standard solution. Perform the test with 1.0 μL each of the test solution and the standard solution as directed under the Gas Chromatography according to the following conditions. Determine the peak areas of each solution by the automatic integration method: the peak area ratio of diethylene glycol to the internal standard obtained from the test solution is NMT the peak area ratio of diethylene glycol to the internal standard from the standard solution (NMT 0.10%), and the peak area ratio of ethylene glycol to the internal standard obtained from the test solution is NMT the peak area ratio of ethylene glycol to the internal standard from the standard solution (NMT 0.10%).

Internal standard—2,2,2-trichloroethanol

Operating conditions

Detector: A flame ionization detector

Column: A quartz glass column 0.53 mm in internal diameter and about 30 m in length, coated the inside with cyanopropylphenyl dimethylpolysiloxane for gas chromatography (6:94) in a 3.0 μm thickness.

Column temperature: Maintain the temperature at 100 °C for 4 minutes, then raise to 120 °C at a rate of 50 °C per minute, and maintain it for 10 minutes. Raise the temperature again to 220 °C at a rate of 50 °C per minute and maintain it at 220 °C for 6 minutes.

Sample injection port temperature: A constant temperature of about 220 °C

Detector temperature: A constant temperature of about 250 °C

Carrier gas: Helium
Flow rate: 4.5 mL/min
Split ratio: About 1 : 10

System suitability

System performance: Proceed with 1 μL of the standard solution according to the above operating conditions; the resolution between the peaks of ethylene glycol and propylene glycol is NLT 5. The relative retention times of ethylene glycol, propylene glycol, 2,2,2-trichloroethanol and diethylene glycol are 0.8, 1.0, 1.7 and 2.4, respectively, and the retention time of propylene

glycol is about 4 minutes

Water NMT 0.5% (2 g, volumetric titration, direct titration).

Residue on ignition Weigh accurately about 20 g of Propylene Glycol in a crucible, previously weighed, and heat to boiling. Stop heating and immediately ignite to burn. Cool, moisten the residue with 0.2 mL of sulfuric acid, and ignite with care to a constant mass: the amount of residue is NMT 0.005%.

Distilling range Between 184 °C and 189 °C, NLT 95 vol%.

Packaging and storage Preserve in tight containers.

Pyroxylin
피록실린

Pyroxylin is a nitric acid ester of cellulose. It is usually moistened with isopropanol or other appropriate solvent.

Description Pyroxylin occurs as white and comes in flocculent or flaky forms.
It is freely soluble in acetone and very slightly soluble in ether.
It is decomposed by heat or light, with evolution of nitrous acid vapors.

Identification Ignite Pyroxylin; it burns very rapidly with a bright flame.

Purity (1) ***Clarity and color of solution*—**Dissolve 1.0 g of Pyroxylin, previously dried at 80 °C for 2 hours, in 25 mL of a mixture of ether and ethanol (3 : 1): the solution is clear.

(2) ***Acid*—**To 1.0 g of Pyroxylin, previously dried at 80 °C for 2 hours, add 20 mL of water, shake well to mix for 10 minutes and filter: the filtrate is neutral.

(3) ***Water-soluble substances*—**Evaporate 10 mL of the filtrate obtained in (2) on a water bath to dryness and dry at 105 °C for 1 hour: the amount of the residue is NMT 1.5 mg.

(4) ***Residue on ignition*—**Weigh accurately about 2 g of Pyroxylin, previously dried at 80 °C for 2 hours, and moisten with 10 mL of a solution of castor oil in acetone (1 in 20) to gelate the sample. Ignite the contents to carbonize the sample, ignite at about 500 °C for 2 hours and allow to cool in a desiccator (silica gel): the amount of the residue is NMT 0.30%.

Packaging and storage Preserve in light-resistant, tight containers, packed loosely, away from fire, and preferably in a cold place.

Rape Seed Oil
채종유

Rape Seed Oil is a fatty oil obtained from the seeds of rapeseed (*Brassica campestris* Linné subsp). *napus* Hooker fi1. et Anderson var. *nippo-oleifera* Makino (Cruciferae).

Description Rape Seed Oil occurs as a clear, pale yellow, slightly viscous oil. It is odorless or has a slight odor and mild taste.
It is miscible with ether and petroleum ether.
It is slightly soluble in ethanol.

Specific gravity d_{25}^{25}: Between 0.906 and 0.920

Saponification value Between 169 and 195.

Unsaponifiable matter NMT 1.5%

Acid value NMT 0.2

Iodine value Between 95 and 127

Packaging and storage Preserve in tight containers.

Rice Starch
쌀전분

Rice Starch is a starch obtained from grains of *Oryza sativa* Linné (Gramineae).

Description Rice Starch is a very fine white or almost white powder that makes a crisp sound when pressed with a finger.
It is practically insoluble in cold water or ethanol (95).
It does not contain starch granules of other origins.
It sometimes contains small amounts of tissue fragments from the original plant.

Identification (1) Drop a mixture of water and glycerin (1 : 1) onto Rice Starch, and observe it under the microscope with a magnification of NLT 20 x; it is a polygon of 1 to 10 μm, mostly composed of grains of 4 to 6 μm in size, and sometimes gathers together to form oval-shaped composite grains of 50 to 100 μm in diameter, without helium or lamination. Rice Starch shows a clear black cross crossing at the hilum between crossed polarizing plates or polarizing prisms.

(2) Boil Rice Starch in 50 mL of water for 1 minute, and cool the solution; it becomes a pale white, turbid, and viscous liquid.

(3) Add 0.05 mL of dilute iodine TS to the paste of Rice Starch obtained in (2); the resulting solution exhibits orange to dark bluish violet, and the color disappears when heated.

Iodine TS—Add water to 12.7 g of iodine and 20 g of potassium iodide to make 1000 mL. Pipet 10 mL of this solution, add 0.6 g of potassium iodide, and add water to make 100 mL. Prepare before use.

pH Place 5.0 g of Rice Starch into a non-metal container, add 25.0 mL of freshly boiled and cooled water, shake gently for 1 minute to make a suspension, and allow it to stand for 15 minutes; the pH of the resulting solution is 5.0 to 8.0.

Purity (1) ***Iron***—Add 15 mL of 2 mol/L hydrochloric acid TS to 1.5 g of Rice Starch, shake to mix, filter, and use the filtrate as the test solution. Add water to 2.0 mL of the iron standard solution to make 20 mL, and use this solution as the control solution. Place 1.0 mL each of the test solution and the control solution into test tubes, add 2 mL of citric acid solution (1 in 5) and 0.1mL of mercaptoacetic acid solution, and mix. Add strong ammonia water to these solutions until the litmus paper exhibits alkalinity, add water to make 20 mL, and mix. Add 10 mL each of these solution to test tubes, allow to stand, and compare each other against a white background; the color of the test solution is not more intense than that of the control solution (NMT 10 ppm).

(2) ***Oxidizing substances***—Add 50.0 mL of water to 4.0 g

of Rice Starch, shake for 5 minutes, and separate the solution by centrifugation. Add 1 mL of acetic acid (100) and 0.5 to 1.0 g of potassium iodide to 30.0 mL of the clear supernatant, shake to mix, and allow the mixture to stand for 25 to 30 minutes in a dark place. Add 1 mL of starch TS, and titrate with 0.002 mol/L sodium thiosulfate VS until the solution turns colorless. Perform a blank test in the same manner and make any necessary correction. The consumed volume of 0.002 mol/L sodium thiosulfate VS is NMT 1.4 mL (NMT 20 ppm, calculated as hydrogen peroxide).

Each mL of 0.002 mol/L sodium thiosulfate VS
= Oxidizing substances converted to 34 μg of hydrogen peroxide

(3) ***Sulfur dioxide***—NMT 50 ppm when tested.

(4) ***Foreign matter***—Under the microscope, Rice Starch does not show other starch. It may contain a minute quantity, if any, of fragments of the tissue of the original plant.

Loss on drying NMT 15.0% (1 g, 130 ℃, 90 minutes).

Residue on ignition NMT 0.6% (1 g).

Microbial limit Perform the microbial limit test; the total aerobic microbial count is NMT 103 CFU and the total combined yeasts/mold count is NMT 102 CFU per 1 g of Rice Starch. No escherichia coli (E.coli) or salmonella is detected.

Packaging and storage Preserve in well-closed containers.

Rosin
송지

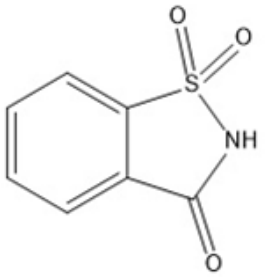
Rosin is the resin obtained from the exudation of plants of Pinus species (Pinaceae), from which essential oils have been removed.

Description Rosin is a pale yellow to pale brown, glassily transparent, brittle mass, and its surface is sometimes covered with a yellow powder, and the fractured surface forms a shell-like pattern with a luster.
It has a slight odor.
It melts easily and burns with a yellowish brown flame.
It is freely soluble in ethanol, acetic acid (100) or ether.
A solution of Rosin in ethanol is acidic.

Acid value Between 150 and 177

Ash NMT 0.1%.

Saccharin
사카린



$C_7H_5NO_3S$: 183.18
1,2-Benzisothiazol-3(2*H*)-one, 1,1-dioxide [*81-07-2*]

Saccharin contains NLT 99.0% and NMT 101.0% of saccharin ($C_7H_5NO_3S$: 183.18), calculated on the dried basis.

Description Saccharin occurs as a colorless to white crystal or crystalline powder with a very sweet taste.
It is sparingly soluble in ethanol and slightly soluble in water.
It is soluble in sodium hydroxide TS.

Identification (1) Determine the infrared spectra of Saccharin and saccharin RS as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

Melting point Between 170 ℃ and 230 ℃

Purity (1) ***Clarity and color of solution***—Weigh accurately about 5.0 g of Saccharin, dissolve in 25 mL of sodium acetate solution (1 in 5), and use this solution as the test solution. Observe this solution on a black background to compare the colors; the test solution is as clear as water or sodium acetate solution (1 in 5) and is less turbid than the control suspension (1). Prepare the control suspension (1), allow it to stand for 5 minutes, and compare with the test solution; The turbidity of the control suspension (1) must be sufficiently distinguishable from that of water and the control suspension (2). The test tube containing the test solution and the reference solution must be identical, colorless, transparent, flat-bottomed and made of glass with a diameter of 15 to 25 mm and a depth of 40 mm.

Control suspension—Pipet 25 mL of hydrazinium sulfate TS left for 4 to 6 hours, mix it with a solution containing 2.5 g of hexamethyltetramine dissolved in 25 mL of water, mix well, and allow the mixture to stand for 24 hours. Store the solution in a glass container and use it within 2 months. Before use, pipet 15 mL of this suspension, add water to make 1000 mL, and use this solution as the stock suspension. Pipet 5 mL of the stock suspension, add water to make exactly 100 mL, and use this solution as the control suspension (1). Pipet 5 mL of the stock suspension, add water to make exactly 50 mL, and use this solution as the control suspension (2).
Observe the test solution for the Clarity and color of solution on a white background; the test solution is as clear as water or sodium acetate solution (1 in 5) and is not more intense than the following control solution.

Control solution—Add hydrochloric acid solution (1 in 100) to a mixture of 3.0 mL of the colorimetric stock solution of iron(III) chloride hexahydrate, 3.0 mL of the colorimetric stock

solution of cobalt(II) chloride hexahydrate, and 2.4 mL of the colorimetric stock solution of copper(II) sulfate pentahydrate to make 10 mL. Before use, pipet 1 mL of this solution, and add hydrochloric acid solution (1 in 100) to make exactly 100 mL.

(2) ***Heavy metals***—Weigh 2.0 g of Saccharin and perform the test according to Method 2 as directed under the Heavy metals. Prepare the control solution by adding 2.0 mL of the lead standard solution (NMT 10 ppm).

(3) ***Benzoate and salicylate***—Add 1 drop each of iron(III) chloride TS to 10 mL of heated saturated aqueous solution of Saccharin; no precipitate is formed, and no purple color appears.

(4) ***Toluenesulfonamide***—Weigh accurately about 10.0 g of Saccharin, add it to 20 mL of water, and dissolve in 5 to 6 mL of 10 mol/L sodium hydroxide TS. If necessary, adjust the pH to 7 to 8 with 1 mol/L sodium hydroxide TS or 1 mol/L hydrochloric acid, and add water to make 50 mL. Extract this solution 4 times with 50 mL each of methylene chloride. Combine all extracts, dry with anhydrous sodium sulfate, filter, and wash the filter paper and sodium sulfate with 10 mL of methylene chloride. Combine the washings and the filtrate, and evaporate to dryness on a water bath at NMT 40 °C. Add a small amount of methylene chloride to the residue, transfer it to an appropriate 10-mL tube, evaporate to dryness under a nitrogen stream, and dissolve the residue in 1.0 mL of the internal standard solution. Use this solution as the test solution. Separately, weigh accurately about 20.0 mg each of *o*-toluenesulfonamide and *p*-toluenesulfonamide, and dissolve in methylene chloride to make exactly 100 mL. Pipet 5.0 mL of this solution, and add methylene chloride to make exactly 50 mL. Evaporate 5.0 mL of this solution to dryness under a nitrogen stream, and dissolve the residue in 1 mL of the internal standard solution. Use this solution as the standard solution. Separately, evaporate 200 mL of methylene chloride to dryness on a water bath at NMT 40 °C, and dissolve the residue in 1 mL of methylene chloride. Use this solution as the blank test solution. Perform the test with 1 μL each of the test solution and the standard solution as directed under the Gas Chromatography according to the following conditions, and determine the ratios, QT and QS, of the peak areas of *o*-toluenesulfonamide and *p*-toluenesulfonamide to that of the internal standard, respectively; each QT is not greater than each QS (NMT 10 ppm each).

Internal standard solution—Methylene chloride solution of caffeine (1 in 4000).

Operating conditions

Detector: A flame ionization detector

Column: A fused silica column about 0.53 mm in internal diameter and about 10 m in length, coated inside with 50% phenyl-50% methylpolysiloxane for gas chromatography, 2 μm in thickness.

Column temperature: A constant temperature of about 180 °C.

Sample injection port temperature: A constant temperature of about 250 °C.

Detector temperature: A constant temperature of about 250 °C.

Carrier gas: Nitrogen

Flow rate: 10 mL/min

Split ratio: 2 : 1

System suitability

System performance: Perform the test with 1 μL of the standard solution according to the above conditions; *o*-toluenesulfonamide, *p*-toluenesulfonamide, and the internal standard are eluted in this order, and the resolution between the *o*-toluenesulfonamide and *p*-toluenesulfonamide peaks is NLT 1.5. Proceed with 1 μL of the blank test solution according to the above conditions; no peaks appear in the retention times of *o*-toluenesulfonamide, *p*-toluenesulfonamide, and the internal standard.

(5) ***Readily carbonizable substances***—Weigh 0.20 g of Saccharin and perform the test. However, allow the solution to stand at 48 to 50 °C for 10 minutes; the color of the solution is not more intense than the Matching fluids A.

Loss on drying NMT 1.0% (1 g, 105 °C, 2 hours).

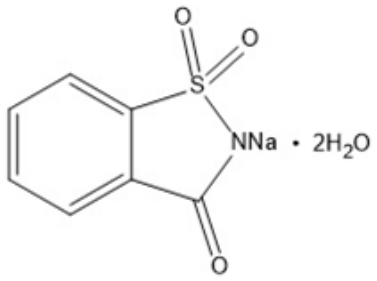
Residue on ignition NMT 0.2% (1 g).

Assay Weigh accurately about 0.5 g of Saccharin, dissolve in 40 mL of ethanol (95), add 40 mL of water to mix, and titrate with 0.1 mol/L sodium hydroxide VS (indicator: 3 drops of phenolphthalein TS). Perform a blank test in the same manner and make any necessary correction.

Each mL of 0.1 mol/L sodium hydroxide VS
= 18.32 mg of $C_7H_5NO_3S$

Packaging and storage Preserve in well-closed containers.

Saccharin Sodium Hydrate
사카린나트륨수화물



$C_7H_4NNaO_3S \cdot 2H_2O$: 241.20

1,2-Benzisothiazol-3(2*H*)-one 1,1-dioxide, sodium salt, dihydrate [*6155-57-3*]

Saccharin Sodium Hydrate contains NLT 99.0% and NMT 101.0% of saccharin sodium ($C_7H_4NNaO_3S$: 205.17), calculated on the anhydrous basis.

Description Saccharin Sodium Hydrate occurs as a colorless crystal or white crystalline powder with a very sweet taste that remains sweet even in 10000 times aqueous solution.
It is freely soluble in water or methanol and sparingly soluble in ethanol (95) or acetic acid (100).
It decomposes slowly in the air and loses about half of its water of crystallization.

Identification (1) Weigh accurately about 1.0 g of Saccharin Sodium Hydrate, and dissolve in 10 mL of water to use as the test solution. Add 2 mL of 15% potassium carbonate solution to 10 mL of this solution, and heat until boiling; no precipitate is formed. Add 4 mL of potassium hexahydroxoantimonate(V) TS to this solution, heat until boiling, and cool with iced water (scrape the inner wall with a glass rod if necessary); a high-density precipitate is formed.

Potassium hexahydroxoantimonate(V) TS: Dissolve 2 g of potassium pyroantimonate in 95 mL of boiling water, and cool quickly. Add 50 mL of potassium hydroxide solution (50 mg/mL) and 1 mL of sodium hydroxide TS (8.5 in 100) to this solution, allow it to stand for 24 hours, filter, and add water to make 150 mL.

(2) Dry Saccharin Sodium Hydrate and saccharin sodium hydrate RS at 105°C to a constant mass, and determine the absorbance as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

(3) The aqueous solution of Saccharin Sodium Hydrate (1 in 10) responds to the Chemical identification reactions (1) for sodium salt.

Purity (1) ***Clarity and color of solution***—Weigh accurately about 5.0 g of Saccharin Sodium Hydrate, dissolve in 25 mL of water, and use this solution as the test solution. Observe this solution against a black background, and compare the color of the solutions; the test solution is as clear as water and is less turbid than the control suspension (1). Prepare the control suspension (1), allow it to stand for 5 minutes, and compare with the test solution; the turbidity of the control suspension (1) must be sufficiently distinguishable from that of water and the control suspension (2). The test tube containing the test solution and the reference solution must be identical, colorless, transparent, flat-bottomed and made of glass with a diameter of 15 to 25 mm and a depth of 40 mm.

Control suspension—Pipet 25 mL of hydrazinium sulfate TS left for 4 to 6 hours, mix it with a solution containing 2.5 g of hexamethyltetramine dissolved in 25 mL of water, mix well, and allow the mixture to stand for 24 hours. Store the solution in a glass container and use it within 2 months. Before use, pipet 15 mL of this suspension, add water to make exactly 1000 mL, and use this as the stock suspension. Pipet 5 mL of this stock suspension, add water to make exactly 100 mL, and use this solution as the control suspension (1). Separately, pipet 5 mL of the stock suspension, add water to make exactly 50 mL, and use this solution as the control suspension (2).

Color Proceed with the test solution for the turbidity test, and observe it on a white background; the test solution is as clear as water and is not more intense than the control solution.

Control solution: Add hydrochloric acid solution (1 in 100) to a mixture of 3.0 mL of the colorimetric stock solution of iron(III) chloride hexahydrate, 3.0 mL of the colorimetric stock solution of cobalt(II) chloride hexahydrate, and 2.4 mL of the colorimetric stock solution of copper(II) sulfate pentahydrate to make 10 mL. Before use, pipet 1 mL of this solution, and add hydrochloric acid solution (1 in 100) to make exactly 100 mL.

(2) ***Acidity or alkalinity***—Dissolve 1.0 g of Saccharin Sodium Hydrate in 10 mL of water, and add 1 drop of phenolphthalein TS; the resulting solution is colorless. Add 1 drop of 0.1 mol/L sodium hydroxide solution; the resulting solution turns red.

(3) ***Heavy metals***—Dissolve 2.0 g of Saccharin Sodium Hydrate in 40 mL of water, add 0.7 mL of dilute hydrochloric acid and water to make 50 mL. Scrape the wall of the vessel with a glass rod. When crystals begin to precipitate, allow it to stand for 1 hour. Next, filter with a dry filter paper, discard the first 10 mL of the filtrate, and add 2 mL of dilute acetic acid and water to the following 25 mL of the filtrate to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution by adding 2 mL of dilute acetic acid and water to 1.0 mL of the lead standard solution to make 50 mL (NMT 10 ppm).

(4) ***Benzoate and salicylate***—Dissolve 0.5 g of Saccharin Sodium Hydrate in 10 mL of water, and add 5 drops of 6 mol/L acetic acid and 3 drops of iron(III) chloride TS; no precipitate is formed, and the resulting solution does not exhibit a purple color.

(5) ***Toluenesulfonamide***—Weigh accurately about 10.0 g of Saccharin Sodium Hydrate, and dissolve in 50 mL of water. If necessary, adjust the pH to 7 to 8 with 1 mol/L sodium hydroxide TS or 1 mol/L hydrochloric acid before final dilution. Extract 50 mL of this solution 4 times with 50 mL each of methylene chloride. Combine all extracts, dry with anhydrous sodium sulfate, filter, and wash the filter paper and sodium sulfate with 10 mL of methylene chloride. Combine the washings and the filtrate, and evaporate to dryness on a water bath at NMT 40 °C. Add a small amount of methylene chloride to the residue, transfer the mixture to an appropriate 10-mL tube, evaporate to dryness under a nitrogen stream, and dissolve the residue in 1.0 mL of the internal standard solution; use this solution as the test solution. Separately, weigh accurately about 20.0 mg each of *o*-toluenesulfonamide and *p*-toluenesulfonamide, and dissolve in methylene chloride to make exactly 100 mL. Pipet 5.0 mL of this solution, and add methylene chloride to make exactly 50 mL. Evaporate 5.0 mL of this solution to dryness under a nitrogen stream, and dissolve the residue in 1 mL of the internal standard solution; use this solution as the standard solution. Separately, evaporate 200 mL of methylene chloride to dryness on a water bath at NMT 40 °C, and dissolve the residue in 1 mL of methylene chloride; use this solution as the blank test solution. Perform the test with 1 μL each of the test solution and the standard solution as directed under the Gas Chromatography according to the following conditions, and determine the ratios, Q_T and Q_S, of the peak areas of *o*-toluenesulfonamide and *p*-toluenesulfonamide to that of the internal standard, respectively; each Q_T is not greater than each Q_S (NMT 10 ppm each).

Internal standard solution—Methylene chloride solution of caffeine (1 in 4000).

Operating conditions

Detector: A flame ionization detector

Column: A fused silica column about 0.53 mm in internal diameter and about 10 m in length, coated inside with 50% phenyl-50% methylpolysiloxane for gas chromatography, 2 μm in thickness.

Column temperature: A constant temperature of about 180 °C.

Sample injection port temperature: A constant temperature of about 250 °C.

Detector temperature: A constant temperature of about 250 °C.

Carrier gas: Nitrogen

Flow rate: 10 mL/min

Split ratio: 2 : 1

System suitability

System performance: Perform the test with 1 μL of the standard solution according to the above conditions; o-toluenesulfonamide, p-toluenesulfonamide, and the internal standard are eluted in this order, and the resolution between the o-toluenesulfonamide and p-toluenesulfonamide peaks is NLT 1.5. Proceed with 1 μL of the blank test solution according to the above conditions; no peaks appear in the retention times of *o*-

toluenesulfonamide, *p*-toluenesulfonamide, and the internal standard.

(6) ***Readily carbonizable substances***—Perform the test with 0.20 g of Saccharin Sodium Hydrate. Allow the solution to stand at 48 to 50 °C for 10 minutes; the color of the solution is not more intense than the control solution A.

Water NMT 15.0% (0.1 g, volumetric titration, direct titration).

Assay Weigh accurately about 0.15 g of Saccharin Sodium Hydrate, add 100 mL of acetic acid (100), heat gently to dissolve if necessary, and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration under the Titrimetry). Perform a blank test in the same manner and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 20.52 mg of $C_7H_4NNaO_3S$

Packaging and storage Preserve in well-closed containers.

Sesame Oil
참기름

Oleum Sesami

Sesame Oil is a fatty oil obtained from the seeds of *Sesamum indicum* Linné (Pedaliaceae).

Description Sesame Oil occurs as a clear, pale yellow oil. It is odorless or has a slight characteristic odor, and has a mild taste.
It is miscible with ether, chloroform, petroleum ether or carbon disulfide.
It is slightly soluble in ethanol.
It congeals at between 0 ℃ and -5 ℃.
Congealing point of fatty acids: Between 20 ℃ and 25 ℃

Identification To 1 mL of Sesame Oil, add 0.1 g of sucrose and 10 mL of hydrochloric acid, and shake for 30 seconds to mix; the acid layer becomes pale red and turns red if allowed to stand.

Saponification value Between 187 and 194

Unsaponifiable matter NMT 2.0%

Specific gravity d_{25}^{25}: Between 0.914 and 0.921

Acid value NMT 0.2

Iodine value Between 103 and 118

Cottonseed oil Put 5 mL of Sesame Oil in a test tube, add 5 mL of a mixture of amyl alcohol and a solution of sulfur in carbon disulfide (1 in 100) (1 : 1), and mix. Carefully warm this solution until carbon disulfide is released, then submerge one third of the test tube from the bottom into a boiling saturated sodium chloride solution; it does not exhibit a red color within 15 minutes.

Triglyceride composition Weigh accurately about 0.2 g of Sesame Oil, transfer to a 10-mL volumetric flask, dissolve with the mobile phase, and use this solution as the test solution. Perform the test with 20 μL of the test solution as directed under the liquid chromatography according to the following conditions, and determine the major peak areas of the eight glycerides. Determine their amounts according to the area percentage; trilinolein is 7.0% to 19.0%, 1,2-dilinoleoyl-3-oleoyl-rac-glycerol is 13.0% to 30.0%, 1,2-dilinoleoyl-3-palmitoyl-rac-glycerol is 5.0% to 9.0%, 1,2-dioleoyl-3-linoleoyl-rac-glycerol is 14.0% to 25.0%, 1-palmitoyl-2-oleoyl-3-linoleoyl-rac-glycerol is 8.0% to 16.0%, triolein 5.0% to 14.0%, 1-linoleoyl-2-oleoyl-3-stearoyl-rac-glycerol 2.0% to 8.0%, 1,2-dioleoyl-3-palmitoyl -rac-glycerol is 2.0% to 8.0%.

Operating conditions

Detector: A differential refractometer

Column: Two stainless steel columns about 4.6 mm in internal diameter and about 250 mm in length, packed with octadecylsilanized silica gel for liquid chromatography.

Column temperature: A constant temperature of about 30 °C

Mobile phase: A mixture of acetonitrile and dichloromethane (60 : 40)

Flow rate: 1.0 mL/min

Time span of measurement: About 40 minutes after the solvent peak

System suitability

System performance: Dissolve 1,2-dioleoyl-3-linoleoyl-rac-glycerol RS and 1,2-dilinoleoyl-3-palmitoyl-rac-glycerol RS in the mobile phase so that each mL contains 3 mg. Proceed with 20 μL of this solution according to the above conditions; the relative retention time of 1,2-dioleoyl-3-linoleoyl-rac-glycerol is 0.93, the relative retention time of 1,2-dilinoleoil-3-palmitoyl-rac-glycerol is 1.0, and the resolution is NLT 1.8.

System repeatability: Repeat the test 6 times with 20 μL each of the test solution according to the above conditions; the relative standard deviation of the peak areas is NMT 1.5%, and the ratio of peak area of 1,2-dioleoyl-3-linoleoyl-rac-glycerol to the that of 1,2-dilinoleoyl-3-palmitoyl-rac-glycerol is NMT 2.2%.

Relative retention time: The relative retention times of trilinolein, 1,2-dilinoleoyl-3-oleoyl-rac-glycerol, 1,2-dilinoleoyl-3-palmitoyl-rac-glycerol, 1,2-dioleoyl-3-linoleoyl-rac-glycerol, 1-palmitoyl-2-oleoyl-3-linoleoyl-rac-glycerol, triolein, 1-linoleoyl-2-oleoyl -3-stearoyl-rac-glycerol with respect to 1,2-dioleoyl-3-palmitoyl-rac-glycerol are about 0.55, about 0.65, about 0.69, 0.77, about 0.82, 0.93, about 0.97, respectively.

Purity ***Heavy metals*—**Proceed with 1.0 g of Sesame Oil according to Method 2, and perform the test. Prepare the control solution with 1.0 mL of lead standard solution (NMT 10 ppm).

Packaging and storage Preserve in tight containers.

Purified Shellac
정제쉘락

Purified Shellac is a resin-like substance obtained by purifying the secretion of *Laccifer lacca* Kerr (Coccidae).

Description Purified Shellac occurs as a pale yellowish brown to brown, scale-shaped chip. It is hard, brittle and lustrous with no odor or a faint characteristic odor.
It is freely soluble in ethanol or anhydrous ethanol and practically insoluble in water or ether.
It dissolves in sodium hydroxide TS.

Acid value Between 60 and 80. Weigh accurately about 1 g of Purified Shellac, add 40 mL of neutralized ethanol, and dissolve by warming. After cooling, titrate with 0.1 mol/L potassium hydroxide VS (potentiometric titration under the Titrimetry).

Purity (1) ***Heavy metals***—Weigh 2.0 g of Purified Shellac and perform the test according to Method 2. Prepare the control solution with 2.0 mL of the lead standard solution (NMT 10 ppm).

(2) ***Lead***—Weigh accurately 5.0 g of Purified Shellac and place it in a platinum crucible. Dry, carbonize, and incinerate at 450 °C to 550 °C. If incineration is not complete, cool and moisten with 2 to 5 mL of nitric acid (1 in 2), 50% magnesium nitrate solution or aluminum nitrate-calcium nitrate solution (prepared by dissolving 40 g of aluminum nitrate and 20 g of calcium nitrate in 100 mL of water) as an incineration supplement, dry, and continue with incineration. If incineration is incomplete, repeat the above procedure once and then add a final 2 mL to 5 mL of nitric acid (1 in 2), if necessary, and incinerate. After incineration, moisten the residue with water, add 2 to 4 mL of hydrochloric acid, and evaporate to dryness. Add 0.5 mol/L nitric acid, dissolve by warming, and filter with filter paper if any insoluble matter exists. Unless otherwise specified, add 0.5 mol/L nitric acid to make 25 mL and use this solution as the test solution. Separately, take 5.0 mL of the lead standard solution to a platinum crucible, proceed in the same manner as the test solution, and use the resulting solution as the standard solution. Perform the test with the test solution and the standard solutions as directed under the Atomic absorption spectroscopy according to the following conditions; the absorbance of the test solution is not greater than that of the standard solution (NMT 2.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(3) ***Arsenic***—Prepare the test solution with 0.40 g of Purified Shellac according to Method 3 and perform the test. Add 10 mL of a solution of magnesium nitrate in ethanol (1 in 50), then add 1.5 mL of concentrated hydrogen peroxide solution, and ignite to burn (NMT 5 ppm).

(4) ***Ethanol-insoluble substances***—Weigh accurately about 5 g of Purified Shellac and dissolve in 50 mL of ethanol on a water bath while shaking. Place a thimble filter, previously dried at 105 °C for 2 hours and tared, in a Soxhlet extractor, pour the ethanol solution prepared above into the thimble filer, extract with ethanol for 3 hours, and dry the thimble filer at 105 ℃ for 3 hours; the amount of the residue is NMT 2.0%. Use a cylindrical weighing bottle for weighing the thimble filer.

(5) ***Rosin***—To 1.0 g of pulverized Purified Shellac, add 10 mL of petroleum ether, shake well to dissolve, and filter. Add 10 mL of copper(II) acetate solution (1 in 200), shake gently to mix, and allow the mixture to stand; the clear supernatant does not exhibit a green color.

(6) ***Wax***—Dissolve 10.0 g of Purified Shellac in 150 mL of sodium carbonate solution (9 in 200) by shaking on a water bath and continue to heat for 2 hours. After cooling, collect the floating wax by filtration, wash the wax and the filter paper with water, transfer it to a beaker, and dry at 65 °C. Transfer the wax and the filter paper to a thimble filter in a Soxhlet extractor. Dissolve the wax remaining in the beaker with a suitable amount of chloroform by warming, transfer the solution to the thimble filter above, and extract with chloroform for 2 hours. Evaporate the chloroform solution to dryness and dry the residue at 105 °C for 3 hours; the amount of the residue is NMT 20 mg.

Loss on drying NMT 2.0%. Weigh accurately about 1 g of moderately fine powder of White Shellac and dry for 4 hours at 40°C, then for 15 hours in a desiccator (calcium chloride).

Ash NMT 1.0% (1 g, proceed as directed in the Ash under the Crude Drugs Test).

Packaging and storage Preserve in well-closed containers.

White Shellac
백색쉘락

White Shellac is a resin-like substance obtained by bleaching the secretion of *Laccifer lacca* Kerr (Coccidae).

Description White Shellac occurs as a yellowish white to pale yellow granule. It is hard and brittle with no odor or a faint characteristic odor.
It is sparingly soluble in ethanol, very slightly soluble in petroleum ether, and practically insoluble in water.
It dissolves in sodium hydroxide TS.

Acid value Between 65 and 90. Weigh accurately about 0.5 g of White Shellac, add 50 mL of neutralized ethanol, and dissolve by warming. After cooling, perform the test as directed in the Acid value under Purified Shellac.

Purity (1) ***Chloride***—Add 5 mL of ethanol to 0.40 g of White Shellac, dissolve by warming and shaking, and add 40 mL of water to cool. Add 12 mL of dilute nitric acid and water to make 100 mL and filter. Use 50 mL of the filtrate as the test solution and perform the test. Prepare the control solution by mixing 0.80 mL of 0.01 mol/L hydrochloric acid, 2.5 mL of ethanol, 6 mL of dilute nitric acid and water to make 50 mL (NMT 0.140%).

(2) ***Sulfate***—Add 5 mL of ethanol to 0.40 g of White Shellac, dissolve by warming and shaking, and add 40 mL of water to cool. Add 2 mL of dilute hydrochloric acid and water to make 100 mL and filter. Use 50 mL of the filtrate as the test solution and perform the test. Prepare the control solution by mixing 0.45 mL of 0.005 mol/L sulfuric acid, 2.5 mL of ethanol, 1 mL of dilute hydrochloric acid and water to make 50 mL (NMT 0.110%).

(3) ***Heavy metals***—Perform the test as directed in the Purity (1) under Purified Shellac.

(4) ***Lead***—Weigh accurately 5.0 g of White Shellac and place it in a platinum crucible. Dry, carbonize, and incinerate at 450 °C to 550 °C. If incineration is not complete, cool and moisten with 2 to 5 mL of nitric acid (1 in 2), 50% magnesium nitrate solution or aluminum nitrate-calcium nitrate solution (prepared by dissolving 40 g of aluminum nitrate and 20 g of calcium nitrate in 100 mL of water) as an incineration supplement, dry, and continue with incineration. If incineration is incomplete, repeat the above procedure once and then add a final 2 mL to 5 mL of nitric acid (1 in 2), if necessary, and incinerate. After incineration, moisten the residue with water, add 2 to 4 mL of hydrochloric acid, and evaporate to dryness. Add 0.5 mol/L nitric acid, dissolve by warming, and filter with filter paper if any insoluble matter exists. Unless otherwise specified, add 0.5 mol/L nitric acid to make 25 mL and use this solution as the test solution. Separately, take 5.0 mL of the lead standard solution to a platinum crucible, proceed in the same manner as the test solution, and use the resulting solution as the standard solution. Perform the test with the test solution and the standard solutions as directed under Atomic absorption spectroscopy according to the

following conditions; the absorbance of the test solution is not greater than that of the standard solution (NMT 2.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(5) ***Arsenic***—Proceed as directed in the Purity (2) under Purified Shellac.

(6) ***Ethanol-insoluble substances***—Proceed as directed in the Purity (3) under Purified Shellac.

(7) ***Rosin***—Proceed as directed in the Purity (4) under Purified Shellac.

(8) ***Wax***—Proceed as directed in the Purity (5) under Purified Shellac.

Loss on drying NMT 6.0%. Weigh accurately about 1 g of moderately fine powder of White Shellac and dry for 4 hours at 40°C, then for 15 hours in a desiccator (calcium chloride).

Ash NMT 1.0% (1 g, proceed as directed in the Ash under Crude Drugs Test).

Packaging and storage Preserve in well-closed containers in a cold place.

Light Anhydrous Silicic Acid
경질무수규산

Light Anhydrous Silicic Acid contains NLT 98.0% and NMT 101.0% of silicon dioxide (SiO_2: 60.08), calculated on the incinerated basis.

Description Light Anhydrous Silicic Acid occurs as a white to bluish white, light and fine powder. It is odorless and tasteless. It is smooth to the touch.
It is practically insoluble in water, ethanol or ether.
It is soluble in hydrofluoric acid, hot potassium hydroxide TS, or hot sodium hydroxide TS and insoluble in dilute hydrochloric acid.

Identification (1) To 0.1 g of Light Anhydrous Silicic Acid, add 20 mL of sodium hydroxide TS, boil to dissolve, and add 12 mL of ammonium chloride TS; a white gel-like precipitate is formed. This precipitate is insoluble in dilute hydrochloric acid.

(2) To the precipitate from (1), add 10 mL of methylene blue solution (1 in 10000), and then wash with water; the precipitate exhibits a blue color.

(3) Prepare a bead by fusing sodium ammonium hydrogen phosphate and place the bead on a platinum loop. Coat the bead with Light Anhydrous Silicic Acid to fuse again; infusible masses appear in the bead, which changes to an opaque bead with clear strips upon cooling.

Purity (1) ***Chloride***—To 0.5 g of Light Anhydrous Silicic Acid, add 20 mL of sodium hydroxide TS, boil to dissolve, cool, and then filter if necessary. Wash with 10 mL of water, combine the washings with the filtrate, add 18 mL of dilute nitric acid, and shake to mix. Then, add water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution by adding 20 mL of sodium hydroxide TS, 18 mL of dilute nitric acid, and water to 0.15 mL of 0.01 mol/L hydrochloric acid to make 50 mL (NMT 0.011%).

(2) ***Heavy metals***—To 0.5 g of Light Anhydrous Silicic Acid, add 20 mL of sodium hydroxide TS, boil to dissolve, and cool. Then, add 15 mL of acetic acid, shake to mix, then filter if necessary, and wash with 10 mL of water. Combine the filtrate and the washings, and add water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution as follows: add acetic acid to 20 mL of sodium hydroxide TS and 1 drop of phenolphthalein TS until the color of this solution disappears, add 2.0 mL of lead standard solution, 2 mL of dilute acetic acid and water to make 50 mL and use this solution as the control solution (NMT 40 ppm).

(3) ***Aluminum***—To 0.5 g of Light Anhydrous Silicic Acid, add 40 mL of sodium hydroxide TS, boil to dissolve, and cool. Then add sodium hydroxide TS to make 50 mL, and filter. Measure 10 mL of the filtrate, add 17 mL of acetic acid, shake, add 2 mL of aluminon TS and water to make 50 mL, and allow to stand for 30 minutes; the color of this solution is not more intense than that of the following control solution.

Control solution—Dissolve 0.176 g of aluminium potassium sulfate in water to make 1000 mL. To 15.5 mL of this solution, add 10 mL of sodium hydroxide TS, 17 mL of acetic acid, 2 mL of aluminon TS, and water to make 50 mL.

(4) ***Iron***—Add 10 mL of dilute hydrochloric acid to 40 mg of Light Anhydrous Silicic Acid, and heat for 10 minutes on a water bath while shaking. After cooling, add 0.5 g of L-tartaric acid, shake to mix, then prepare the test solution according to Method 2, and perform the test under Method B. Prepare the control solution with 2.0 mL of the iron standard solution (NMT 500 ppm).

(5) ***Calcium***—To 1.0 g of Light Anhydrous Silicic Acid, add 30 mL of sodium hydroxide TS, boil to dissolve, and cool. Then, add 20 mL of water and 1 drop of phenolphthalein TS, add dilute nitric acid until the red color of the solution disappears, and immediately add 5 mL of dilute acetic acid. Shake to mix, then add water to make 100 mL, and centrifuge or filter to obtain a clear solution. To 25 mL of this solution, add 1 mL of oxalic acid TS and ethanol to make 50 mL, immediately shake to mix and allow to stand for 10 minutes; the turbidity of this solution is not more intense than that of the following control solution.

Control solution—Dissolve 0.250 g of calcium carbonate, previously dried at 180 ℃ for 4 hours, in 3 mL of dilute hydrochloric acid, and add water to make 100 mL. To 4 mL of this solution, add 5 mL of dilute acetic acid and water to make 100 mL. Take 25 mL of this solution, add 1 mL of oxalic acid TS and ethanol to make 50 mL, and shake to mix.

(6) ***Arsenic***—Transfer 0.40 g of Light Anhydrous Silicic Acid into a porcelain crucible, add 10 mL of sodium hydroxide TS, boil to dissolve, and cool. Then, add 5 mL of water and 5 mL of dilute hydrochloric acid, and shake to mix. Use this solution as the test solution and perform the test (NMT 5 ppm).

Loss on drying NMT 7.0% (1 g, 105 ℃, 4 hours).

Loss on ignition NMT 12.0% (1 g, 850 ℃ to 900 ℃, constant mass).

Volume Slowly transfer 5.0 g of Light Anhydrous Silicic Acid in a 200-mL measuring cylinder, and allow to stand; the volume is NLT 70 mL.

Assay Weigh accurately about 1 g of Light Anhydrous Silicic Acid, add 20 mL of hydrochloric acid, and evaporate to dryness

on a sand bath. Moisten the residue with hydrochloric acid, evaporate to dryness, heat at 110 to 120 ℃ for 2 hours, and cool. Add 5 mL of dilute hydrochloric acid and heat. Allow to cool to room temperature, add 20 mL to 25 mL of hot water, and filter. Wash with warm water until the washings do not respond to the Chemical identification reactions (2) for chloride. Transfer the residue and the filter paper into a platinum crucible, ignite to incinerate, then ignite for 30 minutes, and cool. Then, weigh the mass, a (g). Moisten the residue with water, add 6 mL of hydrofluoric acid and 3 drops of sulfuric acid, evaporate to dryness, and ignite for 5 minutes. After cooling, weigh the residue, b (g).

Amount (g) of silicon dioxide (SiO_2) = a - b

Packaging and storage Preserve in tight containers.

Silicone Resin
실리콘수지

Description Silicone Resin occurs as a pale gray semi-transparent viscous liquid or paste-form substance. It is almost odorless.

Identification Transfer 1 g of Silicone Resin into a platinum dish, and ignite; it burns with the white smoke. To this white smoke, contact a cooled glass plate, and then the powder adheres to the surface of the glass plate. Combine this powder, transfer into a platinum dish, add 3 g of sodium hydroxide, and heat to dissolve. After cooling, dissolve in 50 mL of water, and filter. Slowly add 1 drop of this filtrate and 1 drop of ammonium molybdate TS to a filter paper, then slowly add 1 drop of benzidine TS, and then let it come in contact with the ammonia vapor; a blue precipitate forms.

Purity (1) ***Specific gravity*** d_{20}^{20}: Between 0.98 and 1.02.

(2) ***Viscosity and refractive index of extracted silicone oil—***Transfer 15 g of Silicone Resin into a Soxhlet extractor, extract with 150 mL of carbon tetrachloride for 3 hours, and evaporate the extracts on a water bath; a viscous liquid remains. The viscosity of this solution is 100 mm^2/s to 1100 mm^2/s at 25 ℃. Also, the refractive index n_n^{20} is 1400 to 1410.

(3) ***Silicon dioxide—***Dry the following residue extracted from (2) at about 100 ℃ for 1 hour; the amount is 0.45 g to 2.25 g.

Packaging and storage Preserve in tight containers.

Sodium Acetate Hydrtate
아세트산나트륨수화물

$CH_3COONa \cdot 3\,H_2O$

$C_2H_3NaO_2 \cdot 2H_2O$: L 36.08

Monosodium acetate trihydrate, [*6131-90-4*]

Sodium Acetate Hydrate, when dried, contains NLT 99.5% and NMT 101.0% of sodium acetate ($C_2H_3NaO_2$: 82.03).

Description Sodium Acetate Hydrate occurs as a colorless crystal or white crystalline powder. It has a slight acetic acid smell and a cool salty taste and is slightly bitter.
It is very soluble in water, freely soluble in acetic acid (100), soluble in ethanol, and practically insoluble in ether.
It decomposes in hot and dry air.

Identification An aqueous solution of Sodium Acetate Hydrate (1 in 10) responds to the Chemical identification reactions for acetate and sodium salt.

Purity (1) ***Clarity and color of solution***—Dissolve 2.0 g of Sodium Acetate Hydrate in 20 mL of water; the resulting solution is colorless and clear.

(2) ***Acidity or alkalinity—***Dissolve 1.0 g of Sodium Acetate Hydrate in 20 mL of freshly boiled and cooled water, and add 3 drops of phenolphthalein TS; the resulting solution exhibits a red color. When cooled to 10 ℃ or after cooled to 10 ℃, add 1.0 mL of 0.01 mol/L hydrochloric acid; the red color disappears.

(3) ***Chloride—***Weigh 1.0 g of Sodium Acetate Hydrate and perform the test. Prepare the control solution by adding 0.30 mL of 0.01 mol/L hydrochloric acid (NMT 0.011%).

(4) ***Sulfate—***Weigh 1.0 g of Sodium Acetate Hydrate and perform the test. Prepare the control solution by adding 0.35 mL of 0.005 mol/L sulfuric acid (NMT 0.017%).

(5) ***Heavy metals—***Weigh 2.0 g of Sodium Acetate Hydrate and perform the test according to Method 1. Prepare the control solution by adding 2.0 mL of the lead standard solution (NMT 10 ppm).

(6) ***Calcium and magnesium***—Dissolve 4.0 g of Sodium Acetate Hydrate in 25 mL of water, add and dissolve 6 g of ammonium chloride, 20 mL of strong ammonia water and 0.25 mL of sodium sulfite solution (1 in 10), and titrate with 0.01 mol/L ethylenediaminetetraacetic acid disodium salt VS; the consumed amount of 0.01 mol/L ethylenediaminetetraacetic acid disodium salt VS is NMT 0.5 mL (indicator: 0.1 g of methylthymol blue-potassium nitrate indicator). The endpoint of the titration is when the color of the solution turns from blue to grayish blue.

(7) ***Mercury***—Spread about 1 g of the excipient (a) evenly on a ceramic boat, and apply 10 to 300 mg of Sodium Acetate Hydrate on top of it. Spread about 0.5 g of the excipient (a) and 1 g of the excipient (b) on top of it in turn to form a layer. In the case of an automated mercury analyzer equipped with a separate catalyst in the combustion chamber, take only the sample without adding excipients to a nickel boat. Place the boat inside the combustion furnace and heat to about 900 ℃ with a current of air or oxygen at 0.5 to 1 L/minute. Elute the mercury and collect in a collecting tube. Heat the collecting tube at about 700 ℃ to transfer mercury vapor to a cold vapor atomic absorption spectrophotometer, and determine the absorbance as A. Separately, determine the absorbance in the same manner using only the excipients in a ceramic boat and determine it as Ab. Separately, proceed with mercury standard solution in the same manner and create a calibration curve from the absorbances. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the sample (NMT 1.0 ppm).

Operating conditions

Apparatus: Use an automated mercury analyzer for specimen combustion, trapping with gold amalgam and measurement using a cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Standard mercury stock solution—Dissolve 0.135 g of mercury(II) chloride in 0.001% L-cysteine solution to make 1000 mL. 1 mL of this solution contains 100 μg of mercury(II) chloride.

Mercury standard solution—Dilute the standard mercury stock solution with 0.001% L-cysteine solution to make 0 to 200 ng/mL.

Excipients—Activate excipients at 950 ℃ for 30 minutes when using (a) aluminum oxide and (b) calcium hydroxide-sodium carbonate (1 : 1).

(8) ***Lead***—Weigh accurately 5.0 g of Glacial Acetic Acid, place it into a platinum crucible, dry and carbonate it, and incinerate at 450 to 550 ℃. If incineration is not complete, cool and moisten with 2 to 5 mL of nitric acid (1 in 2), 50% magnesium nitrate solution or aluminum nitrate-calcium nitrate solution (prepared by dissolving 40 g of aluminum nitrate and 20 g of calcium nitrate in 100 mL of water) as an incineration supplement, dry, and continue with incineration. If incineration is incomplete, repeat the above procedure once and then add a final 2 mL to 5 mL of nitric acid (1 in 2), if necessary, and incinerate. After incineration, moisten the residue with water, add 2 to 4 mL of hydrochloric acid, and evaporate to dryness. Add 0.5 mol/L nitric acid, dissolve by warming, and filter with filter paper if any insoluble matter exists. Unless otherwise specified, add 0.5 mol/L nitric acid to make 25 mL and use this solution as the test solution. Separately, take 1.0 mL of lead standard solution to a platinum crucible, proceed in the same manner as the test solution, and use this solution as the standard solution. Perform the test with the test solution and the standard solutions as directed under the Atomic Absorption Spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 2.0 ppm).

Gas: Acetylene or hydrogen- Air.
Lamp: A lead hollow cathode lamp
Wavelength: 283.3 nm

(9) ***Arsenic***—Weigh 1.0 g of Sodium Acetate Hydrate and perform the test according to Method 1 (NMT 2 ppm).

(10) ***Potassium permanganate-reducing substances***—Dissolve 1.0 g of Sodium Acetate Hydrate in 100 mL of water, add and boil 5 mL of dilute sulfuric acid, add 0.50 mL of 0.002 mol/L potassium permanganate solution, and boil again for 5 minutes; the red color of the resulting solution does not disappear.

Loss on drying 39.0% to 40.5% (1 g, 2 hours from the start at 80 ℃, next 2 hours at 130 ℃).

Assay Weigh accurately about 0.2 g of Sodium Acetate Hydrate, previously dried, dissolve in 50 mL of acetic acid (100) and titrate with 0.1 mol/L perchloric acid VS (indicator: 1 mL of α-naphtholbenzein TS). The endpoint of the titration is when the color of the solution changes from yellow to green. Perform a blank test in the same manner and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 8.203 mg of $C_2H_3NaO_2$

Packaging and storage Preserve in tight containers.

Sodium Bisulfite
아황산수소나트륨

Sodium Hydrogen Sulfite $NaHSO_3$: 104.06

Sodium Bisulfite is a mixture of sodium bisulfite and sodium pyrosulfite.

Sodium Bisulfite contains NLT 64.0% and NMT 67.4% of sulfur dioxide (SO_2:64.06).

Description Sodium Bisulfite occurs as a white grain or powder and has an odor of sulfur dioxide.
It is freely soluble in water and practically insoluble in ethanol or ether.
An aqueous solution of Sodium Bisulfite (1 in 20) is acidic.
It is slowly changed by the air or light.

Identification An aqueous solution of Sodium Bisulfite (1 in 20) responds to the Chemical identification reactions for sodium salt and bisulfite.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Sodium Bisulfite in 10 mL of water; the resulting solution is clear and colorless.

(2) ***Thiosulfate***—Dissolve 1.0 g of Sodium Bisulfite in 15 mL of water, add slowly 5 mL of dilute hydrochloric acid, shake slowly to mix, and allow to stand for 5 minutes; the resulting solution doesn't become turbid.

(3) ***Heavy metals***—Dissolve 1.0 g of Sodium Bisulfite in 10 mL of water, add 5 mL of hydrochloric acid, evaporate on a water bath to dryness, and add 2 mL of dilute acetic acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution as follows: evaporate 5 mL of hydrochloric acid on a water bath to dryness and add 2 mL of dilute acetic acid, 2.0 mL of lead standard solution and water to make 50 mL (NMT 20 ppm).

(4) ***Mercury***—Spread evenly about 1 g of excipient (a) into a ceramic boat. In the case of a solid sample, take 10 to 300 mg of the cut and homogenized sample. For a liquid sample, allow 0.1 to 0.5 mL to completely permeate the excipient (a). Spread about 0.5 g of the excipient (a) and 1 g of the excipient (b) on top of it in turn to form a layer. In the case of an automated mercury analyzer equipped with a separate catalyst in the combustion chamber, take only the sample without adding excipients to a nickel boat. Place the boat inside the combustion furnace and heat to about 900 ℃ with a current of air or oxygen at 0.5 to 1 L/minute. Elute the mercury and collect in a collecting tube. Transfer the mercury vapor to a cold vapor atomic absorption spectrometer by heating the collecting tube to about 700 ℃ and determine the absorbance. A. Separately, place only the excipients in a ceramic boat and determine the absorbance, Ab in the same manner. Separately, proceed with mercury standard solution in the same manner and create a calibration curve from the absorbances. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the sample (NMT 1.0 ppm).

Operating conditions

Apparatus: Use an automated mercury analyzer for specimen combustion, trapping with gold amalgam and measurement using a cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Standard mercury stock solution—Dissolve 0.135 g of mercury(II) chloride in 0.001% L-cysteine solution to make 1000

mL. Each mL of this solution contains 100 μg of mercury(II) chloride.

Mercury standard solution—Dilute the standard mercury stock solution with 0.001% L-cysteine solution to make 0 to 200 ng/mL.

Excipients—Activate excipients at 950 ℃ for 30 minutes when using (a) aluminum oxide and (b) calcium hydroxide-sodium carbonate (1 : 1).

(5) ***Lead***—Weigh accurately 5.0 g of Sodium Bisulfite, transfer into a 150-mL beaker, and add 30 mL of water. Add hydrochloric acid in small amounts until the sample is completely dissolved, and then add 1 mL of hydrochloric acid. Boil for about 5 minutes, cool, add water to make about 100 mL, and adjust the pH to 2 to 4 with sodium hydroxide (1 in 4) or hydrochloric acid (1 in 4). Transfer the solution into a 250-mL separatory funnel, add water to make about 200 mL, add 2 mL of 2% of ammonium pyrolidine dithiocarbamate solution (2 in 100), and shake to mix. Extract this solution with 20 mL each of chloroform twice and evaporate the extract to dryness on a water bath. To the residue, add 3 mL of nitric acid, and heat until almost dry. Add 0.5 mL of nitric acid and 10 mL of water, concentrate until the final solution becomes 3 to 5 mL, add water to make 10 mL, and use this solution as the test solution. Separately, proceed with 1.0 mL of the lead standard solution in the same manner as the test solution and use this solution as the standard solution. Determine the absorbances of the test solution and the standard solution as directed under the Atomic absorption spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 2.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: A lead hollow-cathode lamp
Wavelength: 283.3 nm

(6) ***Iron***—Prepare the test solution with 1.0 g of Sodium Bisulfite according to Method 1 and perform the test according to Method A. Prepare the control solution by adding 2.0 mL of the iron standard solution (NMT 20 ppm).

(7) ***Selenium***—Weigh accurately 2.0 g of Sodium Bisulfite, transfer to a 50-mL beaker, add 10 mL of water and 5 mL of hydrochloric acid, and boil to remove sulfur dioxide. Use this solution as the test solution. Separately, place 1.0 g of Sodium Bisulfite and 5 mL of the standard selenium stock solution in a beaker, proceed in the same manner as the preparation of the test solution, and use the resulting solution as the control solution. To each test solution and the control solution, add 2 g of hydrazine sulfate, dissolve by warming, allow to stand for 5 minutes, and transfer to a Nessler tube. Add water to make 50 mL, and compare the colors; the red color of the test solution is not more intense than the color of the control solution (NMT 5 ppm).

(8) ***Arsenic***—Dissolve 0.5 g of Sodium Bisulfite in 10 mL of water, add 1 mL of sulfuric acid, heat the mixture in a sand bath until white smoke appears, and add water to make 5 mL. Use this solution as the test solution and perform the test (NMT 4 ppm).

Assay Weigh accurately about 1.5 g of Sodium Bisulfite, put immediately in an iodine flask containing exactly 50 mL of 0.05 mol/L Iodine solution, stopper, shake to mix, and allow to stand in a dark place for 5 minutes. Add 1 mL of hydrochloric acid and titrate the excessive iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 1 mL of starch TS). Perform a blank test in the same manner.

Each mL of 0.05 mol/L iodine VS
= 3.2032 mg of SO_2

Packaging and storage Fill in a light-resistant, tight container as full as possible, and preserve below 30 ℃.

Sodium Carbonate Hydrate
탄산나트륨수화물

Sodium Carbonate $Na_2CO_3 \cdot 10H_2O$: 286.14

Sodium Carbonate Hydrate contains NLT 99.0% and NMT 103.0% of sodium carbonate hydrate ($Na_2CO_3 \cdot 10H_2O$).

Description Sodium Carbonate Hydrate occurs as a colorless or white crystal.
It is freely soluble in water and practically insoluble in ethanol or ether.
Dissolve 1 g of Sodium Carbonate Hydrate in 10 mL of water; the solution is alkaline.
It effloresces in air.
It dissolves in its water of crystallization at 34 °C, and loses its water of crystallization at above 100 °C.

Identification An aqueous solution of Sodium Carbonate Hydrate (1 in 20) responds to the Chemical identification reactions for sodium salts and carbonates.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Sodium Carbonate Hydrate in 5 mL of water; the resulting solution is clear and colorless.

(2) ***Chloride***—Dissolve 0.5 g of Sodium Carbonate Hydrate in 10 mL of water, add 7 mL of dilute nitric acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution with 1.0 mL of 0.01 mol/L hydrochloric acid (NMT 0.071%).

(3) ***Heavy metals***—Dissolve 2.0 g of Sodium Carbonate Hydrate in 10 mL of water, add 8 mL of dilute hydrochloric acid, and evaporate to dryness on a water bath. Dissolve the residue in 35 mL of water and 2 mL of dilute acetic acid, and add water again to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution by evaporating 8 mL of dilute hydrochloric acid to dryness on a water bath and adding 2 mL of dilute acetic acid, 2.0 mL of lead standard solution and water to make 50 mL (NMT 10 ppm).

(4) ***Mercury***—Spread about 1 g of excipient (a) evenly on a ceramic boat, and place 10 mg to 300 mg of Sodium Carbonate Hydrate on top. Spread about 0.5 g of the excipient (a) and 1 g of the excipient (b) on top of it in turn to form a layer. In the case of an automated mercury analyzer equipped with a separate catalyst in the combustion chamber, take only the sample without adding excipients to a nickel boat. Place the boat inside the combustion furnace and heat to about 900 °C with a current of air or oxygen at 0.5 to 1 L/minute. Elute the mercury and collect in a collecting tube. Transfer the mercury vapor to a cold vapor atomic absorption spectrometer by heating the collecting tube to about 700 °C and determine the absorbance, A. Separately, place only the excipients in a ceramic boat and determine the absorbance, Ab in the same manner. Separately, proceed with mercury standard solution in the same manner and create a calibration curve from the absorbances. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the sample (NMT 1.0

ppm).

Operating conditions

Apparatus: Use an automated mercury analyzer for specimen combustion, trapping with gold amalgam and measurement using a cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Standard mercury stock solution—Dissolve 0.135 g of mercury(II) chloride in 0.001% L-cysteine solution to make 1000 mL. Each mL of this solution contains 100 μg of mercury(II) chloride.

Mercury standard solution—Dilute the standard mercury stock solution with 0.001% L-cysteine solution to make 0 to 200 ng/mL.

Excipients—Activate excipients at 950 ℃ for 30 minutes when using (a) aluminum oxide and (b) calcium hydroxide-sodium carbonate (1 : 1).

(5) ***Lead***—Weigh accurately 5.0 g of Sodium Carbonate Hydrate, transfer to a 150-mL beaker, add 30 mL of water, and add hydrochloric acid in small amounts until the sample is sufficiently dissolved. Then add 1 mL of hydrochloric acid again. Boil for about 5 minutes, cool, add water to make about 100 mL, and adjust the pH to 2 to 4 with sodium hydroxide (1 in 4) or hydrochloric acid (1 in 4). Transfer this solution to a 250-mL separatory funnel, add water to make about 200 mL, add 2 mL of 2% ammonium pyrrolidine dithiocarbamate solution (2 in 100), and shake to mix. Extract this solution twice with 20 mL of chloroform and evaporate the extract to dryness on a water bath. To the residue, add 3 mL of nitric acid and heat until almost dry. Add 0.5 mL of nitric acid and 10 mL of water, concentrate until the final solution becomes 3 mL to 5 mL, add water to make 10 mL, and use this solution as the test solution. Separately, proceed with 2.0 mL of the lead standard solution in a platinum crucible in the same manner as the test solution and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Atomic absorption spectroscopy according to the following operating conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 4.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow-cathode lamp
Wavelength: 283.3 nm

(6) ***Arsenic***—Prepare the test solution with 0.65 g of Sodium Carbonate Hydrate according to Method 1, and perform the test (NMT 3.1 ppm).

Loss on drying 61.0 to 63.0% (1 g, 105 ℃, 4 hours).

Assay Weigh accurately about 3 g of Sodium Carbonate Hydrate, dissolve in 25 mL of water, and titrate with 0.5 mol/L sulfuric acid VS. When the blue color of the solution changes to yellowish green, boil it carefully, cool, and titrate until a greenish yellow color appears (indicator: 2 drops of bromocresol green TS).

Each mL of 0.5 mol/L sulfuric acid VS
= 143.07 mg of $Na_2CO_3 \cdot 10H_2O$

Packaging and storage Preserve in tight containers.

Dried Sodium Carbonate
건조탄산나트륨

Anhydrous Sodium Carbonate
Sodium Carbonate Na_2CO_3:105.99

Dried Sodium Carbonate, when dried, contains NLT 99.0% and NMT 101.0% of sodium carbonate (Na_2CO_3).

Description Dried Sodium Carbonate occurs as a white crystal or crystalline powder.
It is freely soluble in water and practically insoluble in ethanol or ether.
Dissolve 1 g of Dried Sodium Carbonate in 10 mL of water; the solution is alkaline.
It is hygroscopic.

Identification An aqueous solution of Dried Sodium Carbonate (1 in 20) responds to the Chemical identification reactions for sodium salts and carbonates.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Dried Sodium Carbonate in 10 mL of water; the resulting solution is clear and colorless.

(2) ***Chloride***—Dissolve 0.5 g of Dried Sodium Carbonate in 10 mL of water, add 12 mL of dilute nitric acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution with 1.0 mL of 0.01 mol/L hydrochloric acid (NMT 0.071%).

(3) ***Heavy metals***—Dissolve 1.0 g of Dried Sodium Carbonate in 10 mL of water, add 7.5 mL of dilute hydrochloric acid, and evaporate to dryness on a water bath. Dissolve the residue in 35 mL of water and 2 mL of dilute acetic acid, and add water again to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution by evaporating 7.5 mL of dilute hydrochloric acid to dryness on a water bath and adding 2 mL of dilute acetic acid, 2.0 mL of lead standard solution, and water to make 50 mL (NMT 20 ppm).

(4) ***Mercury***—Spread about 1 g of excipient (a) evenly on a ceramic boat, and place 10 mg to 300 mg of Dried Sodium Carbonate on top. Spread about 0.5 g of the excipient (a) and 1 g of the excipient (b) on top of it in turn to form a layer. In the case of an automated mercury analyzer equipped with a separate catalyst in the combustion chamber, take only the sample without adding excipients to a nickel boat. Place the boat inside the combustion furnace and heat to about 900 °C with a current of air or oxygen at 0.5 L/min to 1 L/min. Elute the mercury and collect in a collecting tube. Transfer the mercury vapor to a cold vapor atomic absorption spectrometer by heating the collecting tube to about 700 °C and determine the absorbance, A. Separately, place only the excipients in a ceramic boat and determine the absorbance, Ab in the same manner. Separately, proceed with mercury standard solution in the same manner and create a calibration curve from the absorbances. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the sample (NMT 1.0 ppm).

Operating conditions

Apparatus: Use an automated mercury analyzer for specimen combustion, trapping with gold amalgam and measurement using a cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion

chamber may be used.

Standard mercury stock solution—Dissolve 0.135 g of mercury(II) chloride in 0.001% L-cysteine solution to make 1000 mL. Each mL of this solution contains 100 μg of mercury(II) chloride.

Mercury standard solution—Dilute the standard mercury stock solution with 0.001% L-cysteine solution to make 0 ng/mL to 200 ng/mL.

Excipients—Activate excipients at 950 ℃ for 30 minutes when using (a) aluminum oxide and (b) calcium hydroxide-sodium carbonate (1 : 1).

(5) ***Lead***—Weigh accurately 5.0 g of Dried Sodium Carbonate, transfer to a 150-mL beaker, add 30 mL of water, and add hydrochloric acid in small amounts until the sample is sufficiently dissolved. Then add 1 mL of hydrochloric acid again. Boil for about 5 minutes, cool, add water to make about 100 mL, and adjust the pH to 2 to 4 with sodium hydroxide (1 in 4) or hydrochloric acid (1 in 4). Transfer this solution to a 250-mL separatory funnel, add water to make about 200 mL, add 2 mL of 2% ammonium pyrrolidine dithiocarbamate solution (2 in 100), and shake to mix. Extract this solution twice with 20 mL of chloroform and evaporate the extract to dryness on a water bath. To the residue, add 3 mL of nitric acid and heat until almost dry. Add 0.5 mL of nitric acid and 10 mL of water, concentrate until the final solution becomes 3 mL to 5 mL, add water to make 10 mL, and use this solution as the test solution. Separately, proceed with 1.0 mL of the lead standard solution in a platinum crucible in the same manner as the test solution and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Atomic absorption spectroscopy according to the following operating conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 2.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: A lead hollow-cathode lamp
Wavelength: 283.3 nm

(6) ***Arsenic***—Prepare the test solution with 0.65 g of Dried Sodium Carbonate according to Method 1 and perform the test (NMT 3.1 ppm).

Loss on drying NMT 2.0% (2 g, 105℃, 4 hours).

Assay Weigh accurately about 1.2 g of Dried Sodium Carbonate, previously dried, dissolve in 25 mL of water, and titrate with 0.5 mol/L sulfuric acid VS. When the blue color of the solution changes to yellowish green, boil it carefully, cool, and titrate until a greenish yellow color appears (indicator: 2 drops of bromocresol green TS).

Each mL of 0.5 mol/L sulfuric acid VS
= 52.99 mg of Na_2CO_3

Packaging and storage Preserve in tight containers.

Sodium Caseinate
카세인나트륨

Sodium Caseinate, when dried, contains NLT 14.5% and NMT 15.8% of nitrogen (N: 14.01).

Description Casein occurs as a white to pale yellow powder, granule or flake. It is odorless and tasteless or has a slight, characteristic odor or taste.
It is soluble in water and is practically insoluble in ethanol or in diethyl ether.
It is soluble in sodium hydroxide TS or in dilute hydrochloric acid.

Identification (1) Perform the test as directed in (1), (2) and (3) under the Identification with Casein.
(2) The residue obtained from the Residue on ignition responds to the Chemical identification reactions for sodium salt.

Purity (1) ***Clarity and color of solution***—Perform the test as directed in (1) under the Purity with Casein.
(2) ***Acidity or alkalinity***—Dissolve 1.0 g of Sodium Caseinate in 50 mL of water; the pH of this solution is 6.0 to 7.5.
(3) ***Heavy metals***—Perform the test as directed in (4) under the Purity with Casein (NMT 20 ppm).
(4) ***Arsenic***—Prepare the test solution with 1.0 g of Sodium Caseinate according to Method 3 and perform the test (NMT 2 ppm).
(5) ***Fat***—Perform the test as directed in (6) under the Purity with Casein (NMT 1.5%).

Loss on drying NMT 15.0% (1 g, 100 °C, 3 hours).

Residue on ignition NMT 6.0% (1 g, after drying).

Assay Weigh accurately about 15 mg of Sodium Caseinate, previously dried, and perform the test as directed under the Nitrogen Determination (Semimicro-Kjeldahl Method).

Each mL of 0.005 mol/L sulfuric acid VS
= 0.14007 mg of N

Packaging and storage Preserve in tight containers.

Sodium Hydroxide
수산화나트륨

NaOH: 40.00

Sodium Hydroxide contains NLT 95.0% and NMT 101.0% of sodium hydroxide (NaOH).

Description Sodium Hydroxide occurs as a white mass in small globules, flakes, rods, or other forms and is hard and brittle with a crystalline fracture.
It is freely soluble in water or ethanol and practically insoluble in ether.
It rapidly absorbs carbon dioxide in the air.
It deliquesces in the presence of moisture.

Identification (1) An aqueous solution of Sodium Hydroxide (1 in 500) is alkaline.
(2) An aqueous solution of Sodium Hydroxide (1 in 25)

responds to the Chemical Identification reactions for sodium salt.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Sodium Hydroxide in 20 mL of water; the resulting solution is colorless and clear.

(2) ***Chloride***—Dissolve 2.0 g of Sodium Hydroxide in 100 mL of water. To 25 mL of the solution, add 10 mL of dilute nitric acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution by adding 0.7 mL of 0.01 mol/L hydrochloric acid (NMT 0.050%).

(3) ***Heavy metals***—Dissolve 1.0 g of Sodium Hydroxide in 5 mL of water, add 11 mL of dilute hydrochloric acid, and evaporate to dryness on a water bath. Dissolve the residue in 35 mL of water, add 2 mL of dilute acetic acid and 1 drop of ammonia TS, and add water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution by evaporating 11 mL of dilute hydrochloric acid to dryness on a water bath and adding 2 mL of dilute acetic acid, 3.0 mL of the lead standard solution, and water to make 50 mL (NMT 30 ppm).

(4) ***Potassium***—Dissolve 0.10 g of Sodium Hydroxide in water to make 40 mL. Add 1.0 mL of dilute acetic acid to 4.0 mL of this solution and shake to mix. Add 5.0 mL of sodium tetraphenylborate solution (1 in 30), shake immediately, and allow the mixture to stand for 10 minutes; the turbidity of the resulting solution is not more intense than that of the following control solution.

Control solution—Dissolve 9.5 mg of potassium chloride in water to make 1000 mL. Add 1.0 mL of dilute acetic acid to 4.0 mL of this solution, shake to mix, and proceed in the same manner as described above.

(5) ***Sodium carbonate***—Prepare B (mL) of the solution as directed under the Assay and determine the amount of sodium carbonate (Na_2CO_3: 105.99) according to the following formula; the amount is NMT 2.0%.

$$\text{Amount (mg) of sodium carbonate} = 105.99 \times B$$

(6) ***Mercury***—To 2.0 g of Sodium Hydroxide, add 1 mL of potassium permanganate solution (3 in 50) and 30 mL of water to dissolve. Add slowly purified hydrochloric acid to neutralize the solution, add 5 mL of diluted sulfuric acid (1 in 2), add hydroxylamine hydrochloride solution (1 in 5) until the precipitate of manganese dioxide disappears, and add water to make exactly 100 mL. Use this solution as the test solution. Perform the test with the test solution as directed under the Atomic absorption spectroscopy (cold vapor generation method). Place the test solution in the sample bottle of the atomic absorption spectrophotometer, add 10 mL of tin(II) chloride-sulfuric acid TS, connect immediately to the atomic absorption spectrophotometer, and circulate air. Read the absorbance, A_T, of the test solution at the wavelength of 253.7 nm when the reading of the data collection device rises rapidly to a constant. Separately, take 2.0 mL of the mercury standard solution, add 1 mL of potassium permanganate solution (3 in 50), 30 mL of water and the same amount of purified hydrochloric acid as used in the preparation of the test solution, and proceed in the same manner as directed for the preparation of the test solution. Determine the atomic absorbance, A_S, of the solution; A_T is NMT A_S.

(7) ***Lead***—Weigh accurately 5.0 g of Sodium Hydroxide, transfer it to a platinum crucible, dry, carbonize, and then incinerate at 450 ℃ to 550 ℃. If incineration is not complete, cool and moisten with 2 to 5 mL of nitric acid (1 in 2), 50% magnesium nitrate solution or aluminum nitrate-calcium nitrate solution (prepared by dissolving 40 g of aluminum nitrate and 20 g of calcium nitrate in 100 mL of water) as an incineration supplement, dry, and continue with incineration. If incineration is incomplete, repeat the above procedure once and then add a final 2 mL to 5 mL of nitric acid (1 in 2), if necessary, and incinerate. After incineration, moisten the residue with water, add 2 to 4 mL of hydrochloric acid, and evaporate to dryness. Add 0.5 mol/L nitric acid, dissolve by warming, and filter with filter paper if any insoluble matter exists. Unless otherwise specified, add 0.5 mol/L nitric acid to make 25 mL and use this solution as the test solution. Separately, take 5.0 mL of the lead standard solution to a platinum crucible, proceed in the same manner as the test solution, and use the resulting solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Atomic absorption spectroscopy according to the following conditions; the absorbance of the test solution is not greater than that of the standard solution (NMT 0.5 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow-cathode lamp
Wavelength: 283.3 nm

(8) ***Arsenic***—Dissolve 50 g of Sodium Hydroxide in freshly boiled and cooled water to make 250 mL and use this solution as the test solution. To 2.6 mL of the test solution, add 5 mL of water, neutralize by slowly adding hydrochloric acid, and use this solution as the test solution. Perform the test with the test solution as directed under the Arsenic (NMT 4 ppm).

Assay Weigh accurately about 1.5 g of Sodium Hydroxide, dissolve in 40 mL of freshly boiled and cooled water, and cool to 15 °C. Add 2 drops of phenolphthalein TS and titrate with 0.5 mol/L sulfuric acid VS until the red color of the solution disappears. Determine the amount, A (mL), of 0.5 mol/L sulfuric acid VS consumed. To this solution, add 2 drops of methyl orange TS and titrate with 0.5 mol/L sulfuric acid VS until the solution exhibits a persistent pale red color. Determine the amount, B (mL), of 0.5 mol/L sulfuric acid VS consumed. Determine the amount of Sodium Hydroxide (NaOH) by subtracting B (mL) from A (mL).

$$\text{Each mL of 0.5 mol/L sulfuric acid VS} = 40.00 \text{ mg of NaOH}$$

Packaging and storage Preserve in tight containers.

Sodium Lauryl Sulfate
라우릴황산나트륨

$C_{12}H_{25}NaO_4S$:288.38

Sodium monododecyl sulfate [*151-21-3*]

Sodium Lauryl Sulfate is alkyl sodium sulfate, consisting mainly of sodium lauryl sulfate ($C_{12}H_{25}NaO_4S$: 289.38).

Sodium Lauryl Sulfate contains NLT 85.0% of alkyl sodium sulfate [as sodium lauryl sulfate ($C_{12}H_{25}NaO_4S$)].

Description Sodium Lauryl Sulfate occurs as a white to pale yellow crystal or powder. It has a slightly characteristic odor.
It is sparingly soluble in ethanol (95).
1 g of Sodium Lauryl Sulfate dissolves in 10 mL of water, forming a clear or opalescent solution, and foams when shaking to mix.

Identification (1) Transfer 2.5 g of Sodium Lauryl Sulfate into a platinum or quartz crucible, and add 2 mL of 5 mol/L sulfuric acid TS. Heat on a water bath, place it in a furnace, ignite at 600 ± 25 ℃, and ignite the residue to complete incineration. After cooling, add a few drops of 1 mol/L sulfuric acid TS, and repeat heating and ignition in the same manner. After cooling, add a few drops of ammonium carbonate TS, evaporate to dryness, and then ignite again in the same manner. To the cooled residue, add 50 mL of water, and shake well to dissolve. To 2 mL of this solution, add 4 mL of potassium hexahydroxoantimonate(V) TS; a white crystalline precipitate forms. If necessary, rub the inner wall of the test tube with a glass rod.

(2) To an aqueous solution of Sodium Lauryl Sulfate (1 in 10), add hydrochloric acid to render the solution acidic, and boil for 20 minutes; no precipitate is formed. To this solution, add barium chloride TS; a white precipitate is formed

(3) Determine the infrared spectra of Sodium Lauryl Sulfate and sodium lauryl sulfate RS as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

Purity (1) ***Alkalinity***—Dissolve about 1.0 g of Sodium Lauryl Sulfate in 100 mL of water, and add 2 drops of phenol red TS and 0.60 mL of 0.1 mol/L hydrochloric acid; the resulting solution exhibits a yellow color.

(2) ***Sodium chloride***—Weigh accurately about 5 g of Sodium Lauryl Sulfate, dissolve in 50 mL of water, add dilute nitric acid to neutralize if necessary, and add exactly 5 mL of 0.1 mol/L sodium chloride TS. Titrate with 0.1 mol/L silver nitrate VS (indicator: 2 drops of fluorescein sodium TS). The endpoint of titration is when the color of the solution changes from yellowish green through yellow and then to orange. Perform a blank test in the same manner and make necessary correction.
The amount of sodium chloride (NaCl: 58.44) combined with the amount of sodium sulfate (Na_2SO_4: 142.04) is NMT 8.0%.

Each mL of 0.1 mol/L silver nitrate VS
= 5.844 mg of NaCl

(3) ***Sodium sulfate***—Weigh accurately about 1 g of Sodium Lauryl Sulfate, add 10 mL of water to dissolve, add 100 mL of ethanol (95), heat for 2 hours near the boiling point, and filter the hot precipitate through a glass filter (4 μm to 10 μm). Then, wash with 100 mL of boiling ethanol (95). Add 150 mL of water to dissolve and wash the residue in the glass filter, add 10 mL of dilute hydrochloric acid, heat until boiling, and add 25 mL of barium chloride TS. Then, allow to stand overnight. Filter and take the precipitate, and wash until the washings are not turbid when silver nitrate TS is added. Dry the precipitate with the filter paper, slowly increase the temperature to ignite at 500 ℃ to 600 ℃ to a constant mass, then weigh the mass accurately, and use this value as the amount of barium sulfate ($BaSO_4$: 233.39).
The amount of sodium sulfate (Na_2SO_4: 142.04), combined with the above amount of sodium chloride (NaCl: 58.44), is NMT 8.0%.

Amount (mg) of sodium sulfate (Na_2SO_4)
= Amount (mg) of barium sulfate ($BaSO_4$) × 0.6086

(4) ***Unsulfated alcohol***—Weigh accurately about 10 g of Sodium Lauryl Sulfate, dissolve in 100 mL of water, add 100 mL of ethanol (95), and transfer into a separatory funnel. Extract 3 times each with 50 mL of petroleum ether. If the ether layer is difficult to separate due to emulsification, add sodium chloride. Collect the petroleum ether layer, wash 3 times each with 50 mL of water, then dehydrate the petroleum ether layer with anhydrous sodium sulfate, and filter. Transfer the filtrate in a previously weighed beaker and evaporate to dryness on a water bath until there is no odor of petroleum ether. Dry the residue at 105 ℃ for 30 minutes and weigh the mass; the amount is NMT 4.0%.

Total alcohol Weigh accurately about 5 g of Sodium Lauryl Sulfate, add 150 mL of water and 50 mL of hydrochloric acid, and boil for 4 hours under a reflux condenser. After cooling, wash the condenser with ether, and extract the inside of the flask 2 times each with 75 mL of ether, combine the extracts, and evaporate to dryness on a water bath. Dry the residue at 105 ℃ for 30 minutes and weigh the mass; it is NLT 59.0%.

Assay Weigh accurately about 1.15 g of Sodium Lauryl Sulfate, and warm, if necessary, to dissolve in water to make exactly 1000 mL. Pipet 20 mL of this solution, transfer into a stoppered flask, add 15 mL of dichloromethane and 10 mL of dimidium bromide-sulfan blue TS, and shake to mix. Titrate with 0.004 mol/L benzethonium chloride VS while shaking vigorously to mix, confirm the separation of the layers, and the endpoint of titration is when the pink color of the dichloromethane layer disappears and changes to grayish blue.

A mixture of dimidium bromide-sulfan blue—Dissolve 0.5 g of dimidium bromide and 0.25 g of sulfan blue each in 30 mL of a mixture of warm water and ethanol (99.5) (9 : 1), and mix these solutions, add a mixture of water and ethanol (99.5) (9 : 1) to make 250 mL. Take 20 mL of this solution and add 270 mL of dilute sulfuric acid (7 in 675) and water to make 500 mL. Store away from light.

Each mL of 0.004 mol/L benzethonium chloride VS
= 1.154 mg of $C_{12}H_{25}NaO_4S$

Packaging and storage Preserve in well-closed containers.

Sodium Oleate
올레인산나트륨

Sodium Oleate is the sodium salt of oleic acid ($C_{18}H_{34}O_2$: 282.46).

Description Sodium Oleate occurs as a white to pale yellow powder or a pale yellowish brown coarse powder or lump. It has a characteristic odor and taste.
It is soluble in water or in ethanol and practically insoluble diethyl ether.

Identification (1) To 50 mL of the aqueous solution of Sodium Oleate (1 in 25), add 5 mL of dilute sulfuric acid while shaking to mix and filter through a filter paper, previously moistened with water. Wash the residue with water until the solution used for washing the residue does not exhibit a red color with the dropwise addition of methyl orange solution. Filter the oily residue using a dry filter paper, take 2 to 3 drops of the emulsion in a small test tube, and carefully add about 1 mL of sulfuric acid; a reddish brown band develops at the zone of contact. Also, take 1 to 3 drops of the emulsion, dissolve in 3 to 4 mL of diluted acetic acid (1 in 4), add 1 drop of acetic acid solution in chromium

trioxide (1 in 10), and add 1 mL of sulfuric acid while shaking to mix; the resulting solution exhibits a dark purple color.

(2) The residue obtained from the Residue on ignition responds to the Chemical identification reactions for sodium salt.

Purity (1) ***Clarity and color of solution***—Dissolve 0.5 g of Sodium Oleate in 20 mL of water; the resulting solution is colorless to pale yellow and clear.

(2) ***Free alkali***—Weigh 0.5 g of pulverized Sodium Oleate and dissolve in 100 mL of neutralized ethanol by heating. Filter the insoluble matter while it is hot, wash it with neutralized ethanol at about 40 ℃ until the solution used for washing becomes colorless, and then combine the solution used for washing with the filtrate. After cooling, titrate the combined solution with 0.05 mol/L sulfuric acid to determine the amount consumed as a mL. Also, wash the residue obtained from the previous step 5 times each time with 10 mL of boiling water. Combine all solutions used for washing, cool, and titrate with 0.05 mol/L sulfuric acid to determine the amount consumed as b mL (indicator: 3 drops of bromophenol blue TS). Calculate the amount of free alkali according to the following equation it is NMT 0.05%.

$$\text{Amount (\%) of free alkali} = \frac{0.0040 \times a + 0.0053 \times b}{Amount\ (g)\ of\ sample} \times 100$$

(3) ***Heavy metals***—Proceed with 0.5 g of Sodium Oleate according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 40 ppm).

(4) ***Arsenic***—Weigh 2.5 g of Sodium Oleate and dissolve in 30 mL of boiling water while shaking well to mix. To this solution, add 6 mL of dilute sulfuric acid, remove the precipitated fatty acids by extraction with diethyl ether, and add water to make 50 mL. Pipet 20 mL of this solution and perform the test using this solution as the test solution; the color of the test solution is not more intense than that of the following colorimetric standard.

Colorimetric standard—To 5.0 mL of arsenic standard solution, add 30 mL of water and 6 mL of dilute sulfuric acid and add more water to make 50 mL. Proceed with 20 mL of this solution in the same manner as in the preparation of the test solution (NMT 2 ppm).

Residue on ignition Between 22.0% and 25.0% (1 g).

Packaging and storage Preserve in well-closed containers.

Sodium Starch Glycolate
전분글리콜산나트륨

Starch carboxymethyl ether, sodium salt [9063-38-1]

Sodium Starch Glycolate is the sodium salt of a carboxymethyl ether of starch or of a cross-linked carboxymethyl ether of starch.

There are two neutralization types of Sodium Starch Glycolate, Type A and Type B, and their insoluble matter in a mixture of ethanol (99.5) and water (8:2), than 4.2%, and NLT 2.0% and NMT 3.4% of sodium (Na: 22.99), respectively.

The label states the type of neutralization.

Description Sodium Starch Glycolate occurs as a white powder, and has a characteristic salty taste.
It practically insoluble in ethanol (99.5).
It swells with water, and becomes viscous, pasty liquid.
It is hygroscopic.

Identification (1) Acidify 5 mL of a solution of Sodium Starch Glycolate (1 in 500) with dilute hydrochloric acid, then add one drop of iodine TS, and stir: a blue to violet color is produced.

(2) Determine the infrared absorption spectrum of Sodium Starch Glycolate as directed in the potassium bromide disk method under Infrared Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wave numbers.

(3) The test solution obtained in the Purity (2) responds to Qualitative Tests (2) for sodium salt. Perform the test using 2 mL of the test solution and 4 mL of potassium hexahydroxoantimonate (V) TS.

pH To 1 g of Sodium Starch Glycolate add 30 mL of water and stir: the pH of the resulting suspension of Type A is 5.5 – 7.5, and that of Type B is 3.0 – 5.0.

Purity (1) ***Iron***—(i) Sample solution Take 2.5 g of Sodium Starch Glycolate in a silica or platinum crucible, add 2 mL of 5 mol/L sulfuric acid TS. Heat on a water bath, then ignite cautiously with a gas burner or preferably in an electric furnace at 600 ± 25℃, and incinerate the residue completely. Allow to cool, add a few drops of 1 mol/L sulfuric acid TS, and heat and ignite as above. Allow to cool, add a few drops of ammonium carbonate TS, evaporate to dryness on a water bath, and heat and ignite as above. After cooling, dissolve the residue by adding 50 mL of water.

(ii) Standard solution Weigh accurately 863.4 mg of ammonium Iron(III) sulfate dodecahydrate, dissolve in water, add 25 mL of 1 mol/L sulfuric acid TS, and add water to make exactly 500 mL. Pipet 10 mL of this solution, and add water to make exactly 100 mL. Pipet 5 mL of this solution, and add water to make exactly 100 mL. Each mL of this solution contains 1.0 mg of Iron(Fe).

(iii) Procedure Pipet 10 mL each of the test solution and standard solution, and to each solution add 2 mL of citric acid solution (1 in 5) and 0.1 mL of thioglycolic acid. Then add ammonia solution (28) dropwise to render the solution alkaline, using litmus paper as an indicator. Add water to make 20 mL, and use these solutions as the test solution and the control solution, respectively. Allow these solutions to stand for 5 minutes, and compare the color of the solutions using white background: the color of the test solution is not deeper than that of the control solution (NMT 20 ppm).

(2) ***Sodium glycolate***—Conduct this procedure without exposure to light, using light-resistant vessels.

(i) Sample solution Weigh accurately 0.200 g of Sodium Starch Glycolate in a beaker, add 4 mL of 6 mol/L acetic acid TS and 5 mL of water, and stir to dissolve. Add 50 mL of acetone and 1 g of sodium chloride, stir, and filter through a filter paper previously soaked with acetone. Rinse the beaker and the filter paper with acetone, combine the filtrate and washings, and add acetone to make exactly 100 mL. Allow to stand for 24 hours, and use the supernatant as the test solution.

(ii) Standard solution To exactly 0.310 g of glycolic acid, previously dried in a desiccator (silica gel) for 18 hours, add water to dissolve to make exactly 500 mL. Pipet 5 mL of this solution, add 4 mL of 6 mol/L acetic acid TS, and allow to stand for 30 minutes. Add 50 mL of acetone and 1 g of sodium chloride, proceed as (i) above, and use the supernatant as the standard solution.

(iii) Procedure Pipet 2.0 mL each of the test solution and standard solution into 25-mL stoppered test tubes, and heat on a water bath for 20 minutes to remove acetone. After cooling, add 20.0 mL of 2,7-dihydroxynaphthalene TS to the residue, stopper the test tube, and heat on a water bath for 20 minutes. Cool under running water, and transfer whole quantity of the content to a 25-mL volumetric flask. Maintain the flask under running water, and add sulfuric acid to make 25 mL. Within 10 minutes, determine the absorbance of these solutions at 540 nm using water as the blank as directed under Ultraviolet-visible Spectroscopy ; the absorbance of the test solution is not larger than that of the standard solution (NMT 2.0%).

(4) ***Sodium chloride***—Weigh accurately about 0.5 g of Sodium Starch Glycolate in a beaker, disperse in 100 mL of water, and add 1 ml of nitric acid. Titrate with 0.1 mol/L silver nitrate VS (potentiometric titration): the amount of sodium chloride (NaCl: 58.44) is NMT 7.0%.

Each mL of 0.1 mol/L silver nitrate VS
= 5.844 mg of NaCl

Loss on drying NMT 10.0% (1 g, 130 ℃, 90 minutes).

Microbial limits *Salmonella* and *Escherichia coli* are not observed.

Assay To about 1 g of Sodium Starch Glycolate add 20 mL of a mixture of ethanol (99.5) and water (8:2), stir for 10 minutes, and filter. Repeat this procedure until no more turbidity is produced by adding silver nitrate TS, and dry the residue on the filter paper at 105 ℃ to constant mass. Weigh accurately 0.7 g of the mass, add 80 mL of acetic acid (100), and heat the mixture under a reflux condenser on a water bath for 2 hours. After cooling, titrate with 0.1 mol/L perchloric acid VS (potentiometric titration).

Content (%) of sodium (Na) = V × 2.299 × 100/M

V: Consumed amount (mL) of 0.1 mol/L perchloric acid VS
M: Mass (mg) of the dried residue

Packaging and storage Preserve in well-closed containers.

Dried Sodium Sulfite
건조아황산나트륨

Anhydrous Sodium Sulfite
Sodium sulfite Na_2SO_3: 126.04

Dried Sodium Sulfite contains NLT 97.0% and NMT 101.0% of sodium sulfite (Na_2SO_3).

Description Dried Sodium Sulfite occurs as a white crystal or powder and is odorless.
It is freely soluble in water and practically insoluble in ethanol or ether.
Dissolve 1 g of Dried Sodium Sulfite in 10 mL of water; the pH of this solution is about 10.
It slowly changes in the humid air.

Identification An aqueous solution of Dried Sodium Sulfite (1 in 20) responds to the Chemical identification reactions for sodium salt and sulfite.

Purity (1) ***Clarity and color of solution***—Dissolve 0.5 g of Dried Sodium Sulfite in 10 mL of water; the solution is colorless and clear.

(2) **Thiosulfate**—Dissolve 1.0 g of Dried Sodium Sulfite in 15 mL of water, add gradually 5 mL of hydrochloric acid, shake to mix, and allow to stand for 5 minutes; the solution does not become turbid.

(3) **Heavy metals**—Dissolve 1.0 g of Dried Sodium Sulfite in 5 mL of water, add 2 mL of hydrochloric acid gradually, and evaporate the mixture on a water bath to dryness. Add 3 mL of boiling water and 1 mL of hydrochloric acid to the residue and again evaporate to dryness on a water bath. Dissolve the residue in 2 mL of dilute acetic acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution by evaporating 3 mL of hydrochloric acid to dryness and adding 2 mL of dilute acetic acid, 1.0 mL of the lead standard solution and water to make 50 mL (NMT 10 ppm).

(4) **Mercury**—Spread evenly about 1 g of additive (a) into a ceramic boat and place 10 to 300 mg of Dried Sodium Sulfite on top. Again, spread evenly about 0.5 g of additive (a) and 1 g of additive (b) successfully to form layers. In the case of an automatic mercury analyzer equipped with a separate catalyst in the combustion chamber, take only the sample without adding the additives to a nickel boat. Place the boat inside the combustion furnace and heat to about 900 °C with a current of air or oxygen at 0.5 to 1 L/minute. Elute the mercury and collect in a collecting tube. Transfer the mercury vapor to a cold vapor atomic absorption spectrometer by heating the collecting tube to about 700 °C, and determine the absorbance, A. Separately, place only the additives in a ceramic boat and determine the absorbance, Ab, in the same manner. Separately, proceed with mercury standard solution in the same manner and create a calibration curve from the absorbances. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the sample (NMT 1.0 ppm).

Operating conditions
Apparatus: Use an automated mercury analyzer for specimen combustion, trapping with gold amalgam and measurement using a cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Standard mercury stock solution—Dissolve 0.135 g of mercury(II) chloride in 0.001% L-cysteine solution to make 1000 mL. 1 mL of this solution contains 100 μg of mercury(II) chloride.

Mercury standard solution—Dilute the standard mercury stock solution with 0.001% L-cysteine solution to make 0 to 200 ng/mL.

Excipients—Activate excipients at 950 ℃ for 30 minutes when using (a) aluminum oxide and (b) calcium hydroxide-sodium carbonate (1 : 1).

(5) ***Lead***—Weigh accurately 5.0 g of Dried Sodium Sulfite, transfer to a platinum crucible, dry, carbonize, and incinerate at 450 to 550 °C. If incineration is not complete, cool and moisten with 2 to 5 mL of nitric acid (1 in 2), 50% magnesium nitrate solution or aluminum nitrate-calcium nitrate solution (prepared

by dissolving 40 g of aluminum nitrate and 20 g of calcium nitrate in 100 mL of water) as an incineration supplement, dry, and continue with incineration. If incineration is incomplete, repeat the above procedure once and then add a final 2 to 5 mL of nitric acid (1 in 2), if necessary, and incinerate. After incineration, moisten the residue with water, add 2 to 4 mL of hydrochloric acid, and evaporate to dryness. Add 0.5 mol/L nitric acid, dissolve by warming, and filter with filter paper if any insoluble matter exists. Unless otherwise specified, add 0.5 mol/L nitric acid to make 25 mL and use this solution as the test solution. Separately, take 1.0 mL of lead standard solution to a platinum crucible, proceed in the same manner as the test solution, and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Atomic absorption spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 2.0 ppm).

Gas: Acetylene or hydrogen – Air
Lamp: A lead hollow-cathode lamp
Wavelength: 283.3 nm

(6) ***Iron*—**To 10.0 g of Dried Sodium Sulfite, add 25 mL of water, shake to mix until the mixture almost dissolves, add slowly and carefully 15 mL of hydrochloric acid TS, and boil. After cooling, add water to make 100 mL, and use this solution as the sample stock solution. Use 10.0 mL of this solution as the test solution. Separately, add water to 1 mL of iron standard solution to make 10 mL, and use this solution as the standard solution. This preparation is prepared just before use. To each of the test solution and the standard solution, add 2 mL of citric acid (2 in 10) and 0.1 mL of thioglycolic acid to mix. Alkalify with strong ammonia water, add water to make 20 mL, and allow to stand for 5 minutes; the color of the test solution is not more intense than that of the standard solution (NMT 10 ppm).

(7) ***Selenium*—**Dissolve 3.0 g of Dried Sodium Sulfite in 10 mL of formaldehyde, add slowly and carefully 2 mL of hydrochloric acid, and use this solution as the test solution. Separately, add 20 mL of selenium standard solution and 10 mL of formaldehyde to 1.0 g of Dried Sodium Sulfite, add slowly and carefully 2 mL of hydrochloric acid TS, and use this solution as the standard solution. Heat the test solution and the standard solution on a water bath for 20 minutes; the color of the test solution is not more intense than that of the standard solution (NMT 10 ppm).

(8) ***Zinc*—**To 10.0 g of Dried Sodium Sulfite, add 25 mL of water, shake to mix until the mixture almost dissolves, add slowly and carefully 15 mL of hydrochloric acid TS, and boil. After cooling, add water to make 100 mL, pipet 2.0 mL of this solution, and add water to make 10 mL. Use this solution as the test solution. Separately, dissolve 0.440 g of zinc sulfate ($ZnSO_4 \cdot 7H_2O$) and 1 mL of acetic acid in water to make 100 mL, and use this solution as the standard zinc stock solution. Pipet a suitable amount of this solution and dissolve in water to make a solution containing 25 μg per mL. Take 1.0 mL, 2.0 mL and 4.0 mL of this solution, respectively, add water to make exactly 100 mL so that each mL contains 0.25 μg, 0.5 μg and 1.0 μg of zinc, and use these solutions as the standard solutions. Perform the test with the test solution and the standard solutions as directed under the Atomic absorption spectroscopy. Determine the absorbances of the test solution and the standard solutions at the wavelength of 213.9 nm and calculate the concentration of zinc according to the calibration curve method (NMT 25 ppm).

(9) ***Arsenic*—**Dissolve 0.5 g of Dried Sodium Sulfite in 5 mL of water, add 1 mL of sulfuric acid, heat in a sand bath until white fumes are evolved, and then add water to make 5 mL. Use this solution as the test solution and perform the test (NMT 4 ppm).

Assay Weigh accurately about 0.2 g of Dried Sodium Sulfite, put immediately in an iodine bottle containing exactly 50 mL of 0.05 mol/L iodine solution, stopper the bottle, shake to mix, and allow to stand for 5 minutes in the dark. Add 1 mL of hydrochloric acid and titrate the excessive iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 1 mL of starch TS). Perform a blank test in the same manner.

Each mL of 0.05 mol/L iodine VS
= 6.302 mg of Na_2SO_3

Packaging and storage Preserve in tight containers.

Sorbitan Esters of Fatty Acids
소르비탄지방산에스테르

Description Sorbitan Esters of Fatty Acids occurs as white to yellowish brown powder, piece, granule, and waxy mass or liquid.

Identification (1) Dissolve 0.5 g of Sorbitan Esters of Fatty Acids in ethanol (99.5) by heating, add 5 mL of dilute sulfuric acid, heat on a water bath for 30 minutes, and then cool; an oil droplet or a white to pale yellow solid precipitates. Separate this oil droplet or the solid, add 5 mL of ether, and shake to mix; it dissolves.

(2) Take 2 mL of the remaining solution after separating the oil droplet or the solid from test (1), add 2 mL of freshly prepared catechol solution (1 in 20), shake to mix, and then add 5 mL of sulfuric acid. Shake to mix; the resulting solution exhibits a red to reddish brown color.

Purity (1) ***Acid value*—**Weigh accurately 5 g of Sorbitan Esters of Fatty Acids, add 100 mL of a mixture of ether and ethanol (95) (1 : 1), heat to dissolve, and use this solution as the test solution. Perform the test as directed under the Acid value of Fats and Fatty Oils; the value is NMT 10.

(2) ***Heavy metals*—**Proceed with 2 g of Sorbitan Esters of Fatty Acids according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 10 ppm).

(3) ***Arsenic***—Proceed with about 0.5 g of Sorbitan Esters of Fatty Acids according to Method 3, and perform the test (NMT 4 ppm).

(4) ***Polyoxyethylene***—Weigh 1 g of Sorbitan Esters of Fatty Acids, add 20 mL of water, shake well to mix while warming, and cool. Then, add 10 mL of ammonium thiocyanate-cobalt nitrate TS, shake well to mix, add 10 mL of chloroform, and then shake well to mix. Then, allow to stand; the chloroform layer does not exhibit a blue color.

Residue on ignition NMT 1.5% (2 g).

Sorbitan Sesquioleate
소르비탄세스퀴올레에이트

Anhydrohexitol sesquioleate
Sorbitani sesquioleas

Sorbitan Sesquioleate is a mixture of monoester and diester of sorbitol anhydride, partially esterified with oleic acid.

Description Sorbitan Sesquioleate is a pale yellow to pale yellowish brown, viscous oily liquid, and has a slightly characteristic odor and a slightly bitter taste.
It is freely soluble in ether, slightly soluble in ethanol, and very slightly soluble in methanol.
It is dispersed as fine oily drops in water.

Identification (1) To 0.5 g of Sorbitan Sesquioleate, add 5 mL of ethanol and 5 mL of dilute sulfuric acid, and heat on a water bath for 30 minutes. After cooling, add 5 mL of petroleum ether, shake to mix, allow to stand, and separate the upper layer and the lower layer. Add 2 mL of freshly prepared catechol solution (1 in 10) to 2 mL of the lower layer, shake to mix, add 5 mL of sulfuric acid, and shake to mix; the solution exhibits a red to reddish brown color.

(2) Heat the upper layer from (1) on a water bath to evaporate petroleum ether. To the residue, add 2 mL of diluted nitric acid (1 in 2) and then add 0.5 g of potassium nitrite at 30 to 35 ℃ while stirring; the solution produces white turbidity, and when cooled, crystals are formed.

Saponification value Between 150 and 168.

Specific gravity d_{25}^{25}: Between 0.960 and 1.020.

Purity (1) ***Acid***—To 2.0 g of Sorbitan Sesquioleate, add 50 mL of neutralized ethanol, and heat on a water bath nearly to boiling while shaking 1 to 2 times to mix. After cooling, add 4.3 mL of 0.1 mol/L sodium hydroxide and 5 drops of phenolphthalein TS; the color of the solution is red.

(2) ***Heavy metals***—Proceed with 1.0 g of Sorbitan Sesquioleate according to Method 2 and perform the test. Prepare the control solution with 1.0 mL of lead standard solution (NMT 10 ppm).

(3) ***Arsenic***—Proceed with 1.0 g of Sorbitan Sesquioleate according to Method 2 and perform the test (NMT 2 ppm).

Water NMT 3.0% (1 g, volumetric titration, direct titration, shaking to mix for 30 minutes).

Residue on ignition NMT 1.0% (1 g).

Packaging and storage Preserve in tight containers.

Soybean Oil
콩기름

Soybean Oil is a fatty oil obtained from the seeds of *Glycine max* Merril1 (*Leguminosae*).

Description Soybean Oil occurs as a clear pale yellow oil. It is odorless or has a slight odor, and has a mild taste.
It is miscible with ether or petroleum ether.
It is slightly soluble in ethanol and practically insoluble in water.
It congeals between -10 ℃ and -17 ℃.
Congealing point of fatty acids: Between 22 ℃ and 27 ℃

Saponification value Between l88 and 195

Unsaponifiable matter NMT 1.0%

Specific gravity d_{25}^{25}: Between 0.916 and 0.922

Acid value NMT 0.2

Iodine value Between 126 and 140

Purity (1) ***Free fatty acids***—Neutralize the free fatty acids contained in 10 g of Soybean Oil with 0.020 mol/L sodium hydroxide solution; NMT 2.5 mL of this solution is consumed.

(2) ***Peroxide value***—Weigh accurately 10 g of Soybean Oil, transfer to a stoppered Erlenmeyer flask, and dissolve in 30 mL of a mixture of acetic acid (100) and a chloroform mixture (3 : 2). To this solution, add 0.5 mL of saturated potassium iodide solution, shake to mix for exactly 1 minute, and add 30 mL of water. Titrate this solution with 0.01 mol/L sodium thiosulfate VS. When the solution turns pale yellow, add 0.5 mL of starch TS; it exhibits a blue color. The endpoint of the titration is when the blue color disappears. Perform a blank test in the same way and calculate the peroxide value according to the following formula; the value is NMT 10.0.

$$\text{Peroxide value (mEq/kg)} = [10\times(V_1\text{-}V_0)]/W$$

V_1: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed for titration of the test solution
V_0: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed for titration of the blank test solution
W: Amount (g) of Soybean Oil taken

(3) ***Heavy metals***—Proceed with 2.0 g of Soybean Oil according to Method 2 under the Heavy metals, and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 10 ppm).

Packaging and storage Preserve in tight containers.

Stearic Acid
스테아르산

Stearic Acid is a solid fatty acid obtained from fats and consists chiefly of stearic acid (C18H36O2 : 248.48) and palmitic acid (C16H32O2 : 256.42).

Stearic Acid type	Content
Stearic Acid 50	Stearic acid: 40.0 to 60.0%. The sum of stearic acid and palmitic acid is NLT 90.0%.
Stearic Acid 70	Stearic acid: 60.0 to 80.0%. The sum of stearic acid and palmitic acid is NLT 90.0%.
Stearic Acid 95	Stearic acid: NLT 90.0%. The sum of stearic acid and palmitic acid is NLT 96.0%.

Indicate the type of Stearic Acid (50, 70, 95).

Description Stearic Acid occurs as a white or almost white beeswax or crystalline mass or powder with a slightly fatty odor. It is soluble in ethanol (95) and petroleum ether and practically insoluble in water.

Identification Both the test solution and the standard solution

exhibit the same retention times in the chromatograms obtained by performing the test as directed under the Assay.

Acid value Between 194 and 212. However, weigh accurately 0.5 g of Stearic Acid and perform the test.

Iodine value Weigh accurately about 1.0 g of Stearic Acid, put it in a 250-mL iodine flask, and dissolve in 25 mL of iodine bromide(II) TS. Cover tightly, and allow to stand for 30 minutes, protected from light, with occasional shaking. Add 30 mL of potassium iodide TS and 100 mL of water, shake to mix, and titrate the liberated iodine with 0.1 mol/L sodium thiosulfate VS while shaking to mix. When the color of iodine becomes sufficiently pale, add 3 mL of starch TS, and titrate with 0.1 mol/L sodium thiosulfate VS until the blue color disappears. Perform a blank test in the same manner. At this time, calculate the iodine value according to the item of the iodine value of Fats and Fatty Oils; it meets the requirements.

Iodine(II) bromide TS—Dissolve 1000 mL of acetic acid (31) in 20 g of iodine bromide(II).

Stearic Acid type	Iodine value
Stearic Acid 50	NMT 4.0.
Stearic Acid 70	NMT 4.0.
Stearic Acid 95	NMT 1.5.

Congealing temperature When performing the test, it is as follows:

Stearic Acid type	Congealing temperature (℃)
Stearic Acid 50	53 to 59
Stearic Acid 70	57 to 64
Stearic Acid 95	64 to 69

Purity (1) ***Clarity and color of solution*—**Observe a solution obtained by heating Stearic Acid at 75 ℃ on a white background; the solution is not more intense than the control solution (1) or the control solution (2). The test tubes containing the test solution and the control solution should be identical and are colorless and transparent glass test tubes.

Control stock solution (1)—Mix 2.4 mL of the colorimetric stock solution of iron(III) chloride hexahydrate, 0.6 mL of the colorimetric stock solution of cobalt(II) chloride hexahydrate, and 7 mL of hydrochloric acid solution (1 in 100). The resulting solution exhibits a yellow color.

Control stock solution (2)—Mix 2.4 mL of the colorimetric stock solution of iron(III) chloride hexahydrate, 1 mL of the colorimetric stock solution of cobalt(II) chloride hexahydrate, 0.4 mL of the colorimetric stock solution of copper(II) sulfate pentahydrate, and 6.2 mL of hydrochloric acid solution (1 in 100). The resulting solution exhibits a yellowish brown color.

Control solution (1)—Add 97.5 mL of hydrochloric acid solution (1 in 100) to 2.5 mL of the control stock solution (1).

Control solution (2)—Add 97.5 mL of hydrochloric acid solution (1 in 100) to 2.5 mL of the control stock solution (2).

(2) ***Acidity*—**Warm 5 g of Stearic Acid to melt, add 10 mL of boiled water, shake to mix for 2 minutes, cool, filter, and add 0.05 mL of methyl orange TS to the filtrate; the resulting solution does not exhibit a red color.

Residue on ignition NMT 0.1% (1 g).

Assay Weigh accurately about 0.1 g of Stearic Acid and put in a small Erlenmeyer flask equipped with a reflux condenser. Add 5 mL of boron trifluoride-methanol TS, shake to mix, and heat for about 10 minutes until dissolved. Add 4 mL of heptane through the cooler and heat for about 10 minutes. After cooling, add 20 mL of saturated sodium chloride solution, shake to mix, and allow the layers to separate. Take 2 mL of the separated heptane layer, pass about 0.2 g of anhydrous sodium sulfate, previously washed with heptane, and put it in a separate flask. Transfer 1.0 mL of this solution to a 10-mL volumetric flask, add heptane to make 10 mL, shake to mix, and use this solution as the test solution. Perform the test with 1 μL of the test solution as directed under the Gas Chromatography according to the following conditions and determine the peak area A_S, of methyl stearate, the peak area A_P of methyl palmitate, and the sum of the peak areas of all fatty acid esters A_T. Calculate the amount (%) of stearic acid and palmitic acid in the fatty acid fraction of Stearic Acid according to the following formula.

$$\text{Amount (\%) of stearic acid} = (A_S / A_T) \times 100$$
$$\text{Amount (\%) of palmitic acid} = (A_P / A_T) \times 100$$

Operating conditions

Detector: A flame ionization detector

Column: A fused silica column about 0.32 mm in internal diameter and 30 m in length, coated with polyethylene glycol 20000 for gas chromatography in 0.5 μm thickness.

Column temperature: After injecting the sample, maintain a constant temperature of about 70 ℃ for about 2 minutes, increase the temperature by 5 ℃ per minute until it reaches 240 ℃, and maintain the constant temperature of about 240 ℃ for 5 minutes.

Sample injection port temperature: A constant temperature of about 220 ℃.

Detector temperature: A constant temperature of about 260 ℃.

Carrier gas: Helium

Flow rate: 2.4 mL/min

Split ratio: 2 : 1

System suitability

System performance: Proceed with 1 μL of the system suitability solution according to the above conditions; methyl palmitate and methyl stearate are eluted in this order, the relative retention time of methyl palmitate to methyl stearate is about 0.9 with the resolution being NLT 5.0.

System reproducibility: Repeat the test 6 times with 1 μL of the system suitability solution according to the above conditions; the relative standard deviation of the peak areas of methyl palmitate and methyl stearate is NMT 3.0%, respectively, and the relative standard deviation of the peak area ratio of methyl palmitate to the peak area of methyl stearate is NMT 1.0%.

System suitability solution: Put about 50 mg each of stearic acid RS and palmitic acid RS into a small Erlenmeyer flask equipped with a reflux condenser. Add 5.0 mL of boron trifluoride-methanol TS, shake to mix, and heat for about 10 minutes until dissolved. Proceed in the same manner as the test solution and use this solution as the system suitability solution.

Packaging and storage Preserve in well-closed containers.

Stearyl Alcohol
스테아릴알코올

Stearyl Alcohol is a mixture of solid alcohols and consists chiefly of stearyl alcohol ($C_{18}H_{38}O$:270.49).

Description Stearyl Alcohol occurs as a white, beeswax-like substance with a slightly peculiar odor and no taste.
It is freely soluble in ethanol, anhydrous ethanol or ether and practically insoluble in water.

Melting point Between 56 ℃ and 62 ℃. Prepare the sample according to the operation method in Method 2, attach the capillary to the lower part of the thermometer with a rubber band or other suitable method, and align the lower end of the capillary with the lower end of the thermometer. Insert this thermometer into a test tube 17 mm in internal diameter and about 170 mm in height. Fasten the thermometer with cork stopper so that the lower end of the thermometer is about 25 mm distant from the bottom of the test tube. Suspend the test tube in a beaker containing water, and heat the beaker with constant stirring. When the temperature reaches 5 °C below the expected melting point, continue to heat so that the temperature increases by 1°C per minute. The temperature at which the sample is transparent and no turbidity is produced is taken as the melting point.

Acid Value NMT 1.0.

Hydroxyl value Between 200 and 220.

Ester value NMT 3.0.

Iodine value NMT 2.0.

Purity (1) ***Clarity and color of solution***—Dissolve 3.0 g of Stearyl Alcohol in 25 mL of anhydrous ethanol by warming; the resulting solution is clear.

(2) ***Alkali***—Add 2 drops of phenolphthalein TS; the solution does not exhibit a red color.

Residue on ignition NMT 0.05% (2 g).

Packaging and storage Preserve in well-closed containers.

Succinylated Gelatin
숙신산젤라틴

Succinylated Gelatin is a succinylated derivative of gelatin obtained by reacting gelatin obtained from treating alkaline with succinic anhydride.

Description Succinylated Gelatin occurs as a pale yellow granule or powder.
It is odorless or has a slightly characteristic odor and a slightly salty taste.
It is very soluble in hot water and practically insoluble in ethanol (95) or ether.
It is insoluble in water, but when placed in water, it slowly swells and absorbs 5 to 10 times its mass of water.
The pH of an aqueous solution of Succinylated Gelatin (3 in 200) is 5.5 to 6.5.

Identification (1) To 5 mL of an aqueous solution of Succinylated Gelatin (1 in 100), slowly add about 1.5 mL of picric acid TS; a yellow precipitate forms.

(2) To 5 mL of an aqueous solution of Succinylated Gelatin (1 in 5000), add 2 to 3 drops of tannic acid TS; the resulting solution becomes turbid.

(3) Dissolve 1 g of Succinylated Gelatin in 20 mL of water by warming, and cool. Then, to 5 mL of this solution, add ammonia TS to adjust pH to 7.0, and slowly add 2 to 3 drops of iron(III) chloride TS; a brown precipitate forms.

Purity (1) ***Off-flavor and insolubles***—Dissolve 1.0 g of Succinylated Gelatin in 40 mL of water by heating; the resulting solution is odorless or has a slightly characteristic odor but not an unpleasant odor. Also, this solution is clear or slightly turbid, and the color is not more intense than the Matching fluids for color A in General tests.

(2) ***Sulfite***—Transfer about 20.0 g of Succinylated Gelatin into a round flask, dissolve in 150 mL of hot water, and add 3 to 5 drops of silicone resin, 5 mL of phosphoric acid, and 1 g of sodium bicarbonate. Immediately attach the condenser, add 50 mL of iodine TS to the collector, insert the end of the condenser into the solution, and distill until 50 mL of the distillate is obtained. To the distillate, slowly add hydrochloric acid to make acidic, add 2 mL of barium chloride TS, and heat on a water bath until the color of the iodine TS disappears. Then, filter to collect the precipitate, wash with water, and ignite; the residue is NMT 4.5 mg. Perform a blank test in the same manner and make any necessary correction.

(3) ***Ammonium***—Weigh 0.25 g of Succinylated Gelatin, transfer into 100 mL of water, allow to stand at room temperature for 20 to 30 minutes, and then heat at 45 ℃ to dissolve. Perform the test with 4 mL of this solution as directed under the Ammonium. Prepare the control solution with 3.0 mL of the ammonium standard solution (NMT 0.3%).

(4) ***Heavy metals***—Proceed with 0.5 g of Succinylated Gelatin according to Method 2 and perform the test. Prepare the control solution with 2.5 mL of lead standard solution (NMT 50 ppm).

(5) ***Arsenic***—Dissolve 15.0 g of Succinylated Gelatin in a flask, add 60 mL of diluted hydrochloric acid (1 in 5), and heat to dissolve. Then, add 15 mL of bromine TS, heat, evaporate excess bromine, and then add ammonia TS to make it neutral. Add 1.5 g of sodium phosphate dibasic, cool, then add 30 mL of magnesia TS, and allow to stand for 1 hour. Filter and collect the precipitate, wash 5 times each with 10 mL of ammonia TS (1 in 4), and dissolve in diluted hydrochloric acid (1 in 4) to make 50.0 mL. Take 5 mL of this solution, and perform the test as directed under the Arsenic. Prepare the control solution with 15.0 mL of arsenic standard solution, and proceed in the same manner (NMT 1 ppm).

(6) ***Free succinic acid***—Weigh about 5.00 g of Succinylated Gelatin, transfer into 250 mL of cold water, allow to stand at about 4 ℃ for 3 hours, and then filter through a glass filter. To 50 mL of the filtrate, add 6 mL of tannic acid TS, and shake to mix well. Allow to stand until a precipitate is formed, then filter, and transfer 28 mL of the filtrate into the strong base ion exchange resin column to efflux. Add 100 mL of water to efflux, then add 50 mL of a mixture of 2 mol/L hydrochloric acid and acetone (1 : 1) to efflux. Evaporate the effluent to dryness on a water bath in vacuum. To the residue, add 0.5 mL of anhydrous pyridine and 0.5 mL of bistrimethylsilylacetamide, seal, and heat at 125 ℃ for 10 minutes. After cooling, dissolve in anhydrous pyridine to make 10 mL, and use this solution as the test solution.

Also, to 50 mL of water, add 6 mL of tannic acid TS, proceed in the same manner as in the preparation of the test solution, and use this solution as the blank test solution.

Separately, dissolve about 0.100 g of succinic acid in water to make 100 mL. Take 1.0, 2.0, and 3.0 mL of this solution, evaporate each to dryness on a water bath, and proceed in the same manner as in the preparation of the test solution, and use these solutions as the standard solutions 1, 2, and 3, respectively. Perform the test with 5 μL each of the test solution, the blank test solution, and the standard solution as directed under the Gas Chromatography according to the following conditions, and determine the amount (mg) of succinic acid, A, in the test solution and the amount (mg) of succinic acid, B, in the blank test solution, using the calibration curve obtained from the standard solution. Determine the amount (%) of free succinic acid according to the following equation; it is NMT 0.50%.

$$\text{Amount (\%) of free succinic acid} = \frac{(A - B)}{500} \times 100$$

Operating conditions

Detector: A flame ionization detector

Column: A glass column about 3 mm in internal diameter and about 1.5 m in length, packed with diatomaceous earth for gas chromatography coated at a rate of 1.5% with methyl silicone polymer for gas chromatography (180 μm to 250 μm in particle diameter).

Column temperature: A constant temperature of about 120 ℃.

Carrier gas: Nitrogen

Flow rate: Adjust the flow rate so that the retention time of succinic acid is about 2.5 minutes.

Loss on drying NMT 15.0%.

Weigh accurately about 1 g of Succinylated Gelatin, and transfer into a 200-mL beaker, previously weighed, with 10 g of sea sand, previously dried at 110 ℃ for 3 hours, add 20 mL of water, and allow to stand for 30 minutes while occasionally shaking to mix. Then, evaporate to dryness on a water bath while occasionally shaking to mix, and dry at 110 ℃ for 3 hours.

Residue on ignition NMT 3.5% (0.5 g).

Bonded succinic acid Dissolve 1.0 g of Succinylated Gelatin in 6 mol/L hydrochloric acid to make 50 mL, and heat at 130 ℃ for 24 hours under a reflux condenser. After cooling, filter, then transfer 10 mL of the filtrate into a conical flask, and evaporate to dryness on a water bath in vacuum. To the residue, add about 10 mL of hot water, then evaporate to dryness on a water bath in vacuum. Then, repeat the same procedure, dissolve the residue in 10 mL of hot water, and transfer into a 100-mL beaker. Wash the flask with 50 mL of water and transfer the washings into the beaker. Adjust the pH to 7.0 with 0.1 mol/L sodium hydroxide, and then transfer into the strong base ion exchange resin column to efflux. Then, add 100 mL of water to efflux, then add 50 mL of a mixture of 2 mol/L hydrochloric acid and acetone (1 : 1) to efflux, and obtain 50 mL of the effluent. Transfer 25 mL of this solution into a conical flask, proceed as directed under the Free succinic acid of Purity (6), and use this solution as the test solution. Separately, weigh 0.10 g of succinic acid, and add water to make 100 mL. Take 1.0, 3.0, and 5.0 mL of this solution, evaporate each to dryness on a water bath, and proceed in the same manner as in the preparation of the test solution, and use these solutions as the standard solutions 1, 2, and 3, respectively. Perform the test with 5 μL each of the test solution and the standard solution as directed under the Gas Chromatography according to the conditions in Free succinic acid of Purity (6), and determine the amount (mg) of succinic acid, A, in 10 mL of the test solution, using the calibration curve obtained from the standard solution. Determine the amount (%) of bonded succinic acid according to the following equation; it is NMT 3.0%.

$$\text{Amount (\%) of bonded succinic acid} = \left(\frac{A}{100} \times 100\right) - \text{Amount (\%) of free succinic acid}$$

Replacement ratio Weigh accurately about 5.0 g of Succinylated Gelatin, add 200 mL of water, allow to stand at room temperature for 20 to 30 minutes, then warm at 45 ℃ to dissolve, and add 0.5 mL of dilute hydrochloric acid and water to make 205 mL. Transfer this solution into a 300-mL beaker, add 5 g of strongly acidic cation-exchange resin and 10 g of strong base anion-exchange resin, shake well to mix for about 20 minutes by warming at 45 ℃, and then filter through a glass filter. Use this solution as the test solution. Transfer 200 mL of the test solution into a beaker, and adjust the pH to 7.5 by slowly adding 0.1 mol/L sodium hydroxide VS while warming at 35 ℃ and shaking to mix well. Add 1 mL of formalin, the pH previously adjusted to 7.5 with 0.1 mol/L sodium hydroxide VS, shake for 3 minutes to mix well, then slowly add 0.1 mol/L sodium hydroxide VS, and titrate until the pH becomes 7.5. Separately, perform a blank test with gelatin (pharmacopeia) in the same manner and make any necessary correction. Determine the replacement ratio of Succinylated Gelatin, calculated on the anhydrous basis by loss on drying; it is NLT 95.0%.

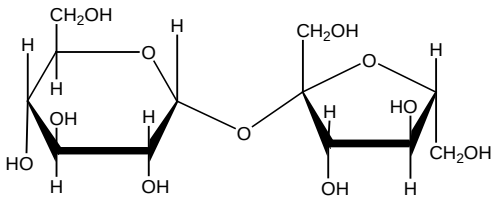
$$\text{Replacement ratio (\%)} = \frac{b - a}{b} \times 100$$

$$a = \frac{volume\ comsumed\ for\ titration\ (mL)\ of\ test\ solution}{100 - loss\ on\ drying\ (\%)\ of\ the\ sample}$$

$$b = \frac{volume\ comsumed\ for\ titration\ (mL)\ of\ blank\ test\ solution}{100 - loss\ on\ drying\ (\%)\ of\ gelatin}$$

Packaging and storage Preserve in tight containers.

Sucrose
백당



$C_{12}H_{22}O_{11}$: 342.30

(2*R*,3*S*,4*R*,5*R*,6*S*)-2-{[(2*R*,3*R*,4*R*,5*S*)-3,4-Dihydroxy-2,5-*bis*(hydroxymethyl)oxolan-2-yl]oxy}-6-(hydroxy-methyl)oxane-3,4,5-triol [*57-50-1*]

Description Sucrose occurs as a colorless or white crystal or crystalline powder. It is odorless and has a sweet taste.

It is very soluble in water, very slightly soluble in ethanol and

practically insoluble in ether.
An aqueous solution of Sucrose (1 in 10) is neutral.

Identification (1) Heat 1 g of Sucrose; it melts to swell, giving off a caramel odor and becoming a bulky carbide.

(2) Boil 0.1 g of Sucrose with 2 mL of dilute sulfuric acid, add 4 mL of sodium hydroxide TS and 3 mL of Fehling's TS, and heat the mixture until boiling; a red to dark red precipitate is formed.

Specific optical rotation $[\alpha]_D^{20}$: Between $+65.0^\circ$ and $+67.0^\circ$ (after drying, 13 g, 50 mL of water, 100 mm).

Purity (1) ***Clarity and color of solution***—Dissolve 100 g of Sucrose in 100 mL of water, pour 50 mL of this solution into a Nessler tube, and observe from the side using a white background; the resulting solution is colorless or exhibits a slightly yellow color with no blue color. Fill a Nessler tube with this solution again, stopper, and allow it to stand for 2 days; no precipitate is formed.

(2) ***Chloride***—Dissolve 10.0 g of Sucrose in water to make 100 mL, and use this solution as the test solution. To 20 mL of this solution, add 6 mL of dilute nitric acid and water to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution by adding 0.30 mL of 0.01 mol/L hydrochloric acid (NMT 0.005%).

(3) ***Sulfate***—Add 1 mL of dilute hydrochloric acid and water to 40 mL of the test solution prepared in (2) to make 50 mL. Use this solution as the test solution and perform the test. Prepare the control solution by adding 0.5 mL of 0.005 mol/L sulfuric acid (NMT 0.006%).

(4) ***Sulfur dioxide***—NMT 20 ppm.

(5) ***Calcium***—Add 1 mL of ammonium oxalate TS to 10 mL of the test solution prepared in (2); the resulting solution does not change immediately.

(6) ***Heavy metals—***Weigh 5.0 g of Sucrose and perform the test according to Method 2. Prepare the control solution by adding 2.5 mL of the lead standard solution (NMT 5 ppm).

(7) **Lead**—Weigh accurately 50 mg of Sucrose and place it in a decomposition container made of polytetrafluoroethylene, add 0.5 mL of nitric acid to dissolve, seal, and heat at 150 °C for 5 hours. After cooling, add water to make exactly 5 mL, and use this solution as the test solution. Take NLT 3 test solutions, and perform the test under the following conditions according to the standard addition method of atomic absorption spectrophotometry (electric heating method). Prepare the standard solution by taking an appropriate amount of the lead standard solution and adding water. Pipet 10.0 mL of nitric acid, and add water to make exactly 100 mL. Perform a blank test in the same manner and make any necessary correction (NMT 0.5 ppm).

Lamp: A lead hollow-cathode lamp
Wavelength: 283.3 nm
Drying temperature: 110 ℃
Incineration temperature: 600 ℃
Atomization temperature: 2100 ℃

(8) ***Arsenic***—Weigh 1.0 g of Sucrose and perform the test according to Method 1 (NMT 2 ppm).

(9) ***Inverted sugar—***Dissolve 5.0 g of Sucrose in water to make 100 mL, and use this solution as the test solution. Separately, place 100 mL of alkaline copper sulfate TS in a 300-mL beaker, cover it with a watch glass, and boil. Add immediately 50.0 mL of the test solution, and boil for exactly 5 minutes. Add immediately 50 mL of freshly boiled and cooled water, put it on a water bath below 10 °C for 5 minutes, and filter the precipitate using a glass filter previously weighed. Wash the filtrate with water until it becomes neutral, wash it again with 10 mL of ethanol and 10 mL of ether, and dry at 105°C for 30 minutes; the amount of the resulting residue is NMT 0.120 g.

Loss on drying NMT 1.30% (15 g, 105 ℃, 2 hours).

Residue on ignition NMT 0.10% (2 g).

Packaging and storage Preserve in well-closed containers.

Powdered Sucrose
분말백당

Powdered Sucrose is a ground powder of sucrose, with corn starch added to prevent clumping.

Powdered Sucrose, when dried, contains 96.0% to 99.0% of sucrose ($C_{12}H_{22}O_{11}$: 342.30) and 1.0% to 4.0% of corn starch.

Description Powdered Sucrose occurs as a white powder.
It is odorless and has a sweet taste.
It is practically insoluble in ethanol (95) or diethyl ether.
It is mostly dissolved in water, leaving a small amount of insoluble.

Identification (1) Heat 1 g of Powdered Sucrose; it melts and expands, producing a caramel aroma.

(2) To 0.1 g of Powdered Sucrose, add 2 mL of dilute sulfuric acid, boil, then add 4 mL of sodium hydroxide TS and 3 mL of Fehling's TS, and boil; a red to dark red precipitate forms.

(3) To 1 g of Powdered Sucrose, add 10 mL of water, shake to mix, and then filter. To the precipitate on the filter paper, add iodine TS; it exhibits a dark bluish purple color.

Loss on drying NMT 2.0% (1 g, 105 ℃, 2 hours).

Particle size distribution estimation by analytical sieving Transfer 5.0 g of Powdered Sucrose into a No. 100 (150 μm) sieve, and lightly rub the sieve with a soft brush; the residue on the sieve is NMT 0.2 g.

Assay (1) ***Sucrose***—Weigh accurately about 13 g of Powdered Sucrose, previously dried, add 50 mL of water, and shake for 30 minutes to mix. Filter this solution through a glass filter (G_4), wash with 30 mL of water, then combine the filtrate and the washings, and add water to make 200 mL. Determine the optical rotation, $[\alpha]_D^{20}$, with this solution as directed under the Optical rotation at the layer length of 100 mm at 20 ± 1 ℃.

$$\text{Amount (\%) of sucrose } (C_{12}H_{22}O_{11}) = \frac{Optical\ rotation\ of\ test\ solution}{66.5} \times 100$$

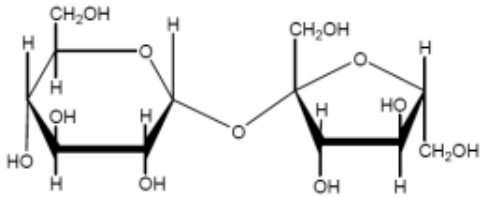
(2) ***Corn starch***—Weigh accurately about 10 g of Powdered Sucrose, previously dried, add 50 mL of water, and shake for 30 minutes to mix. Filter this solution through a glass filter (G_4) with a known mass, wash the residue 5 times each with 10 mL of water, then dry at 105 ℃ for 1 hour, and weigh the mass accurately.

Amount (%) of corn starch

$$= \frac{Amount\ (g)\ of\ dry\ matter}{Amount\ (g)\ of\ sample} \times\ 100$$

Packaging and storage Preserve in well-closed containers.

Purified Sucrose
정제백당



$C_{12}H_{22}O_{11}$: 342.30

Purified Sucrose contains no additives.

The label indicates its use in the preparation of large-volume solutions.

Description Purified Sucrose occurs as a white or almost white crystalline powder or shiny, colorless or white or almost white crystals.

It is very freely soluble in water, slightly soluble in ethanol (95) and practically insoluble in ethanol (99.5).

Identification Determine the infrared spectra of Purified Sucrose and purified sucrose RS as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; both spectra exhibit similar intensities of absorption at the same wavenumbers.

Optical rotation $[\alpha]_D^{20}$: Between + 66.3˚ and + 67.0˚ (26.0 g, water, 100 mL, 100 mm).

Purity (1) ***Color index***—Weigh 50.0 g of Purified Sucrose, add exactly 50 mL of water to mix, filter through a filter with a pore size of 0.45 μm, and use the degassed solution as the test solution. Determine the absorbance of the test solution at a wavelength of 420 nm using a cell with a length of at least 4 cm (a recommended layer of NLT 10 cm) as directed under the Ultraviolet-visible Spectroscopy. Determine the color index according to the following equation; use NMT 45 for the preparation of injections and NMT 75 for other preparations.

Color index = (A ×1000) / (b × c)

A: Absorbance (wavelength: 420 nm)

b: Cell length (cm)

c: Concentration (g/mL) of the test solution calculated from the refractive index (n_D^{20}) as directed under the Refractive index. Extrapolate the values using the following table if necessary.

n_D^{20}	c (g/mL)
1.4138	0.570
1.4159	0.585
1.4179	0.600
1.4200	0.615
1.4221	0.630
1.4243	0.645
1.4264	0.661

System suitability

System repeatability: Repeat the test twice with the test solution under the above conditions; the difference between the result values is within 3.

(2) ***Clarity and color of solution***—Weigh 50.0 g of Purified Sucrose, dissolve in water to make 100 mL, and use this solution as the test solution. The test solution is as clear as water and is less turbid than the control suspension.

Control suspension—Pipet 25 mL of hydrazinium sulfate TS left for 4 to 6 hours, mix it with a solution containing 2.5 g of hexamethyltetramine dissolved in 25 mL of water, mix well, and allow the mixture to stand for 24 hours. Store the solution in a glass container and use it within 2 months. Before use, pipet 15 mL of the suspension, add water to make 1000 mL, and shake well to mix 5 mL of this solution with 95 mL of water. Use this solution as the control suspension.

(3) ***Sulfite***—Sulfurous acid is oxidized to sulfuric acid and hydrogen peroxide by sulfite oxidase, which is then reduced by nicotinamide adenine dinucleotide peroxidase in the presence of reduced nicotinamide adenine dinucleotide (NADH). The amount of NADH oxidized is proportional to the amount of sulfite. The amount of oxidized NADH is calculated from a decrease in absorbance at a wavelength of 340 nm. Appropriate kits are available.

Procedure Weigh 4.0 g of Purified Sucrose, dissolve in freshly prepared distilled water to make 10 mL, and use this solution as the test solution. Separately, weigh 4.0 g of Purified Sucrose, dissolve in freshly prepared distilled water, add 0.5 mL of the standard sulfurous acid solution, and add freshly prepared distilled water to make 10 mL; use this solution as the standard solution. Use freshly prepared distilled water as a blank test solution. Separately, place 2.0 mL each of the test solution, the standard solution, and the blank test solution in a 1-cm cell. Add 1.00 mL of β-nicotinamide adenine dinucleotide (β-NADH) TS and 10 μL of reduced β-NADH oxidase TS, and stir the solution for 20 to 25 minutes. Allow it to stand for 5 minutes at 20 to 25 ℃. Determine the absorbances, A_{T1}, A_{S1} and A_{B1}, at a wavelength of 340 nm with each solution as directed under the Ultraviolet-visible Spectroscopy. Add 50 μL of sulfurous acid oxidase TS to each solution, mix, allow them to stand for 30 minutes at 20 to 25°C, and determine the absorbances, A_{T2}, A_{S2}, and A_{B2}, in the same manner. The difference in the absorbance of the test solution (A_{T1} - A_{T2}) - (A_{B1} - A_{B2}) is not greater than half of the difference in the absorbance of the standard solution (A_{S1} - A_{S2}) - (A_{B1} - A_{B2}) (NMT 10 ppm as SO_2).

Sulfurous acid RS—Weigh accurately 3.150 g of anhydrous sodium sulfite, and dissolve in freshly prepared distilled water to make 100 mL. Add freshly prepared distilled water to 0.5 mL of this solution to make 100 mL. Prepare before use.

Reduced β-nicotinamide adenine dinucleotide TS (reduced β-NADH) TS—Dissolve 0.4 mg of reduced β-nicotinamide adenine dinucleotide (β-NADH)in 1 mL of a solution prepared by dissolving 5.57 g of 2,2',2″-nitrilotriethanol hydrochloric acid in 40 mL of water, adjusting the pH to 8.0 with 5 mol/L sodium hydroxide TS, and adding water to make 50 mL. Prepare before use.

NADH oxidase solution—Suspend NADH oxidase in an ammonium sulfate solution, prepared by dissolving 39.6 g of ammonium sulfate in 70 mL of water, adjusting the pH to 8.0 with sodium hydroxide TS, and adding water to make 100 mL, so that 1 mL of this TS contains 10 units of NADH oxidase activity.

NADH oxidase—Use reduced β-nicotinamide adenine dinucleotide (β-NADH) and hydrogen peroxide as substrates; the enzyme activity that consumes 1 μmol of β-NADH in 1 minute at 25°C and pH 8.0 is considered as 1 unit of NADH oxidase activity.

Sulfurous acid oxidase solution—Suspend sulfurous acid oxidase in ammonium sulfate solution, prepared by dissolving 39.6 g of ammonium sulfate in 70 mL of water, adjusting the pH to 8.0 with sodium hydroxide TS, and adding water to make 100 mL, so that 1 mL of this TS contains 2.5 units of NADH oxidase activity.

Sulfurous acid oxidase—Use sulfur dioxide and oxygen as substrates; the enzyme activity that consumes 1 μmol of oxygen for 1 minute at 25 ℃ and pH 8.0 is considered as 1 unit of sulfite oxidase activity.

(4) ***Reducing sugars***—Take 5 mL of the test solution from (2) into a test tube with a length of about 150 mm and a diameter of about 16 mm, add 5 mL of water, 1.0 mL of 1 mol/L sodium hydroxide TS, and 1.0 mL of methylene blue TS, shake to mix, and warm the mixture on a water bath for exactly 2 minutes. Observe it immediately after taking it out of the water bath; the blue color of the solution does not completely disappear. However, exclude the blue color of the surface in contact with air.

Conductivity Weigh accurately 31.3 g of Purified Sucrose, and dissolve in freshly boiled and cooled water to make 100 mL. Use this solution as the test solution. Determine the conductivity of the test solution (C_1) as directed under the Conductivity by slowly shaking to mix with a magnetic stirrer at 20 ± 0.1 ℃ for 30 seconds, and determine the conductivity of the water (C_2) used to prepare the test solution in the same way. Stabilize the conductivity determined for 30 seconds within 1%; the conductivity of Purified Sucrose is NMT 35 $\mu S \cdot cm^{-1}$ when calculated according to the following formula.

$$\text{Conductivity } (\mu S \cdot cm\text{-}1) = C1 - (0.35 \times C2)$$

C_1: Conductivity of the test solution
C_2: Conductivity of water used to prepare the test solution

Loss on drying NMT 0.1% (2 g, 105 ℃, 3 hours).

Dextrin Add 8 mL of water, 0.05 mL of 2 mol/L hydrochloric acid TS and 0.05 mL of 0.05 mol/L iodine TS to 2 mL of the test solution in (2) for those used for the preparation of large volume parenteral infusions; the yellow color of the solution does not disappear.

0.05 mol/L iodine TS—Dissolve 12.7 g of iodine and 20 g of potassium iodide in water to make 1000 mL.

Standardization. Add 1 mL of dilute acetic acid and 40 mL of water to 10.0 mL of 0.05 mol/L iodine TS. Titrate with 0.1 mol/L sodium thiosulfate VS (indicator: starch solution).

Storage: light-resistant

Bacterial endotoxins Less than 0.25 EU per mg of Purified Sucrose for those used for the preparation of large volume parenteral infusions.

Packaging and storage Preserve in well-closed containers.

Sucrose Esters of Fatty Acids
백당지방산에스테르

Sucrose Esters of Fatty Acids is esters of fatty acid and sucrose.

Description Sucrose Esters of Fatty Acids occurs as white to pale yellowish brown powder, mass, or colorless to pale yellowish brown viscous resin-like substance.
It is odorless or has a characteristic odor and is tasteless or has a bitter taste.
It is very soluble or freely soluble in *n*-butanol and chloroform and soluble or practically insoluble in water, ethanol (95) and diethyl ether.

Identification (1) To 1 g of Sucrose Esters of Fatty Acids, add 25 mL of 0.5 mol/L potassium hydroxide-ethanol TS, heat on a water bath for 1 hour under a reflux condenser, then add 50 mL of water, and heat to concentrate on a water bath until the solution becomes 30 mL. After cooling, add 10 mL of dilute hydrochloric acid to this solution, shake well to mix, and then warm until there is no diethyl ether odor. After cooling, slowly add 1 mL of anthrone TS along the inner wall; the interface exhibits a blue to green color.

(2) Filter the diethyl ether layer obtained from test (1) through an absorbent cotton containing 2 g of anhydrous sodium sulfate, then evaporate diethyl ether on a water bath, and cool the residue at 10 ℃; a colorless to pale yellowish brown oil droplet or white to pale yellowish brown solid precipitates.

Acid value NMT 5.0.

Purity (1) ***Heavy metals***—Proceed with 1.0 g of Sucrose Esters of Fatty Acids according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 20 ppm).

(2) ***Arsenic***—Prepare the test solution with 2.0 g of Sucrose Esters of Fatty Acids according to Method 3 and perform the test (NMT 1 ppm).

(3) ***N,N-Dimethylformamide***—Weigh accurately about 10.0 g of Sucrose Esters of Fatty Acids, transfer into a 200-mL flask, add 100 mL of 0.1 mol/L sodium hydroxide-methanol TS, attach a reflux condenser, and connect a condenser to the reflux condenser. Immerse the end of the condenser in a test tube containing 10 mL of 0.01 mol/L methanolic hydrochloric acid TS. Heat on a water bath for 30 minutes to reflux, remove the coolant from the reflux condenser, and distill until 50 mL of the distillate is obtained. Heat this solution on a water bath, concentrate almost to dryness, then dissolve this residue in 10 mL of water, and transfer into a separatory funnel. Wash the test tube 3 times each with 10 mL of water, collect the washings, combine with the solution in the separatory funnel, and use this solution as the test solution. To the test solution, add 10 mL of a mixture of chloroform and carbon disulfide (20 : 1) and 5 mL of ammonia TS, and shake vigorously for 2 minutes to mix. To this solution, add 1 mL

of copper sulfate-ammonia TS, shake vigorously for 1 minute to mix, then take the lower layer, and dehydrate with anhydrous sodium sulfate; the resulting solution is not more intense than that of the following control solution.

Control solution—Weigh accurately about 1.116 g of dimethylamine hydrochloride, dissolve in water to make exactly 1000 mL, then pipet 1 mL of this solution and add water to make exactly 1000 mL. Transfer 10 mL of this solution into a separatory funnel, add 30 mL of water, and proceed in the same manner as in the preparation of the test solution.

(4) ***Free sucrose***—Weigh about 2.0 g of Sucrose Esters of Fatty Acids, transfer into 40 mL of n-butanol, warm on a water bath to dissolve, and then extract this solution 2 times with 20 mL each of sodium chloride solution (1 in 20). Combine the extracts, add 2 mL of dilute hydrochloric acid, and heat on a water bath for 30 minutes. After cooling, add 2 to 3 drops of phenolphthalein TS, neutralize with sodium hydroxide TS, and add water to make exactly 100 mL. Take 20 mL of this solution, add 40 mL of Fehling's TS, and boil gently for 3 minutes. After cooling, carefully filter the supernatant solution through a glass filter (G_4) so that the precipitate remains in the flask, wash the precipitate with hot water until the washings do not exhibit alkalinity, filter the washings through the used glass filter (G_4), and then wash with water. Combine the filtrate and washings, and heat at 80 ℃. Titrate this solution with 0.02 mol/L potassium permanganate VS; the consumed amount is NMT 6.6 mL.

Water NMT 4.0% (0.5 g, back titration).

Residue on ignition NMT 1.5% (1 g).

Packaging and storage Preserve in well-closed containers.

Talc
탤크

Talc is a triturated, selected, natural hydrous magnesium silicate. The molecular formula of pure talc is $Mg_3Si_4O_{10}(OH)_2$ (379.27). Talc sometimes contains small amounts of chlorite (hydrous aluminum and magnesium silicate), magnesite (magnesium carbonate), calcite (calcium carbonate) and dolomite (calcium carbonate and magnesium carbonate).

Talc does not contain asbestos.

Talc contains NLT 17.0% and NMT 19.5% of magnesium (Mg: 24.31).

If applicable, Talc is labeled that it is suitable for oral or dermal application.

Description Talc occurs as a white to grayish white, fine crystalline powder, and is odorless and tasteless.
It has a smooth texture and adheres easily to the skin.
It is practically insoluble in water, ethanol (95), dilute acid and dilute hydroxide alkaline solution.

Identification (1) Mix 0.1 g of Talc with 0.2 g of anhydrous sodium carbonate and 2.0 g of potassium carbonate, and heat in a platinum crucible until completely dissolved. After cooling, transfer the fused mixture to a beaker with the aid of 50 mL of hot water, add hydrochloric acid until no bubbles appear, then add 10 mL more of hydrochloric acid, and evaporate to dryness on a water bath. After cooling, add 20 mL of water, boil, and filter. To 5 mL of the filtrate, add 1 mL of ammonium chloride TS and 1 mL of ammonia TS, filter if necessary, and add 1 mL of disodium hydrogen phosphate TS; a white crystalline precipitate is formed.

(2) Place the insoluble residue obtained in (1) in a lead or platinum crucible, add 10 mg of sodium fluoride and a few drops of sulfuric acid, and stir with a copper wire to create a slurry state. Cover the crucible with thin, transparent plastic, drop a drop of water underneath it, and then slowly warm it up; a white ring rapidly forms around the water droplet in a short time.

(3) Determine the infrared absorption spectrum of Talc as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; it exhibits maxima at the wavenumbers of 3677 ± 2 cm^{-1}, 1018 ± 2 cm^{-1} and 669 ± 2 cm^{-1}.

Purity (1) ***Acid or alkali***—To 2.5 g of Talc, add 50 mL of freshly boiled and cooled water, heat under a reflux condenser, and filter in a vacuum. To 10 mL of filtrate, add 0.1 mL of bromothymol blue solution, and add 0.01 mol/L hydrochloric acid until the color of the solution turns green; the amount is NMT 0.4 mL. Separately, to 10 mL of filtrate, add 0.1 mL of phenolphthalein TS, and add 0.01 mol/L sodium hydroxide solution until the color of the solution turns pink; the amount is NMT 0.3 mL.

(2) ***Acid soluble substances***—Weigh accurately about 1.0 g of Talc, add 20 mL of dilute hydrochloric acid, warm at 50 ℃ for 15 minutes while stirring to mix. After cooling, add water to make exactly 50 mL, and filter. If necessary, centrifuge the filtrate until it becomes clear. Take 25 mL of this solution, add 1 mL of dilute sulfuric acid, evaporate to dryness, and ignite at 800 ± 25 ℃ to a constant mass; the residue is NMT 2.0%.

(3) ***Water-soluble substances***—To 10 g of Talc, add 50 mL of freshly boiled and cooled water, and heat under a reflux condenser for 30 minutes. After cooling, filter, and add freshly boiled and cooled water to make 50 mL. Take 25.0 mL of the filtrate, evaporate to dryness, and dry the residue at 105 ℃ for 1 hour; the amount is NMT 10.0 mg.

(4) ***Lead***—Weigh 10.0 g of Talc, add 50 mL of 0.5 mol/L hydrochloric acid TS while stirring slowly, attach a reflux condenser, and heat on a water bath for 30 minutes. After cooling, transfer the solution to a beaker, and allow to stand. Filter the clear supernatant, retaining as much as possible of the settled precipitate in the beaker. Wash the beaker and precipitate three times with 10 mL each of hot water, filter, and wash the filter paper with 15 mL of hot water. Cool the filtrate, add water to make 100 mL, and use this solution as the test solution. Separately, place 5.0 mL, 7.5 mL, 10.0 mL and 12.5 mL of lead standard solution in volumetric flasks, each containing 50 mL of 0.5 mol/L hydrochloric acid TS, add water to make 100 mL, and use these solutions as the standard solutions. Perform the test with the test solution and the standard solutions as directed in the calibration curve method under the Atomic absorption spectroscopy, and determine the content of lead in the test solution; NMT 10 ppm.

Gas: Acetylene - Air
Lamp: Lead hollow-cathode lamp
Wavelength: 217.0 nm

(5) ***Aluminum***—[Be careful when handling a mixture of perchloric acid and heavy metals as it is explosive.] Place 0.5 g of Talc in a polytetrafluoroethylene dish, add 5 mL of hydrochloric acid, 5 mL of nitric acid (lead-free) and 5 mL of perchloric acid, and stir slowly. Then add 35 mL of hydrofluoric acid, and slowly evaporate to dryness until the volume of the residue reaches about 0.5 mL. Add 5 mL of hydrochloric acid to the residue, cover with a watch glass, heat to dissolve, and then cool.

Transfer to a volumetric flask, wash the polytetrafluoroethylene dish and the watch glass with water, combine all the washings, and add water to make 50 mL. Take 5 mL of this solution, add 10 mL of cesium chloride TS and 10 mL of hydrochloric acid, then add water to make 100 mL, and use this solution as the test solution. Separately, weigh 8.947 g of aluminum chloride, and add water to make 1000 mL. Just before use, take 10 mL of this solution, add water to make 100 mL, and use this solution as the aluminum standard stock solution. Each mL of this solution contains 100 μg of aluminum. Place 5.0 mL, 10.0 mL, 15.0 mL and 20.0 mL of the aluminum standard stock solution in volumetric flasks, each containing 10 mL of hydrochloric acid and 10 mL of cesium chloride TS, add water to make 100 mL, and use these solutions as the standard solutions. Perform the test with the test solution and the standard solutions as directed in the calibration curve method under the Atomic absorption spectroscopy according to the following conditions, and determine the content of aluminum in the test solution; NMT 2.0%.

Gas: Acetylene – Nitrous oxide
Lamp: Aluminum hollow cathode lamp
Wavelength: 309.3 nm

Cesium chloride TS—Add water to 2.53g of cesium chloride to make 100ml.

(6) ***Iron***—Weigh 10.0 g of Talc, add 50 mL of 0.5 mol/L hydrochloric acid TS while stirring slowly, attach a reflux condenser, and heat on a water bath for 30 minutes. After cooling, transfer the solution to a beaker, and allow to stand. Filter the clear supernatant, retaining as much as possible of the settled precipitate in the beaker. Wash the beaker and precipitate three times with 10 mL of hot water, filter, and wash the filter paper with 15 mL of hot water. Cool the filtrate, and add water to make 100 mL. Take 2.5 mL of this solution, add 50 mL of 0.5 mol/L hydrochloric acid TS, add water to make 100 mL, and use this solution as the test solution. Separately, dissolve 4.840 g of ferric chloride in hydrochloric acid solution (150 in 1000) so that each mL of this solution contains 250 μg of Iron(Fe), and use this solution as the iron standard stock solution. Prepare before use. Place 2.0 mL, 2.5 mL, 3.0 mL and 4.0 mL of the iron standard stock solution in volumetric flasks, each containing 50 mL of 0.5 mol/L hydrochloric acid TS, add water to make 100 mL, and use these solutions as the standard solutions. Perform the test with the test solution and the standard solutions as directed in the calibration curve method under the Atomic absorption spectroscopy according to the following conditions, and determine the content of iron in the test solution; NMT 0.25%.

Gas: Acetylene - Air
Lamp: Iron hollow cathode lamp
Wavelength: 248.3 nm

(7) ***Calcium***—[Be careful when handling a mixture of perchloric acid and heavy metals as it is explosive.] Place 0.5 g of Talc in a polytetrafluoroethylene dish, add 5 mL of hydrochloric acid, 5 mL of nitric acid (lead-free) and 5 mL of perchloric acid, and stir slowly. Then add 35 mL of hydrofluoric acid, and slowly evaporate to dryness until the volume of the residue reaches about 0.5 mL. Add 5 mL of hydrochloric acid to the residue, cover with a watch glass, heat to dissolve, and then cool. Transfer this solution to a 50-mL volumetric flask, wash the polytetrafluoroethylene dish and watch glass with water, combine all the washings, and add water to make 50 mL. Take 5.0 mL of this solution, add 10 mL of hydrochloric acid and 10 mL of lanthanum chloride TS, then add water to make 100 mL, and use this solution as the test solution. Separately, take 3.67 g of calcium chloride dihydrate, and dissolve in dilute hydrochloric acid to make 1000 mL. Just before use, take 10 mL of this solution, add water to make 100 mL, and use this solution as calcium standard stock solution. Each mL of this solution contains 100 μg of calcium. Place 1.0 mL, 2.0 mL, 3.0 mL and 4.0 mL of the calcium standard stock solution in volumetric flasks, each containing 10 mL of hydrochloric acid and 10 mL of lanthanum chloride TS, add water to make 100 mL, and use these solutions as the standard solutions. Perform the test with the test solution and the standard solutions as directed in the calibration curve method under the Atomic absorption spectroscopy according to the following conditions, and determine the content of calcium in the test solution; NMT 0.9%.

Gas: Acetylene - Nitrous oxide
Lamp: Calcium hollow cathode lamp
Wavelength: 422.7 nm

Lanthanum chloride TS—Add slowly 10 mL of hydrochloric acid to 5.9 g of lanthanum oxide and heat. After cooling, add water to make 100 mL.

(8) ***Arsenic***—To 0.5 g of Talc, add 5 mL of dilute sulfuric acid, shake well to mix, and heat gently to boiling. Cool quickly, filter, and wash first with 5 mL of dilute sulfuric acid and then with 10 mL of water. Combine the filtrate and the washings, and evaporate on a water bath to make 5 mL. Use this solution as the test solution and perform the test (NMT 4 ppm).

(9) ***Asbestos***—Perform the test according to Method (A) or (ii) as below; no asbestos is detected. If it is detected in (i) or (ii), continue performing the test according to Method (C); asbestos is not detected.

(i) Determine the infrared absorption spectrum of Talc as directed in the potassium bromide disk method under the Mid-infrared Spectroscopy; check the absorption at the wavenumbers of 757 cm^{-1} to 759 cm^{-1} (amphibole-based asbestos) or 600 cm^{-1} to 650 cm^{-1}(serpentine-based asbestos). If there is an absorption peak at a wave number of 757 cm^{-1} to 759 cm^{-1}, weigh a certain amount of the sample, ignite at 850 ℃ for NLT 30 minutes, and cool. Determine the infrared absorption spectrum again to check the absorption peaks at wave numbers of 757 cm^{-1} to 759 cm^{-1}, which indicates the presence of tremolite among amphibole-based asbestos.

(ii) Determine the powder diffraction of Talc as directed under the Characterization of crystalline and partially crystalline solids by X-ray powder diffraction according to the following operating conditions; check the diffraction peaks with a diffraction angle of 2θ at 10.4° to 10.6° (amphibole-based asbestos), 24.2° to 24.4° and 12.0° to 12.2° (serpentine-based chrysotile).

Operating conditions
X-ray light source: Cu K α monochromator
Tube current and voltage: 24 mA to 30 mA, 40 kV
Angle of incidence: 1°
Measuring angle: 0.2 °
Scanning speed: 0.1°/min
Scanning range (diffraction angle of 2θ): 10° to 13°, 24° to 26°

(iii) Observe the shape and color of asbestos with Talc using an optical microscope, and if the following characteristics are observed, it is assumed that asbestos has been detected.

① The ratio of the length and width of the fiber is within the range of 20 : 1 to 100 : 1, or if the fiber length is longer

than 5 μm, it is NLT 100 : 1.

② The fibers may split and become very thin.

③ They exhibit two or more of the four characteristics below.

㉠ The fiber bundles have parallel fibers inside them

㉡ The fiber bundles are worn or have frayed ends.

㉢ There are fine needle-shaped fibers

㉣ Each fiber is a tangled mass or has a curved shape.

Loss on ignition NMT 7.0% (1 g, 1050 to 1100 ℃, constant mass).

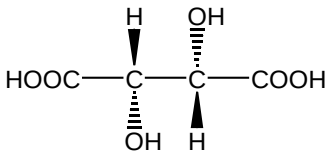
Microbial limit In case of the preparations for cutaneous use, the total aerobic microbial count is NMT 10^2 CFU per 1 g of Talc. In case of the preparations for oral use, the total aerobic microbial count is NMT 10^3 CFU and the total combined yeasts/moulds count is NMT 10^2 CFU per 1 g of Talc.

Assay Be careful when handling a mixture of perchloric acid and heavy metals as it is explosive.
Place 0.5 g of Talc in a polytetrafluoroethylene dish, add 5 mL of hydrochloric acid, 5 mL of nitric acid (lead-free) and 5 mL of perchloric acid, and stir slowly. Then add 35 mL of hydrofluoric acid, and slowly evaporate to dryness until the volume of the residue reaches about 0.5 mL. Add 5 mL of hydrochloric acid to the residue, cover with a watch glass, heat to dissolve, and then cool. Transfer this solution to a 50-mL volumetric flask, wash the polytetrafluoroethylene dish and watch glass with water, combine all the washings into the flask, and add water to make 50 mL. Take 0.5 mL of this solution and add water to make 100 mL. Take 4.0 mL of this solution, add 10 mL of hydrochloric acid and 10 mL of lanthanum chloride TS, then add water to make 100 mL, and use this solution as the test solution. Separately, take 8.3365 g of magnesium chloride, and dissolve in dilute hydrochloric acid to make 1000 mL. Take 5 mL of this solution, add water to make 500 mL, and use this solution as the magnesium standard stock solution. Each mL of this solution contains 10μg of magnesium. Place 2.5 mL, 3.0 mL, 4.0 mL and 5.0 mL of the magnesium standard stock solution in volumetric flasks, each containing 10 mL of hydrochloric acid and 10 mL of lanthanum chloride TS, add water to make 100 mL, and use these solutions as the standard solutions. Perform the test with the test solution and the standard solutions as directed in the calibration curve method under the Atomic absorption spectroscopy, and determine the content of magnesium in the test solution; it is 17.0% to 19.5%.

Gas: Acetylene - Air
Lamp: Magnesium hollow cathode lamp
Wavelength: 285.2 nm

Packaging and storage Preserve in well-closed containers.

Tartaric Acid
타르타르산



$C_4H_6O_6$: 150.09
(2*R*,3*R*)-2,3-Dihydroxybutanedioic acid [*87-69-4*]

Tartaric Acid, when dried, contains NLT 99.7% and NMT 101.0% of tartaric acid ($C_4H_6O_6$).

Description Tartaric Acid occurs as a colorless crystal or white crystalline powder. It is odorless and has a strong sour taste.
It is very soluble in water, freely soluble in ethanol, and slightly soluble in ether.
Dissolve 1 g of Tartaric Acid in 10 mL of water; the solution is dextrorotatory.

Identification (1) Heat Tartaric Acid slowly; it decomposes and produces an odor similar to burning sucrose.

(2) An aqueous solution of Tartaric Acid (1 in 10) changes blue litmus paper to red, and responds to the Chemical identification reactions for tartrate.

Purity (1) ***Sulfate*—**Perform the test with 0.5 g of Tartaric Acid. Prepare the control solution with 0.5 mL of 0.005 mol/L sulfuric acid (NMT 0.048%).

(2) ***Oxalate*—**Dissolve 1.0 g of Tartaric Acid in 10 mL of water, add 2 mL of calcium chloride TS; no turbidity is produced.

(3) ***Heavy metals*—**Proceed with 2.0 g of Tartaric Acid according to Method 4, and perform the test. Prepare the control solution with 2.0 mL of lead standard solution (NMT 10 ppm).

(4) ***Mercury*—**Spread about 1 g of excipient (a) evenly on a ceramic boat, and place 10 mg to 300 mg of Tartaric Acid on top. Then, evenly spread about 0.5 g of excipient (a) and 1 g of excipient (b) on top it in turn to form a layer. However, in the case of an automatic mercury analyzer equipped with a separate catalyst in the combustion chamber, place only the sample in a nickel boat without any excipients. Place the boat inside the combustion furnace, and heat to about 900°C with a current of air or oxygen at 0.5 L/min to 1 L/min. Elute the mercury and collect in a collection tube. Transfer the mercury vapor to a cold vapor atomic absorption spectrometer by heating the collecting tube to about 700 °C and determine the absorbance, A. Separately, place only the excipients in a ceramic boat and determine the absorbance, Ab in the same manner. Separately, proceed with mercury standard solution in the same manner and create a calibration curve from the absorbances. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the sample (NMT 1.0 ppm).

Operating conditions

Apparatus: Use an automated mercury analyzer for specimen combustion, trapping with gold amalgam and measurement using a cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Standard mercury stock solution—Dissolve 0.135 g of mercury(II) chloride in 0.001% L-cysteine solution to make 1000

mL. Each mL of this solution contains 100 μg of mercury(II) chloride.

Mercury standard solution—Dilute the standard mercury stock solution with 0.001% L-cysteine solution to make 0 to 200 ng/mL.

Excipients—Activate excipients at 950 ℃ for 30 minutes when using (a) aluminum oxide and (b) calcium hydroxide-sodium carbonate (1 : 1).

(5) ***Lead***—Weigh accurately 5.0 g of Tartaric Acid, transfer to a platinum crucible, dry, carbonize, and incinerate at 450 °C to 550 °C. If incineration is not complete, cool and moisten with 2 mL to 5 mL of nitric acid (1 in 2), 50% magnesium nitrate solution or aluminum nitrate-calcium nitrate solution (prepared by dissolving 40 g of aluminum nitrate and 20 g of calcium nitrate in 100 mL of water) as an incineration supplement, dry, and continue with incineration. If incineration is incomplete, repeat the above procedure once and then add a final 2 mL to 5 mL of nitric acid (1 in 2), if necessary, and incinerate. After incineration, moisten the residue with water, add 2 4 mL to 4 mL of hydrochloric acid, and evaporate to dryness. Add 0.5 mol/L nitric acid, dissolve by warming, and filter with filter paper if any insoluble matter exists. Unless otherwise specified, add 0.5 mol/L nitric acid to make 25 mL and use this solution as the test solution. Separately, proceed with 5.0 mL of cadmium standard solution in a platinum crucible in the same manner as the test solution, and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Atomic absorption spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 1.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: A lead hollow-cathode lamp
Wavelength: 283.3 nm

(6) ***Calcium***—Dissolve 1.0 g of Tartaric Acid in 10 mL of water and add ammonia TS to make it neutral. Add 1 mL of ammonium oxalate TS; no turbidity is produced.

(7) ***Arsenic***—Proceed with 2.0 g of Tartaric Acid according to Method 1 and perform the test (NMT 1 ppm).

Specific optical rotation $[\alpha]_D^{20}$: Between +12° and +13° (2 g, water, 10 mL, 100 mm).

Loss on drying NMT 0.5% (3 g, silica gel, 3 hours).

Residue on ignition NMT 0.05% (1 g).

Assay Weigh accurately about 1.5 g of Tartaric Acid, previously dried, dissolve in 40 mL of water, and titrate with 1 mol/L sodium hydroxide VS (indicator: 2 drops of phenolphthalein TS).

Each mL of 1 mol/L sodium hydroxide VS
= 75.04 mg of $C_4H_6O_6$

Packaging and storage Preserve in well-closed containers.

Titanium Oxide
산화티탄

TiO_2: 79.87

Titanium Oxide, when dried, contains NLT 98.5% and NMT 101.0% of titanium oxide (TiO_2).

Description Titanium Oxide occurs as a white powder and is odorless and tasteless.
It is practically insoluble in water, anhydrous ethanol or ether.
It dissolves in hot sulfuric acid or hydrofluoric acid and does not dissolve in hydrochloric acid, nitric acid or dilute sulfuric acid.
Add potassium hydrogen sulfate, potassium hydroxide or potassium carbonate to Titanium Oxide, and fuse by heating; it transforms into a soluble salt.
To 1 g of Titanium Oxide, add 10 mL of water, and shake; the solution is neutral.

Identification To 0.5 g of Titanium Oxide, add 5 mL of sulfuric acid, and heat until white fumes evolve. After cooling, dilute cautiously by adding water to make 100 mL and filter. To 5 mL of the filtrate, add 2 to 3 drops of hydrogen peroxide TS; the solution exhibits a yellowish red color.

Purity (1) ***Lead***—Take 1.0 g of Titanium Oxide in a platinum crucible, add 10.0 g of potassium hydrogen sulfate, heat gently with caution at first, and then strongly heat, occasionally shaking until the contents melt and become a clear liquid. After cooling, add 30 mL of ammonium citrate solution (9 in 20) and 50 mL of water, heat on a water bath to dissolve, cool, and add water to make 100 mL. Use this solution as the sample stock solution. Put 25 mL of the sample stock solution into a separatory funnel, add 10 mL of ammonium sulfate solution (2 in 5) and 5 drops of thymol blue TS, neutralize with ammonia TS, and add 2.5 mL of ammonia TS. Then add exactly 20 mL of dithizone in *n*-butyl acetate solution (1 in 500), shake to mix for 10 minutes, and use this *n*-butyl acetate solution as the test solution. Separately, take 1.0 mL of lead standard solution to a platinum crucible, proceed in the same way as in the test solution, and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Atomic Absorption Spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 10 ppm).

Gas: Acetylene or hydrogen – Air
Lamp: A lead hollow-cathode lamp
Wavelength: 283.3 nm

(2) ***Arsenic***—Perform the test using 20 mL of the sample stock solution from (1) as the test solution; the color of the solution is not more intense than the following standard color.

Standard color—Prepare the control solution in the same manner as in the test solution without using Titanium Oxide. Add 0.2 mL of arsenic standard solution and the control solution to make 20 mL, put in a colorimetric flask, and proceed in the same manner as in the test solution (NMT 1 ppm).

(3) ***Water-soluble substances***—To 4.0 g of Titanium Oxide, add 50 mL of water, shake well to mix, and allow to stand overnight. Add 2 mL of ammonium chloride TS, shake well to mix, and add again 2 mL of ammonium chloride TS, if necessary to precipitate titanium oxide. Then, add water to make 200 mL,

shake well to mix, and filter with double filter paper. Discard the first 10 mL of the filtrate, take 100 mL of the clear filtrate, evaporate on a water bath, and heat the residue strongly at 800 ℃ to a constant mass; the residue is NMT 5.0 mg.

(4) ***Antimony***—Perform the test with 25 mL of the sample stock solution of (1), and use this solution as the test solution. Separately, proceed in the same manner with 2 mL of antimony standard solution, and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Atomic Absorption Spectroscopy according to the following conditions; the absorbance of the test solution is NMT that of the standard solution (NMT 2 ppm).

Gas: Acetylene or hydrogen – Air
Lamp: An antimony hollow cathode lamp
Wavelength: 217.6 nm

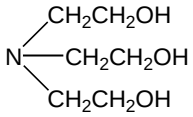
(5) ***Mercury***—Dissolve 2.0 g of Titanium Oxide in 1 mL of potassium permanganate solution (3 in 50) and 30 mL of water. Neutralize gradually with purified hydrochloric acid, add 5 mL of diluted sulfuric acid (1 in 2), and then add hydroxylamine hydrochloride solution (1 in 5) until the precipitate of manganese dioxide disappears. Then, add water to make exactly 100 mL, and use this solution as the test solution. Perform the test with the test solution as directed under the Atomic absorption spectroscopy (cold vapor method). Put the test solution in the test bottle of an atomic absorption spectrophotometer, add 10 mL of tin(II) chloride TS-sulfuric acid TS, connect the bottle immediately to the atomic absorption spectrophotometer, and circulate air. Read the absorbance A_T of the test solution when the reading of the a data collection device rises rapidly and becomes constant at the wavelength of 253.7 nm. Separately, weigh exactly 0.0135 g of mercury(II) chloride, previously dried for 6 hours in a desiccator (silica gel), dissolve in 10 mL of dilute nitric acid, and add water to make exactly 1000 mL. Pipet 10 mL of this solution, and add 1 mL of dilute nitric acid and water to make exactly 100 mL. Pipet 2.0 mL of this solution, and add 1 mL of potassium permanganate solution (3 in 50), 30 mL of water and the same amount of purified hydrochloric acid used in the preparation of the test solution. Proceed in the same manner as the test solution, and determine the absorbance, A_S, of this solution; A_T is NMT A_S (NMT 1 ppm).

Loss on drying NMT 0.5% (1 g, 105 ℃, 3 hours).

Assay Weigh accurately about 0.2 g of Titanium Oxide, previously dried, put in a crucible, add 3 g of potassium pyrosulfate, and stopper. Heat at low heat at first and gradually increase the temperature until the contents melt, continue to heat for 30 minutes, then again apply strong heat for another 30 minutes until the molten substance becomes a clear liquid with an intense yellowish red color. After cooling, transfer the contents from the crucible to a 250-mL beaker, wash the crucible with a mixture of 75 mL of water and 2.5 mL of sulfuric acid, add the washings, and heat on a water bath until the solution becomes almost clear. Add 2 g of L-tartaric acid to dissolve, add 2 to 3 drops of bromothymol blue TS, neutralize with ammonia TS, add 1 to 2 mL of diluted sulfuric acid (1 in 2) to make it acidic, and pass through hydrogen sulfide sufficiently. Add 30 mL of ammonia TS, pass through hydrogen sulfide to saturate, allow to stand for 10 minutes, and filter. Wash the residue on the filter paper 10 times with 25 mL each of L-ammonium tartrate solution (1 in 100) containing 2.5 mL of ammonium sulfide TS. Filter, and wash while keeping the filter paper filled with the solution to prevent the oxidation of iron sulfide. Combine the filtrate and washings, add 40 mL of diluted sulfuric acid (1 in 2), boil to remove hydrogen sulfide, and add water to make 400 mL after cooling. Add 40 mL of cupferron TS slowly while stirring to mix, allow to stand to form yellow precipitates, and then add cupferron TS again until white precipitates are formed. Filter the precipitates with under weak suction using a filter paper for assay, wash 20 times with diluted hydrochloric acid (1 in 10), and filter with strong suction to remove water. Dry the precipitates with the filter paper at 70 ℃, transfer into a crucible previously weighed and ignite initially very gently or strongly if no smoke is produced at 900 to 950 ℃ to a constant mass. After cooling, weigh the mass, and take it as the amount of titanium oxide (TiO_2).

Packaging and storage Preserve in well-closed containers.

Trolamine
트롤아민



Triethanolamine $C_6H_{15}NO_3$: 149.19
2,2′,2″-Nitrilotriethanol [*102-71-6*]

Trolamine largely consists of triethanolamine, usually containing diethanolamine and monoethanolamine.

Trolamine contains NLT 99.0% and NMT 107.4% of alkanolamines, calculated on the anhydrous basis as triethanolamine ($C_6H_{15}NO_3$: 149.19).

Description Trolamine occurs as colorless or pale yellow viscose liquid with a slight odor of ammonia.
It is miscible with water, ethanol or chloroform.

Identification (1) To 1 mL of Trolamine, add 0.1 mL of copper sulfate TS: a deep blue color develops. To this solution, add 5 mL of sodium hydroxide TS, evaporate by heating to 2 mL: the color of the solution does not change.

(2) To 1 mL of Trolamine, add 0.3 mL of cobalt chloride TS: a carmine-red color develops.

(3) Weigh 1 mL of Trolamine and heat gently; the evolved vapor turns moistened red litmus paper blue.

Refractive index n_D^{20}: Between 1.481 and 1.486

Specific gravity d_{20}^{20}: Between 1.120 and 1.128 (Method 1)

Purity ***Heavy metals***—Dissolve 12.0 g of Trolamine in water to make 20 mL. Pipet 5.0 mL of this solution, and add water to make 30 mL. Use this solution as the test solution and perform the test. To 3.0 mL of lead standard solution, add water to make 30 mL and use this solution as the control solution. To 10 mL of this solution, add 2 mL of the test solution. Separately, to 10 mL of water, add 2 mL of the test solution, and use this solution as the blank test solution. To 12 mL each of the test solution, the control solution, and the blank test solution, add 2 mL of pH 3.5 acetate buffer solution, mix, add 1.2 mL of thioacetamide TS, and mix immediately. Allow to stand for 2 minutes; the color of the test solution is not more intense than that of the control solution (NMT 10 ppm).

Water NMT 0.5% (1.0 g, volumetric titration, direct titration.

Use a mixture of 5.0 mL of acetic acid (100) and 20 mL of methanol instead of methanol for Karl Fischer titration).

Residue on ignition NMT 0.05% (2 g).

Assay Weigh accurately about 2 g of Trolamine, dissolve in 75 mL of water, and titrate with 1 mol/L hydrochloric acid VS (Indicator: 2 drops of methyl red TS).

Each mL of 1 mol/L hydrochloric acid VS
= 149.19 mg of $C_6H_{15}NO_3$

Packaging and storage Preserve in light-resistant, tight containers.

Turpentine Oil
테레빈유

Oleum Terebinthinae

Turpentine Oil is the essential oil distilled with steam from the wood or balsam of Pinus genus (Pinaceae).

Description Turpentine Oil occurs as clear, colorless to pale yellow liquid, and has a characteristic odor and pungent, bitter taste.
Turpentine Oil (1 mL) is miscible with 5 mL of ethanol and this solution is neutral.

Refractive index n_D^{20}: Between 1.465 and 1.478.

Specific gravity d_{20}^{20}: Between 0.860 and 0.875.

Purity (1) ***Foreign matter***—Turpentine Oil has no offensive odor. To 5 mL of Turpentine Oil, add 5 mL of a solution of potassium hydroxide (1 in 6), and shake to mix: the water layer does not show a yellowish brown to dark brown color.

(2) ***Hydrochloric acid-coloring substances***—To 5 mL of Turpentine Oil, add 5 mL of hydrochloric acid, shake to mix and allow to stand for 5 minutes: the hydrochloric acid layer shows a pale yellow color and not a brown color.

(3) ***Mineral oil***—Place 5 mL of Turpentine Oil in a cassia flask, cool to a temperature not higher than 15 °C, add slowly 25 mL of fuming sulfuric acid while shaking, warm at 60 °C to 65 °C for 10 minutes and add sulfuric acid to raise the lower level of the oily layer to the graduated volume of the neck: NMT 0.1 mL of oil separates.

(4) ***Peroxide value***—Weigh accurately 2 g of Turpentine Oil, transfer to a stoppered Erlenmeyer flask and dissolve in 50 mL of a mixture of trimethylpentane and acetic acid (100) (2 : 3). To this solution, add 0.5 mL of a saturated potassium iodide solution, stopper the flask, allow to stand for 1 minute, shake continuously and add 30 mL of water. Titrate this solution with 0.01 mol/L sodium thiosulfate VS. However, the endpoint of the titration is when the blue color of the solution disappears after addition of 0.5 mL of starch TS when the color of the solution turns to a pale yellow color. Perform a blank test and make any necessary correction (NMT 0.1 mL of 0.01 mol/L sodium thiosulfate VS is consumed by the blank test solution). The peroxide value calculated by the following equation is NMT 20.0.

$$\text{Peroxide value (mEq/kg)} = [10 \times (V_1 - V_0)]/W$$

V_1: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed in the titration of the test solution
V_0: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed in the titration of the blank test solution
W: Amount (g) of Turpentine Oil taken

Distilling range Between 150 °C and 170 °C, NLT 90 vol%.

Packaging and storage Preserve in light-resistant, tight containers.

Urea
우레아

H_2NCONH_2

CH_4N_2O: 60.06

Urea [*57-13-6*]

Urea contains NLT 99.0% and NMT 101.0% of urea (CH_4N_2O).

Description Urea occurs as a colorless or white crystal or crystalline powder, is odorless and has a fresh salty taste.
It is very soluble in water, freely soluble in boiling ethanol, soluble in ethanol, and very slightly soluble in ether.
An aqueous solution of Urea (1 in 100) is neutral.

Identification (1) Heat 0.5 g of Urea; it liquefies and an odor of ammonia is produced. Continue the heating until the liquid becomes turbid, then cool. Dissolve the resulting mass in a mixture of 10 mL of water and 2 mL of sodium hydroxide TS, and add 1 drop of copper sulfate TS; the resulting solution exhibits a reddish violet color.

(2) Dissolve 0.1 g of Urea in 1 mL of water and add 1 mL of nitric acid; a white crystalline precipitate is produced.

Melting point Between 132.5 ℃ and 134.5 ℃.

Purity (1) ***Chloride***—Perform the test with 2.0 g of Urea. Prepare the control solution by adding 0.40 mL of 0.01 mol/L hydrochloric acid (NMT 0.007%).

(2) ***Sulfate—***Perform the test with 2.0 g of Urea. Prepare the control solution by adding 0.40 mL of 0.005 mol/L sulfuric acid (NMT 0.010%).

(3) ***Heavy metals***—Weigh 1.0 g of Urea and perform the test according to Method 1. Prepare the control solution by adding 2.0 mL of the lead standard solution (NMT 20 ppm).

(4) ***Ethanol-insoluble matter***—Dissolve 5.0 g of Urea in 50 mL of warm ethanol, filter the solution through a glass filter, previously weighed, wash the residue with 20 mL of warm ethanol and dry at 105 °C for 1 hour: the amount of the residue is NMT 2.0 mg.

Residue on ignition NMT 0.1% (1 g).

Assay Weigh accurately about 0.2 g of Urea and dissolve in water to make exactly 200 mL. Pipet 5 mL of this solution into a Kjeldahl flask and perform the test as directed under the Nitrogen Determination (Semimicro-Kjeldahl Method).

Each mL of 0.005 mol/L sulfuric acid VS
= 0.30028 mg of CH_4N_2O

Packaging and storage Preserve in well-closed containers.

Water
상수

H_2O: 18.02

Water meets the quality standards for drinking water in the Management of Drinking Water Act. Water meets the requirements of the following tests in addition to the above standards, when produced using well or industrial water.

Purity (1) ***Ammonium***—Pipet 30 mL of Water and use this solution as the test solution. Prepare the control solution with 0.15 mL of ammonium standard solution, dilute with distilled water for ammonium assay to make 30 mL, and proceed in the same manner as the test solution (NMT 0.05 mg/L).

(2) ***Nitrogen from nitrites***—Put 50 mL of Water in a Nessler tube, add 0.3 g of Griess-Romijin's nitrous acid TS, dissolve by shaking, and allow to stand for 10 minutes; the solution does not exhibit a pale red color.

Water for Injection
주사용수

Water for Injection is prepared by distillation or ultrafiltration from a water obtained by applying appropriate pretreatments such ion exchange or reverse osmosis on Water, or from Purified Water. When preparing Water for Injection by ultrafiltration (a method of purifying water using a reverse osmosis membrane, an ultrafiltration membrane capable of removing substances with a molecular weight of about NLT 6000, or a manufacturing system combining these membranes), care must be taken to avoid microbial contamination of the manufacturing system, and to provide water with equivalent quality to that prepared by distillation. Water for Injection should be used quickly after preparation. However, if there is a system to suppress the growth of microorganisms, such as high-temperature circulation, it can be temporarily preserved.

Description Water for Injection occurs as a clear, colorless liquid, and is odorless.

Purity ***Total organic carbon***—NMT 0.50 mg/L.

Bacterial endotoxins Less than 0.25 EU/mL

Conductivity Perform the test as follows; the conductivity of Water for Injection at 25 °C is NMT 2.1 μS/cm. Transfer an appropriate amount of Water for Injection into a beaker, and shake to mix. Adjust the temperature to 25 ± 1 °C, shake vigorously to mix, and determine the conductivity of this solution at regular intervals. When the change in conductivity becomes NMT 0.1 μS/cm per 5 minutes, adopt the observed value as the conductivity (25 °C) of Water for Injection.

Sterile Water for Injection
멸균주사용수

Sterile Water for Injection is prepared by introducing Water for Injection into a hermetic container and sterilizing, or by introducing pre-sterilized Water for Injection into a sterile container through an aseptic processing and sealing up the container. However, when preparing Sterile Water for Injection using Water for injection made by distillation, it may be labeled with distilled water for injection as a nickname. Also, products prepared by distillation and sterilized in a container can be labeled as distilled water for injection as a nickname.

Description Sterile Water for Injection occurs as a colorless, transparent liquid and is odorless.

Purity ***Potassium permanganate reducing substances***—To 100 mL of Sterile Water for Injection, add 10 mL of dilute sulfuric acid, boil, add 0.10 mL of 0.02 mol/L potassium permanganate solution, and boil again for 10 minutes; the red color of the solution does not disappear.

Sterility Meets the requirements.

Conductivity Perform the test as follows; the conductivity at 25 °C of Sterile Water for Injection is NMT 25 μS/cm for containers with a volume of 10 mL or less, and NMT 5 μS/cm for containers with a volume greater than 10 mL.
Transfer an appropriate amount of Sterile Water for Injection into a beaker, and shake to mix. Adjust the temperature to 25 ± 1℃, shake vigorously to mix, and determine the conductivity of this solution at regular intervals. When the change in conductivity becomes NMT 0.1 μS/cm per 5 minutes, adopt the observed value as the conductivity (25 °C) of Sterile Water for Injection.

Bacterial endotoxins Less than 0.25 EU/mL

Particulate contamination: Visible particles Meets the requirements.

Extractable volume of injections Meets the requirements.

Insoluble particulate matter in injections Meets the requirements.

Packaging and storage Preserve in hermetic containers. However, plastic containers for aqueous infusions may be used.

Purified Water in Bulk
정제수

Purified Water in Bulk is prepared with Water by ion exchange, distillation, reverse osmosis or ultrafiltration, or by a combination of these processes. Purified Water in Bulk should be used quickly after preparation. However, when the growth of microorganisms is suppressed, it can be temporarily preserved.

Description Purified Water in Bulk occurs as a colorless, transparent liquid, and is odorless.

Purity ***Total organic carbon***—It is NMT 0.50 mg/L.

Conductivity Perform the test as follows; the conductivity of Purified Water in Bulk at 25 °C is NMT 2.1 μS/cm. Transfer an appropriate amount of Purified Water in Bulk into a beaker, and shake to mix. Adjust the temperature to 25 ± 1℃, shake vigorously to mix, and determine the conductivity of this solution at

regular intervals. When the change in conductivity becomes NMT 0.1 μS/cm per 5 minutes, adopt the observed value as the conductivity is considered the conductivity (25 °C) of Purified Water in Bulk.

Purified Water in Containers
정제수 (기밀용기 내)

Purified Water in Containers is prepared from Purified Water in Bulk by introducing it into tight containers.

Description Purified Water in Containers occurs as a colorless, transparent liquid, and is odorless.

Purity ***Potassium permanganate reducing substances***—To 100 mL of Purified Water in Containers, add 10 mL of dilute sulfuric acid, boil, add 0.10 mL of 0.02 mol/L potassium permanganate solution, and boil again for 10 minutes; the red color of the solution does not disappear.

Conductivity Perform the test as follows; the conductivity at 25 °C of Purified Water in Containers is NMT 25 μS/cm for containers with a volume of 10 mL or less, and NMT 5 μS/cm for containers with a volume greater than 10 mL.
Transfer an appropriate amount of Purified Water in Containers into a beaker, and shake to mix. Adjust the temperature to 25 ± 1℃, shake vigorously to mix, and determine the conductivity of this solution at regular intervals. When the change in conductivity becomes NMT 0.1 μS/cm per 5 minutes, adopt the observed value as the conductivity (25 °C) of Purified Water in Containers.

Microbial limit The acceptance criteria of the total aerobic microbial count is NMT 1000 CFU per 1 mL of Purified Water in Containers. However, perform the test using soybean-casein digest agar medium.

Packaging and storage Preserve in tight containers.

Sterile Purified Water
멸균정제수

Sterile Purified Water is prepared by introducing Purified Water in Bulk into a hermetic container and sterilizing, or by introducing previously sterilized Purified Water in Bulk into a sterile container through an aseptic processing and sealing up the container.

Description Sterile Purified Water occurs as a colorless, transparent liquid and is odorless.

Purity ***Potassium permanganate reducing substances***—To 100 mL of Sterile Purified Water, add 10 mL of dilute sulfuric acid, boil, add 0.10 mL of 0.02 mol/L potassium permanganate solution, and boil again for 10 minutes; the red color of the solution does not disappear.

Conductivity Perform the test as follows; For products with capacity of NMT 10 mL, the conductivity at 25 °C is NMT 25 μS/cm, and those with capacity of NLT 10 mL, it is NMT 5 μS/cm. Transfer an appropriate amount of Sterile Purified Water into a beaker, and shake to mix. Adjust the temperature to 25 ± 1 ℃, shake vigorously to mix, and determine the conductivity of this solution at regular intervals. When the change in conductivity becomes NMT 0.1 μS/cm per 5 minutes, adopt the observed value as the conductivity (25 °C) of Sterile Purified Water.

Sterility Meets the requirements.

Packaging and storage Preserve in hermetic containers. Plastic containers for aqueous injections may be used.

Wheat Starch
밀전분

Wheat Starch is a starch obtained from the grains of *Triticum aestivum* Linné (*Gramineae*).

Description Wheat Starch occurs as a white or almost white fine powder. It has a crispy sound when pressed with a finger.
It is practically insoluble in cold water and ethanol (95).
Wheat Starch does not include starch from different origins. Occasionally, it may contain small fragments of the original plant tissue.

Identification (1) To Wheat Starch, add a mixture of water and glycerin (1 : 1), and examine under a microscope; it is a large and small grain or, very rarely, medium-sized grain. The grains, usually 10 to 60 μm in diameter, are discoid or, more rarely, reniform when seen face-on. The central hilum and striations are invisible or barely visible and the granules sometimes show cracks on the edges. Seen in profile, the grains are elliptical or fusiform and the hilum appears as a slit along the main axis. The small grains, 2 to 10 μm in diameter, are rounded or polyhedral. Between crossed polarizing prisms, Wheat Starch shows a distinct black cross intersecting at the hilum.

(2) To 1 g of Wheat Starch, add 50 mL of water, boil for 1 minute, and cool; a pale, white-turbid and viscous liquid is formed.

(3) To 1 mL of the pasty Wheat Starch obtained from (2), add 0.05 mL of dilute iodine TS; the resulting solution exhibits a dark bluish purple color, and the color disappears when heated.

pH Transfer 5.0 g of Wheat Starch into a non-metallic container, add 25 mL of freshly boiled and cooled water, shake gently for 1 minute to make the suspension, and allow to stand for 15 minutes; the pH is 4.5 to 7.0.

Purity (1) ***Iron***—To 1.5 g of Wheat Starch, add 15 mL of 2 mol/L hydrochloric acid TS, shake to mix, filter, and use this solution as the test solution. Prepare the control solution by adding water to 2.0 mL of iron standard solution to make 20 mL. Transfer 10 mL each of the test solution and the control solution into test tubes, add 2 mL of citric acid (1 in 5) and 0.1 mL of mercaptoacetic acid, and mix. To this solution, add strong ammonia water until the litmus paper clearly exhibits alkaline, then add water to make 20 mL, and mix. Transfer 10 mL each of these solutions into two test tubes, allow to stand for 5 minutes, and compare the color of the solution against a white background; the color of the test solution is not more intense than that of the control solution (NMT 10 ppm).

(2) ***Oxidizing substances***—To 4.0 g of Wheat Starch, add 50.0 mL of water, shake for 5 minutes, and then centrifuge. To 30.0 mL of the clear supernatant, add 1 mL of acetic acid (100) and 0.5 g to 1.0 g of potassium iodide, shake to mix, and allow to

stand in a dark place for 25 to 30 minutes. Add 1 mL of starch TS, and titrate with 0.002 mol/L sodium thiosulfate VS until the solution becomes colorless. Perform a blank test in the same manner and make any necessary correction. The consumed amount of 0.002 mol/L sodium thiosulfate VS is NMT 1.4 mL (20 ppm, calculated as hydrogen peroxide).

(3) ***Sulfur dioxide***—NMT 50 ppm.

(4) ***Foreign matter***—Examine Wheat Starch under a microscope; no other starches are detected. Also, it contains none or very minimal tissue fragments from the source plant.

(5) ***Total protein***—Weigh accurately 3 g of the sample containing 2 mg of nitrogen, transfer into a Kjeldahl flask, add 4 g of a mixture of 100 g of potassium sulfate, 5 g of copper(II) sulfate pentahydrate, and 3 g of titanium dioxide (IV), previously powdered, and add 3 glass beads. Carefully add 5 mL of sulfuric acid along the inside wall of the flask, wash the sample adhering to the flask neck, and mix it by rotating. Close the mouth of the flask, heat gradually at first, and then increase the temperature until boiling. At this point, take care to prevent the upper part of the flask from being overheated, and heat until the carbonaceous material on the inner wall of the flask disappears and the solution becomes clear. After cooling, carefully dissolve in 25 mL of water, then cool again, and place it in a steam distillation apparatus. Add 30 mL of sodium hydroxide solution (21 in 50), pass the steam through the mixture, and immediately distill. Collect about 40 mL of distillate in 25 mL of 0.01 mol/L hydrochloric acid and sufficient water to cover the tip of the condenser. Towards the end of the distillation, lower the receiver so that the tip of the condenser is placed above the surface of the acid. Take care to prevent any water on the outer surface of the condenser from reaching the contents of the receiver. Titrate the excess hydrochloric acid with 0.025 mol/L sodium hydroxide VS until the color of the solution changes from reddish purple through grayish blue and then to green (indicator: 3 drops of methyl red-methylene blue TS) (b mL). Perform a blank test in the same manner (a mL). An added amount of sodium hydroxide solution (21 in 50) through a funnel is sufficient to change the color of the solution in the flask from bluish green to dark brown or black. Calculate the amount of nitrogen according to the following formula and multiply by the conversion factor 6.25 to determine the total protein; the total protein is NMT 0.3% (equivalent to 0.048% of nitrogen).

$$\text{Amount (\%) of nitrogen} = (a - b) \times 0.035 / W$$

W: Amount (g) of sample taken

a: Volume (mL) of 0.025 mol/L sodium hydroxide VS consumed in the blank test

b: Volume (mL) of 0.025 mol/L sodium hydroxide VS consumed in the sample test

Loss on drying NMT 15.0% (1 g, 130 ℃, 90 minutes).

Residue on ignition NMT 0.6% (1 g).

Microbial limit Perform the microbial limit test; the total aerobic microbial counts is NMT 10^3 CFU, and the total combined yeasts/mould count is NMT 10^2 CFU per 1 g of Wheat Starch. No escherichia coli (E.coli) or salmonella is detected.

Packaging and storage Preserve in well-closed containers.

6) Quasi Drugs

Absorbent Cotton
탈지면

Absorbent Cotton is made by defatting and bleaching the hair of the seed of *Gossypium herbaceum* Linné or that of other species in the same genus (Malvaceae).

Description Absorbent Cotton occurs as white, soft, fine and fibrous hair, odorless and tasteless.
Under a microscope, Absorbent Cotton is flat, lined, twisted and hollow ribbon-shaped, with slightly thick edges.
It is soluble in ammonia copper TS, but is insoluble in ordinary solvents.

Purity Take a certain amount of samples of Absorbent Cotton from 10 different positions in the same package and combine them to make the specified amount.

(1) ***Acid or alkali***—Macerate 10 g of Absorbent Cotton in 100 mL of freshly boiled and cooled water. To 25 mL of the extract, add 3 drops of phenolphthalein TS; the resulting solution does not exhibit a red color. Likewise, to 25 mL of the extract, add 1 drops of methyl orange TS; the resulting solution does not exhibit a red color.

(2) ***Water-soluble substances*—**Place 5 g of Absorbent Cotton in 500 mL of water and boil for 15 minutes, while adding water to maintain the original volume. Pour the extract through a funnel into another container, transfer the Absorbent Cotton to the funnel, press out the water absorbed therein with a glass rod, and wash the Absorbent Cotton twice each time with 150 mL of boiling water. Press out the extract from the Absorbent Cotton after each washing and combine the extracts and the solutions used for washing, and filter. Evaporate the filtrate to concentrate, transfer the concentrated solution in a weighing bottle, and dry at 105 °C to a constant mass; the amount of residue is NMT 14.0 mg. Perform a blank test in the same manner and make any necessary correction.

(3) ***Dyes*—**Macerate 10 g of Absorbent Cotton in 100 mL of ethanol, press out the extract, transfer 50 mL of the extract to a Nessler tube, and observe from above; the resulting solution may exhibit a yellow color, but never a blue or green color.

(4) ***Fluorescent whitening agents*—**Irradiate Absorbent Cotton with ultraviolet light in the dark; it does not emit dye-related fluorescence from any of the surface.

(5) ***Sedimentation rate*—**Prepare a test basket, 3.0 g in mass, 50 mm in diameter, 80 mm in depth and 20 mm between the wires, which is made of copper wire with a diameter of about 0.4 mm. Place 5 g of Absorbent Cotton fittingly in the basket, hold the basket on its side, 12 mm above the surface of water at a temperature between 24 °C and 26 °C, and drop the basket gently into the water, which is 200 mm deep; the basket sinks to the bottom of the water in less than 8 seconds.

(6) ***Absorbency*—**Leave the submerged basket in (5) at the bottom of the water without disturbance for 3 minutes. Take out the basket gently from the water, keeping it horizontally, and allow to drain for 1 minute on the metal mesh of a sieve No.10 in the same horizontal position. Then, transfer the Absorbent Cotton into a beaker and weigh; the mass of water absorbed is NLT 100.0 g.

(7) ***Other fiber filaments*—**Immerse 1.0 g of Absorbent Cotton in 0.5 mol/L iodine TS for 1 minute and wash thoroughly with water; no dyed fiber filament is observed.

(8) ***Neps and adhering impurities*—**Spread evenly about 1 g of Absorbent Cotton between two colorless, transparent plates, 10 cm × 10 cm in size, and examine neps and adhering impurities (fragments of rinds and seeds) through transmitted light; the total number of neps and adhering impurities more than 2.5 mm in diameter is NMT 5.

Ash NMT 0.25% (5 g, proceed as directed in the Ash under the Crude Drugs Test).

Packaging and storage Preserve in well-closed containers.

Sterile Absorbent Cotton
멸균탈지면

Sterile Absorbent Cotton is the sterilized absorbent cotton.

Description Sterile Absorbent Cotton occurs as white, soft, fine and fibrous hair, odorless and tasteless.
Under a microscope, Sterile Absorbent Cotton is flat, lined, twisted and hollow ribbon-shaped, with slightly thick edges.
It is soluble in ammonia copper TS, but is insoluble in ordinary solvents.

Purity Proceed as directed in the Purity under Absorbent Cotton.

Ash NMT 0.25% (5 g, proceed as directed in the Ash under the Crude Drugs Test).

Sterility Proceed with about 0.5 g of Sterile Absorbent Cotton (use the whole amount if the total amount of the sample is less than 0.5 g) as directed in the Sterility under Sterile Absorbent Gauze.

Packaging and storage Preserve in tight containers, impervious to any microbe

Purified Absorbent Cotton
정제탈지면

Purified Absorbent Cotton is the hair of the seed of *Gossypium herbaceum* Linné, or that of other species in the same genus (Malvaceae), carefully selected, free from adhering impurities, defatted, bleached, and then purified.

Description Purified Absorbent Cotton occurs as white, soft, fine and fibrous hair, odorless and tasteless.
Under a microscope, Purified Absorbent Cotton is flat, lined, twisted and hollow ribbon-shaped, with slightly thick edges.
It is soluble in ammonia copper TS, but is insoluble in ordinary solvents.

Purity (1) ***Acid or alkali, Water-soluble substances, Dyes, Fluorescent whitening agents, Sedimentation rate, Absorbency, Other fiber filaments, Neps and Adhering impurities***—Proceed as directed in (1), (2), (3), (4), (5), (6), (7) and (8) of the Purity under Absorbent Cotton.

(2) ***Short fibers***—Take 0.10 g of Purified Absorbent Cotton, divide the fibers into two groups, one consisting of fibers exceeding 6.0 mm in length and the other consisting of fibers NMT 6.0 mm in length, weigh both groups, and determine the percentage (%) of those NMT 6.0 mm in length (short fibers); the amount of short fibers is NMT 10%.

$$\text{Amount (\%) of short fibers} = \frac{W_2}{W_1+W_2} \times 100$$

W_1: Mass of the group of fibers exceeding 6.0 mm in length.

W_2: Mass of the group of fibers NMT 6.0 mm in length.

Ash NMT 0.25% (5 g, proceed as directed in the Ash under the Crude Drugs Test).

Packaging and storage Preserve in well-closed containers.

Sterile Purified Absorbent Cotton
멸균 정제탈지면

Sterile Purified Absorbent Cotton is the sterilized purified absorbent cotton.

Description Sterile Purified Absorbent Cotton occurs as white, soft, fine and fibrous hair, odorless and tasteless.
Under a microscope, Sterile Purified Absorbent Cotton is flat, lined, twisted and hollow ribbon-shaped, with slightly thick edges.
It is soluble in ammonia copper TS, but is insoluble in ordinary solvents.

Purity Proceed as directed in the Purity under Purified Absorbent Cotton.

Ash NMT 0.25% (5 g, proceed as directed in the Ash under the Crude Drugs Test).

Sterility Proceed with about 0.5 g of Sterile Purified Absorbent Cotton (use the whole amount if the total amount of the sample is less than 0.5 g) as directed in the Sterility under Sterile Absorbent Gauze.

Packaging and storage Preserve in tight containers, impervious to any microbe

Absorbent Gauze
거즈

Absorbent Gauze is made by defatting and bleaching the raw fabric woven with pure cotton yarn obtained from the seeds of *Gossypium hirsutum* Linné or other plants of the same genus (*Malvaceae*).

The labeling of contents for Absorbent Gauze includes the thread number, the length and the width. For those folded twice or more to be used as it is for special purposes, the length, width and number of layers in the folded state are indicated in the label.

Description Absorbent Gauze occurs as white cotton cloth, odorless and tasteless.

Purity (1) ***Water-soluble substances***—Place 20 g of Absorbent Gauze in 500 mL of water and boil for 15 minutes, while adding water to maintain the original volume. Pour the extract through a funnel into a 1000-mL flask, transfer the Absorbent Gauze to the funnel, press out the water absorbed therein with a glass rod, and wash the Absorbent Gauze twice each time with 250 mL of boiling water. Press out the extract from the gauze after each washing and combine the extracts and the solutions used for washing, filter and add water to make 1000 mL. Transfer 400 mL of the filtrate into a beaker, evaporate to concentrate, and transfer the concentrated solution into a weighing bottle. Rinse the beaker with a small amount of water, combine the water used for washing with the concentrated solution in the weighing bottle, and dry at 105 °C to a constant mass; the amount of residue is NMT 20.0 mg. Perform a blank test in the same manner and make any necessary correction.

(2) ***Acid or alkali***—Take 200 mL of the extract obtained in (1) and add 5 drops of phenolphthalein TS; the resulting solution does not exhibit a red color. Likewise, to 200 mL of the extract, add 2 drops of methyl orange TS; the resulting solution does not exhibit a red color.

(3) ***Dextrin or starch***—To 200 mL of the extract obtained in (1), add 2 drops of iodine TS; the resulting solution does not exhibit a reddish purple to blue color.

(4) ***Dyes***—Macerate 10 g of Absorbent Gauze in 80 mL of ethanol, press out the extract, transfer 50 mL of the extract to a Nessler tube, and observe from above; the resulting solution may exhibit a yellow, but never a blue or green color.

(5) ***Fluorescent whitening agents***—Irradiate Absorbent Gauze with ultraviolet light in the dark; it does not emit dye-related fluorescence from any of the surface.

(6) ***Sedimentation rate***—Prepare a test basket, 3.0 g in mass, 50 mm in diameter, 80 mm in depth and 20 mm between the wires, which is made of copper wire with a diameter of about 0.4 mm (No. 26 wire). Place 5 g of Absorbent Gauze evenly in the basket, hold the basket on its side, 12 mm above the surface of water at a temperature between 24 °C and 26 °C, and drop the basket gently into the water, which is 200 mm deep; the basket sinks to the bottom of the water in less than 8 seconds.

(7) ***Other fiber filaments***—Immerse 1.0 g of Absorbent Gauze in 0.5 mol/L iodine TS for 1 minute, and then wash thoroughly with water; no dyed fiber filament is observed.

Physical shape The requirements of Absorbent Gauze for physical shape are as follows.

(1) ***Number of threads***—Prepare a frame with an empty space of 2.54 cm × 2.54 cm and place Absorbent Gauze over the frame. Including all threads at the edge of the frame, count the number of threads separately for warp, weft and those within 6.45 cm^2, take the integer digits of each count, and calculate the average from NLT 3 counts. However, closely woven parts are not counted.

(2) ***Mass***—Weigh a piece of Absorbent Gauze with the length of 1 meter and the width labeled and convert the mass in gram (g), equivalent to 1 meter. For those with closely woven parts at both ends lengthwise or widthwise, measure the full length or the full width as it is, while for those without closely woven parts at both ends lengthwise or widthwise, measure the length or the width of the net part alone. Also, fold Absorbent Gauze into about a size of 10 cm^2, place it in a desiccator, previously saturated with the vapor of a saturated solution of sodium nitrate, allow to stand for 4 hours at ordinary temperature, and weigh.

(3) ***Length***—Place Absorbent Gauze on a flat surface,

eliminate unnatural creases or tensions, and measure the full length at the center line. For those with closely woven parts at both ends lengthwise or widthwise, measure the full length or the full width as it is, while for those without closely woven parts at both ends lengthwise or widthwise, measure the length or the width of the net part alone. For those folded twice or more to be used as it is for special purposes, measure the length in the folded state.

(4) ***Width***—Place Absorbent Gauze on a flat surface, eliminate unnatural creases or tensions, measure the full width at more than 3 different positions, and calculate the average value. For those with closely woven parts at both ends lengthwise or widthwise, measure the full length or the full width as it is, while for those without closely woven parts at both ends lengthwise or widthwise, measure the length or the width of the net part alone. For those folded twice or more to be used as it is for special purposes, measure the width in the folded state.

(5) ***Fold***—For those folded twice or more to be used as it is for special purposes, count the number of folds.

Ash NMT 0.25% (5 g, proceed as directed in the Ash under the Crude Drugs Test).

Packaging and storage Preserve in well-closed containers.

Thread number	Number of threads between the 2.54-cm interval		Number of threads per 6.45 cm^2	Mass		Length	Allowable range of width	For those folded twice or more	
	Warp	Weft		g/m^2	Allowable range			Length or width	Number of folds
1	* 41 - 47	33 - 39	75 - 85	49.8	± 12% for those NMT 90 cm in width ± 8% for those exceeding 90 cm in width	NLT 98%	NLT -1.6 mm of the labeled width	NLT 98%	NLT the labeled number of folds
	41 - 47	33 - 39	76 - 84	49.8		NLT 95%	NLT -20% of the labeled width for those NMT 5 cm in width NLT -1.0 cm of the labeled width for those exceeding 5 cm and NMT 30 cm in width NLT -1.5 cm of the labeled width for those exceeding 30 cm in width		
2	30 - 34	26 - 30	57 - 63	37.4					
3	26 - 30	22 - 26	49-55	32.3					
4	22 - 26	18 - 22	41 - 47	27.8					
5	20 - 24	16-20	37-43	25.7					
6	18 - 22	14 - 18	33 - 39	22.5					
7	18 - 22	10 - 14	29-35	20.6					
8	12 - 16	8 - 12	21 - 27	13.8	± 12%				

Sterile Absorbent Gauze
멸균거즈

Sterile Absorbent Gauze is the sterilized absorbent gauze.

Description Sterile Absorbent Gauze occurs as white cotton cloth, odorless and tasteless.

Purity Proceed as directed in the Purity under Absorbent Gauze.

Physical shape Proceed as directed in the Physical shape under Absorbent Gauze.

Ash NMT 0.25% (5 g, proceed as directed in the Ash under the Crude Drugs Test).

Sterility Take Sterile Absorbent Gauze from a package aseptically under an aseptic environment, take out 1.0 g (take out the whole content, if the total amount is less than 1 g) total of Sterile Absorbent Gauze evenly from 5 different positions around the center region, and transfer each sample into test tubes of 25 mm × 200 mm, containing 60 mL each of the thioglycollate medium I for sterility test and the glucose-peptone medium for sterility test. Then, immerse the samples in the medium using an appropriate apparatus and proceed with the bacterial test and the fungal test as directed under the Sterility; it meets the requirements. However, in the test for the growth of fungi, a 200-mL Erlenmeyer flask can be used. When performing a performance test for a medium under a condition in the absence of the sample, the substantial growth of the inoculated microorganisms can be recognized.

The number of Sterile Absorbent Gauze samples used in the Sterility is indicated in the following table.

The number of the same products sterilized at the same time	The number of products used for the test
Less than 100	4
NLT 100 and less than 500	10
NLT 500	20

Packaging and storage Preserve in tight containers, impervious to any microbe

Adhesive Plaster
반창고

Method of preparation Adhesive Plaster is made by evenly spreading an adhesive mixture of carefully selected rubber, resins, zinc oxide and other substances on a cotton fabric.

Description The adhesive surface of Adhesive Plaster occurs as milky white and adheres well to the skin.

Purity Adhesive substance—The adhesive substance contained in Adhesive Plaster does not significantly ooze through the cotton fabric. When unrolled, not so significant amount of the adhesive substance is transferred to the outer surface of the next layer of the fabric. When removed from the skin, no large amount of the

adhesive substance remains on the skin.

Physical shape Adhesive Plaster is usually rectangular. and the length is NLT 98% of the labeled length. Measure the width of Adhesive Plaster at 5 different positions at appropriate intervals; the average of the 5 measurements is NLT 94% of the labeled width.

Tensile strength Cut Adhesive Plaster to a strip parallel with the warp, 12 mm in standard width and about 200 mm in length, allow it to stand in a desiccator, previously saturated with the vapor of a saturated solution of sodium nitrite, at ordinary temperature for 4 hours. Then, set the gauge distance of the tensile tester to 150 mm, fix the sample with a clamp, 25 to 50 mm in width, pull the sample at a speed of 300 mm for 1 minute, and measure the maximum load at the time it breaks; the average of 10 measurements is NLT 7.5 kg. For those with a width narrower than the standard width, calculate with the necessary correction to convert the width to the standard width.

Adhesive strength Cut Adhesive Plaster to a strip parallel with the warp, 12 mm in standard width and about 250 mm in length, and attach quickly one end of the strip, 12 mm in width and 125 mm in length, to a test plate made of phenol resin, about 25 mm in width, 125 mm in length and 5 mm in thickness, previously kept in an incubator at a constant temperature of 37 °C for 30 minutes. Then, promptly pass a rubber roller, 850 g in mass, twice over the Adhesive Plaster at a rate of 300 mm per minute. Leave the Adhesive Plaster in an incubator at a constant temperature of 37 °C for 30 minutes, fold back the free edge of the strip attached to the test plate at an angle of 180°, and peel about 25 mm from the front edge of the test plate. Next, fix firmly the free edge of the Adhesive Plaster with the upper clamp of the tensile tester and the test plate with the lower clamp, then pull at a rate of 300 mm per minute, and measure the load 4 times at an interval of about 20 mm; the average of the load is NLT 150 g. For those with a width narrower than the standard width, calculate with the necessary correction to convert the width to the standard width.

Packaging and storage Preserve in light-resistant, well-closed containers.

Bandage
붕대

Bandage

Bandage is made of Number 1, 2 or 3 absorbent gauze.

The length and width of Bandage are displayed on the package.

Description Bandage is in various lengths and widths. The number of threads between the 2.54-cm interval and the number of threads and mass per 6.45 cm^2 meet the requirements of Absorbent Gauze. Bandage is cut according to the labeled length and width.

Purity Proceed as directed in the Purity under Absorbent Gauze.

Physical shape The length is NLT 98% of the labeled length and the average of the widths measured at 5 positions at appropriate intervals is NMT 1.6 mm narrower than the labeled width.

Packaging and storage Preserve in well-closed containers (by each cut)

Calamine Lotion
칼라민 로션

Method of preparation

Calamine	80 g
Zinc oxide	80 g
Glycerin	20 mL
Bentonite magma	250 mL
Calcium hydroxide solution	A suitable amount
	To make 1000 mL

Dilute bentonite magma with an equal volume of a solution of calcium hydroxide. To prepare a solution of calcium hydroxide, add 1000 mL of cold purified water to 3 g of Calcium Hydroxide, mix with occasional shaking for 1 hour, allow to stand, and use the clear supernatant. Thoroughly and homogeneously mix Calamine and Zinc Oxide with Glycerin and about 100 mL of the diluted bentonite magma until a smooth, uniform paste is formed. Then, slowly add the remainder of the diluted bentonite magma little by little to soften the paste. Lastly, add a suitable amount of a solution of calcium hydroxide to make 1000 mL and mix well. If a lotion with higher viscosity is desired, increase the amount of bentonite magma up to 400 mL.

Microbial limit *Pseudomonas* and *Staphylococcus*—bacteria are not detected in the test.

Packaging and storage Preserve in tight containers.

Cresol
크레솔

C_7H_8O : 108.14

Cresol is a mixture of isomeric cresols

Description Cresol occurs as a clear, colorless or yellow to yellowish brown liquid and has a phenol-like odor.
It is miscible with ethanol or with ether.
It is sparingly soluble in water.
It is soluble in sodium hydroxide TS.
A saturated solution of Cresol shows a neutral reaction to bromocresol purple TS.
It strongly refracts light.
It turns dark brown when exposed to light or left for a long time.

Identification To 5 mL of a saturated solution of Cresol, add 1 to 2 drops of dilute ferric chloride TS; the resulting solution exhibits a bluish purple color.

Specific gravity d_{20}^{20}: Between 1.032 and 1.041

Purity (1) ***Hydrocarbons***—Dissolve 1.0 mL of Cresol in 60 mL of water; the resulting solution is no more turbid than the following reference solution.

Reference solution—To 54 mL of water, add 6.0 mL of 0.005 mol/L sulfuric acid and 1.0 mL of barium chloride TS,

shake well to mix, and allow to stand for 5 minute.

(2) ***Sulfur compounds***—Transfer 20 mL of Cresol in a 100-mL Erlenmeyer flask, place a piece of moistened lead acetate paper on the mouth of the flask, and warm for 5 minutes on a water bath; the lead acetate paper may exhibit a yellow color, but never a brown or dark tint color.

(3) ***Phenol***—Weigh accurately about 2.5 g of Cresol, add 10 mL of a solution of sodium hydroxide (1 in 10), and add water to make 250 mL. To 5 mL of this solution, add 45 mL of water and 1 drop of methyl orange TS, add 20 vol% nitric acid dropwise to neutralize, add water to make 200 mL, and use this solution as the test solution. Separately, to about 1 g of phenol RS, add water to make 100 mL and use this solution as the standard solution. Put 5 mL each of the test solution into two test tubes with a 25-mL graduation line and put 5 mL each of the standard solution into another two test tubes with a 25-mL graduation line. To each test tube, add 5 mL of Millon TS, allowing it to flow down along the inner wall of the test tube, mix, and heat in a boiling water bath for 30 minutes. After cooling in a cold water bath for NLT 10 minutes, add 5 mL of 20 vol% nitric acid to each test tube, and mix. Add 3 mL of formaldehyde solution (1 in 51) to only one of each pair of the test tubes, add water to all test tubes to make 25 mL, and allow to stand for 16 hours; test tubes with formaldehyde exhibits a yellow color and the test tubes without formaldehyde exhibits an orange to red color. Pipet 20 mL from each of the two test tubes containing the standard solution, add 5 mL of 20 vol% nitric acid, and add water to make 100 mL. Transfer the solutions to burets each marked as B_1 and B_2, indicating the solution not treated with formaldehyde and the solution treated with formaldehyde, respectively. Pipet 10 mL from the test tube containing formaldehyde and Cresol and transfer into a test tube marked as N_1. Likewise, pipet 10 mL from the test tube containing only Cresol without formaldehyde and transfer into a test tube marked as N_2.
Observe the colors of test tubes N_1 and N_2 using a colorimeter. Next, add the orange to red solution from buret B_1 to tube N_1 until the colors in tubes N_1 and N_2 becomes the same. Then, add the equal volume of the yellow solution from buret B_2 to tube N_2. (NMT 5.0%)

$$\text{Content (\%) of phenol in Cresol} = 5V/W$$

V: Amount (mL) of the standard phenol solution consumed from buret B_1
W: Amount (g) of Cresol taken

Standardization of the standard phenol solution Pipet 4 mL of standard phenol solution into an iodine flask, add 30 mL of 0.05 mol/L bromine VS and 5 mL of hydrochloric acid, and immediately stopper the flask. Shake for 30 minutes to mix, allow to stand for 15 minutes, add 5 mL of potassium iodide (1 in 5), and stopper the flask, taking extra caution to prevent the escape of bromine vapor. Then, shake well to mix, remove the stopper, rinse the stopper and the neck of the flask with a small amount of water, and combine the water used for washing with the mixture. To the mixture, add 1 mL of chloroform and titrate the liberated iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 3 mL of starch TS). Perform a blank test and make any necessary correction.

$$\text{Each mL of 0.05 mol/L bromine VS} = 1.5685 \text{ mg of } C_6H_6O$$

Distilling range 196 to 206 °C, NLT 90 vol%.

Packaging and storage Preserve in light-resistant, tight containers.

Cresol Solution
크레솔수

Cresol Solution contains NLT 1.25 vol% and NMT 1.60 vol% of cresol.

Method of preparation

Saponated cresol solution	30 mL
Tap water or purified water	A suitable amount
	To make 1000 mL

Prepare by mixing the above ingredients.

Description Cresol Solution occurs as a clear or slightly turbid, yellow solution and has a characteristic odor of cresol.

Identification Take 0.5 mL of the oil layer obtained in the Assay, add 30 mL of water, shake to mix, and filter. Perform the following tests using the filtrate as the test solution.

(1) To 5 mL of the test solution, add 1 to 2 drops of dilute ferric chloride TS; the resulting solution exhibits a bluish purple color.

(2) Take 5 mL of the test solution and add 1 to 2 drops of bromine TS; a pale yellow, flocculent precipitation is formed.

Assay Transfer exactly 200 mL of Cresol Solution to a 500-mL distilling flask, add 40 g of sodium chloride and 3 mL of dilute sulfuric acid, and connect distilling apparatus with the distilling flask. Distill into a Cassia flask which contains exactly 30 g of powdered sodium chloride and exactly 3 mL of kerosene. When the volume of the distillate reaches 90 mL, draw off the water from the condenser and continue the distillation until water vapor begins to come out of the tip of the condenser. After the distillation is completed, place the Cassia flask in warm water for 15 minutes to dissolve sodium chloride with frequent shaking, and allow to stand for 15 minutes. After cooling down to 15 °C, add a saturated solution of sodium chloride and allow to stand for more than 3 hours with occasional shaking. Gently shake the separated oil droplets, allow to stand for 1 to 2 minutes, and combine the separated oil droplets with the oil layer. Subtract 3 (mL) from the volume (mL) of the oil layer; the resulting value represents the amount (mL) of cresol.

Packaging and storage Preserve in tight containers.

Saponated Cresol Solution
크레솔 비누액

Saponated Cresol Solution contains NLT 42 vol% and NMT 52 vol% of cresol.

Method of preparation

Cresol	500 mL
Vegetable oil	300 mL
Potassium hydroxide	A suitable amount

Tap water or purified water	A suitable amount
	To make 1000 mL

Dissolve a suitable amount of Potassium Hydroxide required for saponification in a suitable amount of Tap Water or Purified Water, add this solution to vegetable oil, previously warmed, add a suitable amount of Ethanol, if necessary, heat on a water bath, while stirring thoroughly, and continue the saponification. After the saponification is completed, add Cresol, stir thoroughly until the mixture becomes clear, and add a suitable amount of Tap Water or Purified Water to make 1000 mL. At this time, an equivalent amount of Sodium Hydroxide may be used in place of Potassium Hydroxide.

Description Saponated Cresol Solution occurs as a yellowish brown to reddish brown, viscous liquid and has the characteristic odor of cresol.
It is miscible with water, with ethanol or with glycerin.
It is alkaline.

Identification Proceed with the distillate obtained in (3) of the Purity as directed in the Identification under Cresol.

Purity (1) ***Alkali***—Mix 0.50 mL of Saponated Cresol Solution with 10 mL of neutralized ethanol, add 2 to 3 drops of phenolphthalein TS and 0.10 mL of 1 mol/L hydrochloric acid VS; the resulting solution does not exhibit a red color.

(2) ***Unsaponifiable matter*—**To 1.0 mL of Saponated Cresol Solution, add 5 mL of water, and shake to mix; the resulting solution is clear.

(3) ***Distillation fraction of cresol*—**Transfer 180 mL of Saponated Cresol Solution into a 2000-mL distilling flask, add 300 mL of water and 100 mL of dilute sulfuric acid, and distill with steam until the distillate becomes clear, remove the water from the condenser, and continue the distillation. When water vapor begins to come out of the tip of the condenser, pass through cooling water again, and continue the distillation for 5 more minutes. Dissolve 20 g of sodium chloride per 100 mL of the distillate, allow to stand, and collect the separated clear oil layer. To the oil layer, add about 15 g of powdered calcium chloride for drying little by little with frequent shaking to mix, allow to stand for 4 hours, and filter. Pipet 50 mL of the filtrate and distill; the amount of the distillate is NLT 43 mL at the temperature between 196 °C and 206 °C.

Assay Pipet 5 mL of Saponated Cresol Solution into a 500-mL distilling flask, holding the pipet vertically for 15 minutes to draw off the solution into the flask. Then, add 200 mL of water, 40 g of sodium chloride and 3 mL of dilute sulfuric acid, connect the distilling apparatus with the distilling flask, and distill into a Cassia flask which contains exactly 30 g of powdered sodium chloride and exactly 3 mL of kerosene. When the volume of the distillate reaches 90 mL, remove the water from the condenser, and continue the distillation until water vapor begins to come out of the tip of the condenser. After the distillation is completed, place the Cassia flask in warm water for 15 minutes to dissolve sodium chloride with occasional shaking, and allow to stand for 15 minutes. After cooling down to 15 °C, add a saturated solution of sodium chloride and allow to stand for more than 3 hours with occasional shaking. Gently shake the separated oil droplets, allow to stand for 1 to 2 minutes, and combine the separated oil droplets with the oil layer. Subtract 3 (mL) from the volume (mL) of the oil layer; the resulting value represents the amount (mL) of cresol.

Packaging and storage Preserve in light-resistant, tight containers.

Dental Iodine Glycerin
치과용 요오드·글리세린

Dental Iodine Glycerin contains NLT 9.0 w/v% and NMT 11.0 w/v% of iodine (I: 126.90), NLT 7.2 w/v% and NMT 8.8 w/v% of potassium iodide (KI: 166.00) and NLT 0.9 w/v% and NMT 1.1 w/v% of zinc sulfate hydrate ($ZnSO_4 \cdot 7H_2O$: 287.58).

Method of preparation

Iodine	10 g
Potassium iodide	8 g
Zinc sulfate hydrate	1 g
Glycerin	35 mL
Purified water	A suitable amount
	To make 100 mL

Prepare by dissolving and mixing the above ingredients.

Description Dental Iodine Glycerin occurs as a dark reddish brown liquid and has a characteristic odor of iodine.

Identification (1) The colored solution obtained in (1) under the Assay exhibits a red color. Determine the absorption spectrum of this solution as directed under the Ultraviolet-visible Spectroscopy; it exhibits a maximum at the wavelengths between 510 nm and 514 nm (iodine).

(2) The colored solution obtained in (2) under the Assay exhibits a red color. Determine the absorption spectrum of this solution as directed under the Ultraviolet-visible Spectroscopy; it exhibits a maximum at the wavelengths between 510 nm and 514 nm (potassium iodide).

(3) Take 1 mL of Dental Iodine Glycerin in a glass-stoppered test tube, add 10 mL of ethanol, then add 2 mL of sodium hydroxide TS and 1 mL of a solution of copper (II) chloride in ethanol (1 in 10), and shake to mix; the resulting solution exhibits a blue color (glycerin).

(4) The colored solution obtained (3) under the Assay exhibits a reddish purple to violet color. Determine the absorption spectrum of this solution as directed under the Ultraviolet-visible Spectroscopy; it exhibits a maximum at the wavelengths between 618 nm and 622 nm (zinc sulfate hydrate).

Assay (1) ***Iodine***—Pipet 5.0 mL of Dental Iodine Glycerin and add diluted ethanol (3 in 10) to make exactly 50 mL. Pipet 5 mL of this solution, add water to make exactly 200 mL, and use this solution as the test solution. Separately, weigh accurately about 0.5 g of iodine RS and 0.4 g of potassium iodide RS (previously dried at 105 °C for 4 hours) and dissolve them in diluted ethanol (3 in 10) to make exactly 50 mL. Pipet 5 mL of this solution, add water to make exactly 200 mL, and use this solution as the standard solution. Pipet 10 mL each of the test solution and the standard solution, add exactly 20 mL each of a mixture of chloroform and hexane (2 : 1), shake immediately to mix, take the chloroform-hexane layer [use the aqueous layer in (2)], and filter through absorbent cotton. Determine the absorbances, A_T and A_S, of the filtrates obtained from the test solution and the standard solution, respectively, at 512 nm as directed under the Ultraviolet-visible Spectroscopy, using the mixture of chloroform and

hexane (2 : 1) as the blank.

$$\text{Amount (mg) of iodine (I)} = \text{Amount (mg) of iodine RS} \times \frac{A_T}{A_S}$$

(2) ***Potassium iodide***—Pipet 7 mL each of the water layers obtained from the test solution and the standard solution in (1), add exactly 1.0 mL each of dilute hydrochloric acid (1 in 2), exactly 1 mL each of sodium nitrite TS and exactly 10 mL each of a mixture of chloroform and hexane (2 : 1), and shake immediately to mix. Take the chloroform-hexane layer and filter through absorbent cotton. Determine the absorbances, A_T and A_S, of the filtrates obtained from the test solution and the standard solution, respectively, at 512 nm as directed under the Ultraviolet-visible Spectroscopy, using the mixture of chloroform and hexane (2 : 1) as the blank.

$$\text{Amount (mg) of potassium iodide (KI)} = \text{Amount (mg) of potassium iodide RS} \times \frac{A_T}{A_S}$$

(3) ***Zinc sulfate hydrate***—Pipet 5.0 mL of Dental Iodine Glycerin and add diluted ethanol (3 in 10) to make exactly 50 mL. Pipet 5 mL of this solution, add water to make exactly 100 mL, and use this solution as the test solution. Separately, pipet 10 mL of standard zinc stock solution add diluted ethanol (3 in 200) to make exactly 1000 mL, and use this solution as the standard solution. Pipet 10 mL each of the test solution and the standard solution, add exactly 10 mL each of a mixture of chloroform and hexane (2 : 1), shake to mix, and allow to stand. Pipet 3 mL each of the aqueous layers, add 2 mL each of boric acid-potassium chloride-sodium hydroxide buffer solution (pH 10.0), 2 mL each of zincon TS and water to make exactly 25 mL. With these solutions, determine the absorbances, A_T and A_S, from the test solution and the standard solution, respectively, at 620 nm as directed under the Ultraviolet-visible Spectroscopy, using a solution prepared with 3 mL of water in the same manner as the blank.

$$\text{Amount (mg) of zinc sulfate heptahydrate } (ZnSO_4 \cdot 7H_2O) = \text{Amount (mg) of zinc in 10 mL of standard zinc stock solution} \times \frac{A_T}{A_S} \times 4.397$$

Packaging and storage Preserve in light-resistant, tight containers.

Dental Phenol with Camphor
치과용 페놀·캄파

Method of preparation

Phenol	35 g
d-or *dl*-Camphor	65 g
	To make 100 g

Prepare by melting Phenol by warming, adding *d*-Camphor or *dl*-Camphor, and then mixing.

Description Dental Phenol with Camphor occurs as a colorless or pale red liquid and has a characteristic odor.

Packaging and storage Preserve in light-resistant, tight containers.

Ethanol for Disinfection
소독용 에탄올

Ethanol for Disinfection

Ethanol for Disinfection contains NLT 76.9 vol% and NMT 81.4 vol% of ethanol (C_2H_6O: 46.07) at 15 °C (by specific gravity).

Method of preparation

Ethanol	830 mL
Purified Water	a sufficient quantity
	To make 1000 mL

Prepare by mixing the above ingredients.

Description Ethanol for Disinfection occurs as a clear, colorless liquid.
It is miscible with water.
It burns with a pale blue flame on ignition.
It is volatile.

Identification (1) To 1 mL of Ethanol for Disinfection, add 2 mL of iodine TS and 1mL of sodium hydroxide TS, and shake to mix; a pale yellow precipitate is formed.

(2) To 1 mL of Ethanol for Disinfection, add 1 mL of acetic acid (100) and 3 drops of sulfuric acid and heat; the characteristic odor of ethyl acetate is emitted.

Specific gravity d_{15}^{15}: Between 0.860 and 0.873

Purity Proceed as directed in the Purity under Ethanol.

Packaging and storage Preserve in light-resistant, tight containers.

Zinc Oxide Ointment
아연화 연고

Zinc Oxide Ointment contains NLT 18.5% and NMT 21.5% of zinc oxide (ZnO: 81.41).

Method of preparation

Zinc Oxide	200 g
Liquid Paraffin	30 g
White Ointment	A suitable amount
	To make 1000 g

Prepare as directed under Ointments, with the above ingredients.

Description The color of Zinc Oxide Ointment occurs as white.

Identification Place 1 g of Zinc Oxide Ointment in a crucible, melt by warming, gradually raise the temperature until the content is completely carbonized, and continue to ignite; the resulting residue exhibits a yellow color, which disappears upon cooling. To the residue, add 10 mL of water and 5 mL of dilute

hydrochloric acid, shake well to mix, and filter. To the filtrate, add 2 to 3 drops of potassium ferrocyanide TS; a white precipitate is formed (zinc oxide).

Purity ***Calcium, magnesium and other foreign inorganic matters*****—**Place 2.0 g of Zinc Oxide Ointment in a crucible, melt by warming, gradually raise the temperature until the content is completely carbonized, and continue to ignite until the resulting residue exhibits a yellow color. After cooling, add 6 mL of dilute hydrochloric acid and heat on a water bath for 5 to 10 minutes; the resulting solution is colorless and clear. Filter the solution, add 10 mL of water to the filtrate, and add ammonia TS until the precipitate initially formed disappears. To this solution, add 2 mL each of ammonium oxalate TS and sodium monohydrogen phosphate TS; the resulting solution remains unchanged or becomes slightly turbid within 5 minutes.

Assay Weigh accurately about 2 g of Zinc Oxide Ointment, place in a crucible, melt by warming, gradually raise the temperature until the content is completely carbonized, and continue to ignite until the resulting residue exhibits a yellow color. After cooling, dissolve the residue in 1 mL of water and 1.5 mL of hydrochloric acid and add water to make exactly 100 mL. Pipet 20 mL of this solution, add 80 mL of water and add a solution of sodium hydroxide (1 in 50) until a small amount of precipitates begins to form in the solution. Then, add 5 mL of ammonia-ammonium chloride buffer solution (pH 10.7) and titrate with 0.05 mol/L ethylenediaminetetraacetic acid disodium salt VS (indicator: 40 mg of eriochrome black T-sodium chloride indicator).

Each mL of 0.05 mol/L ehylenediaminetetraacetic acid disodium salt VS = 4.071 mg of ZnO

Packaging and storage Preserve in tight containers.