Monographs, Part II

1) Herbal Drugs and Herbal Drug Preparations

Acanthopanax Root Bark

Acanthopanacis Cortex

Acanthopanax Root Bark is the bark of the root and stem of the *Acanthopanax sessilifolium* Seeman or other species of the same genus (Araliaceae).

Description Acanthopanax Root Bark is the bark of the root and stem, usually cylindrical or semi-cylindrical, 5 to 10 cm in length, 5 to 8 mm in diameter and 1 mm in thickness. Outer surface is yellowish brown to dark gray and flat. Thorns or their marks are on the surface of stem, sporadically. Inner surface of the bark is yellowish white. The texture is fibroid and difficult to be cut.
Acanthopanax Root Bark has a characteristic odor and slightly bitter taste.

Identification Weigh 1 g of pulverized Acanthopanax Root Bark, add 10 mL of methanol, shake thoroughly, 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 a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of chloroform, methanol and water (70:30:4) to a distance of about 10 cm and air-dry the plate. Spray evenly sulfuric acid TS for spraying and heat at 105 °C: 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.

Purity (1) ***Foreign matter*—**(i) Xylem tissue and twigs: Not more than 2.0 %.

(ii) Other foreign matter: Acanthopanax Root Bark contains not more than 1.0 % of the foreign matter other than xylem tissue and twigs.

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Extract Content ***Water-soluble extract***—Not less than 8.0 %.

Acid-insoluble Ash Not more than 1.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Achyranthes Root

Achyranthis Radix

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

Description (1) ***Achyranthes japonica***—Achyranthes Root from *Achyranthes japonica* is a cylindrical main root with numerous lateral roots, 5 cm to 20 cm in length, 3 mm to 5 mm in diameter, with short remains of rhizome at the top. External surface is grayish yellow to pale yellow. Achyranthes Root is hard and brittle and the fractured surface is horn-like, yellowish white to yellowish brown.
Achyranthes Root from *Achyranthes japonica* has a slight, characteristic odor, slightly sweet taste and viscosity.

(2) ***Achyranthes bidentata***—Achyranthes Root from *Achyranthes bidentata* is the root, thin and long cylindrical, slightly curved, slightly thick at the top, relatively thin at the bottom, 15 cm to 50 cm in length and 0.4 cm to 1 cm in diameter. The external surface is grayish yellow to yellowish brown with several longitudinal wrinkles and rarely lateral root scars. The texture is hard and fragile, easy to cut and flexible on soaking in water. The fractured surface is even, yellowish brown, roughly horny and slippery. Under a microscope, the transverse section reveals a cork layer consisting of several rows of cork cells. The cortex is narrow. The stele takes up most of the root with several vascular bundles forming 2 to 4 concentric circles to form abnormal vascular bundes arranged intermittently. Vascular bundles in the outermost circle are relatively small and those in the third circle on the inside are relatively large. The xylem consists of vessels and xylem fiber and vessels are lignified or slightly lignified and sometimes contain deposits. A small number of parenchyma cells contain calcium oxalate crystal sand. In the middle are normal vascular bundles and the primary xylem is diarch.
Achyranthes Root from *Achyranthes bidentata* is nearly odorless, slightly sweet and mucilaginous.

Identification (1) Weigh 0.5 g of pulverized Achyranthes Root, add 10 mL of water and shake vigorously: a lasting fine foam is produced.

(2) Weigh 2 g of pulverized Achyranthes Root, add 10 mL of methanol, sonicate for 1 hour, filter and use

the filtrate as the test solution. Seperately, weigh 1 mg of 20-Hydroxyecdison RS, dissolve in 1 mL of methanol and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 5 μL each of the test solution and the standard solution on a plate of silica gel with a fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of cholroform, methanol and water (8 : 2 : 0.5) to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm) or spray evenly sulfuric acid TS for spray, and heat at 105 °C for 10 minutes: the spot 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*—**(i) Stem: Not more than 5.0 %.

(ii) Other foreign matter: The amount of foreign matter other than stems is not more than 1.0 %.

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): Not more than 0.2 ppm.

(vi) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 17.0 % (6 hours).

Ash Not more than 10.0 %.

Acid-insoluble Ash Not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Akebia Stem

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 external surface is yellowish white to yellowish brown with very numerous longitudinal ribs, nodes may appear expanded and may show scars of lateral branches. Those with the periderm are grayish brown with cylindrical or longitudinally long ellipsoidal lenticels. The texture is light, solid and difficult to cut. The cut surface on both sides is dark grayish brown. Xylem reveals pale brown vessels and grayish white medullary rays lined alternately and radially. Pith is pale grayish yellow and distinct. Under a microscope, a transverse section reveals a wheel shape ring mainly consisting of fiber bundles with crystal cells and stone cell groups and surrounding the relatively broad outside of the phloem in arc shape. Medullary rays of the phloems are consisted of sclerenchymatous cells containing solitary crystals. Cambium is distinct. Cells around the pith are remarkably thick-walled. Relatively well-developed large and small vessels radiate in the xylem and the medullary rays are in 4 to 5 rows. Medullary rays of xylem and parenchymatous cells around the pith contain solitary crystals of calcium oxalate and starch grains.
Akebia Stem is nearly odorless and has a slight acrid taste.

Identification Weigh 0.5 g of pulverized Akebia Stem, add 10 mL of water, boil, allow to cool and shake vigorously: a lasting fine foam is produced.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 10.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Alisma Rhizome

Alismatis Rhizoma

Alisma Rhizome is the tuber of *Alisma orientale* Juzepzuk (Alismataceae), from which periderm has been usually removed.

Description Alisma Rhizome is the 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 yellow-brown, with irregular, transverse, ring-shaped shallow furrows and numerous thin,

small, protruding scars due to rootlets, sometimes with strumous 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.
Alisma Rhizome has a slight, characteristic odor and taste.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): Not more than 0.2 ppm.
(vi) Endrin: Not more than 0.01 ppm.
(vii) Chlorpyrifos: Not more than 0.5 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 5.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Alpina Katsumadai Seed

Alpiniae Katsumadaii Semen

Alpina Katsumadai Seed is the seed of *Alpinia katsumadai* Hayata, removed from pericarp.

Description Alpina Katsumadai Seed is the mass of seeds, subspheroidal, 15 mm to 27 mm in diameter. External surface is grayish brown, with yellowish white septa in central part dividing the masses into three groups, each having numerous sticky seeds, agglutinated closely. Seed is ovoid-polyhedral, 3 mm to 5 mm in length, about 3 mm in diameter, covered with pale brown membranous aril, with raphe occurring as a longitudinal furrow and with hilum present at one end. Texture is hard. On cutting in half longitudinallay along the raphe, the seed shows oblique-cordate in shape. Endosperm is grayish white.
Alpina Katsumadai Seed has a characteristic odor and pungent, slightly bitter tastes.

Identification Weigh 1 g of pulverized Alpina Katsumadai Seed, add 5 mL of methanol and warm on a water bath for 5 minutes. Cool, 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. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 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 cyclohexane, ethyl acetate and acetic acid (100) (14 : 5 : 1) to a distance of about 10 cm and air-dry the plate. Spray evenly 5 % iron (II) chloride-ethanol solution on the plate: 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: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 12.0 %

Containers and Storage ***Containers*—**Well-closed containers.

Alpinia Officinarum Rhizome

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 in length and 10 mm to 15 mm in diameter. External 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 fracture surface is grayish brown to reddish brown and fibrous, in which the stele occupies one third. Under a microscope, the transverse section reveals that the outermost layer consists of the epidermis, with the epidermal cells sometimes containing resinous substances. The cortex and stele consist of parenchyma, separated by the endodermis, scattered with vascular bundles surrounded by fiber. The parenchyma is scattered with parenchyma cells containing a brown oily substance. The parenchyma cells have solitary crystals of calcium oxalate and starch grains. The starch grains are chiefly single grained and also has complex starch grains.
Alpinia Officinarum Rhizome has a characteristic odor and extremely pungent taste.

Identification Weigh 1 g of pulverized Alpinia Officinarum Rhizome, add 10 mL of ether, shake for 10 minites, and filter. To the residue obtained from evaporation of the filtrate, add 2 mL of phosphoric acid, heat and dissolve: yellow color develops. Add 2 mL of water, shake and keep; the solution become cloudy.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide*—**Not more than 30 ppm.

Loss on Drying Not more than 12.0 % (6 hours).

Ash Not more than 6.0 %.

Acid-insoluble Ash Not more than 1.5 %

Essential Oil Content Not less than 0.2 mL (50 g).

Extract Content ***Dilute ethanol-soluble extract***—Not less than 15.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Amomum Fruit

Amomi Fructus

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

Description Ammomum Fruit is the fruit, ellipsoidal or ovoid, indistinctly 3-ridged, 15 mm to 20 mm in length and 10 mm to 15 mm in diameter. External surface is pale brown, densely covered with spiny protrudings, apix with remains of perianth, and base often bearing a fruit stalk. Pericarp is thin and soft. The seeds are concentrated to make a mass with three blunt ridges. The center is divided into 3 loculi by white septa and each loculus contains 5 to 26 seeds. Seed is irregularly polyhedral, about 3 mm in diameter, and external surface is reddish brown or dark brown, the outside layer is pale brown and covered with a membranous aril. The texture is hard and endosperms are grayish white.
Amomum Fruit has a characteristic odor and taste is pungent, cool and slight bitter.

Identification Dissolve 20 μL of Amomum Fruit essential oil in 1 mL of ethanol and use this solution as the test solution. Or, weigh 1 g of pulverized Amomum Fruit, add 50 mL of ether, warm with a reflux condenser in 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. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 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 cycloheane and ethyl acetate (22:1) 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 spots obtained form the test solution shows the same color and R_f value as the spot from the standard solution.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 9.0 % (seed).

Acid-insoluble Ash Not more than 3.0 % (seed).

Essential Oil Content Not less than 0.6 mL (30.0 g, seed).

Containers and Storage ***Containers*—**Well-closed containers.

Amomum Tsao-ko Fruit

Amomi Tsao-ko Fructus

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

Identification 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. Ex-

ternal 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. Pericarp is hard, lasting and easily split 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.
Amomum Tsao-ko Fruit has a characteristic aroma and pungent, slightly bitter tastes.

Identification Dissolve 50 μL of the essential oil, as obtained from the Essential Oil Content, in 1 mL of ethanol and use this solution as the test solution. Separately, dissolve 20 μL of Cineole RS in 1 mL of ethanol and use this solution as the standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 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 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 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: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 12.0 %

Ash Not more than 9.0 %

Acid-insoluble Ash Not more than 3.0 %

Essential Oil Content Not less than 0.3 mL (100 g)

Containers and Storage ***Containers*—**Well-closed containers.

Anemarrhena Rhizome

Anemarrhenae Rhizoma

Anemarrhena Rhizome is the rhizome of *Anemarrhena asphodeloides* Bunge (Liliaceae). Anemarrhena Rhizome, when dried, contains not less than 0.7 % of mangiferin ($C_{19}H_{18}O_{11}$: 422.33).

Description Anemarrhena Rhizome is the slightly flat, thick rod-like rhizome, slightly curved, 3 cm to 15 cm in length and 5 mm to 15 mm in diameter. External surface is yellowish brown to brown. On the upper surface, a longitudinal furrow and hair-like remains or scars of leaf sheath forming fine ring-nodes are present. On the lower surface, scars of root appear as numerous round spot-like hollow. Anemarrhena Rhizome is light and easily broken. Fractured surface is pale yellowish brown. Under a magnifying glass, transverse section reveals an extremely narrow cortex and stele porous, with many irregularly scattered vascular bundles. Under a microscope, the transverse section 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 stele is scattered with multiple collateral vascular bundles and near the stele 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 relatively largely distributed in the cortex and contain calcium oxalate raphide bundles. Many calcium oxalate columnar crystal bundles are scattered throughout the parenchyma surrounding the vascular bundles. The parenchyma cells contain fatty oil drops.
Anemarrhena Rhizome has a slight, characteristic odor and slightly sweet and mucous taste, followed by bitterness.

Identification (1) Weigh 0.5 g of pulverized Anemarrhena Rhizome, add 10 mL of water: 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 dark green precipitate is produced.

(2) Weigh 0.5 g of pulverized Anemarrhena Rhizome with 2 mL of acetic anhydride on a water-bath for 2 minutes while shaking, filter and to the filtrate, add carefully 1 mL of sulfuric acid to make two layer: a red-brown color develops at the zone of contact.

(3) Weigh 2 g each of pulverized Anemarrhena Rhizome and Anemarrhena Rhizome RMPM, add 20 mL of ethanol, heat on a water bath for 40 minutes under a reflux condenser and filter, respectively. Add 1 mL of hydrochoric acid to 10 mL of the filtrate, heat on a water bath for 1 hour under a reflux condenser and filter again. Concentrate to about 5 mL of the filtrate in vaccum, add 10 mL of water and 20 mL of toluene to the concentrated solution, extract and evaporate the toluene layer to dryness. Dissolve the residue to 2 mL

of toluene and use the solutions as the test solution and the standard solution of Anemarrhena Rhizome RMPM. Separately, weigh 5 mg of Sarsasapogenin RS, dissolve in 1 mL of toluene and use this solution as the standard solution. Perform the test with the test solution, the standard solution of Anemarrhena Rhizome RMPM and the standard solution as directed under the Thin-layer Chromatography. Spot 10 μL each of the test solution, the standard solution of Anemarrhena Rhizome RMPM and the standard solution on a plate of silica gel for thin-layer chromatography. Deve1op the plate with a mixture of toluene and acetone (9 : 1) to a distance of about 10 cm and air-dry the plate. Spray the vanillin sulfuric acid TS to the plate, heat the plate at 105 °C for 10 minutes. The spots from the test solution and the spots from the standard solution of Anemarrhena Rhizome RMPM show the same color and the same R_f value. Of these, the spot of sarsasapogenin appears at the R_f value of 0.4.

Purity (1) ***Foreign matter***—The amount of fiber, originating from the dead leaves and other foreign matter is not more than 3.0 %.

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(vi) Cypermethrin: Not more than 0.5 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 7.0 %.

Acid-insoluble Ash Not more than 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, 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 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.

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

Operating conditions

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

Column: A stainless steel column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilanized 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 step or concentration gradient by mixing mobile phases A and B as directed in the following table.

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

Mobile phase B: Methanol

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

Flow rate: 1.0 mL/minute

Containers and Storage ***Containers***—Well-closed containers.

Angelica Dahurica Root

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).

Angelica Dahurica Root, when dried, contains not less than 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).

Description Angelica Dahurica Root is a main root from which many long roots are branched out and nearly fusiform, 10 cm to 25 cm in length and 15 mm to 25 mm in diameter. External surface is grayish brown to dark brown, with longitudinal wrinkles and with numerous scars of rootlets laterally elongated and protruded. A few remains of leaf sheath at the crown and ring-nodes closely protruded near the crown. In a transverse section, the outer region is grayish white and the central region is sometimes dark brown. Under a microscope, transverse section reveal vessels and medullary rays developed radially from the center, much starch grains, and calcium oxalate druses in parenchyma cells.

Angelica Dahurica Root 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. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 2 μL each of the test solution and the Angelica Dahurica Root RMPM standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of hexane and ethyl acetate (2:1) to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): of the spots obtained from the test solution, the spots at the R_f values of 0.45 and 0.7 show the same color as the spot from the Angelica Dahurica Root RMPM standard solution.

Purity (1) ***Foreign matter*—**(i) Leaf sheath: Not more than 3.0 %.

(ii) Other foreign matter: The amount of foreign matter other than leaf sheath contained in Angelica Dahurica Root dose not exceed than 1.0 %.

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 7.0 %.

Acid-insoluble Ash Not more than 2.0 %.

Extract Content ***Dilute ethanol-soluble extract***— Not less than 25.0 %.

Assay Weigh accurately about 1 g of pulverized Angelica Dahurica Root, add 50 mL of methanol, sonicate for 1 hour and filter. To the residue, add 50 mL of methanol and proceed in the same manner. 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 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}, 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 solution.

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

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

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

Operating conditions

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

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

Column temperature: An ordinary temperature

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

Flow rate: 1.0 mL/minute

System suitability

System performance: When the procedure is run with 10 μL of the standard solution under the above operating conditions, oxypeucedanin, imperatorin and isoimperatorin are eluted in this order.

System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions, the relative standard deviation of each peak area of oxypeucedanin, imperatorin and isoimperatorin is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Angelica Gigas Root

Angelicae Gigantis Radix

Angelica Gigas Root is the root of *Angelica gigas* Nakai (Umbelliferae). Angelica Gigas Root, when dried, contains not less than 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 decursenol angelate ($C_{19}H_{20}O_5$: 328.36)].

Description Angelica Gigas Root is the root, conical or narrow long conical in shape, usually branched, 15

cm to 25 cm in length and 2 cm to 5 cm in diameter. The external 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 hard 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 a microscope, the transverse section reveals cork consisting of 5 to 6 layers of cells, cells aligned transversely, parenchymas from primary cortex to xylem aligned systematically in rectangular shape. The cortex has schizogenous intercellular space, secretary canal with yellowish brown ingredient and bast fiber bundles are sparsely scattered. Scalariform or spiral vessel is observed. Numerous starch grains are observed in parenchyma cells.
Angelica Gigas Root has a slight, characteristic odor, and slightly bitter and sweet taste.

Identification Weigh 1 g each of pulverized Angelica Gigas Root and Angelica Gigas Root RMPM, dissolve separately in 5 mL of ethanol, heat in a water-bath for 10 minutes, cool, filter and use the filtrates as the test solution and the Angelica Gigas Root RMPM standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 5 μL each of the test solution and the Angelica Gigas Root RMPM standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of hexane and ethyl acetate (2:1) to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 365 nm): two spots among the spots from the test solution show the same color and Rf 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 stem and woody root.

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

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Methoxyclor: Not more than 1 ppm.

(iv) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(v) Azocyclotin: Not more than 0.2 ppm.

(vi) Azoxystrobin: Not more than 0.1 ppm.

(vii) Aldrin: Not more than 0.01 ppm.

(viii) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): Not more than 0.2 ppm.

(ix) Endrin: Not more than 0.01 ppm.

(x) Terbuconazole: Not more than 1.0 ppm.

(xi) Pendimethalin: Not more than 0.2 ppm.

(xii) Fenpropathrin: Not more than 0.2 ppm.

(xiii) Sethoxydim: Not more than 0.2 ppm.

(xiv) Fluazifop-butyl: Not more than 0.3 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 6.0 %.

Assay Weigh accurately about 0.5 g of pulverized Angelica Gigas Root, add 20 mL of methanol , heat with a reflux condensor for 1 hour, 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 about 10 mg of Nodakenin RS, dissolve in methanol to make 20 mL, take exactly 5 mL of this solution, dissolve acucurately about 10 mg of Decursin RS, add methanol to make exactly 50 mL, and use this solution as the standard solutions. 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 operating 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, the test solution and A_{Sa} and A_{Sb}, of nodakenin and decursin, respectively, 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 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: An ordinary temperature.

Mobile phase: Control gradually or concentration-gradiently with mobile phase A and B as follows.

Mobile phase A: acetonitrile

Mobile phase B: water

Time (min)	Mobile phase A (%)	Mobile phase B (%)

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: When the procedure is run with 10 μL of the standard solution under the above operating conditions, nodakenin and decursin are eluted in this order, clearly dividing each peak.

System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions, the relative standard deviation of each peak area of nodakenin and decursin is not more than 1.5 %.

Containers and Storage ***Containers*****—**Well-closed containers.

Apricot Kernel

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, when dried, contains not less than 3.0 % of amygdalin ($C_{20}H_{27}NO_{11}$: 457.43).

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 acute and other end is rounded, thickened and asymmetric. A short, linear hilum is situated on one side of the acute end and the chalaza is situated at the rounded end. The seed coat is brown and its surface has epidermal cells easily detachable by rubbing, giving a powdery appearance. Several deep brown vascular patterns stretch upwards from the chalaza. The seed coat and pale translucent white albumen easily separate from the cotyledon when softened in hot water. Cotyledons are two, milky white and oily. Under a microscope, the transverse section reveals a single row of epidermal cells on the outside, among which are yellow stone cells protruding. These stone cells are approximately 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 hypodermis consists of 1 row containing yellow substances. The perisperm is composed of several rows of degenerated parenchyma and the endosperm consists of 1 row of rectangular cells containing aleurone grains and fatty oil.

Apricot Kernel is nearly odorless and has a bitter taste.

Identification Weigh 1 g of pulverized Apricot Kernel, add 10 mL of methanol, heat for 10 minutes on a water bath with a reflux condenser. Filter after cooling and use this as the test solution. Separately, dissolve 2 mg of Amygdalin RS in 1 mL of methanol and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 10 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 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 for 10 minutes at 105 °C : one spot among the spots from the test solution and a brown to dark brown spot from the standard solution show the same color and 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: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

(5) ***Mycotoxins***—Total aflatoxin (sum of aflatoxins B_1, B_2, G_1 and G_2): Not more than 15 ppb (aflatoxin B_1 is not more than 10.0 ppb).

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

Assay Weigh accurately about 0.5 g of pulverized Apricot Kernel, add 50 mL of methanol, heat with a reflux condenser for 2 hours and filter. Repeat the above procedure with the residue using 50 mL of methanol. Combine the whole filtrates and evaporate to dryness under reduced pressure. Add 70 mL of water and 70 mL of hexane to the residue, shake well and discard the hexane layer. Add 70 mL of ether to the water layer, shake and discard the ether layer. The remaining water layer is filtered, adjust the total volume to make exactly 100 mL and use this solution as the test solution. Separately, dry the Amygdalin RS for 24 hours in the desiccator (silica gel) and weigh accurately about 10 mg, dissolve in 100 mL of water and use this solution as the standard solution. Perform the test with l0 μL each of the test solution and the standard solution as directed under the Liquid Chromatography accord-

ing to the following operating conditions and determine the peak areas, A_T and A_S, of amygdalin for the test solution and the standard solution, respectively.

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

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 214 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 (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/minute

System suitability

System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions, the relative standard deviation of the peak area of amygdalin is not more than 1.5 %.

Containers and Storage ***Containers*****—**Well-closed containers.

Aralia Continentalis Root

Araliae Continentalis Radix

Aralia Continentalis Root is the root of *Aralia continentalis* Kitakawa (Araliaceae). Aralia Continentalis Root, when dried, contains not less than 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).

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 is grayish-white to grayish-brown, with longitudinal wrinkles and sparse rootlet scars. Fractured surface is fibrous with pale yellow pith and texture is light and loose.

Under a microscope, transverse section reveals small resin canal with secretary cells in collenchyma. The clear cambium consists of 3 to 5 rows. Xylem fibers are developed around vessles in xylem, medullary rays consisting of 3 to 5 rows, are connected from the pith to the phloem.

Aralia Continentalis Root has a characteristic odor and tastes unpleasant and slightly bitter.

Identification (1) Weigh 0.5 g of pulverized Aralia Continentalis Root, add 10 mL of chloroform, extract for 1 hr with agitating, stand for 15 minutes and filter. Take 1.0 mL of the filtrate, add 0.5 mL of anhydrous acetic anhydride, vortex and add carefully 0.5 mL of sulfuric acid to make two layers: a red to dark red color develops at the zone of contact and the upper layer produces yellowish-red to dark yellowish red.

(2) Weigh about 1 g each of pulverized Aralia Continentalis Root and Aralia Continentalis Root RMPM, add 10 mL of methanol, sonicate for 1 hour, filter and use the filtrates as the test solution and the Aralia Continentalis Root RMPM standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 5 μL each of the test solution and the Aralia Continentalis Root RMPM standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of n-hexane and ethyl acetate (2:1) to a distance of about 10 cm and air-dry the plate. Spray evenly sulfuric acid TS for spraying and heat at 105 °C: the spots obtained from the test solution show the same color and R_f value as the spots from the Aralia Continentalis Root RMPM standard solution and of these, a yellow spot appears at the R_f value of about 0.8.

Purity (1) ***Heavy metals*****—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides*****—(**i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 12.0 %. (6 hours).

Ash Not more than 9.0 %.

Acid-insoluble Ash Not more than 2.0 %.

Assay Weigh accurately about 0.2 g of pulverized Aralia Continentalis Root, add 10 mL of methanol, 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 silica gel desiccator 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 standard solution as directed under Liquid Chromatography according to the following operating conditions and determine the peak areas, A_{Ta} and A_{Tb}, of kaurenoic acid and continentalic acid in the test solution and the peak areas, A_{Sa} and A_{Sb}, of kaurenoic acid and continentalic acid in 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 octadecylsilanized 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/minute

Containers and Storage ***Containers*—**Well-closed containers.

Arctium Fruit

Arctium Fructus

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

Description Arctium Fruit is slightly curved, long obovate achene, 5 to 7 mm in length, 2.0 to 3.2 mm in width, 0.8 to 1.5 mm in thickness. External surface is grayish brown to brown, with blackish purple spots. The longitudinal lines are several with 1 to 2 lines in the middle usually distinct. The apex is obtusely round and slightly broad with circular ring patterns on the top surface and spots of stigma scras in the middle. The lower part is slightly narrow compared to the upper part and the surface of attachment is relatively pale in color. The pericarp is relatively hard with 2 cotyledons, pale yellowish white, richly oily. About 100 fruits of Arctium Fruit weigh 1.0 to 1.5 g.
Arctium Fruit has odorless, bitter taste and oily.

Identification Weigh 0.5 g of pulverized Arctium Fruit, add 20 mL of methanol, shake for 10 minutes, filter, and use filtrate as the sample solution. Perform the test with the sample solution as directed under thin-layer chromatography. Spot 5 μL of the sample solution on a plate of silica gel for thin-layer chromatography, develop the plate with a mixture of acetone, ethyl acetate and water (15:10:1) to a distance of about 10 cm, and air-dry the plate. Spray evenly diluted sulfuric acid TS on the plate, and heat at 105 °C for 10 minutes: a red-purple spot appears at around R_f value 0.4.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 12.0 % (6 hours).

Ash Not more than 7.0 %.

Acid-insoluble Ash Not more than 1.0 %.

Extract Content ***Dilute ethanol-soluble extract***—not less than 15.0 %

Containers and Storage ***Containers*—**Well-closed containers.

Areca

Arecae Semen

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

Description Areca is the seed, rounded-conical or flattened nearly spherica1, l5 mm to 35 mm in height and 15 mm to 30 mm in diameter. Hilum is present at the center of its base and usually forms a dent. External surface is grayish reddish brown to grayish yellowish brown, with a network of pale lines. Texture is very hard and difficult to crack. Cross section is dense in texture, exhibiting a marble-like flower pattern of grayish brown seed coat alternating with white albumen. The interior is sometimes hollow.
Areca has a slight, characteristic odor and astringent and slightly bitter taste.

Identification Weigh 3 g of pulverized Areca and Areca RMPM in stoppered centrifuge tube, add 30 mL of ether and 5 ml of sodium hydroxide TS, stoppered, shake for 5 minutes, centrifuge and pipet the supernatant. Evaporate ether in 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. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 5 μL each of the test solution and the Areca RMPM standard solution on a plate of silica gel for thin-layer chromatography. De-

velop the plate with a mixture of acetone, water and acetic acid (100) (10 : 6 : l) to a distance of about 10 cm and air-dry the plate. Spray evenly iodine TS on the plate: the spots obtained from the test solution show the same color and R_f value as the spot from the Areca RMPM standard solution.

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

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

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

(5) ***Mycotoxins***—Total aflatoxin (sum of aflatoxins B_1, B_2, G_1 and G_2): Not more than 15.0 ppb (aflatoxin B_1 is not more than 10.0 ppbw).

Ash Not more than 2.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Areca Peel

Arecae Pericarpium

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

Description (1) ***Daebokpi*—**Daebokpi is the pericarp, usually elliptical or long ovoid gourd-shaped, 4 cm to 7 cm in length, 20 cm to 35 cm in width and 2 mm to 5 mm in thickness. Epicarp is deep brown to black, with irregular longitudinal wrinkles, raised transverse lines on the surfaces, stalk scars at apex, remains of fruit stalk and calyx at the base. Endocarp is dented, brown to deep brown, lustrous, smooth and hard shell-shaped. Texture is light and hard and mesocarp fibers visible torn longitudinally.
Daebokpi has a slight, characteristic odor and slightly astringent taste.

(2) ***Daebokmo*—**Daebokmo is the pericarp, usually elliptical or gourd-shaped. Epicarp is mostly lost or remained. Mesocarp is fibrous, yellowish white or pale brown, sparce and soft. Endocarp is hard shell-shaped, yellowish brown to deep brown. Inner surface is lustrous, smooth, and sometimes broken in longitudinal.
Daebokmo has a slight, characteristic odor and weak taste.

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

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

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 12.0 % (6 hours).

Ash Not more than 7.0 %.

Extract Content ***Dilute ethanol-soluble extract***—Not less than 5.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Arisaema Rhizome

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 removed.

Description Arisaema Rhizome is irregular oblate rhizome, 1.5 cm to 7 cm in diameter and 1 cm to 3 cm in height. The external surface is white to pale brown, relatively smooth with dented stem scars at the top 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.
Arisaema Rhizome has a slight pungent odor and taste.

Identification (1) Weigh 0.5 g of pulverized Arisaema Rhizome, add 10 mL of water, 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 for 2 minutes on a water-bath, filter and add carefully 0.5 mL of sulfuric acid to the filtrate: a pale brown color develops at the zone of contact.

(3) On a section of Arisaema Rhizome, add dilute iodine TS drop-wise: a dark blue-purple color is produced on the surface.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 15.0 % (6 hours).

Ash Not more than 5.0 %.

Acid-insoluble Ash Not more than 1.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Asiasarum Root and Rhizome

Asiasari Radix et Rhizoma

Asiasarum Root and Rhizome is the root and rhizome of *Asiasarum heteropoides* F. Maekawa var. *mandshuricum* F. Maekawa or *Asiasarum sieboldi* 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 is grayish brown with ring-shaped nodes, intermodal distance of 0.2 cm to 0.3 cm, 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 is grayish yellow, smoothly elongated or longitudinally wrinkled and with rootlets and rootlet scars. The texture is fragile and easy to cut. The cut surface is even and yellowish white or white. Under a microscope, the transverse section reveals the bark consisting of a single row of cells, longitudinally long or close to transversely long rectangular, with some epidermal cells remaining on the outside. The cortex consists of 10 to 17 layers of cells and the intracellcular space is distinct. Cells in the outside layer are dense and include a few cells containing yellow or yellow-brown substances. The cortex is scattered with numerous oil cells, and the oil cell walls are suberified or slightly suberified. The parenchyma cells of the cortex are filled with starch grains. The endodermal layer is distinct and shows a casparian strip. The stele 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 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 sieboldi* is very similar to *Asiasarum heteropoides* but has 25 to 43 vessels at the top of the root and the diameter of the largest vessel is larger than that of *Asiasarum heteropoides.*
Asiasarum Root and Rhizome has a characteristic odor and pungent taste with a slight numbness on the tongue.

Purity (1) ***Foreign matter***—(i) Terrestrial part: Asiasarum Root and Rhizome contains less than 10.0 % of its terrestrial part such as leaves and petioles.

(ii) Other foreign matter: Asiasarum Root and Rhizome contains less than 1.0 % of foreign matter other than terrestrial part.

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 10.0 %.

Acid-insoluble Ash Not more than 3.0 %.

Essential Oil Content Not less than 0.6 mL (30.0 g).

Containers and Storage ***Containers*—**Well-closed containers.

Asparagus Tuber

Asparagi Tuber

Asparagus Tuber is the tuber of *Asparagus cochinchinensis* Merill (Liliaceae). The cork layer has been removed and dried after boiling or steaming by hot water.

Description Asparagus Tuber is a fusiform to globular tuber, somewhat curved, 5 cm to 15 cm in length and 5 mm to 20 mm in diameter. External surface is pale yellow-brown to pale brown, semi-translucent, smooth or longitudinally wrinkled at irregular depths, sometimes with the grayish brown epidermis remaining. The texture is hard or soft, lustrous and viscous. The cut surface is horny and the stele is yellowish white. Under a microscope, a transverse section reveals the root bark sometimes remaining. The epidermis is composed of a single layer of root bark cells, the walls sclerified. The cortex takes up 2/3 of the root and stone cell groups form a single row of a ring on the outside. The stone cells are pale yellowish brown. Mucous cells are scattered in the cortex and contain calcium oxalate raphide bundles. The endodermis is distinct with a single row of pericycle forming a ring immediately below. The protoxylem and sieve tubes are immediately below with tracheids surrounding the vessels. The parenchyma cells of the pith are scattered with mucous cells, which contain calcium oxalate raphide bundles.
Asparagus Tuber has a slight, characteristic odor and tastes sweet followed by 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, filter, add 1 mL of Fehling solution TS to 3 mL of the filtrate and warm on a water-bath: a red-brown precipitate is produced.

(2) Weigh 1 g each of pulverized Asparagus Tuber and Asparagus Tuber RMPM, add 5 mL of a mixture of butanol, water (40 : 7), shake for 30 minutes and filter. Use the filtrates as the test solution and the Asparagus Tuber RMPM standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 μL each of the test solution and the Asparagus Tuber RMPM standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of butanol, water and acetic acid (100) (10 : 6 : 3) to a distance of about 10 cm and air-dry the plate. Spray dilute sulfuric acid TS to the plate, heat the plate at 105 °C for 2 minutes. The several spots obtained from the test solution show the same color and R_f value as the spots from the Asparagus Tuber RMPM standard solution.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 18.0 % (6 hours).

Ash Not more than 3.0 %.

Extract Content ***Dilute ethanol-soluble extract***—Not less than 25.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Aster Root and Rhizome

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 cm to 15 cm in length and 0.1 cm to 0.3 cm in diameter. The external surface is red-purple to grayish red and longitudinally wrinkled. The texture is relatively soft and tough and the fractured surface is fibrous.
Aster Root and Rhizome has a characteristic odor and tastes slightly bitter and acrid.

Identification (1) Weigh 0.2 g of pulverized Aster Root and Rhizome, add 10 mL of water, shake vigorously to mix, and filter. Add 1 to 2 drops of iron (III) chloride TS to 2 mL of the filtrate; a blue-violet color develops.

(2) Add 10 mL of water to 0.5 g of pulverized Aster Root and Rhizome and shake vigorously: a lasting fine foam is produced.

(3) Weigh 0.2 g of pulverized Aster Root and Rhizome, add 2 mL of acetate anhydride, shake the solution on a water bath and mix. Warm for 2 minutes and filter. Add slowly 0.5 mL of sulfuric acid to make two layers: a red-brown color develops at the zone of contact

Purity (1) ***Foreign matter***—Less than 5.0 % of stem and other foreign material.
(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.
(4) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 15.0 % (6 hours).

Ash Not more than 15.0 %

Acid-insoluble Ash Not more than 8.0 %.

Extract Content ***Dilute ethanol-soluble extract*—** Not less than 30.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Astragalus Root

Astragali Radix

Astragalus Root is the root or the root removed the periderm of *Astragalus membranaceus* Bunge or *Astragalus membranaceus* Bunge var. *mongholicus* Hsiao (Leguminosae)

Description Astragalus Root is nearly 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, twisted near the crown. External surface is pale grayish yellow to pale yellow-brown and covered with irregular, dispersed longitudinal wrinkles and horizontal lenticel-like patterns. Texture is dense and difficult to break and 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 somewhat 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. Usually pith is unobservable.
Astragalus Root has a slight, characteristic odor and sweet taste.

Identification Weigh 3 g of pulverized Astragalus Root, add 20 mL of methanol, heat with a reflux condenser for 1 hour, filter and evaporate the filtrate to dryness. Dissolve the residue in 0.5 mL of methanol and use this solution as the test solution. Separately, dissolve 1 mg of Formononetin RS in 1 mL of methanol and use this solution as the standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 5 μL each of the test solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of toluene and methanol (9 : 1) to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 254nm): 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: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Napropamide: Not more than 0.1 ppm.
(ii) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(iii) Dieldrin: Not more than 0.01 ppm.
(iv) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(v) Acetamiprid: Not more than 0.1 ppm.
(vi) Azoxystrobin: Not more than 0.1 ppm.
(vii) Aldrin: Not more than 0.01 ppm.
(viii) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): Not more than 0.2 ppm.
(ix) Endrin: Not more than 0.01 ppm.
(x) Imidacloprid: Not more than 0.3 ppm.
(xi) Triflumizole: Not more than 0.1 ppm.
(xii) Thiamethoxam: Not more than 0.1 ppm.
(xiii) Fenarimol: Not more than 0.5 ppm.
(xiv) Pymetrozine: Not more than 0.05 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.
(4) ***Root of Hedysarum species and others***—Under a microscope, a vertical section of Astragalus Root reveals no crystal fiber containing solitary crystals of calcium oxalate outside the fiber bundle.

Loss on Drying Not more than 13.0 % (6 hours).

Ash Not more than 5.0 %.

Acid-insoluble Ash Not more than 1.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Atractylodes Rhizome

Atractylodis Rhizoma

Atractylodes Rhizome is the rhizome of *Atractylodes lancea* De Candlle or of *Atractylodes chinensis* Koidzumi (Compositae).

Description Atractylodes Rhizome is cylindrical and irregularly curved rhizome, 3 cm to 10 cm in length and 10 mm to 25 mm in diameter. External surface is dark grayish brown to dark yellow-brown. A transverse section reveals nearly orbicular, with pale brown to red-brown secretes as fine points. Often white cotton-like crystals are produced on its surface if Atractylodes Rhizome is stored long time. Under a microscope, a transverse section usually reveals no fiber in parenchyma of cortex, and the end region of medullary rays reveals oil sacs containing pale brown to yellow-brown

substances. Xylem exhibits vessels surrounded by fiber bundles and arranged radially on the region adjoining the cambium. Pith and medullary rays exhibit the same oil sacs as in the cortex. Parenchyma cells contain spherocrystals of inulin and fine needles of calcium oxalate.
Atractylodes Rhizome has a characteristic odor and slightly bitter taste.

Identification Weigh 0.5 g of pulverized Atractylodes Rhizome, add 2 mL of hexane, sonicate for 15 minutes, filter and use the filtrate as the test solution. Perform the test with this solution as directed under Thin-layer Chromatography. Spot 5 μL of the test solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of petroleum ether and ethyl acetate (50 : 1) 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: a gray spot appears at the R_f value of about 0.5.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(vi) Tolylfluanid: Not more than 1 ppm.
(vii) Procymidone: Not more than 0.1 ppm.
(3) ***Sulfur dioxide***—Not more than 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: no red to red-purple color develops within l minute.

Ash Not more than 7.0 %.

Acid-insoluble Ash Not more than 1.5 %.

Essential Oil Content Not less than 0.7 mL (50.0 g).

Containers and Storage ***Containers—***Well-closed containers.

Atractylodes Rhizome White

Atractylodis Rhizoma Alba

Atractylodes Rhizome White is the rhizome, with or without periderm, of *Atractylodes japonica* Koidzumi or *Atractylodes macrocephala* Koidzumi (Compositae), or from which the periderm has been removed.

Description (1) ***Atractylodes japonica***—Atractylodes Rhizome White from *Atractylodes japonica* is rhizome, in a irregular mass or curved cylinder, 3 cm to 8 cm in length and 2 cm to 3 cm in diameter. For those without peridom, the external surface is pale grayish yellow to pale yellowish white and grayish brown here and there When periderm is remained, the external surface is balckish brown, sometimes in a protruding knot-shape, with coarse wrinkles. Texture is difficult to break and the fractured surface is fibrous. Under a microscope, a transverse section reveals periderm with stone cell layers, often fiber bundles at the outside of the phloem in the parenchyma of the cortex, and oil sacs containing pale brown to brown substances at the end of medullary rays. The xylem reveals small and radially lined vessels surrounding pith, and distinct fiber bundle surrounding these vessels. The pith and medullary rays contain oil sacs similar to those in cortex. The parenchyma tissues contain small crystals of inulin and needle crystals of calcium oxalate.
Atractylodes Rhizome White from *Atractylodes japonica* has a characteristic odor and somewhat bitter taste.
(2) ***Atractylodes macrocephala***—Atractylodes Rhizome White from *Atractylodes macrocephala* is the rhizome, in a shape of irregularly enlarged mass, 3 cm to 13 cm in length and 15 mm to 70 mm in diameter. External surface is grayish yellow or dark brown, having sporadic, knob-like small protrusions, interrupted longitudinal wrinkles and grooves, and scars of fibrous rootlets. Remains of stems and bud scars are attached to the apex. Texture is hard and difficult to be broken. The fractured surface is not flat and yellowish white to pale brown, and scatterd with yellowish brown oil sacs. The fractured surface of dried ones by baking is horny, relatively deep colored or cracked. Under a microscope, the transverse section reveals a stone cell layer in the periderm, usually none in the cortex. Oil sacs containing yellowish brown substances are present in the phloem rays and their tips. The cambium ring is distinct. The external vessels of the xylem are mostly radiating in 1 to 3 rows with no 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 needle crystals of calcium oxalate.
Atractylodes Rhizome White from *Atractylodes macrocephala* has a characteristic odor, sweet taste, and viscosity on chewing.

Identification Weigh 0.5 g of pulverized Atractylodes Rhizome White, add 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: a red to red-purple color develops and persists.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(vi) Captan: Not more than 2 ppm.

(vii) Procymidone: Not more than 0.1 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

(4) ***Atractylodes lancea rhyzome***—Weigh 2 g of pulverized Atractylodes Rhizome White, add 5.0 mL of hexane, shake for 5 minutes, filter and use this filtrate as the test solution. Perform the test with this solution as directed under the Thin-layer Chromatography. Spot 10 μL of the test solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of hexane and acetone (7 : 1) 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 °C for 5 minutes: no green to grayish green spot appears between R_f 0.3 and 0.6.

Ash Not more than 7.0 %.

Acid-insoluble Ash Not more than 1.0 %.

Essential Oil Content Not less than 0.5 mL (50.0 g).

Containers and Storage ***Containers*—**Well-closed containers.

Belladonna Extract

Belladonna Extract contains not less than 0.85 % and not more than 1.05 % of total alkaloids [as 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 % Ethanol and digest for 3 days. Press the mixture, add 2000 mL of 35 % Ethanol to the residue and digest again for 2 days. Combine all the extract and allow to stand for 2 days. Filter and prepare the viscous extract as directed under Extract. A appropriate quantity of Ethanol and Purified Water may be used in place of 35 % Ethanol.

Description Belladonna Extract is a dark brown, has a characteristic odor and bitter taste.

Identification Mix 0.5 g of Belladonna Extract with 30 mL of ammonia TS in a flask, transfer the mixture to a separatory funnel, then add 40 mL of ethyl acetate and shake the mixture. Drain off the ethyl acetate layer, add 3 g of anhydrous sodium sulfate to the ethyl acetate, shake and filter after the ethyl acetate become clear. Evaporate the filtrate to dryness in vaccum, 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: Not more than 30 ppm.

(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

Assay Weigh accurately about 0.4 g of Belladonna Extract, place in a glass-stopperd centrifuge tube, add 15 mL of ammonia TS and shake. Add 25 mL of ether, stopper tightly, shake for 15 minutes, centrifuge and separate the ether layer. Repeat this procedure twice with the water layer, using 25 mL each of ether. Combine 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 to make exactly 25 mL. 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 dihyrate in the mobile phase (1 in 2500)

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light-resistant, and in a cold place.

Belladonna Root

Belladonnae Radix

Belladonna Root is the root of *Atropa belladonna* Linné (Solanaceae). Belladonna Root, when dried, contains not less than 0.4 % of total alkaloids [as hyoscyamine ($C_{17}H_{23}NO_3$: 289.37)].

Description Belladonna Root is the root, cylindrical, sometimes cut crosswise or lengthwise, 10 cm to 30 cm in length and 5 mm to 40 mm in diameter. External surface is grayish brown to grayish yellow. Periderm of

Belladonna Root is often removed. Fractured surface is pale yellow to pale yellow-brown and much powdery. Belladonna Root is 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 in separatory funnel, add 40 mL of ethyl acetate, shake, separate the ethyl acetate layer, add 3 g of anhydrous sodium sulfate, shake and filter after the solution is clear. Evaporate ethyl acetate in vaccum, 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 in 1 mL of ethanol and use this solution as the standard solution. Perform the test with the test solution and the standard soltuion as directed under the Thin-layer Chromatography. Spot 5 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 acetone, water and ammonia water (90 : 7 : 3) to a distance of about 10 cm and dry the plate at 80 °C for 10 minutes. Spray evenly Dragendorff's TS for spraying: one spot among the spots 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: Less than 10.0 %.

(ii) Other foreign matter: The amount of foreign matter other than stems and crowns contained in Belladonna Root is not more than 2.0 %.

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

Ash Not more than 6.0 %.

Acid-insoluble Ash Not more than 4.0 %.

Assay Dry the pulverized Belladonna Root at 60 °C for 8 hours, weigh accurately about 0.7 g of pulverized Belladonna Root, place in a sotppered centrifuge tube and moisten with 15 mL of ammonia TS. Add 25 mL of ether, stopper, shake for 15 minutes, centrifuge and take the ether layer. To 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 mobile phase, add 3.0 mL of the internal standard solution and add mobile phase to make exactly 25 mL. Filter this solution with filter paper (not more than 0.8 μm in diameter), discard 2 mL of first filtrate and use the subsequent filtrate as the test solution. Separately, weigh accurately about 25 mg of Atropine Sulfate RS, previously determine loss on drying, dissolve in 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 mobile phase to make exactly 25 mL and use this solution as the standard solution. Perform the test with l0 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, Q_T and Q_S, of hyoscyamine (atropine) 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 mobile phase (1 in 2500).

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 ge1 (5 μm in particle diameter).

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

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

Selection of column: When the procedure is run with 10 μL of the standard solution under the above operating conditions, atropine and the internal standard are eluted in this order with a resolution between their peaks being not less than 4.0.

Containers and Storage ***Containers*—**Well-closed containers.

Benzoin

Benzoinum

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

Description Benzoin is the resin, grayish brown to dark red-brown block varying in size. The fractured surface exhibits white to pale yellow-red grains. Ben-

zoin is hard and tender at ordinary temperature but softened by heat.
Benzoin has a characteristic odor and slightly pungent and acrid taste.

Identification (1) Heat a fragment of Benzoin in a test tube: it evolves an irritating vapor and a crystalline sublimate is produced.

(2) Weigh 0.5 g of Benzoin, add 10 mL of ether, take 1 mL of the solution on a porcelain dish and add 2 to 3 drops of sulfuric acid: a deep red-brown to deep red-purple color develops.

Purity ***Ethanol-insoluble substances***—Weigh gently 1 g of Benzoin, boil with 30 mL of ethanol on a water-bath for 15 minutes under a reflux condenser. After cooling, collect the insoluble substances through a tared glass filter (G3) and wash three times with 5 mL volumes of ethanol. Dry the residue at 105 °C for 4 hours: the residue is not more than 0.3 g.

Ash Not more than 2.0 %.

Acid-insoluble Ash Not more than 1.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Bitter Cardamon

Alpiniae Oxyphyllae Fructus

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

Description Bitter Cardamon is spherical to fusiform fruit, with both ends somewhat pointed; 1 to 2 cm in length, and 7 to 10 mm in width. External surface is brown to dark brown, with numerous longitudinal, knob-like protruding lines. Pericarp is 0.3 to 0.5 mm in thickness. Inside of the Bitter Cardamon is divided vertically into three loculi by thin membranes, each loculus containing 5 to 8 seeds adhering by aril. Seeds are irregularly polygonal, about 3.5 mm in diameter, brown to dark brown, and texture is hard.
Bitter Cardamon has a characteristic odor and 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. Perform the test with this solution as directed under Thin-layer Chromatography. Spot 10 μL of the test solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. 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 green fluorescent spot appears at the R_f value of 0.3.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 10.0 %.

Acid-insoluble Ash Not more than 2.5 %.

Essential Oil Content Not less than 0.4 mL (50.0 g).

Containers and Storage ***Containers*—**Well-closed containers.

Bupleurum Root

Bupleuri Radix

Buplerum Root is the root of *Bupleurum falcatum* Linné or its varieties (Umbelliferae). Buplerum Root, when dried, contains not less than 0.3 % of saikosaponin *a* ($C_{42}H_{68}O_{13}$: 780.97).

Description Bupleurum Root is the root, in long cone or column-shape, single or branched, 10 cm to 15 cm in length and 5 mm to 15 mm in diameter. Upper part is thick and lower part thin. Apex has numerous hairy fibres from withered leaves. External surface is pale brown to brown with deep wrinkles. Texture is easily broken and the fractured surface is somewhat fibrous. Under a microscope, a transverse section reveals the thickness of cortex reaching 1/3 to 1/2 of the radius, tangentially extended clefts in cortex. Cortex is scattered with a good many intercellular schizogenous oil canals 15 μm to 35 μm in diameter. In xylem, vessels are lined radially or step-wise and fiber groups are scattered. The pith at the crown reveals the same oil canals, as in the cortex. Parenchyma cells contain fully starch grains and oil droplets.
Bupleurum Root has a characteristic order and 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 separately in 1 mL of methanol and use these solutions as standard solution (1) and standard solution (2). Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 μL each of the test solution, standard solution (1) and standard solution (2) on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, ethanol and water (8:2:1) to a distance of about 10 cm and air-dry the plate. Spray evenly dilute sulfuric acid TS and heat at 105 °C: two of the spots obtained from the test solution show the same color and Rf value as the purple spots obtained from standard solution (1) and standard solution (2).

Purity (1) ***Foreign matter*—**(i) Stem and leaf: Less than 10.0 %.

(ii) Other foreign matter: The amount of foreign matter other than sterns and leaves is not more than 1.0 %.

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(vi) Pendimethalin: Not more than 0.2 ppm.

(vii) Fosthiazate: Not more than 0.02 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 6.5 %.

Acid-insoluble Ash Not more than 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. To the filtrate, add methanol to make exactly 50 mL. Take 30.0 mL of this solution and evaporate. 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 desiccator of silica gel for 24 hours), add methanol to make exactly 20 mL and use this solution as 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 operating conditions and determine the peak areas, A_T and A_S, for the test solution and the standard solution, respectively.

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

Operating conditions

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

Column: A stainless steel column, 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica ge1 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/minute.

Containers and Storage ***Containers*—**Well-closed containers.

Capsicum

Capsici Fructus

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

Description Capsicum is elongated conical to fusiform fruit, often bent, about 3 cm to 10 cm in length and about 0.8 cm in width. The external surface is dark red to dark yellow-red and lustrous, usually with remains of calyx and peduncle. The interior is hollow and divided into two loculi, containing numerous seeds. The seeds are nearly circular and compressed, pale yellow-red and about 5 mm in diameter.

Capsicum has a characteristic odor and extremely pungent taste.

Identification Weigh 2.0 g of pulverized Capsicum, add 5 mL of ethanol, warm on a water-bath for 5 minutes, cool, centrifuge and use the supernatant liquid as the test solution. Separately, dissolve l mg of Capsaicin RS in l mL of ethanol and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 10 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 ether and methano1 (19 : 1) to a distance of about 12 cm and air-dry the plate. Spray evenly 2,6-dibromo-*N*-chloro-1,4-benzoquinonemonoimine TS on the plate and allow to stand in ammonia gas: a spot from the test solution and a blue spot from the standard solution show the same color and the same R_f value.

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

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—Proceed as directed in "Capsicum (Dried)" in [Attachment 4] MRLs for Agricultural Products in KFDA Notice "Standards and Specifications for Food."

Ash Not more than 6.0 %.

Acid-insoluble Ash Not more than 1.2 %.

Extract Content ***Ether-soluble extract***—Not less than 9.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Capsicum Tincture

Method of Preparation

Capsicum in medium cutting	100 g
Ethanol	a sufficient quantity
	To make 1000 mL

Prepare as direction under Tinctures, with the above ingredients.

Description Capsicum Tincture is yellowish red liquid and has extreamly pungent taste.

Specific gravity—d_{20}^{20} : About 0.82.

Identification Proceed as directed in the Identification under Capsicum. Spot 20 μL each of the solution and the standard solution.

Alcohol Number Not less than 9.7 (Method 2)

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light-resistant.

Cardamon

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 surfaceis pale yellow and has three blunt ridges and many longitudinal lines. At the upper end, small protrusion is present. Interior is longitudinally divided into three loculi by thin membranes, each loculus containing 3 to 7 seeds joining by aril. Seed is ovoid to long ovoid or irregularly angular, 3 mm to 4 mm in length, dark brown to blackish brown. The dorsal side is convex, the ventral side is longitudinally grooved and coarsely tuberculated.

Cardamon has a characteristic odor and tastes pungent and slightly bitter. Pericarp is odorless and tasteless.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 6.0 % (seed).

Acid-insoluble Ash Not more than 4.0 % (seed).

Essential Oil Content Not less than 1.0 mL (30.0 g, seed).

Containers and Storage ***Containers*—**Well-closed containers.

Cassia Seed

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 short cylindrical seed, relatively small, 3mm to 5 mm in length and 2 mm to 3 mm in diameter. Both sides of the external ridge have a wide, pale yellow-brown band.

When cracked, Cassia Seed has a characteristic odor and taste.

(2) ***Cassia obtusifolia***—Cassia Seed from *Cassia obtusifolia* is rectangular or short cylindrical seed with both slopes sloping in parallel, 3 mm to 7 mm in length

and 2 mm to 4 mm in width. The external surface is greenish brown or dark brown, smooth and lustrous. One end is relatively flat and the other is oblique and acuminate. A ridgeline is prominent at the back and belly. Each side of the ridgeline 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, on a slide glass, put a glass ring, 10 mm in both internal diameter and height on it, then cover 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 l 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: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endosulfan (sum of α, β-endosulfan and endosulfan sulfate): Not more than 0.2 ppm.

(vi) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

(5) ***Mycotoxins***—Total aflatoxin (sum of aflatoxin B_1, B_2, G_1 and G_2): Not more than 15.0 ppb (aflatoxin B_1 is not more than 10.0 ppb).

Ash Not more than 5.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Cattle Gallstone

Bovis Calculus

Cattle Gallstone is a stone formed in the gall sac of *Bos taurus* Linné var. *domesticus* Gmelin (Bovidae). Cattle Gallstone, when dried, contains not less than 20.0 % of conjugated bilirubin ($C_{33}H_{36}N_4O_6$: 584.66).

Description Cattle Gallstone is the stone, mostly ovate, close to spherical, triangular or quadrilateral cylindrical, irregular in size, rarely cylindrical or broken pieces, 0.6 cm to 4.5 cm in diameter. The external surface is red to yellow-brown, sometimes covered in a black shiny film; this is known as "Ogeumui." Some are coarse, strumous, sometimes with a cracked pattern. The body is light and the texture is fragile, easily detached by layer. The cut surface is golden with fine concentric lamella visible, sometimes with a narrow white core.

Cattle Gallstone has a clear aroma and tastes slightly bitter, later sweet and cool, easily broken when chewed and does not adhere to teeth.

Identification (1) Weigh 0.1 g of pulverized Cattle Gallstone, add 10 mL of petroleum ether, shake for 30 minutes, filter and wash the residue with 10 mL of petroleum ether. Shake 10 mg of the residue with 3 mL of acetic anhydride for 1 to 2 minutes, add a mixture of 0.5 mL of acetic anhydride and 2 drops of sulfuric acid and shake: a yellow-red to deep red color develops and changes through dark red-purple to dark red-brown.

(2) Weigh 10 mg of Cattle Gallstone, add 10 mL of chloroform, shake well and discard extracted chloroform. Shake well the residue with 1 mL of hydrochloric acid and 10 mL of chloroform, separate the chloroform layer when it acquires a yellow-brown color and shake with 5 mL of barium hydroxide TS: a yellow-brown precipitate is produced.

Purity (1) ***Curcuma Root, Curcuma Longa Rhizome***—Weigh 0.1 g of pulverized Cattle Gallstone, add 50 mL of methanol and heat for 30 minutes in a water-bath with reflux condensor. Filter after cooling, concentrate by evaporating the filtrate to 1 mL and use this as the test solution. Seperately weigh 1 mg of Curcumin RS, disslve in methanol to make 5 mL and use this as the standard solution. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 10 μL each of the test solution and the standard solution on a plate 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) to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 365 nm): the spot from the test solution dose not show a yellowish fluorescent spot at the same R_f value of the standard solution.

(2) ***Synthesized pigments***—Weigh 2 mg of pulverized Cattle Gallstone and add 1 mL of dilute hydrochloric acid: the solution does not show purple color.

(3) ***Starch***—Weigh 5 mg of pulverized Cattle Gallstone, add 2 mL of water, heat for 5 minutes in a water-bath. After cooling, add 2 to 3 drops of iodine TS: the solution does not show blue-purple color.

(4) ***Sucrose***—Weigh 20 mg of pulverized Cattle Gallstone, add 2 mL of water, shake for 15 minutes and filter. To 1 mL of the filtrate, add 2 mL of anthrone TS and shake: the solution does not show deep blue-green to dark green color.

Ash Not more than 10.0 %.

Assay Proceed under the dark place as fast as possible. Weigh accurately about 0.1 g of the fine powder of Cattle Gallstone to a Erlenmeyer flask, add 10 mL of diluted hydrochloric acid (1 in 5) and 200 mL of chloroform, mix well, warm with a reflux condenser in a water-bath at 61 ± 2 °C for 90 minutes and cool. Transfer the chloroform extract into a separatory funnel. The flask is washed with a small volume of chloroform and place the chloroform layer into the separatory funnel. Allow to stand for 10 minutes and take the chloroform layer. The remaining water layer is back extracted with chloroform. Combine all of the chloroform layer, add 5 g of anhydrous sodium sulfate, mix well and filter. Add chloroform to the filtrate to make 200 mL and use this solution as test solution for total bilirubin. Separately, weigh accurately about 0.1 g of pulverized Cattle Gallstone, dissolve in 200 mL of chloroform, filter and use this filtrate as test solution for free bilirubin. Separately, Weigh accurately about 20 mg of Bilirubin RS, dissolve in chloroform to make 100 mL, use this solution as standard solution. Pipet 5 μL each 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_T and A_S, of bilirubin for the test solution and the standard solution, respectively.

$$\text{Amount (mg) of total bilirubin or free bilirubin} = \text{amount (mg) of Bilirubin RS} \times \frac{A_T}{A_S} \times 2$$

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

Operating conditions

Detector: A visible absorption photometer (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 (5 μm to 10 μm in particle diameter).

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

Mobile phase: A mixture of methanol, water and acetic acid (900 : 98 : 2).

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

System suitability

System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions, the relative standard deviation of the peak area of bilirubin is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Cibot Rhizome

Cibotii Rhizoma

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

Description Cibot Rhizome is the rhizome, irregularly long mass-shaped, 10 cm to 30 cm in length and 2 cm to 10 cm in diameter. External surface is deep brown, with remains of golden hairs. The upper part is exhibiting several reddish brown woody petioles and the lower part is showing remains of black fibrous roots. Texture is hard and uneasily broken.
Cibot Rhizome is nearly odorless and the taste is weak and slightly astringent.

Identification (1) Weigh 1 g of pulverized Cibot Rhizome, add 10 mL of methanol, boil in the water bath for 15 minutes and filter. Drop the filtrate on the filter paper and examine under a ultraviolet light (365 nm): fluorescence of bluish white color develp.

(2) Weigh 1 g of pulverized Cibot Rhizome, add 10 mL of water, boil in the water bath for 15 minutes and filter. Drop the 1 % iron (III) chloride solution to 2 mL of the filtrate:: the filtrate turn to dark green.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 11.0 % .

Ash Not more than 2.5 %.

Extract Content ***Dilute ethanol-soluble extract*—** Not less than 22.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Cimicifuga Rhizome

Cimicifugae Rhizoma

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

Description Cimicifuga Rhizome is the rhizome, irregular, long masses, frequently branched, knotted, 10 cm to 20 cm in length and 2 cm to 4 cm in diameter. The external surface is blackish brown to maroon, slightly coarse and uneven. The upper surface has several empty circular holes and stem scars. The inside wall of the holes have a distinct, slightly dented mesh-like pattern. The ottom part has scars of rootlets. The body is light, hard and not easy to cut. The cut surface is uneven with open clefts, fibrous, yellowish green to pale yellowish white.
Cimicifuga Rhizome is nearly odorless and has bitter and slightly astringent taste.

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

Ash Not more than 9.0 %.

Acid-insoluble Ash Not more than 1.5 %.

Extract Content ***Dilute ethanol-soluble extract***—Not less than 18.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Cinnamon Bark

Cinnamomi Cortex

Cinnamon Bark is the bark of the trunk of *Cinnamomum cassia* Presl or other species of the same genus (Lauraceae), or such bark from which a part of the periderm has been removed. Cinnamon Bark contains not less than 0.03 % of cinnamic acid ($C_9H_8O_2$: 148.16), calculated on the dried basis.

Description Cinnamon Bark is usually cylindrical or cylindrically rolled pieces of bark, 5 cm to 50 cm in length and 15 cm to 50 cm in diameter, 1 mm to 5 mm in thickness. External surface is dark red-brown and the inner surface is red-brown and smooth. Cinnamon Bark is brittle and the fractured surface is slightly fibrous, red-brown, exhibiting a pale brown, thin layer. Under a microscope, a transverse section reveals a primary cortex and a secondary cortex divided by an almost continuous ring consisting of stone cells. Nearly round bundles of fibers are in the outer region of the ring and wall of each stone cell is often thickened in a U-shape. Secondary cortex of Cinnamon Bark lacks stone cells and is with a small number of sclerenchymatous fibers coarsely scattered. Parenchyma tissue is scattered with oil cells, mucilage cells and cells containing fine needles of calcium oxalate and parenchyma cell contains starch grains.
Cinnamon Bark has a characteristic aroma, sweet and pungent taste at first, later rather mucilaginous and slightly astringent.

Identification Weigh about 2 g each of pulverized Cinnamon Bark and Cinnamon Bark RMPM, add 10 mL of ether, shake for 3 minutes, filter and use the filtrates as the test solution and the Cinnamon Bark RMPM standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 μL each of the test solution and the Cinnamon Bark RMPM standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of petroleum ether and ethyl acetate (17:3) to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): the spots obtained from the test solution show the same color and R_f value as the spots from the Cinnamon Bark RMPM standard solution and of these, the spot of cinnamaldehyde appears at the R_f value of 0.7.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 15.5 % (6 hours).

Ash Not more than 5.0 %.

Assay Weigh about 1.0 g of pulverized Cinnamon Bark, add 50 mL of methanol, sonicate, filter, add methanol to make exactly 50 mL and use this solution as the test solution. Separately, weigh about 10 mg of Cinnamic Acid RS (previously dried in a silica gel desiccator for not less than 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 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.

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

Operating conditions

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

Column: A stainlees 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: An ordinary temperature.

Mobile phase: A mixture of water, acetonitrile and acetic acid (100) (68:30:2)

Flow rate: 1.0 mL/minute.

System suitability

System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions, the relative standard deviation of the peak area of cinnamic acid is not more than 1.5 %.

Containers and Storage ***Containers*****—**Well-closed containers.

Citrus Unshiu Immature Peel

Citri Unshius Pericarpium Immaturus

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

Description Citrus Unshiu Immature Peel is the irregularly shaped pieces of pericarp, about 2 mm in thickness. The external surface is grayish green to blue-green, rough and wrinkled, with dented scars due to oil sacs. The inside is white to grayish white. The texture is light and easily broken.
Citrus Unshiu Immature Peel has a characteristic odor, bitter and slightly pungent tastes.

Identification (1) Weigh 0.3 g of pulverized Citrii Unshiu Immature Peel, add 10 mL of methanol, heat with a reflux condenser for 20 minutes and filter. Add small amount of magnesium powder to 1 ml of the filtrate and add 3 to 5 droplets of hydrochloric acid: a scarlet color slowly appears.

(2) Evaporate 5 mL of the filtrate of (1) to about 1 mL, use this solution as the test solution. Separately, use the saturated solution of Hesperidin RS in methanol as the standard solution. Perform the test with the test solution and the standard solution as directed under Thin-layer Chromatography. Spot 5 μL each of the test solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. 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 on a plate and heat at 105 °C: a fluorescent spot among the several spots form the test solution and a spot from the standard solution show the same color and the some R_f value.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(vi) Methidathion: Not more than 10 ppm.

(vii) Tetradifon: Not more than 25 ppm.

(viii) Triazophos: Not more than 5 ppm.

(ix) Fenitrothion: Not more than 10 ppm.

(x) Phenthoate: Not more than 10 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 12.0 %

Ash Not more than 5.0 %

Acid-insoluble Ash Not more than 1.0 %

Essential Oil Content Not less than 0.2 mL (50 g)

Extract Content ***Dilute ethanol-soluble extract***— Not less than 12.0 %

Containers and Storage ***Containers*****—**Well-closed containers.

Citrus Unshiu Peel

Citri Unshius Pericarpium

Citrus Unshiu Peel is the ripe pericarp of *Citrus unshiu* Markovich or *Citrus reticulate* Blanco (Rutaceae). Citrus Unshiu Peel contains not less than 4.0 % of hesperidin ($C_{28}H_{34}O_{15}$: 610.56), calculated on the dried basis.

Description Citrus Unshiu Peel is irregular, plate-shaped pieces of pericarp, about 2 mm in thickness. External surface is yellow-red to dark yellow-red to dark yellow-brown, with numerous small dents associated with oil sacs. Interior is white to pale grayish yellow-brown. Texture is light and brittle.
Citrus Unshiu Peel has a characteristic odor and bitter and slightly pungent taste.

Identification (1) Weigh 0.5 g of pulverized Citrus Unshiu Peel, add 10 mL of methanol, warm 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 to stand: a red-purple color develops.

(2) Weigh 0.5 g of pulverized Citrus Unshiu Peel and Citrus Unshiu Peel RMPM, add 10 mL of methanol, sonicate for 20 minutes and filter. Use the filtrates as the test solution and the Citrus Unshiu Peel RMPM standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 μL each of the test solution and the Citrus Unshiu Peel standard solution on a plate of silica gel with a fluorescent indicator for thin-layer chromatography. Deve1op the plate with a mixture of ethyl acetate, methanol and water (100 : 17 : 3) to a distance of about 10 cm and air-dry the plate. Spray evenly the aluminium chloride TS to the plate and examine under ultraviolet light (main wavelength: 365 nm): the several spots from the test solution show the same color and R_f value as the spots from the Citrus Unshiu Peel RMPM standard solution.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(vi) Methoxychlor: Not more than 1 ppm.
(vii) Methidathion: Not more than 6 ppm.
(viii) Tetradifon: Not more than 15 ppm.
(ix) Triazophos: Not more than 2 ppm.
(x) Fenitrothion: Not more than 10 ppm.
(xi) Phenthoate: Not more than 6 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 13.0 % (6 hours).

Ash Not more than 4.0 %.

Assay Weigh accurately about 0.5 g of pulverized Citrus Unshiu Peel, add 60 mL of methanol, heat with a reflux condenser for 2 hours and filter. To the residue, add 30 mL of methanol and proceed in same manner. Combine all filtrates, add methanol to make exactly 100 mL and use this solution as the test solution. Weigh accurately about 20 mg of Hesperidin RS, dissolve in methanol to make 100 mL and use this solution as the standard solution. Perform the test with l0 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_T and A_S, of hesperidin for 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 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica ge1 (5 μm to l0 μm in particle diameter).
Column temperature: An ordinary temperature.
Mobile phase: A mixture of water and methanol (60:40).
Flow rate: 1.0 mL/minute.
System suitability
System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions, the relative standard deviation of he peak area of hesperidin is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Clove

Syzygii Flos

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

Description Clove is the bud, slightly trimmed club-shaped, l cm to 2 cm in length. The corolla is globose, 0.3 cm to 0.5 cm in diameter. There are 4 petals, overlapping in a bottle shape, maroon to yellow-brown. Inside the petals are the stamen and style with numerous fine, yellow, granular pollen visible on breaking by rubbing. The calyx tube is cylindrical, slightly flat, sometimes slightly curved, 0.7 cm to 1.4 cm in length

and 0.3 cm to 0.6 cm in diameter. The external surface is red-brown or maroon. The calyx of the upper part divides into 4 and the lobe is triangular. The texture is solid and richly oily. Under a microscope, a transverse section reveals irregular, long ovoid oil sacs in the outer surrounding surface, two layered vascular bundles surrounded by collenchyma in the inner surface, bast fiber in phloem, and spongy tissue in vascular bundles. Parenchyma cell surrounded by vascular bundles contains aggregate crystals of calcium oxalates and oil droplets of essential oil.
Clove has strong, characteristic odor and pungent taste, followed by a slight numbness of the tongue.

Identification Take 0.1 mL of the mixture of xylene and essential oi1, obtained in the Essential oil content, add 2 mL of ethanol and add l to 2 drops of iron (III) chloride TS: a green to blue color develops.

Purity (1) ***Foreign matter*—**(i) Stem: Less than 5.0 %.

(ii) Other foreign matter: The amount of foreign matter other than the stem is not more than 1.0 %.

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 7.0 %.

Acid-insoluble Ash Not more than 0.5 %.

Essential Oil Content Not less than 1.6 mL (10.0 g).

Containers and Storage ***Containers*—**Well-closed containers.

Cnidium Rhizome

Cnidii Rhizoma

Cnidium Rhizome is the rhizome or the rhizome passed through hot water of *Cnidium officinale* Makino or *Ligusticum chuanxiong* Hort. (Umbelliferae).

Description Cnidium Rhizome is irregular massive, knotted rhizome, 5 cm to 10 cm in length and 3 cm to 5 cm in diameter. The external surface is grayish brown to dark brown, coarse, 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, a transverse section reveals a cork layer composed of about 10 rows of flat cork cells. The cortex is narrow, scattered with root-trace vascular bundles, the cells are transversely long with many nearly orbicular oil sacs. The phloem is relatively broad, scattered with sieve tube groups. The cambium forms a ring. In the xylem, the 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, sometimes calcium oxalate crystals, nearly orbicular masses or nearly rosette crystals. Cnidium Rhizome from *Cnidium officinale* and Cnidium Rhizome from *Ligusticum chuanxiong* are generally similar.
Cnidium Rhizome has a characteristic odor and slightly bitter taste.

Identification Weigh 1 g each of pulverized Cnidium Rhizome and Cnidium Rhizome RMPM, add 10 mL of diluted ethanol (7 in 10), sonicate for 60 minutes, filter and use the filtrates as the test solution and the Cnidium Rhizome RMPM standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 2 μL each of the test solution and the Cnidium Rhizome RMPM standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of hexane, ethyl acetate and methanol (10 : 2 : 1) 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 Cnidium Rhizome RMPM standard solution and among these, the spot of ligustilide appears at the R_f value of about 0.6.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Bifenthrin: Not more than 0.5 ppm.

(v) Aldrin: Not more than 0.01 ppm.

(vi) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): Not more than 0.2 ppm.

(vii) Endrin: Not more than 0.01 ppm.

(viii) Chlorpyrifos: Not more than 0.5 ppm.

(ix) Chlorfenapyr: Not more than 2.0 ppm.

(x) Tebufenpyrad: Not more than 0.1 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 6.0 %.

Acid-insoluble Ash Not more than 1.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Codonopsis Pilosula Root

Codonopsis Pilosulae Radix

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

Description (1) ***Root of Codonopsis pilosula*—** Codonopsis Pilosula Root from *Codonopsis pilosula* consists of the root, long cylindrical, slightly curved, 10 cm to 35 cm in length and 4 mm to 20 mm in diameter. The external surface is yellowish brown to grayish brown. The root stock has numerous warty prominent stem scars and buds, and the apex of each stem scar is sunkenly dotted. Dense, ring-shaped transverse annulations are occurring below the root stock, gradually sparse downwards. These transverse annulations are sometimes up to half of the entire length. These transverse annulations are rare in cultivars, which are hairless, the whole root showing longitudinal wrinkles and scattered transversely long lenticels, usually with blackish brown gelatinous substances at the fractured area of the rootlet. 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.
Codonopsis Pilosula Root has the characteristic odor and slightly sweet taste.

(2) ***Root of 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. External surface is yellowish white to grayish yellow. Dense transverse annulations are occurring below the root stock, frequently up to over half length of the root. Fractured surface has more clefts and the cortex is grayish white or pale brown.

(3) ***Root of Codonopsis tangshen*—**Codonopsis Pilosula Root from *Codonopsis tangshen* consists of the root, long cylindrical in shape, 10 cm to 45 cm in length and 5 mm to 20 mm in diameter. External surface is grayish yellow to yellowish brown, with distinctly longitudinal wrinkles. Texture is slightly soft and tenacious. The fractured surface has less clefts, and the cortex is yellowish white.

Identification Weigh 1 g of pulverized Codonopsis Pilosula Root, add 10 mL of water, boil and cool. Pour this solution to test tube and shake vigorously: the fine persistent foam is rising.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): Not more than 0.2 ppm.
(vi) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 13.0 % .

Ash Not more than 6.0 %.

Acid-insoluble Ash Not more than 2.0 %

Extract Content ***Dilute ethanol-soluble extract***—Not less than 35.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Coix Seed

Coicis Semen

Coix Seed is the ripe seed of *Coix lachryma-jobi* Linné var. *ma-yuen* Stapf (Gramineae), from which the seed coat has been removed.

Description Coix Seed is ovoid or broad ovoid seed, about 6 mm in length and about 5 mm in width with a slightly hollowed apex and base. Dorsal side is distended and ventral side is longitudinally and deeply furrowed in the center. Dorsal side is mostly white and powdery. In the furrow on the ventral surface, brown and membranous pericarp and seed coat are attached. Under a magnifying glass, a transverse section reveals white endosperm in the dorsal side and pale yellow scutellum in the hollow of the ventral side.
Coix Seed has a slight, characteristic odor and slightly sweet taste. Coix Seed adheres to the teeth on chewing.

Identification (1) Add iodine TS drop-wise to a transverse section of Coix Seed: a dark red-brown color develops in the endosperm and a dark gray color develops in the scutellum.

(2) Place a small amount of Coix Seed on a slide glass, add drop-wise iodine TS and examine under a microscope: nearly equi-diameter and obtuse polygonal simple starch grains, usually 10 μm to 15 μm in diame-

ter and compound starch grains have a reddish brown color, and small spheroidal starch grains, coexisting with fixed oil and with aleuron grains in parenchyma cells, have a blue-purple color.

(3) Weigh 1 g of pulverized Coix Seed, add 10 mL of methanol, heat on a water bath for 10 minutes, and filter. Evaporate the filtrate to make 2 mL, and use this solution as the test solution. Perform the test with the test solution as directed under the Thin-layer Chromatography. Spot 5 μL of the test solution on the plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of petroleum ether, ethylacetate, and acetic acid (10 : 3 : 0.1) to a distance of about 10 cm and air-dry the plate. Spray the iodide steam to the plate; the yellow spot shows the R_f value about 0.63

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—Proceed with Coix Seed as directed in "Coix lachryma-jobi" in [Attachment 4] MRLs for Agricultural Products in KFDA Notice "Standards and Specifications for Food."

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 14.0 % (6 hours).

Ash Not more than 3.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Condurango

Condurango Cortex

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

Description Condurango is cylindrical or semi-cylindrical pieces of bark, 4 cm to 15 cm in length and 1 mm to 6 mm in thickness. External surface is grayish brown to dark brown, nearly smooth and with numerous lenticels, or more or less scaly and rough. Inner surface is pale grayish brown and longitudinally striated. Fractured surface is fibrous on the outer region and generally granular in the inner region. 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 contains phloem fiber bundles scattered inside the starch sheath consisting of one-cellular layer. Articulate latex tubes are scattered in both cortices. Parenchyma cells contain starch grains or crystal druses of calcium oxalate.

Condurango has a slight, characteristic odor and bitter taste.

Identification Weigh l g of pulverized Condurango, add 5 mL of water, cool and filter: the clear filtrate becomes turbid on heating, but becomes clear again upon cooling.

Purity (1) ***Foreign matter***—The xylem and other foreign matter do not exceed 2.0 %.

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

Ash Not more than 12.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Condurango Fluid Extract

Method of Preparation Take medium powder of Condurango and the fluid extract as directed under Fluid Extracts using a suitable quantity of a mixture of purified water, ethanol and glycerin (5 : 3 : 2) as the first solvent and a suitable quantity of a mixture of purified water and ethanol (3 : 1) as the second solvent.

Description Condurango Fluide extract is brown liquid. Condurango Fluidextract has a characteristic odor and bitter taste.

Identification Mix 1 mL of Condurango Fluidextract with 5 mL of water, filter, if necessary, and heat the clear solution: turbidity is produced, but it becomes almost clear upon cooling.

Purity (1) ***Heavy metals***—Total heavy metals: Not more than 30 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

Containers and Storage ***Containers*—**Tight containers.

Coptis Rhizome

Coptidis Rhizoma

Coptis Rhizome is the rhizome of *Coptis japonica* Makino, *Coptis chinensis* Franchet, *Coptis deltoidea* C. Y. Cheng et Hsiao or *Coptis teeta* Wallich (Ranunculaceae), from which the roots have been removed practically.
Coptis Rhizome contains not less than 4.2 % of berberine [as berberine chloride ($C_{20}H_{18}ClNO_4$: 371.81)], calculated on the dried basis.

Description Coptis Rhizome is irregular, cylindrical rhizome, 2 cm to 4 cm, rarely up to 10 cm in length and 2 mm to 7 mm in diameter, slightly curved and often branched. External surface is grayish yellow-brown, with ring nodes and with numerous remains of rootlets. Generally, remains of petiole are present at one end. Fractured surface is rather fibrous. Cork layer is pale grayish brown, cortex is yellowish brown to reddish-yellowish brown, xylem is yellow to red-yellow, and pith is yellow-brown. 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 wood 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.
Coptis Rhizome has a slight, characteristic odor and extremely bitter and lasting taste. Coptis Rhizome colors the saliva yellow on chewing.

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: a red-purple color develops.

(2) Weigh 0.5 g of pulverized Coptis Rhizome, add 20 mL of methanol, shake for 2 minutes, filter and use the filtrate as the test solution. Separately, weigh l mg of Berberine Chloride RS, dissolve in l mL of methanol and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 5 μL each of the test solution and the standard solution on a plate of silica gel with a fluorescent indicator for thin-layer chromatography. Develop the chromatogram with a mixture of *n*-butanol, water and acetic anhydride (7 : 2 : 1) to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 365 nm): one spot among the spots from the test solution and a yellow to yellow-green fluorescent spot from the standard solution show the same color and the same R_f value.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 11.0 % (105 °C, 6 hours).

Ash Not more than 4.0 %.

Acid-insoluble Ash Not more than 1.0 %

Assay Weigh accurately about 0.5 g of pulverized Coptis Rhizome, add 30 mL of a mixture of methanol and dilute hydrochloric acid (100 : 1), heat with a reflux condenser for 30 minutes and filter. Repeat the above procedure twice with the residue, using 30 mL and 20 mL volumes of a mixture of methanol and dilute hydrochloric acid (100 : 1). To the last residue, add 10 mL of methanol, shake well and filter. Combine all 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), dissolve 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 the Liquid Chromatography according to the following operating conditions. Determine the peak areas, A_T and A_S, of berberine for the test solution and the standard solution, respectively.

$$\text{Amount (mg) of berberine [berberine chloride } (C_{20}H_{18}ClNO_4)] = \text{amount (mg) of Berberine Chloride RS} \times \frac{A_T}{A_S}$$

Operating conditions
Detector: An ultraviolet absorption photometer (wavelength: 345 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 (5 μm to 10 μm in particle diameter).
Column temperature: A constant temperature of about 40 °C.
Mobile phase: Dissolve 3.4 g of monobasic potassium phosphate and 1.7 g of sodium lauryl sulfate in l000 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 Chloride RS and Palmatine RS in 10 mL of methanol. When the procedure is run with 20 μL of this solution under the above operating conditions, palmatine and berberine are eluted in this order and clearly dividing each peak.

System repeatability: When the test is repeated 6 times with 20 μL each of the standard solution under the above operating conditions, the relative deviation of the peak area of berberine is not more than 1.5 %.

Containers and Storage ***Containers*****—**Well-closed containers.

Cornus Fruit

Corni Fructus

Cornus Fruit is the ripe fruit of *Cornus officinalis* Siebold et Zuccarini (Cornaceae), from which the seeds have been removed. Cornus Fruit, when dried, contains not less than 1.2 % in total of loganin ($C_{17}H_{26}O_{10}$: 390.38) and morroniside (C17H26O11: 406.38).

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. External surface is dark red-purple to dark purple, lustrous and with coarse wrinkles. A scar is formed by removal of true fruit. A scar of calyx is present at upper part and a scar of fruit stalk at base. The texture is soft.
Cornus Fruit 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, filter and use these solutions as the test solution and Cornus Fruit RMPM standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 μL each of the test solution and Cornus Fruit RMPM standard solution on a 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) to a distance of about 10 cm and air-dry the plate. Examine 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 fruit stalk and other foreign matter contained in Cornus Fruit is less than 2.0 %.

(2) ***Heavy metals*****—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*****—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Methoxychlor: Not more than 1 ppm.

(iv) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(v) Aldrin: Not more than 0.01 ppm.

(vi) Endrin: Not more than 0.01 ppm.

(vii) Myclobutanil: Not more than 2.0 ppm.

(vii) Triforine: Not more than 0.2 ppm.

(ix) Triflumizole: Not more than 0.2 ppm.

(x) Hexaconazole: Not more than 0.3 ppm.

(4) ***Sulfur dioxide*****—**Not more than 30 ppm.

Ash Not more than 5.0 %.

Assay Weigh accurately about 2 g of pulverized Cornus Fruit, add 100 mL of methanol, warm 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. Collect all of the filtrates, vacuum-concentrate, 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 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 operating 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/minute.

System suitability

System performance: When the procedure is run with 10 μL of the standard solution under the above operating conditions, morroniside and loganin are elut-

ed in this order.

System repeatability: When the test is repeated 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 not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Corydalis Tuber

Corydalis Tuber

Corydalis Tuber is the tuber of *Corydalis ternata* Nakai or *Corydalis yanhusuo* W.T.Wang (Papaveraceae). Corydalis Tuber from *Corydalis ternata* contains not less than 0.03 % of coptisine ($C_{19}H_{14}NO_4$: 320.32) and not less than 0.02 % of berberine [berberine chloride ($C_{20}H_{18}ClNO_4$: 371.81)], and Corydalis Tuber from *Corydalis yanhusuo* contains not less than 0.03 % of coptisine ($C_{19}H_{14}NO_4$: 320.32) and not less than 0.05 % of tetrahydropalmatine ($C_{21}H_{25}NO_4$: 355.43).

Description (1) ***Corydalis ternata*—**Corydalis Tuber from *Corydalis ternata* is nearly flattened spherical or polygonal tuber, l cm to 2 cm in diameter, with stem scar at one end and with several warty protrusion at the base. External surface is grayish yellow to grayish brown. Texture is hard. Fractured surface is smooth or granular and yellow to grayish yellow-brown.
Corydalis Tuber from *Corydalis ternat* is almost odorless and has a bitter taste.

(2) ***Corydalis yanhusuo***—Corydalis Tuber from *Corydalis yanhusuo* is the tuber, mostly uneven, flattened globose, 0.5 cm to 1.5 cm in diameter. The external surface is yellow to yellow-brown, the texture is hard and crunchy with irregular reticular wrinkles and the cut surface is yellow and horny with a wax-like luster.
Corydalis Tuber from *Corydalis yanhusuo* is 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, cool and filter. To 5 mL of the filtrate, add 3 drops of Meyer's TS: a yellow-brown cotton-shaped precipitate is produced.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 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 each. 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*. 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*. 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}, A_{Tb} and A_{Tc}, of coptisine, berberine chloride and tetrahydropalmatine in the test solution and the peak areas, A_{Sa}, A_{Sb} and A_{Sc}, of coptisine, berberine and tetrahydropalmatine in the standard solution.

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

$$\text{Amount (mg) of berberine } (C_{20}H_{18}NO_4) = \text{amount (mg) of Berberine RS} \times \frac{A_{Tb}}{A_{Sb}} \times \frac{1}{40}$$

$$\text{Amount (mg) of tetrahydropalmatine } (C_{21}H_{25}NO_4) = \text{amount (mg) of Tetrahydropalmatine RS} \times \frac{A_{Tc}}{A_{Sc}} \times \frac{1}{20}$$

Operating conditions

Detector: An ultraviolet absorption photomter (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 octadecylsilanized 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 step or concentration gradient by mixing mobile phases A and B as directed in the following table.

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

Flow rate: 1.0 mL/minute

System suitability

System performance: When the procedure is run with 10 μL of the standard solution under the above operating conditions, coptisine, berberine and tetrahydropal-matine are eluted in this order with the resolution between their peaks being not less than 3.

System repeatability: When the test is repeated 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 not more than 1.5 %.

Containers and Storage ***Containers*****—**Well-closed containers.

Croton Seed

Crotonis Semen

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

Description Croton Seed is the ovate seed with three edges, 18 mm to 22 mm in length and 14 mm to 20 mm in diameter. The external surface is grayish yellow or slightly deeper and coarse. Six lines run longitudinally and the apex is cut flat. When the pericarp is craked, it reveals three loculi containing one seed each. The seed is slightly flattened elliptic, 1.2 mm to 1.5 mm in length and 0.7 mm to 0.9 mm in diameter. The external surface of the seed is brown or grayish brown with the hilum and caruncle or caruncle scar appearing as small spots at one end. The seed coat is thin and hard but brittle. The seed kernel is yellowish white and very oily. Croton Seed is nearly odorless and tastes oily at first and 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. Perform the test with this solution as directed under Thin-layer Chromatography. Spot 10 μL of the test solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of petroleum ether, ethyl acetate and formic acid (10 : 1 : 0.5) 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: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Mycotoxins***—Total aflatoxin (sum of aflatoxins B_1, B_2, G_1 and G_2): Not more than 15 ppb (aflatoxin B_1 is not more than 10.0 ppb).

Ash Not more than 3.5 %.

Extract Content ***Dilute ethanol–soluble extract***—Not less than 18.0 %.

Containers and Storage ***Containers*****—**Well-closed containers.

Crude Licorice Extract

Crude Licorice Extract contains not less than 6.0 % of glycyrrhiza acid ($C_{42}H_{62}O_{16}$: 822.94).

Method of Preparation Boil coarse powder of Licorice with Water or Purified Water, filter the solution under pressure and evaporate the filtrate.

Description Crude Licorice Extract is lustrous, dark yellow-red to blackish brown plates, rods, or masses. Crude Licorice Extract dissolves in water with turbidity. Crude Licorice Extract is brittle when colded, the fractured surface is dark yellow-red, lustrous like shell. Crude Licorice Extract soften when warmed.
Crude Licorice Extract has a characteristic odor and sweet taste.

Identification Weigh 0.6 g of Crude Licorice Extract add 10 mL of a mixture of ethanol and water (7 : 3), dissolve by warming if necessary, cool, centrifuge and use the supernatant liquid as the test solution. Proceed as directed in the Identification under Licorice.

Purity (1) ***Insoluble substances***—Boil 5.0 g of pul-

verized Crude Licorice Extract with 100 mL of water. After cooling, filter the mixture through tared filter paper, wash with water and dry the residue at 105 °C for 5 hours: the residue is not more than 1.25 g.

(2) ***Starch***—Weigh about 1 g of pulverized Crude Licorice Extract, add water to make 20 mL, shake the mixture thoroughly and filter. Examine the soluble substance on filter paper under a microscope: the residue contains no starch grains.

(3) ***Foreign matter***—The filtrate obtained in (1) does dot have strong bitter taste.

(4) ***Heavy metals***—Total heavy metals: Not more than 30 ppm.

(5) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

Ash Not more than 12.0 % (1 g, proceed as directed in the Ash under Test for Herbal Drugs).

Assay Weigh accurately about 0.15 g of Crude Licorice Extract and proceed as directed in the Assay under Licorice Extract.

$$\text{Amount (mg) of glycyrrhizic acid } (C_{42}H_{62}O_{16}) = \text{amount (mg) of Glycyrrhizic Acid RS, calculated on the anhydrous basis} \times \frac{A_T}{A_S}$$

Containers and Storage ***Containers***—Well-closed containers.

Curcuma Longa Rhizome

Curcumae Longae Rhizoma

Curcuma Longa Rhizome is the rhizome, boiled or steamed thoroughly, and dried, of *Curcuma longa* Linné (Zingiberaceae).

Curcuma Longa Rhizome contains not less than 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 in length and 1 cm to 3 cm in diameter. Secondary rhizome is cylindrical with two obtuse ends, slightly curved, about 1 cm in diameter and 2 cm to 6 cm in length, with ring-nodes. The rhizome with cork layer is yellowish red and lustrous. The rhizome without cork layer is dark yellowish red and powdery. The texture is hard and difficult to break and the fractured surface is horny and lustrous like wax. Under a magnifying glass, endodermis ring is distinct and parenchyma is star-like scattered. Under a microscope, the outermost layer usually consists of cortex layer with 4 to 10 layers or a part of cortex. One layer of endodermis divides cortex and stele. The cortex and stele consist of parenchyma tissues and vascular bundles scattered. Oil cells are scattered in parenchyma tissue. Yellow substances, sand crystals or solitary crystals of calcium oxalate and gelatinized starch grains are observed in the parenchyma cells.

Curcuma Longa Rhizoma has a characteristic odor and bitter and irritating taste and the color of saliva becomes 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 purplish red 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 saturated boric acid solution and boil: reddish orange color develops. Add 1 drop of ammonia TS: the color develops as dark blue-black immediately, gradually changes into brown color and returns to reddish orange color 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, filter, vacuum-concentrate, dissolve in 2 mL of methanol and use these solutions as the test solution and the Curcuma Longa Rhizome RMPM standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 μL each of the test solution and the Curcuma Longa Rhizome RMPM standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of dichloromethane, methanol and formic acid (94:4:0.7) to a distance of about 10 cm and air-dry the plate. Examine 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 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: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide*—**Not more than 30 ppm.

Ash Not more than 7.0 %.

Acid-insoluble Ash Not more than 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 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}, 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 solution.

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

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

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

Operating conditions
Detector: An ultraviolet absorption photometer (wavelength: 420 nm)
Column: A stainless steel column 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilanized 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 diluted acetic acid (2 in 100) (65:35)
Flow rate: 1.0 mL/min
System suitability
System performance: When the procedure is run with 10 μL of the standard solution under the above operating conditions, bis-demethoxy curcumin, demethoxy curcumin and curcumin are eluted in this order.
System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution, the relative standard deviation of the peak area of each of bis-demethoxy curcumin, demethoxy curcumin and curcumin is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Curcuma Root

Curcumae Radix

Curcuma Root is the dried steamed root tuber, or the root tuber from which the periderm has been removed, 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).

Description (1) ***Curcuma wenyujin***—Curcuma Root from *Curcuma wenyujin* is ellipsoidal and ovoid root tuber, 35 mm to 70 mm in length, 12 mm to 25 mm in diameter. External surface is grayish brown, with uneven longitudinal and pale brown wrinkles. Texture is hard and a transverse section is grayish brown with horn. Endodermis ring is distinct.
Curcuma Root from *Curcuma wenyujin* has a characteristic odor and slight bitter taste.
(2) ***Curcuma longa***—Curcuma Root from *Curcuma longa* is pyramidal root tuber, 25 mm to 45 mm in length, 10 mm to 15 mm in diameter, one end thin or long. External surface is grayish brown, or grayish yellow with longitudinal winkles. A transverse section is orange in center, and yellowish brown to reddish brown in outer surface.
Curcuma Root from *Curcuma longa* has a characteristic odor and very pungent taste.
(3) ***Curcuma kwangsiensis***—Curcuma Root from *Curcuma kwangsiensis* is long conical, or long globular root tuber, 20 mm to 65 mm in length, 10 mm to 18 mm in diameter. External surface is shallow longitudinal, or coarse reticular wrinkled.
Curcuma Root from *Curcuma kwangsiensis* has a characteristic odor and slight pungent and bitter taste.
(4) ***Curcuma phaeocaulis***—Curcuma Root from *Curcuma phaeocaulis* is long ellipsoidal root tuber, 15 mm to 35 mm in length, 10 mm to 12 mm in diameter, slightly thick and big.
Curcuma Root from *Curcuma phaeocaulis* has a characteristic odor and weak taste

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

(4) ***Mycotoxins***—Total aflatoxin (sum of aflatoxins B_1, B_2, G_1 and G_2): Not more than 15.0 ppb (aflatoxin B_1 is not more than 10.0 ppb).

Loss on Drying Not more than 16.0 %.

Ash Not more than 9.0 %

Containers and Storage ***Containers*—**Well-closed containers.

Cynomorium Herb

Cynomorii Herba

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

Description Cynomorium Herb is the stem, flat cylindrical, slightly curved, 5 cm to 20 cm in length and 2 cm to 5 cm in diameter. The external surface is 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 hard and uneasily broken. The fractured surface is pale brown to maroon. The vascular bundles are yellow and appear triangular.
Cynomorium Herb has a slight aroma and slightly bitter and astringent taste.

Identification (1) Weigh 1 g of pulverized Cynomorium Herb, add 10 mL of water, allow to stand for 30 minutes, filter and use the filtrate as the test solution. Separately, dissolve 2 mg of L-Proline RS in 1 mL of water and use this solution as the standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 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 propanol, water, acetic acid (100), ethanol and water (4:2:1:1) to a distance of about 10 cm and air-dry the plate. Spray evenly 2 % ninhydrin-ethanol solution and heat at 105 °C: the test solution shows a purple spot at the R_f value of 0.5. One of the spots obtained from the test solution shows the same color and R_f value as the spot from the standard solution.

(2) Weigh 1 g of pulverized Cynomorium Herb, add 20 mL of ethyl acetate, sonicate for 30 minutes, filter, concentrate the filtrate to make 1 mL and use as the test solution. Separately, dissolve 0.5 mg of Ursolic Acid RS in 1 mL of methanol and use this solution as the standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 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, ethyl acetate and formic acid (15:5:0.5) to a distance of about 10 cm and air-dry the plate. Spray evenly sulfuric acid TS for spraying and heat 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***—Cynomorium Herb contains less than 5.0 % of flower stalk and the other foreign matters.

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide*—**Not more than 30 ppm.

Loss on Drying not more than 10.0 %

Ash not more than 8.0 %

Extract Content ***Dilute ethanol–soluble extract*—** Not less than 25.0 %

Containers and Storage ***Containers*—**Well-closed containers.

Cyperus Rhizome

Cyperi Rhizoma

Cyperus Rhizome is the rhizome of *Cyperus rotundus* Linné (Cyperaceae), from which rootlets have been 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 is 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 that are 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 yellow-brown or red-brown and horny. The cut surface of those that have been dried is white and distinctly powdery. The endodermis has distinct ring patterns and the stele has a relatively intense color and is scattered with spot-like vascular bundles.
Cyperus Rhizome has a characteristic aroma and slightly bitter taste.

Identification Weigh 1 g each of pulverized Cyperus Rhizome and Cyperus Rhizome RMPM, add 5 mL of ether, shake for 1 hour and filter, respectively. Eavaporate the filterates to dryness. Dissolve each of the residues to 0.5 mL of ethyl acetate and use these solution as the test solution and the standard solution of Cyperus Rhizome RMPM. Perform the test with the test solution and the standard solution of Cyperus Rhizome RMPM 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 plate with fluorescent indicator for thin-layer chromatography. Deve1op the plate with a mixture of hexane, ethyl acetate and formic acid (14 : 4 : 0.5) to a distance of about 10 cm and air-dry the plate. Examine the plate under ultraviolet light (wavelength: 254 nm). The spots obtained from the test solution and one of the spots from the Cyperus Rhizome RMPM standard solution shows a blackish brown spot at the R_f value of about 0.7. Spray evenly dilute sulfuric acid TS on the plate and heat at 105 °C: the spots from the test solution show the same color and R_f value as the spots from the Cyperus Rhizome RMPM standard solution.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): Not more than 0.2 ppm.
(vi) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 3.0 %.

Acid-insoluble Ash Not more than 1.5 %.

Essential Oil Content Not less than 0.3 mL (50.0 g, 1 mL of silicon resin).

Containers and Storage ***Containers*—**Well-closed containers.

Dictamnus Root Bark

Dictamni Cortex

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

Description Dictamnus Root Bark is the root bark, cylindrical, 5 cm to 15 cm in length, 1 cm to 2 cm in diameter and 2 mm to 5 mm in thickness. External surface is grayish white or grayish yellow, longitudinally wrinkled, with rootlet scars, frequently and small protruding granular dots. Inner surface is almost pale yellow. Texture is weak and easy to be broken and powdery. Fracture is uneven, somewhat lamellar, and when outer layer is peeled off, numerous glittering small spots are observed on exposing to light.
Under a microscope, the transverse section reveals the remaining cork layers consisting of about 3 to 10 rows of flat rectangular or elliptic cork cells. The cortex is narrow, consisting of elliptic parenchyma cells, which usually contain oil drops. There is a large, transversely long intracellular space. The phloem is broad, taking up most of the entirety, the cells are contracted circular or close to circular, sparsely arranged, with large torn gaps. The phloem rays are curved and consist of 1 to 3 rows of cells. Fibers are mostly scattered individually in the cortex and phloem, polygonal or rectangular in shape, with very thick cell walls forming the shape of stone cells. The parenchyma cells contain small starch grains, calcium oxalate druses and oil drops.
Dictamnus Root Bark has a characteristic odor and slightly bitter taste.

Identification Weigh 0.5 g of pulverized Dictamnus Root Bark, add 10 mL of diuted acetic acid, heat in a water bath for 3 minutes and filter. Add 1 to 2 drops of Mayer TS to 5 mL of the filtrate: a yellowish white precipitate is produced.

Purity (1) ***Foreign matter***—Dictamnus Root Bark contains not more than 5.0 % of the xylem tissue and other foreign matter.
(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(4) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 12.0 %.

Ash Not more than 8.0 %.

Acid-insoluble Ash Not more than 1.0 %.

Extract Content ***Dilute ethanol-soluble extract—*** Not less than 14.0 %.

Containers and Storage ***Containers—***Well-closed containers.

Dioscorea Rhizome

Dioscoreae Rhizoma

Dioscorea Rhizome is the rhizome or the dried steamed rhizome (rhizophore) of *Dioscorea japonica* Thunberg or *Dioscorea baratas* Decaisne (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 external surface is white to pale yellow, longitudinally grooved, longitudinally wrinkled with root hair scars or pale brown bark remaining. The body is heavy and the texture is solid and difficult to cut. The cut surface is white, powdery, sometimes horny. Under a microscope, the transverse section reveals nearly circular mucous cells in the basic tissue, containing calcium oxalate raphides. The vascular bundles are collateral, surrounded by a row of parenchymatous vascular bundles. Resin canals are distributed between parenchyma cells and are filled with brown resinous substances. There are many starch grains. The starch grains are close to circular, long circular or triangular ovoid, with distinct striae visible in large ones.
Dioscorea Rhizome is nearly 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 and add 0.5 mL of sulfuric acid carefully to make two layers: a very pale red to red-brown color appears at the zone of contact and blue-green to green color at upper layer.

(2) Weigh 0.5 g of pulverized Dioscorea Rhizome, add 10 mL of water, heat 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, heat under a reflux condensor for 30 minutes, filter and evaporate on a water-bath to dryness. To the each residue, add 2 mL of ethanol and use each solution as the test solution and Dioscorea Rhizome RMPM standard solution. Perform the test with the test solution and Dioscorea Rhizome RMPM standard solution as directed under the Thin-layer Chromatography. Spot 10 μL each of the test solution and Dioscorea Rhizome RMPM standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of cyclohexane and ethyl acetate (3 : 1) to a distance of about 10 cm and air-dry the plate. Spray evenly diluted sulfuric acid TS and heat at 105 °C for 10 minutes: the spots from the test solution and the spots from Dioscorea Rhizome RMPM standard solution show the same colors and the same R_f values

Purity (1) ***Heavy metals—***(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—Proceed with Dioscorea Rhizome as directed in "*Dioscorea baratas* Decaisne" in [Attachment 4] MRLs for Agricultural Products in KFDA Notice "Standards and Specifications for Food."
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 14.0 % (6 hours).

Ash Not more than 6.0 %.

Containers and Storage ***Containers—***Well-closed containers.

Dolichos Seed

Dolichoris Semen

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

Description Dolichos Seed is the seed, flattend-ellipsoidal or flatten-ovoid, 8 mm to 12 mm in length, 6 mm to 9 mm in diameter, and 4 mm to 7 mm in thickness. External surface is yellowish white, smooth, lustrous, with a white, prominent, eyebrow-shaped caruncle at the edge of one side. Texture is hard. Testa is thin and brittle, with 2 plump, yellowish white cotyledons inside.
Dolichos Seed has a characteristic odor and taste is weak and bean-like on chewing.

Identification Weigh 1 g of pulverized Dolichos Seed, add 10 mL of diluted ethanol (7 in 10) and warm in 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. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 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 butanol, acetic acid and water (3:1:1) to a distance of about 10 cm and air-dry the plate. Spray evenly ninhydrin-ethanol solution

(2 in 100) 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: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—Proceed with Dolichos Seed as directed in "Other Legumes" in [Attachment 4] MRLs for Agricultural Products in KFDA Notice "Standards and Specifications for Food."
(3) ***Sulfur dioxide***—Not more than 30 ppm.
(4) ***Mycotoxins***—Total aflatoxin (sum of aflatoxins B_1, B_2, G_1 and G_2): Not more than 15.0 ppb (aflatoxin B_1 is not more than 10.0 ppb).

Loss on Drying Not more than 12.0 %.

Ash Not more than 5.0 %.

Acid-insoluble Ash Not more than 1.0 %.

Extract Content ***Water-soluble extract***—Not less than 14.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Drynaria Rhizome

Drynaria Rhizoma

Drynaria Rhizome is the rhizome of *Drynaria foutunei* J. Smith (Polyodiaceae), from which the scaly pieces (ramenta) have been burned off.

Description Drynaria Rhizome is the rhizome, flattened cylindrical, mostly curved, branched, 5 cm to 15 cm in length, 10 mm to 15 mm in width, and 2 mm to 5 mm in thickness. External surface is closely covered with deep brown to dark brown hairy ramenta, and is soft like flocky hair. The burnt one is reddish brown or dark brown, the upper surface and both side are evenly marked by raised of depressed circular frond scar, rarely by remains of frond-bases and fibrous roots. Texture is light, fragile and easily broken. Fracture is reddish brown, with vascular bundles yellow dotted and arranged in a ring.
Drynaria Rhizome has a slight, characteristic odor and weak and slight astringent taste.

Identification Weigh 0.5 g of pulverized Drynaria Rhizome, add 30 mL of methanol, sonicate, filter and evaporate the filtrate. To the residue, add 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. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 5 μL each of the test solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with the upper layer of the mixture of ethyl acetate, methanol, acetic acid and water (9:1:1:0.2) to a distance of about 12 cm and air-dry the plate. Spray with 1 % Aluminium chloride TS in ethanol on the plate and examine under ultraviolet light at (main wavelength: 365 nm): a spot 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: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Containers and Storage ***Containers*—**Well-closed containers.

Ephedra Herb

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, when dried, contains not less than 0.7 % of total alkaloids [as ephedrine ($C_{10}H_{15}NO$: 165.23) and as pseudoephedrine ($C_{10}H_{15}NO$: 165.23)].

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 of internode. Ephedra Herb is pale green to yellowish green. Numerous parallel vertical furrows are on the surface. Scaly leaves are at the node volume. 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 appears as circle and ellipse, the outer volume grayish green to yellow-green and the center filled with a red-purple substance or hollow. Around the fractured surface is fibrous and easily split vertically.
Ephedra Herb has a slight odor and astringent and

slightly bitter taste and paralyzes slightly sensation on the tongue.

Identification Weigh 0.5 g of pulverized Ephedra Herb, add 10 mL of methanol, shake for 2 minutes, filter and use the filtrate as the test solution. Perform the test with the test solution as directed under the Thin-layer Chromatography. Spot 10 μL of the test solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of *n*-butanol, water and acetic acid (100) (7 : 2 : 1) to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with 2 % ninhydrin-ethanol solution and heat at 105 °C for 10 minutes: spot of the R_f value about 0.35 is red- purple.

Purity (1) ***Foreign matter***—Woody stem: Less than 5.0 %.

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 11.0 %.

Acid-insoluble Ash Not more than 2.0 %.

Assay Weigh accurately about 0.5 g of pulverized Ephedra Herb, previously dried in a desiccator (silica gel) for 24 hours, in a glass-stoppered centrifuge tube, add 20 mL of diluted methanol (1 in 2), shake for 30 minutes, centrifuge and separate the supernatant liquid. Repeat this procedure twice with the residue using 20 mL volumes of diluted methanol (l in 2). 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 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 2.0 mL of the 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 operating conditions. Determine the peak areas, A_{Ta} and A_{Tb}, of ephedrine and pseudoephedrine (the relative retention time to ephedrine is about 0.9), respectively, in the test solution and the peak area, A_S, of ephedrine in the standard solution.

$$\text{Amount (mg) of total alkaloids} = \text{amount (mg) of Ephedrine Hydrochloride RS} \times \frac{A_{Ta} + A_{Tb}}{A_S} \times \frac{1}{10} \times 0.819$$

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 °C.

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 l mg of Ephedrine Hydrochloride RS and 4 mg of Atropine Sulfate RS, dissolve each in diluted methanol (l in 2) to make 100 mL. When the procedure is run with 10 μL of this solution under the above operating conditions, ephedrine and atropine are eluted in this order, clearly dividing each peak.

System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions, the relative standard deviation of the peak area of ephedrine is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Epimedium Herb

Epimedii Herba

Epimedium Herb is the aerial part of *Epimedium koreanum* Nakai, *Epimedium brevicornum* Maximowicz, *Epimedium pubescens* Maximowicz, *Epimedium wushanense* T. S. Ying, or *Epimedium sagittatum* Maximowicz. Epimedium Herb (Berberidaceae). Epimedium Herb contains not less than 0.3 % of icariin ($C_{33}H_{40}O_{15}$: 676.66), calculated on the basis of the dried material.

Description (1) ***Epimedium koreanum***—Epimedium Herb from *Epimedium koreanum* is the aerial part, composed of a stem and biternate compound leaf. The stem is thin and long, cylindrical, 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 middle and upper part has white pith and the external surface is brown or yellowish brown. 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 is yellow-brown and needle-like serrated. The external surface is deep green or yellowish green, smooth and lustrous. The back surface is grayish green, leaf veins protruding, sparsely pilose with soft yellowish brown hairs and the hairs of the middle vein relatively dense. The leaf is thin and the texture is paper-like.

(2) ***Epimedium brevicornum***—Epimedium Herb from *Epimedium brevicornum* is composed of a biternate compound leaf, similar to Epimedium Herb from *Epimedium koreanum* but the texture of the leaf is close to leathery.

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

(4) ***Epimedium wushanense***—Epimedium Herb from *Epimedium wushanense* is composed of a ternate compound leaf, the leaflets are lanceolate to narrow lanceolate, 9 cm to 23 cm in length, 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 a ternate compound leaf, the leaflets are 4 cm to 12 cm in length, 2.5 cm to 5 cm in width, gradually acute towards the apex, the base of the leaflets on both sides 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.

Epimedium Herb has a slight, characteristic odor and 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, weigh1 mg of Icariin RS, add in 1 mL of methanol and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 10 μL each of the test solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of ethylacetate, ethanol, and water (8 : 2 : 1) to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): one spot among the 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: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 12.0 % (6 hours).

Ash Not more than 8.0 %.

Acid-insoluble Ash Not more than 1.0 %.

Extract Content ***Dilute ethanol-soluble extract*—** Not less than 15.0 %.

Assay Weigh accurately about 1 g of pulverized Epimedium Herb, add 50 mL of diluted ethanol (7 in 10), heat 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 filtrate, 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, 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 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.

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

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 acetonitrile and water (72:28)

Flow rate: 1.0 mL/min.

System suitability

System repeatability: When the test is repeated six times with 10 μL each of the standard solution under the above operating conditions: the relative standard deviation of the peak area of icariin is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Eribotrya Leaf

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. Apex is acute, margin is sparsely serrate and entire is near base. Upper surface is graeyish brown, yellowish brown to greenigh brown, lustrous, and smooth. Lower surface is pale colored and densely yellow tomentose. Petiols is very short, with yellowish brown tomentose. Under a microscope, transverse section reveals thin cuticle, 4 to 5 layers of palisade tissue, sparsely scattered large cells without chloroplast. Midrib appears the vascular bundle collateral, curved into the xylem tissue, formig interrupted ring and pericycle fibre bundles arranged in phloem. Upper part of small vescular bundle has lignified tissue, surrounded by solitary crystal of calium oxalate. Mesophyll appears the solitary and clustered crystals. Tomentum is unicellular, curved, 25 μm in thickness and about 1.5 mm in length.
Eriobotrya Leaf is nearly 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 and filter. Add 0.5 mL of lead subacetate TS to 2 mL of the filtrate: pale yellowish brown precipitation is produced.

(2) Weigh 0.3 g of pulverized Eriobotrya Leaf, add 10 mL of methanol, heat for 5 minutes in a water-bath, cool, filter and use the filtrate as the test solution. Separately, weigh 5 mg of Ursolic Acid RS, dissolve it in 5 mL of methanol and use this solution as standard solution. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 10 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) to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with sulfuric acid TS for spray and heat at 105 °C for 10 minutes: one of the spots from the test solution and the spot from the standard solution are the same color and the R_f value.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 15.0 % (6 hours).

Ash Not more than 10.0 %.

Extract Content ***Dilute ethanol-soluble extract*—** Not less than 15.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Eucommia Bark

Eucommiae Cortex

Eucommia Bark is the stem bark of *Eucommia ulmoides* Oliver (Eucommiaceae), from which periderm is removed. Eucommia Bark contains not less than 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 curved inwards, varying in length and width and 3 mm to 7 mm in thickness. The external surface is pale brown or grayish brown, sometimes with distinct wrinkles or longitudinal cracks, sometimes relatively flat. Those with coarse bark remaining have distinct lenticels. The inner surface is smooth, brown or dark brown, with fine wrinkeld longitudinally. Inner texture is fragile and easily broken. Fracture is linked up by fine, dense, silvery white and elastic rubber threads. Under a microscope, the outermost layer reveals a thick rhytidome. Several layers of cork cells are regularly arranged inside the rhytidome. The cell walls of these cells are lignified and located underneath is the phelloderm. The phloem takes up most of the area with stone cell rings in a transverse arrangement of 5 to 7 rows, each ring with 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, including white gutta percha, can be observed near the pith. These parenchyma cells are particularly numerous inside the phloem.
Eucommia Bark has charateristic odor and slightly bitter taste.

Identification Weigh 1 g each of pulverized Eucommia Bark and Eucommia Bark RMPM, add 10 mL of methanol, sonicate for 60 minutes, filter and use the filtrates as the test solution and the Eucommia Bark RMPM standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 μL each of the test solution and the Eucommia

Bark RMPM standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of chloroform, methanol and water (10:5:1) 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 spots obtained from the test solution show the same color and Rf value as the spots from the Eucommia Bark RMPM standard solution and of these, a dark brown spot appears at each of the R_f values of 0.55 and 0.7.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 10.0 %.

Ash Not more than 8.0 %.

Acid-insoluble Ash Not more than 6.0 %.

Extract Content ***Dilute ethanol-soluble extract*—** Not less than 9.0 %.

Assay Weigh accurately about 1.0 g of pulverized Eucommia Bark, add 20 mL of diluted ethanol (75 in 100), sonicate for 30 minutes, filter and use the filtrate as the test solution. Separately, weigh accurately about 10 mg of Pinoresinol Diglucoside RS and dissolve in 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 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 pinoresinol diglucoside ($C_{32}H_{42}O_{16}$)
= Amount (mg) of Pinoresinol Diglucoside RS
$$\times \frac{A_T}{A_S} \times \frac{1}{25}$$

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 octadecylsilanized silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).
Column temperature: A constant temperature of about 35 °C
Mobile phase: Control the step or concentration gradient by mixing mobile phases A and B as directed in the following table.
Mobile phase A: Diluted formic acid (1 in 1000)
Mobile phase B: Acetonitrile

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

Flow rate: 1.0 mL/minute

Containers and Storage ***Containers*—**Preserve in well-closed containers.

Euryale Seed

Euryales Semen

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

Description Euryale Seed is the seed, close to spherical, 5 mm to 8 mm in diameter. It sometimes breaks to form small masses. The unbroken Euryale Seed has an external surface (inner seed coat) in the form of a thin membrane, closely adhered to the endosperm, red-brown or dark purple in color, sometimes with an 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.
Euryale Seed is odorless and has week taste.

Purity (1) ***Foreign matter***—The amount of cortex and other foreign matter contained in Euryale Seed is not more than 2.0 %.
(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(4) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 14.0 %.

Ash Not more than 2.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Evodia Fruit

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). Evodia fruit, when dried, contains not less than 0.1 % of a mixture of the contents of evodiamine ($C_{19}H_{21}N_3O$: 307.39) and rutecarpin ($C_{18}H_{13}N_3O$: 287.32).

Description Evodia Fruit is flattened, spheroidal or slightly pentagonal, flattened spheroidal fruit, 2.5 mm to 5 mm in diameter. The external surface is dark brown to grayish brown, with many oil sacs appearing as hollow pits. Sometimes it has a peduncle, 2 mm to 5 mm in length, covered densely with hairs. Matured pericarp is split to reveal five loculi and each loculus containing seeds. Seeds are obovoid or globular, brown to blackish brown or bluish black and lustrous. Under a microscope, a transverse section reveals hard hairs of epidermis of epicarp.
Evodia Fruit has a characteristic odor and purgent taste followed by lasting bitterness 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 tests using the filtrate as the test solution.

(i) Spot one drop of the test solution on a filter paper, air-dry, spray Dragendorff's TS for spraying and allow to stand: a yellow-red color develops.

(ii) Take 0.2 mL of the test solution and add 0.8 mL of dilute acetic acid. To this solution, add gently 2 mL of 4-dimethylaminobenzaldehyde TS and warm on a water-bath: a purple-brown ring develops at the zone of contact.

(2) Weigh 1 g of pulverized Evodia Fruit, add 20 mL of methanol, heat for 5 minutes in water-bath, filter after cooling and use the filtrate as the test solution. Seperately, weigh 1 mg of evodiamine RS and 1 mg of rutecarpin RS, dissolve in 1 mL of methanol and use these solutions as the standard solution (1) and the standard solution (2), respectively. Perform the test with the test solution 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 with a fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of hexane and ethyl acetate (3 : 2) to a distance of about 10 cm and air-dry the plate. Spray evenly dilute sulfuric acid TS on the plate and examine under ultraviolet light (main wavelength: 365 nm): two spot among the several spots from the test solution and the spot from the standard solution (1) and the standard solution (2) show the same color and the same R_f value.

Purity (1) ***Foreign matter*—**(i) Peduncle: Less than 5.0 %.

(ii) Other foreign matter: The amount of foreign matter other than peduncles contained in Evodia Fruit is not more than 1.0 %.

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Methoxychlor: Not more than 1 ppm.

(iv) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(v) Aldrin: Not more than 0.01 ppm.

(vi) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): Not more than 0.2 ppm.

(vii) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 8.0 %.

Assay Weigh accurately about 0.5 g of the fine powder of Evodia Fruit, add 25 mL of methanol, sonicate for 1 hour and filter. To 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 rutecarpin RS, dissolve to make exactly 50 mL of methanol. Take exactly 2 mL each of these solutions, add exactly 20 mL of methanol and use this solution as the standard solution. Pipet 10 μL each 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 the test solution and, A_{Sa} and A_{Sb}, of the standard solution, respectively.

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

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

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 acetonitrile and water (50:50).

Flow rate: 1.0 mL/min.

System suitability

System performance: When the procedure is run with 10 μL of the standard solution under the above operating conditions, evodiamine and rutecarpin are eluted in this order with and clearly dividing each peak.

System repeatability: When the test is repeated six times with 10 μL each of the standard solution under the above operating conditions: the relative standard deviation of the peak area of evodiamine and rutecarpin is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Farfarae Flower

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 mm to 25 mm in length and 5 mm to 10 mm in diameter. The upper part is broader and the lower part is gradually slender or has short stems. On the outer surface, numerous scaly bracts are attached. The outer side of bracts is purple-red or pale red, and the inner 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 calyx, and thickend cell wall in subsequently strung beads.

Farfarae Flower is aromatic and taste is slightly bitter and pungent.

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

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 8.0 %.

Ash Not more than 5.0 %.

Acid-insoluble Ash Not more than 1.5 %.

Extract Content ***Dilute ethanol-soluble extract***—Not less than 18.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Fennel

Foeniculi Fructus

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

Description Fennel is cylindrical cremocarp fruit, 3 mm to 8 mm in length, 1 mm to 3 mm in width and occasionally with 2 mm to 10 mm of a fruit stalk. External surface is grayish yellow-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 ventral side.

Fennel has a characteristic odor and sweet taste at first, followed by bitterness.

Identification Weigh 0.5 g each of pulverized Fennel and Fennel RMPM, add 10 mL of hexane, shake well, allow to stand for 5 minutes, filter and use the filtrates as the test solution and the standard solution of Fennel RMPM. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 5 μL each of the test solution and the standard solution of Fennel RMPM on a plate of silica gel with a fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of hexane and ethyl acetate (20 : l) to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): the several spots from the test solution and the spots from the standard solution of Fennel RMPM show the same color and the same R_f value.

Purity (1) ***Foreign matter*—**(i) Fruit stalk: The amount of fruit stalk is not more than 3.0 %.

(ii) Other foreign matter: The amount of foreign matter other than the fruit stalk is not more than 1.0 %.

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

(5) ***Estragole***—To the essential oil as obtained from the Essential Oil Content, add xylene to make 5.0 mL and use this solution as the test solution. Separately, dissove 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 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: the peak area of estragole ($C_{10}H_{12}O$: 148.20) in the essential oil of Fennel is not more than 10.0 %.

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

Operating conditions
Detector: A hydrogen flame ionization detector
Column: A capillary column about 0.3 mm in internal diameter and 30 m to 60 m in length, coated with polyethylene glycol 20M for gas chromatography.
Column temperature: Maintain at 60 °C for 4 minutes then raise the temperature to 170 °C over 22 minutes and maintain at 170 °C for 15 minutes.
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/minute

Ash Not more than 10.0 %.

Acid-insoluble Ash Not more than 1.5 %.

Essential Oil Content Not less than 0.7 mL (50.0 g).

Containers and Storage ***Containers—***Well-closed containers.

Forsythia Fruit

Forsythiae Fructus

Forsythia Fruit is the fruit of *Forsythia virdissima* 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 from *Forsythia virdissima* contains not less than 0.4 % of arctigenin ($C_{21}H_{24}O_6$: 372.42) and Forsythia Fruit from *Forsythia suspensa* contains not less than 0.25 % of forsythiaside A ($C_{29}H_{36}O_{15}$:624.59).

Description (1) ***Forsythia virdissima***—Forsythia Fruit from *Forsythia virdissima* is the fruit, close to ovate, slightly broad, flat, 10 mm to 17 mm in length and 5 mm to 12 mm in diameter. The tip is very acute and open like a bird's beak. The lower part is slightly round, with the peduncle remaining or fallen off. The external surface is brown or green, slightly bulging and unevenly wrinkled.
Forsythia Fruit from *Forsythia virdissima* has a slight, characteristic odor and bitter taste.

(2) ***Forsythia suspensa***—Forsythia Fruit from *Forsythia suspense* 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 external surface has irregular longitudinal wrinkles and several small, protruding spots. Each side has 1 distinct longitudinal furrow. The apex is dull and acute 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 to stand for 2 minutes and filter. To l mL of the filtrate, add gently 0.5 mL of sulfuric acid to form two layers: a red-purple color develops at the zone of contact.

(2) Weigh l g of pulverized Forsythia Fruit, add l0 mL of methanol, warm in a water-bath for 2 minutes and filter. To 5 mL of the filtrate, add 0.1 g of magnesium and l mL of hydrochloric acid and allow to stand: a pale red to yellow-red color develops.

Purity (1) ***Foreign matter—***(i) Branchlet: Less than 5.0 %.
(ii) Other foreign matter: The amount of foreign matter other than branchlets contained in Forsythia Fruit is not more than 1.0 %.

(2) ***Heavy metals—***(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides—***(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide—***Not more than 30 ppm.

Ash Not more than 5.0 %.

Extract Content ***Dilute ethanol-soluble extract*—** Not less than 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, filter and use the filtrate as the test solution. Separately, weigh accurately about 10 mg of Arctigenin 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 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.

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

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 octadecylsilanized 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 diluted acetic acid (100) (3 in 1000) and methanol (55:45)

Flow rate: 1.0 mL/minute

(2) ***Forsythiaside A*—**Weigh accurately about 0.1 g of pulverized Forsythia Fruit, add 50 mL of diluted methanol (1 in 2), sonicate for 30 minutes, filter and use the filtrate as the test solution. Separately, weigh accurately about 10 mg of Forsythiaside A 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 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.

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

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 octadecylsilanized silica gel for liquid chromatography (5 μm to 10 μm particle diameter).

Column temperature: A constant temperature of about 25 °C

Mobile phase: A mixture of diluted acetic acid (100) (3 in 1000) and methanol (55:45)

Flow rate: 1.0 mL/minute

Containers and Storage ***Containers*—**Well-closed containers.

Fritillaria Bulb

Fritillariae Cirrhosae Bulbus

Fritillaria Bulb is the 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 by figures, Songpae or Cheongpae.

Description (1) ***Songpae*—**Songpae is conical to nearly globose bulb, 3 mm to 8 mm in height, 3 mm to 8 mm in diameter. External surface is white. Externally the outer scale leaves are two, varying considerably in size, with the large scale closely embracing the small one, the uncovered part appearing crescent. The apex is closed, with cylindrical and slightly tapering buds and 1 to 2 scales inside. The base is globular, even and slightly pointed with a grayish brown disk at central part. Occasionally the remains of fibrous roots are found. Texture is hard and fragile, and the fractured surface is white and starchy.

Songpae is nearly odorless and has a slightly pungent taste.

(2) ***Cheongpae*—**Cheongpae is slightly oblate bulb, 4 mm to 11 mm in height and 4 mm to 16 mm in diameter. Outer scale leaves are two, almost uniform in size, embraced. The apex is open with buds, 1 to 2 small scales inside and slender cylindrical remains of a stem.

Identification (1) Weigh 0.5 g of Fritillaria Bulb, add 5 mL of acetic anhydride, shake for 5 minutes and filter. Add 1 mL of sulfuric acid carefully to 2 mL of the filtrate: a red color develops at the zone of contact. Let stand for a while: the upper layer shows green.

(2) Weigh 0.5 g of Fritillaria Bulb, add 10 mL of dilute acetic acid, shake for 5 minutes and filter. Add 1 to 2 droplets of Mayer TS carefully to 2 mL of the filtrate: the solution becomes turbid. Let stand for a while: a white precipitate is produced.

(3) Weigh 2 g of Fritillaria Bulb, add 20 mL of methanol, warm for 5 minutes on a water-bath with occasional shaking, cool and filter. Evaporate all the filtrate to dryness, add 3 mL of dilute hydrochloride to the residual, warm for 2 minutes in a water bath, cool and filter. Drop one drop of this filtrate on the filter paper, air-dry and spray Dragendorff's TS: a yellowish red color is produced.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5

ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.

Loss on Drying Not more than 15.0 %.

Ash Not more than 5.0 %.

Extract Content ***Dilute ethanol-soluble extract***— Not less than 9.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Fritillaria Thunbergii Bulb

Fritillariae Thunbergii Bulbus

Fritillaria Thunbergii Bulb is the bulb of *Fritillaria thunbergii* Miquel (Liliaceae) and other species of the same genus. The larger one is removed from the central bud and commonly known as Daepae, the smaller one, with the central bud not removed, is commonly known as Jupae. The bulb with central bud removed, regardless of the size, cut into thick slice freshly is called Jeolpaepyeon.

Description (1) ***Daepae*—**Daepae is bulb and the outer single scale leaf of a bulb, almost in crescent shape, 1 cm to 2 cm in height and 20 mm to 35 mm in diameter, outer surface is milky white to pale yellow, inner surface is white or pale brown, covered with white powder. Texture is hard and brittle, fracture surface is white to yellowish white, highly starchy.
Daipae has a slight, characteristic odor and a slightly bitter taste.
(2) ***Jupae*—**Jupae is whole bulb, and oblate, 10 mm to 15 mm in height and 10 mm to 25 mm in diameter. Outer surface is milky white, the outer scale leaves 2, plump and fleshy, almost in reniform holding to each other, containing 2 to 3 small scale leaves and dried shrunken stem remains.
Jupae has a slight, characteristic odor and a slightly bitter taste.
(3) ***Jeolpaepyeon*—**Jeolpaepyeon is bulb, and slices cut from the outer single scale leaf of a bulb, elliptical or subrounded, 1 cm to 2 cm in diameter. The surface of edge is pale yellow. Texture is fragile and easy to cut. The fractured surface is powdery-white, highly starchy.
Jeolpaepyeo has a slight, characteristic odor and a slightly bitter taste.

Identification (1) Weigh 0.5 g of Fritillaria Thunbergii Bulb, add 5 mL of acetic anhydride, shake for 5 minutes and filter. Add 1 mL of sulfuric acid carefully to 2 mL of the filtrate: a red color develops at the zone of contact. Let stand for a while: the upper layer shows green.
(2) Weigh 0.5 g of Fritillaria Thunbergii Bulb, add 10 mL of dilute acetic anhydride, shake for 5 minutes and filter. Add 1 drop of Mayer TS carefully to 2 mL of the filtrate: the solution becomes turbid. Let stand for a while: a white precipitate is produced.
(3) Weigh 2 g of Fritillaria Thunbergii Bulb, add 20 mL of methanol, warm for 5 minutes on a water-bath with occasional shaking, cool and filter. Evaporate all the filtrate, add 3 mL of dilute hydrochloride to the residual, warm for 2 minutes in a water-bath, cool and filter. Drop one drop of this filtrate on the filter paper, air-dry and spray Dragendorff's TS: a yellow-red color is produced.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): Not more than 0.2 ppm.
(vi) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 15.0 %.

Ash Not more than 5.0 %.

Extract Content ***Dilute ethanol-soluble extract***— Not less than 9.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Gambir

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

Description Gambir is the aqueous extract from the leaves and young twigs, in masses of irregular shapes. The external surface is brown to dark brown and the

inside is pale brown. The texture is brittle and easily broken.
Gambir has a slight, characteristic odor and extremely astringent and bitter taste.

Identification (1) Weigh 0.2 g of pulverized Gambir, add 10 mL of water, warm in a water-bath for 5 minutes with occasional shaking and filter. Cool the filtrate and add 2 to 3 drops of gelatin TS: a white turbidity or precipitate is produced.

(2) Weigh 0.1 g of pulverized Gambir, dissolve with 20 mL of dilute ethanol for 2 minutes and filter. Mix l mL of the filtrate with 9 mL of dilute ethanol and to this solution, add 1 mL of vanillin-hydrochloric acid TS: a pale red to red-brown color develops.

Ash Not more than 6.0 %.

Acid-insoluble Ash Not more than 1.5 %.

Extract Content ***Dilute ethanol-soluble extract—*** Not less than 70.0 %.

Containers and Storage ***Containers—***Well-closed containers.

Gamisoyosan Extract Granules

Gamisoyosan Extract Granules contains not less than 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 herbal drugs to coarse powder, weigh each herbal drugs, put into the extractor, add eight to ten fold of water, extract for 2 to 3 hours at 80 to 100 °C and filter. Vacuum-concentrate the filtrate under 60 °C until it becomes 1.64 g to 2.45 g of Viscous extract or concentrate in a suitable method until it becomes 0.91 g to 1.36 g of Dry extract. Gamisoyosan Extract Granules is prepared 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, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 pulverized Angelica Gagas Root, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 toluene, ether, water and acetic acid (500 : 500 : 5 : 2) 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 spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(2) ***Atractylodes Rhizome White—***Pulverize Gamiso-yosan Extract Granuels, weigh an amount, equivalent to 1 g of Atractylodes Rhizome White, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 pulverized Atractylodes Rhizome White, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 acetone (7 : 1) to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with the solution made by dissolving 5 g of 4-dimethylamino-benzaldehyde in 100 mL of dilute sulfuric acid and heat at 105 °C for 10 minutes: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(3) ***Poria—***Pulverize Gamisoyosan Extract Granules, weigh an amount, equivalent to 1 g of Poria, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 pulverized Poria, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of hexane and acetone (7 : 3) 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 °C for 10 minutes. Examine under ultraviolet light (main wavelength: 254 nm): one spot among the spots from the test solution and a spot from the standard solution

show the same color and the same R_f value.

(4) ***Bupleurum Root*—**Pulverize Gamisoyosan Extract Granules, weigh an amount, equivalent to 1 g of Bupleurum Root, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 pulverized Bupleurum Root RMPM, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 chloroform, methanol and water (30 : 10 : 1) to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with sulfuric acid TS for spray and heat at 105 °C for 10 minutes: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(5) ***Peony Root*—**Pulverize Gamisoyosan Extract Granules, weigh an amount, equivalent to 1 g of Peony Root, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 pulverized Peony Root, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 lower layer of the mixture of chloroform, methanol and water (26 : 14 : 5) 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 °C for 10 minutes: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(6) ***Licorice*—**Pulverize Gamisoyosan Extract Granules, weigh an amount, equivalent to 1 g of Licorice, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 pulverized Licorice, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 chloroform and methanol (95 : 5) 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 °C for 10 minutes: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(7) ***Moutan Root Bark*—**Pulverize Gamisoyosan Extract Granules, weigh an amount, equivalent to 1 g of Moutan Root Bark, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 pulverized Moutan Root Bark, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 ethyl formate, chloroform, toluene and formic acid (6 : 6 : 5 : 3) 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 °C for 10 minutes: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(8) ***Gardenia Fruit*—**Pulverize Gamisoyosan Extract Granules, weigh an amount, equivalent to 1 g of Gardenia Fruit, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 pulverized Gardenia Fruit RMPM, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 chloroform and methanol (3: 1) 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 °C for 10 minutes: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(9) ***Ginger*—**Pulverize Gamisoyosan Extract Granules, weigh an amount, equivalent to 1 g of Ginger, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 pulverized Ginger, add 100 mL of methanol, heat with a reflux condenser for 1

hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 (85 : 15) 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 °C for 10 minutes: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(10) ***Mentha Herb*—**Pulverize Gamisoyosan Extract Granules, weigh an amount, equivalent to 1 g of Mentha Herb, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 pulverized Mentha Herb, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 ethyl acetate-acetone (10 : 3) 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 °C for 10 minutes: one spot among the 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) Total heavy metals: Not more than 30 ppm

(ii) Lead: Not more than 5 ppm

(iii) Arsenic: Not more than 3 ppm

Disintegration Test It meets the requirement.

Particle Size Distribution Test for Preparations It meets the requirement.

Uniformity of Dosage Units (divided) It meets the requirement.

Microbial Limit It meets the requirement.

Assay (1) ***Total paeoniflorin of Peony Root and Moutan Root Bark*—**Take not less than about 20 sachets of Gamisoyosan Extract Granules, weigh accurately and pulverize. Weigh accurately equivalent to about 10 mg of paeoniflorin, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat 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 filtrate, Vacuum-concentrate the filtrate 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. Pipet 20 μL each 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_T and A_S, of the test solution and the standard solution, respectively

$$\text{Amount (mg) of paeoniflorin } (C_{23}H_{28}O_{11}) = \text{amount (mg) of Paeoniflorin RS, calculated on the anhydrous basis} \times \frac{A_T}{A_S}$$

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 methanol and water (60:40)

Flow rate: 1.0 mL/min

(2) ***Glycyrrhizic Acid of Licorice*—**Take not less than about 20 sachets of Gamisoyosan Extract Granules, weigh accurately and pulverize. Weigh accurately equivalent to about 10 mg of glycyrrhizic acid, add 50 mL of water, heat with a reflux condenser in a water bath for 3 hour, add 50 mL of 3 mol/mL sulfuric acid TS and hydrolyze in a water bath for 1 hour. After cooling, add 50 mL of chloroform, warm with a reflux condenser for 30 minutes. After cooling, take the chloroform latyer in separatory funnel, add 30 mL of chloroform, extract three times repetitively and combine chloroform layers and filter through anhydrous sodium sulfurate. Vacuum-concentrate the filtrate. To the residue, add methanol to make exactly 50 mL and use the solution as the standard solution. Separately, weigh accurately about 10 mg of Glycyrrhizic acid RS (previously dried in a silica gel desiccator for 24 hours), prepare the solution, prepared in the same manner as the test solution, and use this solution as the standard solution. Pipet 10 μL each 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_T and A_S, of the test solution and the standard solution, respectively.

$$\text{Amount (mg) of glycyrrhizic } (C_{42}H_{62}O_{16}) = \text{amount (mg) of Glycyrrhizic acid RS, calculated on the anhydrous basis} \times \frac{A_T}{A_S}$$

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 in particle diameter).

Mobile phase: A mixture of methanol, water and acetic acid (100) (78:19:3)

Flow rate: 1.0 mL/min

(3) ***Geniposide of Gardenia Fruit***—Take not less than about 20 sachets of Gamisoyosan Extract Granules, weigh accurately and pulverize. Weigh accurately equivalent to about 10 mg of geniposide, add 10 mL of water, shake for 5 minutes, add 100 mL of water, heat 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 filtrates, 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 Geniposide RS (previously dried in a silica gel desiccator for 24 hours), add methanol to make exactly 50 mL and use the solution as the standard solution. Pipet 20 μL each 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_T and A_S, of the test solution and the standard solution, respectively

$$\text{Amount (mg) of geniposide } (C_{17}H_{24}O_{10}) = \text{amount (mg) of Geniposide RS, calculated on the anhydrous basis} \times \frac{A_T}{A_S}$$

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 to10 μm in particle diameter).

Mobile phase: A mixture of water, methanol and acetic acid (100) (95:15:1)

Flow rate: 1.0 mL/min

Containers and Storage ***Containers***—Tight containers.

Gardenia Fruit

Gardeniae Fructus

Gardenia Fruit is the well ripe fruit or the fruit passed through hot water of *Gardenia jasminoides* Ellis (Rubiaceae). Gardenia Fruit, when dried, contains not less than 3.0 % of geniposide ($C_{17}H_{24}O_{10}$: 388.37) and not less than 1.8 % of gardenoside ($C_{17}H_{24}O_{11}$: 404.37).

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 is yellow-brown to red-brown, usually with 5 to 7 distinct wing-shaped longitudinal ridges. The calyx or its scar is present at the top and the lower part is slightly acute and sometimes has the fruit stalk remaining. The pericarp is thin and easily broken. 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 yellow-red with a dense arrangement of thin, small strumae on the external surface.

Gardenia Fruit 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 between 60 °C and 70 °C 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 resulting solution is yellow and is not lighter than that of the following control solution.

Control solution—Dissolve 2.0 mg of potassium bichromate in water to make exactly l0 mL.

(2) Weigh 1.0 g each of pulverized Gardenia Fruit and Gardenia Fruit RMPM, add 20 mL of methanol, warm for 3 minutes on a water-bath, cool, filter and use the filtrates as the test solution and the standard solution of Gardenia Fruit RMPM, respectively. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 μL each of the test solution and the Gardenia Fruit RMPM standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate and methanol (3 : 1) 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 l0 minutes: The several spots from the test solution and the spots from the standard solution of Gardenia Fruit RMPM show the same color and the same R_f value. Among the spots from the test solution, the spot of geniposide can be observed at the R_f value of about 0.5.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not

more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 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, filter and use the filtrate as the test solution. Separately, weigh accurately about 1.0 mg each of Geniposide RS and Gardenoside 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 Liquid Chromatography according to the following operating conditions and determine the peak areas, A_{Ta} and A_{Tb}, of geniposide and gardenoside in the test solution and the peak areas, A_{Sa} and A_{Sb}, of geniposide and gardenoside in the standard solution.

$$\text{Amount (mg) of geniposide } (C_{17}H_{24}O_{10}) = \text{amount (mg) of Geniposide RS} \times \frac{A_{Ta}}{A_{Sa}} \times 50$$

$$\text{Amount (mg) of gardenoside } (C_{17}H_{24}O_{11}) = \text{amount (mg) of Geniposide RS} \times \frac{A_{Tb}}{A_{Sb}} \times 50$$

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 octadecylsilaznied silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 35 °C

Mobile phase: Control the step or concentration gradient by mixing mobile phases A and B 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 (%)	Mobile phase B (%)
0	90	10
8	85	15
35	85	15
40	80	20
45	0	100

Flow rate: 0.6 mL/minute

Containers and Storage ***Containers***—Well-closed containers.

Gastrodia Rhizome

Gastrodiae Rhizoma

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

Description Gastrodia Rhizome is the rhizome, slightly curved and flattened cylindrical to fusiform, 3 cm to 15 cm in length, 1.5 cm to 5 cm in width and 0.5 cm to 2 cm in thickness. The external surface is pale yellowish white to yellow-brown, with irregular longitudinal wrinkles and several turns of transverse rings from the latent bud, sometimes with maroon root-shaped hyphae. The top part has red-brown to dark 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, yellowish white to pale brown and horny. Under a microscope, a transverse section reveals epidermal tissue sometimes remaining on the outermost layer, pale brown in color. The cortex has transversely long cells, the cell wall of the outermost 1 to several rows of cells slightly thickened, pits rarely observed. The stele has relatively large parenchyma cells, nearly orbicular or polygonal, sometimes with pitted patterns observed. The vascular bundles are collateral or amphicribral, scattered, vessels forming groups of 2 or more, polylgonal in shape. The parenchyma cells have polysaccharide masses and sometimes contain calcium crystal raphide bundles.
Gastrodia Rhizome has a slight, characteristic odor and 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; a red-purple color develops.

(2) Weigh 0.5 g each of pulverized Gastrodia Rhizoma and Gastrodia Rhizoma RMPM, add 5 mL of diluted methanol (7 in 10), warm on a water bath under a reflux condenser for 1 hour and filter, respectively. Use the filterates as the test solution and the standard solution of Gastrodia Rhizoma RMPM. Perform the test with the test solution and the standard solution of Gastrodia Rhizoma RMPM as directed under the Thin-layer Chromatography. Spot 5 μL each of the test solution and the standard solution of Gastrodia Rhizoma RMPM on a plate of silica gel for thin-layer chromatography. Deve1op the plate with a mixture of dichloromethane, methanol and water (7 : 2.5 : 0.25) to a distance of about 10 cm and air-dry the plate. Spray dilute sulfuric acid TS to the plate, heat the plate at 105 °C for 10 minutes. The spots from the test solution and

the spots from the standard solution of Gastrodia Rhizoma RMPM show the same color and the same R_f value.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 13.0 %.

Ash Not more than 6.0 %.

Extract Content ***Dilute ethanol-soluble extract***—Not less than 17.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Gentian

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 external surface is dark brown with lateral roots and sometimes longitudinally divided. The rhizome is short with fine transverse wrinkles and sometimes with buds and remains of leaves at the upper edge. The root is longitudinally and deeply wrinkled and slightly twisted. The fractured surface is flat and yellow-brown, and the cortex and xylum is dark brown at the cambium. Under a microscope, a transverse section of the root reveals several layers of collenchyma adjoined internally to 4 to 6 layers of thin-walled cork. The secondary cortex of the parenchyma is with irregularly distributed phloem. The xylem consists chiefly of parenchyma with individual or clustered vessels and tracheids. A small number of sieve tubes are present in xylem. The parenchyma cells of the cortex and xylem contain oil droplets and minute needle crystals of calcium oxalate. Starch grains are very rare. Gentian has a characteristic odor and sweet at first, later persistently bitter taste.

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

(2) Weigh 0.5 g of pulverized Gentian, add 10 mL of methanol, shake for 5 minutes and filter and use the filtrate as the test solution. Perform the test with the test solution as directed under the Thin-layer Chromatography. Separately, dissolve 1 mg of gentiopicroside RS in 10 mL of methanol and use this solution as the standard solution. Spot 10 μL each of the test solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, anhydrous ethanol and water (8 : 2 : 1) to a distance of about 10 cm and air-dry the plate. Examine 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: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.

Ash Not more than 6.0 %.

Acid-insoluble Ash Not more than 3.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Gentian Root and Rhizome

Gentianae Scabrae Radix et Rhizoma

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

Description Gentian Root and Rhizome is the root and rhizome. The rhizome 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 is dark grayish brown or deep brown and has 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 is pale yellow to yellow-brown with distinct many transverse wrinkles at the top and also longitudinal wrinkles. The texture is fragile and easy to cut. The cut surface is slightly even, the cortex is yellowish white or pale yellow-brown and the xylem is relatively pale in color with spot-shaped rings. Under a microscope, the transverse section reveals bark cells, close to circular or flattened circular, the outer wall is slightly thick, usually containing drops 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 sieve tube groups are scattered in the direction of the diameter. The cambium usually forms a discontinuous ring. In the xylem, 3 to 10 vessels form groups and sometimes form 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 contain drops of fatty oil and needle crystals and prismatic crystals of calcium oxalate. Starch grains are not apparent.
Gentian Root and Rhizome has a slight, characteristic odor and extremely bitter and lasting taste.

Identification Weigh 0.5 g of pulverized Gentian Root and Rhizome, add 10 mL of methanol, shake for 20 minutes, filter, and use the filtrate as the test solution. Perform the test with 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 of the test solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, dehydrated ethanol, and water (8 : 2 : 1) to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): one spot among the several spots form the test solution and a dark purple spot from the standard solution show the same color and the some R_f value.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 7.0 %.

Acid-insoluble Ash Not more than 3.0 %

Containers and Storage ***Containers*—**Well-closed containers.

Geranium Herb

Geranii Herba

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

Description Geranium Herb is the aerial part of stem with leaves opposite. Stem is slender and long, green-brown. Stem and leaf are covered with soft hairs. Leaf is divided palmately into 3 to 5 lobes and 2 cm to 4 cm in length, grayish yellow-green to grayish brown. Each lobe is oblong to obovate and its upper margin is crenate.
Geranium Herb is nearly odorless and has an astringent taste.

Identification Weigh 0.1 g of Geranium Herb, add 10 mL of water, boil, filter and to the filtrate, add l drop of iron (III) chloride TS: a blackish blue color develops.

Purity (1) ***Foreign matter***—The amount of the root and other foreign matter contained in Geranium Herb is not more than 2.0 %.
(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): Not more than 0.2 ppm.
(vi) Endrin: Not more than 0.01 ppm.

Ash Not more than 10.0 %.

Acid-insoluble Ash Not more than 1.5 %.

Extract Content ***Dilute ethanol-soluble extract*—** Not less than 15.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Ginger

Zingiberis Rhizoma

Ginger is the dried rhizome of *Zingiber officinale* Roscoe (Zingiberacece). Ginger, when dried, contains not less than 0.4 % of 6-gingerol ($C_{17}H_{26}O_4$: 294.39).

Description Ginger consists of rhizome and is in the shape of flat, irregular masses with finger-shaped branches. Ginger is 2 cm to 4 cm in length and 1 cm to 2 cm in diameter. The external surface is grayish white to pale gray-brown with white powder and with or without the pale gray-yellow periderm. The branched parts are slightly compressed and slightly curved ovoid or oblong-ovoid, sometimes with attached buds at both ends, swelled warty. The texture is solid. The cut surface is slightly fibrous, powdery and yellowish white or grayish white. Under a magnifying glass, the transverse section reveals a distinct ring pattern in the endodermis with scattered vascular bundles and yellow oil spots. Under a microscope, the transverse section of Ginger reveals a cork layer consisting of several rows of flat cork cells. The cortex is scattered with several leaf vascular bundles with parenchyma cells visible throughout. The endodermis is distinct and shows a casparian strip. The stele takes up most of the underground stems and is scattered with collateral vascular bundles. Vascular bundles near the stele are small and relatively densely arranged. Unlignified fiber and parenchyma cells are present inside and around the xylem. The parenchyma contains starch grains.
Ginger has a characteristic odor and extremely pungent taste.

Identification Weigh 2 g of pulverized Ginger, add 5 mL of acetone, shake for 3 minutes, filter and use the filtrate as the test solution. Separately, dissolve 1 mg of 6-Gingerol RS in l mL of acetone and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 10 μL 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, acetone and acetic acid (100) (10 : 7 : 1) to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with 2,4-dinitrophenylhydrazine TS and heat at 105 °C 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: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—Proceed with Ginger as directed in "Ginger" in [Attachment 4] MRLs for Agricultural Products in KFDA Notice "Standards and Specifications for Food."
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 8.0 %.

Assay Weigh accurately about 2.0 g of pulverized Ginger, add 60 mL of methanol, heat with a reflux condensor 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 10 mg of 6-Gingerol RS, dissolve in methanol to make exactly 100 mL and use this solution as the standard solution. Pipet 10 μL each of the test 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_T and A_S, of 6-gingerol of 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: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions, the relative standard deviation of the peak area of 6-gingerol is not more than 1.5 %.

Containers and Storage ***Containers***—Well-closed containers.

Ginkgo Leaf

Ginkgo Folium

Ginkgo Leaf is the leaf of *Ginkgo biloba* Linné (Ginkgoaceae). Ginkgo Leaf contains not less than 0.5 % of total flavonoids, calculated on the basis of the dried material.

Description Ginkgo Leaf is the leaf, mostly crinkled

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

Identification Weigh 0.5 g of pulverized Ginkgo leaf, add 10 mL of ethanol, warm and extract and filter. Add a small amount of magnesium and 1 drop of hydrogen chloride to 1 mL of the filtrate: a red color develops.

Purity (1) ***Foreign matter*—**not more than 5.0 % of stems and not more than 2.0 % of other foreign matter.
(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.

Loss on Drying not more than 11.0 % (1.0 g, 100 °C to 105 °C, 2 hours)

Ash not more than 11.0 %

Assay Weigh accurately about 2.5 g of pulverized Ginkgo Leaf, add 50 mL of diluted acetone (3 in 5), heat with a reflux condenser for 30 minutes and filter. To the residue, add 40 mL of diluted acetone (3 in 5) and proced in the same manner. Combine the filtrates and add diluted acetone (3 in 5) to make exactly 100 mL. Evaporate 50 ml of the solution to eliminate the acetone, rinsing with 30 ml of methanol. Add 4.4 ml of hydrochloric acid, dilute to 50 ml with water and centrifuge. Pipette 10 ml of the supernatant liquid in a 10 ml brown-glass vial. Stopper and heat on a water bath for 25 minutes. Allow to cool to room temperature and use this solution as the test solution. Separately, weigh accurately 10.0 mg of Quercetin Dihydrate RS, and dissolve in 20 ml of methanol. Add 15 ml of dilute hydrochloric acid and 5 ml of water and dilute to 50.0 ml with methanol. Use this solution as the standard solution. Pipet 10 μL each 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}, A_{Tb}, and A_{Tc}, of quercetin, kaempferol (the relative retention time to quercetin is about 1.4) and isoramnetin (the relative retention time to quercetin is about 1.5), respectively, in the test solution and the peak area, A_S, of quercetin in 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 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 °C .
Mobile phase: Control the mobile phase A and B stepwise or gradient as the following conditions.
Mobile phase A: dissolve 0.3 of phosphoric acid to 1000 mL of water and adjust with phosphoric acid to make a pH of 2.0.
Mobile phase B: methanol

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

Flow rate: 1.0 mL/min.
System suitability
System performance: The retention time of quercetin adjust at about 12.5 minutes. The resolution between the peaks, of kaempferol and of isoramnetin, is not less than 1.5

Containers and Storage ***Containers*—**Well-closed containers.

Ginseng

Ginseng Radix

Ginseng is the root of *Panax ginseng* C. A. Meyer (Araliaceae), from which rootlets and cork layer has been removed. Ginseng contains not less than 0.10 % of ginsenoside Rg_1 ($C_{42}H_{72}O_{14}$: 801.01) and not less than 0.20 % of ginsenoside Rb_1 ($C_{54}H_{92}O_{23}$: 1109.29), calculated on the dried basis.

Description Ginseng is thin and long cylindrical roots, often branching 2 to 5 lateral roots from the middle. Ginseng is 5 cm to 20 cm in length, main root,

5 mm to 30 mm in diameter. External surface is pale yellow-brown to pale grayish brown, with longitudinal wrinkles and scars of rootlets, sometimes with curved crown and with short remains of rhizome. Fractured surface is practically flat, light yellow-brown, and brown in the neighborhood of the cambium. Under a microscope, a transverse section reveals thin-walled parenchyma cell containing filled with starch grains, and cortex is scattered secret vessels filled with yellow to yellow-red secretion. Aggregate crystal of calcium oxalate is observed in parenchyma cell of phloem.
Ginseng has a characteristic odor and taste, at first slightly sweet, followed by a slight bitterness.

Identification (1) On a section of Ginseng, add dilute iodine TS drop-wise: a dark blue color is produced on the surface.

(2) Weigh 2g of pulverized Ginseng, add 20 mL of methanol, heat under a reflux condenser in a water-bath for 15 minutes, cool, filter, and use the filtrate as the test solution. Separately, weigh l mg of Ginsenoside Rg_1 RS, add l mL of methanol, and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 10 μL each of the test solution and the standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with the lower layer of a mixture of ethyl acetate, methanol, and water (14:5:4) 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 10 minutes: one of the spots 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 Ginseng is not more than 2.0 %.

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—Proceed with Ginseng as directed in "Dried Ginseng" in [Attachment 5] MRLs for Ginseng in KFDA Notice "Standards and Specifications for Food."

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 15.0 % (6 hours)

Ash Not more than 5 %.

Extract Content ***Dilute ethanol-soluble extract*—** Not less than 14.0 %.

Assay (1) ***Ginsenoside Rg_1***—Weigh accurately about 1.0 g of pulverized Ginseng, put in a glass-stoppered centrifuge tube, add 30 mL of diluted methanol (3 in 5), shake for 15 minutes, centrifuge, and separate the supernatant liquid. Repeat the procedure with the residue using 15 mL of diluted methanol (3 in 5), combine the supernatant liquids, and add diluted methanol (3 in 5) to make exactly 50mL. Pipet 10mL 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 and diluted methanol (3 in 5) to make exactly 20 mL, and use this solution as the sample solution. Separately, weigh accurately about 10 mg of Ginsenoside Rg_1 RS (previously dried in a silica gel desiccator for 24 hours), dissolve in diluted methanol (3 in 5) to make exactly 100mL, and use this solution as the standard solution. Perform the test with exactly 10 mL each of the sample solution and standard solution as directed under Liquid Chromatography according to the following conditions, and determine the peak areas, A_T and A_S, of ginsenoside Rg_1.

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

Operating conditions

Detector: An ultraviolet absorption photometer (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 °C.

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. When the procedure is run with 10 mL 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 not less than 1.5.

System repeatability: When the test is repeated 6 times with 10 mL each of the standard solution under the above operating conditions, the relative standard deviation of the peak area of ginsenoside Rg1 is not more than 1.5 %.

(2) ***Ginsenoside Rb_1***—Use the sample solution obtained in (1) as the sample solution. Separately, weigh accurately about 10 mg of ginsenoside Rb_1 RS (previously dried in a silica gel desiccator for 24 hours) dissolve in diluted methanol (3 in 5) to make exactly 100 mL, and use this solution as the standard solution. Perform the test with exactly 10 mL each of the sample solution and standard solution as directed under Liquid Chromatography according to the following conditions, and determine the peak areas, A_T and A_S, of ginsenoside Rb_1

$$\text{Amount (mg) of ginsenoide } Rb_1 \ (C_{42}H_{72}O_{14}) = \text{amount (mg) of Ginsenoide } Rb_1 \text{ RS} \times \frac{A_T}{A_S}$$

Operating conditions

Detector: An ultraviolet absorption photometer (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 °C.

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. When the procedure is run with 10 mL 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 not less than 3.

System repeatability: When the test is repeated 6 times with 10 mL each of the standard solution under the above operating conditions, the relative standard deviation of the peak area of ginsenoside Rb_1 is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Gleditsia Spine

Gleditsiae Spina

Gleditsia Spine is the thorn of *Gleditsia japonica* Miquel var. *koraiensis* Nakai 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. Main spines are flattened long conical or conical, 3 cm to 15 cm in length or longer, 3 mm to 10 mm in width. Branched small spines are 1 cm to 6 cm in length, acute at apex. The external surface is purple-brown to deep brown. The body is light and the texture is hard and uneasily broken. Fractured surface is 1 mm to 3 mm in thickness, usually tapering-tipped. Xylem is yellowish white, pith is lax, pale red-brown, and texture is fragile, easily broken.

Gleditsia Spine from *Gleditsia japonica* is odorless and has weak taste.

(2) ***Gleditsia sinensis***—Gleditsia Spine from *Gleditsia sinensis* has more circular, hard main spines and branched small spines than those of Gleditsia Spine from *Gleditsia japonica*.

Purity (1) ***Foreign matter***—Not more than 3.0 % of stem and other foreign material.

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 10.0 %

Ash Not more than 2.0 %

Extract Content ***Dilute ethanol-soluble extract***—Not less than 10.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Glehnia Root

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 is pale yellowish white, frequently with the epidermis remaining, those with the epidermis not removed is yellow-brown on the outside. The entire root has thin longitudinal wrinkles and longitudinal furrows, yellow-brown in color, with spot-like scars of thin roots. The yellow-brown rhizome usually remains at the apex, the upper part slightly slender, the middle part slightly thick, gradually thinner towards the lower part. The texture is hard but easily fractured. The fractured surface is powdery and the cortex is pale white to pale yellow, sometimes cracked, brown secretory canals scattered as small dots. The xylem is pale yellow and the texture is dense.

Glehnia Root has a slight, characteristic odor and slightly sweet taste.

Identification Weigh 1 g each of pulverized Glehnia Root and Glehnia Root RMPM, add 10 mL of acetone, sonicate for 20 minutes and filter, respectively. Eavaporate the filterates to dryness. Dissolve each of

the residues to 1 mL of ethanol and use these solutions as the test solution and the standard solution of Glehnia Root RMPM. Perform the test with the test solution and the standard solution of of Glehnia Root RMPM as directed under the Thin-layer Chromatography. Spot 5 μL each of the test solution and the standard solution of Glehnia Root RMPM on a plate for thin-layer chromatography. Deve1op the plate with a mixture of cyclohexane and ethyl acetate (2 : 1) to a distance of about 10 cm and air-dry the plate. Spray evenly dilute sulfuric acid TS to the plate, heat the plate at 105 °C for 10 minutes. The several spots from the test solution and the spots from the standard solution of Glehnia Root RMPM show the same color and the same R_f value.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 6.0 %.

Acid-insoluble Ash Not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Hawthorn Fruit

Crataegi Fructus

Hawthorn Fruit is the ripe fruit of *Crataegus pinnatifida* Bunge and its varieties (Rosaceae).

Description Hawthorn Fruit is the fruit, circular or long circular, 1 cm to 2.5 cm in diameter. The external surface is reddish brown to dark red with sparse white, round spots. The apex has a persistent calyx, which is deeply concave with a fruit stalk 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 have fallen out, the texture is hard and long kidney-shaped, the dorsal side is roughly round with a valley and two peaks in the center.
Hawthorn Fruit 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, and filter. After removing the ether layer, the residue is dissolved in 1 mL of anhydrous acetic acid, and add 1 to 2 drops of sulfuric acid: a red- 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 it in 1 mL of ethyl acetate and use this solution as standard solution. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 4 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 ethyl acetate, hexane, methanol and formic acid (10:4:4:0.5) to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with diluted sulfuric acid TS and heat at 105 °C for 10 minutes: one of the spots from the test solution and the red-purple spot from the standard solution are the same color and the R_f value.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 6.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Hyeonggaeyeongyotang Extract Granules

Hyeonggaeyeongyotang Extract Granules contains no less than 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 (a 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 0.5 g

Pulverize the above herbal drugs to coarse powder, weigh each herbal drugs, put into the extractor, add eight to ten fold of water, extract for 2 to 3 hours at 80 ~ 100 °C and filter. Evaporate the filtrate to dryness in vacuum under 60 °C until it becomes 1.94 g to 2.75 g of Viscous extract or concentrate in a suitable method until it becomes 0.92 g to 1.30 g of Dry extract. Hyeonggaeyeongyotang Extract Granules is prepared as directed under Granules.

Indentification (1) ***Platycodon Root*—**Pulverize Hyeonggaeyeongyotang Extract Granules, weigh an amount, equivalent to 1 g of Platycodon Root, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 accurately about 1 g of pulverized Platycodon Root, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 chloroform, methanol and water (65 : 35 : 10) to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with sulfuric acid TS for spray and heat the plate at 105 °C for 10 minutes: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(2) ***Angelica Dahurica Root*—**Pulverize Hyeonggaeyeongyotang Extract Granules, weigh an amount, equivalent to 1 g of Angelica Dahurica Root, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 accurately about 1 g of pulverized Angelica Dahurica Root, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 with a fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of hexane and ethyl acetate (1 : 1) to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength : 365 nm) : one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(3) ***Bupleurum Root*—**Pulverize Hyeonggaeyeongyotang Extract Granules, weigh an amount, equivalent to 1 g of Bupleurum Root, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 accurately about 1 g of pulverized Bupleurum Root RMPM, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 chloroform, methanol and water (30 : 10 : 1) to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with sulfuric acid TS for spray and heat at 105 °C for 10 minutes: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(4) ***Licorice*—**Pulverize Hyeonggaeyeongyotang Extract Granules, weigh an amount, equivalent to 1 g of Licorice, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 accurately about 1 g of pulverized Licorice, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 chloroform and methanol (95 : 5) 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 °C for 10 minutes: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(5) ***Angelica Gigas Root*—**Pulverize Hyeonggaeyeongyotang Extract Granules, weigh an amount, equivalent to 1 g of Angelica Gigas Root, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, extract with a reflux condenser 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 accurately about 1 g of pulverized Angelica Gigas Root, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 20 μL each of the test solution and the standard solu-

tion on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of toluene, ether, water and acetic acid (500 : 500 : 5 : 2) 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 spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(6) ***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 with a reflux condenser 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 accurately about 1 g of pulverized Saposhnikovia Root, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 with a fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of hexane and ethyl acetate (1 : 1) to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 365 nm): one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(7) ***Forsythia Fruit*—**Pulverize Hyeonggaeyeongyotang Extract Granules, weigh an amount, equivalent to 1 g of Forsythia Fruit, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 accurately about 1 g of pulverized Forsythia Fruit, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 with a 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) to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 365 nm): one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(8) ***Peony Root*—**Pulverize Hyeonggaeyeongyotang Extract Granules, weigh an amount, equivalent to 1 g of Peony Root, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 accurately about 1 g of pulverized Peony Root, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 with a fluorescent indicator for thin-layer chromatography. Develop the plate with a lower layer of the mixture of chloroform, methanol and water (26 : 14 : 5) 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 °C for 10 minutes: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(9) ***Poncirus Immature Fruit Root*—**Pulverize Hyeonggaeyeongyotang Extract Granules, weigh an amount, equivalent to 1 g of Poncirus Immature Fruit Root, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 accurately about 1 g of pulverized Poncirus Immature Fruit Root RMPM, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 with a fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate and hexane (5 : 1) 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 °C for 10 minutes. Examine under ultraviolet light (main wavelength: 365 nm): one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(10) ***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 with a reflux condenser 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 accurately about 1 g of pulverized Cnidium Rhizome, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 with a fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of cyclohexane and ethyl acetate (9 : 1) to a distance of

about 10 cm and air-dry the plate. Spray evenly the plate with sulfuric acid TS for spray and heat at 105 °C for 10 minutes. Examine under ultraviolet light (main wavelength: 365 nm): one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(11) ***Gardenia Fruit***—Pulverize Hyeonggaeyeongyotang Extract Granules, weigh an amount, equivalent to 1 g of Gardenia Fruit, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 pulverized Gardenia Fruit RMPM, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 chloroform and methanol (3 : 1) 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 °C for 10 minutes: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(12) ***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 with a reflux condenser 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 accurately about 1 g of pulverized Schizonepeta Spike, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 (17 : 3) 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 °C for 10 minutes: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(13) ***Scutellaria Root***—Pulverize Hyeonggaeyeongyotang Extract Granules, weigh an amount, equivalent to 1 g of Scutellaria Root, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 accurately about 1 g of pulverized Scutellaria Root RMPM, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 with a fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of toluene, acetone and chloroform (40 : 35 : 25) to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with iron (II) chloride-methanol TS: one spot among the 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) Total heavy metals: Not more than 30 ppm.

(ii) Lead: Not more than 5 ppm.

(iii) Arsenic: Not more than 3 ppm.

Disintegration Test It meets the requirement.

Particle Size Distribution Test for Preparation It meets the requirement.

Uniformity of Dosage Units (divided) It meets the requirement.

Microbial Limit It meets the requirement.

Assay (1) ***Glycyrrhizic Acid of Licorice***—Take not less than about 20 sachets of Hyeonggaeyeongyotang Extract Granules, weigh and pulverize. Weigh accurately equivalent to about 10 mg of glycyrrhizic acid, add 50 mL of water, heat with a reflux condenser for 3 hours, add 50 mL of 3 mol/mL sulfuric acid TS and hydrolyzed in a water bath for 1 hour. After cooling, add 50 mL of chloroform, heat with a reflux condenser for 30 minutes. After cooling, take the chloroform layer in separatory funnel, add 30 mL of chloroform, extract three times repetitively, combine chloroform layers and filter through anhydrous sodium sulfurate. 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 silica gel desiccator for 24 hours), use the solution, prepared in the same manner as the test solution, as the standard solution. Pipet 10 μL each 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_T and A_S, of 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 (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*—**Take not less than about 20 sachets of Hyeonggaeyeongyotang Extract Granules, weigh and pulverize. Weigh accurately equivalent to about 10 mg of paeoniflorin, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat 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, vacuum-concentrate the filtrate 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. Afterward examine the test followed by the assay of Peony Root.

(3) ***Geniposide of Gardenia Fruit*—**Take not less than about 20 sachets of Hyeonggaeyeongyotang Extract Granules, weigh and pulverize. Weigh accurately equivalent to about 50 mg of geniposide, add 70 mL of methanol, and heat with a reflux condenser for 1 hour, cool and 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 the solution as the standard solution. Pipet 20 μL each 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_T and A_S,, of the test solution and the standard solution, respectively

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

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/min

(4) ***Baicalin of Scutellaria Root*—**Take not less than about 20 sachets of Hyeonggaeyeongyotang Extract Granules, weigh and pulverize. Weigh accurately equivalent to about 50 mg of baicalin. Afterward examine the test followed by the assay of Scutellaria Root

Containers and Storage ***Containers***—Tight containers.

Imperata Rhizome

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 thinly cylindrical, 30 cm to 60 cm in length and 2 mm to 4 mm in diameter. The external surface is 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 slightly fragile. The cut surface has a white cortex and several clefts. The stele is pale yellow with an easily removable outer cortex. It is nearly odorless and tastes slightly sweet. Under a microscope, the transverse section reveals epidermal cells in a single row, close to quadrilateral, small, 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 foliar-trace vascular bundles. The vascular bundles are closed collateral usually surrounded by clefts. The endodermal cells are thickened and contain silicon masses. Several closed collateral vascular bundles are scattered inside the stele, vascular bundle sheath fibers are arranged in a ring and lignified, and the outer vascular bundles and fibers are connected to each other, forming a ring.
Imperata Rhizome is odorless and the taste is weak at first, but slightly sweet later.

Identification (1) Weigh l.0 g of pulverized Imperata Rhizome, add 20 mL of hexane, allow the mixture to stand for 30 minutes with occasional shaking and filter. Evaporate the filtrate to dryness, dissolve the residue in 5 mL of chloroform, place 0.5 mL of this solution in a test tube and mix with 0.5 mL of acetic anhydride by shaking and add carefully 0.5 mL of sulfuric acid to make two layers: a red-brown color develops at the zone of contact 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. . Perform the test with the test solution as directed under the Thin-layer Chromatography. Spot 5 μL of the test solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of hexane and acetone (9:1) to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with diluted sulfuric acid TS and heat at 105 °C: spot of the R_f value about 0.3 is purple.

Purity (1) ***Foreign matter***—(i) Rootlet and scaly leaf: Less than 3.0 %.

(ii) Other foreign matter: The amount of foreign matter other than rootlets and scaly leaves is not more than 1.0 %.

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 5.0 %.

Acid-insoluble Ash Not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Ipecac

Ipecacuanhae Radix et Rhizoma

Ipecac is the root and rhizome of *Cephaelis ipecacuanha* A. Richard or *Cephaelis acuminate* Karsten (Rubiaceae).

Ipecac contains not less than 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 basis dried material.

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 is gray, dark gray or red-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 is grayish brown and the xylem is 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 raphides of calcium oxalate. In the cortex, sclerenchyma cells are absent. In the xylem, vessels and tracheids are arranged alternately.

Ipecac has a slight, characteristic odor and the taste is slightly bitter and unpleasant. The powder 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: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

Loss on Drying Not more than 12.0 % (6 hours).

Ash Not more than 5.0 %.

Acid-insoluble Ash Not more than 2.0 %.

Assay Weigh accurately about 0.5 g of pulverized Ipecac, in a glass-stoppered centrifuge tube, add 30 mL of 0.01 mol/L hydrochloric acid TS, shake for 15 minutes, centrifuge and separate the supernatant liquid. Repeat this procedure twice with the residue using 30 mL volumes 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 mo1/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.

$$\text{Amount (mg) of total alkaloids (emetine and cephaeline)} = \text{amount (mg) of Emetine Hydrochloride RS} \times \frac{A_{Ta} + A_{Tb} \times 0.971}{A_S} \times 0.868$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 283 nm).

Column: A stainless steel 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 °C.

Mobile phase: Dissolve 2 g of sodium 1-heptane sulfonate in 500 mL of water, adjust the pH 4.0 with acetic acid (100) and add 500 mL of methanol.

Flow rate: Adjust the flow rate so that the retention time of emetine is about 14 minute.

System suitability

System performance: Dissolve l mg each of Emetine Hydrochloride RS and cephaeline hydrofluoric acid in 10 mL of 0.01 mol/L hydrochloric acid TS. When the procedure is run with 10 μL of this solution under the above operating conditions, cephaeline and emetine are eluted in this order with clearly dividing each peak.

System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions: the relative standard deviation of the peak area of emetine is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Jujube

Zizyphi Fructus

Jujube is the ripe fruit of *Zizyphus jujuba* Miller var. *inermis* Rehder or *Zizyphus jujube* Miller var. hoonensis T. B. Lee (Rhamnaceae).

Description Jujube is the fruit, ellipsoidal or spherical, 2 cm to 3 cm in length and 1 cm to 2 cm in diameter. External surface is red-brown to dark red with wrinkles and lustrous. Both ends of the Jujube is slightly dented, with a scar of style on one end and a scar of fruit stalk on the other. Epicarp is thin and leather. Mesocarp is thick, dark grayish brown spongy, soft and adhesive. Endocarp is extremely hard, fusiform and divided into two loculi containing flat and ovoid seeds and the texture is hard.
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, cool, filter, concentrate the filtrate until it becomes 2 mL and use 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. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 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, ethyl acetate and formic acid (15:5:0.5) to a distance of about 10 cm and air-dry the plate. Spray evenly sulfuric acid TS for spraying and heat at 105 °C: 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.

Purity (1) ***Rancidity***—Jujube has no unpleasant, rancid odor and taste.

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***— Proceed with Jujube as directed in "Jujube (Dried)" in [Attachment 4] MRLs for Agricultural Products in KFDA Notice "Standards and Specifications for Food."

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 3.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Juncus Medulla

Junci Medulla

Juncus Medulla is the stem pith of *Juncus effusus* Linné (Juncaceae).

Description Juncus Medulla is the stem pith, slenderly cylindrical, 30 cm to 60 cm in length and about 2 mm in diameter. External surface is white to pale yellowish white, flattened on touching, with fine longitudinal wrinkles. Under a magnifying glass, transverse section reveals numerous fine pits and loose and light fractured surface like sponge. Under a microscope, transverse section reveals that the whole consists of aerenchymas. Each cells is nearly quadrilateral or rectangle, branched. Lacuna of cells forms in triangular or quadrilateral shape.
Juncus Medulla is nearly odorless and taste is weak.

Identification Weigh 1 g of pulverized Juncus Medulla, add 100 mL of methanol, heat under a reflux condenser in a water bath for 1 hour, filter and evaporate the filtrate to dryness. Wash the residue with 2 mL of ether, dissolve it in 1 mL of anhydrous ethanol, use this solution as the test solution. Perform the test with

the test solution as directed under the Thin-layer Chromatography. Spot 5 μL of the test solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of cyclohexane and ethyl acetate (10 : 7) to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with sulfonic acid for spray and heat at 105 °C for 10 minutes: spot of the R_f value about 0.5 is purple.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 11.0 %.

Ash Not more than 5.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Kalopanax Bark

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, 1 mm to 4 mm in thickness. The external surface is grayish white to grayish brown, coarse, with grayish black longitudinal clefts and transversely open patterns. It is scattered with yellow, round, spot-like lenticels, 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, longtitudinally oblong. The inner bark with the spines fallen off is yellow. The inner surface is yellow-brown or purple-brown, smooth, with a distinct fine longitudinal pattern. The texture is hard, tough and difficult to cut. The cut surface is grayish brown on the outside, grayish yellow on the inside, very fibrous with distinct lamellae.
Kalopanax Bark 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 and filter. Add slowly 1 mL of sulfuric acid to 2 mL of the filtrate: a red-purple color develops at the zone of contact and a green color at the upper layer.
(2) Weigh 1 g of pulverized Kalopanax Bark, add 10 mL of methanol, sonicate for 60 minutes, filter and use the filtrate as the test solution. Perform the test with this solution as directed under Thin-layer Chromatography. Spot 10 μL of the test solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of chloroform, methanol and water (10 : 5 : 1) to a distance of about 10 cm and air-dry the plate. Spray evenly dilute sulfuric acid on the plate and heat at 105 °C for 10 minutes: a dark brown spot appears at the R_f value of about 0.55.

Purity (1) ***Foreign matter*—**The amount of cork layer and other foreign matter contained in Kalopanax Bark is not more than 1.0 %
(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(4) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 9.0 %

Ash Not more than 10.0 %

Extract Content ***Dilute ethanol-soluble extract*—** Not less than 8.0 %

Containers and Storage ***Containers*—**Well-closed containers.

Kochia Fruit

Kochiae Fructus

Kochiae Fruit is the well ripe fruit of *Kochia scoparia* Schrader (Chenopodiaceae).

Description Kochia Fruit is oblate, five-pointed star shape fruit, 1 mm to 3 mm in diameter. External surface is grayish green to pale brown with five membranous winglets, surrounded by a persistent perianth. The center of dorsal surface has a slightly prominent, pointed fruit stalk scar and 5 to 10 radial veins. When the perianth stripped, translucent membranous pericarp is visible. The seed is flattened ovoid, about 1 mm in length, black.
Kochia Fruit has a slight, characteristic odor and slightly bitter taste.

Identification Weigh 2 g of pulverized Kochia Fruit, add 20 mL of ethanol and 1.5 mL of hydrochloric acid, heat with a reflux condenser for 2 hours and filter. Concentrate to about 5 mL of the filtrate in vaccum, add 10 mL of water, transfer the filtrate to a separatory funnel and extract with 20 mL of petroleum ether. Evaporate the ether layer to dryness, dissolve the residue to 2 mL of ethanol and use the solution as the test solution. Separately, weigh 5 mg of Oleanolic acid RS, add 5 mL of ethanol and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 2 μL each of the test solution and the standard solution on a plate of silica gel for thin-layer chromatography. Deve1op the plate with a mixture of cyclohexane, acetone and ethyl acetate (5:2:1) to a distance of about 10 cm and air-dry the plate. Spray dilute sulfuric acid TS and heat the plate at 105 °C. One spot among several spots from the test solution and the spot from the standard solution show the same color and the same R_f value.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 10.0 %

Ash Not more than 10.0 %

Acid-insoluble Ash Not more than 5.0 %

Extract Content ***Dilute ethanol-soluble extract***—Not less than 15.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Leonurus Herb

Leonuri Herba

Leonurus Herb is the aerial part collected before or when flowering of *Leonurus japonicus* Houttuyn (Labiatae). Leonurus Herb, when dried, contains not less than 0.05 % of leonurine ($C_{14}H_{21}N_3O_5$: 311.33).

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, 1 mm to 5 mm in diameter, the external surface is yellow-green to green-brown, densely covered with white and short hairs. White huge pith is in fractured surface of the stem and texture is pliable. Leaves are opposite, palmately ternate to lobed-ternate, the upper surface is pale yellow, and the lower surface is densely pubescent and grayish green. Flowers are pubescent verticillatelly on axil, calyx is cylindrical, usually 5-lobed at the apex and pale green to green-brown.
Leonurus Herb has a slight, characteristic odor and tastes bitter and astringent.

Identification Weigh 3 g each of pulverized Leonurus Herb and Leonurus Herb RMPM, add 30 mL of methanol, sonicate for 1 hour, and filter, respectively. Use the filtrates as the test solution and the standard solution of Leonurus Herb RMPM. Perform the test with the test solution and the standard solution of Leonurus Herb RMPM as directed under Thin-layer Chromatography. Spot 10 μL of test solution and the standard solution of Leonurus Herb RMPM on the plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of buthanol, formic acid, and water (4 : 1 : 0.5) to a distance of about 10 cm and air-dry the plate. Spray the iodide-bismuth potassium TS to the plate; the several spots from the test solution and the spots from the standard solution of Leonurus Herb RMPM show the same color and the same R_f value and of these, the spot of stachydrine hydrochloride appears at the R_f value of 0.15.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 13.0 % (6 hours).

Ash Not more than 10.0 %.

Acid-insoluble Ash Not more than 2.0 %.

Extract Content ***Dilute ethanol–soluble extract***—Not less than 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, 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 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.

$$\text{Amount (mg) of leonurine } (C_{14}H_{21}N_3O_5) = \text{Amount (mg) of Leonurine RS} \times \frac{A_T}{A_S} \times \frac{1}{20}$$

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 octadecylsilanized silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Column temperature: A constant temperature of about 30 °C

Mobile phase: Control the step or concentration gradient by mixing mobile phases A and B as directed in the following table.

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 (%)	Mobile phase B (%)
0	75	25
30	45	55
35	75	25

Flow rate: 1.0 mL/minute

System suitability

System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions, the relative standard deviation of the peak area of leonurine is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Licorice

Glycyrrhizae Radix et Rhizoma

Licorice is the root and rhizome with or without the periderm, of *Glycyrrhiza uralensis* Fisher, *Glycyrrhiza glabra* Linné or *Glycyrrhiza inflate* Batal. (Leguminosae). Licorice contains not less than 2.5 % of glycyrrhizic acid ($C_{42}H_{62}O_{16}$: 822.93) and not less than 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 rhizome. The root is cylindrical pieces, 25 cm to 100 cm in length and 5 mm to 35 mm in diameter. The external surface is red-brown to yellow-brown with distinct longitudinal wrinkles, dents and lenticels, and has sparse, thin root scars. The texture is hard. The transverse section is fibrous and yellowish white, much 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 has pith. Under a microscope, the transverse section reveals severa1 layers of yellow-brown cork layers and 1 to 3 cellular layer of cork cortex inside the cork layer. The cortex exhibits groups of phloem fibers with thick but incompletely lignified walls and surrounded by crystal 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 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 cells are scattered between vessels. The rhizome has pith and the parenchyma cells of the cortex and xylem contain solitary crystals of calcium oxalate and starch grains. Peeled Licorice sometimes lacks periderm and apart of phloem.

(2) ***Glycyrrhiza glabra***—Licorice from *Glycyrrhiza glabra* Linné consists the root and 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 inflate***—Licorice from *Glycyrrhiza inflate* Batal. consists the root and rhizome. Its texture is relatively touch and is sometimes branched. The external peel is rough and largely grayish brown. The lenticel is slender and not distinct.

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. Perform the test with the test solution and the standard solutions as directed under the Thin-layer Chromatography. Spot 2 μL each of the test solution and the standard solutions on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, water, formic acid and acetic acid (100) (15 : 2 : 1 : 1) to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): one spot

among the several spots from the test solution and spots from the standard solution show the same colors and the same R_f values.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Difenoconazole: Not more than 0.05 ppm.

(iv) Methoxychlor: Not more than 1 ppm.

(v) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(vi) Azoxystrobin: Not more than 0.5 ppm.

(vii) Aldrin: Not more than 0.01 ppm.

(viii) Endrin: Not more than 0.01 ppm.

(ix) Acetamiprid: Not more than 0.1 ppm.

(x) Imidacloprid: Not more than 0.1 ppm.

(xi) Chlorothalonil: Not more than 0.05 ppm.

(xii) Thiamethoxam: Not more than 0.1 ppm.

(xiii) Fenpyroximate: Not more than 0.1 ppm.

(xiv) Pymetrozine: Not more than 0.5 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

(4) ***Mycotoxins***—Total aflatoxins (sum of aflatoxins B_1, B_2, G_1 and G_2): Not more than 15.0 ppb (aflatoxin B_1 is not more than 10.0 ppb).

Loss on Drying Not more than 12.0 % (6 hours).

Ash Not more than 7.0 %.

Acid-insoluble Ash Not more than 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 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 silica gel desiccator for 24 hours), dissolve in diluted ethanol (7 in 10) to make exactly 100 mL and use this solution as the standard solution. Pipet 20 μL each 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_T and A_S, of 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 mm to 6 mm in internal diameter and l5 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 dilute 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 l mg of propylparaben in diluted ethanol (7 in 10) to make 20 mL. When the procedure is run with 20 μL of this solution under the above operating conditions, glycyrrhizic acid and propylparaben are eluted in this order with clearly dividing each peak.

System repeatability: When the test is repeated six times with 20 μL each of the standard solution under the above operating conditions: the relative standard deviation of the peak area of glycyrrhizic acid is not more than 1.5 %.

(2) ***Liquiritigenin***—Weigh accurately about 0.5 g of pulverized Licorice, add 100 m L of 2 mol/L hydrochloric acid and heat with a reflux condenser at 90 °C for 1 hour. To the extract, add 100 mL of dichloromethane and heat with a reflux condenser 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 the dichloromethane layer, 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 silica gel desiccator 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 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.

$$\text{Amount (mg) of liquiritigenin } (C_{15}H_{12}O_4) = \text{amount (mg) of Liquiritigenin RS} \times \frac{A_T}{A_S}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 276 nm).

Column: A stainless steel column, about 4 mm to 6 mm in internal diameter and l5 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 diluted acetic acid (1 in 100) and acetonitrile (75:25)

Flow rate: 1.0 mL/min.

System suitability

System repeatability: When the test is repeated six times with 20 μL each of the standard solution under the above operating conditions: the relative standard deviation of the peak area of liquiritigenine is not more than 1.5 %.

Containers and Storage ***Containers*****—**Well-closed containers.

Licorice Extract

Licorice Extract contains not less than 4.5 % of glycyrrhizic acid. ($C_{42}H_{62}O_{16}$: 822.93).

Method of Preparation Weigh 1 kg of fine cutting licorice, add 5 L of water or purified water and digest for 2 days. Filter the digested solution through a cloth filter. Add 3 L of Water or Purified Water to the residue, digest again for 12 hours and filter through a cloth filter. Evaporate the combined filtrates until the whole volume becomes 3000 mL. After cooling, add 1 L of ethanol and allow to stand in a cold place for 2 days. Filter and evaporate the filtrate to a viscous extract.

Description Licorice Extract is brown to blackish brown, viscous extract and has a characteristic odor and sweet taste. Licorice Extract dissolves in water, forming a clear solution, or with a slight turbidity.

Identification Weigh 0.8 g of Licorice Extract, add 10ml of a mixture of ethanol and water (7 : 3), shake for 2 minitues, centrifuge and use the supernatant liquid as the test solution. Proceed as directed in the Identification under Licorice.

Purity (1) ***Insoluble matter*****—**Dissolve 2.0 g of Licorice Extract in 18ml of water and filter. To 10 mL of the filtrate, add 5 mL of ethanol: the solution is clear.

(2) ***Heavy metals*****—**Total heavy metals: Not more than 30 ppm.

(3) ***Residual pesticides*****—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

Assay Weigh accurately about 0.15 g of Licorice Extract, place in a glass-stoppered, centrifuge tube, add 25 mL of dilute ethanol and heat at 50 °C for 30 minute with occasional shaking. After cooling, centrifuge and separate the supernatant liquid. 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 silica gel desiccator 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 under Licorice.

$$\text{Amount (mg) of glycyrrhizic acid } (C_{42}H_{62}O_{16}) = \text{amount (mg) of Glycyrrhizic Acid RS, calculated on the anhydrous basis} \times \frac{A_T}{A_S}$$

Containers and Storage ***Containers***—Tight containers.

Lindera Root

Linderae Radix

Lindera Root is the root of *Lindera strychnifolia* Fernandez-Villar (Lauraceae).

Description Lindera Root is the fusiform of rosary-like root, 10 to 15 cm in length, 10 to 25 mm in diameter, slightly curved and bead-threaded shpae. External surface is yellowish brown to brown, with lateral wrinkles and with scattering scars of rootlets. Lindera Root, is hard and dense in texture, is difficult to break. The fractured surface is pulverized. Under a magnifying glass, a transverse section reveals brown to light yellowish brown and concentric circles and radially arranged lines brown.

Under a microscope, a transverse section reveals a cork layer consisting of partially cork stone cells and parenchyma cells is composed of oil cells and fibers. In xylem, vessels, xylem fibers and rays are arranged alternately. Parenchyma cells of cortex and xylem contain sandy and columnar crystals of calcium oxalate, simple starch grains, 1 to 15 μm in diameter and starch grains, 2 to 4 compound.

Lindera Root has an aroma, cool and slightly bitter taste.

Identification (1) Weigh 1 g of pulverized Lindera Root, add 10 mL of chloroform and 1 mL of ammonia TS, and allow to stand for 1 hour with occasional shaking. Transfer the filtrate into the separatory funnel, add 2 mL of dilute hydrochloric acid, and allow to stand after shaking well. Pipet the water layer, add 1 to 2 drops of Mayer TS: the solution develops a white turbidity.

(2) Weigh 1 g of pulverized Lindera Root, add 30 mL of petroleum ether, allow to stand for 30 minutes, sonicate for 10 minutes, 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. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 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 acetone (9:1) to a distance of about 10 cm and air-dry the plate. Spray evenly dilute sulfuric acid TS and heat 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) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 14.0 % (6 hours)

Ash Not more than 2.5 %.

Extract Content ***Dilute ethanol-soluble extract*—** Not less than 7.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Linseed

Lini Semen

Linseed is the ripe seed of *Linum usitatissimum* Linné (Linaceae).

Description Linseed is the seed, flattened ovate, obtusely round at one end and pointed and slightly flat and slanted at the other end. It is 4 mm to 6 mm in length and 2 mm to 3 mm in width. The external surface is reddish brown to grayish brown, slightly smooth and lustrous. The hilum is located at the slightly dented area of the pointed end, the dorsal ridge of the seed is pale brown and located at the margin of one side. Under a microscope, the transverse section reveals relatively large epidermal cells, close to rectangular. The cell walls contain mucous, expanding on contact with water, making the lamella distinct. The outside is covered by a horny layer. The hypodermis consists of 1 to 5 rows of parenchyma cells and the cell walls are slightly thick. The fiber layer consists of 1 row of dense fiber cells. The degenerate layer does not have a clear cell boundary. The pigment layer consists of 1 layer of flat parenchyma cells containing reddish brown substances. The endosperm and cotyledon cells are polygonal, containing fatty oil, aleurone grains and 1 to 2 pseudocrystals.
Linseed is nearly odorless and produces mucus when immersed in water.

Identification Weigh 0.5 g of pulverized Linseed, add 5 mL of dichloromethane, macerate for 20 minutes, filter and use the filtrate as the test solution. Perform the test with this solution as directed under Thin-layer Chromatography. Spot 10 μL of the test solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of hexane, ether and acetic acid (7:3:0.1) 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 the R_f values of 0.3 and 0.7.

Purity (1) ***Foreign matter***—Not more than 2.0 %.
(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(4) ***Sulfur dioxide*—**Not more than 30 ppm.

Ash Not more than 5.0 %.

Extract Content ***Ether-soluble extract***—Not less than 30.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Liriope Tuber

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 tuber, with the shape of long rectangle or round rectangle, 12 mm to 40 mm in length and 4 mm to 9 mm in diameter. External surface is pale yellow, wrinkled longitudinally. The texture is soft and tough. The fractured surface is yellowish white, slightly transparent. The stele is the thin, strong, tough core. Under a microscope, transverse section appears epidermis, rectangular or polygonal, with 1 line of cells. Exodermal cells are lined in 1

to 2 rows, bigger than epidermal cells, lignified. The cortex is very broad, composed of about 30 rows of cells containing mucous and calcium oxalate raphide bundles. The outer layer of the endodermis has 1 to 3 rows of stone cells. The endodermal cells have evenly thickened cell walls and have passage cells. The vascular bundles are radial and there are 12 to 20 phloem bundles, each of which are located in the bow-like dents of the xylem bundles. Lignified tissue of the xylem bundles are connected to each other, forming rings. They are small in number.
Liriope Tuber has a slight, characteristic odor and slightly sweet and mucous taste.

(2) ***Ophiopogon japonicus***—Liriope Tuber from *Ophiopogon japonicus* is the tuber, long rectangular cylindrical or fusiform, 10 mm to 25 mm in length and 3 mm to 5 mm in diameter, somewhat sharp at one end and somewhat rounded at the other. External surface is pale yellow to pale yellow-brown, with longitudinal wrinkles of various sizes. It is easily fractured. The fractured surface is yellowish white, slightly transparent. The stele is the thin, strong and tough core. Under a microscope, a transverse section reveals 1 row of rectangular to polygonal epidermal cells. There are 3 to 5 rows of exodermal cells and the cell wall is lignified. The cortex is broad, composed of 14 to 27 rows of cells containing mucous and calcium oxalate raphide bundles. The outer layer of the endodermis has 1 row of stone cells. The endodermal cells have evenly thickened cell walls and have passage cells. The stele is very small and the stele sheath is composed of 1 to 2 rows of parenchyma cells. The vascular bundles are radial and there are 13 to 22 phloem bundles, each of which are located in the bow-like dents of the xylem bundles. Lignified tissue of the xylem bundles are connected to each other, forming rings. They are small in number.
Liriope Tuber has a slight, characteristic odor and slightly sweet and mucous taste.

Identification Weigh 2 g of pulverized Liriope Tuber, add 20 mL of methanol, allow to stand for 3 hours, sonicate for 30 minutes, filter and evaporate the filtrate to dryness. Dissolve the residue in 1 mL of methanol and use this solution as the test solution. Perform the test with this solution as directed under Thin-layer Chromatography. Spot 10 μL of the test solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of toluene, methanol and acetic acid (100) (80:5:0.1) to a distance of about 10 cm and air-dry the plate. Spray evenly sulfuric acid TS for spraying and heat at 105 °C: of the spots obtained from the test solution, a green spot appears at the R_f value of 0.45.

Purity (1) ***Foreign matter***—Liriope Tuber contains less than 1.0 % of rootlets.

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(vi) Pendimethalin: Not more than 0.2 ppm.
(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 3.0 %.

Containers and Storage ***Containers—***Well-closed containers.

Lithospermum Root

Lithospermi Radix

Lithospermum Root is the root of *Lithospermum erythrorhizon* Siebold et Zuccarini, *Arnebia euchroma* Johnst., and *Arnebia guttata* Bunge (Boraginaceae).

Description (1) ***Lithospermum erythrorhizon—***Litho-spermum Root from *Lithospermum erythrorhizon* is rather slender conical root, often branched, 6 cm to 10 cm in length and 5 mm to 15 mm in diameter. External surface is dark purple to purple-brown, and cortex is coarse, thin and easily peeled. Lithospermum Root is mostly with twisted and deep longitudinal furrows which sometimes reach to xylem. Sometimes remains of stem are present at the crown. Lithospermum Root is easily broken. Fractured surface is granular and with many clefts. Under a magnifying glass, a transverse section reveals a dark purple color at the outer volume of cortex and pale brown inner volume making irregular wave, and xylem is yellow. The center of the crown is often cracked and the surrounding part is red-purple.
Lithospermum Root from *Lithospermum erythrorhizon* has a slight, characteristic odor and slight sweet taste.

(2) ***Arnebia euchroma***—Lithospermum Root from *Arnebia euchroma* is irregular cylindrical root, almost twisted, 7 cm to 20 cm in length and 10 mm to 25 mm in diameter. External surface is red-purple to purple-brown and lax, bar-shaped, normally ten layers overlapped and easily peeled off. Apex sometimes bears branched remains of stems. Texture is lax, soft and light. Under a magnifying glass, a transverse surface reveals uneven surface and relatively small, yellowish white to yellow xylem.
Lithospermum Root from *Arnebia euchroma* has a characteristic odor and slight bitter and astringent tastes.

(3) ***Arnebia guttata—***Lithospermum Root from *Arnebia guttata* is conical to cylindrical and twisted root, 6 cm to 20 cm in length, 15 mm to 40 mm in diameter. Crown is slightly large and apex bears one or

more remains of a stem covered with short and stiff hairs. External surface is red-purple or dark purple, slightly thin, normally several layers overlapped and easily peeled off. Txture is hard, fragile and easily broken. Under a magnifying glass, a transverse section reveals clear surface, red-purple cortex and slightly small and yellowish white xylem.
Lithospermum Root from *Arnebia guttata* has a characteristic odor and astringent taste.

Identification (1) Weigh 0.5 g of pulverized Lithospermum Root, heat in a test tube: red vapor evolves, which condenses on the wall of the upper part of the tube into red-brown oil drops.

(2) Weigh 0.5 g of pieces or powder of Lithospermum Root, add l mL of ethanol and to the red solution thereby obtained, add l drop of sodium hydroxide TS: the red color changes to blue-purple. To this solution, add l to 2 drops of dilute hydrochloric acid: the color turns red again.

(3) Weigh 0.5 g of pulverized Lithospemum Root, add 5mL of ethanol, shake for 30 minutes, filter, evaporate in vaccum at not more than 40 °C, then add 1 mL of ethanol and use this solution as the test solution. Perform the test with the test solution as directed under the Thin-layer Chromatography. Spot 5 μL of the test solution on a plate of silica gel for thin-layer chromatography. 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: the spot of the R_f value about 0.75 is red-purple.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(vi) Thifluzamide: Not more than 1.0 ppm.
(vii) Pencycuron: Not more than 1.0 ppm.
(viii) Hexaconazole: Not more than 0.2 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 11.0 %.

Acid-insoluble Ash Not more than 3.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Longan Arillus

Longan Arillus

Longan Arillus is the arill of *Dimorcarpus longan* Loureiro (Sapindaceae).

Description Longan Arillus is longitudinally broken and irregular thin slices, frequently several slices agglutinated, 2 to 4 cm in length and 1 cm to 2 cm in width and 2 mm to 4 mm in thickness. External surface is dark reddish brown to blackish brown and semi-transluscent. One surface is shrunkened, the other surface is lustrous, with longitudinally fine wrinkles. Texture is soft and sticky.
Longan Arillus has a slight, characteristic odor and sweet taste.

Identification Weigh 1 g of Longan Arillus, add 10 mL of water, shake well and filter. To 3 mL of filtrate, add 3 mL of Fehling TS and warm in a water-bath: a red precipitate is produced.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 15.0 % (60 °C, 6 hours).

Ash Not more than 5.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Longgu

Fossila Ossis Mastodi

Longgu is the ossified bone of large mammal and is mainly composed of calcium carbonate.

Description Longgu is irregular masses or fragments, occasionally cylindrical masses as the ossified bone of large mammal. External surface is pale grayish white, sometimes with grayish black or yellow-brown spots here and there. The outer part consists of a layer 2 mm

to 10 mm in thickness and is dense in texture, and the inner part consists of pale brown porous volume. The texture is heavy and hard, but somewhat fragile. When crushed, it changes into pieces and powder.
Longgu is odorless, tasteless and strongly adhesive to the tongue on licking.

Identification (1) Weigh 0.5 g of pulverized Longgu, dissolve in 10 mL of dilute hydrochloric acid: it evolves a gas and forms slightly brown and turbid solution. Pass the gas evolved through calcium hydroxide TS: a white precipitate is produced.

(2) The turbid solution, obtained in (1), has a characteristic odor. Filter this solution and neutralize with ammonia TS: the solution responds to the Qualitative Tests 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 produced.

Purity (1) ***Heavy metals***—Weigh 2 g of pulverized Longgu, add 5 mL of water, shake to mix, add carefully 6 mL of hydrochloric acid and evaporate on a water-bath to dryness. 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. Prepare the control solution as follows: evaporate 3 mL of hydrochloric acid on a water–bath to dryness, add 2 mL of dilute acetic acid and 2.0 mL of standard lead solution and add water to make 50 mL (not more than 20 ppm).

(2) ***Arsenic***—Prepare the test solution with 0.2 g of pulverized Longgu according to Method 2 and perform the test (not more than 10 ppm).

Containers and Storage ***Containers*—**Well-closed containers.

Lonicera Flower

Lonicerae Flos

Lonicera Flower is the flower bud or the flower starting to bloom of *Lonicera japonica* Thunberg (Caprifoliaceae).

Description Lonicera Flower is the flower bud or the flower starting to bloom. The flower buds are small clavate or conical in shape and the flowers are lip-shaped. It is 15 mm to 35 mm in length and about 3 mm in diameter in upper part and 1.5 mm in diameter in lower part. External surface is yellowish-white or greenish-white, gradually darken on keeping. Under a magnifying glass, pale brownish hair is densely pubescent. The calyx is green, 5-lobed at the apex, lobes are pubescent, about 2 mm in length. Numbers of stamens are 5, pistil 1, ovary glabrous.
Lonicera Flower has a slight, characteristic odor and weak and slightly bitter taste.

Identification (1) Weigh 0.5 g of pulverized Lonicera Flower, add 10 mL of water, heat and filter. Add 1 to 2 drops of iron (III) chloride TS to the filtrate: a blue-violet color develops.

(2) Weigh 2.0 g of pulverized Lonicera Flower, add 10 mL of ethanol, heat for 2 minutes in a water-bath and filter. Add 0.1 g of magnesium and 2 to 3 drops of hydrochloric acid to the filtrate: a pale brown to reddish brown color develops.

(3) Weigh 0.1 g of pulverized Lonicera Flower and Lonicera Flower RMPM, add 10 mL of diluted ethanol (7 in 10), sonicate for 60 minutes, filter and use these solutions as the test solution and the Lonicera Flower RMPM standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 2 μL each of the test solution and the Lonicera Flower RMPM standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, methanol and water (8:2:1) to a distance of about 10 cm and air-dry the plate. Examine 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 Lonicera Flower RMPM Standard Solution, and the spot of chlorogenic acid appears at the R_f value of about 0.3.

Purity (1) ***Foreign matter***—Stems and leaves: Less than 5.0 %.

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 15.0 % (6 hours).

Ash Not more than 9.0 %.

Extract Content ***Dilute ethanol-soluble extract***—Not less than 16.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Lonicera Leaf and Stem

Lonicerae Folium et Caulis

Lonicera Leaf and Stem is the leaves and climbing

stems of *Lonicera japonica* Thunberg (Caprifoliaceae). Lonicera Leaf and Stem, when dried, contains not less than 0.1 % of loganin ($C_{17}H_{26}O_{10}$: 390.38).

Description Lonicera Leaf and Stem is the leaves and climbing stems. The leaves is round and entire, 3 cm to 7 cm in length, 1 cm to 3 cm in width, with short petiole. The upper surface is greenish brown, and the lower surface is pale grayish green. Under a magnifying glass, both surfaces are pubescent. The stem is long cylindrical, frequently branched, usually forming tangled bundles, 1.5 mm to 6 mm in diameter. The external surface is red-brown to dark brown, sometimes grayish green. The epidermis is easily peeled and fallen off. There are many nodes on the branches with an internode distance of 6 cm to 9 cm. The texture is fragile and easy to cut. The cut surface is yellowish white and hollow in the middle. Under a microscope, the transverse section reveals medullary rays of the stem consisting of 1 to 2 rows of cells. The vessels are up to about 160 μm in diameter and contain yellow-brown or red-brown substances. The xylem fiber is polygonal and the cell wall is very thick. The parenchyma cells of the xylem have very thick, lignified walls and sometimes contain prismatic crystals of calcium oxalate. The pith has irregular polygonal parenchyma cells, varying in size, the walls slightly lignified and pitted.
Lonicera Leaf and Stem has almost odorless and slightly astringent, followed by 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, filter and use the filtrates as the test solution and the Lonicera Leaf and Stem RMPM standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 μL each of the test solution and the Lonicera Leaf and Stem RMPM standard solution on a plate of silica gel for thin-layer chromatography. 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 spots obtained from the test solution show the same color and R_f value as the spots from the Lonicera Leaf and Stem RMPM standard solution and of these, the spot of loganin appears at the R_f value of 0.25.

Purity (1) ***Foreign matter***—Lonicera Leaf and Stem does not contains the stems larger than 5 mm in diameter.

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 12.0 % (6 hours).

Ash Not more than 9.0 %.

Acid-insoluble Ash Not more than 1.0 %.

Extract Content ***Dilute ethanol-soluble extract***—Not less than 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, 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.

$$\text{Amount (mg) of loganin } (C_{17}H_{25}O_{10}) = \text{Amount (mg) of Loganin RS} \times \frac{A_T}{A_S} \times \frac{1}{10}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 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 octadecylsilanized silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Mobile phase: Control the step or concentration gradient by mixing mobile phases A and B as directed in the following table.

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 (%)	Mobile phase B (%)
0	100	0
15	70	30
25	30	70
30	30	70
35	100	0

Flow rate: 1.0 mL/minute

Containers and Storage ***Containers*—**Well-closed containers.

Lycium Fruit

Lycii Fructus

Lycium Fruit is the dried fruit of *Lycium chinense* Miller or *Lycium barbarum* Linné (Solanaceae). Lycium Fruit, when dried, contains not less than 0.5 % of betain ($C_5H_{11}NO_2$: 117.15).

Description Lycium Fruit is the fruit, nearly fusiform or elliptical, 6 mm to 20 mm in length and 3 mm to 10 mm in diameter. External surface is red to dark red, with a scar of pistil stalk like small projection at the end and a scar of fruit stalk on the base. Pericarp is soft, tough and crumpled. Sarcocarp is pulpy, soft and tender. 20 to 50 Seeds are present inside. Seed is kidney-shaped, nearly flat, about 2 mm in length and 1 mm to 2 mm in width. External surface of seed is pale yellow or yellowish brown.
Lycium Fruit has a slight, characteristic odor and sweet taste.

Identification Weigh 1 g of pulverized Lycium Fruit, add 5 mL of ethyl acetate, extract by shaking for 15 minutes, filter and use the filtrate as the test solution. Perform the test with this solution as directed under Thin-layer Chromatography. Spot 20 μL of the test solution on a plate of silica gel for thin-layer chromatography. 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. It shows 1 yellowish spot at the R_f value of about 0.6.

Purity (1) ***Foreign matter***—Lycium Fruit contains less than 3.0 % of branch, fruit stalk and other foreign matter.

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—Proceed as directed in "Lycium Fruit (Dried)" in [Attachment 4] MRLs for Agricultural Products in KFDA Notice "Standards and Specifications for Food."

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 8.0 %.

Assay Weigh accurately about 1.0 g of pulverized Lycium Fruit, add 50 mL of diluted methanol (1 in 2), heat with a reflux condenser for 2 hours and filter. Repeat the above procedure with the residue using 50 mL of diluted methanol (1 in 2). Combine all filtrates, evaporate to dryness in vaccum, add 30 mL of deionized water to the residue and adjust pH to 3.0 with dilute hydrogen chloride. Load the suspension on to the column I and elute with 60 mL of deionized water and discard it. The column is eluted with 15 mL of diluted ammonia TS (2 in 5) and 15 mL of deionized water, successively. The eluate is collected and concentrated to 5 mL in vaccum. The concentrated solution is loaded on to column II and eluted with 10 mL of deionized water. The eluate is combined and evaporated to dryness. Dissolve the residue in 1.0 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 L 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 betaine of 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 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with dimethylaminopropylsilylated silica ge1 (5 μm to 10 μm in particle diameter).

Mobile phase: A mixture of acetonitrile and water (85 : 15).

Flow rate: 1.0 mL/minute.

Column I: A glass tube 10 mm to 12 mm in internal diameter and 10 cm in length, packed 5 cm high with strong cationic ion-exchange resin (H^+ form).

Column II: A glass 10 mm to 12 mm in internal diameter and 10 cm in length, packed 5 cm high with 1:2 ratio of cationic ion-exchange resin (H^+ form) and strong basic anionic ion-exchange resin (OH^- form), respectively.

System suitability

System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions, the relative standard deviation of the peak area of betaine is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Lycium Root Bark

Lycii Radicis Cortex

Lycium Root Bark is the rhizome of *Lycium chinense* Miller or *Lycium barbarum* Linné (Solanaceae).

Description Lycium Root Bark is the cylindrical to semi-cylindrical or fragmentary rhizome, 3 cm to 10 cm in length, 5 mm to 15 mm in width and 1 mm to 3

mm in thickness. The external surface is grayish yellow to brownish yellow and coarse with irregular transversus torn patterns, the periderm is scale-shaped and easily taken off. The inside is yellowish white to grayish yellow and slightly even with fine longitudinal striations. The body is light and the texture is fragile and easily broken. Fractured surface is grayish white to yellowish brown, not fibrous. The texture is light and coarse. Under a microscope, the transverse section reveals a rhytidome on the outer layer. The rhytidome consists of 2 to 3 bands of cork tissue layers, the innermost layer forming a whole and even ring and occurring deep within the phloem. Degenerated sieve tubes and 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 one row of cells and the parenchyma cells contain calcium oxalate crystal sand and starch grains. Fibers and stone cells appear scattered. Fibers exist individually or in bundles and the cell wall is lignified or slightly lignified.
Lycium Root Bark has a characteristic odor and tastes slightly sweet, later bitter.

Identification (1) Weigh 0.5 g of pulverized Lycium Root Bark, add 10 mL of acetic anhydride, heat on a water bath for 2 minutes and filter. Add carefully 2 mL of sulfuric acid to 2 mL of the filtrate: a red-brown color appears at the zone of contact, a green color at the upper layer after allowing to stand.

(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. Add 1 mL of ninhydrin TS to 2 mL of the filtrate, heat on a water bath for 2 to 3 minutes: a purple color appears.

(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. Perform the test with this solution as directed under Thin-layer Chromatography. Spot 10 μL of the test solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of 1-butanol, water, pyridine and acetic acid (3:1:1:1) to a distance of about 10 cm and air-dry the plate. Spray evenly Dragendorff's TS, 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 0.5.

Purity (1) ***Foreign matter***—Not more than 5.0 % of xylem and other foreign matter

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 12.0 %

Ash Not more than 18.0 %

Acid-insoluble Ash Not more than 3.0 %.

Extract Content ***Dilute ethanol-soluble extract***—Not less than 8.0 %

Containers and Storage ***Containers*—**Well-closed containers.

Lycopus Herb

Lycopi Herba

Lycopus Herb is the aerial part, before flowering, of *Lycopus lucidus* Turczaininov (Labiatae).

Description Lycopus Herb is the aerial part, rectangular cylindrical with a few branches, 50 cm to 100 cm in length and 2 mm to 6 mm in diameter. The external surface is yellow-green or purple with a distinct purple 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 is 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 is blackish green and the lower surface is grayish green, densly glandular-dotted. Both surfaces are covered in equally short hairs. Apex is acute and margin is serrate. Flowers are aggregated in leaf axils in verticillate cymes, corolla is mostly fallen off, and bracts and calyx are yellow-brown.
Lycopus Herb is odorless and has weak 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, dissolve 0.5 mg of Ursolic Acid RS in 1 mL of ethanol and use this solution as the standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 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, ethyl acetate and formic acid (15 : 5 : 0.5) 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: 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: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 10.0 %

Ash Not more than 5.0 %

Acid-insoluble Ash Not more than 2.0 %

Extract Content ***Dilute ethanol-soluble extracts*** Not less than 20.0 %

Containers and Storage ***Containers*—**Well-closed containers.

Magnolia Bark

Magnoliae Cortex

Magnolia Bark is the bark of the trunk of *Magnolia obovata* Thunberg, *Magnolia officinalis* Rehder et Wilson or *Magnolia officinalis* Rehder et Wilson var. *biloba* Rehder et Wilson (Magnoliaceae). Magnolia Bark contains not less than 0.8 % of magnolol ($C_{18}H_{18}O_2$: 266.33). Magnolia Bark contains not less than 1.0 % in total of magnolol ($C_{18}H_{18}O_2$: 266.33) and honokiol ($C_{18}H_{18}O_2$: 266.33).

Description Magnolia Bark is plate-like or semi-cylindrical bark, 2 mm to 7 mm in thickness. External surface is grayish white to grayish brown and rough, sometimes cork layer removed and externally red-brown. Interior surface is pale brown to dark purplish brown. Cut surface is extremely fibrous and it is pale red-brown to purple-brown. Under a microscope, a transverse section reveals a thick cork layer or several thin cork layers and internally adjoining the circular tissue of stone cells of approximately equal diameter. Primary cortex is thin. Fiber groups are 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.

Magnolia Bark has a slight odor and bitter taste.

Identification Weigh 1 g of pulverized Magnolia Bark, add 10 mL of methanol, shake for 10 minutes, centrifuge and use the supernatant liquid 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). Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 μL of the test solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of *n*-hexane and ethyl acetate (3 : 1) 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: two of thespots obtained from the test solution show the same color and R_f value as the spots from test solution (1) and test solution (2).

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 6.0 %.

Assay Weigh accurately about 0.1 g of pulverized Magnolia Bark, add 100 mL of diluted methanol (7 in 10), sonicate to extract for 20 minutes and filter. Combine 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 not less than 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, A_{Ta} and A_{Tb}, of magnolol and honokiol in the test solution, and the peak areas, A_{Sa} and A_{Sb}, of magnolol and honokiol 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 steel column, 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel (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) (70 : 30 : 1)

Flow rate: 0.3 mL/minute

System suitability

System performance: Dissolve l mg each of Magnolol RS and Honokiol RS in diluted methanol (7 in 10) to make 10 mL. When the procedure is run with 10 μL of this solution under the above operating conditions, honokiol and magnolol are eluted in this order with a resolution between their peaks being not less than 5.0.

System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions, the relative standard deviation of each peak area of magnolol and honokiol is not more than 1.5 %.

Containers and Storage ***Containers*****—**Well-closed containers.

Mentha Herb

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 with opposite leaves. Stem is square column, 15 cm to 40 cm in length and 0.2 cm to 0.4 cm in diameter. The external surface is purplish brown to pale green with short hairs near the edge and spacing of 2 cm to 5 cm between nodes. Texture is weak. The cut surface is white and the pith is hollow in the middle. The leaves are opposite with short petioles, and the leaf lobes are crushed and rolled together. A whole leaf, when smooth, is long ovoid to ovoid, 2 cm to 7 cm in length and 1 cm to 3 cm in width. The front surface of the leaf is deep green, the back surface is grayish green, rarely covered in short hairs, with concave spot-like scales. The flowers are in axillary verticils, the calyx is bell-shaped, dividing into 5 at the tip, and the corolla is pale purple.

Mentha Herb has a characteristic, refreshing aroma when rubbed by hand, tastes pungent and gives a cool feeling.

Identification Take l mL of the mixture of essential oil and xylene, obtained in the Essential oil content, and add carefully 2 mL of sulfuric acid to make two layers: a deep red to red-brown color develops at the zone of contact.

Purity (1) ***Foreign matter*****—**The amount of roots and other foreign matter contained in Mentha Herb is less than 2.0 %.

(2) ***Heavy metals*****—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*****—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Methoxychlor: Not more than 1 ppm.

(iv) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(v) Aldrin: Not more than 0.01 ppm.

(vi) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): Not more than 0.2 ppm.

(vii) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 15.0 % (6 hours).

Ash Not more than 11.0 %.

Acid-insoluble Ash Not more than 2.5 %.

Essential Oil Content Not less than 0.4 mL (50.0 g, 1 mL of silicon resin).

Containers and Storage ***Containers*****—**Well-closed containers.

Morinda Root

Morindae Radix

Morinda Root is the root of *Morinda* officinalis How (Rubiaceae), from which the rootlets removed. Morinda Root is pressed flatly and dried.

Description Morinda Root is compressed cylindrical root, 5 mm to 20 mm in diameter, somewhat bented and varying in length. External surface is grayish yellow or dark gray, with longitudinal wrinkles and transverse cracks. Some bark is transversly broken and xylem is exposed. Texture is tough. The transverse section has a thick cortex, purple or pale purple and easily separatled 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.
Morinda Root is odorless and has a sweet, slightly astringent taste.

Identification Weigh 2.0 g of pulverized Morinda Root, add 15 mL of ethanol, warm on a water bath for 1 hour with a reflux condenser and filter. Evaporate the filtrate to dryness, dissovle the residue to 1 mL of ethanol and use this solution as the test solution. Perform the test with the test solution as directed under Thin-layer Chromatography. Spot 10 μL of the test solution on a plate of slica gel for thin-layer chromatography. Develop the plate with a mixture of hexane and ethyl acetate (3 : 2) to a distance of about 10 cm and air-dry the plate. Spray dilute sulfuric acid TS, and heat at 105 °C for l0 minutes: a purple spot appears at about 0.6 of R_f value.

Purity (1) ***Foreign Matter*—**Xylem: The amount of the xylem contained in Morinda Root is not more than 35.0 %.

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 13.0 %

Ash Not more than 6.0 %

Acid-insoluble Content Not more than 1.0 %

Extract Content ***Dilute ethanol-soluble extract*—** Not less than 52.0 %

Containers and Storage ***Containers*—**Well-closed containers.

Moutan Root Bark

Moutan Radicis Cortex

Moutan Root Bark is the root bark of *Paeonia suffruticosa* Andrews (Paeoniaceae). Moutan Root Bark, when dried, contains not less than 1.0 % of paeonol ($C_9H_{10}O_3$: 166.17).

Description Moutan Root Bark is the root bark, cylindical to semi-cylindrical, slightly curved inwards or longitudinally open 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 external surface is grayish brown to yellowish brown with several transversely long lenticels and scars of rootlets. Those without the outer bark are pink on the outside. The inside is pale grayish yellow or brown, not dark, usually with sparkling crystals. The texture is firm and easy to cut. The cut surface is pale pink and powdery.
Moutan Root Bark has a characteristic odor and slight 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 filtrate as the test solution and Moutan Root Bark RMPM standard solution. Perform the test with the test solution and Moutan Root Bark RMPM standard solution as directed under the Thin-layer Chromatography. Spot 10 μL of the test solution and Moutan Root Bark RMPM standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of hexane and ethyl acetate (1 : 1) to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): several spots from the test solution and the spots from Moutan Root Bark RMPM standard solution are 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 not more than 1.0 %.

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(vi) Chlorpyrifos:Not more than 0.5 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 6.0 %.

Acid-insoluble Ash Not more than 1.0 %.

Assay Weigh accurately about 0.3 g of pulverized Moutan Bark, add 40 mL of methanol, heat under a reflux condenser in a water-bath for 30 minutes, cool and filter. Repeat the above procedure with the residue, using 40 mL of methanol. 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, dry Paeonol RS in a desiccator (calcium chloride for dryness) for more than l hour. Weigh accurately about 10 mg of it, dissolve 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 the Liquid Chromatography according to the following operating conditions and determine the peak areas, A_T and A_S, of paeonol for 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 ge1 (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 paraoxybenzoate in 25 mL of methanol. When the procedure is run with 10 μL of this solution under the above operating conditions and calculate the resolution, paeonol and butylparaben are eluted in this order, with the resolution between their peaks being not less than 2.0.

System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions, the relative deviation of the peak area of paeonol is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Mulberry Root Bark

Mori Radicis Cortex

Mulberry Root Bark is the root bark of *Morus alba* Linné (Moraceae), from which 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 is white to pale yellow-brown, and its periderm is yellow-brown, easy to peel, with numerous longitudinal, fine wrinkles and numerous red-brown lenticels, in the case of the bark with periderm. The inner surface is yellowish white or grayish yellow with a fine longitudinal pattern. The body is light and tough, strongly fibrous and difficult to cut. The cut surface is white to pale brown and fibrous. Under a microscope, the transverse section 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 bark many years of age contain a small number of stone cell groups, most cell cavities containing prismatic crystals. Stone cell groups are intermittently arranged inside the phloem to form a ring shape.

Mulberry Root Bark has a slight, characteristic odor and is nearly tasteless.

Identification (1) Weigh 1 g of pulverized Mulberry Root Bark, add 20 mL of hexane, boil under a reflux condenser in a water–bath for l5 minutes and filter. Evaporate the filtrate to dryness, dissolve the residue in l0 mL of chloroform, mix 0.5 mL of the solution with 0.5 mL of acetic anhydride in a test tube and add carefully 0.5 mL of sulfuric acid to make two layers: 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 of methanol, heat in a water-bath for 30 minutes, cool and filter. Evaporate the filtrates to dryness, dissolve the residues in 1 mL of ethanol and use these solutions as the test solution and the Mulberry Root Bark RMPM standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 μL each of the test solution and the Mulberry Root RMPM standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of cyclohexane and ethyl acetate (3 : 1) to a distance of about 10 cm and air-dry the plate. Spray evenly diluted sulfuric acid TS and heat 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 Mulberry Root RMPM standard solution and of these, a purple spot appears at the R_f value of 0.5.

Purity (1) ***Foreign matter***—The amount of the root

xylem and other foreign matter contained in Mulberry Root Bark is not more than l.0 %.

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 11.0 %.

Acid-insoluble Ash Not more than 1.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Mume Fruit

Mume Fructus

Mume Fruit is the unripe fruit of the *Prunus mume* Siebold et Zuccarini (Rosaceae) and fumed.

Description Mume Fructus is usually the spherical or spheroid fruit, 2 to 3 cm in length, 15 to 20 mm in diameter. The external surface is black to blackish brown, wrinkled, with a circular fruit stem scar at the bottom. The core is very hard, elliptic, yellow-brown with many dented spots on the outer surface, 10 to 14 mm in length, 10 mm in diameter and 5 mm in thickness. The seed is flattened ovoid and pale yellow.
Mume Fructus has a characteristic odor and acidic taste.

Identification (1) Weigh 0.5 g of pulverized Mume Fructus, add 10 mL of water, shake well for 5 minutes and filter. Add dilute hydrochloride to the filtrate to acidify. To the vaporized product, add water: a white precipitate is produced after adding acetate-lead TS.

(2) Weigh 1.0 g of pulverized Mume Fructus, add 2 mL of acetic anhydride, shake for 5 minutes. To 1 mL of the filtrate, add gently 0.5 mL of sulfuric acid to form two layers: a reddish brown color develops at the zone of contact and a dark greenish-brown at the upper layer.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 19.0 % (6 hours).

Ash Not more than 5.0 %

Acid-insoluble Ash Not more than 1.5 %.

Extract Content ***Dilute ethanol-soluble extract***—Not less than 27.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Myrrh

Myrrha

Myrrh is the gum-resin collected from the *Commiphora myrrha* Engler or *Commiphora molmol* Engler (Burseraceae). The former is known as Gum Myrrh, and the latter Gum Opoponax.

Description (1) ***Gum Myrrh***—Gum Myrrh appears as irregular granular masses, varying in size, larger ones being 6 cm or more in diameter. The external surface is yellowish brown or reddish brown, those that are close to translucent are blackish brown, covered in yellow dust. The texture is brittle and the fractured surface is uneven and without luster.
Gum Myrrh has a characteristic aroma and tastes bitter and slightly pungent.

(2) ***Gum Opoponax***—Gum Opoponax appears as irregular masses, granular, mostly adhered to each other, forming masses of varying sizes, larger ones reaching 6 cm in diameter. The external surface is yellowish brown to maroon and opaque. The texture is solid or sparse.

Identification (1) Triturat Myrrh with water: a yellowish brown emulsion is produced.

(2) Weigh 1 g of pulverized Myrrh, add 5 mL of ether, vortex and filter. The filtrate is evaporated to dryness: the residue becomes purplish red when contacted with bromine gas.

Purity ***Ethanol-insoluble substances***—Weigh accurately about 2 g of finely pulverized Myrrh, add 30 mL of ethanol and warm for 30 minutes with occasional stirring. Ethanol extract is filtered with pre-weighed filtrator, repeat the above extract procedure three times

with the residue with 15 mL of each ethanol for 5 minutes. The residue on the filtrator is washed several times with 5 mL of warm ethanol: the remaining insoluble substance is not more than 70 % after oven drying at 100 °C and cooling in the desiccator (silica gel).

Ash Not more than 15.0 %.

Acid-insoluble Ash Not more than 5.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Nelumbo Seed

Nelumbinis Semen

Nelumbo Seed is the well-ripe seed of *Nelumbo nucifera* Gaertner (Nymphaeaceae), usually with the endocarp, sometime being removed the 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 is pale yellow-brown to red-brown with fine longitudinal striations and relatively wide veins. The center of one end is papillate, deep brown, mostly with cracks, somewhat dented around edge. Seed coat is thin, yellowish brown and hard to peeled off. Two plump cotyledons are yellowish white with green embryo at the space between two cotyledons.
Nelumbo Seed is nearly odorless and slightly oily and has sweet taste. Its embryo is extremely bitter.

Identification (1) Weigh 1.0 g of pulverized Nelumbo Seed, add 10 mL of dilute acetic acid, heat for 3 minutes with occasional stirring in a water-bath, cool and filter. Add 2 to 3 drops of Dragendorff's TS to 2 mL of the filtrate: a brownish-yellow precipitate is produced.

(2) Shake thoroughly a small quantity of pulverized Nelumbo Seed and some water and add several drops of iodide solution: a bluish-purple color is produced, and the color abate gradually after heating, allow to cool, the blue-purple color produces again.

(3) Weigh 0.5 g of pulverized Nelumbo Seed, add 0.5 mL of water, shake for 5 minutes, and centrifuge. To 0.5 mL of the supernatant liquid add 1 droplet of 1-naphthol solution, mix well and add gently 1 mL of sulfuric acid: a purple color ring is produced at the contact of the two layers.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

(4) ***Mycotoxins***—Total aflatoxin (sum of aflatoxins B_1, B_2, G_1 and G_2): Not more than 15.0 ppb (aflatoxin B_1 is not more than 10.0 ppb).

Ash Not more than 5.5 %.

Extract Content ***Dilute ethanol–soluble extract***— Not less than 12.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Nutmeg

Myristicae Semen

Nutmeg is the ripe seed of *Myristica fragrans* Houttuyn (Myristicaceae). The aril and seed coat is removed when used.

Description Nutmeg is the ovoid or ellipsoidal seed, 2 cm to 3 cm in length and 1.5 cm to 2.5 cm in diameter. The external surface is yellowish brown to grayish yellow, the outer coat sometimes covered in white powder. The entire seed has longitudinal furrows of a pale color and irregular reticular furrows. The hilum is located at the end of the broad area, appearing as a circular projection of a pale color, and the chalaza is dented and appears dark. The seed ridge is a longitudinal furrow and the single furrow connecting the two ends is shallow and wide. Thin, narrow, reticular furrows are visible throughout. A small endosperm is bent near the hilum. Under a magnifying glass, a transverse section shows thin, dark brown perisperm. The perisperm has a number of irregular protrusions and inserted to the pale yellowish white endosperm to make marble-like striations. Under a microscope, the perisperm is divided into the inner layer and outer layer. The outer layer has flat cells containing brown substances. The inner layer has rectangular cells containing red-brown substances, protruding into the endosperm to form an irregular tissue. This tissue has one vascular bundle and is scattered with multiple oil cells. The oil cells contain essential oil. The endosperm cells are polygonal and contain large amounts of fatty oil, starch grains and aleurone grains. The aleurone grains contain pseudocrystals. The endosperm is scattered with cells containing brown substances.
Nutmeg has a characteristic odor and tastes pungent and slightly bitter.

Identification (1) Weigh 1 g of pulverized Nutmeg, add 10 mL of methanol, warm for 3 minutes in a water-bath and filter while warm. Stand the filtrate for 10 minutes in an ice water-bath: a white precipitate is produced.

(2) Dissolve precipitate produced in (1) 5 mL of chloroform and use this solution as the test solution. Perform the test with this solution as directed under the Thin-layer Chromatography. Spot 2 μL of the test solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of hexane and acetone (4:1) to a distance of about l0 cm and air-dry the plate. Expose the plate to iodine vapor: a yellow spot appears in the region of R_f value 0.3.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

(4) ***Mycotoxins***—Total aflatoxin (sum of aflatoxins B_1, B_2, G_1 and G_2): Not more than 15.0 ppb (aflatoxin B_1 is not more than 10.0 ppb).

Ash Not more than 3.0 %.

Essential Oil Content Not less than 0.5 mL (10.0 g).

Containers and Storage ***Containers*—**Well-closed containers.

Nux Vomica

Strychni Semen

Nux Vomica is the well ripe seed of *Strychnos nux-vomica* Linné (Loganiaceae). Nux Vomica, when dried, contains not less than 1.05 % of strychnine ($C_{21}H_{22}N_20_2$: 334.42).

Description Nux Vomica is button-shaped, 1 cm to 3 cm in diameter and 3 mm to 5 mm in thickness, and prominent at the one side and slightly dented at the other side. External surface is pale grayish yellow-green to pale grayish brown, covered densely with lustrous suppressed hairs radiating from the center to the circumference. On both sides, the margin and the central part are bulged a little. The dot-like micropyle is situated at one point on the margin and from which usually a raised line runs to the center on one side. Texture is extremely hard. When cracked upon soaking in water, the seed coat is thin and the interior consists of two horny, pale grayish yellow endosperms and leaving a central narrow cavity at the center. A white embryo, about 7 mm in length, is situated at one end between the inner surfaces of the endosperms.

Nux Vomica is nearly odorless and it has very bitter and persisting taste.

Identification (1) 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 in a water-bath, add 5 mL of diluted sulfuric acid (1 in 10) and warm in a water-bath while shaking well until the odor of chloroform is no longer perceptible. After cooling, filter through a pledget of absorbent cotton and add 2 mL of nitric acid to l mL of the filtrate: a red color develops.

(2) Take the remaining filtrate obtained in (l), add 1 mL of potassium bichromate TS and allow to stand for 1 hour: a yellow-red precipitate is produced. Collect the precipitate by filtration and wash with l mL of water. Transfer a part of the precipitate to a small test tube, add l mL of water, dissolve by warming, cool and add 5 drops of sulfuric acid drop-wise carefully along the wall of the test tube: the layer of sulfuric acid shows a purple color which turns immediately form red to red-brown.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

Ash Not more than 3.0 %.

Assay Weigh accurately about 1 g of pulverized Nux Vomica, previously dried at 60 °C for 8 hours, place in a glass-stoppered centrifuge tube and moisten with l mL of strong ammonia water. To this solution, add 20 mL of ether, stopper the centrifuge tube tightly, shake for 15 minutes, centrifuge and separate the supernatant liquid. Repeat this procedure three times with the residue using 20 mL volumes of ether. Combine all the extracts and evaporate the ether in a water-bath. Dissolve the residue in 10 mL of the mobile phase, add exactly 10 mL of the internal standard solution and add the mobile phase to make exactly 100 mL. Filter this solution through a membrane filter with a porosity not

more than 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 (determined the loss on drying before use) and dissolve in the mobile phase to make exactly 50 mL. Pipet 10.0 mL of this solution, add exactly 10 mL of the internal standard solution, then add the mobile phase to make exactly 100 mL and use this 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 following operating conditions. Determine the ratios, Q_T and Q_S, of the peak area of strychnine to that of the internal standard for the test solution and the standard solution, respectively.

$$\text{Amount (mg) of strychnine } (C_{21}H_{22}N_2O_2) = \text{amount (mg) of Strychnine Nitrate RS, calculated on the dried basis} \times \frac{Q_T}{Q_S} \times \frac{1}{5} \times 0.8414$$

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 monobasic potassium phosphate in water to make 1000 mL and a mix with acetonitrile and triethylamine (45 : 5 : 1) and adjust the mixture with phosphoric acid to a pH of 3.0.

Flow rate: Adjust the flow rate so that the retention time of strychnine is about l7 minutes.

Selection of column: Proceed with 5 μL of the standard solution under the above operating conditions, use a column giving elution of the internal standard and strychnine in this order and clearly dividing each peak

Containers and Storage ***Containers*—**Well-closed containers.

Nux Vomica Extract

Nux Vomica Extract contains 6.15 % to 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, digest by the percolation method, 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 the extracts and prepare the dry extract as directed under Extracts. May be prepared with an appropriate quantity of ethanol and purified water.

Description Nux Vomica Extract is yellow-brown to brown powder. Nux Vomica Extract has a characteristic odor and an extremely bitter taste.

Identification Extract 0.5 g of Nux Vomica Extract with 0.5 mL of ammonia TS and 10 mL of chloroform with occasional shaking. Filter the chloroform extract, evaporate the filtrate on a water-bath until most of the chloroform is removed and proceed as directed in the Identification under Nux Vomica.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

Assay Weigh accurately about 0.2 g of Nux Vomica Extract, place in a glass-stoppered centrifuge tube, add 15 mL of ammonia TS and shake. Add 20 mL of ether, stopper tightly, shake for 15 minutes, centrifuge to disperse the ether layer. Repeat this procedure 3 times with the water layer, using 20-mL volumes of ether. Combine the extracts and evaporate the 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 the mobile phase to make exactly 100 mL. Proceed as directed in the Assay under Nux Vomica.

$$\text{Amount (mg) of strychnine } (C_{21}H_{22}N_2O_2) = \text{amount (mg) of Strychnine Nitrate RS, calculated on the dried basis} \times \frac{Q_T}{Q_S} \times \frac{1}{5} \times 0.8414$$

Internal standard solution—A solution of barbital sodium in the mobile phase (1 in 500).

Containers and Storage ***Containers***—Tight containers.

***Storage*—**Light-resistant.

Ostericum Root

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* Boissier (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 cm to 30 cm in length and 2 cm to 5 cm in diameter. The external surface is yellow-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 frond bases. The texture is hard but fragile. The transverse section has a pale brown or yellow-brown cortex, is relatively sparse with several clefts and white or yellowish white xylem. Under a microscope, the outermost layer of the root consists of 3 to 4 rows of cork cells and underneath are 4 to 8 layers of collenchymas. Secretory canals are sparsely arranged in the collenchymas or 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 numerous at the cortex and the parenchyma is filled with starch grains.
Ostericum Root from *Ostericum koreanum* has a characteristic odor and sweet and cooling taste at first and followed by a slight bitterness.

(2) ***Notopterygium incisum***—Ostericum Root from *Notopterygium incisum* consists of rhizome and root, somewhat curved cylindrical, occasionally branched, 4 cm to 13 cm in length and 6 mm to 25 mm in diameter. The external surface is maroon to blackish brown and yellow where it has been peeled. Those with short nodes and tightly packed ring patterns giving the appearance of a silkworm are called Jamgang, and those with long nodes giving the appearance of bamboo joints are called Jukjeolgang. The nodes have marks of roots protruding in the shape of spots or nodules, is brown with several broken scale pieces. The body is light and has a fragile texture, making it easy to cut. The transverse section is irregular with several clefts. The cortex is yellow-brown to dark brown and slippery with brown oil drops. The xylem is yellowish white with clear medullary rays. The pith is yellow to yellow-brown. Under a microscope, the transverse section has a cork layer consisting of about 10 rows of cork cells. The cortex is narrow with several clefts in the phloem and the cambium forms a ring. It has a relatively large number of xylem vessels. The oil sacs are large and particularly numerous at the phloem and also present at the pith and medullary rays. The oil sacs contain a yellow-brown oil-like substance.
Ostericum Root from *Notopterygium incisum* has an odor and a slightly bitter and pungent taste.

(3) ***Notopterygium forbesii***—Ostericum Root from *Notopterygium forbessi* is the rhizome and root. The rhizoma is close to cylindrical and the top of the rhizoma has scars of stem or leaf sheath. The root is conical with longitudinal wrinkles and lenticels. The external surface is maroon with relatively dense circular patterns near the rhizoma. Roots 8 cm to 15 in length and 1 cm to 3 cm in diameter are called Jogang. Thick, large, irregularly knotted rhizoma with several stem bases at the top and relatively thin roots are called Daedugang. Texture is lax and weak, making it easy to cut. The cut surface is slightly flat, the cortex is pale brown and the xylem is yellowish white. Under a microscope, the transverse surface reveals that the outermost layer of the root consists of 3 to 4 rows of cork cells, and underneath are 4 to 8 layers of collenchymas with secretory canals. The phloem has a sparse arrangement of secretory canals. 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 numerous at the cortex and the parenchyma is filled with starch grains.
Ostericum Root from *Notopterygium forbessi* has a slight odor and a relatively plain taste.

Identification Weigh 1 g of pulverized Ostericum Root, add 10 mL of ether, and extract this at ordinary temperature. Examine the extract solution under ultraviolet light; it has strong fluorescence.

Purity (1) ***Foreign matter***—Ostericum Root contains not more than 5.0 % of stem base and other foreign matters.
(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): Not more than 0.2 ppm.
(vi) Endrin: Not more than 0.01 ppm.
(vii) Oxolinic acid: Not more than 7.0 ppm.
(4) ***Sulfur dioxide***—Not less than 30 ppm.

Loss on Drying Not more than 13.0 %.

Ash Not more than 10.0 %.

Acid-insoluble Ash Not more than 2.0 %.

Extract Content *Dilute ethanol-soluble extract*—Not less than 20.0 %.

Containers and Storage *Containers*—Well-closed containers.

Oyster Shell

Ostreae Testa

Oyster Shell is the shell of *Ostrea gigas* Thunberg, *Ostrea talienwhanensis* Crosse or *Ostrea rivularis* Gould (Ostreidae).

Description (1) ***Oyster Shell of Ostrea gigas***—Oyster Shell from *Ostrea gigas* is the shell consisting of long pieces, the dorsal and ventral edges almost parallel, 10 cm to 50 cm in length and 4 cm to 15 cm in height. The right valve is relatively small and the scales are firm and thick, layered or lamellar. The external surface is flat or has several dents, pale purple, grayish white or yellowish brown, the inside is white, both apexes without fine serration. The left valve is deeply concave, the scales of the right valve are relatively coarse and large, the apexes of the shell attached to each other in a small area. The texture is fragile and the cross-section forms layers, white in color.
Oyster Sheel is nearly odorless and tastes slightly saline.

(2) ***Oyster Shell of Ostrea talienwhanensis***—Oyster shell from *Ostrea talienwhanensis* is the shell, subtriangular, dorsal and ventral edges are V-shaped. The outer surface of the right valve is pale yellow and concentric scales are arranged loosely and the bottom of scales are undulated. The inner surface is white. The left valve bears strong and thick concentric scales, with several distinct ribs radiated from the apex of the shell. The inner surface is concaved in box-shaped. Hinge surface is small.

(3) ***Oyster Shell of Ostrea rivularis***—Oyster shell from *Ostrea rivularis* is the shell, rounded, oval or triangular. The external surface of the right valve is uneven, gray, purple, brown and yellow, concentric scales forming a ring. It is thin and fragile when immature and overlapped one another after several years growth. The inner surface is white and sometimes its edge is pale purple.

Identification (1) Weigh l g of pulverized Oyster Shell, dissolve in 10 mL of dilute hydrochloric acid by 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 solution obtained in (1) has a slight, characteristic odor. Filter this solution and neutralize with ammonia TS: the solution responds to the Qualitative Tests for calcium salt.

(3) Ignite l g of pulverized Oyster Shell: it turns blackish brown at first and evolves characteristic odor. Ignite it further: it becomes almost white.

Purity ***Barium***—Weigh 1 g of pulverized Oyster Shell, dissolve in 10 mL of dilute hydrochloric acid: the solution does not respond to the Qualitative Tests (1) for barium salt.

Loss on Drying Not more than 4.0 % (1 g, 180 °C, 4 hours).

Containers and Storage ***Containers—***Well-closed containers.

Peach Kernel

Persicae Semen

Peach Kernel is the ripe seed of *Prunus persica* Batsch or *Prunus davidiana* Franchet (Rosaceae). Peach Kernel, when dried, contains not less than 0.5 % of amygdalin ($C_{20}H_{27}NO_{11}$: 457.43).

Description Peach Kernel is the seed, flattened, asymmetric ovoid seed, 12 mm to 20 mm in length, 6 mm to 12 mm in width and 3 mm to 7 mm in thickness. The shape of the seed is somewhat sharp at one end and round at the other end with chalaza. Seed coat is red-brown to pale brown and its surface is powdery and easily detachable with stone cells of epidermis. Dented longitudinal wrinkles are distributed from the chalaza through the seed coat. When soaked in boiling water and softened, the seed coat and thin, translucent, white endosperm are easily separated from the cotyledons. There are 2 cotyledons, close to white and very oily. Under a microscope, the outer surface of the seed coat reveals protruding stone cells, polygonal, long polygonal or obtuse triangular according to position and their membranes almost equally thickened.
Peach Kernel has a slight, characteristic odor and tastes bitter.

Identification Weigh 1 g each of pulverized Peach Kernel or Peach Kernel RMPM, add 10 mL of methanl and heat under a reflux condenser in a water bath for 10 minutes. After then, filter and use these as the test solution and Peach Kernel RMPM standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 μL each of the test solution and the Peach Kernel RMPM standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, methanol and water (12 : 2 : l) 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 for 10 minutes: the spots from the test solution and the spots from Peach Kernel RMPM standard solution show the same color and the same R_f value, and 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 endocarp or other foreign matter.

(2) ***Heavy metals—***(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides—***(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(vi) Chlorothalonil: Not more than 0.1 ppm.
(4) ***Sulfur dioxide***—Not more than 30 ppm.
(5) ***Mycotoxins***—Total aflatoxin (sum of aflatoxins B_1, B_2, G_1 and G_2): Not more than 15.0 ppb (aflatoxin B_1 is not more than 10.0 ppb).
(6) ***Rancidity***—Grind Peach Kernel with boiling water: no odor of rancid oil is perceptible.

Assay Weigh accurately about 2.0 g of pulverized Peach Kernel, add 50 mL of methanol, heat with a reflux condenser for 3 hours and filter. Repeat the above procedure with the residue using 50 mL of methanol. Combine the whole filtrates, evaporate to dryness in vaccum. Add 70 mL of water and 70 mL of hexane to the residue, shake well and discard the hexane layer. Add 70 mL of ether to the water layer, shake and discard the ether layer. The remaining water layer is filtered, adjust the total volume to 100 mL and use this solution as the test solution. Seperately, dry the Amygdalin RS for 24 hours in the desiccator (silica gel) and weigh accurately about 10 mg, dissolve in 100 mL of water and use this solution as 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 amygdalin for the test solution and the standard solution, respectively.

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

Operating conditions
Detector: An ultraviolet absorption photometer (wavelength: 214 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 l0 μm in particle diameter).
Column temperature: An ordinary temperature.
Mobile phase: A mixture of water and methanol (80:20).
Flow rate: 1.0 mL/minute.
System suitability
System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions, the relative standard deviation of the peak area of amygdalin is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Peony Root

Paeoniae Radix

Peony Root is the root of *Peonia lactiflora* Pallas or allied plants (Paeoniaceae). Peony Root contains not less than 2.3 % of total sum 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 cm to 20 cm in length and 10 mm to 25 mm in diameter, and large root is cut lengthwise. External surface is white or brown, with distinct longitudinal wrinkles, often with wrinkles or scars of lateral roots and with laterally elongated lenticels. The upper part of root often remains scars of stems or unremoved brown cortex. Texture is hard, difficult to fracture. The transverse section is granular and very dense. Under a magnifying glass, cambium is distinct, milky white or brown, and radial pith is observed.
Peony Root has a characteristic odor and taste, slightly sweet at first, followed by an astringency and a slight bitterness.

Identification (1) Weigh 0.5 g of pulverized Peony Root, add 30 mL of ethanol, shake for 15 minutes and filter. Shake 3 mL of the filtrate with l drop of iron (III) chloride TS: a blue-purple to blue-green color is produced and it changes to dark blue-purple to dark green.
(2) Weigh 2 g each of pulverized Peony Root and Peony Root RMPM, add 10 mL of methanol, warm in a water-bath for 5 minutes, cool, filter and use the filtrates as the test solution and the Peony Root RMPM standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 μL each of the test solution and the Peony Root RMPM standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of acetone, ethyl acetate and acetic acid (100) (10:10:1) 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 spots obtained from the test solution show the same color and R_f value as the spots from the Peony Root RMPM standard solution and of these, the spot of paeoniflorin appears at the R_f value of 0.25.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides*—**(i) Napropamide—Not more than 0.1 ppm.
(ii) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(iii) Dieldrin: Not more than 0.01 ppm.
(iv) Myclobutanil: Not more than 0.1 ppm.

(v) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(vi) Cyprodinil: Not more than 0.1 ppm.
(vii) Aldrin: Not more than 0.01 ppm.
(viii) Endrin: Not more than 0.01 ppm.
(ix) Iminoctadine: Not more than 0.3 ppm.
(x) Carbendazim: Not more than 0.05 ppm.
(xi) Triadimenol: Not more than 0.1 ppm.
(xii) Triadimefon: Not more than 0.5 ppm.
(xiii) Triforine: Not more than 0.1 ppm.
(xiv) Triflumizole: Not more than 1.0 ppm.
(xv) Pendimethalin: Not more than 0.2 ppm.
(xvi) Propineb: Not more than 0.2 ppm.
(xvii) Fludioxonil: Not more than 0.1 ppm.
(xviii) Dithianon: Not more than 0.3 ppm.
(xix) Azoxystrobin: Not more than 0.1 ppm.
(xx) Cadusafos: Not more than 0.01 ppm.
(xxi) Terbufos: Not more than 0.05 ppm.
(xxii) Thiram: Not more than 0.2 ppm.
(xxiii) Fenarimol: Not more than 0.1 ppm.
(xxiv) Fosthiazate: Not more than 0.01 ppm.
(xxv) Prochloraz: Not more than 0.1 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 14.0 % (6 hours).

Ash Not more than 6.5 %.

Assay Weigh accurately about 0.5 g of pulverized Peony Root, add 50 mL of diluted methanol (1 in 2), heat with a reflux condenser in 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 the filtrates, add diluted methanol (1 in 2) to make exactly 100 mL and use this solution as the test solution. Separaetly, weigh accurately about 10 mg each of Paeoniflorin RS (previously dried in a silica gel desiccator for 24 hours) and Albiflorin RS (previously dried in a silica gel desiccator for 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 Liquid Chromatography according to the following operating 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 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 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica gel (5 μm to 10 μm in diameter).

Column temperature: An ordinary temperature.

Mobile phase: Control gradually or concentration-gradiently with mobile phase A and B as follows.

Mobile phase A: water

Mobile phase B: acetonitrile

Time (min)	Mobile phase A (%)	Mobile phase B (%)
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: Perform the test with the standard solution under the above operating conditions. Use a column giving elution of albiflorin and paeoniflorin in this order with the resolution between their peaks being separated completely.

System repeatability: When the test is repeated 6 times with 20 μL each of the standard solution under the above operating conditions: the relative standard deviation of the peak area of paeoniflorin is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Perilla Leaf

Perillae Folium

Perilla Herb is the leaf and twig of *Perilla frutescens* Britton var. *acuta* Kudo or *Ferilla frutescens* Britton var. *crispa* Decaisne (Labiatae).

Description Perilla Herb is leaves and twigs. Both surfaces of the leaf are brownish purple, or the upper surface is grayish green to brownish green and the lower surface is brownish purple. When smoothed by immersion in water, the lamina is ovate to obcordate, 5 to 12 cm in length and 5 cm to 8 cm in width. The apex is acuminate and the margin is serrate. The base is broadly cuneate, and has petiole, 3 cm to 5 cm in length. Cross sections of stem and petiole are square. Under a magnifying glass, hairs are observed on both surfaces of the leaf, but abundantly on the vein and

sparsely on other parts. Small glandular hairs are observed on the lower surface.
Perilla Herb has a characteristic odor and slightly bitter taste.

Identification Take 0.3 mL of a mixture of essential oil and xylene, obtained in Essential oil content, add l mL of acetic anhydride, shake and add l drop of sulfuric acid: a red-purple to dark red-purple color develops.

Purity (1) ***Foreign matter*—**(i) Stem: The amount of its stems, which are not less than 3 mm in diameter, contained in Perilla Herb is not more than 3.0 %.

(ii) Other foreign matter: The amount of foreign matter other than the stems is not more than 1.0 %.

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 13.0 % (6 hours).

Ash Not more than 16.0 %.

Acid-insoluble Ash Not more than 2.5 %.

Essential Oil Content Not less than 0.2 mL (50.0 g, 1 mL of silicon resin).

Containers and Storage ***Containers*—**Well-closed containers.

Pharbitis Seed

Pharbitidis Semen

Pharbitis Seed is the ripe seed of *Pharbitis nil* Choisy or *Pharbitis purpurea* Voigt (Convolvulaceae).

Description Pharbitis Seed is the seed like longitudinally quartered or sexpartite globe, 6 mm to 8 mm in length and 3 mm to 5 mm in width. External surface is black to grayish reddish brown or grayish white, smooth or slightly shrunken. The transverse section is almost fan-shaped, pale yellow-brown to pale grayish brown 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. Seed coat is thin and the outer layer is dark gray and the inner layer is pale gray. Two irregularly folded cotyledons and two thin membranes from the center of the dorsal side to the ridge separating cotyledons are present in the transverse section of one end, but they are not observed in the transverse section of the other end with the hilum. Dark gray secretary pits are located in the section of the cotyledon.
When cracked, Pharbitis Seed has a characteristic odor and oily and slightly pungent taste.

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. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 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 dichloromethane, methanol and formic acid (10:9:4) 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: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 6.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Phellodendron Bark

Phellodendri Cortex

Phellodendron Bark is the bark of *Phellodendron amurense* Ruprecht or *Phellodendron chinense* Schneide (Rutaceae), from which the periderm has been removed.
Phellodendron Bark contains not less than 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 to semi-cylindrical pieces of bark, 2 mm to 4 mm in thickness, 5 to 15 cm in width and 20 cm to 40 cm in length, and sometimes fragments are mixed. External surface is grayish yellow-brown to grayish brown, with numerous traces of lenticel. The internal surface is yellow to dark yellow-brown, with five vertical lines and smooth. 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 yellow-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 tangential direction, crossed over the secondary medullary rays and then these tissues show a latticework.
Phellodendron Bark has a slight, characteristic odor and extremely bitter taste and is mucilaginous. Phellodendron Bark colors the saliva yellow on chewing.

Identification (1) 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. To 2 to 3 drops of the filtrate, add 1 mL of hydrochloric acid, add 1 to 2 drops of hydrogen peroxide TS and shake: a red–purple color develops.

(2) Use the filtrate obtained in (1) as the test solution. Separately, weigh 1 mg of Berberine Chloride RS, dissolve in l mL of methanol and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 5 μL each of the test solution and the standard solution on a plate of silica gel with a fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of *n*-butanol, water and acetic acid (100) (7 : 2 : 1) to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 365 nm): one spot among the spots from the test solution and a spot with yellow to yellow-green fluorescence from the standard solution show the same color and the same R_f value.

(3) Stir up pulverized Phellodendron Bark with water: the solution becomes gelatinous owing to mucilage.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 11.0 % (105 °C, 6 hours).

Ash Not more than 7.5 %.

Assay Weigh about about 0.5 g of Phellodendron Bark, proceed as described in the Assay of Coptis Rhizoma.

Containers and Storage ***Containers*—**Well-closed containers.

Picrasma Wood

Picrasmae Lignum

Picrasma Wood is the heartwood of *Picrasma quassioides* Bennet (Simaroubaceae).

Description Picrasma Wood the heartwood consisting of chips, slices or short pieces of wood. The external surface is pale yellow and the texture is dense. Under a magnifying glass, the transverse section reveals distinct annual rings and thin medullary rays. Under a microscope, Picrasma Wood reveals medullary rays consisting of l to 5 cells wide for transverse section and 5 to 50 cells high for longitudinal section. The vessels of spring wood are up to about 150 μm in diameter, but those of autumn wood are only one-fifth as diameter. These vessels are all single or in groups, scattered in the xylem parenchyma. The wood fibers are extremely thickened, and medullary rays and xylem parenchyma cells contain rosette aggregates of calcium oxalate and starch grains. Vivid yellow or red-brown, resinous substances are often present in the vessels.
Picrasma Wood is odorless, taste, extremely bitter and lasting.

Identification Weigh 1 g of pulverized Picrasma Wood, add 10 mL of methanol, overnight, filter and evaporate to dryness. Dissolve the residue in 1 mL of methanol and use this solution as the test solution. Perform the test with this solution as directed under Thin-layer Chromatography. Spot 10 μL of the test solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, methanol and water (100:13.5:10) to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 365 nm): a spot with yellow-green fluorescence is observed at the R_f value of about 0.2.

Purity (1) ***Foreign matter***—Not more than 1.0 %.
(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.

Ash Not more than 4.0 %.

Acid-insoluble Content Not more than 1.0 %

Containers and Storage ***Containers*—**Well-closed containers.

Pinellia Tuber

Pinelliae Tuber

Pinellia Tuber is the tuber of *Pinellia ternata* Breitenbach (Araceae), from which periderm has been removed.

Description Pinellia Tuber is the tuber, completely removed the periderm, slightly flattened spherical to irregular spherical, 7 mm to 25 mm in diameter and 7 mm to 15 mm in height. External surface is white to grayish white-yellow. The upper end is dented where the stem has been removed, and with root scars dented densely as numerous small spots on the circumference. Texture is dense and difficult to cut. The cross section is white and powdery. Under a microscope, the transverse section reveals cork cells remaining on the outside. The 10 to 12 layers of parenchyma cells near the cork cells contain a relatively small amount of starch grains or sometimes none, but parenchyma cells on the inside are filled with starch grains. The parenchyma cells contain raphides of calcium oxalate and mucilage. The vascular bundles are collateral and amphivasal, distributed transversly and longitudinally. The vessels are mostly spiral vessels, sometimes with annular vessels.
Pinellia Tuber is nearly odorless and the taste is weak at first, slightly mucous, but leaving 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 and use the filtrates as the test solution and the Pinellia Tuber RMPM standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 μL each of the test solution and the Pinellia Tuber RMPM standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of chloroform and methanol (95:0.5) 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 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: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.
(4) ***Mycotoxins***—Total aflatoxin (sum of aflatoxins B_1, B_2, G_1 and G_2): Not more than 15.0 ppb (aflatoxin B_1 is not more than 10.0 ppb).

Loss on Drying Not more than 14.0 % (6 hours).

Ash Not more than 3.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Plantago Seed

Plantaginis Semen

Plantago Seed is the ripe seed of *Plantago asiatica* Linné or *Plantago depressa* Willdenow (Plantaginaceae).

Description Plantago Seed is flattened ellipsoidal seed, 2 mm to 2.5 mm in length, 0.7 mm to 1 mm in width and 0.3 mm to 0.5 mm in thickness. External surface is brown to yellow-brown and lustrous. 100 seeds 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. Micropyle and raphe is not observed.
Under a microscope, a transverse section reveals an outermost mucous layer and a very thin cell wall, which swells on contact with water, and a pigment layer below. The pigment cells are trapezoid with a straight lower surface, containing brown pigments. The endosperm cells are in several rows with slightly thick cell walls, close to rectangular, in a mosaic-like arrangement, containing fatty oil droplets. The cotyledon cells are regularly arranged and contain aleurone grains.

Plantago Seed is odorless and tastes slightly bitter and mucous.

Identification (1) Weigh 1 g of Plantago Seed, add 2 mL of warm water and allow the mixture 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 produced.

Purity (1) ***Foreign matter***—Not more than 2.0 %.

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Methoxychlor: Not more than 1 ppm.

(iv) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(v) Aldrin: Not more than 0.01 ppm.

(vi) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 5.5 %.

Acid-insoluble Ash Not more than 2.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Platycodon Fluid Extract

Method of Preparation Weigh coarse powder of Platycodon Root and prepare the fluid extract as directed under Fluid Extracts using 25 % Ethanol. An appropriate quantity of Ethanol and Purified Water may be used in place of 25 % Ethanol.

Description Platycodon Fluid Extract is red-brown liquid. Platycodon Fluid Extract is miscible with water and produces slight turbidity. Platycodon Fluid Extract has a mild taste at first, followed by an acrid and bitter taste.

Identification (1) Shake vigorously 0.5 mL of Platycodon Fluid Extract with 10 mL of water: a lasting fine foam is produced.

(2) Dissolve 1 drop of Platycodon Fluid Extract in 2 mL of acetic anhydride and add gently 0.5 mL of sulfuric acid: a red to red-brown color is observed at the zone of contact.

Purity (1) ***Starch***—Mix 1 mL of Platycodon Fluid Extract with 4 mL of water and add 1 drop of dilute iodine TS: no purple or blue color is observed.

(2) ***Heavy metals***—Total heavy metals: Not more than 30 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

Content of the Active Principle Transfer 5 mL of Platycodon Fluid Extract, accurately measured, to a tared beaker, evaporate to dryness on a water-bath and dry at 105 °C for 5 hours: the residue is not less than 0.50 g

Containers and Storage ***Containers***—Tight containers.

***Storage*—**Light-resistant.

Platycodon Root

Platycodonis Radix

Platycodon Root is the root, with or without periderm, of *Platycodon grandiflorum* A. De Candolle (Campanulaceae).

Description Platycodon Root is the root, thin, long fusiform or conical, often branched. The main root is 10 cm to 15 cm in length and 1 cm to 3 cm in diameter. The external surface is grayish brown, pale brown or white. The upper end of the root has dented scars of removed stems. The neighborhood of the root has fine lateral wrinkles and longitudinal furrows. The greater part of the root, except the crown, is covered with coarse longitudinal wrinkles, lateral furrows and lenticel-like lateral lines. The texture is hard is but easy to break. The transverse section is not fibrous, the cortex is slightly thinner than the xylem, almost white with scattered cracks. The neighborhood of the cambium is brown. The xylem is white to pale brown and the tissue is slightly denser than cortex. Under a microscope, the transverse section reveals a yellow-brown cork layer, mostly removed. The phloem is wide, the phloem rays on the outside are bent and phloem bundles are mostly compressed and degenerated. The lactiferous tubes are scattered in bundles and contain a yellow-brown granular substance. Bundles of lactiferous tubes are arranged in the inside phloem along with sieve tubes. The cambium forms a ring. The xylem has wide medullary rays and polygonal vessels, solitary or several gathered together in a radiating arrangement.
Platycodon Root has a slight, characteristic odor and the taste is weak at first, later acrid and bitter.

Identification (1) Weigh 0.5 g of pulverized

Platycodon Root, boil with 10 mL of water for a while, 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 in a water bath for 2 minutes and filter. To l mL of the filtrate, add carefully 0.5 mL of sulfuric acid to make two layers: a red to red-brown color develops at the zone of contact and the upper layer acquires a blue-green to green color.

(3) To 1 g each of pulverized Platycodon Root and Platycodon Root RMPM, add 50 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate until the filtrate becomes 5 mL, add 20 mL of ether, collect the precipitate, dissolve in 2 mL of ethanol and use these solutions as the test solution and the Platycodon Root RMPM standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 μL each of the test solution and the Platycodon Root RMPM standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of cyclohexane and acetone (4:1) 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 spots obtained from the test solution show the same color and R_f value as the spots obtained form the Platycodon Root RMPM standard solution and of these, a red-brown spot appears at the R_f values of about 0.25 and 0.4.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—(i) Napropamide: Not more than 0.1 ppm.

(ii) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(iii) Dieldrin: Not more than 0.01 ppm.

(iv) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(v) Aldrin: Not more than 0.01 ppm.

(vi) Endrin: Not more than 0.01 ppm.

(vii) Thiomethionate: Not more than 0.3 ppm.

(3) **Sulfur dioxide**—*Not more than 30 ppm.*

Ash Not more than 6.0 %.

Extract Content ***Dilute ethanol-soluble extract***— Not less than 25.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Pogostemon Herb

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 opposite leaves. Stems are square cylindrical, frequently branched, 30 cm to 60 cm in length and 0.2 cm to 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 pith. Leaves are opposite, crumpled into masses, when whole ovate or elliptical, 4 cm to 9 cm in length and 3 cm to 7 cm in width. The leaves are covered with grayish white pubescent on both surfaces. The apex is short-acute or obtuse-rounded, the base is wedge-shaped or obtuse-rounded, the margin is irregular in size and blunt serrated. The petioles are slender, 2 cm to 5 cm in length, coated with soft hairs.
Pogostemon Herb has a characteristic odor and slightly bitter taste.

Identification Add 20 mL of water to the 2 mg of pulverized Pogostemon Herb, shake well and heat for distillating. Take 2 mL of distillate and add 0.5 mL of dinitrophenylhydrazine solution and shake well: it becomes turbid brown.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 13.0 % (6 hours).

Ash Not more than 13.0 %.

Essential Oil Content Not less than 0.3 mL (50.0 g).

Containers and Storage ***Containers*—**Well-closed containers.

Polygala Root

Polygalae Radix

Polygala Root is the root of *Polygala tenuifolia* Willdenow (Polygalaceae).

Description Polygala Root is the root, cylindrical, long, thin and curved. The main root is 10 cm to 20 cm

in length and 2 mm to 10 mm in diameter. The external surface is pale grayish yellow to grayish brown with a relatively dense and deeply dented transverse wrinkles, longitudinal wrinkles and open gaps. Older roots have relatively dense transverse wrinkles, even more deeply dented, slightly knotted. The texture is hard, fragile and easy to cut. The cut surface has a yellowish brown cortex and yellowish white xylem. The cortex and xylem are separate and easily detached, sometimes with the core already removed. Under a microscope the transverse section reveals a cork layer consisting of about 10 rows of cork cells. The cells on the 1 to 2 outside rows are rectangular. The cortex is narrow and the phloem is relatively wide with open gaps throughout. The cambium forms a ring. Those from 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 drops, sometimes containing calcium oxalate druses or solitary crystals.
Polygala Root 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: a lasting fine foam is produced

(2) Weigh 0.5 g of pulverized Polygala Root, add 2 mL of acetic anhydride. After shaking well, allow to stand for 2 minutes and filter. To the filtrate, add carefully 1 mL of sulfuric acid to make two layers: a red-brown color develops at the zone of contact and changes to dark green.

(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), respectively, heat with a reflux condenser for 30 minutes, and filter. To the filtrate, add 30 mL of water and extract with two 20 mL volumes of ethyl acetate. Combine the extracts and evaporate to dryness, shake and separate the dichloromethane layer. Dissolve the residues to 1 mL of ethylacetate, filter, and use each of the filtrates as the test solution and the Polygala Root RMPM standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 5 μL each of the test solution and the Polygala Root RMPM standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Deve1op the plate with a mixture of ethyl acetate, hexane and formic acid (10:4:0.5) to a distance of about 10 cm and air-dry the plate. Spray diluted sulfuric acid TS to the plate, heat the plate at 105 °C and examine under ultraviolet light (main wavelength: 365 nm). The spots from the test solution and the spots from the Polygala Root RMPM standard solution show the same color and the same R_f value.

Purity (1) ***Foreign matter*—**(i) Stem: Not more than 10.0 %.

(ii) Other foreign matter: The amount of foreign matter other than the stems contained in Polygala Root is not more than 1.0 %.

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): Not more than 0.2 ppm.

(vi) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

(5) ***Mycotoxins***—Total aflatoxin (sum of aflatoxins B_1, B_2, G_1 and G_2): Not more than 15.0 ppb (aflatoxin B_1 is not more than 10.0 ppb).

Ash Not more than 6.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Polygonatum Rhizome

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 irregular cylinder or tubercle-shaped rhizome, 3 cm to 10 cm in length and 5 mm to 30 mm in diameter, sometimes furcated. External surface is yellow-brown to blackish brown, with ring shaped transverse nodes. The upper part of the node shows stem scar orbiculate with a sunken 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, semi-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, and 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.
Polygonatum Rhizome has a slight sweet odor and tastes sweet and viscous on chewing.

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

red-brown color appears at the zone of contact.

(2) Weigh 1 g of pulverized Polygonatum Rhizome, add 10 mL of dilute hydrochloric acid, heat carefully for 2 minutes and filter. To the filtrate, add sodium hydroxide TS to neutralize. Add 1 mL of Fehling's TS to 3 mL of this solution and heat: red precipate appears

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 5.0 %.

Acid-insoluble Ash Not more than 1.0 %

Containers and Storage ***Containers*—**Well-closed containers.

Polygonum Multiflorum Root

Polygoni Multiflori Radix

Polygonum Multiflorum Root is the tuber of *Polygonum multiflorum* Thunberg (Polygonaceae). Polygonum Multiflorum Root, when dried, contains not less than 0.75 % of 2,3,5,4'-tetrahydroxy-stilbene-2-O-β-D-glucoside ($C_{20}H_{22}O_9$: 406) and not less than 0.10 % in total of emodin ($C_{15}H_{10}O_5$: 270.24) and physcion ($C_{16}H_{12}O_5$: 284.27).

Description Polygonum Multiflorum Root is fusiform or massive tuber, 5 cm to 15 cm in length, 3 cm to 10 cm in diameter. External surface is red-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 cut. The cut surface is pale yellow-brown or pale redd-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. The phloem is relatively broad and is scattered with 4 to 11 nearly orbicular hetero-vascular bundles, or complex vascular bundles. A type of solitary vascular bundle is present at the center of the root. 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.
Polygonum Multiflorum Root is 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. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 μL each of the test solution and the Polygonum Multiflorum Root RMPM standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, methanol, water and acetic acid (200 : 10 : 10 : 3) 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: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 14.0 %

Ash Nor more than 5.0 %

Acid-insoluble Ash Not more than 1.5 %

Extract Content ***Dilute ethanol-soluble extract*—**

Not less than 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.

$$\text{Amount (mg) of 2,3,5,4'-tetrahydroxystilbene-2-O-β-D-glucoside} = \text{Amount (mg) of 2,3,5,4'-Tetrahydroxystilbene-2-O-β-D-glucoside RS} \times \frac{A_T}{A_S} \times \frac{1}{8}$$

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 octadecylsilanized silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Column temperature: A constant temperature of about 30 °C

Mobile phase: Control the step or concentration gradient by mixing mobile phases A and B as directed in the following table.

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 (%)	Mobile phase B (%)
0	85	15
35	60	40
40	85	15

Flow rate: 1.0 mL/minute

System suitability

System repeatability: When the test is repeated 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 not more than 1.5 %.

(2) ***Emodin and physcion***—Weigh accurately 1 g of pulverized Polygonum Multiflorum Root, add 50 mL of methanol, heat 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 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 solution layer with three 15 mL volumes of chloroform, combine the chloroform layers and vacuum-concentrate. To the residue, add methanol to make exactly 10 mL and use this solution as the test solution. Separately, weigh 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 physicion 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 octadecylsilanized 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 water, acetonitrile and phosphoric acid (850 : 150 : 1)

Flow rate: 0.5 mL/minute

System suitability

System performance: When the procedure is run with 10 μL of the standard solution, emodin and physcion are eluted in this order with the resolution between their peaks being not less than 3.

System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions, the relative standard deviation of each peak area of emodin and physcion is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Polyporus Sclerotium

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 cm to 25 cm in length and 2 cm to 6 cm in diameter. The external surface is black, gray or blackish brown, wrinkled or strumous. The body is light and the texture is hard. The cut surface is close to white or yellowish white and usually granular.
Polyporus Sclerotium is odorless and tasteless.

Identification Weigh 0.5 g of pulverized Polyporus Sclerotium, add 5 mL of acetone and heat on a water-bath for 2 minutes, filter and evaporate the filtrate to dryness. Dissolve the residue in 5 drops of acetic anhydride and add 1 drop of sulfuric acid: a red-purple color develops and immediately changes to dark green.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 16.0 %.

Acid-insoluble Ash Not more than 4.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Poncirus Immature Fruit

Ponciri Fructus Immaturus

Poncirus Immature Fruit is the unripe whole fruit or halved fruit of *Poncirus trifoliata* Rafinesque (Rutaceae). Poncirus Immature Fruit, when dried, contains not less than 2.0 % of poncirin ($C_{28}H_{34}O_{14}$: 594.28) and not less than 0.7 % of naringin ($C_{27}H_{32}O_{14}$: 580.55).

Description Poncirus Immature Fruit is almost ball-shaped, unripe fruit, 1 cm to 2 cm in diameter. External surface is brown to deep brown, coarse and has a number of dented spots due to the oil cavity. Epidermal side of the transverse section is yellowish brown, inner side is pale grayish-brown and the center is composed of about 8 small cells, each cell is yellowish brown and dented, sometimes unriped seed is contained.
Poncirus Immature Fruit has a characteristic odor and bitter taste.

Identification (1) Weigh 0.5 g of pulverized Poncirus Immature Fruit, add 10 mL of the methanol, heat gently for 2 minutes and filter. Add 0.1g of magnesium powder and 1 mL of hydrochloric acid to the 5 mL of the filtrate: the liquid becomes reddish purple.
(2) Weigh 0.5 g each of pulverized Poncirus Immature Fruit and Poncirus Immature Fruit RMPM, add 10 mL of ethanol, shake well, allow to stand for 30 minutes and filter, respectively. Use the filterates as the test solution and the standard solution of Poncirus Immature Fruit RMPM. Perform the test with the test solution, the standard solution of Poncirus Immature Fruit RMPM and the standard solution as directed under the Thin-layer Chromatography. Spot 10 μL each of the test solution, the standard solution of Poncirus Immature Fruit RMPM and the standard solution on a plate of silica gel for thin-layer chromatography. Deve1op the plate with a mixture of dichloromethane, methanol and water (30 : 10.5 : 1) to a distance of about 10 cm and air-dry the plate. Spray the dilute sulfuric acid TS to the plate, heat the plate at 105 °C for 10 minutes. The spots from the test solution and the spots from the standard solution of Poncirus Immature Fruit RMPM show the same color and the same R_f value. Among those spots, the spots of naringin and poncirin appear at the R_f values of about 0.45 and 0.6, respectively.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): Not more than 0.2 ppm.
(vi) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 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 and filter. Combine

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 silica gel desiccator 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 Liquid Chromatography according to the following operating conditions and determine the peak areas, A_{Ta} and A_{Tb}, of poncirin and naringin in the test solution and the peak areas, A_{Sa} and A_{Sb}, of poncirin and naringin in the standard solution.

$$\text{Amount (mg) of poncirin } (C_{28}H_{34}O_{14}) = \text{amount (mg) of Poncirin RS} \times \frac{A_{Ta}}{A_{Sa}}$$

$$\text{Amount (mg) of naringin } (C_{27}H_{32}O_{14}) = \text{amount (mg) of Naringin RS} \times \frac{A_{Tb}}{A_{Sb}}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 313 nm).

Column: A stainless steel column, 4 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: Control the step or concentration gradient by mixing mobile phases A and B 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 (100:1).

Time (min)	Mobile phase A (%)	Mobile phase B (%)
0	90	10
30	35	65
35	10	90
40	10	90
45	90	10

Flow rate: 1.0 mL/minute

System suitability

System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions, the relative standard deviation of the peak area of poncirin is not more than 1.5 %.

Containers and Storage ***Containers*****—**Well-closed containers.

Poria

Poria Sclerotium

Poria is the sclerotium of *Poria cocos* Wolf (Polyporaceae).

Description Poria is the sclerotium, in masses, usually as broken or chipped pieces, unbroken ones are 10 cm to 30 cm in diameter and 0.1 kg to 2 kg in mass. Remaining outer layer is dark brown to dark reddish brown, coarse, which fissures. The inside is white or pale reddish white. The texture is hard, but brittle.
Poria is nearly odorless, the taste is weak and slightly mucous.

Identification (1) Weigh 1 g of pulverized Poria, add 5 mL of acetone, warm in a water-bath for 2 minutes with 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: a pale red color develops, which changes immediately to dark green.

(2) Take a section or powder of Poria and add 1 drop of iodine TS: a deep red-brown color is produced.

Purity (1) ***Heavy metals*****—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides*****—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 1.0 %.

Containers and Storage ***Containers*****—**Well-closed containers.

Prepared Aconite

Aconiti Lateralis Radix Preparata

Prepared Aconite is the prepared daughter root of *Aconitum carmichaeli* (Ranunculaceae). According to 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 by size the daughter root of f *Aconitum*

carmichaeli, harvested between June and August and with the parent root, rootlets and soil removed. Wash with water and immerse overnight in brine. Add salt, immerse, take out and sun-dry. Repeat the above process and gradually prolong the sun-drying time until a lot of salt is crystallized on the surface and its texture becomes hard. This is known as Yeombuja.

(2) ***Bujapyeon (Sliced Aconite)***—Immerse Yeombuja in water several times to rinse out the salt and cut longitudinally into slices 3 mm to 5 mm in thickness. Immerse in water and steam until cooked through. Take out, bake to half-dryness then sun-dry.

(3) ***Pobuja (Boiled Aconite)***—Immerse Yeombuja in water, replacing the water 2 to 3 times a day until the salt is completely rinsed out. Boil with Licorice Root and black bean until cooked through. Take out when the slice becomes numbless to the tongue , remove the periderm and make slices or cut into several pices and sun-dry. 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. External surface is grayish-black, covered with fine powder of salt, topped with depressed bud scars and encircled with tuberculated short rootlets or rootlet scars. Texture is heavy and hard. Transversely cut surface is grayish-brown, showing small clefts filled with fine powder of salt and a polyangular cambium ring and vascular bundles are arranged irregularly inside the ring.
It has a slight odor and tastes salty, numb and pungent.

(2) ***Bujapyeon (Sliced Aconite)***—Bujapyeon is processed Yeombuja, cut longitudinally, 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. Texture is hard and fragile. Fractured surface is horn-like.
It is nearly odorless and its taste is weak.

(3) ***Pobuja* (*Boiled Aconite*)**—Pobuja is the processed Yeombuja, sliced 3 mm to 5 mm in thickness with irregular shapes and sizes. External surface is pale brown to dark brown or black. Texture is hard and semi-translucent and it is 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 and filter. Transfer the filtrate to a separatory funnel, add 20 mL of 0.25 mol/L sulfuric acid solution, shake and stand. Separate the acid solution and determine the spectrum of the solution as directed under the Ultraviolet-visible Spectrophotometry: the spectrum exhibits maximum between 231 nm and 274 nm.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.
(4) ***Aconitine***—Weigh 20 g of the pulverized Prepared Aconite to a stoppered Erlenmeyer flask, add 150 mL of ether, shake for 10 minutes, add 10 mL of ammonia TS, shake for 30 minutes and allow to stand for 1 to 2 hours. Evaporate the ether layer to dryness. Dissolve the residue in 2 mL of dehydrated 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. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 5 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 ethylacetate (1 : 1) to a distance of about 10 cm and air-dry the plate. Spray evenly Dragendorff's TS on the plate: the spot of the test solution is not more dense than that of the standard solution.

Containers and Storage ***Containers*—**Well-closed containers.

Prepared Rehmannia Root

Rehmanniae Radix Preparata

Prepared Rehmannia Root is the root of *Rehmannia glutinosa* Liboschitz ex Steudel (Scrophulariaceae), with the application of steaming. Steamed Rehmannia Root, when dried, contains not less than 0.1 % of 5-hydroxymethyl-2-puraldehyde ($C_6H_6O_3$: 126.11).

Method of Preparation Select well cleaned rehmannia root, steam with wine, ammomum fruit and citrus unshiu peel. Take it out and sun-dry. Repeat the above process until the inside and outside of the root becomes blackish and shiny and until the texture becomes soft and flexible.

Description Prepared Rehmannia Root is the steamed root as an irregular mass, with various size. External surface is black, lustrous and sticky. The texture is soft and flexible, uneasily broken and the fracture is black and lustrous.
Prepared Rehmannia Root has a slight, characteristic odor and the sweet taste.

Identification Weigh 1 g of the Prepared Rehmannia Root, add 20 mL of water or dilute ethanol, shake well and filter. Add 10 mL of Fehling's TS to the filtrate and

heat for a while: red-purple to red-brown precipitate is produced.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.
(4) ***Benzopyrene***—Not more than 5 ppb.

Ash Not more than 6.0 %.

Acid-insoluble Ash Not more than 2.5 %.

Assay Weigh accurately about 2 g of finely cut Prepared Rehmannia Root, add 100 mL of diluted methanol (1 in 2), heat with a reflux condenser for 3 hours and filter. Repeat the above process with the residue. Combine the filtrate, extract twice with 200 mL volumes of hexane and discard the hexane layer. Remaining water layer is evaporated under reduced pressure until the volume is less than half of the initial volume. Extract the water layer twice with 100 mL volumes of ethylacetate, the extract is combined and is evaporated under reduced pressure. The residue is dissolved in methanol to make 20 mL and use this solution as the test solution. Weigh accurately about 10 mg of 5-Hydroxymethyl-2-furaldehyde RS, dissolve in 100 mL of methanol 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, for the test solution and the standard solution, respectively.

$$\text{Amount (mg) of 5-hydroxymethyl-2-furaldehyde } (C_6H_6O_3) = \text{amount (mg) of 5-Hydroxymethyl-2-furaldehyde RS} \times \frac{A_T}{A_S} \times \frac{1}{5}$$

Operating conditions
Detector: An ultraviolet absorption photometer (wavelength: 280 nm).
Column: A stainless steel column, 4 mm to 6 mm in internal diameter and 15 cm to 25 cm in length, packed with octadecylsilyl silica ge1 for liquid chromatography (5 μm to l0 μm in particle diameter).
Column temperature: 25 °C.
Mobile phase: A mixture of water and acetonitrile (95 : 5).
Flow rate: 1.0 mL/minute.
System suitability
System repeatability: When the test is repeated 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 not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Prunella Spike

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 is grayish brown to red-brown, and texture is light. Spikes are composed of a floral axis having numerous bracts and calyxes. Corollas are often remained on the upper part. A calyx usually enclosed four mericarps. Bract is cordate to eccentric and exhibiting white hairs on the vein, as on the calyx.
Prunella Spike is almost odorless and tasteless.

Identification Weigh 1 g of pulverized Prunella Spike, add 20 mL of ethanol, heat on a water bath for 1 hour under a reflux condenser and filter. Evaporate the filtrate to dryness, dissolve the residue to 15 mL of petroleum ether, and shake for 2 minutes. Remove the petroleum ether layer, dissolve the residue to 1 mL of ethanol and use the solution as the test solution. Separately, take 1 mg of Ursolic acid RS, add 1 mL of ethanol and use the solution as the standard solution. Perform the test with the test solution and the standard solution as directed under Thin-layer Chromatography. Spot 2 μL each of the test solution and the standard solution on a plate of silica gel for thin-layer chromagraphy. Develop the plate with a mixture of ethyl acetate and methanol (40 : 1) 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: One spot of the several spots from the test solution and the reddish spot spot from the standard solution show the same color and the same R_f value.

Purity (1) ***Foreign matter*—**(i) Stem: The amount of the stems contained in Prunella Spike is not more than 5.0 %.
(ii) Other foreign matter: The amount of foreign matter other than the stems contained in Prunella Spike is not more than 1.0 %.

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 13.0 %.

Acid-insoluble Ash Not more than 5.0 %.

Containers and Storage ***Containers—***Well-closed containers.

Pueraria Root

Puerariae Radix

Pueraria Root is the root, with or without periderm, of *Pueraria lobata* Ohwi (Leguminosae). Pueraria Root contains not less than 2.0 % of puerarin ($C_{21}H_{20}O_9$: 416.38) and not less than 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 cm to 30 cm in length and about 1 cm in thickness, and the latter are close to hexahedrons of irregular sizes. External surface is grayish white to pale brown, longitudinal 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. The phloem shows light grayish yellow, and the xylem shows numerous vessels appearing as small dots. The medullary rays are light grayish yellow and slightly dented. Under a microscope, the cortex is mostly 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 bundless. 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. Pueraria has a slight odor and it tastes slightly sweet.

Identification Weigh 2 g each of pulverized Pueraria Root and Pueraria Root RMPM, add 10 mL of methanol, shake for 3 minutes, filter and use the filtrates as the test solution and Pueraria Root RMPM standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 2 μL each of the test solution and the Pueraria Root RMPM standard solution on a plate of silica gel with fluorescent indiator for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, methanol and water (12:2:1) to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 254nm): the spots from the test solution and the spots from Pueraria Root RMPM standard solution show the same color and the same R_f value. The spots of puerarin and diadzin appear at the R_f value of about 0.5 and 0.55, respectively.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endosulfan (sum of α and β-endosulfan and endosulfan sulfate): Not more than 0.2 ppm.
(vi) Endrin: Not more than 0.01 ppm.
(vii) Captan: Not more than 2 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not less than 13.0 % (6 hours).

Ash Not more than 6.0 %.

Assay Weigh accurately about 2 g of pulverized Pueraria Root, add 60 mL of methanol, heat 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 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 silica gel desiccator 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 Liquid Chromatography according to the following operating conditions and determine the peak areas, A_{Ta} and A_{Tb}, of puerarin and daidzin in the test solution and the peak areas, A_{Sa} and A_{Sb}, of puerarin and daidzin in 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 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: Control the step or concentration gradient by mixing mobile phases A and B as directed in the following table.

Mobile phase A: Methanol

Mobile phase B: Water

Time (min)	Mobile phase A (%)	Mobile phase B (%)
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: When the procedure is run with 10 μL of the standard solution under the above operating conditions, puerarin and daidzin are eluted in this order, clearly dividing each peak.

System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions, the relative standard deviation of each peak area of puerarin and daidzin is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Raphanus Seed

Raphani Semen

Raphanus Seed is the ripe seed of *Raphanus sativus* Linné (Cruciferae).

Description Raphanus Seed is theseed, nearly subovoid or ellipsoidal, slightly flattened, 2.5 mm to 4 mm in length, and 2 mm to 3 mm in width. External surface is yellowish brown to reddish brown, or grayish brown, with a deep brown round hilum at one end and several longitudinal furrows on one side. Testa is thin and brittle, coryledons is 2, yellowish white and oily. Under a microscope, transverse section reveals a pigment layer adhering to palisade layer and atrophying, with reddish brown substance inside and endosperm flattened in a line, with starch grains inside.

Raphanus Seed is nearly odorless and the taste is weak, slightly bitter and pungent.

Identification Weigh 1 g each of pulverized Raphanus Seed and Raphanus Seed RMPM, dissolve separately in 50 mL of diluted methanol (4 in 5), heat with a reflux condenser for 1 hour, 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. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 5 μL each of the test solution and the Raphanus Seed RMPM standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, water and formic acid (10:3:2) to a distance of about 10 cm and air-dry the plate. Spray evenly *p*-anisaldehyde-sulfuric acid TS and heat at 105 °C: the 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: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 8.0 %.

Ash Not more than 7.0 %.

Acid-insoluble Ash Not more than 1.0 %.

Extract Content ***Ether-soluble extract***—Not less than 31.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Red Ginseng

Ginseng Radix Rubra

Red Ginseng is the root of *Panax ginseng* C. A. Meyer (Araliaceae), after being steamed.
It contains not less than 0.10 % of ginsenoside Rg_1 ($C_{42}H_{72}O_{14}$: 801.01) and not less than 0.20 % of ginsenoside Rb_1 ($C_{54}H_{92}O_{23}$: 1109.29), calculated on the dried basis.

Description Red Ginseng is thin and long cylindrical to fusiform steamed root, often branching out into 2 to 3 lateral roots from the middle. Red Ginseng is 5 cm to 25 cm in length, main root is 5 to 30 mm in diameter. External surface is pale yellow-brown to red-brown and semi-translucent and with longitudinal wrinkles and with thin scars.of root. Crown is somewhat constricted and sometimes with short remains of stem. Fractured surface is flat. Texture is horny and hard.
Red Ginseng has a characteristic odor and taste, 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 in a water-bath for 2 minutes and filter. To 1 mL of the filtrate, add gently 0.5 mL of sulfuric acid to make two layers: a red–brown color develops at the zone of contact.

(2) Weigh 2 g of pulverized Red Ginseng, add 20 mL of methanol, heat with a reflux condenser 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. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 10 μL each of the test solution and the standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with the lower layer of a mixture of chloroform, methanol and water (13 : 7 : 2) to a distance of about 10 cm, spray evenly sulfuric acid TS for spray on the plate and heat at 110 °C for 5 minutes: one spot among the spots from the test solution and a red-purple spot from the standard solution show the same color and the same R_f value.

Purity (1) ***Foreign matter***—The amount of stems and other foreign matter contained in Red Ginseng is not more than 2.0 %.

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—Proceed with Red Ginseng as directed under "Red Ginseng" in [Attachment 4] MRLs for Agricultural Products in KFDA Notice "Standards and Specifications for Food."

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 15.5 % (6 hours)

Ash Not more than 4.5 %.

Extract Content ***Dilute ethanol-soluble extract***—Not less than 18.0 %.

Assay (1) ***Ginsenoside Rg_1***—Weigh 1 g of pulverized Red Ginseng, and perform the test as directed under the assay of [Ginseng]

(2) ***Ginsenodise Rb_1***—Use the solution of (1) as the test solution, and perform the test as directed under the assay of [Ginseng]

Containers and Storage ***Containers***—Well-closed containers.

Rehmannia Root

Rehmanniae Radix

Rehmannia Root is the root of *Rehmannia glutinosa* Liboschitz ex Steudel (Scrophulariaceae).

Description Rehmannia Root is cylindrical to fusiform root, 5 cm to 15 cm in length, 5 mm to 15 mm in diameter, and often broken or markedly deformed in shape. External surface is yellow-brown to black-brown, with deep, longitudinal wrinkles, laterally scars of lateral roots, and lenticel. The texture is soft and breakable. Under a magnifying glass, a transverse section reveals yellowish brown to blackish brown, cortex darker than xylem in color, hardly observable pith. Under a microscope, the transverse section reveals a cork layer consisting of several rows of cork cells. The cortex has a sparse arrangement of parenchyma cells and is scattered with numerous secreting cells, which contain orange oil drops. Nearly orbicular stone cells can be seen at the upper part of the tuberous root. The phloem has relatively fewer secreting cells. The cambium forms a ring. The xylem rays are broad and in 2 to 4 rows. Vessels are rare, radiating in an intermittent and sparse arrangement.
Rehmannia Root has a characteristic odor, and slightly sweet taste at first and followed by a slight bitterness.

Identification Weigh 2 g each of pulverized Rehmannia Root and Rehmannia Root RMPM, add 20 mL of methanol, warm with a reflux condenser for 1 hour, filter and evaporate the filtrates to dryness. Dissolve the residues in 5 mL of methanol and use these solutions as the test solution and the Rehmannia Root RMPM standard solution. Perform the test with the test solution and the standard solution of Rehmannia Root RMPM as directed under Thin-layer Chromatography. Spot 10 μL of the test solution and the standard solution of Rehmannia Root RMPM on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, hexane and formic acid (10 : 4 : 0.5) to a distance of about 10 cm and air-dry the plate. Spray evenly dilute sulfuric acid TS to the plate, heat the plate at 105 °C for 10 minutes. Several spots from the test solution and the spots from the standard solution of Rehmannia Root RMPM show the same

color and the same R_f value.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(vi) Difenoconazole: Not more than 0.3 ppm.
(vii) Iminoctadine: Not more than 0.1 ppm.
(viii) Kresoxim-methyl: Not more than 0.1 ppm.
(ix) Thiram: Not more than 0.5 ppm.
(x) Pyrimethanil: Not more than 0.2 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.
(4) ***Benzopyrene***—Not more than 5 ppb.

Ash Not more than 6.0 %.

Acid-insoluble Ash Not more than 2.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Rhubarb

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 not less than 0.02 % of sennoside A ($C_{42}H_{38}O_{20}$: 862.74), and not less than 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 crosswise or longitudinally, 5 cm to 15 cm in length and 4 cm to 10 cm in diameter. The outside is without most of the bark. In the case of Rhubarb without most part of cortex, the outer surface is yellow-brown to pale brown, exhibiting white, fine reticulations, and texture is thick and hard. In the case of Rhubarb with cork layer, externally dark brown or blackish-red and with coarse wrinkles, and texture is rough and brittle. The transverse section is not fibrous. It is pale grayish brown or brown, having patterns of blackish brown tissue complicated with white and pale brown tissues. This pattern sometimes radiates near the cambium. The pith consists of whirls of tissues radiated from the center of a small brown circle, 1 mm to 3 mm in diameter and arranged in a ring or scattered irregularly. Under a microscope, the transverse section of Rhubarb from *Rheum palmatum* reveals the cork layer of the rhizome and cortex mostly removed, sometimes partially remaining. The phloem rays are in 3 to 4 rows, relatively linear and containing brown substances. The cambium consists of flat cells. The xylem rays are relatively dense, consisting of 2 to 4 rows of cells and containing deep brown substances. Vessels are rare, sparse and arranged towards the center. The pith is wide, mainly consisting of parenchyma cells with multiple complex vascular bundles inside a single fence or scattered. The complex vascular bundles have a ring-shaped cambium with the phloem at the center, sometimes with mucilage cavities visible near the cambium. Outside the cambium is the xylem with medullary rays stretching out in a star shape and containing deep brown substances inside. The parenchyma cells contain many starch grains and large calcium oxalate crystal druses. Rhubarb from *Rheum tanguticum* has phloem rays of the rhizome consisting of 2 to 3 rows, curved wavy. The phloem has many mucilage cavities, arranged in concentric rings. There are no xylem rays and many mucilage cavities inside the star spot. Rhubarb from *Rheum officinale* has phloem rays of the rhizome consisting of 1 to 2 rows, linear, with no mucilage cavities in the xylem, no xylem rays and no mucilage cavities in the star spot.
Rhubarb has a characteristic odor and a astringent and bitter taste. When chewed, it is gritty between the teeth and colors 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 and centrifuge. Transfer the supernatant liquid to a separatory funnel, add 13 g of sodium chloride and shake for 30 minutes. Separate the water layer with undissolved sodium chloride and adjust the pH to 1.5 by adding l mol/L hydrochloric acid TS. Transfer this solution to another separatory funnel, add 30 mL of tetrahydrofuran, shake for 10 minutes, separate 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. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 40 μL each of the test solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, *n*-propanol, water and acetic anhydride (40 : 40 : 30 : 1) to a distance of about 15 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 365 nm): one of the spots 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) ***Raponticin***—Weigh 0.5 g of pulverized

Rhubarb, add 10 mL of ethanol, heat in a water-bath with a reflux condenser for 10 minutes and filter. Perform the test as directed under the Thin-layer Chromatography, using the filtrate as the test solution. Spot 10 μL of the test solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of isopropyl ether, *n*-butanol and methanol (26 : 7 : 7) to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 365 nm): no spot with blue-purple 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: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 13.0 % (6 hours).

Ash Not more than 13.0 %.

Acid-insoluble Ash Not more than 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 1000), shake for 30 minutes, 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 in sodium bicarbonate solution (1 in 1000) to make exactly 50 mL. Pipet 5.0 mL of this solution, add sodium bicarbonate solution (1 in 1000) 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. Determine the peak areas, A_T and A_S, of sennoside A of the test solution and the standard solution, respectively.

$$\text{Amount (mg) of sennoside A } (C_{42}H_{38}O_{20}) = \text{amount (mg) of Sennoside A RS} \times \frac{A_T}{A_S} \times 0.25$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 340 nm).

Column: A stainlee 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 °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 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 1000) to make 10 mL. When the procedure is run with 20 μL of this solution under the above operating conditions, sennoside A and naringin are eluted in this order with the resolution between their peaks being not less than 3.0.

System repeatability: When the test is repeated 6 times with 20 μL each of the standard solution under the above operating conditions: the relative standard deviation of the peak area of sennoside A is not more than 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 with a reflux condenser for 1 hour and filter. Pipet 5 mL of the filtrate, vacuum-concentrate, add 10 mL of 8 % hydrochloric acid solution and sonicate for 2 minutes. Add 10 mL of chloroform and heat with a reflux condenser for 1 hour. Transfer to a separatory funnel, wash the container with a small amount of chloroform, combine the washings in the separatory funnel, shake and take the chloroform layer. Extract the hydrochloric acid layer with three 10 mL volumes of chloroform, combine the chloroform layers and vacuum-concentrate. To the residue, add methanol 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 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 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}, 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 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 octadecylsilanized 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 acetonitrile, water and phosphoric acid (850:150:1)

Flow rate: 0.5 mL/min

System suitability

System performance: When the procedure is run with 10 μL of the standard solution under the above operating conditions, aloe emodin, rhein, emodin, chrysophanol and physcion are eluted in this order with the resolution between their peaks being not less than 3.

System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions, the relative standard deviation of each peak area of aloe emodin, rhein, emodin, chrysophanol and physicion is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Rhus Galls

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 form it is divided into Dubae and Gakbae.

Description (1) ***Dubae***—Dubae is the oblong or spindle-globular gall, 25 mm to 90 mm in length, 15 mm to 40 mm in diameter. External surface is grayish brown, slightly pubescent. Texture is hard and fragile, easily broken. Fractured surface is horny-like, lustrous with 2 mm to 3mm thick of gall wall. Inner surface of gall is smooth and contains black-brown killed aphids and gray excreta.

Dubae has a characteristic odor and astringent taste

(2) ***Gakbae*—**Gakbae is the rhombic gall with irregular obtuse branchings and distinct pubescences. Gall walls are relatively thin.

Identification Weigh 0.5 g of pulverized Rhus Gallas, macerate with 10 mL of water, and filter. Add iron (III) chloride TS into the filtrate: a blue-violet precipitate is produced.

Ash Not more than 5.0 %

Containers and Storage ***Containers*—**Well-closed containers.

Rosa Fruit

Rosae Laevigatae Fructus

Rosa Fruit is the ripe fruit of *Rosa laevigata* Michaux (Rosaceae).

Description Rosa Fruit is the fruit, obovoid, 20 mm to 35 mm in length and 1 cm to 2 cm in diameter. External surface is yellowish red to reddish brown, with scars of the fallen bristles appeared as brown small raised dots. A dish like calyx is remained at the apex, with a yellow stalk base in the middle and the lower part is gradually tapered. Texture is hard. The cut surface reveals the wall of calyx, 1 mm to 2 mm in thickness, with numerous small achenes inside with yellow tomenta.

Rosa Fruit has a slight odor and sweet and slightly astringent taste.

Purity (1) ***Foreign matter***—Rosa Fruit contains less than 2.0 % of fruit stalk and bristle.

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 5.0 %.

Extract Content ***Dilute ethanol-soluble extract***—Not less than 34.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Round Amomum Fruit

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 from *Amomum kravanh* is the fruit, nearly spherical, 1 cm to 2 cm in diameter. The external surface is yellowish white to pale yellowish brown with three relatively deep longitudinal ridges, protruding stigmata at the apex, a dented scar of fruit stalk at the lower part and an even growth of pale brown cilia at both ends. The pericarp is thin and light, easily split longitudinally. The inside of the pericarp is divided into 3 loculi with about 10 seeds in each loculus. The seeds are irregular polyhedral with wrinkles and aril remaining.
Round Amomum Fruit from *Amomum kravanh* is aromatic, has a slightly camphor-like odor and tastes pungent and cool.

(2) ***Amomum compactum***—Round Amomum Fruit from *Amomum compactum* is the fruit, smaller than *Amomum kravanh*. External surface is yellowish white, sometimes purplish brown. Pericarp is relatively thin and seeds are thin and blighted.
Round Amomum Fruit from *Amomum compactum* has a weaker odor and taste compared to *Amomum kravanh*.

Identification Dissolve 20 μL of Round Amomum Fruit essential oil 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. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 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 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 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***—Round Amomum Fruit contains not more than 2.0 % of the pericarp, fruit stalk and other foreign matter.

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 12.0 % (seed).

Acid-insoluble Ash Not more than 5.0 % (seed).

Essential Oil Content 0.4 mL (50.0 g) (seed).

Containers and Storage ***Containers*—**Well-closed containers.

Rubus Fruit

Rubi Fructus

Rubus Fruit is the unripe fruit of *Rubus coreanus* Miquel (Rosaceae).

Description Rubus Fruit is the fruit as aggregate consisting of numerous small drupes, mostly round and 7 mm to 9 mm in diameter. External surface is pale green, grayish brown or red-brown to red-purple, surrounded by numerous drupelets, nearly hairless. The calyx divides into 5 and is brown with a fruit stalk scar at the bottom. Individual druplets are easily separated, close to spherical, relatively flat at the bottom and about 4 mm in diameter.
Rubus Fruit is nearly odorless, and the taste is slightly sour and sweet.

Identification Weigh 0.5 g of pulverized Rubus Fruit, add 10 mL of ethanol, heat for about 2 minutes and filter. To 5 mL of the filtrate, add a little of magnecium powder and 2 to 3 drops of hydrochloric acid: the color develops deep red.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—Proceed with Rubus Fruit as directed in "Rubus Fruit" in [Attachment 4] MRLs for Agricultural Products in KFDA Notice "Standards and Specifications for Food."

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 17.0 %.

Ash Not more than 8.0 %.

Extract Content ***Dilute ethanol-soluble extract***—Not less than 20.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Safflower

Carthami Flos

Safflower is the tubulous flower of *Carthamus tinctorius* Linné (Compositae).

Description Safflower is the tubulous flower without the ovary, 1 cm to 2 cm in length. The external surface is red to redd-brown. The corolla tube is slender and long and divides into 5 lobes at the apex. The lobe is in the shape of a narrow cord, 5 mm to 8 mm in length. There are 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.
Safflower has a characteristic odor and slightly bitter taste.

Identification (1) Weigh 0.2 g of Safflower, add 10 mL of dilute ethanol, heat with a reflux condenser for 15 minutes and filter. Place 3 mL of the filtrate in a small glass vessel about 30 mm in both internal diameter and height, 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. Transfer 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 colored pale yellow and the lower volume, 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 and filter, respectively. Use these solutions as the test solution and the standard solution of Safflower RMPM. Perform the test with the test solution and the standard solution of Safflower RMPM as directed under the Thin-layer Chromatography. Spot 5 μL each of the test solution and the standard solution of Safflower RMPM on a plate with fluorescent indicator for thin-layer chromatography. Deve1op the plate with a mixture of ethyl acetate, water, formic acid and methanol (7 : 3 : 2 : 0.4) to a distance of about 10 cm and air-dry the plate. Examine the plate under ultraviolet light (wavelength: 365 nm). The several spots from the test solution and the spots from the standard solution of Safflower RMPM show the same color and the same R_f value.

Purity (1) ***Foreign matter***—The amount of ovaries, stems, leaves and other foreign matter is not more than 2.0 %.

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(vi) Quintozene (sum of quintozene, pentachloroaniline and methyl pentachlorophenyl sulfide): Not more than 0.1 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

(5) ***Mycotoxins***—Total aflatoxin (sum of aflatoxins B_1, B_2, G_1 and G_2): Not more than 15 ppb (aflatoxin B_1 is not more than 10.0 ppb).

Ash Not more than 18.0 %.

Containers and Storage ***Containers*—**Well-closed containers.
***Storage*—**Light-resistant.

Saffron

Crocus

Saffron is the stigma of *Crocus sativus* Linné (Iridaceae).

Description Saffron is the stigma, thin cord-like shaped, 20 mm to 35 mm in length, tripartite or separate. It is dark yellowish red to reddish brown overall and the upper part is relatively broad and slightly flat. The apex is tooth-shaped with uneven margin and has one short lacunae on the inside. The lower part sometimes has a small, yellow base of the style remaining. The body is light and the texture is soft with no luster. When dried, the texture is fragile and easily cut.
Saffron has strong and characteristic odor, bitter taste and the color of saliva becomes yellow.

Identification (1) Add l drop of sulfuric acid to Saffron: The color changes to dark blue which gradually turns red–brown through purple.

(2) ***Crocin***—Dry Saffron in a desiccator (silica gel) for 24 hours and powder. Weigh 0.1 g of pulverized Saffron, add 150 mL of warm water, warm the mixture between 60 °C and 70 °C for 30 minutes with frequent shaking and filter. To 1.0 mL of the filtrate, add water to make 10 mL: the solution is not more intense than the following control solution.

Control solution— Weigh 5 mg of potassium

bichro-mate, dissolve it in water to make exactly l0 mL.

Purity (1) ***Foreign matter*—**(i) Aniline dyes: Shake 50 mg of Saffron with 10 mL of chloroform: the solution is colorless or only slightly yellow.

(ii) Glycerol, sugar or honey: Saffron has no sweet taste. Press it between two pieces of paper: no spot is left on the paper.

(iii) Yellow style: The yellow style in Saffron is not more than 10.0 %.

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

Loss on Drying Not more than 12.0 % (6 hours).

Ash Not more than 7.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

***Storage*—**Light-resistant.

Salvia Miltiorrhiza Root

Salviae Miltiorrhizae Radix

Salviae Miltiorrhizae Root is the root of *Salvia miltiorrhiza* Bunge (Labiatae).
Salvia Miltiorrhiza Root, when dried, contains not less than 4.1 % of salvianolic acid B ($C_{36}H_{30}O_{16}$: 718.62).

Description Salvia Miltiorrhiza Root is the root, long cylindrical in shape, 10 cm to 20 cm in length and 3 mm to 15 mm in diameter. The root consists of 1 to 2 or several branches, slightly curved, some with thin, hair-like rootlets. The external surface is coarse, red-brown or dark red-brow, longitudinally wrinkled. The bark of old roots is soft and tender. The texture is hard and fragile. The cut surface is soft with clefts or slightly even and dense, with red-brown cortex and gray-yellow or purple-brown xylem, showing bundles of yellowish white vessels arranged radially.
Under a microscope, the transverse section reveals a cork layer consisting of 4 to 6 rows of cork cells. The Cortex is wide and the phloem is semi-circular. The cambium forms rings and the interfascicular cambium is not very distinct. The xylem consists of 8 to 10 bundles in a radial arrangement. The vessels are concentrated near the cambium and become a single row towards the center. The xylem fibers are in bundles, distributed near the primary xylem in the center.
Salvia miltiorrhiza Root has a slight, characteristic odor and slightly bitter and astringent taste.

Identification (1) Weigh 1 g of pulverized Salvia Miltiorrhiza Root, add 10 mL of ethanol, boil shortly and filter: the filtrate shows brownish yellow. Add 1 mL of diluted sulfuric acid and 0.5 g of zinc powder: the filtrate turn to yellow.

(2) Weigh 2 g of pulverized Salvia Miltiorrhiza Root, add 10 mL of methanol, sonicate for 1 hour, filter and use the fitrate as the test solution. Separately, dissolve 1 mg of tansinone II A RS in 1 mL of methanol and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of hexane and ethyl acetate (4 : 1) to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm) or spray evenly the plate with sulfuric acid TS for spray and heat: one spot among the 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: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 12.0 % .

Ash Not more than 7.0 %.

Extract Content ***Dilute ethanol-soluble extract***—Not less than 25.0 %.

Assay Weigh accurately about 0.3 g of pulverized Salvia Miltiorrhiza Root, add 50 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 operating conditions and determine the peak areas, A_T and A_S, of the test solution and the standard solution.

$$\text{Amount (mg) of salvianolic acid B } (C_{36}H_{30}O_{16}) = \text{Amount (mg) of Salvianolic Acid B RS} \times \frac{A_T}{A_S}$$

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 octadecylsilanized silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Column temperature: An ordinary temperature

Mobile pahse: Control the step or concentration gradient by mixing mobile phases A and B as directed in the following table.

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 (%)	Mobile phase B (%)
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: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions, the relative standard deviation of the peak area of salvianolic acid B is not more than 1.5 %.

Containers and Storage ***Containers*****—**Well-closed containers.

Saposhnikovia Root

Saposhnikoviae Radix

Saposhnikovia Root is the root of *Saposhnikovia divaricata* Schischkin (Umbelliferae).

Description Saposhnikovia Root is the root, thin, long conical, thinner towards the bottom, 15 cm to 20 in length and 7 mm to 15 mm in diameter. External surface is pale brown. There are several longitudinal wrinkles and rootlet scars. The body is light and the texture is soft and easy to cut. The cut surface reveals a pale brown cortex and many lacunae, and the xylem is pale yellow. Under a microscope, the transverse section reveals a cork layer consisting of several rows of cork cells, and the phelloderm is narrow. The cortex reveals an irregular and relatively large elliptic lactiferous tube. The phloem is relatively broad with multiple lactiferous tubes, close to circular, with 4 to 8 secreting cells nearby, and the lactiferous tubes are filled with golden yellow, oil-like substance. The medullary rays are curved, usually separated from the phloem tissue to form a lacuna. The cambium is ring-shaped and distinct. The xylem has very numerous vessels, radiating one by one or in groups of 2 to 3. The medullary rays of the xylem consists of 1 to 2 rows of cells with several lacunae and radiating. The center of the crown has pith. Saposhnikovia Root has a characteristic odor and slightly sweet taste.

Identification Weigh 1 g each of pulverized Saposhnikovia Root and Saposhnikovia Root RMPM, add 10 mL of methanol, sonicate for 60 minutes, filter and use the filtrates as the test solution and the Saposhnikovia Root RMPM standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 μL each of the test solution and the Saposhnikovia Root RMPM standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of dichloromethane, methanol and water (45:10:1) to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): the spots obtained from the test solution show the same color and R_f value as the spots from the Saposhnikovia Root RMPM standard solution and of these, a deep blue spot appears at the R_f values of 0.25 and 0.5.

Purity (1) ***Foreign matter*****—**The amount of stems and other foreign matter contained in Saposhnikovia Root is not more than 2.0 %.

(2) ***Heavy metals*****—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*****—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): Not more than 0.2 ppm.

(vi) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 7.0 %.

Acid-insoluble Ash Not more than 1.5 %.

Extract Content ***Dilute ethanol-soluble extract—*** Not less than 20.0 %.

Containers and Storage ***Containers—***Well-closed containers.

Sappan Wood

Sappan Lignum

Sappan Wood is the heartwood of *Caesalpinia sappan* Linné (Leguminosae).

Description Sappan Wood is the heartwood, long cylindrical, semicylindrical or stick-like in shape. External surface is yellowish red to grayish brown, with sometimes traces of sapwood in pale brown to grayish brown. It is often cut transversely or longitudinally. Texture is hard but longitudinally cut texture is easy to be broken. Transversely cut surface has distinct annual rings. Under a microscope, the transverse section reveals medullary rays consisting of 1 to 2 rows of cells. The vessels are about 160 μm in diameter and contain yellow-brown or red-brown substances. The xylem fiber is polygonal and very thick-walled. The xylem parenchymal 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, walls slightly lignified with pitting.
Sappan Wood is nearly odorless and slightly astringent.

Identification Weigh 0.5 g of pulverized Sappan Wood, add 10 mL of diluted ethanol, shake and filter. To 5 mL of filtrate, add 2 to 3 drops of sodium hydroxide TS: dark red color develops.

Purity (1) ***Foreign matter—***(i) Sapwood: Sappan Wood contains less than 3.0 % of sapwood other than heartwood.
(2) ***Heavy metals—***(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(3) ***Residual pesticides—***(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(4) ***Sulfur dioxide—***Not more than 30 ppm.
(5) Put a small piece of Sappan Wood in calcium hydroxide TS: no purpe-blue color develops.

Loss on Drying Not more than 13.0 % (6 hours).

Ash Not more than 2.5 %.

Extract Content ***Dilute ethanol–soluble extract—*** Not less than 5.0 %

Containers and Storage ***Containers—***Well-closed containers.

Schisandra Fruit

Schisandrae Fructus

Schisandra Fruit is the well ripe fruit of *Schisandra chinensis* Baillon (Schisandraceae). Schisandra Fruit, when dried, contains not less than 0.7 % of total sum of the contents 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).

Description Schisandra Fruit is sap fruit of irregular sphere or spheroid, about 6 mm in diameter. External surface is dark red to blackish brown, with wrinkles and occasionally with white powder. The flesh is pliant with 1 to 2 seeds present. The seeds are 2 mm to 5mm in length, kidney-shaped, externally yellow-brown to dark red-brown, lustrous, with distinct raphe on the dorsal side.
Schisandra Fruit is nearly odorless and has an acidic, later astringent and bitter taste.

Identification Weigh 1.0 g each of pulverized Schisandra Fruit and Schisandra Fruit RMPM, add 20 mL of dichloromethane, heat on a water bath for 30 minutes under a reflux condenser, filter and evaporate the filterate to dryness, respectively. Add 1 mL of methanol to each of the residue and use these solutions as the test solution and the standard solution of Schisandra Fruit RMPM, respectively. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 2 μL each of the test solution and the Schisandra Fruit RMPM standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with the upper layer of a mixture of petroleum ether, ethyl formate and formic acid (15:5:1) to a distance of about 10 cm and air-dry the plate. Spray evenly dilute sulfuric acid TS and heat at 105 °C: the spots obtained from the test solution show the same color and R_f value as the spots from the Schisandra Fruit RMPM standard solution and of these, the spots of schisandrin, gomisin A and gomisin N appear at the R_f values of 0.2, 0.25 and 0.45, respectively.

Purity (1) ***Foreign matter***—The amount of receptacle, peduncle and other foreign matter contained in Schisandra Fruit is not more than 1.0 %.
(2) ***Heavy metals—***(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 5.0 %.

Assay Weigh accurately about 0.5 g of the fine powder of Schisandra Fruit, add 20 mL of methanol, sonicate for 20 minutes and filter. To 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 10 mg of schisandrin RS, 10 mg of gomisin A RS and 10 mg of gomisin N RS, dissolve to make exactly 25 mL of methanol. Take exactly 2 mL each of these solutions, add exactly 20 mL of methanol and use this solution as the standard solution. Pipet 10 μL each 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}, A_{Tb} and A_{Tc}, of the test solution and A_{Sa}, A_{Sb} and A_{Sc}, of the standard solution, respectively.

$$\text{Amount (mg) of schisandrin } (C_{24}H_{32}O_7) = \text{amount (mg) of Schisandrin RS} \times \frac{A_{Ta}}{A_{Sa}} \times \frac{1}{5}$$

$$\text{Amount (mg) of gomisin A } (C_{23}H_{28}O_7) = \text{amount (mg) of Gomisin A RS} \times \frac{A_{Tb}}{A_{Sb}} \times \frac{1}{5}$$

$$\text{Amount (mg) of gomisin N } (C_{23}H_{28}O_6) = \text{amount (mg) of Gomisin N RS} \times \frac{A_{Tc}}{A_{Sc}} \times \frac{1}{5}$$

Operating conditions

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

Column: A stainless column, 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).

Mobile phase: A mixture of acetonitrile, water and formic acid (70:30:0.1).

Flow rate: 0.6 mL/min.

System suitability

System performance: When the procedure is run with 10 μL of the standard solution under the above operating conditions, schisandrin, gomisin A and gomisin N are eluted in this order with the resolution between their peaks being not less than 1.6.

System repeatability: When the test is repeated six times with 10 μL each of the standard solution under the above operating conditions: the relative standard deviation of the peak area of schisandrin, gomisin A and gomisin N is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Schizonepeta Spike

Schizonepetae Spica

Schizonepeta Spike is the spike of *Schizonepeta tenuifolia* Briquet (Labiatae).

Description Schizonepeta Spike is a thin, long barley ear-like shaped spike, purplish green-brown to green-brown, 5 cm to 10 cm in length, with calyx-tubes containing small labiate flower or often fruits and with short milky white hairs at the whole root.
Schizonepeta Spike 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 and distill. To 3 mL of the distillate, add 2 or 3 drops of 2,4-dinitrophenylhydrazine-ethanol TS: an orange-red precipitate is formed.

(2) Weigh 0.8 g of pulverized Schizonepeta Spike, add 20 mL of petroleum ether, 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. Perform the test with this solution as directed under Thin-layer Chromatography. Spot 10 μL of the test solution on a plate of silica gel with fluorescent indicator 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 shade. Spray evenly sulfuric acid TS for spraying on the plate and examine under ultraviolet light (main wavelength: 365 nm): a green spot appears at the R_f value of about 0.4.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 11.0 %.

Acid-insoluble Ash Not more than 3.0 %.

Extract Content ***Dilute ethanol-soluble extract—*** Not less than 8.0 %.

Containers and Storage ***Containers—***Well-closed containers.

Scopolia Extract

Scopolia Extract contains not less than 0.90 % and not more than 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 Extract the coarse powder of Scopolia Rhizome with 35 % ethanol, water, or purified water and prepare the viscous extract as directed under Extracts.

Description Scopolia Extract is brown to dark brown, and has a characteristic odor and bitter taste.
Scopolia Extract dissolves in water with a slight turbid.

Identification (1) Weigh 4 g of Scopolia Extract, dissolve in 10 mL of water, add 8 mL of ammonia TS and 80 mL of ether, stopper tightly, shake for 1 hour, add 2.5 g of powdered tragacanth, shake vigorously, allow to stand for 5 minutes and separate the ether layer into a porcelain dish. Evaporate the ether 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 tetraethyl-lammonium hydroxide TS: a red-purple to purple color is observed.

(2) Weigh 0.5 g of Scopolia Extract, add 30 mL of Ammonia TS, shake and transfer the mixture to a separatory funnel. Add 40 mL of ethyl acetate, shake, separate the ethyl acetate layer, add 3 g of anhydrous sodium sulfate, shake and filter after the solution is clear. Take the filterate, evaporate ethyl acetate in vaccum, dissolve the residue in 1 mL of ethanol and use this solution as the test solution. Proceed as directed in the Identification (2) under Scopolia Rhizome.

Purity (1) ***Heavy metals—***Total heavy metals: Not more than 30 ppm.

(2) ***Residual pesticides—***(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

Assay Weigh accurately about 0.4 g of Scopolia Extract, place in a glass-stoppered centrifuge tube, add 15 mL of ammonia TS and shake. To this solution, add 25 mL of ether, stopper tightly, shake for 15 minutes, centrifuge and separate the ether layer. Repeat this procedure twice with the water layer, using the ether on a water-bath. Dissolve the residue in 5 mL of the mobile phase, add 3.0 μL of the internal standard solution, add the mobile phase to the internal standard solution and add the mobile phase to make 25 mL. Proceed as directed under Scopolia Rhizome.

$$\text{Amount (mg) of hyoscyamine } (C_{17}H_{23}NO_3) = \text{amount (mg) of Atropine Sulfate RS, calculated on the dried basis} \times \frac{Q_{TA}}{Q_{SA}} \times \frac{1}{5} \times 0.8551$$

$$\text{Amount (mg) of scopolamine } (C_{17}H_{21}NO_4) = \text{amount (mg) of Scopolamine Hydrobromide RS, calculated on the dried basis} \times \frac{Q_{TS}}{Q_{SS}} \times \frac{1}{25} \times 0.7894$$

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

Containers and Storage ***Containers***—Tight containers.

Storage—Light-resistant, and in a cold place.

10 % Scopolia Extract Powder

10 % Scopolia extract powder contains not less than 0.09 % and not more than 0.11 % of total alkaloids [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 Hydrate or their mixture	a sufficient quantity
To make	1000 g

Take Scopolia Extract, add 100 mL of purified water, warm and soften the mixture with stirring. After cooling, add 800 g of Starch, Lactose Hydrate or their mixture little by little and mix well. Dry preferably at a low temperature and dilute with a sufficient additional quantity of Starch, Lactose Hydrate or their mixture to make 1000 g of homogeneous powder.

Description 10 % Scopolia Extract powder is a brownish yellow to grayish yellowish brown powder, and has a slight, characteristic odor and slightly bitter taste.

Identification (1) Weigh 20 g of 10 % Scopolia Extract powder add 15 mL of water and 8 mL of ammonia TS, mix homogeneously, add 100 mL of ether and 7 g of sodium chloride, stopper tightly, shake for 1 hour, add 5 g of powdered tragacanth and shake vigorously. Allow to stand for 5 minutes, take the clearly separated ether layer and filter. Proceed with the filtrate as directed in the identification (1) under Scopolia Extract.

(2) Weigh 5 g of 10 % Scopolia Extract Powder, transfer to a stoppered centrifuge tube, add 30 mL of ammonia TS, sonicate for 5 minutes, filter and centrifuge the filterate. Transfer the supernatant liquid to a separatory funnel, add 40 mL of ethyl acetate, shake, separate the ethyl acetate layer, add 3 g of anhydrous sodium sulfate, shake and filter after the ethyl acetate solution is clear. Evaporate ethyl acetate in vaccum, dissolve the residue in 1 mL of ethanol and use this solution as the test solution. . Proceed as directed in the Identification (2) under Scopolia Rhizome.

Purity (1) ***Heavy metals***—Total heavy metals: Not more than 30 ppm.

(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

Assay Weigh accurately about 4.0 g of 10 % Scopolia Extract powder, place 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 centrifuge to take the ether layer. Repeat this procedure three times with the water layer, using 25 mL volumes of ether. 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 to make exactly 25 mL. Proceed as directed under Scopolia Rhizome..

$$\text{Amount (mg) of hyoscyamine } (C_{17}H_{23}NO_3) = \text{amount (mg) of Atropine Sulfate RS, calculated on the dried basis} \times \frac{Q_{TA}}{Q_{SA}} \times \frac{1}{5} \times 0.8551$$

$$\text{Amount (mg) of scopolamine } (C_{17}H_{21}NO_4) = \text{amount (mg) of Scopolamine Hydrobromide RS, calculated on the dried basis} \times \frac{Q_{TS}}{Q_{SS}} \times \frac{1}{25} \times 0.7894$$

Containers and Storage ***Containers***—Tight containers.

Scopolia Rhizome

Scopoliae Rhizoma

Scopolia Rhizome is the rhizome of *Scopolia japonica* Max. or *Scopolia carniolica* Jacquin (Solanaceae). Scopolia Rhizome, when dried, contains not less than 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)].

Description Scopolia Rhizome is the rhizome, chiefly irregularly branched, slightly curved, about 5 cm to l5 cm in length and 1 cm to 3 cm in diameter. Constrictions make the rhizome appear nodular. Rarely, stem base is present at one end. Scars of stem are present at upper side of each node, and scars of roots or root are on lower surface of rhizome. External surface is grayish brown to blackish brown, with wrinkles. Fractured surface is grayish white to pale brown, granular and compact, with lighter colored cortex. Under a microscope, transverse sectioin reveals xylem with groups of vessels arranged stepwise and accompanied with xylem sieve tubes in medullary rays. Parenchyma cells contain starch grains and sometimes sand crystals of calcium oxalate.
Scopolia Rhizome has a characteristic odor and slightly sweet taste, later slightly bitter.

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 l0 mL of ether, transfer the filtrate and the washing to a separatory funnel, add 20 mL of diluted sulfuric acid (l in 50), shake well and drain off the acid extract into another separatory funnel. Render the solution slightly alkaline with ammonia TS, add 10 mL of ether, shake well, 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. Cool, dissolve the residue in 1 mL of dimethylformamide and add 5 to 6 drops of tetraethylammonium hydroxide TS: a red-purple to purple color develops.
(2) Weigh 2 g of pulverized Scopolia Rhizome, transfer to a stoppered centrifuge tube, add 30 mL of ammonia TS, sonicate for 5 minutes, filter and centrifuge the filterate. Transfer the supernatant liquid to a separatory funnel, add 40 mL of ethyl acetate, shake, separate the ethyl acetate layer, add 3 g of anhydrous sodium sulfate, shake and filter after the ethyl acetate solution is clear. Evaporate ethyl acetate in vaccum, 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 (l) and (2). Perform the test with the test solution and the standard solutions (1) and (2) as directed under the Thin-layer Chromatography. Spot 5 μL each of the test solution and the standard solutions (1) and (2) on a plate 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 °C for 10 minutes. After cooling, spray evenly Dragendorff's TS on the plate: two principal spots from the test solution and each yellow-red spot from the standard solutions (1) and (2) show the same color and the same R_f value.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

Ash Not more than 7.0 %.

Assay Weigh accurately about 0.7 g of pulverized Scopolia Rhizome, previously dried at 60°C for 8 hours, in a glass-stoppered centrifuge tube and moisten with l5 mL of ammonia TS. To this, add 25 mL of ether, stopper the centrifuge tube tightly, shake for 15 minutes, centrifuge and separate the ether layer. Repeat this procedure twice with the residue using 25 mL volumes of ether. 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 to make 25 mL. Filter this solution through a filter having a porosity of not more than 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 in the mobile phase to make exactly 25 mL and use this solution as the standard stock solution A. Weigh accurately about 25 mg of Scopolamine Hydrobromide RS (determined the loss on drying before use), dissolve in the mobile phase to make exactly 25 mL and use this solution as the standard stock solution B. Pipet 5 mL of standard stock solution A and 1 mL of standard stock solution B, add 3.0 mL of the internal standard solution, then add 25 mL of the mobile phase 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. Determine the ratios, Q_{TA} and Q_{SA}, for the test solution and the standard solution, respectively, of the peak area of hyoscyamine (atropine) and the ratios, Q_{TS} and Q_{SS}, for the test solution and the standard solution, respectively, of the peak area of scopolamine to that of the internal standard, calculate the amounts of hyoscyamine and scopolamine by the following equations and designate the total as the amount of tota1 alkaloids.

$$\text{Amount (mg) of hyoscyamine } (C_{17}H_{23}NO_3) = \text{amount (mg) of Atropine Sulfate RS, calculated on the dried basis} \times \frac{Q_{TA}}{Q_{SA}} \times \frac{1}{5} \times 0.8551$$

$$\text{Amount (mg) of scopolamine } (C_{17}H_{21}NO_4) = \text{amount (mg) of Scopolamine Hydrobromide RS, calculated on the dried basis} \times \frac{Q_{TS}}{Q_{SS}} \times \frac{1}{25} \times 0.7894$$

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 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 monobasic potassium phosphate in 900 mL of water, add 10 mL of triethylamine adjust with phosphoric acid to make a pH of 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 operating conditions use a column giving elution of scopolamine, atropine and the internal standard in this order with, clearly dividing each peak.

Containers and Storage ***Containers*—**Well-closed containers.

Scrophularia Root

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 is yellowish brown-brown, with rough longitudinal wrinkles, transverse lenticels and sparse rootlet scars. Texture is compact and flexible, hard to be fractured, and the fractured surface is black to blackish

brown.
Scrophularia Root 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 for 2 to 3 minutes in a water-bath and filter. Add 2 mL of Fehling TS to 4 mL of the filtrate and heat in 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 in a water-bath and filter. The filtrate is evaporated to dryness, add 4 mL of acetic anhydride to the residue, warm for 2 minutes and filter. After cooling, add carefully 1 mL of sulfuric acid to the filtrate: a red-brown color develops at the zone of contact.

(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 Harpagoside RS in 1 mL of diluted ethanol (7 in 10) and use this solution as the standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 2 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 butanol, water and acetic acid (7 : 2 : 1) 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: one of the spots obtained from the test solution shows the same color and R_f value as the pink spot from the standard solution.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 17.0 % (6 hours).

Ash Not more than 6.0 %.

Acid-insoluble Ash Not more than 2.0 %.

Extract Content ***Dilute ethanol-soluble extract***—Not less than 24.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Scutellaria Root

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 not less than 10.0 % of total sum of baicalin ($C_{21}H_{18}O_{11}$: 446.37), baicalein ($C_{15}H_{10}O_5$: 270.24) and woogonin ($C_{16}H_{12}O_5$: 284.28), calculated on the dried basis.

Description Scutellaria Root is the conical root, twisted and curved, 8 cm to 25 cm in length and 1 cm to 3 cm in diameter. The external surface is yellow-brown or deep yellow, sparsely scattered with strumous rootlet scars. The upper part has relatively coarse, twisted, curved longitudinal wrinkles or an irregular reticulation. The texture is hard but brittle and easily cut. The cut surface is yellow and the middle part is red-brown. Older roots are decayed or hollow in the middle and dark brown or red-brown in color.
Scutellaria Root is almost odorless and it has a slightly bitter taste.

Identification (1) Weigh 0.5 g of pulverized Scutellaria Root, add 20 mL of ether, heat with a reflux condenser for 5 minutes, cool and filter. Evaporate the filtrate, dissolve the residue in 10 mL of ethanol and to 3 mL of the solution, add 1 to 2 drops of dilute iron (III) chloride TS: a grayish green color develops and changes to purple-brown.

(2) Weigh 1 g each of pulverized Scutellaria Root and Scutellaria Root RMPM, add 30 mL of a mixture of ethyl acetate and methanol (3 : 1), heat on a water bath for 30 minutes under a reflux condenser and filter, respectively. Evaporate the filtrates to dryness, dissolve the residues to 5 mL of methanol and use these solutions as the test solution and the standard solution of Scutellaria Root RMPM. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 2 μL each of the test solution and the Scutellaria Root RMPM standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, methanol and water (100 : 17 : 13) 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 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: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not

more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 15.0 %.

Ash Not more than 6.0 %.

Acid-insoluble Ash Not more than 1.0 %.

Assay Weigh accurately about 0.5 g of pulverized Scutellaria Root, add 40 mL of diluted ethanol (7 in 10), heat 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 each of Baicalin RS (previously dried in a silica gel desiccator for not less than 24 hours), Baicalein RS (previously dried in a silica gel desiccator for not less than 24 hours) and Woogonin RS (previously dried in a silica gel desiccator for not less than 24 hours). To each, add 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 test solution 1, test solution 2 and the standard solution as directed under Liquid Chromatography according to the following operating conditions and determine the peak areas, A_{Tb} and A_{Tc}, of baicalein and woogonin in test solution 1, the peak area, A_{Ta}, of baicalin in test solution 2, and the peak areas, A_{Sa}, A_{Sb} and A_{Sc}, of baicalin, baicalein and woogonin in the standard solution.

$$\text{Amount (mg) of baicalin } (C_{21}H_{18}O_{11}) = \text{amount (mg) of Baicalin RS} \times \frac{A_{Ta}}{A_{Sa}} \times 5$$

$$\text{Amount (mg) of baicalein } (C_{15}H_{10}O_5) = \text{amount (mg) of Baicalein RS} \times \frac{A_{Tb}}{A_{Sb}} \times \frac{1}{2}$$

$$\text{Amount (mg) of woogonin } (C_{16}H_{12}O_5) = \text{amount (mg) of Woogonin RS} \times \frac{A_{Tc}}{A_{Sc}} \times \frac{1}{2}$$

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 277 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 to 10 μm in particle diameter).

Column temperature: 40 °C.

Mobile phase: Control the mobile phase A and B stepwise or gradient as the following conditions.

Mobile phase A: Diluted acetic acid (1 in 100)

Mobile phase B: A mixture of acetonitrile, methanol and acetic acid (7 : 3 : 0.01)

Time (min)	Mobile phase A (%)	Mobile phase B (%)
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, Baiclein RS, Woogonin RS and methyl *p*-hydroxybenzoate in methanol to make 100 mL. When the procedure is run with 10 μL of this solution under the above operating conditions, use a column giving elution of methyl *p*-hydroxybenzoate, baicalin, baicalein and woogonin in this order and clearly dividing each peak.

System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions, the relative standard deviation of the peak area of baicalin, baicalein and woogonin is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Senega

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 is pale grayish brown to grayish brown, 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 a grayish brown cortex,

the xylem is nearly white, usually circular, occasionally dented cuneate to semi-circular. The cortex on the opposite side is thick. Under a microscope, the transverse section 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 medullary rays, all containing substances in the shape of oil droplets. The sieve tubes are gathered only on the outside of normally developed xylem. The xylem is usually circular, occasionally cuneate to semi-circular, the cortex on the opposite side forming a thick ridge. The cuneate part is filled with unlignified parenchyma cells, the membrane wall usually not lignified in *Polygala senega* Linné and slightly lignified in *Polygala senega* Linné var. *latifolia* Torrey et Gray. The medullary rays are difficult to distinguish from other tissue but consist of slightly thin membranes radiating, with no pith visible. The parenchyma cells contain oil droplets, but starch grains and calcium oxalate crystals are absent.
Senega 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 30 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 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 Spectrophotometry: it exhibits a maximum at about 317 nm.

Purity (1) ***Foreign matter*—**(i) Stem: Senega contains lessthan 2.0 % of stems.

(ii) Other foreign matter: Senega contains less than 1.0 % of foreign matter other than stems.

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

Loss on Drying Not more than l3.0 % (6 hours).

Ash Not more than 5.0 %.

Acid-insoluble Ash Not more than 2.0 %.

Extract Content ***Dilute ethanol–soluble extract*—**Not less than 30.0 %

Containers and Storage ***Containers*—**Well-closed containers.

Senna Leaf

Sennae Folium

Senna Leaf is the leaflets of *Cassia angustifolia* Vahl or *Cassia acutifolia* Delile (Leguminosae). Senna Leaf contains not less than 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, pale grayish yellow to pale grayish yellow-green. Margin is entire and apex is acute. The base is asymmetric and primary lateral veins are running toward the apex along the margin and joining together. The upper surface is flat, the lower surface has slight hairs, vein of lower surface is marked and petiole of leaflet is short. Under a microscope, a transverse section reveals epidermis with thick cuticle, with numerous stomata and with thick-walled, warty unicellular hairs. Epidermal cells are often separated into two loculi by a septum which is in parallel with the surface of the leaf and contain mucilage in the inner loculus. Palisade of a single layer is under each upper and lower epidermis. Spongy tissue is consisted of 3 to 4 layers and contains clustered or solitary crystals of calcium oxalate. Cells adjacent to vascular bundle forms crystal cell rows.
Senna Leaf has a slight odor and bitter taste.

Identification (1) Weigh 0.5 g of pulverized Senna Leaf, add 10 mL of ether for 2 minutes and filter. Add 5 mL of ammonia TS to the filtrate: a yellow-red color is produced in the water layer. To the residue of maceration, add 10 mL of water and macerate for 2 minutes. Filter and add 5 mL of ammonia TS: a yellow-red color is produced in the water layer.

(2) Weigh 2 g of pulverized Senna Leaf, add 40 mL of a mixture of tetrahydrofuran and water (7 : 3), shake for 30 minutes and centrifuge. Transfer the supernatant liquid to a separatory funnel, add 13 g of sodium chloride and shake for 30 minutes. Separate the water layer with 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, separate the tetrahy-drofuran 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. Perform the test as directed under the Thin-layer Chromatography with the test solution and the standard solution. Spot 10 μL each of the test solution and the

standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, *n*-propanol, water and acetic acid (100) (40 : 40 : 30 : 1) to a distance of about 15 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 365 nm): one spot among the spots from the test solution and a red fluorescent spot from the standard solution show the same color and R_f value.

Purity (1) ***Foreign matter*—**(i) Rachis and fruit: Senna Leaf contains less than 5.0 % of petiols and fruits.

(ii) Other foreign matter: Senna Leaf contains less than 1.0 % of foreign matter other than petiols and fruits.

(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

Loss on Drying Not more than 12.0 % (6 hours).

Ash Not more than 12.0 %.

Acid-insoluble Ash Not more than 2.0 %.

Assay Weigh accurately about 0.5 g of pulverized Senna Leaf in a glass-stoppered centrifuge tube, add 25 mL of diluted methanol (7 in 10), shake for 30 minutes, centrifuge and separate the supernatant liquid. To the residue, add twice 10 mL of diluted methanol (7 in l0), shake each for 10 minutes, centrifuge and separate the supernatant liquid, respectively. 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 silica gel desiccator for not less than 24 hours), dissolve in diluted 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 silica gel desiccator for not less than 24 hours), dissolve in diluted sodium bicarbonate (1 in 100) to make exactly 20 mL and use this solution as the standard stock solution (2). Pipet 5.0 mL of the standard stock solution (1) and 10.0 mL of the standard stock solution (2), add methanol to make exactly 50 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_{Sa}, of sennoside A, for the test solution and the standard solution, respectively and the peak areas, A_{Tb} and A_{Sb}, of sennoside B for the test solution and the standard solution, respectively,, calculate the amounts of sennoside A and sennoside B by the following equations and designate the total as the amount of total 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 20 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm to l0 μm in particle diameter).

Column temperature: A constant temperature of about 50 °C.

Mobile phase: Dissolve 2.45 g of tetra-*n*-heptylammonium bromide in l00 mL of a mixture of diluted 1 mo1/L acetic acid-sodium acetate buffer solution, pH 5.0 (1 in 10) and acetonitrile (17 : 8).

Flow rate: Adjust the flow rate so that the retention time of sennoside A is about 26 minutes.

System suitability

System performance: When the procedure is run with 10 μL of the standard solution under the above operating conditions: sennoside B and sennoside A are eluted in this order, clearly dividing each peak.

System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions: the relative standard deviation of the peak area of sennoside A is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Sinomenium Stem and Rhizome

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 stem and rhizome, long cylindrical, slightly curved, 20 cm to 70 cm in length and 0.5 cm to 2 cm in diameter. The external surface is greenish brown, maroon or

grayish brown, with the nodes slightly expanded. The body is light and the texture is firm but fragile and easy to cut. The cut surface is grayish yellow or pale grayish brown, the cortex is narrow, medullary rays radiate in the xylem and the pith is pale yellowish white or yellowish brown. Under a microscope, the transverse section reveals the epidermis of the outermost layer covered in a thick horny layer or consisting of a cork layer. The cortex is scattered with fibers and stone cells. The stele sheath fiber bundles are crescentic with 2 to 5 rows of stone cells on the inside. The stone cells reach out to connect with stone cell bundles in the medullary rays, forming a ring. The vascular bundles are collateral. In the phloem, the medullary rays become gradually broader towards the outside 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 individual vessels or several vessels connecting down lengthwise. The pith cells have thick cell walls and distinct pits. The parenchyma cells contain starch grains and needle crystals of calcium oxalate.
Sinomenium Stem and Rhizome is nearly odorless and has bitter taste.

Identification Weigh 0.5 g of pulverized Sinomenium Stem and Rhizome add 10 mL of dilute acetic acid, heat for 2 minutes in a water-bath with frequent shaking, cool and filter. To 5 mL of the filtrate, add 2 drops of Dragendorff's TS: immediately, an orange-yellow precipitate is produced.

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, dissolve 1 mL of ethanol and use this solution as the test solution. Separately, weigh 1 mg of Aristolokinic acid RS, dissolve it in 1 mL of ethanol, and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 5 μL each of the test solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of toluene, ethyl acetate, methanol and formic acid (20 : 10 : 1 : 1) to a distance of about 10 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm) or spray evenly aluminum chloride TS and examine under ultraviolet light (main wavelength: 365 nm): one spot among the spots 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: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 6.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Sophora Flower

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 mm to 6 mm in length, about 2 mm in diameter. The lower part of calyx has several longitudinal scars. The upper part of calyx has yellowish white petals. The stalk is thin and small. The body is light and breaks upon rubbing by hand.
Koemi has a slight, characteristic odor and tastes slightly bitter and astringent.

(2) ***Koehwa***—Koehwa is the flower, wrinkled and rolled. Calyx is campanulate and yellowish-green, to lobed at the apex. Numbers of petals are 5, yellow to yellowish white. One of petal is relatively large, nearly round, with apex hollowed slightly. The others are long round. Numbers of stamens are 10, accreted at the base of 9 stamens. The stalk of stamen is thin and long. Pistil is cylindrical and curved.
Koehwa has a slight, characteristic odor and tastes slightly bitter.

Identification (1) Weigh 0.5 g of the pulverized Sophora Flower, add 10 mL of methanol and extract and filter. Perform the following test with the filtrate.

(i) Take 2.0 mL of the filtrate, add a small amount of magnesium powder and 2 to 3 drops of hydrochloric acid: a red color is produced.

(ii) Drop 2 to 3 drops of the filtrate on the filter paper and drop 1 % alum solution onto the filtrate: at the zone of two liquids yellow color is produced. And examine under ultraviolet light: test solution produces yellowish-brown and the zone of two liquids produces yellow fluorescence.

(2) Weigh 0.2 g of pulverized Sophora Flower, add 5 mL of methanol, shake for 10 minutes, 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. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 10 μL each of the test solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer

chromatography. Develop the plate with a mixture of ethyl acetate, formic acid and water (8 : 1 : l) 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): one spot among the spots 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***—Sophora Flower contains not more than 10 % of flower stalk and other foreign matter.

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 9.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Sophora Root

Sophorae Radix

Sophora Root is the root of *Sophora flavescens* Solander ex Aiton (Leguminosae), with or without periderm. Sophora Root, when dried, contains not less than 1.0 % of the sum of oxymatrine ($C_{15}H_{24}N_2O_2$: 264.36), and matrine ($C_{15}H_{24}N_2O$: 248.36).

Description Sophora Root is cylindrical root, 5 cm to 20 cm in length and 2 cm to 3 cm in diameter. External surface is dark brown to yellow-brown, with distinct longitudinal wrinkles and with laterally extended lenticels. External surface of root without periderm is yellowish white, with somewhat fibrous surface. The transverse section is pale yellow-brown. The cortex is 1 mm to 2 mm in thickness, slightly tinged with dark color near cambium, forming a crack between xylem. Sophora Root has a slight odor and tastes extremely bitter and lasting.

Identification (1) Weigh 0.5 g of pulverized Sophora Root, add 10 mL of dilute acetic acid, heat in a water-bath for 3 minutes with occasional 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 1 g of pulverized Sophora Root and Sophora Root RMPM, add 50 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrates, dissolve in 1 mL of methanol and use these solutions as the test solution and the Sophora Root RMPM standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 4 μL each of the test solution and the Sophora Root RMPM standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of cyclohexane, acetone and ethyl acetate (5:2:1) 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 spots obtained from the test solution show the same color and Rf value as spots from the Sophora Root RMPM standard solution, and a red-brown spot is observed at the R_f value of about 0.7.

Purity (1) ***Foreign matter*—**(i) Stem: Not more than 10.0 %.
(ii) Other foreign matter: The amount of foreign matter other than stems contained in Sophora Root is not more than 1.0 %.

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 6.0 %.

Acid-insoluble Ash Not more than 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 dryness in vacuum, and add water to dissolve completely, followed by addition of 10 % hydrochloride solution to adjust to pH 2. Add 50 mL of dichloromethane to wash, collect the aqueous layer, add 50mL of dichloromethane and proceed in the same manner. Add potassium carbonate to aqueous layer to adjust pH to 10, add 50 mL of dichloromethane, extract with occasional shaking. Add 50mL dichloromethane to aqueous layer, and proceed in the same manner. Combine all the extracts and evaporate to dryness in vacuum. To the residue, add methanol to make exactly 10 mL, and use this solution as the test solution. Separately, weigh accurately 20 mg of Oxymatrine RS and 10mg of

Matrine RS, dissolve in methanol to make exactly 10 mL, and use this solution as the standard solutions. 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 operating conditions. 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 solution.

$$\text{Amount (mg) of oxymatrine } (C_{15}H_{24}N_2O_2) = \text{Amount (mg) of Oxymatrine RS} \times \frac{A_{Ta}}{A_{Sa}}$$

$$\text{Amount (mg) of matrine } (C_{15}H_{24}N_2O) = \text{Amount (mg) of Matrine RS} \times \frac{A_{Tb}}{A_{Sb}}$$

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: When the procedure is run with 10 μL of the standard solutions under the above operating conditions, oxymatrine and matrine are eluted in this order with clearly dividing each peak.

System repeatability: When the test is repeated six times with 10 μL each of the standard solutions under the above operating conditions: the relative standard deviation of the peak area of oxymatrine and matrine is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Sparganium Rhizome

Sparganni 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. External surface is yellowish white or graysh yellow, with marks pared with a knife and fibrous root scars spotted, slightly ringed-arranged transversely. The body is heavy and the texture is compact. Under a microscope, the transverse section reveals the cortex consisting of aerenchyma tissue, parenchyma cells are irregular in shape with large cavities between cells. Cells are tightly arranged in the endodermis. Parenchyma cells of the stele are close to circular, the cell walls mostly thickened, containing starch grains inside. The vascular bundles are collateral and amphivasal, scattered, and vessels are not lignified. Secretory cells are evely scattered in the cortex and stele and contain red-brown secretion.

Sparganium Rhizome is nearly odorless and taste is week, slightly numb on chewing.

Identification Weigh 2 g each of pulverized Sparganium Rhizome and Sparganium Rhizome RMPM, add 30 mL of ethanol, heat under a reflux condensor for 10 minutes on a water-bath, filter and evaporate to dryness. To the each residue, add 2 mL of ethanol and use each solution as the test solution and Sparganium Rhizome RMPM standard solution. Perform the test with the test solution and Sparganium Rhizome RMPM standard solution as directed under the Thin-layer Chromatography. Spot 10 μL each of the test solution and Sparganium Rhizome RMPM standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of cyclohexane and ethyl acetate (4 : 1) to a distance of about 10 cm and air-dry the plate. Spray evenly diluted sulfuric acid TS and heat at 105 °C for 10 minutes: the spots from the test solution and the spots from Sparganium Rhizome RMPM standard solution show the same color and the same R_f value.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 10.0 %.

Ash Not more than 5.0 %.

Acid-insoluble Ash Not more than 1.0 %.

Extract Content ***Dilute ethanol-soluble extract***— Not less than 11.0 %

Containers and Storage ***Containers*—**Well-closed containers.

Ssanghwatang Extract Granules

Ssanghwatang Extract Granules contains not less than 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 for a 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 herbal drugs to coarse powder, weigh each herbal drugs, put into the extractor, add eight to ten fold of water, extract for 2 to 3 hours at 80 ~ 100 °C and filter. Vacuum-concentrate the filtrate under 60 °C until it becomes 3.37 g to 5.05 g of Viscous extract or concentrate in a suitable method until it becomes 1.32 g to 1.98 g of Dry extract. Ssanghwatang Extract Granules is prepared as directed under Granules.

Identification (1) ***Peony Root***—Pulverize Ssanghwa-tang Extract Granules, weigh an amount, equivalent to 1 g of Peony Root, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 pulverized Peony Root, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under Thin-layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a lower layer of the mixture of chloroform, methanol and water (26 : 14 : 5) to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with *p*-anisaldehydesulfuric acid TS and heat at 105 °C for 10 minutes: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(2) ***Angelica Gigas Root***—Pulverize Ssanghwatang Extract Granules, weigh an amount, equivalent to 1 g of Angelica Gigas Root, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 pulverized Angelica Gigas Root, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under 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 toluene, ether, water and acetic acid (500 : 500 : 5 : 2) 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 spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(3) ***Prepared Rehmannia Root***—Pulverize Ssanghwatang Extract Granules, weigh an amount, equivalent to 1 g of Prepared Rehmannia Root, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 pulverized Prepared Rehmannia Root, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under Thin-layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of chloroform and methanol (20 : 1) to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with 2,4-dinitrophenylhydrazine TS. Examine under ultraviolet light (main wavelength: 254 nm): one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(4) ***Cnidium Rhizome***—Pulverize Ssanghwatang 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 with a reflux condenser 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 pulverized Cnidium Rhizome, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under Thin-layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of cyclohexane and ethyl acetate (9 : 1) to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with sulfuric acid TS for spray and heat at 105 °C for 10 minutes. Examine under ultraviolet light (main wavelength: 365 nm): one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(5) ***Astragalus Root***—Pulverize Ssanghwatang Ex-

tract Granules, weigh an amount, equivalent to 1 g of Astragalus Root, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 pulverized Astragalus Root, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 20 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under Thin-layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a plate 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 evenly the plate with vanillin-sulfuric acid TS and heat at 105 °C for 10 minutes. Examine under ultraviolet light (main wavelength: 365 nm): one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(6) ***Licorice***—Pulverize Ssanghwatang Extract Granules, weigh an amount, equivalent to 1 g of Licorice, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 pulverized Licorice, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under 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 chloroform and methanol (95 : 5) to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with *p*-anisaldehydesulfuric acid TS and heat at 105 °C for 10 minutes: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(7) ***Cinnamon Bark***—Pulverize Ssanghwatang Extract Granules, weigh an amount, equivalent to 1 g of Cinnamon Bark, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 pulverized Cinnamon Bark, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under 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 (85 : 15) to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with a saturated solution of *o*-dianisidine-acetic acid (100) freshly prepared: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(8) ***Jujube***—Pulverize Ssanghwatang Extract Granules, weigh an amount, equivalent to 1 g of Jujube, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 pulverized Jujube, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under 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 chloroform and methanol (6 : 1) 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 °C for 10 min: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(9) ***Ginger***—Pulverize Ssanghwatang Extract Granules, weigh an amount, equivalent to 1 g of Ginger, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 pulverized Ginger, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under 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 (85 : 15) 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 °C for 10 min: one spot among the 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) Total heavy metals: Not more than 30 ppm.

(ii) Lead: Not more than 5 ppm.

(iii) Arsenic: Not more than 3 ppm.

Disintegration Test It meets the requirement.

Particle Size Distribution Test for Preparations It meets the requirement.

Uniformity of Dosage Units (divided) It meets the

requirement.

Microbial Limit It meets the requirement.

Assay (1) ***Paeoniflorin of Peony Root***—Take not less than 20 sachets of Ssanghwatang Extract Granules, weigh accurately and pulverize. Weigh accurately equivalent to about 10 mg of paeoniflorin, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat 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, vacuum-concentrate the filtrate 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.

(2) ***Glycyrrhizic acid of Licorice***—Take not less than 20 sachets of Ssanghwatang Extract Granules, weigh accurately and pulverize. Weigh accurately equivalent to 10 mg of glycyrrhizic acid, add 50 mL of water, warm with a reflux condenser for 3 hours, add 50 mL of 3 mol/L of sulfuric acid TS and hydrolyze in a water bath for 1 hour. After cooling, add 50 mL of chloroform, warm with a reflux condenser for 30 minutes. After cooling, take the chloroform layer in separatory funnel, add 30 mL of chloroform, extract three times repetitively, combine chloroform layers and filter through anhydrous sodium sulfurate. 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 silica gel desiccator for 24 hours), use the solution, prepared prepare the solution, prepared in the same manner as the test solution, and use this 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 operating conditions. Determine the peak areas, A_T and A_S, of 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 (5 μm in particle diameter).

Mobile phase: A mixture of methanol, water and acetic acid (100) (78 : 19 : 3)

Flow rate: 1.0 mL/min

Containers and Storage ***Containers***—Tight containers.

Ssanghwatang Solution

Ssanghwatang Solution contains not less than 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 for a dose (one bottle).

Method of Preparation for a dose (one bottle)

Peony Root	3.13 g
Angelica Gigas Root, Prepared Rehmannia Root, Cndium Rhizome, Astragalus Root	1.25 g
Licorice, Cinnamon Bark	0.94 g
Jujube	0.67 g
Ginger	0.50 g
Purified water	a sufficient quantity
	To make 100 mL

Pulverize the above herbal drugs to coarse powder, weigh each herbal drugs, put into the extractor, add eight to ten fold of water, extract for 2 to 3 hours at 80 ~ 100 °C and filter. Ssanghwatang Solution is prepared as directed under Solutions.

Identification (1) ***Peony Root***—Take equivalent to 1 g of Peony Root, add 100 mL of methanol, shake for 1 hour, vacuum-concentrate the filtrate until the filtrate becomes 20 mL and use this solution as the test solution. Separately, weigh 1 g of pulverized Peony Root, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Evaporate the filtrate to dryness in vacuum until the filtrate becomes 20 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 lower layer of the mixture of chloroform, methanol and water (26 : 14 : 5) 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 °C for 10 minutes: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(2) ***Angelica Gigas Root***—Take equivalent to 1 g of Angelica Gigas Root, add 100 mL of methanol, shake for 1 hour, vacuum-concentrate the filtrate until the filtrate becomes 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 with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 20 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 toluene, ether, water and acetic acid (500 : 500 : 5 : 2) 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 spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(3) ***Prepared Rehmannia Root***—Take equivalent to 1 g of Prepared Rehmannia Root, add 100 mL of methanol, shake for 1 hour, vacuum-concentrate the filtrate until the filtrate becomes 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 with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 20 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of chloroform and methanol (20 : 1) to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with 2,4-dinitrophenylhydrazine TS. Examine under ultraviolet light (main wavelength: 254 nm): one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(4) ***Cnidium Rhizome***—Take equivalent to 1 g of Cnidium Rhizome, add 100 mL of methanol, shake for 1 hour, vacuum-concentrate the filtrate until the filtrate becomes 20 mL and use this solution as the test solution. Separately, weigh 1 g of pulverized Cnidium Rhizome, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 20 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of cyclohexane and ethyl acetate (9 : 1) to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with sulfuric acid TS for spray and heat at 105 °C for 10 minutes. Examine under ultraviolet light (main wavelength: 365 nm): one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(5) ***Astragalus Root***—Take equivalent to 1 g of Astragalus Root, add 100 mL of methanol, shake for 1 hour, vacuum-concentrate the filtrate until the filtrate becomes 20 mL and use this solution as the test solution. Separately, weigh 1 g of pulverized Astragalus Root, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 20 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 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 evenly the plate with vanillin-sulfuric acid TS and heat at 105 °C for 10 minutes. Examine under ultraviolet light (main wavelength: 365 nm): one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(6) ***Licorice***—Take equivalent to 1 g of Licorice, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 pulverized Licorice, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 chloroform and methanol (95 : 5) 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 °C for 10 minutes: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(7) ***Cinnamon Bark***—Take equivalent to 1 g of Cinnamon Bark, add 100 mL of methanol, shake for 1 hour, vacuum-concentrate the filtrate until the filtrate becomes 20 mL and use this solution as the test solution. Separately, weigh 1 g of pulverized Cinnamon Bark, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 20 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 (85 : 15) to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with a saturated solution of *o*-dianisidine-acetic acid (100) (make when used): one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(8) ***Jujube***—Take equivalent to 1 g of Jujube, add 100 mL of methanol, shake for 1 hour, vacuum-concentrate the filtrate until the filtrate becomes 20 mL and use this solution as the test solution. Separately, weigh 1 g of pulverized Jujube, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate be-

comes 20 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 chloroform and methanol (6 : 1) 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 °C for 10 min: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(9) ***Ginger***—Take equivalent to 1 g of Ginger, add 100 mL of methanol, shake for 1 hour, vacuum-concentrate the filtrate until the filtrate becomes 20 mL and use this solution as the test solution. Separately, weigh 1 g of pulverized Ginger, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 20 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 (85 : 15) 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 °C for 10 min: one spot among the 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) Total heavy metals: Not more than 30 ppm.

(ii) Lead: Not more than 5 ppm.

(iii) Arsenic: Not more than 3 ppm.

pH 3.0 ~ 5.0

Specific Gravity $[\alpha]_D^{20}$: 0.980 ~ 1.080

Uniformity of Dosage Units (divided) It meets the requirement.

Microbial Limit It meets the requirement.

Assay (1) ***Paeoniflorin of Peony Root***—Pipet an amount of Ssasnghwatang Solution, equivalent to about 10 mg of paeoniflorin, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat 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, vacuum-concentrate the filtrate 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. Perform the test as directed in the Assay under Peony Root.

(2) ***Glycyrrhizic acid of Licorice***—Take accurately equivalent to 1 g of glycyrrhizic acid, warm with a reflux condenser for 3 hours, add 50 mL of 3 mol/L of sulfuric acid TS and hydrolyze in a water bath for 1 hour. After cooling, add 50 mL of chloroform, heat and warm with a reflux condenser for 30 minutes. After cooling, take the chloroform layer in separatory funnel, add 30 mL of chloroform, extract three times repetitively, combine chloroform layers and filter through anhydrous sodium sulfurate. Vacuum-concentrate the filtrate, dissolve the residue in methanol to make exactly 50 mL, and use this solution as the test solution. Separatly, weigh accurately about 10 mg of Glycyrrhizic acid RS (previously dried in a silica gel desiccator for 24 hours), prepare the solution, prepared in the same manner as the test solution, and use this solution as the standard solution. Pipet 10 μL each 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_T and A_S, of 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 (5 μm in particle diameter).

Mobile phase: A mixture of methanol, water and acetic acid (100) (78 : 19 : 3)

Flow rate: 1.0 mL/min

Containers and Storage ***Containers***—Tight containers.

Star Anis Fruit

Illici Veri Fructus

Star Anis Fruit is the dried fruit or the dried fruit passed through hot water of *Illicium verum* Hook. fil..

Description Star Anis Fruit is mostly aggregate fruit of 8 follicles radiated from the central axis. Each follicle is 1 cm to 2 cm in length, 3 mm to 5 mm in width, and 6 mm to 10 mm in height. External surface is reddish brown, irregularly wrinkled, with summit beaked and upper part mostly dehiscent. Inner surface is pale brown, smooth and lustrous. Texture is hard and brittle. Fruit stalk is 3 cm to 4 cm in length, connected to the base of the fruit, curved, and usually deciduous. Each follicle contains a compressed-ovoid seed which is about 6 mm in length, reddish brown, lustrous, with a

hilum at the acute end. Endosperm is white and oily.
Star Anis Fruit is aromatic and taste is pungent and sweet.

Identification Weigh 1 g of pulverized star anis fruit, add 10 mL of hexane, shake to mix, allow to stand for 5 minutes and filter. Use this solution as the test solution. Separately, dissolve 1 mg each of Anetole RS and Anesaldehyde RS to 1 mL of ethanol and use these solutions as the standard solution (1) and the standard solution (2), respectively. Perform the test with the test solution and the standard solution (1), (2) as directed under Thin-layer Chromatography. Spot 5 μL each of the test solution and the standard solution (1), (2) on a plate of silica gel with a fluorescent indicator for thin-layer chromatography. Develop the plater 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 (main wavelength: 254 nm): Two spots among the several spots from star anis fruit and the each spot from the standard standard (1) and (2) show the same color and the same R_f value.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 11.0 %

Ash Not more than 4.0 %

Essential Oil Content Not less than 0.4 mL (10.0 g)

Extract Content ***Dilute ethanol-soluble extract*** Not less than 15.0 %

Containers and Storage ***Containers*—**Well-closed containers.

Swertia Herb

Swertia Herba

Swertia Herb is the whole herb of *Swertia japonica* Makino (Gentianaceae) collected during the blooming season. Swertia Herb contains not less than 2.0 % of swertiamarin ($C_{16}H_{22}O_{10}$: 374.34), calculated on the dried basis.

Description Swertia Herb is the whole herb, consisted of flowers, opposite leaves, stems, usually short, lignified roots, and 20 cm in length. The leaves and stems are dark green to dark purple or yellow-brown, and the flowers are white to whitish. The stems are cylindrical, about 2 mm in diameter, occasionally with branches. The root is yellow-brown. The leaves are crumpled and when smoothed by immersion in water, the leaves are liniear to narrow lanceolate, 1 cm to 4 cm in length, 1 mm to 5 mm in width, entire and sessile. The corolla is split deeply as 5 lobes, the lobes are narrow and oblong with distinct peduncles. There are five stamens growing on the tube of the corolla and stand alternately in a row with corolla-lobes. Under a magnifying glass, the corolla reveals two elliptical nectarines juxtaposed at the base of the inner surface, and the margin of the lobe resembles eyelashes.
Swertia Herb has a slight, characteristic odor and an extremely bitter and lasting taste.

Identification Weigh 2 g of pulverized Swertia Herb, add 10 mL of ethanol, shake for 5 minutes, filter, and use the filtrate as the test solution. Separately, weigh 2 mg of Swertiamarin RS, dissolve in 1 mL of ethanol, and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under Thin-layer Chromatography. Spot 10 μL each of the test solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Then develop the plate with a mixture of ethyl acetate, *n*-propanol and water (6 : 4 : 3) to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (broad spectrum wavelength): one spot among the several spots from the test solution and a red spot from the standard solution show the same color and the same R_f value.

Purity (1) ***Foreign matter***—The amount of straw and other foreign matters contained in Swertia Herb is not more than 1.0 %.
(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.

Loss on Drying Not more than 12.0 % (6 hours).

Ash Not more than 6.5 %.

Assay Weigh accurately about 1 g of pulverized

Swertia Herb in a glass-stoppered centrifuge tube, add 40 mL of methanol, shake for 15 minutes, centrifuge, and separate the supernatant liquid. To the residue add 40 mL of methanol 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), add methanol to make exactly 20 mL. Pipet exactly 5.0 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 the Liquid Chromatography according to the following operating conditions, and determine A_T and A_S, of the peak area of swertiamarin 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 column, 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: Dissolve 1 mg each of Swertiamarin RS and theophylline in the mobile phase to make 10 mL. Whe the procedure is run with 10 μL of this solution according to the above operating conditions, theophylline and swertiamarin are eluted in this order, with clearly dividing each peak.

System repeatability: When the test is repeated six times with 10 μL each of the standard solution under the above operating conditions, the relative standard deviation of the peak area of swertiamarin is not more than 1.5 %.

Containers and Storage ***Containers*—**Well-closed containers.

Terminalia Fruit

Terminaliae Fructus

Terminalia Fruit is the ripe fruit of *Terminalia chebula* Retzins or *Terminalia chebula* Retzins var. *tomentella* Kurt. (Combretaceae).

Description Terminalia Fruit is the fruit, oblong or ovoid, 2 cm to 4 cm in length, 20 mm to 25 mm in diameter. External surface is yellowish-brown to dark brown, ofter lustrous, with 5 to 6 ridgeline and irregular wrinkles, and basal scar of peduncle. Texture is hard. Sarcocarp is 2 mm to 4 mm in thickness, 10 mm to 15 mm in diameter, pale yellow, coarse and hard. Kernel is narrow, ellipsoidal, about 10 mm in length, and 2 mm to 4 mm in diameter. Seed coat is yellowish brown and 2 cotyledons are white, overlapped and rolled.

Terminalia Fruit has a slight, characteristic odor and sour, pungent, and later sweet taste.

Identification Weigh 0.5 g of pulverized Terminalia Fruit, add 10 mL of water, shake and mix well, and filter. Add 1 to 2 drops of iron (III) chloride TS to the filtrate: a dark purple color develops

Purity (1) ***Foreign matter***—Terminalia Fruit contains less than 2.0 % of peduncle and other foreign matter.

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm

(ii) Dieldrin: Not more than 0.1 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm

(iv) Aldrin: Not more than 0.01 ppm

(v) Endrin: Not more than 0.01 ppm

(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 12.0 % (6 hours).

Ash Not more than 5.0 %.

Extract Content ***Dilute ethanol-soluble extract***—Not less than 35.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Thuja Seed

Thujae Semen

Thuja Seed is the seed of *Thuja orientalis* Linné (Cupressaceae), from which seed coat is removed.

Description Thuja Seed is the seed, long ovoid or long elliptical, 4 mm to 7 mm in length and 1.5 mm to

3 mm in diameter. External surface is yellowish white or pale yellowish brown, covered with membranous tegmen. Apex is slightly acute, with a small deep brown spot and base obtusely rounded. Texture is soft and oily.
Under a microscope, the transverse section reveals a tegmen on the outside consisting of a single row, cells are long rod-shaped, usually connected with hypodermal cells containing a brown pigment. The endosperm is close to polygonal or close to circular, cell cavities filled with relatively large aleurone grains and fatty oil drops, aleurone grains leaving a mesh pattern after dissolution. The cotyledon cells are rectangular, the cell cavities filled with relatively small aleurone grains and fatty oil drops. The cotyledon structure reveals 2 vascular bundles.
Thuja Seed has a characteristic odor and weak taste.

Purity (1) ***Foreign matter***—Thuja Seed contains not more than 1.0 % of the endocarp and other foreign matter.
(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(4) ***Sulfur dioxide***—Not more than 30 ppm.
(5) ***Mycotoxins***—Total aflatoxin (sum of aflatoxins B_1, B_2, G_1 and G_2): Not more than 15.0 ppb (aflatoxin B_1 is not more than 10.0 ppb).

Loss on Drying Not more than 7.0 %.

Ash Not more than 6.0 %.

Acid-insoluble Ash Not more than 1.0 %.

Extract Content ***Ether-soluble extract*—**Not less than 50.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Toad Venom

Bufonis Venenum

Toad Venom is the venomous secretion of *Bufo bufo gargarizans* Cantor or *Bufo melanostictus* Schneider (Bufonidae). When dried, Toad Venom contains not less than 5.8 % of bufosteroid.

Description Toad Venom is the collected secretion, flat circular masses or pieces, irregular in length and width. The external surface is maroon or red-brown. The masses are hard and difficult to cut. The cut surface is maroon, horny and slightly lustrous. The pieces are fragile and easily broken. The cut surface is red-brown and translucent.
Toad Venom has a fish-like odor and tastes sweet at first, later irritating with a lasting sensation of numbness. Pulverized Toad Venom stimulates the olfactory sense and causes sneezing.

Identification (1) Weigh 0.l g of pulverized Toad Venom, add 5 mL of chloroform, warm under a reflux condenser in a water-bath for l0 minutes, filter and perform the following tests using the filtrate as the test solution.
(i) Take 1 mL of the test solution and add carefully 1 mL of sulfuric acid to make two layers: a yellow color develops at the zone of contact, then changes to red after standing for l5 to 20 minutes and the chloroform layer acquires a pale red color.
(ii) Evaporate 1 mL of the test solution in a waterbath to dryness, dissolve the residue in 25 mL of methanol and determine the absorption spectrum of the solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits a maximum at about 300 nm.
(2) Warm 0.1 g of pulverized Toad Venom with 5 mL of a solution of tartaric acid (1 in 100) in a waterbath for 10 minutes and filter. To 1 mL of the filtrate, add carefully 1 mL of 4-dimethylaminobenzaldehyde TS, heat for l0 minutes in a waterbath and add l0 mL of water: a blue color develops.

Ash Not more than 5.0 %.

Acid-insoluble Ash Not more than 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 under a reflux condenser in a water-bath for 1 hour, cool and filter. Wash the residue with 30 mL of methanol, combine the washing and filtrate. To this solution, add methanol to make exactly l00 mL. Pipet 10.0 mL of this solution, add 5.0 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 of silica gel for 24 hours), about 20 mg of Cinobufagin RS (previously dried in a desiccator of silica gel for 24 hours), and about 20 mg of Redibufogenin RS (previously dried in a desiccator of 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 the test solution and use these solutions as the standard solution. Perform the test with 10 μL each of the test solution and the standard solutions as directed under the Liquid Chromatog-

raphy according to the following operating conditions. Determine the ratios, Q_{TB} and Q_{SB}, of the peak area of bufalin, for the test solution and the standard solution, respectively, Q_{TC} and Q_{SC}, of the peak area of cinobufagin, for the test solution and the standard solution, respectively and Q_{TR} and Q_{SR}, of the peak area of resibufogenin, for the test solution and the standard solution, respectively, to that of the internal standard in each solution and designate the total amount as an amount of bufosteroid.

$$\text{Amount (mg) of bufalin } (C_{24}H_{34}O_4) = \text{amount (mg) of Bufalin RS} \times \frac{Q_{TB}}{Q_{SB}}$$

$$\text{Amount (mg) of cinobufagin } (C_{26}H_{34}O_6) = \text{amount (mg) of Cinobufagin RS} \times \frac{Q_{TC}}{Q_{SC}}$$

$$\text{Amount (mg) of redibufogenin } (C_{24}H_{32}O_4) = \text{amount (mg) of Redibufogenin RS} \times \frac{Q_{TB}}{Q_{SB}}$$

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 30cm 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 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 under the above operating conditions, bufalin, cinobufagin. Use a column giving elution of resibufogenin and the internal standard in this order and clearly dividing each peak.

Containers and Storage ***Containers*—**Well-closed containers.

Tribulus Fruit

Tribulus Fruit

Tribulus Fruit is the ripe fruit of *Tribulus terrestris* Linné.

Description Tribulus Fruit is pentagonal star shaped fruit composed of five mericarps, 7 mm to 12 mm in diameter. Each mericarp is axe-shaped, 3 mm to 6 mm in length. The dorsal part is yellow-green, protrudent, with a longitudinal edge and several small spines. The spines are in symmetric pairs of longer and shorter spines. The two sides are coarse, reticulated, grayish white. The texture is hard. Under a microscope, a transverse section reveals several triangular mericarp connected by the pericarp. The pericarp is composed of a single-layered epidermis, the mesocarp is composed of parenchyma and a 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 and stone cell groups below. The seed coat consists of a single layer of cells in a tight arrangement. The parenchyma cells of the cotyledon contain oil droplets.
Tribulus Fruit is nearly odorless and tastes mild at first, followed by bitterness.

Identification Weigh 0.2 g of pulverized Tribulus Fruitm, add 3 mL acetic anhydride, warm on a water bath for 2 minutes and filter. Add carefully 1 mL of sulfuric acid to 1 mL of the filtrate: a red-brown color appears at the zone of contact, a blue-purple to green color at the upper layer.

Purity (1) ***Foreign matter***—(i) Fruit Stalk: Tribulus Fruit has Less than 4.0 % of fruit stalk.

(ii) Other foreign matter: The amount of foreign matter other than fruit stalk contained in Tribulus Fruit is not more than 1.0 %.

(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 7.0 %

Ash Not more than 13.0 %

Acid-insoluble Ash Not more than 2.5 %

Extract Content ***Water-soluble extract*** Not less than 12.0 %

Containers and Storage ***Containers*—**Well-closed

containers.

Trichosanthes Root

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 cm to 16 cm in length and 1.5 cm to 5.5 cm in diameter. The external surface is yellowish white or pale yellow-brown with longitudinal wrinkles and slightly concave, transversely long lenticels, some with remains of the maroon cortex. The texture is solid. The cut surface is white or pale yellow and largely powdery. Under magnifying glass, the transverse section reveals the wide medullary rays and yellowish brown spots or small holes formed by vessels.
Trichosanthes Root has a slight, characteristic odor and slightly bitter taste.

Identification Weigh 2 g of pulverized Trichosanthes Root, add 20 mL of diluted ethanol (1 in 2), sonicate for 30 minutes, 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. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 2 μL of the test solution and 1 μL of the standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of 1-butanol, water, ethanol and acetic acid (100) (8:3:2:2) to a distance of about 10 cm and air-dry the plate. Spray evenly Ninhydrin TS and heat at 105 °C: 1 spot 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: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): Not more than 0.2 ppm.
(vi) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 4.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Trichosanthes Seed

Trichosanthis Semen

Trichosanthes Seed is the ripe seed of *Trichosanthes kirilowii* Maximowicz or *Trichosanthes rosthornii* Harms (Cucurbitaceae).

Description (1) ***Trichosanthes kirlowii***—Trichosanthes Seed from *Trichosanthes kirilowii* is flat elliptical, about 12 mm to 15 mm in length, 6 mm to 10 mm in width and about 3.5 mm in thickness. External surface is pale brown to deep brown, flat, slippery, with dented scar along the margin of the seed. The upper side is relatively sharp, with hilum and the base is obtusely round or relatively narrow. The seed coat is tough and hard. The inner seed coat is membranous and grayish green. Two grayish cotyledons are largely oily.
Tricosanthes Seed has the characteristic odor and a slightly bitter taste.
(2) ***Trichosanthes rosthornii***—Trichosanthes Seed from *Trichosanthes rosthornii* is a relatively large and flat seed, 15 mm to 19 mm in length, 8 mm to 10 mm in width, and about 2.5 mm in thickness. External surface is deep brown, with distinctly dented scars surrounding the edge in a ring shape. This pattern is relatively wide. The upper side appears as if it has been cut.

Identification Weigh 0.1 g of powdered Trichosanthes Seed, add 2 mL of acetic anhydride, shake well and heat for 2 minutes in a water-bath and filter. Add 0.5 mL of sulfuric acid to the fitrate: a red brown to red color is produced at the zone of the two liquids.

Purity (1) ***Foreign matter***—Not more than 1.0 % of fragments of unriped seed coat.
(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(3) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(4) ***Sulfur dioxide***—Not more than 30 ppm.
(5) ***Mycotoxins***—Total aflatoxins (sum of aflatoxins B_1, B_2, G_1 and G_2): Not more than 15.0 ppb (aflatoxin B_1 is not more than 10.0 ppb).

Ash Not more than 4.0 %.

Extract Content ***Water-soluble extract*—**Not less than 6.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Valerian Root and Rhizome

Valerianae Radix et Rhizoma

Valerian Root is the root and rhizome of *Valerianae faurei* Briquet or other species of the same genus (Valerianaceae).

Description Valerian Root and Rhizome is the root and rhizome, usually the short rhizome with numerous fine, long roots. The rhizome is obovoid, 1 cm to 2 cm in length and 1 mm to 3 mm in diameter. The crown has buds and remains of stem and the flank of the rhizome is sometimes accompanied 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 cm to 15 cm in length and 1 mm to 3 mm in diameter. The external surface has fine, longitudinal wrinkles and is brittle. Under a magnifying glass, the transverse section reveals a thick, pale grayish brown cortex and a grayish brown stele.
Valerian Root has strong and characteristic odor and slightly bitter taste.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.

Ash Not more than 10.0 %.

Acid–insoluble Ash Not more than 5.0 %.

Essential Oil Content Not less than 0.3 mL (50.0 g, 1 mL of silicon resin).

Containers and Storage ***Containers*—**Tight containers.

Vitex Fruit

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 and 4 mm to 6 mm in diameter. External surface is grayish brown to blackish brown, covered with grayish white frost-like hairs, bearing 4 longitudinal shallow furrows. Apex is slightly concave, with grayish white persistent calyx and short peduncle at base. Calyx is 1/3 to 2/3 length of the fruit, with 5 crenatures, of which 2 is relatively deep, and densely pubescent. Texuture is light, rough and uneasily broken. Transverse section is showing 4 loculi, each with a white seed.
Vitex Fruit has a characteristic odor and the taste is weak and slightly pungent.

Identification Weigh 0.5 g of pulverized Vitex Fruit, add 10 mL of ethanol, shake well and filter. Add 0.1 g of magnesium and 0.3 mL of hydrochloric acid to 5 mL of the fitrate: a pale red to red-purple color appears.

Purity (1) ***Foreign matter***—(i) Fruit stalk and leaves: Less than 4.0 %.
(ii) Other foreign matter: Vitex Fruit contains other foreign matter less than 1.0 %.
(2) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(4) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 12.0 % (6 hours).

Ash Not more than 9.0 %

Acid-insoluble Ash Not more than 3.5 %.

Extract Content ***Dilute ethanol-soluble extract***—Not less than 8.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Xanthium Fruit

Xanthii Fructus

Xanthium Fruit is the ripe fruit of *Xanthium strumarium* Linné (Compositae).

Description Xanthium Fruit is the fusiform or ovoid fruit, 10 mm to 15 mm in length, 4 mm to 7 mm in diameter. External surface is yellowish brown to yellowish green with hooked spines throughout. The apex has two relatively thick spines, sperated or linked up and the base has a fruit stalk scar. Texture is hard and rigid. The center of a transverse section show a septum and two loculi, each having an achene. An achene is slightly fusiform, relatively even at the one side. The apex of an achene has protruding remains of style. The pericarp is thin, grayish black with longitudinal wrinkles and the seed coat is membranous, pale gray and oily with two cotyledons.
Xanthium Fruit has a characteristic odor and bitter taste.

Identification Weigh 0.5 g of pulverized Xanthium Fruit, macerate with 10 mL of water and filter. To the filtrate, add one droplet of iron (III) chloride TS: a grayish green color appears.

Purity (1) ***Heavy metals***—(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides***—(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endosulfan (sum of α,β-endosulfan and endosulfan sulfate): Not more than 0.2 ppm.
(vi) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide***—Not more than 30 ppm.

Loss on Drying Not more than 7.0 %

Ash Not more than 7.0 %

Acid-insoluble Ash Not more than 1.0 %

Extract Content ***Dilute ethanol-soluble extract*** Not less than 10.0 %

Containers and Storage ***Containers*—**Well-closed containers.

Yukmijihwangtang Extract Granules

Yukmijihwangtang Extract Granules contains no less than 1.4 mg of paenioflorin ($C_{23}H_{28}O_{11}$: 480.46) in Moutan Root Bark for a dose (a sachet)

Method of Preparation for a dose (one sachet)

Prepared Rehmannia Root	2.00 g
Moutan Root Bark, Poria, Cornus Fruit, Dioscorea Rhizome, Alisma Rhizome	1.00g

Pulverize the above herbal drugs to coarse powder, weigh each herbal drugs, put into the extractor, add eight to ten fold of water, extract for 2 to 3 hours at 80 ~ 100 °C and filter. Vacuum-concentrate the filtrate under 60 °C until it becomes 1.57 g to 2.34 g of Viscous extract or concentrate in a suitable method until it becomes 0.82 g to 1.22 g of Dry extract. Hyunggeyungyo Extract Granules is prepared as directed under Granules.

Indentification (1) ***Prepared Rehmannia Root***—Pulverize Yukmijihwangtang Extract Granules, weigh an amount, equivalent to 1 g of Prepared Rehmannia Root, add 100 mL of methanol, heat with a reflux condenser for 1 hour, cool and filter. Evaporate the filtrate to dryness in vacuum, add water to the residue and dissolve. Add 30 mL of ethyl acetate and extract in separatory funnel. Evaporate the layer of ethyl acetate to dryness, add 2 mL of ethanol and use this solution as the test solution. Separately, weigh accurately about 1 g of pulverized Prepared Rehmannia Root and use this solution, proceeding in the same manner as the test solution, as the standard solution. Perform the test with the test solution and the standard solution 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 with a fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of chloroform and methanol (20 : 1) to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with 2, 4-dinitrophenylhydrazine TS and examine under ultraviolet light (main wavelength: 254 nm): one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.
(2) ***Moutan Root Bark***—Pulverize Yukmijihwangtang Extract Granules, weigh an amount, equivalent to 1 g of pulverized Moutan Root Bark, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, heat with a reflux condenser 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 accurately about 1 g of pulverized Moutan Root Bark RMPM, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 ethyl formate, chloroform, toluene and formic acid (6 :

6 : 5 : 3) 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 °C for 10 minutes: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(3) ***Poria***—Pulverize Yukmijihwangtang Extract Granules, weigh an amount, equivalent to 1 g of Poria, add 10 mL of water, shake for 5 minutes, add 100 mL of methanol, extract with a reflux condenser 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 accurately about 1 g of pulverized Poria, add 100 mL of methanol, heat with a reflux condenser for 1 hour and filter. Vacuum-concentrate the filtrate until the filtrate becomes 10 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution 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 with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of hexane and acetone (7 : 3) 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 °C for 10 minutes. Examine under ultraviolet light (main wavelength: 254 nm): one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(4) ***Cornus Fruit***—Pulverize Yukmijihwangtang Extract Granules, weigh an amount, equivalent to 1 g of Cornus Fruit, add 100 mL of methanol, shake thoroughly and filter. Evaporate the filtrate to concentrate until the filtrate becomes about 2 mL and use this solution as the test solution. Separately, weigh accurately about 1 g of pulverized Cornus Fruit RMPM, 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 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 dichloromethane, methanol and water (60 : 35 : 15) 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 °C for 10 minutes: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(5) ***Dioscorea Rhizome***—Pulverize Yukmijihwangtang Extract Granules, weigh an amount, equivalent to 1 g of Dioscorea Rhizome, add 50 mL of ethanol and 5 mL of acetic acid, heat with a reflux condenser for 1 hour, cool and filter. Evaporate the filtrate to dryness in vacuum, add 2 mL of ethanol, dissolve and use this solution as the test solution. Separately, weigh accurately about 1 g of pulverized Dioscorea Rhizome RMPM, 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 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 cyclohexane and ethyl acetate (3 : 1) to a distance of about 10 cm and air-dry the plate. Spray evenly the plate with sulfuric acid TS for spray and heat at 105 °C for 10 minutes: one spot among the spots from the test solution and a spot from the standard solution show the same color and the same R_f value.

(6) ***Alisma Rhizome***—Pulverize Yukmijihwangtang Extract Granuels, weigh an amount, equivalent to 1 g of Alisma Rhizome, add 100 mL of ether, heat with a reflux condenser for 1 hour, cool and filter. Evaporate the filtrate to concentrate until the filtrate becomes about 2 mL and use this solution as the test solution. Separately, weigh accurately about 1 g of pulverized Alisma Rhizome, 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 Thin-layer Chromatography. Spot 20 μL each of the test solution and the standard solution on a plate of silica gel with a fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of chloroform and acetone (1 : 1) 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 °C for 10 minutes: one spot among the 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) Heavy metals: Not more than 30 ppm.

(ii) Lead: Not more than 5 ppm.

(iii) Arsenic: Not more than 3 ppm.

Disintegration Test It meets the requirement.

Particle Size Distribution Test for Preparations It meets the requirement.

Uniformity of Dosage Units (divided) It meets the requirement.

Microbial Limit It meets the requirement.

Assay (1) ***Paenioflorin of Moutan Root Bark***—Take not less than about 20 sachets of Yukmijihwangtang Extract Granules, weigh and pulverize. Weigh accurately equivalent to about 5 mg of paenioflorin, add 40 mL of water, extract with a sonicator for 30 minutes and filter. Extract the residue with chloroform, remove the layer of chloroform, add water in the layer of water to make exactly 50 mL and use this solution as the test solution Separately, weigh accurately about 5 mg of Paenioflorin RS (previously dried in a silica gel desiccator for 24 hours), add water to make exactly 50 mL and use the solution as the standard solution. Pipet 10 μL each 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_T and A_S, of the test solution and the standard solution, respectively

$$\text{Amount (mg) of paenioflorin } (C_{42}H_{62}O_{16}) = \text{amount (mg) of Paenoiflorin RS, calculated on the anhydrous basis} \times \frac{A_T}{A_S}$$

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

Containers and Storage ***Containers***—Tight containers.

Zanthoxylum Peel

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) ***Pericarp of Zanthoxylum piperitum***—Zanthoxylum Peel from *Zanthoxylum piperitum* is pericarp, consisting of 2 to 3 follicles. Each mericarp is flattened spheroidal, dehiscent in 2 pieces and about 5 mm in diameter. The outer surface of pericarp is dark yellow-red to dark red-brown, with numerous dented spots originated from oil sacs: the inner surface is pale yellowish white. Under a microscope, transverse section reveals the external epidermis and the adjoined unicellular layer containing red-brown tannin: the pericarp holds oil sacs being up to approximately 500 μm in diameter and sporadically vascular bundles consisting mainly of spiral vessels: the endocarp consists of stone cell layers: inner epidermal cells very small.
Zanthoxylum Peel from *Zanthoxylum piperitum* has a characteristic odor and purgent taste with a sensation of numbness on the tongue.

(2) ***Pericarp of Zanthoxylum schinifolium***—Zantho-xylum Peel from *Zanthoxylum schinifolium* is pericarp, consisting of 2 to 3 small follicles which is apocarpous at the upper part and grouped on a fruit stalk. Follicles are spherical, splitting along the ventral suture and 3 mm to 4 mm in diameter. External surface is grayish green to dark green,, scattered with numerous oil dots and fine reticulated and raised wrinkles. Inner surface is almost white and smooth. Endocarp is commonly separated from pericarp at the base. Remains of seed are ovoid, 3 mm to 4 mm in length, 2 cm to 3 cm in diameter, with black and lustrous surface.
Zanthoxylum Peel from *Zanthoxylum schinifolium* has a characteristic odor and slight sweet and pungent taste.

(3) ***Pericarp of Zanthoxylum bungeanum***—Zantho-xylum Peel from *Zanthoxylum bungeanum* is pericarp, consisting of follicles, mostly singly and 4 mm to 5 mm in diameter. Exernal surface is red-purple to red-brown, scattered with numerous warty oil dots, 0.5 mm to 1 mm in diameter, translucent when observed against light. Inner surface is pale yellow.
Zanthoxylum Peel from *Zanthoxylum bungeanum* has strong and characteristic odor and taste lastingly pungent and numb.

Identification Weigh 0.5 g of pulverized Zanthoxylum Peel, add 10 mL of diluted ethanol (7 in l0), stopper the vessel tightly, shake for 30 minutes, filter and use this filtrate as the test solution. Perform the test with the test solution as directed under the Thin-layer Chromatography. Spot 10 μL of the test solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate, ethanol and water (8 : 2 : 1) 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 at the R_f value of about 0.7 appears.

Purity (1) ***Foreign matter—***(i) Seed: Less than 20.0 %.

(ii) Fruit stalk and twig: Less than 5.0 %.

(iii) Other foreign matter: The amount of foreign matter other than fruit stalk and twigs contained in Zanthoxylum Peel is not more than 1.0 %.

(2) ***Heavy metals—***(i) Lead: Not more than 5 ppm.

(ii) Arsenic: Not more than 3 ppm.

(iii) Mercury: Not more than 0.2 ppm.

(iv) Cadmium: Not more than 0.3 ppm.

(3) ***Residual pesticides—***(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Methoxychlor: Not more than 1 ppm.

(iv) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(v) Aldrin: Not more than 0.01 ppm.

(vi) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

Ash Not more than 6.0 %.

Acid-insoluble Ash Not more than 1.5 %.

Essential Oil Content Not less than 1.0 mL (30.0 g).

Containers and Storage ***Containers*—**Well-closed containers.

Zedoary

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), usually dried after steamed.

Description (1) ***Rhizome of Curcuma phaeocaulis***—Zedoary from *Curcuma phaeocaulis* is the rhizome, ovoid, elongated ovoid, conical or elongated fusiform, 2 cm to 8 cm in length, 15 mm to 40 mm in diameter. The external surface is grayish yellow to grayish brown, the upper part is frequently acute and obtuse and the base, obtuse and round. The upper part is conspicuously raised-annulated, circular, with slightly dented rootlet scars or rootlets remaining. Some have 1 row of dented sprout scars on both sides and nearly circular outer rhizome scars, sometimes with knife marks remaining. The body is heavy and the texture is solid. The transverse section is waxy, grayish brown to bluish brown, usually with grayish brown powder. The cortex and stele are easily detachable and the endodermal ring is deep brown.
Zedoary from *Curcuma phaeocaulis* has a slight, characteristic odor and slightly bitter and pungent taste.

(2) ***Rhizome of Curcuma kwangsiensis***—Zedoary from *Curcuma kwangsiensis* is the rhizome, slightly raised-annulated. Fractured surface is yellowish brown to brown, with commonly pale yellow powder. Endodermal ring is yellowish white.

(3) ***Rhizome of Curcuma wenyujin***—Zedoary from *Curcuma wenyujin* is the rhizome and its fractured surface is commonly yellowish brown to deep brown, with commonly pale yellow to yellowish brown powder. Zedoary from *Curcuma wenyujin* has a slight, characteristic odor.

Identification Weigh 1 g of pulverized Zedoary, add 50 mL of petroleum ether, sonicate for 1 hour, filter, concentrate the filtrate to make 2 mL and use as the test solution. Separately, dissolve 0.4 mg of Germacron RS in 1 mL of methanol and use this solution as the standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 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 petroleum ether and ethyl acetate (9:1) 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 spots obtained from the test solution shows the same color and Rf value as the spot from the standard solution.

Purity (1) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(2) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.
(ii) Dieldrin: Not more than 0.01 ppm.
(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.
(iv) Aldrin: Not more than 0.01 ppm.
(v) Endrin: Not more than 0.01 ppm.
(3) ***Sulfur dioxide*—**Not more than 30 ppm.

Ash Not more than 7.0 %.

Essential Oil Content Not less than 0.5 mL (50.0 g, 1 mL of silicon resin

Containers and Storage ***Containers*—**Well-closed containers.

Zizyphus Seed

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 circular or flattened elliptic, 5 mm to 9 mm in length, 5 mm to 7 mm in width and 3 mm in thickness. The external surface is redd-purple or purple-brown, smooth, lustrous, sometimes with open patterns. One side is relatively even and the other side is slightly bumpy with one raised longitudinal line in the middle. One end shows a concave, linear hilum and the other end shows a small, protruding chalaza. The testa is relatively flexible and covers grayish endosperm and pale yellow cotyledons.
Zizyphus Seed has a slight oily odor and the taste is weak.

Identification Weigh 0.5 g of Zizyphus Seed, add 5 mL of ether, shake for 2 minutes and filter. The filtrate is removed, 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 it slowly change to red-purple.

Purity (1) ***Foreign matter*—**Zizyphus Seed contains not more than 3 % of endocarp and other foreign matter.
(2) ***Heavy metals*—**(i) Lead: Not more than 5 ppm.
(ii) Arsenic: Not more than 3 ppm.
(iii) Mercury: Not more than 0.2 ppm.
(iv) Cadmium: Not more than 0.3 ppm.
(3) ***Residual pesticides*—**(i) Total DDT (sum of p,p'-DDD, p,p'-DDE, o,p'-DDT and p,p'-DDT): Not more than 0.1 ppm.

(ii) Dieldrin: Not more than 0.01 ppm.

(iii) Total BHC (sum of α, β, γ and δ-BHC): Not more than 0.2 ppm.

(iv) Aldrin: Not more than 0.01 ppm.

(v) Endrin: Not more than 0.01 ppm.

(4) ***Sulfur dioxide***—Not more than 30 ppm.

(5) ***Mycotoxins***—Total aflatoxin (sum of aflatoxins B_1, B_2, G_1 and G_2): Not more than 15.0 ppb (aflatoxin B_1 is not more than 10.0 ppb).

Ash Not more than 7.0 %.

Containers and Storage ***Containers—***Well-closed containers.

2) Biological Preparations, etc.

Adsorbed Diphtheria-Tetanus Combined Vaccine for Adult

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 conforms to the requirements of Adsorbed Diphtheria-Tetanus Combined Vaccine for Adult in the Specifications and Test Methods for Biological Products of Korea.

Adsorbed Diphtheria-Tetanus-Acellular Pertussis Combined Vaccine

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 Biological Products of Korea.

Adsorbed Diphtheria-Tetanus-Acellular Pertussis-Enhanced Inactivated Poliomyelitis Combined Vaccine

Adsorbed Diphtheria-Tetanus-Acellular Pertussis-Enhanced Inactivated Poliomyelitis Combined Vaccine is a liquid preparation obtained by adding aluminum salt to adsorb and mix detoxified toxoid solutions of diphtheria toxin and tetanus toxin, purified pertussis antigens from which pertussis defense antigens, etc. have been separated, purified and detoxified, and a solution containing poliovirus of type I, II and III inactivated in a suitable manner.
Adsorbed Diphtheria-Tetanus-Acellular Pertussis-Enhanced Inactivated Poliomyelitis Combined Vaccine conforms to the requirements of Adsorbed Diphtheria-Tetanus-Acellular Pertussis-Enhanced Inactivated Poliomyelitis Combined Vaccine in the Specifications and Test Methods for Biological Products of Korea.

Clostridium botulinum Toxin Type A

Clostridium botulinum Toxin Type A is a freeze-dried preparation containing *Clostridium botulinum* toxin type A. It becomes a liquid preparation on addition of solvent.
Clostridium botulinum Toxin Type A conforms to the requirements of *Clostridium botulinum* Toxin Type A in the Specifications and Test Methods for Biological Products of Korea.

Enhanced Inactivated Poliomyelitis Vaccine

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 conforms to the requirements of Enhanced Inactivated Poliomyelitis Vaccine in the Specifications and Test Methods for Biological Products of Korea.

Erythropoietin Concentrated Solution (rDNA)

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 effect of increasing a number of reticulocytes.
Erythropoietin Concentrated Solution (rDNA) has a potency of not less than 100000 IU per mg of protein determined using the conditions described under Assay and in the test for protein.

Description Erythropoietin Concentrated Solution (rDNA) appears as clear or slightly turbid, colorless solution.

Identification (1) ***Biological identification***—When examined using the conditions described under Assay, increase in red blood cell count is observed.

(2) ***Capillary electrophoresis***

Test solution: Dilute Erythropoietin Concentrated Solution (rDNA) with water to a protein concentration of 1 mg/mL. Desalt 0.25 mL of this solution by passage through a centrifuge cartridge with a molecular mass cut-off of not more than 10,000. Add 0.2 mL of water to sample and desalt again. Repeat this process once more. Determine the protein content as directed in the Assay, dilute with water to a concentration of 1 mg/mL and use this solution as the test solution.

Standard solution: Dilute Erythropoietin RS with water to a protein concentration of 1.0 mg/mL. Desalt this solution as described for the test solution.Examine with the test standard solution and compare the electropherogram obtained with the test solution with that with the standard solution: 8 isoform peaks are observed in the test solution. The percentage 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.

Capillary temperature: A constant temperature of about 35 °C

Store the test solution and the standard solution at 4 °C during the analysis.

Concentrated capillary electrophoresis buffer: 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: Dissolve 21.0 g of urea in 25 mL of water at 30 °C. Add 5 mL of the concentrated capillary electrophoresis buffer and 125 μL of 1 mol/L putrescine solution, and add water to make 50 mL. Adjust to pH 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	60	Pressure
	Capillary electrophoresis buffer	60	Pressure
	Capillary electrophoresis buffer	720	20 kV
Between-run washing	Water	10	Pressure
	0.1 mol/L sodium hydroxide	5	Pressure
	Capillary electrophoresis buffer	10	Pressure
Injection	Injection of test solution and standard solution	-	Pressure or vacuum
Separation	Capillary electrophoresis buffer	80	143 V/cm (15.4 kV)

System suitability

System performance: The peaks of the standard solution are well separated and the height of the largest peak is at least 50 times greater than the baseline noise. If necessary, adjust the sample loading to give peaks of sufficient height. In the eletropherogram of standrad solution, peaks corresponding to isoforms 1 to 8 are observed. Isoform 1 may not be visible and isoform 8 is detected. The height of the peak of isoform 6 is the greatest and the resolution between the peaks of isoform 5 and 6 is not less than 1.

System repeatability: Repeat the test at least3 times with the standard solution. The baseline is stable, showing almost no drift, 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: 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 water to make 100 mL. Adjust to pH 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) in water to a protein concentration of 1.0 mg/mL. Mix equal volumes of this solution and the dilution buffer.

Test solution (b): Dilute Erythropoietin Concentrated Solution (rDNA) in water to a protein concentration of 0.1 mg/mL. Mix equal volumes of this solution and the dilution buffer.

Standard solution (a): Dissolve Erythropoietin RS in water to a protein concentration of 1.0 mg/mL. Mix equal volumes of this solution and the dilution buffer.

Standard solution (b): Dissolve Erythropoietin RS in water to a protein concentration of 0.1 mg/mL. Mix equal volumes of this solution and the dilution buffer.

Standard solution (c): Use a solution of molecular mass markers suitable for calibrating sodium dodecyl sulfate-polyacrylamide gel electrophoresis in the range of molecular mass 10000 to 70000 Dalton.

Standard solution (d): Use a solution of molecular mass markers suitable for calibrating sodium dodecyl sulfate-polyacrylamide gel electrophoresis in the range of molecular mass 10000 to 70000 Dalton and suitable for membrane blotting.

Boil the test solutions and the standard solutions in a water bath for 2 minutes. Load 20 μL of each samples in the following order: standard solution (c), standard solution (a), test solution (a), empty well, standard solution (b), test solution (b) and standard solution (d) to the wells of the gel, 0.75 mm in thickness, about 16 cm^2 in area, composed of 12 % acrylamide. After electrophoresis, cut the empty well between test solution (a) and standard solution (b) into two parts. Stain the gel containing standard solution (c), standard solution (a) and test solution (a) with the Coomassie Brilliant Blue staining solution.

Test solution (a) shows a single diffuse band corresponding in position and intensity to the band of standard solution (a).

(b) ***Immunoblotting***

After polyacrylamide gel electrophoresis, transfer the gel containing the standard solution (b), test solution (b) and standard solution (d) onto a membrane suitable for protein immobilization and start the electrotransfer. After the electrotransfer, incubate the membrane in a blocking solution containing 50 g/L of skim dry milk or 10 vol % bovine serum albumin for 1 to 2 hours shaking. Incubate the membrane in a solution of anti-erythropoietin antibody diluted with the same blocking solution for 1 to 14 hours. Detect the erythropoietin-bound antibody using asecondary antibody labeled with an enzyme such as alkaline phosphatase or with a radioactive material. Test solution (b) shows a single broad band corresponding in position and intensity to the band of standard solution (b).

System suitability

System performance: The molecular mass markers of standard solution (d) are resolved into discrete bands with a linear relationship between the distance migrated and logarithmic value of the molecular mass.

(4) ***Peptide map***

Test solution: Dilute Erythropoietin Concentrated Solution (rDNA) with tris-acetate buffer (pH 8.5) water to a protein concentration of 1.0 mg/mL. Equilibrate this solution with tris-acetate buffer (pH 8.5) using a suitable procedure such as dialysis or membrane filtration. Transfer 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, then stop the reaction immediately by freezing.

Standard solution: Dissolve Erythropoietin RS in water to a protein concentration of 1.0 mg/mL. Prepare as for the test solution simultaneously.

Examine with 50 μL each of the test solution and the standard solution as directed under Liquid Chromatography according to the following operating conditions: the profile of the chromatogram obtained with the test solution corresponds to that of the chromatogram with 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 about 25 cm in length, packed with butylsilyl silica gel for liquid chromatography (5 μm to 10 μm in particle diameter).

Mobile phase: Control the step or concentration gradient by mixing mobile phases A and B as directed in the following table.

Mobile phase A: A mixture of water and trifluoroacetic acid (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 %)	Elution condition
0-10	0.75	100	0	Isocratic
10-125	0.75	100→39	0→61	Linear gradient
125-135	1.25	39→17	61→83	Linear gradient
135-145	1.25	17→0	83→100	Linear gradient
145-150	1.25	100	0	Isocratic

Column equilibration: Equilibrate by running mobile phase A for at least 15 minutes.

System suitability

System performance: The chromatograms obtained with the test solution and the standard solution are qualitatively similar to the standard chromatogram of Erythropoietin RS.

(5) ***N-terminal sequence analysis***

Dilute a volume of Erythropoietin Concentrated Solution (rDNA), equivalent to 50 μg of erythropoietin, in 1 mL of diluted trifluoroacetic acid (1 in 1000). Desalt this solution by applying to 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 discard the washings. Elute the cartridge with a mixture of water and acetonitrile (50 : 50), and lyophilize the collect.

Redissolve the lyophilised sample in 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 Reverse-phase Liquid Chromatography: Ala-Pro-Pro-Arg-Leu-Ile-(unsequenced amino acid)-Asp-Ser-Arg-Val-Leu-Glu-Arg-Tyr. Set the analyzing conditions of reverse-phase liquid chromatography to separate all amino acids using amino acids reference standards.

Purity (1) ***Dimers and related substances of higher molecular mass***

Test solution: Dilute Erythropoietin Concentrated Solution (rDNA) with the mobile phase water to a protein concentration of 0.2 mg/mL.

Standard solution: To 0.02 mL of the test solution, add the mobile phase to make 1 mL.

Examine with 100 μL each of the test solution and the standard solution as directed under Liquid Chromatography according to the following operating conditions: the total area of all peaks eluted before the principal peak from the test solution is not more than the area of the principal peak obtained with the standard solution (not more than 2 %).

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 suitable for fractionation of globular protein in the molecular mass 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/minute

Run time : 60 minutes after loading of the test solution

System suitability

System performance: The area of the principal peak obtained with standard solution is 1.5 % to 2.5 % of the area of the principal peak obtained with the test solution.

(2) ***Sialic acids***

Test solution (a): Dilute Erythropoietin Concentrated Solution (rDNA) with the mobile phase used in "Dimers and related substances of higher molecular mass" to a protein concentration of 0.3 mg/mL.

Test solution (b): To 0.5 mL of test solution (a), add 0.5 mL of the mobile phase from "Dimers and related substances of higher molecular mass."

Standard solution (a): Dissolve a suitable amount of *N*-acetylneuraminic acid in water to obtain a concentration of 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 each standard solutions and test solution in triplicate. Transfer 100 μL each of the test solutions and standard solutions to 10 mL glass-stoppered test tubes. To each tube add 1.0 mL of resorcinol. Stopper the tubes and heat at 100 °C for 30 minutes. Cool on ice and to each tube add 2.0 mL of a mixture of butyl acetate and butanol (48 : 12). Mix vigorously and allow to separate into two layers. Ensuring that the supernatant liquid is completely clear and carefully take the supernatant liquid, making sure to exclude any of the lower liquid. Measure the absorbances at 580 nm of the test solutions and the standard solutions as directed under Ultraviolet-visible Spectrophotometry. Using the calibration curve obtained with the absorbances of the standard solutions. Determine the content of sialic acids of test solutions (a) and (b) and calculate the mean. Calculate the number of moles of silaic acids per mole of erythropoietin assuming that the molecular mass of erythryopoetin is 30600 and that the molecular mass of *N*-acetylneuraminic acid is 309: not less than 10 mole of sialic acids per mole of erythropoietin.

System suitability

System performance: The resulting value of standard solution (a) is 1.5 to 3.3 times the resulting value of test solution (a).

System repeatability: Individual 3 replicates of the standard solutions and the test solutions agree to within 10 %.

Bacterial Endotoxins Less than 20 EU per volume equivalent to 10000 IU 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.

Record the absorbance at 250 nm to 400 nm as directed under Ultraviolet-visible Spectrophotometry. After correction for light scattering up to 400 nm, it exhibits a maximum absorbance between 276 nm and 280 nm. Calculate the concentration of erythropoietin using the specific absorbance: 80 % to 120 % of the labeled concentration.

(2) ***Potency—***The activity of Erythropoeitin Concentrated Solution (rDNA) is compared with that of Eryth-ropoetin RS and expressed in International Units (IU).

Concentrated staining solution: Use a solution of thiazole orange suitable for the determination of reticu-

locytes. Prepared at a concentration twice that necessary for the analysis.

Test solution (a): Dilute Erythropoietin Concentrated Solution (rDNA) with bovine serum albumin in saline solution to a concentration of 80 IU/mL.

Test solution (b): Mix equal volumes of test solution (a) and bovine serum albuminin saline solution.

Test solution (c): Mix equal volumes of test solution (b) and bovine serum albuminin saline solution.

Standard solution (a): Dilute Erythropoietin RS with bovine serum albuminin saline solution to a concentration of 80 IU/mL.

Standard solution (b): Mix equal volumes of standard solution (a) and bovine serum albumin in saline solution.

Standard solution (c): Mix equal volumes of standard solution (b) and bovine serum albumin in saline solution.

The exact concentrations of the test solutions and the standard solutions may be modified according to the response range of the animals used.

At the beginning of the assay, randomly distribute mice of a suitable age and strain (for example, 8-weekold B6D2F1 mice) with 6 mice in each group. Inject each animal subcutaneously with 0.5 mL each of the test and the standard solutions (one solution per group). Collect blood samples from the animals 4 days after the injections.

Dilute the whole blood 500-fold with the buffer used to prepare the thiazole orange staining solution. Mix equal volumes of this solution and the concentrated staining solution. Stain for 3 to 10 minutes then determine the reticulocyte count with a flow cytometer. Determine the percentage of reticulocytes using a biparametric histogram: number of cells and red fluorescence (620 nm). Calculate the potency by the parallel line assay method: between 80 % and 125 % of the labeled potency. The confidence interval (P = 0.95) of the calculated potency is 64 % to 156 % of the labeled potency.

Containers and Storage ***Containers***—Hermetic containers.

Storage—At a temperature below -20 °C, and avoid repeated freezing and thawing.

Freeze-Dried Agkistrodon (Salmusa) Antivenom (Equine)

Freeze-Dried Agkistrodon (Salmusa) Antivenum (Equine) is a freeze-dried preparation that contains Agkistrodon (Salmusa) Antivenum in immunoglobulin of horse origin. It becomes a liquid preparation on addition of solvent.

Freeze-Dried Agkistrodon (Salmusa) Antivenum (Equine) conforms to the requirements of Freeze-Dried Agkistrodon (Salmusa) Antivenum (Equine) in the Specifications and Test Methods for Biological Products of Korea.

Freeze-dried BCG Vaccine for Intradermal Use

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 addition of solvent.

Freeze-dried BCG Vaccine for Intradermal Use conforms to the requirements of Freeze-dried BCG Vaccine for Intradermal Use in the Specifications and Test Methods for Biological Products of Korea.

Freeze-dried BCG Vaccine for Percutaneous Use

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 addition of solvent.

Freeze-dried BCG Vaccine for Percutaneous Use conforms to the requirements of Freeze-dried BCG Vaccine for Percutaneous Use in the Specifications and Test Methods for Biological Products of Korea.

Freeze-dried Concentrated Human Antithrombin 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 conforms to the requirements of Freeze-dried Concentrated Human Antithrombin III in the Specifications and Test Methods for Biological Products of Korea.

Freeze-dried Concentrated Human Blood Coagulation Factor VIII

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 addition of solvent.

Freeze-dried Concentrated Human Blood Coagulation Factor VIII conforms to the requirements of Freeze-dried Concentrated Human Blood Coagulation Factor

VIII in the Specifications and Test Methods for Biological Products of Korea.

Freeze-dried Human Blood Coagulation Factor IX Complex

Freeze-dried Human Blood Coagulation Factor IX Complex is a freeze-dried preparation that contains blood coagulation factor IX complex of human plasma. It becomes a liquid preparation on addition of solvent.
Freeze-dried Human Blood Coagulation Factor IX Complex conforms to the requirements of Freeze-dried Human Blood Coagulation Factor IX Complex in the Specifications and Test Methods for Biological Products of Korea.

Freeze-dried Human Fibrinogen

Freeze-dried Human Fibrinogen is a freeze-dried preparation that contains fibrinogen of human plasma. It becomes a liquid preparation on addition of solvent.
Freeze-dried Human Fibrinogen conforms to the requirements of Freeze-dried Human Fibrinogen in the Specifications and Test Methods for Biological Products of Korea.

Freeze-Dried Live Attenuated Measles-Mumps-Rubella Combined Vaccine

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 addition of solvent.
Freeze-Dried Live Attenuated Measles-Mumps-Rubella Combined Vaccine conforms to the requirements of Freeze-Dried Live Attenuated Measles-Mumps-Rubella Combined Vaccine in the Specifications and Test Methods for Biological Products of Korea.

Freeze-dried Smallpox Vaccine

Freeze-dried Smallpox Vaccine is a freeze-dried preparation containing live vaccinia virus. It becomes a liquid preparation on addition of solvent.
Freeze-dried Smallpox Vaccine conforms to the requirements of Freeze-dried Smallpox Vaccine in the Specifications and Test Methods for Biological Products of Korea.

Haemophilus influenzae type b Conjugated to Diphtheria CRM197 Vaccine (Aluminum Adjuvanted)

Hemophilus influenzae type b Conjucated to Diphtheria CRM197 Vaccine (Aluminum Adjuvanted) is a liquid preparation containing *Haemophilus influenzae* type b oligosaccharides conjugated to a non-toxic mutant of diphtheria toxin (CRM197).
Hemophilus influenzae type b Conjucated to Diphtheria CRM197 Vaccine (Aluminum Adjuvanted) conforms to the requirements of *Hemophilus influenzae* type b Conjucated to Diphtheria CRM197 Vaccine (Aluminum Adjuvanted) in the Specifications and Test Methods for Biological Products of Korea.

Haemophilus influenzae type b Conjugated to Meningococcal Protein Vaccine

Haemophilus influenzae type b Conjugated to Meningococcal Protein Vaccine is a liquid preparation containing *Haemophilus influenzae* type b polysaccharides conjugated to a meningococcal protein.
Haemophilus influenzae type b Conjugated to Meningococcal Protein Vaccine conforms to the requirements of *Haemophilus influenzae* type b Conjugated to Meningococcal Protein Vaccine in the Specifications and Test Methods for Biological Products of Korea.

Haemophilus influenzae type b Conjugated to Tetanus Toxoid Vaccine

Haemophilus influenzae type b Conjugated to Tetanus Toxoid Vaccine is a dried preparation containing *Haemophilus influenzae* type b polysaccharides conjugated to tetanus toxoids. It becomes a liquid preparation on addition of solvent.
Haemophilus influenzae type b Conjugated to Tetanus Toxoid Vaccine conforms to the requirements of *Haemophilus influenzae* type b Conjugated to Tetanus Toxoid Vaccine in the Specifications and Test Methods for Biological Products of Korea.

Hepatitis A Vaccine (Adsorbed, Inactivated)

Hepatitis A Vaccine (Adsorbed, Inactivated) is a liquid preparation containing inactivated hepatitis A virus antigens.
Hepatitis A Vaccine (Adsorbed, Inactivated) conforms to the requirements of Hepatitis A Vaccine (Adsorbed, Inactivated) in the Specifications and Test Methods for Biological Products of Korea.

Hepatitis A Vaccine (Virosome, Inactivated)

Hepatitis A Vaccine (Virosome, Inactivated) is a liquid preparation obtained by adsorbing inactivated hepatitis A virus antigens to virosomes composed of influenza hemagglutinin and phospholipids.
Hepatitis A Vaccine (Virosome, Inactivated) conforms to the requirements of Hepatitis A Vaccine (Virosome, Inactivated) in the Specifications and Test Methods for Biological Products of Korea.

Hepatitis B Vaccine (rDNA)

Hepatitis B Vaccine (rDNA) is a liquid preparation containing surface antigens of recombinant hepatitis B virus.
Hepatitis B Vaccine (rDNA) conforms to the requirements of Hepatitis B Vaccine (rDNA) in the Specifications and Test Methods for Biological Products of Korea.

Human Hepatitis B Immunoglobulin

Human Hepatitis B Immunoglobulin is a liquid preparation containing hepatitis B immunoglobulin which is a kind of human serum immunoglobulin G.
Human Hepatitis B Immunoglobulin conforms to the requirements of Human Hepatitis B Immunoglobulin in the Specifications and Test Methods for Biological Products of Korea.

Human Hepatitis B Immunoglobulin for Intravenous Administration

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 conforms to the requirements of Human Hepatitis B Immunoglobulin for Intravenous Administration in the Specifications and Test Methods for Biological Products of Korea.

Human Normal Immunoglobulin

Human Normal Immunoglobulin is a liquid preparation containing immunoglobulin G in human serum globulin.
Human Normal Immunoglobulin conforms to the requirements of Human Normal Immunoglobulin in the Specifications and Test Methods for Biological Products of Korea.

Human Normal Immunoglobulin in Maltose (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) conforms to the requirements of Human Normal Immunoglobulin in Maltose (pH 4.25) in the Specifications and Test Methods for Biological Products of Korea.

Human Papillomavirus Vaccine (rDNA)

Human Paillomavirus Vaccine (rDNA) is a liquid preparation containing recombinant capsid (L1) of papillomavirus.
Human Paillomavirus Vaccine (rDNA) conforms to the requirements of Human Paillomavirus Vaccine (rDNA) in the Specifications and Test Methods for Biological Products of Korea.

Human Serum Albumin

Human Serum Albumin is a liquid preparation containing albumin of human serum.
Human Serum Albumin conforms to the requirements of Human Serum Albumin in the Specifications and Test Methods for Biological Products of Korea.

Human Tetanus Immunoglobulin

Human Tetanus Immunoglobulin is a liquid preparation containing anti-tetanus human immunoglobulin G among human serum globulin.
Human Tetanus Immunoglobulin conforms to the requirements of Human Tetanus Immunoglobulin in the Specifications and Test Methods for Biological Products of Korea.

Human Varicella Immunoglobulin

Human Varicella Immunoglobulin is a liquid preparation containing human varicella antibody among human serum immunoglobulin G.
Human Varicella Immunoglobulin conforms to the requirements of Human Varicella Immunoglobulin in the Specifications and Test Methods for Biological Products of Korea.

Inactivated Oral Cholera Vaccine

Inactivated Oral Cholera Vaccine is a preparation containing inactivated *Vibrio cholerae* and recombinant cholera toxin B subunit (rCTB-213).
Inactivated Oral Cholera Vaccine conforms to the requirements of Inactivated Oral Cholera Vaccine in the Specifications and Test Methods for Biological Products of Korea.

Influenza HA Vaccine

Influenza HA Vaccine is a liquid preparation containing hemagglutinin of inactivated influenza virus.
Influenza HA Vaccine conforms to the requirements of Influenza HA Vaccine in the Specifications and Test Methods for Biological Products of Korea.

Influenza Vaccine (Split Virion, Inactivated)

Influenza Vaccine (Split Virion, Inactivated) is a liquid preparation containing influenza virions, split and inactivated to maintain antigenicity.
Influenza Vaccine (Split Virion, Inactivated) conforms to the requirements of Influenza Vaccine (Split Virion, Inactivated) in the Specifications and Test Methods for Biological Products of Korea.

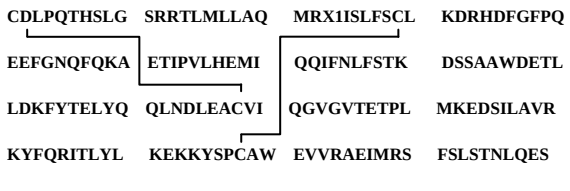
Influenza Vaccine (Surface Antigen, Inactivated)

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) conforms to the requirements of Influenza Vaccine (Surface Antigen, Inactivated) in the Specifications and Test Methods for Biological Products of Korea.

Influenza Vaccine (Surface Antigen-Virosome, Inactivated)

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) conforms to the requirements of Influenza Vaccine (Surface Antigen-Virosome, Inactivated) in the Specifications and Test Methods for Biological Products of Korea.

Interferon alpha-2 Concentrated Solution (rDNA)



Interferon alpha-2 Concentrated Solution (rDNA) is the concentrated solution of a recombinant protein that is produced according to the code information by the interferon alpha-2 gene and that has non-specific antiviral activity and antiproliferative activity. Two types of interferon alpha-2 exist depending on the amino acid residue at position 23.

Designation	Amino acid residue at position 23 (X_1)
alpha-2a	Lys
alpha-2b	Arg

Interferon alpha-2 Concentrated Solution (rDNA) contains not less than 1.4×10^8 IU per mg of protein and not less than 2×10^8 IU of interferon alpha-2 per mL.

Description Interferon alpha-2 Concentrated Solution (rDNA) appears as clear, colorless to pale yellow liquid.

Identification (1) ***Inhibition of virus multiplication***—Perform the test as directed under Potency : multiplication of viruses such as the vesicular stomatitis virus, is inhibited.

(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 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 pI range 3.0 to 10.0. Examine with these solutions as directed in 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 solution (98 g/L H_3PO_4) as the anode solution and 1 mol/L sodium hydroxide solution as the cathode solution. Apply 15 μL each of the test solution and the standard solution on filter paper and place the filter paper on the gel close to the cathode. Start the isoelectric focusing at 1500 V, 50 mA. Turn off the power after 30 minutes, remove the filter paper and reconnect the power supply for 1 hour. Keep the power constant during the focusing process. After focusing, place the gel in a suitable amount of the solution containing 115 g of trichloroacetic acid and 34.5 g of sulfosalicylic acid in 1 L of water and gently shake the container for 60 minutes. Immerse the gel in a mixture of water, ethanol (99.5) and acetic acid (100) (268 : 100 : 32) for 5 minutes. Immerse the gel for 10 minutes in a pre-warmed staining solution containing 1.2 g of Coomassie Brilliant Blue R-250 in 1 L of a mixture of water, ethanol (99.5) and acetic acid (268 : 100 : 32). Wash the gel several times with a mixture of water, ethanol (99.5) and acetic acid (100) (268 : 100 : 32) and keep the gel in this mixture until the gel background is clear (12 to 24 hours). After adequate destaining, immerse the gel for 1 hour in a 10 % v/v solution of glycerol in a previously mixture of water, ethanol (99.5) and acetic acid (100) (268 : 100 : 32). Plot the migration distance of the isoelectric point markers versus their isoelectric points. Determine the isoelectric points of the test solution and the standard solution: the position of the principal band of the test solution corresponds to that of the principal band of the standard solution and they do not differ by more than 0.2 pH units. Isoelectric point markers are distributed along the entire length of the gel and the isoelectric points of the principal bands of the standard solution are between 5.8 and 6.3.

(3) ***Electrophoresis***—Examine the electropherograms obtained under the reducing conditions in the impurities of molecular masses differing from that of interferon alpha-2. The principal band in the electropherogram obtained with test solution (a) corresponds in position to the principal band in the electropherogram obtained with standard solution (a).

(4) ***Peptide map***—Dilute Interferon alpha-2 Concentrated Solution (rDNA) in water to a protein concentration of 1.5 mg/mL. Transfer 25 μL to a 1.5 mL polypropylene or glass tube. Add 1.6 μL of 1 mol/L phosphate buffer, pH 8.0, 2.8 μL of a freshly prepared 1.0 mg/mL trypsin solution and 3.6 μL of water and mix vigorously. Cap the tube and place it 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) solution, mix well place the capped tube at 95 °C to 100 °C for 1 minute. Cool to room temperature and use this solution as the test solution. Separately, dissolve Interferon alpha-2 RS in water to a concentration of 1.5 mg/mL. Proceed in the same manner at the same time as the test solution and use this solution as the standard solution. Examine with 100 μL each of the test solution and the standard solution as directed under Liquid Chromatography: the profile of the chromatogram obtained with the test solution corresponds to that of the chromatogram obtained with 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 about 10 cm in length, packed with octadecylsilyl silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 30 °C

Mobile phase: Control the step or concentration gradient by mixing mobile phases A and B as directed in the following table.

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 %)	Elution condition
0-8	100	0	Isocratic
8-68	100→40	0→60	Linear gradient
68-72	40	60	Isocratic
72-75	40→100	60→0	Linear gradient
75 ~ 80	100	0	Isocratic

Flow rate: 1.0 mL/minute. Equilibrate the column with mobile phase A for at least 15 minutes.

Purity (1) ***Impurities of molecular mass differing from that of interferon alpha-2***—Examine with reducing and non-reducing condition as following procedure to identify impurities of molecular mass differing from that of interferon alpha-2. In the test solution (a) under reducing conditions, in addition to the principal band, less intense bands with molecular masses lower than the principal band are not more intense than the principal band obtained with the standard solution (d) (1.0 %). Not more than 3 bands are more intense than the principal band obtained with the standard solution (e) (0.2 %). In the test solution (a) under non-reducing conditions, in addition to the principal band, less intense bands with molecular masses higher than the principal band are not more intense than the principal band obtained with the standard solution (d) (1.0 %). Not more than 3 bands are more intense than the principal band obtained with thestandard solution (e) (0.2 %). Prepare the test solutions and the standardsolutions under non-reducing and reducing conditions and examine as directed under Electrophoresis using resolving gels of 14 % acrylamide and silver staining.

Concentrated sodium dodecyl sulfate-polyacrylamide gel electrophoresis buffer: Dissolve 1.89 g of Tris, 5.0 g of sodium dodecyl sulfate and 50 mg of bromophenol blue in water, add 25.0 mL of glycerol and add water to mamke 100 mL. Adjust the pH to 6.8 with hydrochloric acid and 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 water and concentrated sodium dodecyl sulfate-polyacrylamide gel electrophoresis buffer containing 2-mercaptoethanol as the reducing agent.

Test solution (a): Dilute Interferon alpha-2 Concentrated Solution (rDNA) with the dilution buffer water to a protein concentration of 0.5 mg/mL.

Test solution (b): Dilute 0.20 mL of test solution (a) to 1 mL with the dilution buffer.

Standard solution (a): Dilute Interferon alpha-2 RS in the dilution buffer to a concentration of 0.625 mg/mL.

Standard solution (b): Dilute 0.20 mL of the standrad solution (a) in1 mL with the dilution buffer.

Standard solution (c): Dilute 0.20 mL of the standard solution (b) in 1 mL with the dilution buffer to make 1 mL.

Standard solution (d): Dilute 0.20 mL of the standard solution (c)in 1 mL with the dilution buffer.

Standard solution (e): Dilute 0.20 mL of the standard solution (d)in 1 mL with the dilution buffer.

Molecular mass standard solution (f): Use a solution of molecular mass standards suitable for calibrating sodium dodecyl sulfate-polyacrylamide gel electrophoresis in the range 15000 to 67000 Dalton.

Place the test and the standard solution on a water bath for 2 minutes. Apply 10 μL of the molecular mass standard solution (f) and 20 μL to 50 μL each of the test solutions and the standard solutions to the wells of the concentrated gel. After performing electrophoresis, detect the gel by silver staining: a band is seen in the standard solution (e). A gradation of intensity of staining is seen in the test solutions (a) and (b) and standard solutions (a) through (e), respectively.

(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. To the test solution, add suitable volume of 0.25 % hydrogen peroxide to give a final concentration of 0.005 % and allow to stand at room temperature for 1 hour, or for the length of time that will generate about 5 % oxidized interferon, and use this solution as the standard solution. Add 12.5 mg of L-methionine per mL of the standard solution and allow to stand at room temperature for 1 hour. Store the solutions for no longer than 24 hours at 2 – 8 °C. Examine with 50 μL each of the test solution and the standard solution as directed under Liquid Chromatography. In the chromatograms obtained, interferon alpha-2 elutes at a retention time of about 20 minutes. In the chromatogram obtained with the standard solution, a peak related to the oxidized interferon appears at a retention time of about 0.9 relative to the principal peak. The total area of any peaks other than the principal peak is not greater than 5.0 % of the total area of all of the peaks.

Operating conditions

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

Column: A stainless steel column 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).

Mobile phase: Control the step or concentration gradient by mixing mobile phases A and B as directed in the following table.

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 %)	Elution condition
0-1	72	28	Isocratic
1-5	72→67	28→33	Linear gradient
5-20	67→63	33→37	Linear gradient
20-30	63→57	37→43	Linear gradient
30-40	57→40	43→60	Linear gradient
40-42	40	60	Isocratic
42-50	40→72	60→28	Linear gradient

50-60	72	28	Isocratic

Flow rate: 1.0 mL/minute. Equilibrate the column with the initial gradient ratio for at least 15 minutes.

System suitability

The resolution between the principal peak of interferon and the peak of oxidized interferon is at least 1.0. The retention time of the peak of oxidized interferon is 0.7 to 1.4 relative to the principal peak. In the chromatogram obtained with the test solution, the area of any peak apart from the principal peak is not greater than 3.0 % of the total area of all of the peaks.

Bacterial Endotoxins Less than 100 EU/mg of interferon alpha-2.

Assay (1) ***Protein content***—Dilute Interferon alpha-2 Concentrated Solution with water to an interferon alpha-2 concentration of about 0.5 mg/mL, and use this solution as the test solution. Prepare 0.5 mg/mL bovine albumin solution as the stock solution. Dilute the stock solution to concentration between 3 μg/mL and 30 μg/mL to prepare standard solutions of 8 different concentrations. Examine with the test solution and the standard solutions as directed below. Prepare 30-fold and 50-fold dilutions of the test solution. Combine 2.0 mL of 20 g/L copper sulfate solution and 2.0 mL of 40 g/L tartrate solution and mix with 96.0 mL of a solution of 40 g/L sodium carbonate solution in 0.2 mol/L sodium hydroxide. To 1.25 mL of this solution, add 1.5 mL of water, or add 1.5 mL of the diluted test solutions or standard solutions. After about 10 minutes, add to each test tube 0.25 mL of a mixture of equal volumes of water and phosphomolybdotungstic reagent. After about 30 minutes, Measure the absorbance of each solution at 750 nm using the blank as the compensation liquid. Draw calibration curve from the absorbances of the 8 standard solutions and the corresponding protein contents, and determine the protein content of the test solution from this calibration curve.

(2) ***Potency***—The potency of interferon alpha-2 is estimated by comparing its effect to protect cells against a viral cytopathic effect with the same effect of the International Standard of human recombinant interferon alpha-2 or of a reference preparation calibrated in International Units. Carry out the potency based on the following conditions. Incubate a constant number of cells in each well of the microplate and include appropriate controls of untreated cells. Treat the cells with more than 3 different concentrations of the test solutions and the standard solutions. Perform the test at least 4 series to calculate the potency. Select the interferon concentrations such that the lowest concentration produces some protection and the largest concentration produces not more than maximum protection against the viral cytopathic effect. Add at a suitable time the cytophathic virus to all wells with the exception of the untreated control cells. When the cytophathic effect is confirmed, stain with a suitable solution, wash and dry the microplate at room temperature. Extract with 2-methoxyethanol, measure the absorbances at 550 nm and quantitatively determine the cytopathic effect. Calculate the potency of the test solution by the usual statistical methods for a parallel line assay. The estimated potency is not less than 80 % and not more than 120 % of the labeled potency,The confidence limit (P = 0.95) is not less than 64 % and not more than 156 % of the labeled potency. Use a established cell line sensitive to cytopathic effect of a suitable virus in standard culture conditions. The following cells 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 virus strain; or A549 cells (ATCC No. CCL-185) responsive to interferon as the cell culture and encephalomyocarditis virus (ATCC No. VR-129B) as the virus strain.

Containers and Storage ***Containers***—Hermetic containers.

Storage—Light-resistant, and below -20 °C.

Japanese Encephalitis Vaccine

Japanese Encephalitis Vaccine is a liquid preparation obtained by cultivating Japanese Encephalitis virus then separating, purifying and inactivating the antigen.
Japanese Encephalitis Vaccine conforms to the requirements of Japanese Encephalitis Vaccine in the Specifications and Test Methods for Biological Products of Korea.

Live Attenuated Oral Rotavirus Vaccine

Live Attenuated Oral Rotavirus Vaccine is a freeze-dried preparation or liquid preparation containing live attenuated rotavirus.
Live Attenuated Oral Rotavirus Vaccine conforms to the requirements of Live Attenuated Oral Rotavirus Vaccine in the Specifications and Test Methods for Biological Products of Korea.

Live Attenuated Varicella Vaccine

Live Attenuated Varicella Vaccine is a freeze-dried preparation containing live attenuated varicella virus. It becomes a liquid preparation on addition of solvent.
Live Attenuated Varicella Vaccine conforms to the requirements of Live Attenuated Varicella Vaccine in the Specifications and Test Methods for Biological Products of Korea.

Oral Typhoid Vaccine

Oral Typhoid Vaccine is a filled capsule preparation or enteric-coated preparation containing freeze-dried live attenuated strain *Salmonella typhi* Ty21a.
Oral Typhoid Vaccine conforms to the requirements of Oral Typhoid Vaccine in the Specifications and Test Methods for Biological Products of Korea.

Pneumococcal Polysaccharide Vaccine

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 conforms to the requirements of Pneumococcal Polysaccharide Vaccine in the Specifications and Test Methods for Biological Products of Korea.

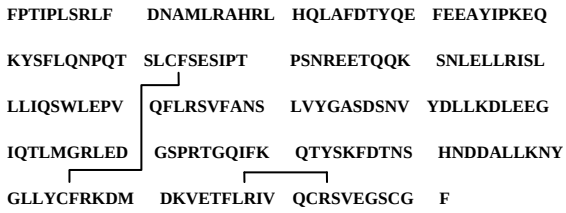
Pneumococcus Conjugated to Diphtheria CRM197 Vaccine

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 mutant of diphtheria toxin.
Pneumococcus Conjugated to Diphtheria CRM197 Vaccine conforms to the requirements of Pneumococcus Conjugated to Diphtheria CRM197 Vaccine in the Specifications and Test Methods for Biological Products of Korea.

Purified Vi Polysaccharide Typhoid Vaccine

Purified Vi Polysaccharide Typhoid Vaccine is a liquid preparation containing inactivated purified Vi capsular polysaccharide of *Salmonella typhi*.
Purified Vi Polysaccharide Typhoid Vaccine conforms to the requirements of Purified Vi Polysaccharide Typhoid Vaccine in the Specifications and Test Methods for Biological Products of Korea.

Somatropin (rDNA)



$C_{990}H_{1528}N_{262}O_{300}S_7$: 22125

Somatropin (rDNA) is a recombinant protein of the human growth hormone having 191 amino acid residues.
Somatropin (rDNA) contains not less than 91.0 % and not more than 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 biological activity.

Description Somatropin (rDNA) appears as white powder.

Identification (1) Examine as directed in Method (1) or (2) in Charged variants under Purity.

(1-1) ***Capillary electrophoresis***—To the test method described in Capillary electrophoresis in Charged variants under Purity, apply a modification of the following:

Injection: Inject the test solution (b) for minimum 3 seconds under pressure or vacuum then inject the capillary electrophoresis buffer solution for 1 second. One principal peak corresponding to somatropin should be observed.

(1-2) ***Isoelectric focusing***—Examine the eletropherogram of Isoelectric focusing in Charged variants under Purity: the principal band obtained with the test solution (a) corresponds in position to that with the standard solution (a).

(2) ***Reversed-phase liquid chromatography***—Exam-ine the chromatograms obtained with the test for related substances: the retention time of the principal peak in the chromatogram obtained with the test solution is similar to that of the principal peak in the chromatogram with the standard solution.

(3) ***Peptide map***

Test solution: Dilute Somatropin (rDNA) with 0.05 mol/L tris-hydrochloric acid buffer (pH 7.5) to a somatropin concentration of 2.0 mg/mL. Transfer about 1.0 mL to a tube made of a suitable material such as polypropylene. Prepare a 1 mg/mL trypsin solution diluted with 0.05 mol/L tris-hydrochloric acid buffer (pH 7.5) and add 30 μL of this solution to the test specimen. Cap the tube and place in a water bath at 37 °C for 4 hours. Remove from the water bath and stop the reaction immediately, for example by freezing. Analyze immediately using an automatic injector, maintaining the temperature at 2 °C to 8 °C.

Standard solution: Prepare in the same manner at the same time as for the test solution, using Somatropin Reference Standard (RS).

Examine with 100 μL each of the test and the standard solution as directed under Liquid Chromatography according to the following operating conditions: The profile of the chromatogram obtained with the test solution corresponds to that with 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 about 25 cm in length, packed with octylsilyl silica gel for the liquid chromatography (5 μm to 10 μm in particle diameter).

Column temperature: A constant temperature of about 30 °C

Mobile phase: Control the concentration gradient by mixing mobile phases A and B as directed in the following table.

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 %)	Elution condition
0-20	100→80	0→20	Linear gradient
20-40	80→75	20→25	Linear gradient
40-65	75→50	25→50	Linear gradient
65-70	50→20	50→80	Linear gradient

Flow rate: 1 mL/minute

(4) ***Size-exclusion liquid chromatography***—Examine the chromatograms as obtained with the Assay: the retention time and size of the principal peak in the chromatogram obtained with the test solution are similar to those of the principal peak in the chromatogram from 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 a somatropin concentration of 2.0 mg/mL. If the concentration of the test solution is lower, adjust the injection volume.

Standard solution: Dilute Somatropin RS with 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) to a protein concentration of 2.0 mg/mL.

System suitability resolution solution: Dilute Somatropin/Desamino-somatropin Mixture RS with 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) to a somatropin concentration of 2 mg/mL.

Examine with 20 μL each of the test and the standard solution as directed under Liquid Chromatography according to the following operating conditions: the amount of related substances is not more than 6.0 %.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 220 nm)

Column: A stainless steel column 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 mL to 500 mL of a mixture of 50 % acetonitrile and 0.1 % trifluoroacetic acid. If necessary, repeat the equilibration to improve column performance.

Flow rate: 0.5 mL/minute

System suitability

System performance: The retention time of desamido-somatropin in the standard solution is about 0.85 relative to the principal peak (the retention time of somatropin is about 33 minutes; if necessary, adjust the volume of propanol in the mobile phase). The test is not valid unless the resolution between the peak of desamido and the peak of somatropin is not less than 1.0 with the symmetry factor of the peak of somatropin being to 1.8.

(2) ***Dimers and related substances of higher molecular mass***

Test solution: Dilute Somatropin (rDNA) with 0.025 mol/L phosphate buffer (pH 7.0) to a somatropin concentration of 1.0 mg/mL.

Standard solution: Dilute Somatropin RS with 0.025 mol/L phosphate buffer (pH 7.0) to a somatropin concentration of 1.0 mg/mL.

System suitability resolution 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 contents in phosphate buffer (pH 7.0) to a somatropin concentration of 1.0 mg/mL.

Examine with 20 μL each of the test solution and standard solution as directed under Liquid Chromatography according to the following operating conditions: the total area of all peaks appeared before the principal peak of the test solution is not more than 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 and capable of eluting proteins having a molecular mass 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/minute

System suitability

System performance: The retention time of the peak of higher molecular mass related substance is about 0.65 relative to the retention time of somatropin monomer (12 minutes to 17 minutes). The retention time of the peak of the somatropin dimer is about 0.9 relative to the retention time (12 minutes to 17 minutes) of the somatropin monomer from the system suitability resolution solution. The peak-to-valley ratio is not less than 2.5.

(3) ***Charged variants***—Use Method (1) or (2).

Method (1) Capillary electrophoresis

Test solution (a): Dilute Somatropin (rDNA) with water to a somatropin concentration of 1 mg/mL.

Test solution (b): Mix equal volumes of test solution (a) and the standard solution.

Standard solution: Dilute Somatropin RS with water to a somatropin concentration of 1 mg/mL.

Capillary electrophoresis buffer: Adjust the pH of 13.2 g/L ammonium phosphate buffer to 6.0 with phosphoric acid then filter through a membrane.

Examine with the test solution and the standard solution as directed under Capillary electrophoresis according to the following operating conditions: deamidated forms are not more than 5.0 %, each impurity is not more than 2.0 % and total charged variants is not more than 10.0 %.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 200 nm)

Capillary: An uncoated silica gel capillary about 50 μm in internal diameter and about 70 cm in effective length.

Capillary temperature: A constant temperature of about 30 °C

Step	Solution	Time	Condition
Capillary equilibration	1 mol/L sodium hydroxide	20 min	Pressure or vacuum
	Water	10 min	
	Capillary electrophoresis buffer	20 min	
Between-run washing	0.1 mol/L sodium hydroxide	2 min	Pressure or vacuum
	Capillary electrophoresis buffer	6 min	
Injection	Injection of test solution and standard solution	3 sec	Pressure or vacuum
		1 sec	
	Capillary electrophoresis buffer		
Separation	Capillary electrophoresis buffer	80 min	217 V/cm

System suitability

System performance: The relative migration distance of the deamidated form is 1.02 to 1.11 relative to the migration distance of somatropin, and the electropherograms of the standard solution and that of the test solution are similar. Two peaks (I1, I2) are detected before the principal peak and more than two peaks (I3, I4) are detected after the principal peak. I2 is fragmented form and I4 is deamidated form, detected as two peaks.

Method (2) Isoelectric focusing

Test solution (a): Dilute Somatropin (rDNA) with 0.025 mol/L phosphate buffer (pH 7.0) to a somatropin concentration of 2.0 mg/mL.

Test solution (b): Mix 1.9 mL of 0.025 mol/L phosphate buffer (pH 7.0) with 0.1 mL of test solution (a).

Standard solution (a): Dilute Somatropin RS with 0.025 mol/L phosphate buffer (pH 7.0) to a somatropin concentration of 2.0 mg/mL.

Standard solution (b): Use an isoelectric point calibration solution in the pH range of 2.5 to 6.5.

Operating conditions

Examine by isoelectric focusing using polyacrylamide gel in the pH range of 4.0 to 6.5. Apply 15 μL each of the test and the standard solution to the gel. Use a 14.7 g/L solution of glutamic acid in phosphoric acid (50 g/L H_3PO_4) as the anode solution and 89.1 g/L β-alanine solution as the cathode solution. Adjust the operating conditions to 2000 V, 25 mA. Allow focusing by maintaining a constant voltage for 2.5 hours with the power not exceeding 25 W. After focusing is complete, immerse the gel in a suitable volume of a solution containing 115 g/L trichloroacetic acid solution and 34.5 g/L sulfosalicylic acid for 30 minutes then transfer to a mixture of water, ethanol (99.5) and acetic acid (100) (67 : 25 : 8) and immerse for 5 minutes. Stain the gel by immersing for 10 minutes in the staining solution, prepared 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) at 60 °C. After staining, place the gel in de-stain solution with a mixture of water, ethanol (99.5) and acetic acid (100) (67 : 25 : 8) until excess stain is removed.

No band apart from the principal band obtained with the test solution (a) is more intense than the principal band of the test solution (b) (not more than 5 %).

System suitability

System performance: Each band of the isoelectric point calibration solution of the standard solution (b) is well separated. The standard solution (a) shows a principal band at the isoelectric points of approximately 5.0, and minor band at the isoelectric points of approximately 4.8, respectively.

Water Not more than 10.0 %

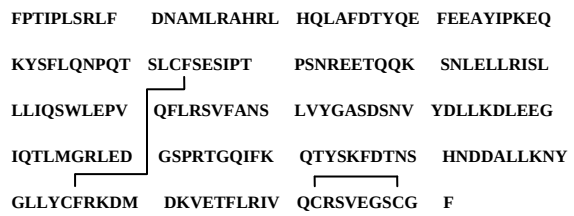
Bacterial Endotoxins Less than 5 EU/mg of somatropin.

Assay Use the chromatograms from the test for the dimers and related substances of higher molecular mass to calculate the amount of somatropin in the test specimen from the labeled amount of Somatropin RS.

Containers and Storage ***Containers***—Hermetic containers.

Storage—At a temperature between 2 °C and 8 °C.

Somatropin Concentrated Solution (rDNA)



$C_{990}H_{1528}N_{262}O_{300}S_7$: 22125

Somatropin Concentrated Solution (rDNA) is a solution containing recombinant protein of the human growth hormone having 191 amino acid residues.
Somatropin (rDNA) contains not less than 91.0 % and not more than 105.0 % of the amount of somatropin stated on the label. 1 mg of anhydrous somatropin ($C_{990}H_{1528}N_{262}O_{300}S_7$) is equivalent to 3.0 IU of biological activity.

Description Somatropin Concentrated Solution (rDNA) appears as clear or slightly turbid, colorless liquid.

Identification (1) Examine as directed in Method (1) or (2) in Charged variants under Purity.

(1-1) ***Capillary electrophoresis***—To the test method described in Capillary electrophoresis in Charged variants under Purity, apply a modification of the following:

Injection: Inject the test solution (b) for minimum 3 seconds under pressure or vacuum then inject the capillary electrophoresis buffer solution for 1 second. One principal peak corresponding to somatropin should be observed.

(1-2) ***Isoelectric focusing***—Examine the electropherogram of Isoelectric focusing in Charged variants under Purity: the principal band obtained with the test solution (a) corresponds in position to that with the standard solution (a).

(2) ***Reversed-phase liquid chromatography***—Exam-ine the chromatograms obtained with the test for related substances: the retention time of the principal peak in the chromatogram obtained with the test solution is similar to that of the principal peak in the chromatogram from the standard solution.

(3) ***Peptide map***

Test solution: Dilute Somatropin Concentrated Solution (rDNA) with 0.05 mol/L tris-hydrochloric acid buffer (pH 7.5) to a somatropin concentration of 2.0 mg/mL. Transfer about 1.0 mL to a tube made of a suitable material, such as polypropylene. Prepare a 1 mg/mL trypsin solution diluted with 0.05 mol/L tris-hydrochloric acid buffer (pH 7.5) and add 30 μL of this solution to the test specimen. Cap the tube and place in a water bath at 37 °C for 4 hours. Remove from the water bath and stop the reaction immediately, for example by freezing. Analyze immediately using an automatic injector, maintaining the temperature at 2 °C to 8 °C.

Standard solution: Prepare in the same manner at the same time as for the test solution, using Somatropin Reference Standard(RS).

Examine with 100 μL each of the test and the standard solution as directed under Liquid Chromatography according to the following operating conditions: The profile of the chromatogram obtained with the test solution corresponds to that with 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 about 25 cm in length, packed with octylsilyl silica gel for the liquid chromatography (5 μm to 10 μm in particle diameter).

Column temperature: A constant temperature of about 30 °C

Mobile phase: Control the concentration gradient by mixing mobile phases A and B as directed in the following table.

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 %)	Elution condition
0-20	100→80	0→20	Linear gradient
20-40	80→75	20→25	Linear gradient
40-65	75→50	25→50	Linear gradient
65-70	50→20	50→80	Linear gradient

Flow rate: 1 mL/minute

(4) ***Size-exclusion liquid chromatography***—Examine the chromatograms as obtained with the Assay: the retention time and size of the principal peak in the chromatogram obtained from the test solution are similar to those of the principal peak in the chromatogram from 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 a somatropin concentration of 2.0 mg/mL. If the concentration of the test solution is lower, adjust the injection volume.

Standard solution: Dilute Somatropin RS with 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) to a protein concentration of 2.0 mg/mL.

System suitability resolution solution: Dilute Somatropin/Desamino-somatropin Mixture RS with 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) to a somatropin concentration of 2 mg/mL.

Examine with 20 μL each of the test and the standard solution as directed under Liquid Chromatography according to the following operating conditions: the amount of related substances is not more than 6.0 %.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 220 nm)

Column: A stainless steel column 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 equlibration: Equilibrate the column using 200 mL to 500 mL of a mixture of 50 % acetonitrile and 0.1 % trifluoroacetic acid. If necessary, repeat the equilibration to improve column performance.

Flow rate: 0.5 mL/minute

System suitability

System performance: The retention time of desamino-somatropin in the system suitability resolution solution is about 0.85 relative to the principal peak (the retention time of somatropin is about 33 minutes; if necessary, adjust the volume of propanol in the mobile phase). The test is not valid unless, the resolution between the peak of desamido and the peak of somatropin is not less than 1.0 with the symmetry factor of the peak of somatropin being 0.9 to 1.8.

(2) ***Dimers and related substances of higher molecular mass***

Test solution: Dilute Somatropin Concentrated Solution (rDNA) with 0.025 mol/L phosphate buffer (pH 7.0) to a somatropin concentration of 1.0 mg/mL.

Standard solution: Dilute Somatropin RS with 0.025 mol/L phosphate buffer (pH 7.0) to a somatropin concentration of 1.0 mg/mL.

System suitability resolution 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 contents in phosphate buffer (pH 7.0) to a somatropin concentration of 1.0 mg/mL.

Examine with 20 μL each of the test solution and standard solution as directed under Liquid Chromatography according to the following operating conditions: the total area of all peaks appeared before the principal peak of the test solution is not more than 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 and capable of eluting proteins having a molecular mass 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/minute

System suitability

System performance: The retention time of the peak of higher molecular mass, related substance is about 0.65 relative to the retention time of somatropin monomer, (12 minutes to 17 minutes) The retention time of the peak of the somatropin dimer is about 0.9 relative to the retention time (12 minutes to 17 minutes) of the somatropin monomer from the system suitability resolution solution, . The peak-to-valley ratio is not less than 2.5.

(3) ***Charged variants***—Use Method (1) or (2).

Method (1) Capillary electrophoresis

Test solution (a): Dilute Somatropin Concentrated Solution (rDNA) with water to a somatropin concentration of 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 a somatropin concentration of 1 mg/mL.

Capillary electrophoresis buffer: Adjust the pH of 13.2 g/L ammonium phosphate buffer to 6.0 with phosphoric acid then filter through a membrane .

Examine with the test solution and the standard solution as directed under Capillary electrophoresis according to the following operating conditions: deamidated forms are not more than 5.0 %, each impurity is not more than 2.0 % and total charged variants is not more than 10.0 %.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 200 nm)

Capillary: An uncoated silica gel capillary about 50 μm in internal diameter and about 70 cm in effective length.

Capillary temperature: A constant temperature of about 30 °C

Step	Solution	Time	Condition
Capillary equilibration	1 mol/L sodium hydroxide	20 min	Pressure or vacuum
	Water	10 min	
	Capillary electrophoresis buffer	20 min	
Between-run washing	0.1 mol/L sodium hydroxide	2 min	Pressure or vacuum
	Capillary electrophoresis buffer	6 min	
Injection	Injection of test solution and standard solution	3 sec	Pressure or vacuum
	Capillary electrophoresis buffer	1 sec	
Separation	Capillary electrophoresis buffer	80 min	217 V/cm

System suitability

System performance: The relative migration distance of the deamidated form is 1.02 to 1.11 relative to the migration distance of somatropin, and the electropherograms of the standard solution and that of the test solution are similar. Two peaks (I1, I2) are detected before the principal peak and more than two peaks (I3, I4) are detected after the principal peak. I2 is fragmented form and I4 is deamidated form, detected as two peaks.

Method (2) Isoelectric focusing

Test solution (a): Dilute Somatropin Concentrated Solution (rDNA) with 0.025 mol/L phosphate buffer (pH 7.0) to a somatropin concentration of 2.0 mg/mL.

Test solution (b): Mix 1.9 mL of 0.025 mol/L phosphate buffer (pH 7.0) with 0.1 mL of test solution (a).

Standard solution (a): Dilute Somatropin RS with 0.025 mol/L phosphate buffer (pH 7.0) to a somatropin concentration of 2.0 mg/mL.

Standard solution (b): Use an isoelectric point calibration solution in the pH range of 2.5 to 6.5.

Operating conditions

Examine by isoelectric focusing using polyacrylamide gel in the pH range of 4.0 to 6.5. Apply 15 μL each of the test and the standard solution to the gel. Use a 14.7 g/L solution of glutamic acid in phosphoric acid (50 g/L H_3PO_4) as the anode solution and 89.1 g/L β-alanine solution as the cathode solution. Adjust the operating condition to 2000 V, 25 mA. Allow focusing by maintaining a constant voltage for 2.5 hours with the power not exceeding 25 W. After focusing is complete, immerse the gel in a suitable volume of a solution containing 115 g/L trichloroacetic acid and 34.5 g/L sulfosalicylic acid for 30 minutes then transfer to a mixture of water, ethanol (99.5) and acetic acid (100) (67 : 25 : 8) and immerse for 5 minutes. Stain the gel by immersing for 10 minutes in the staining solution, prepared 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) at 60 °C. After staining, place the gel in –de-stain solution with a mixture of water, ethanol (99.5) and acetic acid (100) (67 : 25 : 8) until excess stain is removed.

No band apart from the principal band obtained with test solution (a) is more intense than the principal band of test solution (b) (not more than 5 %).

System suitability

System performance: Each band of the isoelectric point calibration solution of the standard solution (b) is well separated. The standard solution (a) shows a principal band at the isoelectric points of approximately 5.0, and minor -band at the isoelectric points of approximatelyt 4.8, respectively.

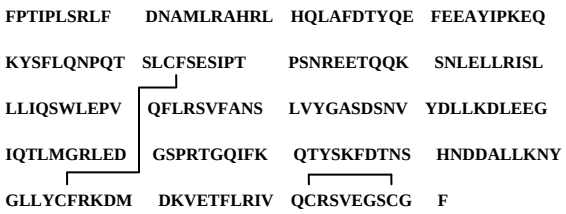
Bacterial Endotoxins Less than 5 EU/mg of somatropin.

Assay Use the chromatograms from the test for the dimers and related substances of higher molecular mass to calculate the amount of somatropin in the test specimen from the labeled amount of Somatropin RS.

Containers and Storage ***Containers***—Hermetic containers.

Storage—At -20 °C, and avoid repeated freezing and thawing.

Somatropin for Injection (rDNA)



$C_{990}H_{1528}N_{262}O_{300}S_7$: 22125

Somatropin for Injection (rDNA) is a preparation for injection which is reconstituted before use.

Somatropin (rDNA) contains not less than 89.0 % and not more than 105.0 % of the amount of somatropin stated on the label. 1 mg of anhydrous somatropin ($C_{990}H_{1528}N_{262}O_{300}S_7$) is equivalent to 3.0 IU of biological activity.

Description Somatropin for injection (rDNA) appears as white powder.

Identification (1) Examine as directed in Method (1) or (2) in Charged variants under Purity.

(1-1) ***Capillary electrophoresis***—To the test method described in Capillary electrophoresis in Charged variants under Purity, apply a modification of the following: .

Injection: Inject the test solution (b) for minimum 3 seconds under pressure or vacuum then inject the capillary electrophoresis buffer solution for 1 second. One principal peak corresponding to somatropin should be observed.

(1-2) ***Isoelectric focusing***—Examine the elecrtopherogram of Isoelectric focusing in Charged variants under Purity: the principal band obtained with test solution (a) corresponds in position to that with the standard solution (a).

(2) ***Reversed-phase liquid chromatography***—Exam-ine the chromatograms obtained with the test for related substances: the retention time of the principal peak in the chromatogram obtained with the test solution is similar to that of the principal peak in the chromatogram withthe standard solution.

(3) ***Peptide map***

Test solution: Dilute Somatropin for Injection (rDNA) with 0.05 mol/L tris-hydrochloric acid buffer (pH 7.5) to a somatropin concentration of 2.0 mg/mL. Transfer about 1.0 mL to a tube made of a suitable material, such as polypropylene. Prepare a 1 mg/mL trypsin solution diluted with 0.05 mol/L tris-hydrochloric acid buffer (pH 7.5) and add 30 μL of this solution to the test specimen. Cap the tube and place in a water bath at 37 °C for 4 hours. Remove from the water bath and stop the reaction immediately, for example by freezing. Analyze immediately using an automatic injector, maintaining the temperature at 2 °C to 8 °C.

Standard solution: Prepare in the same manner at the same time as the test solution, using Somatropin RS .

Examine with 100 μL each of the test solution and the standard solution as directed under Liquid Chromatography according to the following operating conditions: The profile of the chromatogram obtained with the test solution corresponds to that with 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 about 25 cm in length, packed with octylsilyl silica gel for the liquid chromatography (5 μm to 10 μm in particle diameter).

Column temperature: A constant temperature of about 30 °C

Mobile phase: Control the concentration gradient by mixing mobile phases A and B as directed in the following table.

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 %)	Elution condition
0-20	100→80	0→20	Linear gradient
20-40	80→75	20→25	Linear gradient
40-65	75→50	25→50	Linear gradient
65-70	50→20	50→80	Linear gradient

Flow rate: 1 mL/minute

(4) ***Size-exclusion liquid chromatography***—Examine the chromatograms as obtained with the Assay: the retention time and size of the principal peak in the chromatogram obtained from the test solution are similar to those of the principal peak in the chromatogram from 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 a somatropin concentration of 2.0 mg/mL. If the concentration of the test solution is lower, adjust the injection volume.

Standard solution: Dilute Somatropin RS with 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) to a protein concentration of 2.0 mg/mL.

System suitability resolution solution: Dilute Somatropin/Desamino-somatropin Mixture RS with 0.05 mol/L tris-hydrochloric acid buffer solution (pH 7.5) to a somatropin concentration of 2 mg/mL.

Examine with 20 μL each of the test and the standard solution as directed under Liquid Chromatography according to the following operating conditions: the amount of related substances is not more than 13.0 %.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 220 nm)

Column: A stainless steel column 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 mL to 500 mL of a mixture of 50 % acetonitrile and 0.1 % trifluoroacetic acid. If necessary, repeat the equilibration to improve column performance.

Flow rate: 0.5 mL/minute

System suitability

System performance: The retention time of desamido-somatropin in the system suitability resolution solution is about 0.85 relative to the principal peak (the retention time of somatropin is about 33 minutes; if necessary, adjust the volume of propanol in the mobile phase). The test is not valid unless the resolution between the peak of desamido and the peak of somatropin is not less than 1.0 with the symmetry factor of the peak of somatropin being 0.9 to 1.8.

(2) ***Dimers and related substances of higher molecular mass***

Test solution: Dilute Somatropin for Injection (rDNA) with 0.025 mol/L phosphate buffer (pH 7.0) to a somatropin concentration of 1.0 mg/mL.

Standard solution: Dilute Somatropin RS with 0.025 mol/L phosphate buffer (pH 7.0) to a somatropin concentration of 1.0 mg/mL.

System suitability resolution solution: Place Som-atropin 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 contents in phosphate buffer (pH 7.0) to a somatropin concentration of 1.0 mg/mL.

Examine with 20 μL each of the test solution and standard solution as directed under Liquid Chromatography according to the following operating conditions: the total area of all peaks appeared before the principal peak of the test solution is not more than 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 and capable of eluting proteins having a molecular mass 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/minute

System suitability

System performance: The retention time of the peak of higher molecular mass, related substance is about 0.65 relative to the retention time of somatropin monomer, (12 minutes to 17 minutes) The retention time of the peak of the somatropin dimer is about 0.9 relative to the retention time (12 minutes to 17 minutes) of the somatropin monomer from the system suitability resolution solution. The peak-to-valley ratio is not less than 2.5.

(3) ***Charged variants***—Use Method (1) or (2).

Method (1) Capillary electrophoresis

Test solution (a): Dilute Somatropin for Injection (rDNA) with water to a somatropin concentration of 1 mg/mL.

Test solution (b): Mix equal volumes of test solution (a) and the standard solution.

Standard solution: Dilute Somatropin RS with water to a somatropin concentration of 1 mg/mL.

Capillary electrophoresis buffer: Adjust the pH of 13.2 g/L ammonium phosphate buffer to 6.0 with phosphoric acid then filter through a membrane.

Examine with the test solution and the standard solution as directed under Capillary electrophoresis according to the following operating conditions: deamidated forms are not more than 6.5 %, each impurity is not more than 2.0 % and total charged variants is not more than 11.5 %.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 200 nm)

Capillary: An uncoated silica gel capillary about 50 μm in internal diameter and about 70 cm in effective length.

Capillary temperature: A constant temperature of about 30 °C

Step	Solution	Time	Condition
Capillary equilibration	1 mol/L sodium hydroxide	20 min	Pressure or vacuum
	Water	10 min	
	Capillary electrophoresis buffer	20 min	
Between-run washing	0.1 mol/L sodium hydroxide	2 min	Pressure or vacuum
	Capillary electrophoresis buffer	6 min	
Injection	Injection of test solution and standard solution	3 sec	Pressure or vacuum
	Capillary electrophoresis buffer	1 sec	
Separation	Capillary electrophoresis buffer	80 min	217 V/cm

System suitability

System performance: The relative migration distance of the deamidated form 1.02 to 1.11 relative to the migration distance of somatropin, and the electropher-ograms of the reference solution and that of the test solution are similar. Two peaks (I1, I2) are detected before the principal peak and more than two peaks (I3, I4) are detected after the principal peak. 2 is fragmented form and I4 is deamidated form, detected as two peaks.

Method (2) Isoelectric focusing

Test solution (a): Dilute Somatropin for Injection (rDNA) with 0.025 mol/L phosphate buffer (pH 7.0) to a somatropin concentration of 2.0 mg/mL.

Test solution (b): Mix 1.9 mL of 0.025 mol/L phosphate buffer (pH 7.0) with 0.1 mL of test solution (a).

Standard solution (a): Dilute Somatropin RS with 0.025 mol/L phosphate buffer (pH 7.0) to a somatropin concentration of 2.0 mg/mL.

Standard solution (b): Use an isoelectric point calibration solution in the pH range of 2.5 to 6.5.

Operating conditions

Examine by isoelectric focusing using polyacrylamide gel in the pH range of 4.0 to 6.5. Apply 15 μL each of the test and the reference solution to the gel. Use a 14.7 g/L solution of glutamic acid in phosphoric acid (50 g/L H_3PO_4) as the anode solution and 89.1 g/L β-alanine solution as the cathode solution. Adjust operating conditions to 2000 V, 25 mA. Allow focusing by maintaining a constant voltage for 2.5 hours with the power not exceeding 25 W. After focusing is complete, immerse the gel in a suitable volume of a solution containing 115 g/L trichloroacetic acid and 34.5 g/L sulfosalicylic acid for 30 minutes then transfer to a mixture of water, ethanol (99.5) and acetic acid (100) (67 : 25 : 8) and immerse for 5 minutes. Stain the gel by immersing for 10 minutes in staining solution, prepared 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) at 60 °C. After staining, place the gel in with a mixture of water, ethanol (99.5) and acetic acid (100) (67 : 25 : 8) until excess stain is removed.

No band apart from the principal band obtained with test solution (a) is more intense than the principal band of test solution (b) (not more than 6.25 %).

System suitability

System performance: Each band of the isoelectric point calibration solution of the standard solution (b) is well separated. The standard solution (a) shows a principal band at the isoelectric points of approximately 5.0 and minor -band at the isoelectric points of approximately 4.8, respectively.

Water Not more than 3.0 %

Sterility Test It meets the requirement.

Bacterial Endotoxins Less than 5 EU/mg of somatropin.

Foreign Insoluble Matter Test It meets the requirement.

Insoluble Particulate Matter Test for Injections It meets the requirement.

Determination of Volume of Injection in Containers It meets the requirement.

Assay Use the chromatograms from the test for the dimers and related substances of higher molecular mass to calculate the amount of somatropin in the test specimen from the labeled amount of Somatropin RS.

Containers and Storage ***Containers***—Hermetic containers.

Storage—At a temperature between 2 °C and 8 °C.3

Tetanus Antitoxin (Equine)

Tetanus Antitoxin (Equine) is a liquid preparation containing tetanus antitoxin of animal immunoglobulin.
Tetanus Antitoxin (Equine) conforms to the requirements of Tetanus Antitoxin (Equine) in the Specifications and Test Methods for Biological Products of Korea.

3) Compound Preparations

Absorptive Ointment

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	a sufficient quantity
	To make 1000 g

Melt White Petroleum, Cetanol, White Beeswax, Sorbitan Sesquioleate, and Lauromacrogol by heating on a water-bath, mix, and maintain at about 75 °C. Add Methylparaben or Ethylparaben and Propylparaben or Butylparaben to Purified Water, dissolve by warming at 80 °C. Combine both solutions, mix to make emulsion, cool, and stir thoroughly until it congeals.

Description Absorptive Ointment is a white, lustrous, and has a slight, characteristic odor.

Containers and Storage ***Containers***—Tight containers.

Alum Solution

Alum Solution contains not less than 0.27 % and not more than 0.33 % of aluminum potassium sulfate hydrate [$AlK(SO_4)_2 \cdot 12H_2O$: 474.39].

Method of Preparation

Aluminum Potassium Sulfate Hydrate	3 g
Mentha Water	50 mL
Water or Purified Water	a sufficient quantity
	To make 1000 mL

Dissolve and mix the above ingredients.

Description Alum Solution is a clear, colorless liquid. Alum Solution has a mentha oil-like odor and an astringent taste.

Identification (1) Take 5 mL of Alum Solution, add 3 mL of ammonium chloride TS and 1 mL of ammonia TS: a white, gelatinous precipitate is produced, which changes to red upon the addition of 5 drop of alizarin S TS (Aluminum sulfate).

(2) Alum Solution responds to the Qualitative Tests (1) and (2) for sulfate.

(3) Place 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 solution responds to the Qualitative Tests for potassium salt.

Assay Pipet 50.0 mL of Alum Solution, add 30.0 mL of 0.02 mol/L disodium ethylene-diaminetetraacetate VS and add 20 mL of acetic acid-ammonium acetate buffer solution, pH 4.8. Boil for 5 minutes, cool, add 55 mL of ethanol (95) and titrate with 0.02 mol/L zinc acetate VS (indicator: 2 mL of dithizone TS) until the color of the solution changes from light dark green to pale red. Perform a blank determination and make any necessary correction.

Each mL of 0.02 mol/L
disodium ethylenediaminetetraacetate VS
= 9.488 mg of $AlK(SO_4)_2 \cdot 12H_2O$

Containers and Storage ***Containers***—Tight containers.

Epinephrine Solution

Epinephrine Hydrochloride Solution
Epirenamine Hydrochloride Solution
Adrenaline Hydrochloride Solution

Epinephrine Solution contains not less than 0.085 % and not more than 0.115 % of epinephrine ($C_9H_{13}NO_3$: 183.20).

Method of Preparation

Epinephrine	1 g
Sodium Hydroxide	8.5 g
Diluted hydrochloric acid (9 in 100)	10 mL
Preservative	a suitable amount
Stabilizer	a suitable amount
Purified Water	sufficient quantity
	To make 1000 mL

Description Epinephrine Solution is a clear, colorless or pale reddish solution.
Epinephrine Solution turns to pale red and brown due to air or light.

pH—2.3 ~ 5.0.

Identification Proceed as directed in the Identification under Epinephrine Injection.

Assay Proceed as directed in the Assay under Epinephrine Injection.

Containers and Storage ***Containers***—Tight containers.

Storage—Light-resistant.

Hydrophilic Ointment

Method of Preparation

White Petrolatum	250 g
Stearyl Alcohol	200 g
Propylene Glycol	120 g
Polyoxyethylene hydrogenated castor oil 60	40 g
Glycerin Monostrateae	10 g
Methylparaben	1 g
Propylparaben	1 g
Purified Water	a sufficient quantity
	To make 1000 g

Melt White Petrolatum, Stearyl Alcohol, polyoxyethylene hydrogenated castor oil 60 and Glycerin Monostearate by heating on a water-bath, stir and keep temperature of the mixture at about 75 °C. To Propylene Glycol, add Methylparaben and Propylparaben, melt by warming, if necessary, dissolve in purified water and warm to about 75 °C. Add this solution to the above mixture, stir to form emulsion, cool and stir thoroughly until it congeals.

Description Hydrophilic Ointment is white, has a slight, characteristic odor.

Containers and Storage ***Containers*—**Tight containers.

Hydrophilic Petrolatum

Method of preparation

White Beeswax	80 g
Stearyl Alcohol or Cetanol	30 g
Cholesterol	30 g
White Petrolatum	A sufficient quantity
	To make 1000 g

Melt and mix Stearyl Alcohol or Cetanol, White Beeswax and White Petrolatum on a water-bath. Add Cholesterol and melt completely by stirring. Stop warming and stir until the mixture congeals.

Description Hydrophilic Petrolatum is white, has a slight, characteristic odor.
When mixed with an equal volume of water, Hydrophilic Petrolatum retains the consistency of ointment.

Containers and Storage ***Containers*—**Tight containers.

Compound Iodine Glycerin

Compound Iodine Glycerin contains not less than 1.1w/v
% and not more than 1.3w/v % of iodine (I: 126.90), not less than 2.2w/v % and not more than 2.6w/v % of potassium iodide (KI: 166.00), not less than 2.7w/v % and not more than 3.3w/v % of total iodine (as I) and not less than 0.43w/v % and not more than 0.53w/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
Liquefied Phenol	5 mL
Purified Water	a sufficient quantity
	To make 1000 mL

Dissolve Potassium Iodide and Iodine in about 25 mL of Purified Water. After adding Glycerin, add Mentha Water, Liquefied Phenol and add sufficient Purified Water to make 1000 mL, mixing thoroughly. It may be prepared with an appropriate quantity of Concentrated Glycerin and Purified Water in place of Glycerin.

Description Compound Iodine Glycerin is red-brown, viscous liquid, has a characteristic odor.

Specific gravity— d_{20}^{20} : About 1.23.

Identification (1) The colored solution obtained in the Assay (1) has a red color. Determine the absorption spectrum of this solution as directed under the Ultraviolet-visible Spectrophotometry : it exhibits a maximum between 510 nm and 514 nm (iodine).

(2) The colored solution obtained in the Assay (2) has a red color. Determine the absorption spectrum of this solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits maxima between 510 nm and 514 nm (potassium iodide).

(3) The colored solution obtained in the Assay (4) has a yellow color. Determine the absorption spectrum of this solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits maxima between 401 nm and 405 nm (phenol).

(4) Take 1 mL of Compound Iodine Glycerin in a glass-stoppered test tube, add 10 mL of ethanol (95) and mix. Then add 2 mL of sodium hydroxide TS, add 1 mL of a solution of cupric chloride in ethanol (95) (1 in 10) and shake: a blue color is observed (glycerin).

Assay (1) ***Iodine***—Measure the specific gravity of Compound Iodine Glycerin according to Method 2. Weigh accurately an amount, equivalent to about 7 mL of Compound Iodine Glycerin, add ethanol (95) to make exactly 200 mL and use this solution as the test solution. Separately, weigh accurately about 80 mg of

Iodine RS and about 0.17 g of 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 exactly 3 mL each of the test solution and the standard solution into 50-mL separatory funnel, to each add exactly 10 mL of a mixture of chloroform and hexane (2 : 1) and exactly 15 mL of water successively and shake immediately and vigorously, separate the chloroform-hexane layers [use the water layers in (2)] and filter through absorbent cotton. Determine the absorbances of the filtrates, A_T and A_S, for the test solution and the standard solution, respectively, at 512 nm as directed under the Ultraviolet-visible Spectrophotometry, using a 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***—Separate the water layers of the test solution and the standard solution obtained in (1), pipet exactly 10 mL of each of the water layers and to each, add 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) and shake immediately and vigorously. Separate the chloroform-hexane layers and filter through absorbent cotton. Determine the absorbances, A_T and A_S, for the test solution and the standard solution, respectively, at 512 nm as directed under the Ultraviolet-visible Spectrophotometry, using a mixture of chloroform and hexane (2 : 1) as the blank,

$$\text{Amount (mg) of iodine (KI)} = \text{amount (mg) of Potassium Iodine RS} \times \frac{A_T}{A_S}$$

(3) ***Total iodine***—Measure the specific gravity of Compound Iodine Glycerin according to Method 2 under Specific Gravity and Density. Weigh accurately an amount, equivalent to about 5 mL of Compound Iodine Glycerin and add water to make exactly 50 mL. Pipet exactly 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 until the color of iodine disappears and heat under a reflux condenser on a water-bath for 30 minutes. Wash the condenser with 10 mL of hot water and filter through a glass filter (G3). Wash the flask twice with 10 mL volumes of warm water and combine the filtrate and the washings. After cooing, 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, dissolve in water to make exactly 50 mL. Pipet exactly 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 exactly 4 mL each of the test solution and the standard solution into 30-mL separatory funnel and to each add 5 mL of water, 1 mL of diluted dilute hydrochlorlc acid (1 in 2), 1 mL of sodium nitrite TS and 10 mL of a mixture of chloroform and hexane (2 : 1) shake well immediately and proceed as directed in (2).

$$\text{Amount (mg) of total iodine (I)} = \text{amount (mg) of Potassium Iodine RS} \times \frac{A_T}{A_S} \times 0.7644$$

(4) ***Phenol***—Measure the specific gravity of Compound Iodine Glycerin according to Method 2 under Specific Gravity and Density. Weigh accurately an amount, equivalent to about 2 mL of Compound Iodine Glycerin, add 3 mL of 0.1 mol/L sodium thiosulfate VS, mix with shaking, add 2 mL of dilute hydrochloric acids, extract twice with 10 mL volumes of chloroform. Combine all of the chloroform extract and extract twice with 10 mL of 0.5 mol/L sodium hydroxide TS. Combine all of the water extracts, add water to make exactly 500 mL and use this solution as the test solution. Separately, dissolve about 0.5 g of Phenol RS, accurately weighed, in ethanol (95) to make exactly 100 mL, pipet exactly 2 mL of this solution, proceed in the same manner as the test solution and use this solution as the standard solution. Pipet exactly 3 mL each of the test solution and the standard solution, to each add 2 mL of dilute hydrochloric acid and place in a water-bath at 30 °C. Allow to stand for 10 minutes and add exactly 2 mL of a solution of sodium nitrite (1 in 100), shake and allow to stand at 30 °C for 60 minutes. Add dilute potassium hydroxide·ethanol TS to make exactly 25 mL. Determine the absorbances, A_T and A_S, of the test solution and the standard solution, respectively, at 403 nm as directed under Ultraviolet-visible Spectrophotometry, using the solution prepared in the same manner with 3 mL of water instead of the test solution as the blank.

$$\text{Amount (mg) of phenol } (C_6H_6O) = \text{amount (mg) of Phenol RS} \times \frac{A_T}{A_S} \times \frac{1}{50}$$

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light-resistant.

Iodine Tincture

Iodine Tincture contains not less than 5.7 w/v % and not more than 6.3 w/v % of Iodine (I: 126.90) and not less than 3.8 w/v % and not more than 4.2w/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 Tinctures, with the above in gradients. It may be prepared with an appropriate quantity of Ethanol or a mixture of Ethanol for Disinfection and Purified Water in place of 70vol % ethanol.

Description Iodine Tincture is dark red-brown liquid and has a characteristic odor.

Specific gravity— d_{20}^{20} : About 0.97.

Identificatien (1) Take a mixture of 1 mL of starch TS and 9 mL of water, and add 1 drop of Iodine Tincture: a dark blue-purple color develops.

(2) Evaporate 3 mL of Iodine Tincture to dryness on a water-bath and heat gently over a free flame: a white residue is produced which responds to the Qualitative Tests for potassium salt and iodide.

Alcohol Number Not less than 6.6 (Method 2). Perform the pretreatment (ii) in the Method 1.

Assay (1) ***Iodine***—Pipet exactly 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***—Pipet exactly 5 mL of Iodine Tincture into 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 red-purple color disappears from the chloroform layer, with agitating the mixture vigorously and continuously. After the chloroform layer has been decolorized, allow the mixture to stand for 5 minutes. If the color reappears, the mixture should be titrated further with 0.05 mol/L potassium iodate VS. Calculate the amount (mg) of potassium iodide from the volume (*a* mL) of 0.05 mol/L potassium iodate VS used as above and the volume (*b* mL) of 0.1 mol/L sodium thiosulfate VS used in the titration under the Assay (1).

Amount (mg) of potassium iodide (KI)
$$= 16.600 \times (a - \frac{b}{2})$$

Containers and Storage ***Containers*—**Tight containers.

Dilute Iodine Tincture

Dilute Iodine Tincture contains not less than 2.8 w/v % and not more than 3.2 w/v % of iodine (I: 126.90). and not less than 1.9 w/v % and not more than 2.1 w/v % of potassium iodide (KI: 166.00).

Method of Preparation

Iodine	30 g
Potassium Iodide	20 g
70vol % Ethanol	a sufficient quantity

To make 1000 mL

Prepare as directed under Medicated Spirits, with the above ingredients. It may be prepared with an appropriate quantity of Ethanol or a mixture of Ethanol for Disinfection and Purified Water in place of 70 % ethanol. It may also be prepared by adding 70 % ethanol to 500 mL of iodine tincture to make 1000 mL.

Description Dilute Iodine Tincture is dark red-brown liquid. Dilute Iodine Tincture has a characteristic odor.

Specific gravity— d_{20}^{20} : About 0.93.

Identification (1) Take a mixture of 1 mL of starch TS and 9 mL of water and add 1 drop of Dilute Iodine Tincture: a dark blue-purple color develops.

(2) Evaporate 3 mL of Diluted Iodine Tincture to dryness on a water-bath and heat gently
over a free flame: a white residue is produced which responds to the Qualitative Tests for potassium salt and iodide

Alcohol Number Not less than 6.7 (Method 2). Perform the pretreatment (ii) in the Method 1

Assay (1) ***Iodine***—Pipet exactly 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) ***Potassiun iodide***—Pipet exactly 10 mL of Dilute Iodine Tincture into 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 red-purple color in the chloroform layer disappears while agitating vigorously and continuously. After the chloroform layer has been decolorized, allow the mixture to stand for 5 minutes. If the color reappears, the mixture should be titrated further with 0.05 mol/L potassium iodate VS. Calculate the amount (mg) of potassium iodide from the volume (*a* mL) of 0.05 mol/L potassium iodateVS consumed as above and the volume (*b* mL) of 0.1 mol/L sodium thiosulfate VS consumed in the titration under the Assay (1).

Amount (mg) of potassium iodide (KI)

$$= 16.600 \times (a - \frac{b}{2})$$

Containers and Storage ***Containers*—**Tight containers.

Morphine and Atropine Injection

Morphine and Atropine Injection is an aqueous solution for injection. Morphine and Atropine Injection contains not less than 0.91 % and not more than 1.09 % of morphine hydrochloride hydrate ($C_{17}H_{19}NO_3 \cdot HCl \cdot 3H_2O$: 375.84) and not less than 0.027 % and not more than 0.033 % of atropine sulfate hydrate [$(C_{17}H_{23}NO_3)_2 \cdot H_2SO_4 \cdot H_2O$: 694.84]

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 direction under Injections, with the above ingredients.

Description Morphine and Atropine Injection is clear, colorless liquid.
Morphine and Atropine Injection is slowly affected by light.
pH—2.5 ~ 5.0.

Identification To 2 mL of Morphine and Atropine Injection, add 2 mL of ammonia TS and extract with 10 mL of ether. Filter the extract with a filter paper, evaporate the filtrate on a water-bath to dryness, dissolve the residue in 1 mL of dehydrated ethanol, and use this solution as the test solution. Separately, dissolve 0.1 g of Morphine Hydrochloride Hydrate RS in 10 mL of water, perform with 2 mL of this solution the same procedure as used for preparation of the test solution, and use the solution so obtained as the standard solution (1). Separately, dissolve 3 mg of Atropine Sulfate Hydrate RS in 10 mL of water, perform with 2 mL of this solution the same procedure as used for preparation of the test solution, and use the solution so obtained as the standard solution (2). Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 10 μL each of the test solution and standard solutions (1) and (2) on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of methanol and ammonia solution (200:3) to a distance of about 10 cm, and air-dry the plate. Spray evenly Dragendor's TS on the plate: the two spots obtained from the test solution show the same color tone and the same R_f value with either spot of orange color obtained from the standard solution (1) or the standard solution (2) (morphine and atropine).

Sterility Test It meets the requirement.

Foreign Insoluble Matter Test It meets the requirement.

Insoluble Particulate Matter Test for Injections It meets the requirement.

Determination of Volume of Injection in Containers It meets the requirement.

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, add exactly 10 mL of the internal standard solution to dissolve, then add water to make 50 mL and use this solution as the standard solution. Perform the test with 20 μL of the test solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions and calculate the ratio, Q_T and Q_S of the peak area of morphine to that of the internal standard for the test solution and the standard solution, respectively.

$$\text{Amount (mg) of morphine hydrochloride hydrate } (C_{17}H_{19}NO_3 \cdot HCl \cdot 3H_2O) = \text{amount (mg) of Morphine Hydrochloride Hydrate RS, calculated on the anhydrous basis} \times \frac{Q_T}{Q_S} \times 1.1679$$

Internal standard solution—A solution of etilefrine hydrochloride (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 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: Dissolve 1.0 g of sodium lauryl sulfate in 500 mL of diluted phosphoric acid (1 in 1000), and adjust the pH with sodium hydroxide TS to 3.0. 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

System Performance: When the procedure is run with 20 μL of the standard solution under the above operating conditions, morphine and the internal standard are eluted in this order with resolution between the two peaks being not less than 3.

System repeatability: When the test is repeated 6 times with 20 μL each of the standard solution under the above operating conditions, the relative standard deviation of the ratios of the peak area of morphine to that of the internal standard is not more than 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 this 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, and calculate the ratios, Q_T and Q_S of the peak areas of atropine to that of the internal standard.

Amount (mg) of atropine sulfate hydrate $[(C_{17}H_{23}NO_3)_2 \cdot H_2SO_4 \cdot H_2O]$
= amount (mg) of Atropine Sulfate Hydrate RS,
calculated 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

Column, column temperature, and mobile phase: Proceed as directed in the operating conditions in the Assay (1).

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: When the procedure is run with 20 μL of the test solution under the above operating conditions, morphine, the internal standard and atropine are eluted in this order, and the resolution between morphine and the internal standard is not less than 3.

System repeatability: When the test is repeated 6 times with 20 μL each of the standard solution under the above operating conditions, the relative standard deviation of the ratios of the peak area of atropine to that of the internal standard is not more than 1.0 %.

Containers and Storage ***Containers*—**Hermetic containers.

***Storage*—**Light-resistant.

Phenolated Water

Phenolated Water contains not less than 1.8 w/v % and not more than 2.3 w/v % of phenol (C_6H_6O: 94.11).

Method of Preparation

Liquefied Phenol	22 mL
Water or Purified Water	a sufficient quantity
	To make 1000mL

Mix the above ingredients.

Description Phenolated Water is clear, colorless liquid, and has the odor of phenol.

Identification (1) Take 10 mL of Phenolated Water, and add 1 drop of iron (III) chloride TS: a blue-purple color is observed.

(2) Proceed with 5 mL of a solution of Phenolated Water for Disinfection (1 in 200) as directed in the Identification (2) under Phenol for Disinfection.

Assay Take exactly 2 mL of Phenolated Water into an iodine flask, add 25 mL of water, then add exactly 40 mL of 0.05 mol/L bromine VS and 5 mL of hydrochloric acid and proceed 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

Containers and Storage ***Containers*—**Tight containers.

Polyethylen Glycol Ointment

Macrogol Ointment

Method of Preparation

Polyethylene Glycol 4000	500 g
Polyethylene Glycol 400	500 g
	Total 1000 g

Melt Polyethylene Glycol 4000 and Polyethylene Glycol 400 by warming on a water-bath to 65 °C. Mix well and allow to cool and stir until congealed. If necessary to get a proper viscosity, adjust amounts of the Polyethylene Glycol 400 and Polyethylene Glycol 4000 up to 100 g with total amount of 1000 g.

Description Polyethylene Glycol Ointment is a white, and has a slight, characteristic odor.

Identification Take 50 mg of Polyethylene Glycol Ointment, add 5 mL of dilute hydrochloric acid, add 1 mL of barium chloride, mix with shaking and filter if necessary. Add 1 mL of phosphomolybdic acid (1 in 10) to the filtrate: yellowish green precipitate is produced.

Containers and Storage ***Containers*—**Tight containers.

Ringer's Solution

Ringer's Solution is an aqueous solution for injection. Ringer's Solution contains not less than 0.53 % and not more than 0.58 % of chlorine [as (Cl: 35.45)] and not less than 0.030 % and not more than 0.036 % of calcium chloride ($CaCl_2 \cdot 2H_2O$: 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 Injection, with the above ingredients. No preservative may be added.

Description Ringer's solution is a clear, colorless liquid, and has a saline taste.

Identification (1) Ringer's Solution responds the Qualitative Tests for sodium salt and chloride.

(2) Evaporate 10 mL of Ringer's Solution to 5 mL: the solution responds to the Qualitative Tests for potassium salt and calcium salt.

pH 5.0 ~ 7.5.

Purity (1) ***Heavy metals***—Evaporate 100 mL of Ringer's Solution to about 40 mL on a water bath, add 2 mL of dilute acetic acid and water to make 50 mL and perform the test. Prepare the control solution as follows: to 3.0 mL of standard lead solution, add 2 mL of dilute acetic and add water to make 50 mL.(not more than 0.3 ppm).

(2) ***Arsenic***—Perform the test with 20 mL of Ringer's Solution and as the test solution (not more than 0.1 ppm).

Sterility Test It meets the requirement.

Bacterial Endotoxins Less than 0.50 EU/mL of Ringer's Solution.

Foreign Insoluble Matter Test It meets the requirement.

Insoluble Particulate Matter Test for Injections It meets the requirement.

Determination of Volume of Injection in Containers It meets the requirement.

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 (indicator: 3drops of sodium fluorescein 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 50 mg of NN indicator and titrate immediately with 0.01 mol/L disodium ethylenediaminetetraacetate VS until the color of the solution changes from red-purple to blue.

Each mL of 0.01 mol/L
disodium ethylenediaminetetraacetate VS
= 1.4701mg of $CaCl_2 \cdot 2H_2O$

Containers and Storage ***Containers—***Hermetic containers. Plastic containers for aqueous infusions may be used.

Salicylic Acid Adhesive Plaster

Method of Preparation

Salicylic Acid, finely powdered	500 g
Adhesive plaster base	a sufficient quantity
To make	1000 g

Adhesive plaster consists of a mixture of the following ingredient with carefully selected rubber, resin, zinc oxide and other substances. Salicylic Acid Adhesive Plaster has adhesive properties. Salicylic Acid Adhesive Plaster spreads evenly on a fabric.

Description The surface of Salicylic Acid Adhesive Plaster is white and adheres well to the skin.

Containers and Storage ***Containers—***Well-closed containers.

Storage—Light-resistant.

Salicylic Acid Spirit

Salicylic acid spirit contains not less tha 2.7 % and not more than 3.3 % 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 Medicated Spirits, with the above ingredients.

Description Salicylic Acid Spirit is clear, colorless

liquid.

Specific gravity— d_{20}^{20} : About 0.86.

Identification The solution obtained in the Assay is red-purple. Determine the absorption spectrum of the solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits a maximum between 520 nm and 535 nm (salicylic acid).

Alcohol Number Not less than 8.8 (Method 2).

Assay Take 10.0 mL of Salicylic Acid Spirit, add 10 mL of ethanol (95) and water to make exactly 100 mL. Pipet 3.0 mL of this solution, add with hydrochloric acid-potassium chloride buffer solution, pH 2.0, to make exactly 100 mL and use this solution as the test solution. Separately, dissolve 0.3 g of Salicylic Acid RS, previously dried in a desiccator (silica gel) for 3 hours and accurately weighted, in 10 mL of ethanol (95) and add 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 of the test solution and the standard solution, add 5 mL of a solution of iron (III) nitrate nonahydrate (1 in 200) and add hydrochloric acid-potassium chloride buffer solution, pH 2.0, to make exactly 25 mL. 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 Spectrophotometry, respectively, using a blank solution prepared in the same manner with water instead of the test solution.

Amount (mg) of salicylic acid ($C_7H_6O_3$)
= amount (mg) of Salicylic Acid RS $\times \frac{A_T}{A_S}$

Containers and Storage ***Containers*—**Tight containers.

Silver Nitrate Ophthalmic Solution

Silver Nitrate Ophthalmic Solution is an aqueous eye lotion containing not less than 0.95w/v % and not more than 1.05w/v % of silver nitrate ($AgNO_3$: 169.87).

Method of Preparation

Silver Nitrate	10g
Sterile Purified Water	a sufficient quantity
To make	1000 mL

Prepare as directed under Ophthalmic Solution, with the above ingredients.

Description Silver Nitrate Ophthalmic Solution is clear, colorless liquid .

Identification Silver Nitrate Ophthalmic Solution responds to the Qualitative Tests for silver salt and for nitrate.

Sterility Test It meets the requirement.

Foreign Insoluble Matter Test It meets the requirement.

Insoluble Particulate Matter Test for Ophthalmic Solutions It meets the requirement.

Assay Weigh accurately 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: 2mL of iron (III) ammonium sulfate TS).

Each mL of 0.1 mol/L ammonium thiocyanate VS
= 16.987 mg of $AgNO_3$

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light-resistant.

Silver Protein Solution

Silver Protein Solution contains not less than 0.22 % and not more than 0.26 % of silver (Ag: 107.87).

Method of Preparation

Silver Protein	30 g
Glycerin	100 mL
Mentha Water	a sufficient quantity
To make	1000 mL

Dissolve and mix the above ingredients.

Description Silver Protein Solution is clear, brown liquid, and has the odor of mentha oil.

Identification (1) Take 1 mL of Silver Protein Solution, add 10 mL of ethanol (95), mix and add 2 mL of sodium hydroxide TS. Add immediately 1 mL of a solution of cupric chloride in ethanol (95) (1 in 10), shake and filter: the filtrate is blue (glycerin).

(2) Take 3 mL of Silver Protein Solution, add water to make 10 mL, add 2 mL of dilute hydrochloric acid, shake frequently for 5 minutes and filter. Add 5 mL of a solution of sodium hydroxide (1in 10) to the filtrate and add 2 mL of diluted cupric sulfate (2 in 25): a purple color is observed (silver protein).

(3) Take 5 mL of the test solution obtained in (2) and add iron (III) chloride TS dropwise: a brown precipitate is produced (silver protein).

(4) Place 3 mL of Silver Protein Solution in a crucible, heat cautiously and evaporate almost to dryness. Then ignite gradually to ash, dissolve the residue in 1 mL of nitric acid by warming and add 10 mL of water: the solution responds to the Qualitative Tests (1) for silver salt.

Assay Pipet exactly 25 mL of Silver Protein Solution into a 250 mL Kjeldahl flask and heat cautiously until a white gas of glycerin is evolved. After cooling, add 25 mL of sulfuric acid, cover the flask with a small funnel and heat gently for 5 minutes. After cooling, drop gradually 5 mL of nitric acid, heat with occasional shaking in a water-bath for 45 minutes and cool. Add 2 mL of nitric acid, boil gently and repeat this operation until the solution becomes colorless upon cooling. Transfer cautiously the cooled content in the flask into a 500-mL Erlenmyer flask with 250 mL of water. Boil gently for 5 minutes, cool and titrate with 0.1 mol/L ammonium thiocyanate VS (indicator: 3mL of iron (III) ammonium sulfate TS).

Each mL of 0.1mol/L ammonium thiocyanate VS
= 10.787 mg of Ag

Containers and Storage ***Containers*****—**Tight containers.

Storage**—**Light-resistant.

Simple Ointment

Method of Preparation

Yellow Beeswax	330 g
Fixed oil	a sufficient quantity
To make	1000 g

Prepare as directed under Ointments, with the above ingredients.

Description Simple Ointment is yellow and has a slight, characteristic odor.

Containers and Storage ***Containers*****—**Tight containers.

Simple Syrup

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 direction under Syrup, with the above materials.

Description Simple Syrup is a colorless to pale yellow, viscous liquid, is odorless, and has a sweet taste.

Identification (1) Evaporate Simple Syrup on a water-bath to dryness. 1 g of residue so obtained, when ignited, melted to swell and decomposes, emitting an odor of caramel, to bulky charcoal.

(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} : 1.310 ~ 1.325.

Purity (1) ***Artificial sweetening agent*****—**Take 100 mL of Simple Syrup, add 100 mL of water, shake, acidify 50 mL volume of the solution with dilute sulfuric acid and make another 50 mL volume alkaline with sodium hydroxide TS. To each volume, add 100 mL of ether, shake, separate the ether layer, combine the two ether layers and evaporate the ether extract on a water-bath to dryness: the residue has no sweet taste.

(2) ***Salicylic acid*****—**Take the residue obtained in (1) and add 2 to 3drops of dilute iron (III) chloride TS: no purple color is observed.

Containers and Storage ***Containers*****—**Tight containers.

White Ointment

Method of Preparation

White Beeswax	50g
Sorbitan Sesquioleate	20g
White Petrolatum	a sufficient quantity
To make	1000g

Prepare as directed under Ointments, with the above materials.

Description White ointment is white, and has a slight, characteristic odor.

Containers and Storage ***Containers*****—**Tight containers.

4) Excipients

Acacia

Acacia is the secretion obtained from the stems and branches of *Acacia senegal* Willdenow or other species of the same genus (Leguminosae).

Description Acacia is colorless or pale yellow-brown, translucent or somewhat opaque spheroidal tears, or angular fragments with numerous fissures on the surface, is very brittle and the fractured surface is glassy and occasionally irridescent.
Acacia is odorless, tasteless, but produces a mucilaginous sensation on a tongue.
One g of pulverized Acacia dissolves almost completely in 2.0 mL of water and the solution is acidic.
Acacia is practically insoluble in ethanol.

Identification To 1 g of powdered Acacia, add 25 mL of water and 1 mL of sulfuric acid and heat with a reflux condenser in a boiling water bath for 60 minutes. After cooling, add gently 2.0 g of anhydrous sodium carbonate. To 1 mL of this solution, add 9 mL of methanol, shake, centrifuge and use the clear supernatant liquid 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 standard solution (1). Proceed with L-arabinose and L-rhamnose hydrate in the same manner and use these solutions as standard solution (2) and standard solution (3), respectively. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 10 μL each of the test solution, standard solution (1), standard solution (2) and standard solution (3) on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of acetone and water (9 : 1) to a distance of about 10 cm and air-dry the plate. Spray evenly 1-naphthol-sulfuric acid TS on the plate and heat at 105 °C for 5 minutes: the 3 spots obtained from the test solution show the same color and R_f value as the spots of D-galactose, L-arabinose and L-rhamnose from the standard solutions.

Purity (1) ***Insoluble residue***—To 5.0 g of pulverized Acacia, add 100 mL of water and 10 mL of dilute hydrochloric acid and dissolve by gentle boiling for 15 minutes with swirling. Filter the warm mixture through a tared glass filter (G3), wash the residue thoroughly with hot water and dry at 105 °C for 5 hours: the residue is not more than 10.0 mg.

(2) ***Tannin-bearing gums***—Take 10 mL of a solution of Acacia (1 in 50) and add 3 drops of ferric chloride TS: no dark green color is produced.

(3) ***Heavy metals***—Proceed with 1.0 g of Acacia according to Method 2 and perform the test. Prepare the control solution with 4.0 m L of standard lead solution (not more than 40 ppm).

(4) ***Glucose***—Use the test solution as obtained in the Identification as the test solution. Separately, 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 as directed under Thin-layer Chromatography in the dentification: any spot at the R_f value corresponding to glucose from the standard solution does not appear from the test solution.

(5) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat and place 10 mg to 300 mg of Acacia on top. Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. Place the boat inside the furnace and heat to about 900 °C with a current of air or oxygen at 0.5 L/minute to 1 L/minute. Elute the mercury and sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions
Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 ng to 200 ng.

Additive—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.

(6) ***Cadmium***—Weigh accurately 5.0 g of Acacia 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 mL 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 supplement, dry and

continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 1.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Cadmium hollow cathode lamp
Wavelength: 228.8 nm

(7) ***Lead***—Weigh accurately 5.0 g of Acacia 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 0.5 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 solution as directed under Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 1.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(8) ***Arsenic***—Proceed with 0.25 g of Acacia according to Method 3 and perform the test (not more than 4 ppm).

(9) ***Starch and dextrin***—Dissolve 1 g of Acacia in 50 mL of water, boil, cool and add a few drops of iodine TS: no blue or red color is produced.

Loss on Drying Not more than 17.0 % (6 hours).

Ash Not more than 4.0 %.

Acid-insoluble Ash Not more than 0.5 %.

Microbial Limit The total aerobic microbial count is not more than 1000 CFU/g, the total combined yeasts/mould count is not more than 100 CFU/g and *Escherichia coli*, *Salmonella* species, *Staphylococcus aureus* and *Pseudomonas aeruginosa* are not observed.

Acetic Acid

Acetic Acid contains not less than 30.0 w/v % and not more than 32.0 w/v % of acetic acid ($C_2H_4O_2$: 60.05).

Description Acetic Acid is a clear, colorless liquid and has a pungent, characteristic odor and an acid taste. Acetic Acid is miscible with water, with ethanol or with glycerin.

Specific gravity—d_{20}^{20} : About 1.04.

Identification Acetic Acid changes blue litmus paper to red and responds to the Qualitative Tests for acetate.

Purity (1) ***Chloride, Sulfate and Potassium permanganate-reducing substances***—To 20 mL of Acetic Acid, add 40 mL of water and use this solution as the test solution. Proceed as directed in the Purity (1), (2) and (4) under Glacial Acetic Acid.

(2) ***Heavy metals***—Evaporate 10 mL of Acetic Acid Solution on a water-bath to dryness and to the residue, add 2 mL of dilute acetic acid and water to make 50 mL. Perform the test. Prepare the control solution with 3.0 mL of standard lead solution by adding 2 mL of dilute acetic acid and water to make 50 mL (not more than 5 ppm).

(3) ***Arsenic***—Proceed with 0.5 g of Acetic Acid according to Method 3 and perform the test (not more than 4 ppm).

(4) ***Non-volatile residue***—Proceed with 30 mL of Acetic Acid as directed in the Purity (5) under Glacial Acetic Acid.

Assay Take 5.0 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 $C_2H_4O_2$

Containers and Storage ***Containers***—Tight containers.

Glacial Acetic Acid

$C_2H_4O_2$: 60.05

Acetic acid [*64-19-7*]

Glacial Acetic Acid contains not less than 99.0 % and not more than 101.0 % of glacial acetic acid ($C_2H_4O_2$).

Description Glacial Acetic Acid is a clear, colorless, volatile liquid, or colorless or white, crystalline mass and has a pungent and characteristic odor.
Acetic acid is miscible with water, with ethanol or with ether.

Boiling point—About 118 °C.

Specific gravity— d_{20}^{20} : About 1.049.

Identification A solution of Glacial Acetic Acid (1 in 3) changes blue litmus paper to red and responds to the Qualitative Tests for acetate.

Congealing point Not below 14.5 °C.

Purity (1) ***Chloride***—Take 10 mL of Glacial Acetic Acid, add water to make 100 mL and use this solution as the test solution. To 10 mL of the test solution, add 5 drops of silver nitrate TS: no opalescence is produced.

(2) ***Sulfate***—Take 10 mL of the test solution obtained in (1) and add 1 mL of barium chloride TS: 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 and perform the test. Prepare the control solution with 1.0 mL of standard lead solution by adding 2.0 mL of dilute acetic acid and water to make 50 mL (not more than 5 ppm).

(4) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat and place 10 mg to 300 mg of Glacial Acetic Acid on top. Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. Place the boat inside the furnace and heat to about 900 °C with a current of air or oxygen at 0.5 L/minute to 1 L/minute. Elute the mercury and sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 ng to 200 ng.

Additive—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 Glacial Acetic Acid 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 0.25 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 0.5 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(6) ***Arsenic***—Proceed with 1.54 g of Glacial Acetic Acid according to Method 1 and perform the test (not more than 1.3 ppm).

(7) ***Potassium permanganate-reducing substances***—Take 20 mL of the test solution obtained in (1) and add 0.10 mL of 0.02 mol/L potassium permanganate VS: the red color does not disappear within 30 minutes.

(8) ***Non-volatile residue***—Evaporate 10 mL of Glacial Acetic Acid on a water-bath to dryness and dry

at 105 °C for 1 hour: the residue is not more than 1.0 mg.

Assay Place 10 mL of water in a glass-stoppered flask and weigh accurately. Add about 1.5 g of Glacial Acetic Acid, weigh accurately again, then 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 $C_2H_4O_2$

Containers and Storage ***Containers*****—**Tight containers.

Agar

Agar is the solid residue obtained by freeze-drying of a mucilage derived from *Gelidium amansii* Lamouroux, other species of the same genus (Gelidiaceae), or other red algae (Rhodophyta).

Description Agar is white, semi-translucent rectangular column, string or flakes.
Rectangular column of Agar is about 26 cm in length, 4 cm^2 in cross section and a string of Agar is about 35 cm in length and about 3 mm in width and flakes of Agar are about 3 mm in length, externally, with wrinkles and somewhat lustrous, light and pliable.
Agar is practically insoluble in organic solvents.
A boiling solution of Agar (1 in 100) is neutral.
Agar is odorless, tasteless and mucilagenous.

Identification (1) Take a fragment of Agar and add drop-wise iodine TS: a dark blue to reddish purple 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 refill the water lost by evaporation: the solution is clear. Cool the solution between 30 °C and 39 °C : the solution forms a firm, resilient gel, which does not melt below 85 °C.

Purity (1) ***Sulfuric acid***—Dissolve 1.0 g of Agar in 100 mL of water by boiling: the solution is not acidic.

(2) ***Sulfurous acid and starch***—Take 5 mL of the solution obtained in (1) and add 2 drops of iodine TS: the solution does not decolorize at once and does not show a blue color.

(3) ***Insoluble matter***—Take 7.5 g of Agar, add 500 mL of water, boil for 15 minutes and add water to make exactly 500 mL. Take exactly 100 mL of the solution, add 100 mL of hot water, heat to boiling, filter while hot through a tared glass filter (G3), wash the residue with a small amount of hot water and dry the residue at 105 °C for 4 hours: the residue is not more than 15.0 mg.

(4) ***Water absorption***—Take 5.0 g of Agar, add water to make 100 mL, shake well, allow to stand at 25 °C for 24 hours and filter through moistened glass wool in a 100-mL graduated cylinder: the volume of the filtrate is not more than 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 standard lead solution (not more than 40 ppm).

(6) ***Arsenic***—Proceed with 0.67 g of Agar according to Method 2 and perform the test (not more than 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 Not more than 22.0 % (6 hours).

Ash Not more than 4.5 %.

Acid-insoluble Ash Not more than 0.5 %

Microbial Limit The total aerobic microbial count is not more than 1000 CFU/g, the total combined yeasts/mould count is not more than 100 CFU/g and *Escherichia coli*, *Salmonella* species, *Staphylococcus aureus* and *Pseudomonas aeruginosa* are not observed.

Aluminum Monostearate

Aluminum Monostearate is mainly aluminum compounds of stearic acid ($C_{18}H_{36}O_2$: 284.48) and palmitic acid ($C_{16}H_{32}O_2$: 256.42). Aluminum Monostearate, when dried, contains not less than 7.2 % and not more than 8.9 % of aluminum (Al: 26.98).

Description Aluminum Monostearate is a white to pale yellow powder, is odorless or has a slight, characteristic odor.
Aluminum Monostearate is practically insoluble in water, in ethanol or in ether.

Identification (1) Heat 3 g of Aluminum Monostearate with 30 mL of hydrochloric acid on a water-bath with occasional shaking for 10 minutes. After cooling, shake the mixture vigorously with 50 mL of water and 30 mL of ether for 3 minutes and allow to stand. To the separated aqueous layer, add sodium hydroxide TS until the solution becomes slightly turbid and filter: the filtrate responds to the Qualitative Tests for aluminum salt.

(2) Wash the ether layer separated in (1) twice with 20 mL volumes of water and evaporate the ether layer on a water-bath: the residue melts at above 54 °C (Method 2).

Acid Value for Fatty Acid 193 ~ 210. Weigh accu-

rately about 1 g of fatty acid obtained in the Identification (2), transfer a 250 mL glass-stoppered flask, add 100 mL of a mixture of ether and ethanol (2 : 1), warm to dissolve, add several drops of phenolphthalein TS and proceed as directed in the Acid value under the Fats and Fatty Oils.

Purity (1) ***Free fatty acid***—Mix 1.0 g of Aluminum Monostearate with about 50 mL of a mixture of neutralized ethanol and ether (1 : 1), filter through dry filter paper, wash the vessel and the filter paper with a small amount of a mixture of neutralized ethanol and ether (1 : 1), combine the filtrate and the washings and add 2.1 mL of 0.1 mol/L potassium hydroxide VS: a red color develops.

(2) ***Water-soluble salts***—Heat 2.0 g of Aluminum Monostearate with 80 mL of water in a loosely stoppered Erlenmeyer flask 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 not more than 10.0 mg.

(3) ***Heavy metals***—Heat 1.0 g of Aluminum Monostearate over a small flame with caution at the beginning, continue the heating and gradually raising the temperature to ash. After cooling, add 10 mL of diluted hydrochloric acid (1 in 2), evaporate on a water-bath and boil the residue with 20 mL of water for 1 minute. Cool, filter, wash the residue with water, combine the filtrate and the washing and add 2 mL of dilute acetic acid and water to make 50 mL. Perform the test. Evaporate 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 standard lead solution, dilute with water to make 50 mL and use this solution as the control solution (not more than 50 ppm).

(4) ***Arsenic***—Mix 1.0 g of Aluminum Monostearate with 2 g of magnesium nitrate, ignite over a small flame, moisten the residue after cooling with 0.5 mL of nitric acid and heat. Heat again the residue with 10 mL of dilute sulfuric acid until white fumes evolve, add water to make 5 mL and perform the test (not more than 2 ppm).

Loss on Drying Not more than 3.0 % (1 g, 105 °C, 3 hours).

Assay Weigh accurately about 1 g of Aluminum Monostearate, previously dried, ignite gently to ash and cool. Add drop-wise 0.5 mL of nitric acid, evaporate on a water-bath by heating and then heat strongly between 900 °C and 1100 °C to a constant weight. After cooling, weigh rapidly the ignited residue and designate the mass as aluminum oxide (Al_2O_3: 101.96).

Amount (mg) of aluminum (Al)
= amount (mg) of aluminum oxide (Al_2O_3) × 0.5293

Containers and Storage ***Containers*—**Well-closed containers.

Aluminum Potassium Sulfate Hydrate

Alum
Aluminum Potassium Sulfate

$AlK(SO_4)_2 \cdot 12H_2O$: 474.39

Aluminum Potassium Sulfate Hydrate contains not less than 99.5 % and not more than 101.0 % of aluminum potassium sulfate hydrate [$AlK(SO_4)_2 \cdot 12H_2O$].

Description Aluminum Potassium Sulfate Hydrate appears as colorless or white crystals or powder, is odorless and has a slightly sweet, strongly astringent taste.
Aluminum Potassium Sulfate Hydrate is freely soluble in water and practically insoluble in ethanol or ether.
A solution of Aluminum Potassium Sulfate Hydrate (1 in 20) is acid.

Identification A solution of Aluminum Potassium Sulfate Hydrate (1 in 10) responds to the Qualitative Tests for aluminum salt, to the Qualitative Tests (1), (3) and (4) for potassium salt and to the Qualitative Tests (1) and (3) for sulfate.

Purity (1) ***Clarity and color of solution and water-insoluble substances***—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. After cooling, filter through a glass filter, wash the insoluble residue with 100 mL of water and dry at 105 °C for 2 hours: not more than 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 standard lead solution (not more than 20 ppm).

(3) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat and place 10 mg to 300 mg of Aluminum Potassium Sulfate Hydrate on top. Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. Place the boat inside the furnace and heat to about 900 °C with a current of air or oxygen at 0.5 L/minute to 1 L/minute. Elute the mercury and sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 ng to 200 ng.

Additive—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 test specimen is completely dissolved 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 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 solution (2 in 100) and shake. Extract this solution with two 20 mL volumes of chloroform and evaporate the extract to dryness in 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 standard lead 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 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 standard iron solution (not more than 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 in 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 solution. To the test solution, the control solution and the blank 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). Add 0.2 g of hydroxylamine chloride to each solution, 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 more than that of the control solution (not more than 30 ppm).

Standard solution—Dilute selenium standard solution with water to make 3 ppm.

2,3-Diaminonaphthalene—Dissolve 0.1 g of 2,3-diaminonaphthalene and 0.5 g of hydroxylamine chloride in 0.1 mol/L hydrochloric acid to make 100 mL.

(7) ***Arsenic***—Prepare the test solution with 0.6 g of Aluminum Potassium Sulfate Hydrate, according to Method 1 and perform the test (not more than 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 (1 in 4) and 10 mL of disodium ethylenediaminetetraacetate (1 in 40) and shake. 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, determine the potential using a fluoride electrode and determine the amount of fluoride from the calibration curve: not more than 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 1 L (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) and 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 each and use these solutions as the standard solutions. Pipet 50 mL of each standard solution into polyethylene containers. 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 exactly 20 mL of this solution, add exactly 30 mL of 0.05 mol/L disodium ethylenediaminetetraacetate VS and 20 mL of acetic acid-ammonium acetate buffer solution, pH 4.8, boil for 5 minutes and cool. Add 55 mL of ethanol and titrate with 0.05 mol/L zinc acetate VS (indicator: 2 mL of dithizone TS), until the color of the solution changes from light dark green to pale red. Perform a blank determination and make any necessary correction.

Each mL of 0.05 mol/L
disodium ethylenediaminetetraacetate VS
= 23.719 mg of $AlK(SO_4)_2 \cdot 12H_2O$

Containers and Storage ***Containers*—**Tight containers.

Dried Aluminum Potassium Sulfate

Burnt Alum
Anhydrous Aluminum Potassium Sulfate
Aluminum Potassium Sulfate $AlK(SO_4)_2$: 258.21

Dried Aluminum Potassium Sulfate, when dried, contains not less than 98.0 % and not more than 101.0 % of aluminum potassium sulfate [$AlK(SO_4)_2$].

Description Dried Aluminum Potassium Sulfate is a white mass or white powder, is odorless and has a slightly sweet, astringent taste.
Dried Aluminum Potassium Sulfate is freely soluble in hot water and practically insoluble in ethanol.
Dried Aluminum Potassium Sulfate dissolves slowly in water.

Identification A solution of Dried Aluminum Potassium Sulfate (1 in 20) responds to the Qualitative Tests for aluminum salt, to the Qualitative Tests (1), (3) and (4) for potassium salt and to the Qualitative Tests (1) and (3) for sulfate.

Purity (1) ***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 matter with 100 mL of water and dry with the glass filter at 105 °C for 2 hours: not more than 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 (G4), wash with 50 mL of water and dry at 105 °C for 2 hours: the residue is not more than 50 mg.

(3) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat and place 10 mg to 300 mg of Dried Aluminum Potassium Sulfate on top. Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. Place the boat inside the furnace and heat to about 900 °C with a current of air or oxygen at 0.5 L/minute to 1 L/minute. Elute the mercury and sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions
Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 ng to 200 ng.

Additive—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 test specimen is completely

dissolved 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 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 solution (2 in 100) and shake. Extract this solution with two 20 mL volumes of chloroform and evaporate the extract to dryness in 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 standard lead 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 5.0 ppm).

Gas: Acetylene or hydrogen – Air
Lamp: 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, if necessary. Add 2 mL of dilute acetic acid and water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution with 2.0 mL of standard lead solution, 2 mL of dilute acetic acid and water to make 50 mL (not more than 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 in 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 solution. To the test solution, the control solution and the blank 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). Add 0.2 g of hydroxylamine chloride to each solution, 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 more than that of the control solution (not more than 30 ppm).

Standard solution—Dilute selenium standard solution with water to make 3 ppm.

2,3-Diaminonaphthalene—Dissolve 0.1 g of 2,3-diaminonaphthalene and 0.5 g of hydroxylamine chloride 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 standard iron solution (not more than 37 ppm).

(8) ***Arsenic***—Prepare the test solution with 0.40 g of Dried Aluminum Potassium Sulfate according to Method 1 and perform the test (not more than 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 (1 in 4) and 10 mL of disodium ethylenediaminetetraacetate (1 in 40) and shake. 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, determine the potential using a fluoride electrode and determine the amount of fluoride from the calibration curve: not more than 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 1000 mL 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 the standard 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) and 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 each and use these solutions as the standard solutions. Pipet 50 mL of each standard solution into polyethylene containers. Determine the potential using a fluoride electrode and plot a calibration curve with the log values of the fluoride concentrations.

Loss on Drying Not more than 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 and 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
disodium ethylenediaminetetraacetate VS
= 12.910 mg of $AlK(SO_4)_2$

Containers and Storage ***Containers*—**Tight containers.

Apricot Kernel Water

Apricot Kernel Water contains not less than 0.09 % and not more than 0.11 % of hydrogen cyanide (HCN: 27.03).

Method of Preparation Prepare by one of the following methods.

(1) Take Apricot Kernels, previously crushed and pressed to remove fixed oils as much as possible, add a suitable amount of Water or Purified Water, and carry out steam distillation. Determine the amount of hydrogen cyanide in the distillate by the method as directed in the Assay, and carry on the distillation until the content of hydrogen cyanide in the distillate is about 0.14 %. To the distillate, add ethanol of about 1/3 of the volume of the distillate, and dilute with 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 amount of hydrogen cyanide in the solution as directed in the Assay, and, if the amount is more than that specified above, dilute the solution to the specified concentration by the addition of the mixture of Purified Water and Ethanol (3 : 1).

Description Apricot Kernel Water is a clear, colorless or pale yellow liquid, has an odor of benzaldehyde and characteristic taste.

pH—3.5 ~ 5.0.

Identification Take 2 mL of Apricot Kernel Water, add 1 mL of ammonia TS, and allow to stand for 10 minutes: a slight turbidity is produced. Alow to stand for 20 minutes: the turbidity becomes more intense.

Specific Gravity d_{20}^{20} : 0.968 ~ 0.978.

Purity (1) ***Sulfate***—Add a few drops of 0.1 mol/L sodium hydroxide VS to 5.0 mL of Apricot Kernel Water to make slightly alkaline, evaporate on a water-bath to dryness, and ignite between 450 °C and 550 °C. Dissolve the residue in 1 mL of dilute hydrochloric acid, and add water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution with 0.50 mL of 0.005 mol/L sulfuric acid VS (not more than 0.005 %).

(2) ***Heavy metals***—Evaporate 50 mL of Apricot Kernel Water on a water-bath to dryness, ignite between 450 °C and 550 °C, 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, dilute the subsequent 20 mL to 50 mL with water, and perform the test using this solution as the test solution. Prepare the control solution with 2.0 mL of standard lead solution, adding 2 mL of dilute acetic acid and water to make 50 mL (not more than 1ppm).

(3) ***Free hydrogen cyanide***—Take 10 mL of Apricot Kernel Water, add 0.8 mL of 0.1 mol/L silver nitrate VS and 2 to 3 drops of nitric acid at 15 °C, filter, and add 0.1 mol/L silver nitrate VS to the filtrate: no change occurs.

(4) ***Residue on evaporation***—Evaporate 5.0 mL of Apricot Kernel water to dryness, and dry: the residue is not more than 1.0 mg.

Assay Take exactly 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 yellow turbidity persists.

Each mL of 0.1 mol/L silver nitrate VS
= 5.405 mg of HCN

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light-resistant.

Beef Tallow

Beef Tallow is a purified fat obtained by wet steam rendering from the fresh fatty tissues of *Bos taurus* Linné var. *domesticus* Gmelin (Bovidae).

Description Beef Tallow is a white, uniform mass, has a characteristic odor and mild taste.

Beef Tallow is freely soluble in ether or in petroleum ether, very slightly soluble in ethanol and practically insoluble in water.

Beef Tallow is breakable at a low temperature, but softens above 30 °C.

Melting point—42 ~ 50 °C (Method 2).

Saponification Value 193 ~ 200.

Acid Value Not more than 2.0.

Iodine Value 33 ~ 50 (When the sample is insoluble in 20 mL of cyclohexane, dissolve it by shaking a glass-stoppered flask in warm water. If it is still insoluble, increase the volume of solvent.).

Purity (1) ***Moisture and coloration*—**Take 5.0 g of Beef Tallow and melt by heating on a water-bath: the melting solution is clear and no water separates from the melting solution. And observe the melting solution

in a 10-mm thick layer of the liquid: it is colorless to pale yellow.

(2) ***Alkali***—Take 2.0 g of Beef Tallow, add 10 mL of water, melt by heating on a water-bath and shake vigorously. After cooling, add 1 drop of phenolphthalein TS to the separated water layer: no color develops.

(3) ***Chloride***—Take 1.5 g of Beef Tallow, add 30 mL of ethanol, boil for 10 minutes under a reflux condenser and 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 mixture is not more than that of the following control solution.

Control solution—Take 1.0 mL of 0.01 mol/L hydrochloric acid VS, add ethanol to make 20 mL and add 5 drops of the solution of silver nitrate in ethanol (1 in 50)

Containers and Storage ***Containers***—Well-closed containers.

White Beeswax

White Beeswax is bleached Yellow Beeswax.

Description White Beeswax is a white to yellowish white mass and has a characteristic odor.
White Beeswax is slightly soluble in ether and practically insoluble in water or in dehydrated ethanol.
White Beeswax is comparatively brittle when cooled and the fractured surface is granular and non-crystalline.

Saponification Value 80 ~ 100. Weigh accurately about 3.0 g of White Beeswax, place in a 250 mL glass-stoppered flask and add 25 mL of 0.5 mol/L potassium hydroxide-ethanol VS and 50 mL of ethanol, heat for 4 hours on a water-bath under a reflux condenser and proceed as directed in the Saponification value under the Fats and Fatty Oils.

Acid Value 5 ~ 9 or 17 ~ 22. Weigh accurately about 6 g of White Beeswax, place in a glass-stoppered 250 mL flask and add 50 mL of dehydrated ethanol. Warm the mixture to dissolve the wax, add 1 mL of phenolphthalein TS and proceed as directed in the Acid value under the Fats and Fatty Oils. Perform a blank determination using solvent which is not previously neutralized and make any necessary correction.

Melting Point 60 ~ 67 °C (Method 2).

Ester value 72~29

Purity (1) ***Paraffin, fat, Japan wax or resin, ceresin and other wax***—Melt White Beeswax at the lowest possible temperature, drip the liquid into a glass vessel containing ethanol to form granules and allow then to stand in air for 24 hours, Drop the granules into two mixtures of ethanol and water, one adjusted so as to have a specific gravity of 0.95 and the other 0.97: the granules sink or are suspended in the mixture with the specific gravity of 0.95 and float or are suspended in the other mixture.

(2) ***Glycerine and polyol***—Weigh accurately 0.2 g of White Beeswax, add 10 mL of potassium hydroxide-ethanol solution and heat with a reflux condenser on a steam bath for 30 minutes. Add 50 mL of dilute sulfuric acid, cool and filter. Wash the flask, combine the washings with the filtrate, dilute with 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 and allow to stand for 5 minutes. Add 1.0 mL of bleached fuchsin TS 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 1000 mL, 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 (not more than 0.5 % of glycerin).

(3) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat, place 10 mg to 300 mg of White Beeswax on top then 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, place only the test specimen in a nickel boat without the additives. Place the boat inside the furnace and heat to about 900 °C with a current of air or oxygen at 0.5 L/minute to 1 L/minute. Elute the mercury and sample in a sampling tube. Transfer the mercury vapor to a cold vapor atomic absorption spectrometer by heating the sampling 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 plot a calibration curve from the absorbances. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard stock solution—Dissolve 0.135 g of mercury (II) chloride in 0.001 % L-cysteine to make 1000 mL (1 mL of mercury standard stock solution = 100 μg Hg).

Mercury standard solution—Dilute mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 ng to 200 ng.

Additive—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 White Beeswax 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 standard lead 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 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 White Beeswax according to Method 3 and perform the test (not more than 4 ppm).

(6) ***Peroxide value***—Weigh accurately 5 g of White 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 the flask and shake vigorously. Titrate with 0.01 mol/L sodium thiosulfate VS (indicator: starch TS) and calculate the peroxide value according to the following equation: not more than 5. Separately, perform a blank determination and make any necessary correction.

$$\text{Peroxide value} = \frac{\text{Volume (mL) of 0.01mol/L sodium thiosulfate VS consumed}}{\text{Amount (g) of White Beeswax taken}} \times 10$$

Containers and Storage ***Containers—***Well-closed containers.

Yellow 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 is a pale yellow to brownish yellow mass, and has a characteristic odor, which is not rancid. Yellow Beeswax is comparatively brittle when cooled and the fractured surface is granular and non-crystalline.

Saponification Value 80 ~ 100. Weigh accurately about 3 g of Yellow Beeswax, place in a 259-mL stoppered flask and add 25 mL of 0.5 mol/L potassium hydroxide-ethanol VS and 50 mL of ethanol, insert a reflux condenser, heat for 4 hours on a water-bath and proceed as directed in the Saponification value under the Fats and Fatty Oils.

Acid Value 5 ~ 9 or 17 ~ 22. Weigh accurately about 6 g of Yellow Beeswax, place in a glass-stoppered 250-mL flask and add 50 mL of dehydrated ethanol. Warm the mixture to dissolve the wax, add 1 mL of phenolphthalein TS and proceed as directed in the Acid value under the Fats and Fatty Oils. Perform a blank determination using solvent which is not previously neutralized and make any necessary correction.

Ester Value 72 ~ 77

Melting Point 60 ~ 67 °C (Method 2).

Purity (1) ***Paraffin, fat, Japan wax or resin***—Melt Yellow Beeswax at the lowest possible temperature, drip the liquid into a glass vessel containing ethanol to form granules and allow then to stand in air for 24 hours, Drop the granules into two mixtures of ethanol and water, one adjusted so as to have a specific gravity of 0.95 and the other 0.97, the granules sink or are suspended in the mixture with the specific gravity of 0.95 and float or are suspended in the other mixture.

(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 washing 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 (10.7 in 1000), shake and allow to stand for 5 minutes. Add 1.0 mL of bleached fuchsin TS 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 (not more than 0.5 % of glycerin).

(3) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat and place 10 mg to 300 mg of Yellow Beeswax on top. Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. Place the boat inside the furnace and heat to about 900 °C with a current of air or oxygen at 0.5 L/minute to 1 L/minute. Elute the mercury and sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 ng to 200 ng.

Additive—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 achieved, 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 (dissolve 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 if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 solution as directed under Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 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 (not more than 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 the flask and shake vigorously. Titrate with 0.01 mol/L sodium thiosulfate VS (indicator: starch TS) and calculate the amount of peroxide by the following formula: not more than 5. Separately, perform a blank determination and make any necessary correction.

$$\text{Peroxide value} = \frac{\text{Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed}}{\text{Amount (g) of Yellow Beeswax taken}} \times 10$$

Containers and Storage ***Containers*—**Well-closed containers.

Bentonite

Bentonite is a natural, colloidal, hydrated aluminum silicate.

Description Bentonite is a very fine, white to pale yellow-brown powder, is odorless. Bentonite has a slightly earthy taste.
Bentonite is practically insoluble in water or in ether.
Bentonite swells in water.

Identification (1) Add 0.5 g of Bentonite to 3 mL of diluted sulfuric acid (1 in 3) and heat until white fumes are evolved. Cool, add 20 mL of water and filter. To 5 mL of the filtrate, add 3 mL of ammonia TS: a white, gelatinous precipitate is produced, which turns red on the addition of 5 drops of alizarin S TS.

(2) Wash the residue obtained in (1) with water, add 2 mL of methylene blue solution (1 in 10000) and wash again with water: the residue is blue.

pH Take 1.0 g of Bentonite, add 50 mL of water and shake: the pH of the suspension is between 9.0 and 10.5.

Purity (1) ***Heavy metals***—Take 1.5 g of Bentonite, add 80 mL of water and 5 mL of hydrochloric acid and boil gently for 20 minutes with thorough stirring. Cool, centrifuge, collect the clear supernatant liquid, wash the residue twice with 10 mL volumes of water and centrifuge. Combine the clear supernatant liquid and the washings and add drop-wise strong ammonia water. When a precipitate is produced, add drop-wise dilute hydrochloric acid with vigorous stirring and dissolve. To the solution, add 0.45 g of hydroxylamine hydrochloride and heat. Cool and add 0.45 g of sodium acetate, 6 mL of dilute acetic acid and water to make 150 mL. Pipet 50.0 mL, use this solution as the test solution and perform the test. Prepare the control solution as follows: to 2.5 mL of standard lead solution, add 0.15 g of hydroxylamine hydrochloride, 0.15 g of sodium acetate and 2 mL of dilute acetic acid and add water to make 50 mL (not more than 50 ppm).

(2) ***Arsenic***—Take 1.0 g of Bentonite, add 5 mL of dilute hydrochloric acid and gently heat to boil while stirring well. Cool immediately and centrifuge. To the residue, add 5 mL of dilute hydrochloric acid, shake well and centrifuge. To the residue, add 10 mL of water and perform the same operations. Combine all the extracts and heat on a water-bath to concentrate to 5 mL. Perform the test(not more than 2 ppm).

(3) ***Foreign matter***—Place 2.0 g of Bentonite in a mortar, add 20 mL of water to swell, disperse evenly with a pestle and add water to make 100 mL. Pour the suspension through a No. 200 (74 μm) sieve and wash the sieve thoroughly with water: no grit is felt when the fingers are rubbed over the wire mesh of the sieve.

Loss on Drying 5.0 ~ 10.0 % (2 g, 105 °C, 2 hours).

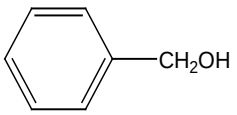
Gel Formation Mix 6.0 g of Bentonite with 0.30 g of magnesium oxide. Add the mixture, in several volumes, to 200 mL of water contained in a glass-stoppered 500 mL cylinder. Agitate for 1 hour, transfer 100 mL of the suspension to a 100 mL mass cylinder and allow to stand for 24 hours: not more than 2 mL of supernatant appears on the surface.

Swelling Power Weigh 2.0 g of Bentonite, add to 100 mL of water in a glass-stoppered 100 mL cylinder, in ten volumes, allowing each volume to settle before adding the next and allow to stand for 24 hours: the apparent volume of the sediment at the bottom is not less than 20 mL.

Microbial Limit The total aerobic microbial count is not more than 1000 CFU/g, the total combined yeasts/mould count is not more than 100 CFU/g, and *Escherichia coli*, *Salmonella*, *Pseudomonas aeruginosa* and *Staphylococcus aureus* are not observed.

Containers and Storage ***Containers***—Well-closed containers.

Benzyl Alcohol



C_7H_8O: 108.14

Benzyl alcohol [*100-51-6*]

Benzyl Alcohol contains not less than 98.0 % and not more than 100.5 % of benzyl alcohol (C_7H_8O).
The label states, where applicable, that it is suitable for the manufacture of injection forms.

Description Benzyl Alcohol is a clear, colorless liquid.
Benzyl Alcohol is miscible with ethanol, with fatty oils and with essential oils.
Benzyl Alcohol is soluble in water.

Specific gravity d_{20}^{20} : 1.043 ~ 1.049.

Identification Determine the infrared spectra of Benzyl Alcohol and Benzyl Alcohol RS, both previously dried, as directed in the liquid film method under the Infrared Spectrophotometry: both spectra exhibit similar intensities of absorption at the same wavenumbers.

Refractive Index n_D^{20} : 1.538 ~ 1.541.

Purity (1) ***Clarity and color of solution***—Dissolve 2.0 mL of Benzyl Alcohol in 60 mL of water: the solution is clear and colorless.

(2) ***Acid***—Take 10 mL of Benzyl Alcohol, add 10 mL of neutralized ethanol, 2 drops of phenolphthalein TS and 1.0 mL of 0.1 mol/L sodium hydroxide VS: a red color develops.

(3) ***Benzaldehyde and other related substances***—Use Benzyl Alcohol as the test solution. Separately, weigh exactly 0.750 g of benzaldehyde and 0.500 g of cyclohexylmethanol, add Benzyl Alcohol to make exactly 25 mL. Pipet 1 mL of this solution, add exactly 2 mL of the ethylbenzene internal standard solution and exactly 3 mL of the dicyclohexyl internal standard solution, 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 standard solution (1) as directed under the Gas Chromatography according to the following operating conditions: no peak of ethylbenzene or dicyclohexyl appears in the chromatogram obtained from the test solution. Proceed with injection of 0.1 μL of the standard solution (1) and adjust the sensitivity of the detector so that the peak height of ethylbenzene is not more than 30 % of the full scale of the recorder. The peak area of benzaldehyde obtained from the test solution is not more than the difference between the peak areas of benzaldehyde of the test solution and the standard solution (1) (0.15 %), and the peak area of cyclohexylmethanol from the test solution is not more than the difference between the peak areas of cyclohexylmethanol of the test solution and the standard solution (1) (0.10 %). The total area of the peaks having the retention time smaller than benzyl alcohol and other than benzaldehyde and cyclohexylmethanol obtained from the test solution is not more than 4 times the peak area of ethylbenzene obtained from the standard solution (1) (0.04 %). The total area of the peaks having the retention time larger than benzyl alcohol obtained from the test solution is not more than the peak area of dicyclohexyl from the standard solution (1) (0.3 %). Disregard any peak having the area not more than 1/100 times the peak area of ethylbenzene from the standard solution (1) for these calculations.

Benzyl Alcohol labeled that it is suitable for use in the manufacture of injection forms meets the following requirements.

Use Benzyl Alcohol as the test solution. Separately, weigh exactly 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 ethylbenzene internal standard solution and exactly 2 mL of the dicyclohexyl internal standard solution, then 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 standard solution (2) as directed under the Gas Chromatography according to the following operating conditions: no peak of ethylbenzene or dicyclohexyl appears on the chromatogram obtained from the test solution. Proceed with injection of 0.1 μL of the standard solution (2) and adjust the sensitivity of the detector so that the peak height of ethylbenzene is not more than 30 % of the full scale of the recorder. The peak area of benzaldehyde of obtained from the test solution is not more than the difference between the peak areas of benzaldehyde of the test solution and the standard solution (2) (0.05 %), and the peak area of cyclohexylmethanol from the test solution is not more than the difference between the peak areas of cyclohexylmethanol of the test solution and the standard solution (2) (0.10 %). The total area of the peaks having the retention time smaller than benzyl alcohol and other than benzaldehyde and cyclohexylmethanol obtained from the test solution is not more than 2 times the peak area of ethylbenzene from the standard solution (2) (0.02 %). The total area of the peaks having the retention time larger than benzyl alcohol obtained from the test solution is not more than the peak area of dicyclohexyl from the standard solution (2) (0.2 %). Disregard any peak having the area not more than 1/100 times the peak area of ethylbenzene from the standard solution (2) for these calculations.

Ethylbenzene internal standard solution—Dissolve exactly 0.100 g of ethylbenzene in Benzyl Alcohol to make exactly 10 mL. Pipet 2 mL of this solution and add Benzyl Alcohol to make exactly 20 mL.

Dicyclohexyl internal standard solution—Dissolve exactly 2.000 g of dicyclohexyl 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 hydrogen 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 20 M for gas chromatography in 0.5 μm thickness.

Column temperature: Raise the temperature at a rate of 5 °C per minutes from 50 °C to 220 °C, and maintain at 220 °C for 35 minutes.

Temperature of injection port: A constant temperature of about 200 °C.

Temperature of detector: A constant temperature of about 310 °C.

Carrier gas: Helium

Flow rate: Adjust the flow rate so that the retention time of benzyl alcohol is between 24 and 28 minutes.

Split ratio: Splitless

System suitability

System performance: When the procedure is run with the standard solution (1) under the above operating conditions, the relative retention times of ethylbenzene, dicyclohexyl, benzaldehyde and cyclohexylmethanol with respect to benzyl alcohol are about 0.28, about 0.59, about 0.68 and about 0.71, respectively, with the resolution between the peaks of benzaldehyde and cyclohexylmethanol being not less than 3.0. In the case of Benzyl Alcohol labeled to use for injection, proceed with the standard solution (2) instead of the standard solution (1).

(4) ***Peroxide value***—Dissolve 5 g of Benzyl Alcohol, accurately weighed, in 30 mL of a mixture of glacial acetic acid and chloroform (3 : 2) in a glass-

stoppered conical flask. Add 0.5 mL of potassium iodide saturated solution, shake exactly for 1 minute, add 30 mL of water, and titrate with 0.01 mol/L sodium thiosulfate VS until the blue color of the solution disappears after addition of 10 mL of starch TS near the end point where the solution is a pale yellow color. Perform a blank determination and make any necessary correction. Calculate the amount of peroxide by the following formula: not more than 5.

$$\text{Amount (mEq/kg) of peroxide} = \frac{[10 \times (V_I - V_0)]}{W}$$

V_I: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed in the test

V_0: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed in the blank

V: Amount (g) of Benzyl Alcohol taken

(5) ***Residue on evaporation***—Perform the test after confirmation that the test specimen meets the requirement of the peroxide value. Transfer 10.0 g of Benzyl Alcohol to a porcelain or quartz crucible or platinum dish, previously weighed accurately, and heat on a hotplate at not exceeding 200 °C, taking care to avoid boiling, to evaporate to dryness. Dry the residue on the hot -plate for 1 hour, and allow to cool in a desiccator: not more than 5 mg.

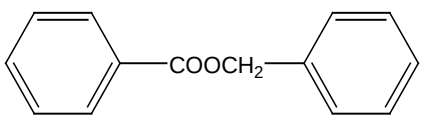
Assay Weigh accurately about 0.9 g of Benzyl Alcohol, add exactly 15 mL of a mixture of pyridine and acetic anhydride (7 : 1) and heat in a water-bath under a reflux condenser for 30 minutes. Cool, add 25 mL of water and titrate the excess acetic acid with 1 mol/L sodium hydroxide VS (indicator: 2 drops of phenolphthalein TS). Perform a blank determination and make any necessary correction.

Each mL of 1 mol/L sodium hydroxide VS
= 108.14 mg of C_7H_8O

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light resistant.

Benzyl Benzoate



$C_{14}H_{12}O_2$: 212.24

Benzyl benzoate [*120-51-4*]

Benzyl Benzoate contains not less than 99.0 % and not more than 101.0 % of benzyl benzoate ($C_{14}H_{12}O_2$).

Description Benzyl Benzoate is a colorless, clear, viscous liquid, has a faint, aromatic odor and a pungent, burning taste.

Benzyl Benzoate is practically insoluble in water.

Benzyl Benzoate is miscible with ethanol or with ether.

Congealing point—About 17 °C.

Specific gravity—d_{20}^{20}: about 1.123.

Boiling point—About 323 °C.

Identification (1) Heat gently 1 mL of Benzyl Benzoate with 5 mL of sodium carbonate TS and 2 mL of potassium permanganate TS: the odor of benzaldehyde is perceptible.

(2) Warm the titrated mixture obtained in the Assay on a water-bath to remove ethanol and add 0.5 mL of ferric chloride TS: a pale yellow-red precipitate is produced, which turns white on the addition of dilute hydrochloric acid.

Refractive Index n_D^{20}: 1.568 ~ 1.570.

Purity **(1)** ***Acid***—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 VS: a red color develops.

(2) ***Aldehyde***—Weigh accurately 10.0 g of Benzyl Benzoate, transfer to a 125 mL conical flask, add 50 mL of alcohol and 5 mL of hydroxylamine hydrochloride solution (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 solution changes to light green. Perform a blank determination and make any necessary correction. Not more than 0.50 mL of 0.1 mol/L sodium hydroxide VS is consumed (not more than 0.05 % of benzaldehyde).

Residue on Ignition Not more than 0.05 % (2 g).

Assay Weigh accurately about 2 g of Benzyl Benzoate, add 50.0 mL of 0.5 mol/L potassium hydroxide-ethanol VS and boil gently for 1 hour under a reflux condenser with a carbon dioxide-absorbing tube (soda lime). Cool and titrate the excess potassium hydroxide with 0.5 mol/L hydrochloric acid VS (indicator: 2 drops of phenolphthalein TS). Perform a blank determination and make any necessary correction.

Each mL of 0.5 mol/L potassium hydroxide-ethanol VS
= 106.12 mg of $C_{14}H_{12}O_2$

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light resistant.

Cacao Butter

Oleum Cacao

Cacao Butter is the fat obtained from the seed of *Theobroma cacao* Linné (Sterculiaceae).

Description Cacao Butter is pale yellow, hard, brittle mass, has a slight, chocolate-like odor and has no odor of rancidity.
Cacao Butter is freely soluble in ether or in chloroform, soluble in boiling dehydrated ethanol and very slightly soluble in ethanol.

Congealing point of the fatty acids—45 ~ 50 °C.

Melting point—31 ~ 35 °C (cram the sample into a capillary tube without melting the sample, then follow Method 2).

Saponification Value 188 ~ 195.

Specific Gravity d_{20}^{40}: 0.895 ~ 0.904.

Acid Value Not more than 3.0.

Iodine Value 35 ~ 43.

Containers and Storage ***Containers*—**Well-closed containers.

Calcium Hydroxide

Slaked Lime $Ca(OH)_2$: 74.09

Calcium Hydroxide contains not less than 90.0 % and not more than 101.0 % of calcium hydroxide [$Ca(OH)_2$].

Description Calcium Hydroxide is a white powder and has a slightly bitter taste.
Calcium Hydroxide is slightly soluble in water, very slightly soluble in boiling water and practically insoluble in ethanol or in ether.
Calcium Hydroxide absorbs carbon dioxide from air.
Calcium Hydroxide dissolves in dilute acetic acid, in dilute hydrochloric acid and in dilute nitric acid.

Identification (1) Mix Calcium Hydroxide with 3 to 4 times its mass of water: the mixture is slushy and is alkaline.

(2) Dissolve 1 g of Calcium Hydroxide in 30 mL of dilute acetic acid and boil. After cooling, neutralize with ammonia TS: the solution responds to the Qualitative Tests (2) and (3) for calcium salt.

Purity (1) ***Acid-insoluble substances***—Take 5 g of Calcium Hydroxide, add 100 mL of water, add hydrochloric acid drop-wise with stirring until the solution becomes acidic and further add 1 mL of hydrochloric acid. Boil this solution for 5 minutes, cool and filter through a tared glass filter (G4). Wash the residue with boiling water until the last washing exhibits no turbidity upon addition of silver nitrate TS and dry at 105 °C to constant weight: the amount is not more than 25 mg.

(2) ***Fluoride***—Weigh 1 g of Calcium Hydroxide, 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 (1 in 4) and 10 mL of disodium ethylenediaminetetraacetate (1 in 40) and shake. 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, determine the potential using a fluoride electrode and determine the amount of fluoride from the calibration curve: not more than 50 ppm.

Standard solution—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 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).

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 (1 in 4) and 10 mL of disodium ethylenediaminetetraacetate (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. Pipet 50 mL each of these solutions and transfer to polyethylene containers. Determine the potential using a fluoride electrode and plot 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 on a water-bath to dryness, dissolve the residue in 40 mL of water and filter. To 20 mL of the filtrate add 2 mL of dilute acetic acid and water to make 50 mL and perform the test. Prepare the control solution as follows: evaporate 5 mL of dilute hydrochloric acid on a water-bath to dryness and add 2 mL of dilute acetic acid, 2.0 mL of standard lead solution and add water to make 50 mL (not more than 20 ppm).

(4) ***Lead***—Weigh accurately 5.0 g of Calcium Hydroxide, transfer to a 150 mL beaker and add 30 mL of water. Add hydrochloric acid in small amounts until the test specimen is completely dissolved 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 between 2 and 4 with sodium hydroxide (1 in 4) or hydrochloric acid (1 in 4). Transfer 250 mL of this solu-

tion to a separatory funnel, add water to make about 200 mL, add 2 mL of 2 % ammonium pyrrolidine dithiocarbamate solution (2 in 100) and shake. Extract this solution with two 20 mL volumes of chloroform and evaporate the extract to dryness in 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 1.0 mL of standard lead 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 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 to stand for 15 minutes. The turbidity of this solution is not more than that of the following solution: to 0.3 mL of barium standard solution, add water to make 20 mL and proceed in the same manner as above (not more than 0.03 %).

(6) ***Magnesium and alkali metals***—Dissolve 1.0 g of Calcium Hydroxide in a mixture of 20 mL of water and 10 mL of dilute hydrochloric acid, boil, neutralize with ammonia TS and precipitate calcium oxalate completely by adding drop-wise ammonium oxalate TS. Heat the mixture on a water-bath 1 hour, cool, add water to make 100 mL, shake 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 weight: the residue is not more than 24 mg.

(7) ***Arsenic***—Dissolve 0.5 g of Calcium Hydroxide in 5 mL of dilute hydrochloric acid and perform the test (not more than 4 ppm).

(8) ***Carbonate***—To 2.0 g of Calcium Hydroxide, add 50 mL of water, shake and add an excess 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 100 mL. Piper 10.0 mL of this solution, add 90 mL of water and 1.5 mL of 8 mol/L potassium hydroxide TS, shake, allow to stand for 3 minutes to 5 minutes and then add 0.1 g of NN indicator. Titrate immediately with 0.05 mol/L disodium ethylenediamine tetraacetate VS, until the color of the solution changes from red-purple to blue.

Each mL of 0.05 mol/L disodium ethylenediamine tetraacetate VS = 3.7046 mg of $Ca(OH)_2$

Containers and Storage ***Containers***—Tight containers.

Calcium Oxide

Quick Lime CaO: 56.08

Calcium Oxide, when ignited, contains not less than 98.0 % and not more than 101.0 % of calcium oxide (CaO).

Description Calcium Oxide is a hard, white mass, containing powder and odorless.
Calcium Oxide is very slightly soluble in boiling water and practically insoluble in ethanol.
One gram of Calcium Oxide dissolves almost completely in 2500 mL of water.
Calcium Oxide slowly absorbs moisture and carbon dioxide from air.

Identification (1) Moisten Calcium Oxide with water: heat is generated and a white powder is obtained. Mix the powder with about 5 times its mass of water: the mixture is alkaline.

(2) Dissolve 1 g of Calcium Oxide in 20 mL of water by adding a few drops of acetic acid: the solution responds to the Qualitative Tests for calcium salt.

Purity (1) ***Acid-insoluble substances***—Disintegrate 5.0 g of Calcium Oxide with a small amount of water, add 100 mL of water and drop-wise hydrochloric acid with stirring until the solution becomes acidic and further add 1 mL of hydrochloric acid. Boil the solution for 5 minutes, cool, filter through a glass filler (G4), wash the residue with boiling water until no turbidity is produced when silver nitrate TS is added to the last washing and dry at 105 °C to a constant weight: the residue is not more than 10.0 mg.

(2) ***Carbonate***—Disintegrate 1.0 g of Calcium Oxide with a small amount of water, mix thoroughly with 50 mL of water, allow to stand for a while, remove most of the supernatant milky liquid by decantation and add an excess of dilute hydrochloric acid to the residue: no vigorous foam is produced.

(3) ***Magnesium and alkali metals***—Dissolve 1.0 g of Calcium Oxide in 75 mL of water by adding drop-wise hydrochloric acid and further add 1 mL of hydrochloric acid. Boil for 1 minute to 2 minutes, neutralize with ammonia TS, add drop-wise an excess of hot ammonium oxalate TS, heat the mixture on a water-bath for 2 hours, cool, add water to make 200 mL, mix thoroughly and filter. Evaporate 50 mL of the filtrate with 0.5 mL of sulfuric acid to dryness and ignite the residue at 600 °C to a constant weight: the residue is not more than 15 mg.

Loss on Ignition Not more than 10.0 % (1 g, 900 °C, constant weight).

Assay Weigh accurately about 0.7 g of Calcium Oxide, previously ignited at 900 °C to a constant weight and cooled in a desiccator (silica gel), and dissolve in 50 mL of water and 8 mL of diluted hydrochloric acid (1 in 3) by heating. Cool and add water to make exactly 250 mL. Pipet 10 mL of the solution, add 50 mL of water, 2 mL of 8 mol/L potassium hydroxide TS and 0.1 g of NN indicator and titrate with 0.02 mol/L disodium ethylenediaminetetraacetate VS, until the color of the solution changes from red-purple to blue.

Each mL of 0.02 mol/L
disodium ethylenediaminetetraacetate VS
= 1.1215 mg of CaO

Containers and Storage ***Containers*—**Tight containers.

Anhydrous Dibasic Calcium Phosphate

$CaHPO_4$: 136.06

[*7757-93-9*]

Anhydrous Dibasic Calcium Phosphate, when dried, contains not less than 98.0 % and not more than 103.0 % of dibasic calcium phosphate ($CaHPO_4$).

Description Anhydrous Dibasic Calcium Phosphate appears as white crystalline powder or granules.
Anhydrous Dibasic Calcium Phosphate is practically insoluble in water or in ethanol
Anhydrous Dibasic Calcium Phosphate dissolves in dilute hydrochloric acid or in dilute nitric acid.

Identification Proceed as directed in the Identification under Dibasic Calcium Phosphate Hydrate.

Purity (1) ***Acid-insoluble substances and Chloride***—Proceed as directed in the Purity (1) and (2) under Dibasic Calcium Phosphate Hydrate.

(2) ***Sulfates***—Weigh 0.80 g of Anhydrous Dibasic Calcium Phosphate, and proceed as directed in the Purity (3) under Dibasic Calcium Phosphate Hydrate (not more than 0.200 %).

(3) ***Carbonate, Heavy metals, Barium Mercury, Cadmium, Lead, Arsenic and Fluoride***—Proceed as directed in Purity (4), (5), (6), (7), (8), (9), (10) and (11) under Dibasic Calcium Phosphate Hydrate.

Loss on Ignition 6.6 ~ 8.5 % (1 g, 800 ~ 825 °C, constant mass)

Assay Proceed as directed in the Assay under Dibasic Calcium Phosphate Hydrate.

Each mL of 0.02 mol/L
disodium ethylenediaminetetraacetate VS
= 2.7211 mg of $CaHPO_4$

Containers and Storage ***Containers*—**Well-closed containers.

Dibasic Calcium Phosphate Hydrate

$CaHPO_4 \cdot 2H_2O$: 172.09

[*7789-77-7*]

Dibasic Calcium Phosphate Hydrate contains not less than 98.0 % and not more than 105.0 % of dibasic calcium phosphate ($CaHPO_4$: 136.06).

Description Dibasic Calcium Phosphate Hydrate is white, crystalline powder.
Dibasic Calcium Phosphate Hydrate is practically insoluble in water or in ethanol.
Dibasic Calcium Phosphate Hydrate dissolves in dilute hydrochloric acid or in dilute nitric acid.

Identification (1) Dissolve 0.1 g of Dibasic Calcium Phosphate Hydrate in 1.0 mL of diluted hydrochloric acid (1 in 6) by warming, add 2.5 mL of ammonia TS dropwise with shaking and add 5 mL of ammonium oxalate TS: a white precipitate is produced.

(2) Dissolve 0.1 g of Dibasic Calcium Phosphate Hydrate in 5 mL of dilute nitric acid, warm for 1 to 2 minutes at 70 °C, and add 2 mL of ammonium molybdate TS: a yellow precipitate is produced.

Purity (1) ***Acid-insoluble substance***—Dissolve 5.0 g of Dibasic Calcium Phosphate Hydrate in 40 mL of water and 10 mL of hydrochloric acid and boil for 5 minutes. After cooling, collect the insoluble substance by filtration using filter paper for quantitative analysis. Wash with water until no more turbidity of the washing is produced by adding silver nitrate TS. Ignite to incinerate the residue and filter paper: the mass is not more than 2.5 mg (not more than 0.05 %).

(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 using 50 mL of this solution as the test solution. Prepare the control solution with 0.70 mL of 0.01 mol/L hydrochloric acid (not more than 0.248 %).

(3) ***Sulfate***—Dissolve by warming 1.0 g of Dibasic Calcium Phosphate Hydrate in 5 mL of water and 5 mL of dilute hydrochloric acid, add water to make 100 mL

and filter, if necessary. Take 30 mL of the filtrate and add 1 mL of dilute hydrochloric acid and add water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution with 1.0 mL of 0.005 mol/L sulfuric acid (not more than 0.160 %).

(4) ***Carbonate***—Dissolve 1.0 g of Dibasic Calcium Phosphate Hydrate with 5 mL of water and add immediately 2 mL of hydrochloric acid: no foam is produced.

(5) ***Heavy metals***—Dissolve 0.65 g of Dibasic Calcium Phosphate Hydrate in a mixture of 5 mL of water and 5 mL of dilute hydrochloric acid by warming, cool and add ammonia TS until precipitates begin to form in the solution. Dissolve the precipitates by adding a small amount of dilute hydrochloric acid dropwise, add 10 mL of hydrochloric acid-ammonium acetate buffer solution, pH 3.5, and water to make 50 mL and perform the test using this solution as the test solution. Prepare the control solution by adding 10 mL of hydrochloric acid-ammonium acetate buffer solution, pH 3.5, to 2.0 mL of standard lead solution and add water to make 50 mL (not more than 31 ppm).

(6) ***Barium***—Heat 0.5 g of Dibasic Calcium Phosphate Hydrate with 10 mL of water, add 1 mL of hydrochloric acid dropwise with stirring and filter, if necessary. Add 2 mL of potassium sulfate TS of the filtrate and allow to stand for 10 minutes: no turbidity is produced.

(7) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat and place 10 to 300 mg of Dibasic Calcium Phosphate Hydrate on top. Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. 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 sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 to 200 ng.

Additive—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.

(8) ***Cadmium***—Use the test solution from Purity (9) as the test solution. Separately, proceed with 5.0 mL of cadmium 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 1.0 ppm).

Gas: Acetylene or hydrogen – Air
Lamp: Cadmium hollow cathode lamp
Wwavelength: 228.8 nm

(9) ***Lead***—Weigh accurately 5.0 g of Dibasic Calcium Phosphate Hydrate, transfer to a 150 mL beaker and add 30 mL of water. Add hydrochloric acid in small amounts until the test specimen is completely dissolved 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 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 solution (2 in 100) and shake. Extract this solution with two 20 mL volumes of chloroform and evaporate the extract to dryness in 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 2.0 mL of standard lead 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 4.0 ppm).

Gas: Acetylene or hydrogen – Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(10) ***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 (not more than 2 ppm).

(11) ***Fluoride***—Weigh 1 g of Dibasic Calcium 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 (1 in 4) and 10 mL of disodium ethylenediaminetetraacetate (1 in 40) and shake. 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, determine the potential using a fluoride electrode and determine the amount of fluoride from the calibration curve: not more than 50 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 1000 mL 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 (1 in 4) and 10 mL of disodium ethylenediaminetetraacetate (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), add water to make 100 mL each and use these solutions as the standard solutions. Pipet 50 mL of each standard solution into polyethylene containers. Determine the potential using a fluoride electrode and plot a calibration curve with the log values of the fluoride concentrations.

Loss on Ignition 24.5 ~ 26.5 % (1 g, 800 ~ 825 °C, constant mass)

Assay Weigh accurately about 0.4 g of Dibasic Calcium Phosphate Hydrate, dissolve in 12 mL of dilute hydrochloric acid and add water to make exactly 200 mL. Pipet exactly 20 mL of this solution, add exactly 25 mL of 0.02 mol/L disodium ethylenediaminetetraacetate VS, 50 mL of water and 5 mL of ammonia-ammonium chloride buffer solution, pH 10.7, and titrate the excess disodium ethylenediaminetetraacetate with 0.02 mol/L zinc sulfate VS (indicator: 25 mg of eriochrome black T-sodium chloride indicator). Perform a blank determination and make any necessary correction.

Each mL of 0.02 mol/L
disodium ethylendiaminetetraacetate VS
= 3.442 mg of $CaHPO_4 . 2H_2O$

Containers and Storage ***Containers*—**Well-closed containers.

Monobasic Calcium Phosphate Hydrate

$Ca(H_2PO_4)_2 \cdot H_2O$: 252.07

Monobasic Calcium Phosphate, when dried, contains not less than 90.0 % and not more than 101.0 % of Monobasic calcium phosphate hydrate $[Ca(H_2PO_4)_2 \cdot H_2O]$.

Description Monobasic Calcium Phosphate Hydrate appears as white crystals or crystalline powder, odorless and has acid taste.
Monobasic Calcium Phosphate Hydrate is sparingly soluble in water and practically insoluble in ethanol or in ether.
Monobasic Calcium Phosphate Hydrate dissolves in dilute hydrochloric acid or in dilute nitric acid.
Monobasic Calcium Phosphate Hydrate is slightly deliquescent.

Identification Proceed as directed in the Identification under Dibasic Calcium Phosphate Hydrate.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Monobasic Calcium Phosphate Hydrate in 19 mL of water and 2 mL of diluted hydrochloric acid (3 in 4) and heat on a water-bath for 5 minutes with occasional shaking: the solution is clear and colorless.

(2) ***Dibasic phosphate and acid***—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: a red color develops. Then add 1.0 mL of 1 mol/L sodium hydroxide VS: the color changes to yellow.

(3) ***Chloride***—Dissolve 1.0 g of Monobasic Calcium Phosphate Hydrate in 20 mL of water and 12 mL of dilute nitric acid, add water to make exactly 100 mL and filter, if necessary. Perform the test using 50 mL of this solution as the test solution. Prepare the control solution with 0.25 mL of 0.01 mol/L hydrochloric acid (not more than 0.018 %).

(4) ***Sulfate***—Dissolve 1.0 g of Monobasic Calcium Phosphate Hydrate in 20 mL of water and 1 mL of hydrochloric acid, add water to make 100 mL and filter, if necessary. Perform the test using 50 mL of this solution as the test solution. Prepare the control solution with 0.5 mL of 0.005 mol/L sulfuric acid (not more than 0.048 %).

(5) ***Heavy metals***—Proceed as directed in the Purity (5) under Dibasic Calcium Phosphate Hydrate.

(6) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat and place 10 to 300 mg of Monobasic Calcium Phosphate Hydrate on top. Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. 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 sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 to 200 ng.

Additive—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 from 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 1.0 ppm).

Gas: Acetylene or hydrogen – Air
Lamp: Cadmium hollow cathode lamp
Wwavelength: 228.8 nm

(8) ***Lead***—Weigh accurately 5.0 g of Monobasic Calcium Phosphate Hydrate, transfer to a 150 mL beaker and add 30 mL of water. Add hydrochloric acid in small amounts until the test specimen is completely dissolved 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 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 solution (2 in 100) and shake. Extract this solution with two 20 mL volumes of chloroform and evaporate the extract to dryness in 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 standard lead 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 4.0 ppm).

Gas: Acetylene or hydrogen – Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(9) ***Arsenic***—Dissolve 1.0 g of Monobasic Calcium Phosphate Hydrate with this solution as the test solution in 5 mL of dilute hydrochloric acid and perform the test (not more than 2 ppm).

(10) ***Fluoride***—Weigh 1 g of Monobasic Calcium 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 (1 in 4) and 10 mL of disodium ethylenediaminetetraacetate (1 in 40) and shake. 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, determine the potential using a fluoride electrode and determine the amount of fluoride from the calibration curve: not more than 10 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 1000 mL 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 (1 in 4) and 10 mL of disodium ethylenediaminetetraacetate (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), add water to make 100 mL each and use these solutions as the standard solutions. Pipet 50 mL of each standard solution into polyethylene containers. Determine the potential using a fluoride electrode and plot a calibration curve with the log values of the fluoride concentrations.

Loss on Drying Not more than 3.0 % (1 g, silica gel,

24 hours).

Loss on Ignition 14.0 ~ 15.5 %, when anhydrous of Monobasic Calcium Phosphate Hydrate is ignited at 800 °C for 30 minutes.

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. Pipet exactly 20 mL of this solution and proceed as directed in the Assay under Dibasic Calcium Phosphate Hydrate.

Each mL of 0.02 mol/L
disodium ethylendiaminetetraacetate VS
= 5.041 mg of $Ca(H_2PO_4)_2 \cdot H_2O$

Containers and Storage ***Containers*—**Tight containers.

Calcium Stearate

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 not less than 6.4 % and not more than 7.1 % of calcium (Ca: 40.08).

Description Calcium Stearate is a white, light, bulky powder. Calcium Stearate feels smooth when touched and is adhesive to the skin. Calcium Stearate is odorless or has a faint, characteristic odor.
Calcium Stearate is practically insoluble in water, in ethanol or in ether.

Identification (1) Shake vigorously 3 g of Calcium Stearate with 20 mL of diluted hydrochloric acid (1 in 2) and 30 mL of ether for 3 minutes and allow to stand: the separated aqueous layer responds to the Qualitative Tests (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 not below 54 °C (Method 2).

Purity (1) ***Heavy metals***—Heat gently 1.0 g of Calcium Stearate with caution at first and then ignite gradually to ash. 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, add water to make 50 mL and perform the test. Prepare the control solution by evaporating 2 mL of hydrochloric acid on a water-bath to dryness and by adding 2 mL of dilute acetic acid, 2.0 mL of standard lead solution and add water to make 50 mL (not more than 20 ppm).

(2) ***Arsenic***—Take 1.0 g of Calcium Stearate, add 5 mL of diluted hydrochloric acid (1 in 2) and 20 mL of chloroform, shake vigorously for 3 minutes, allow to stand and separate the water layer. Perform the test with the water layer as the test solution (not more than 2 ppm).

Loss on Drying Not more than 4.0 % (1 g, 105 °C, 3 hours)

Microbial Limit The total aerobic microbial count is not more than 1000 CFU/g, the total combined yeasts/mould count is not more than 100 CFU/g and *Escherichia coli*, *Salmonella* species, *Staphylococcus aureus* and *Pseudomonas aeruginosa* are not observed.

Assay Weigh accurately about 0.5 g of Calcium Stearate, previously dried, heat gently with caution at first and then ignite gradually to ash. Cool, 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 VS, 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 rapidly the excess disodium ethylenediamine tetraacetate with 0.05 mol/L magnesium chloride VS, until the green color of the solution disappears and a red color develops. Perform a blank determination and make any necessary correction.

Each mL of 0.05 mol/L disodium ethylenediamine
tetraacetate VS = 2.0039 mg of Ca

Containers and Storage ***Containers*—**Well-closed containers.

Camellia Oil

Camellia Oil is the fixed oil obtained from the peeled seeds of *Camellia japonica* Linné or other allied plants (Theaceae).

Description Camellia Oil is a colorless or pale yellow, clear oil, is nearly odorless and tasteless.
Camellia Oil is miscible with ether and petroleum ether.
Camellia Oil slightly soluble in ethanol.

Congealing point—Partially at -10 °C and completely -15 °C.

Specific Gravity d_{25}^{25} : 0.910~ 0.914.

Identification Take 2 mL of Camellia Oil, add dropwise 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 188 ~ 194.

Unsaponifiable Matters Not more than 1.0 %.

Acid Value Not more than 2.8.

Iodine Value 78 ~ 83.

Containers and Storage ***Containers*—**Tight containers.

Capsules

Capsules are made of gelatin or a suitable material and their shape is a pair of cylinders with one end closed which can be overlapped on each other.

Method of Preparation Dissolve Gelatin or the like in water by warming, add Glycerin or D-Sorbitol, emulsifier, preservatives, coloring substances and so forth, if necessary, to make a thick gluey solution and form into capsules while warm. Capsules may be coated with a lubricant, if necessary.

Description Capsules are odorless and elastic.

Purity ***Odor, Solubility and acidity or alkalinity***— Place, without overlapping of the parts, 1 piece (1 pair) of Capsules in a 100 mL Erlenmeyer flask, add 50 mL of water and shake often, keeping the temperature at 37 °C ± 2 °C. Perform this test 5 times: they all dissolve within 10 minutes and all these solutions are odorless and neutral or slightly acidic.

Containers and Storage ***Containers*—**Well-closed containers.

Carboxymethylcellulose

Carmellose
CMC

[*9000-11-7*]

Carboxymethylcellulose is a polycarboxymethylether of cellulose.

Description Carboxymethylcellulose is white powder, is odorless and tasteless.
Carboxymethylcellulose is practically insoluble in ethanol and in ether.
Carboxymethylcellulose swells with water to form suspension.
Carboxymethylcellulose becomes viscose in sodium hydroxide TS.
The pH of a suspension, obtained by shaking 1 g of Carboxymethylcellulose with 100 mL of water, is between 3.5 and 5.0.
Carboxymethylcellulose is hygroscopic.

Identification (1) Shake well 0.1 g of Carboxymethylcellulose with 10 mL of water, add 2 mL of sodium hydroxide TS, shake, allow to stand for 10 minutes and use this 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: a red-purple color develops.

(2) Shake 5 mL of the test solution obtained in (1) with 10 mL of acetone: a white, flocculent precipitate is produced.

(3) Shake 5 mL of the test solution obtained in (1) with 1 mL of ferric chloride TS: a brown, flocculent precipitate is produced.

Purity (1) ***Chloride***—Shake well 0.8 g of Carboxymethylcellulose with 50 mL of water, dissolve in 10 mL of sodium hydroxide TS and add water to make 100 mL. Heat 20 mL of this solution with 10 mL of dilute nitric acid on a water-bath until a flocculent precipitate is produced, cool, centrifuge and take the clear supernatant liquid. Wash the precipitate three times with 10 mL volumes of water by centrifuging each time, combine the clear supernatant liquid and the washings and add water to make 100 mL. Pipet 25 mL of this solution and add 6 mL of dilute nitric acid and water to make 50 mL. Perform the test. Prepare the control solution with 0.40 mL of 0.01 mol/L hydrochloric acid (not more than 0.360 %).

(2) ***Sulfate***—Shake well 0.40 g of Carboxymethylcellulose with 25 mL of water, dissolve in 5 mL of sodium hydroxide TS and add 20 mL of water. Heat this solution with 2.5 mL of hydrochloric acid on a water-bath until a flocculent precipitate is produced. Cool, centrifuge and take the clear supernatant liquid. Wash the precipitate three times with 10 mL volumes of water by centrifuging each time, combine the clear supernatant liquid and the washings and add water to make 100 mL. Filter this solution, discard 5 mL of the first filtrate, take 25 mL of the subsequent filtrate and add 1 mL of dilute hydrochloric acid and add water to make 50 mL. Perform the test. Prepare the control solution with 1.5 mL of 0.005 mol/L sulfuric acid (not more than 0.720 %).

(3) ***Silicate***—Weigh accurately about 1 g of Carboxymethylcellulose, ignite in a platinum crucible, add 20 mL of dilute hydrochloric acid, cover with a watch glass and boil gently for 30 minutes. Remove the watch glass and evaporate on a water-bath to dryness with the aid of a current of air. Continue heating further for 1 hour, add 10 mL of hot water, stir well and filter through a filter paper for quantitative analysis. Wash the residue with hot water, dry the residue together with the filter paper when no turbidity is produced on the addition of silver nitrate TS to the last washing and

ignite to constant weight: the residue is not more than 0.5 %.

(4) ***Heavy metals***—Proceed with 1.0 g of Carboxymethylcellulose according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 20 ppm).

(5) ***Arsenic***—Prepare the test solution with 1.0 g of cabroxymethylcellulose according to Method 3 and perform the test (not more than 2 ppm).

Loss on Drying Not more than 8.0 % (1 g, 105 °C, 4 hours)

Residue on Ignition Not more than 1.5 % (after drying, 1 g)

Containers and Storage ***Containers***—Tight containers.

Carboxymethylcellulose Calcium

Carmellose Calcium
CMC Calcium

[*9050-04-8*]

Carboxymethylcellulose Calcium is the calcium salt of a polycarboxymethylether of cellulose.

Description Carboxymethylcellulose Calcium is white to yellow powder and is odorless.
Carboxymethylcellulose Calcium is practically insoluble in ethanol or in ether.
Carboxymethylcellulose Calcium swells with water to form a suspension.
The pH of a suspension, obtained by shaking 1 g of Carboxymethylcellulose Calcium with 100 mL of water, is between 4.5 and 6.5.
Carmellose Calcium is hygroscopic.

Identification (1) Shake thoroughly 0.1 g of Carboxymethylcellulose Calcium with 10 mL of water, add 2 mL of sodium hydroxide TS, allow to stand for 10 minutes and use this 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: a red-purple color develops.

(2) Shake 5 mL of the test solution obtained in (1) with 10 mL of acetone: a white, flocculent precipitate is produced.

(3) Shake 5 mL of the test solution obtained in (1) with 1 mL of ferric chloride TS: a brown, flocculent precipitate is produced.

(4) Ignite 1 g of Carboxymethylcellulose Calcium to ash, dissolve the residue in 10 mL of water and 6 mL of acetic acid and filter, if necessary. Boil the filtrate, cool and neutralize with ammonia TS: the solution responds to the Qualitative Tests (1) and (3) for calcium salt.

Purity (1) ***Alkali***—Shake thoroughly 1.0 g of Carboxy-methylcellulose Calcium with 50 mL of water, freshly boiled and cooled and add 2 drops of phenolphthalein TS: no red color develops.

(2) ***Chloride***—Shake thoroughly 0.8 g of Carboxyme-thylcellulose Calcium with 50 mL of water, dissolve in 10 mL of sodium hydroxide TS, add water to make 100 mL and use this solution as the test stock solution. Heat 20 mL of the test stock solution with 10 mL of dilute nitric acid on a water-bath until a flocculent precipitate is produced. After cooling, centrifuge and take the clear supernatant liquid. Wash the precipitate three times with 10 mL volumes of water by centrifuging each time, combine the clear supernatant liquid and washings and add water to make 100 mL. Take 25 mL of this solution and add 6 mL of dilute nitric acid, water to make 50 mL and use this solution as the test solution. Perform the test. Prepare the control solution with 0.40 mL of 0.01mol/L hydrochloric acid (not more than 0.36 %).

(3) ***Sulfate***—Heat 10 mL of the test stock solution obtained in (2) with 1 mL of hydrochloric acid on a water-bath until a flocculent precipitate is produced. Cool, centrifuge and take out the clear supernatant liquid. Wash the precipitate three times with 10 mL volumes of water by centrifuging each time, combine the clear supernatant liquid and the washings and add water to make 100 mL. Take 25 mL of this solution, Perform the test using 25 mL of this solution as the test solution and prepare the control solution with 0.42 mL of 0.005mol/L sulfuric acid. Add 1 mL of 3 mol/L hydrochloric acid and 3 mL each of brium chloride TS to the test solution and the standard solution, add water to make 50 mL, and mix. Allow to stand for 10 minutes, and compare the turbidity. The turbidity from the test solution is not more dense than the turbidity from the control solution (not more than 1.0 %).

(4) ***Heavy metals***—Proceed with 1.0 g of Carboxymethylcellulose Calcium according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 20 ppm).

(5) ***Arsenic***—Proceed with 0.5 g of Carboxylmethylcellulose Sodium according to Method 1 and perform the test (not more than 4 ppm).

(6) ***Lead***—Weigh accurately 5.0 g of Carboxylmethylcellulose Sodium and transfer to a platinum crucible. Dry, carbonize and incinerate at 450 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 supplement, 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, 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 solution as directed under Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 2.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

Loss on Drying Not more than 10.0 % (1 g, 105 °C, 4 hours).

Residue on Ignition 10.0 ~ 20.0 % (after drying, 1 g)

Containers and Storage ***Containers*—**Tight containers.

Carboxymethylcellulose Sodium

Carmellose Sodium
CMC Sodium

[*9004-32-4*]

Carboxymethylcellulose Sodium is the sodium salt of a polycarboxymethylether of cellulose. Carboxymethylcellulose Sodium, when dried, contains not less than 6.5 % and not more than 8.5 % of sodium (Na: 22.99).

Description Carboxymethylcellulose Sodium appears as white to yellow powder or granules and has no taste.
Carboxymethylcellulose Sodium is practically insoluble in methanol, in ethanol, in acetic acid (100) or in ether.
Carboxymethylcellulose Sodium forms a viscid solution in water or warm water.
Carboxymethylcellulose Sodium is hygroscopic.

Identification (1) Dissolve 2.0 g of Carboxymethylcellulose Sodium in 20 mL of warm water with stirring, cool and use this 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: a red-purple color develops.

(2) To 10 mL of the test solution obtained in test (1), add 1 mL of cupric sulfate TS: a blue flocculent precipitate is produced.

(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 Qualitative Tests for sodium salt.

pH Add 1.0 g of Carboxymethylcellulose Sodium in small volumes to 100 mL of warm water with stirring, dissolve and cool: the pH of this solution is between 6.5 and 8.0.

Viscosity Weigh an amount of Carboxymethylcellulose Sodium, equivalent to 2.00 g calculated on the dried basis. Add slowly to 50 mL of water while stirring well using a stirrer. If necessary for the preparation of a low-viscosity substance, dilute to the corresponding concentration. Heat slowly to about 90 °C while stirring, 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 Determination at 20 °C: not less than 75.0 % and not more than 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. This is used 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. Dissolve 1.0 g of Carboxymethylcellulose Sodium in 100 mL of water, pour this solution into the outer tube and place on a piece of white paper on which 15 parallel black line, 1 mm in width and 1 mm in interval are drawn. Moving the inner tube up and down observing from the upper part, determine the height of the solution up to the lower edge of the inner tube when the distinction of the lines becomes impossible. Repeat the operation 3 times and calculate the mean value: it is not larger than that calculated from the same operation, using the following control solution.

Control solution—Take 5.50 mL of 0.005mol/L sulfuric acid, add 1 mL of dilute hydrochloric acid, 5 mL of ethanol and add water to make 50 mL. Add 2 mL of barium chloride TS, mix well and allow to stand for 10 minutes. Shake well this solution before use.

(2) ***Chloride***—Dissolve 0.5 g of Carboxymethylcellulose Sodium in 50 mL of water and use this solution as the test stock solution. Shake 10 mL of the test solution with 10 mL of dilute nitric acid, heat to produce a flocculent precipitate on a water-bath, cool and centrifuge. Separate the clear supernatant liquid, wash the precipitate three times with 10 mL volumes of water, centrifuging each time, combine the clear supernatant liquid and the washings, add water to make 200

mL and use 50 mL of this solutions as the test solution. Perform the test. Prepare the control solution with 0.45 mL of 0.01mol/L hydrochloric acid (not more than 0.640 %).

(3) ***Sulfate***—Shake throughly 1 mL of hydrochloric acid to 10 mL of the test stock solution obtained in (2), heat to produce a flocculent precipitate on a water-bath, cool and centrifuge. Separate the clear supernatant liquid, wash the precipitate three times with 10 mL volumes of water, centrifuging each time, combine the clear supernatant liquid and the washings and add water to make 50 mL. Take 10 mL of this solution, add water to make 50 mL and use this solution as the test solution. Perform the test. Prepare the control solution with 0.40 mL of 0.005 mol/L sulfuric acid (not more than 0.960 %).

(4) ***Silicate***—Weigh accurately about 1 g of Carbox-ymethylcellulose Sodium, ignite in a platinum crucible, add 20 mL of dilute hydrochloric acid, cover with a watch glass and boil gently for 30 minutes. Remove the watch glass and evaporate on a water-bath to dryness with the aid of a current of air. Continue heating for further 1 hour, add 10 mL of hot water, stir well and filter through a filter paper for quantitative analysis. Wash the residue with hot water, dry together with the filter paper after no turbidity is produced on the addition of silver nitrate TS to the last washing and then ignite to constant weight: the residue is not more than 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 standard lead solution (not more than 20 ppm).

(6) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat and place 10 to 300 mg of Carboxylmethylcellulose Sodium on top. Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. 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 sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 to 200 ng.

Additive—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***—Weigh accurately 5.0 g of Carboxyl-methylcellulose Sodium and transfer to a platinum crucible. Dry, carbonize and incinerate at 450 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 supplement, 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, 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 5 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 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 Carboxyl-methylcellulose Sodium and transfer to a platinum crucible. Dry, carbonize and incinerate at 450 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 supplement, 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, 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 solution as directed under Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 2.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(9) ***Arsenic***—Take 1.0 g of Carboxymethylcellulose Sodium, add 20 mL of nitric acid, heat gently until it becomes fluid, cool, add 5 mL of sulfuric acid and heat until white fumes are evolved. Cool, if necessary, add 5 mL of nitric acid and heat again. Repeat this operation until the solution becomes colorless or pale yellow. After cooling, add 15 mL of a saturated solution of ammonium oxalate and heat until white fumes are evolved again, cool and add water to make 25 mL. Take 5 mL of this solution and use this solution as the test solution and perform the test. The solution has no more color than the following standard stain.

Standard stain—Without using Carboxymethylcellulose Sodium, proceed in the same manner, then transfer 5 mL of this solution to a generator bottle, add exactly 2 mL of standard arsenic solution and proceed as directed for the test with the test solution (not more than 10 ppm).

(10) ***Starch***—Add step-wise 2 drops of iodine TS to 10 mL of the test solution obtained in (2): no blue color develops.

Loss on Drying Not more than 10.0 % (1 g, 105 °C, 4 hours).

Assay Weigh accurately about 0.5 g of Carboxymethylcellulose Sodium, previously dried, add 80 mL of acetic acid (100), connect with a reflux condenser and heat on an oil bath maintained at 130 °C for 2 hours. Cool and titrate with 0.1 mol/L perchloric acid (potentiometric titration). Perform a blank determination and make any necessary correction.

Each mL of 0.1mol/L perchloric acid
= 2.2990 mg of Na

Containers and Storage ***Containers*—**Tight containers.

Carboxymethylcellulose Sodium Tablets

Carmellose Sodium Tablets
CMC Sodium Tablets

Carboxymethylcellulose Sodium Tablets contain equivalent to not less than 6.5 % and not more than 9.5 % of the labeled amount of carboxymethylcellulose sodium (Na: 22.99).

Method of Preparation Prepare as directed under Tablets, with Carboxymethylcellulose Sodium.

Identification Dissolve a quantity of powdered Carboxymethylcellulose Sodium Tablets, equivalent to about 1 g of Carboxymethylcellulose Sodium, in 50 mL of water and filter: the filtrate responds to the following tests.

(i) Take 30 mL of the filtrate and add 3 mL of hydrochloric acid: a white precipitate is produced.

(ii) Take a volume of the fitrate and add an equal volume of barium chloride TS: a fine, white precipitate is formed.

(iii) The filtrate obtained from (i) responds to the Qualitative Tests for sodium salt.

Disintegration Test It meets the requirement, provided that the time limit is 2 hours.

Uniformity of Dosage Units It meets the requirement.

Assay Weigh accurately not less than 20 Carboxymethylcellulose Sodium Tablets and powder. Weigh accurately a volume of the powder, equivalent to about 0.5 g of Carboxymethylcellulose Sodium, add 80 mL of acetic acid (100), heat the mixture on a water-bath for 2 hours, cool and titrate with 0.1 mol/L perchloric acid (potentiometric titration, End point Detection Method in Titrimetry). Perform a blank determination and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid
= 2.2990 mg of Na

Containers and Storage ***Containers*—**Tight containers.

Carnauba Wax

Cera Carnauba

Carnauba Wax is the wax obtained from the leaves of Copernicia cerifera Mart (Palmae).

Description Carnauba Wax is pale yellow to pale

brown, hard and brittle mass or white to pale yellow powder. Carnauba Wax has a slight, characteristic odor and is tasteless.
Carnauba Wax is practically insoluble in water, ethanol, ether or xylene.

Specific gravity— d_{20}^{20} : 0.990 ~ 1.002.

Melting point—80 ~ 86 °C.

Saponification Value 78 ~ 95. Weigh accurately about 3 g of Carnauba Wax in a 300-mL flask, add 25 mL of xylene and dissolve by warming. To this solution, add 50 mL of ethanol and 25 mL of 0.5 mol/L potassium hydroxide-ethanol VS and proceed as directed in the Saponification value under the Fats and Fatty Oils. The time of heating should be 2 hours and the titration should be done by warming.

Acid Value Not more than 10.0. Use a mixture of xylene and ethanol (2 : 1) as a solvent.

Iodine Value 5 ~ 14 (Dissolve Carnauba Wax in by shaking a glass-stoppered flask in warm water).

Purity ***Heavy metals*—**Proceed with 1.0 g of Carnauba Wax according to Method 2 under Heavy Metals Limit Test and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 20 ppm).

Residue on Ignition Weigh accurately about 2 g of Carnauba Wax, place in a porcelain or platinum dish and heat: it volatizes without emitting an acrid odor. Ignite to constant mass: the weight of the residue is not more than 5 mg (not more than 0.25 %).

Containers and Storage ***Containers*—**Well-closed containers.

Castor Oil

Oleum Ricini

Castor Oil is the fixed oil obtained by compression from the seeds of *Ricinus Communis* Linné (Euphorbiaceae).

Description Castor Oil is a colorless or pale yellow, clear, viscous oil, has a slight, characteristic odor and has bland at first and afterwards slightly acrid taste.
Castor Oil is miscible with dehydrated ethanol or ether.
Castor Oil is freely soluble in ethanol and practically insoluble in water.
When cooled to 0 °C, Castor Oil becomes more viscous and turbidity is gradually formed.

Identification Take 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 176 ~ 187.

Specific Gravity d_{25}^{25} : 0.953 ~ 0.965.

Acid Value Not more than 1.5.

Hydroxyl Value 155 ~ 177.

Iodine Value 80 ~ 90.

Purity (1) ***Adulteration***—Shake to mix 1.0 g of Castor Oil with 4.0 mL of ethanol: it dissolves clearly. Add 15 mL of ethanol more: no turbidity is produced.

(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 standard lead solution (not more than 10 ppm).

(3) ***Peroxide value***—Weigh accurately 5 g of Castor Oil, transfer to a stoppered conical 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 solution of potassium iodide, stopper the flask, allow to stand for 1 minute, shake continuously and add 30 mL of water. Titrate with 0.01 mol/L sodium thiosulfate VS until the blue color of the solution disappears after addition of 0.5 mL of starch TS near the end point where the solution is a pale yellow color. Perform a blank determination and make any necessary correction (not more than 0.1 mL of 0.01 mol/L sodium thiosulfate VS is consumed by the blank solution). Calculate the amount of peroxide by the following formula: not more than 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 test
V_0: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed in the blank
W: Amount (g) of Castor Oil taken

Containers and Storage ***Containers*—**Tight containers.

Cellacefate

Cellulose Acetate Phthalate

[*9004-38-0*]

Cellacefate is a reaction product of phthalic anhydride and partially acetylated cellulose. Cellacefate contains not less than 21.5 % and not more than 26.0 % of acetyl group (-$COCH_3$: 43.05) and not less than 30.0 %

and not more than 40.0 % of carboxybenzoyl group (-COC_6H_4COOH: 149.12), calculated on the anhydrous and free acid-free basis.

Description Cellacefate appears as white powder or granules.
Cellacefate is freely soluble in acetone and practically insoluble in water, in methanol or in dehydrated ethanol.

Identification Determine the infrared spectra of Cellacefate and Cellacefate RS as directed in the potassium bromide disk method under the Infrared Spectrophotometry : both spectra exhibit similar intensities of absorption at the same wavenumbers.

Viscosity Weigh accurately a portion of Cellacefate, equivalent to 15 g calculated on the anhydrous basis, dissolve in 85 g of a mixture of acetone and water (249: 1 in mass) and perform the test at 25 ± 0.2 °C as directed in Method 1 under the Viscosity Determination to obtain the kinematic viscosity ν. Separately, determine the density, ρ, of Cellacefate as directed under the Determination of Specific Gravity and Density and calculate the viscosity, η, as $\eta = \rho\nu$: not less than 45 mPa·s and not more than 90 mPa·s.

Purity (1) ***Heavy metals***—Proceed with 2.0 g of Cellacefate according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 10 ppm).

(2) ***Free acids***—Weigh accurately about 3.0 g of Cellacefate, put in a glass-stoppered Erlenmeyer flask, add 100 mL of diluted methanol (1 in 2), stopper tightly and filter after shaking for 2 hours. Wash both the flask and residue twice with 10 mL volumes 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 the blank determination with 120 mL of diluted methanol (1 in 2) and make any necessary correction: the amount of free acids is not more than 3.0 %, calculated as phthalic acid ($C_8H_6O_4$: 166.13).

$$\text{Amount (\%) of free acids} = \frac{0.8306 \times A}{W}$$

A : Amount (mL) of 0.1 mol/L sodium hydroxide consumed,
W : Amount (g) of Cellacefate taken, calculated on the anhydrous basis.

Water Not more than 5.0 % (1 g, volumetric titration, direct titration, using a mixture of dehydrated methanol and dichloromethane (3 : 2) instead of methanol for water determination).

Residue on Ignition Not more than 0.1 % (1 g)

Assay (1) ***Carboxybenzoyl group***—Weigh accurately about 1.0 g of Cellacefate, dissolve in 50 mL of a mixture of ethanol and acetone (3:2) and titrate with 0.1 mol/L sodium hydroxide VS (indicator: 2 drops of phenolphthalein TS). Perform a blank determination and make any necessary correction.

$$\text{Amount (\%) of carboxybenzoyl group}(C_8H_5O_3) = \frac{\dfrac{1.491 \times A}{W} - 1.795 \times B}{100 - B} \times 100$$

A : Amount (mL) of 0.1 mol/L sodium hydroxide consumed
B : Amount (%) of free acids obtained in the Free acids under the Purity
W : Amount (g) of Cellacefate taken, calculated on the anhydrous basis.

(2) ***Acetyl group***—Weigh accurately about 0.1 g of Cellacefate, place in a glass-stoppered Erlenmeyer flask, add 50 mL of water and exactly 25 mL of 0.1 mol/L sodium hydroxide VS and boil for 30 minutes under a reflux condenser. After cooling, add 5 drops of phenolphthalein TS and titrate with 0.1 mol/L hydrochloric acid VS. Perform a blank determination and make any necessary correction.

$$\text{Amount (\%) of free acids and bound acetyl group } (C_2H_3O) = \frac{0.4305 \times A}{W}$$

A : Amount(mL) of 0.1 mol/L sodium hydroxide consumed, corrected after the blank determination
W : Amount(g) of Cellacefate taken, calculated on the anhydrous basis

$$\text{Amount (\%) of acetyl group}(C_2H_3O) = \frac{100 \times (P - 0.5182 \times B)}{(100 - B)} - 0.5772 \times C$$

B : Amount (%) of free acids obtained in the Free acids under the Purity
C : Content (%) of carboxybenzoyl group
P : Content (%) of free acids and bound acetyl group (C_2H_3O)

Containers and Storage ***Containers—***Tight containers.

Microcrystalline Cellulose

[*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 is a white crystalline powder having fluidity.
Microcrystalline Cellulose swells with sodium hydroxide TS on heating.
Microcrystalline Cellulose is practically insoluble in water, in ethanol or in ether.

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. Place about 10 mg of Microcrystalline Cellulose on a watch glass and disperse in 2 mL of this solution: the substance develops a blue-violet color.

(2) Sieve 20 g of Microcrystalline Cellulose for 5 minutes on an air-jet sieve equipped with a screen (No. 400 and about 20 cm in internal diameter). If more than 5 % is retained on the screen, mix 30 g of Microcrystalline Cellulose with 270 mL of water: otherwise, mix 45 g with 255 mL of water. Perform the mixing for 5 minutes in a high-speed (18000 revolutions per minute or more) power blender. Transfer 100 mL of the dispersion to a 100-mL mass cylinder and allow to stand for 3 hours: a white, opaque, bubble-free dispersion, which does not form a supernatant liquid at the surface, is obtained.

(3) Transfer 1.3 g of Microcrystalline Cellulose, accurately weighed, to a 125-mL Erlenmeyer flask and add 25.0 mL each of water and 1 mol/L cupriethylenediamine TS. Immediately purge the solution with nitrogen, insert the stopper and shake on a suitable mechanical shaker to dissolve. Perform the test with this solution according to Method 1 under the Viscosity Determination using a capillary viscometer having the viscometer constant (K), 0.03, at 25 ± 0.1 °C and determine the kinematic viscosity, ν. Separately, perform the test with a mixture of 25.0 mL each of water and 1 mol/L cupriethylenediamine TS in the same manner as above, using a capillary viscometer having *K*, 0.01 and determine the kinematic viscosity, ν_0.

Calculate the relative viscosity, η_{rel}, of Microcrystalline Cellulose by the formula:

$$\eta_{rel} = \frac{\nu}{\nu_0}$$

Obtain the product, $[\eta]\cdot C$, of limiting viscosity $[\eta]$ (mL/g) and concentration *C* (g/100 mL) from the value η_{rel} of the Table shown somewhere below. When calculated the degree of polymerization, *P*, by the following formula, *P* is not more than 350 and within the labeled range.

$$P = \frac{95[\eta]C}{\text{amount (g) of the test calculated on the dried basis}}$$

pH Shake 5.0 g of Microcrystalline Cellulose with 40 mL of freshly boiled and cooled water for 20 minutes and centrifuge: the pH of the clear supernatant liquid is between 5.0 and 7.0.

Purity (1) ***Heavy metals***—Proceed with 2.0 g of Microcrystalline Cellulose according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 10 ppm).

(2) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat, place 10 mg to 300 mg of Microcrystalline Cellulose on top. For a liquid sample, allow 0.1 mL to 0.5 mL to completely permeate additive (a). 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, place only the test specimen in a nickel boat without the additives. Place the boat inside the furnace and heat to about 900 °C with a current of air or oxygen at 0.5 L/minute to 1 L/minute. Elute the mercury and sample in a sampling tube. Transfer the mercury vapor to a cold vapor atomic absorption spectrometer by heating the sampling 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 plot a calibration curve from the absorbances. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 ng to 200 ng.

Additive—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.

(3) ***Cadmium***—Weigh accurately 5.0 g of Microcrystalline Celluluose 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 5 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 1.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Cadmium hollow cathode lamp
Wavelength: 228.8 nm

(4) ***Lead***—Use the test solution obtained in (3). Transfer 1.0 mL of standard lead 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 Atomic Absorption Spectrophotometery according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 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 Microcrystalline Cellulose according to Method 3 and perform the test (not more than 4 ppm).

(6) ***Water-soluble substances***—Shake 5.0 g of Microcrystalline Cellulose with 80 mL of water for 10 minutes, filter with the aid of vacuum through filter paper into a vacuum flask. Evaporate the clear filtrate in a beaker, previously weighed, to dryness without charring, dry at 105 °C for 1 hour, cool in a desiccator (silica gel) and weigh: the residue is not more than 12.5 mg. Perform a blank determination and make any necessary correction.

(7) ***Ether-soluble substances***—Place 10.0 g of Microcrystalline Cellulose in a column, about 20 mm in inner diameter, and pass 50 mL of peroxide-free ether through the column. Evaporate the eluate to dryness in a previously dried and tared evaporation dish. Dry the residue at 105 °C for 30 minutes, allow to cool in a desiccator, and weigh accurately: the residue is not more than 5.0 mg. Perform a blank determination and make any necessary correction.

(8) ***Starch***—To 30 g of Microcrystalline Cellulose, add 270 mL of water and mix at about 3000 rpm for 5 minutes. To 30 mL of this solution, add 2 drops of iodine TS: blue-purple to blue color is not observed.

Conductivity (1) ***Standard potassium chloride solution***—Weigh accurately 0.744 g of powdered potassium chloride, previously dried at 500 °C to 600 °C for 4 hours, and dissolve in water at 20 ± 1 °C to make exactly 1000 mL. Take exactly 100 mL of this solution, add water at 20 ± 1 °C to make exactly 1000 mL and determine the conductivity: the conductivity constant of this solution, χ_{KCl}, at 25 °C is 146.9 μS·cm^{-1}.

(2) ***Apparatus***—Use an appropriate conductivity meter having the cell constant of between 0.01 and 0.1 cm^{-1}. Usually, the conductivity meter consists of detector and indicator. The detector consists of a cell including electrodes in it. The cell with a temperature compensation circuit is preferable.

(3) ***Procedure***—Wash 2 to 3 times the cell, previously washed well with water, with standard potassium chloride solution and fill up with the standard potassium chloride solution. Determine the conductivity and the standard potassium chloride solution is kept at 25 ± 0.1 °C. Replace the standard potassium chloride solution, repeat the determination in same manner and measure the conductivity of the standard potassium chloride solution, G_{x_0} (μS), after a stable reading of ± 3 % is obtained. The cell constant, J, is calculated by the following:

$$J = \frac{x_{KCl} + x_{H_2O}}{G_{x_0}}$$

J : cell constant (cm^{-1}).

x_{KCl} : conductivity constant of the potassium chloride conductivity calibration standard solution (μS·cm^{-1}) (25°C)

x_{H_2O} : conductivity constant of water used for preparation of the potassium chloride conductivity calibration standard solution (μS·cm^{-1}) (25°C),

G_{x_0} : conductivity measured (μS).

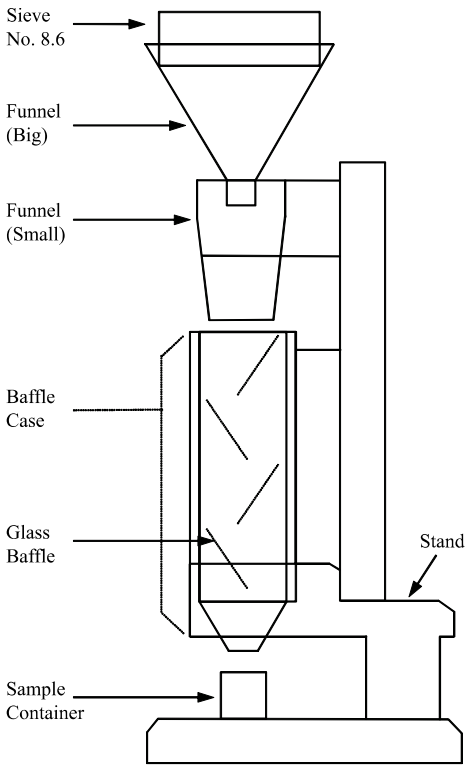
Use the clear supernatant liquid obtained in the pH test as the test solution. After washing well the cell with water, rinse the cell with the test solution 2 to 3 times, fill up with the test solution and determine the conductivity of the test solution, G_T (μS), kept at 25 ± 0.1 °C. Determine the conductivity of water used for the preparation of the test solution, G_0 (μS), in the same manner as above and calculate the conductivity constants, x_T (μS·cm^{-1}) and x_0 (μS·cm^{-1}), by the following expressions: the value $x_T - x_0$, is not more than 75 μS·cm^{-1}.

$$x_T \ (\mu S \cdot cm^{-1}) = JG_T$$
$$x_0 \ (\mu S \cdot cm^{-1}) = JG_0$$

Loss on Drying Not more than 7.0 % and within a range as specified on the label (1 g, 105 °C, 3 hours).

Residue on Ignition Not more than 0.1 % (2 g).

Bulk Density (1) ***Apparatus***—Use a volumeter shown in the figure.



Put a No. 8.6 sieve (2000 μm) on top of the volumeter. A funnel is mounted over a baffle box, having four glass baffle plates inside which the sample powder slides as it passes. At the bottom of the baffle box is a funnel that collects the powder, and allows it to pour into a sample receiving cup mounted directly below it.

(2) ***Procedure***—Weigh accurately the mass of a brass or stainless steel cup, which has a capacity of 25.0 ± 0.05 mL and an internal diameter of 30.0 ± 2.0 mm in inner diameter, and put the cup directly below the funnel of the volumeter. From a position apart 5.1 cm from the top edge of the funnel, slowly pour Microcrystalline Cellulose through the sieve, at a rate suitable to prevent clogging, until the cup overflows. Level the excess powder with the aid of a slide glass, weigh the filled cup and weigh accurately the content of the cup and then calculate the bulk density by the following expression: the bulk density is within the labeled specification.

$$\text{Bulk density (g/cm}^3\text{)} = \frac{A}{25}$$

A: Measured mass of the content of the cup (g)

Microbial Limit When tested, the total aerobic microbial count is not more than 1000 CFU/g, the total combined yeasts/mould count is not more than 100 CFU/g, and *Escherichia coli*, *Salmonella* species, *Staphylococcus aureus* and *Pseudomonas aeruginosa* are not observed.

Containers and Storage ***Containers***—Tight contaiers.

Table for Conversion of Relative Viscosity (η_{rel}) into the Product of Limiting Viscosity and Concentration ($[\eta]C$)

η_{rel}	$[\eta]$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

[*9004-34-6*, Cellulose]

Powdered Cellulose is purified, mechanically disintegrated α-cellulose obtained as a pulp from fibrous plant materials.
The label indicates the mean degree of polymerization value with the range.

Description Powdered Cellulose is a white powder.
Powdered Cellulose is practically insoluble in water, in ethanol or in ether.

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. Place about 10 mg of Powdered Cellulose on a watch glass and disperse in 2 mL of this solution: the substance develops a blue-purple color.

(2) Mix 30 g of Powdered Cellulose with 270 mL of water in a high-speed (18000 revolutions per minute or more) blender for 5 minutes, transfer 100 mL of the dispersion to a 100 mL mass cylinder and allow to stand for 1 hour: a supernatant liquid appears above the layer of the cellulose and the supernatant liquid produces a precipitate.

(3) Weigh accurately about 0.25 g of Powdered Cellulose, transfer to a 125 mL Erlenmeyer flask, add 25.0 mL each of water and 1 mol/L cupriethylenediamine TS and proceed as directed in the Identification (3) under Microcrystalline Cellulose : the mean degree of polymerization, *P*, is not less than 440 and is within the labeled specification.

pH Mix 10 g of Powdered Cellulose with 90 mL of freshly boiled and cooled water and allow to stand for 1 hour with occasional stirring: the pH of the clear supernatant liquid is between 5.0 and 7.5.

Purity (1) ***Heavy metals***—Proceed with 2.0 g of Powdered Cellulose according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 10 ppm).

(2) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat and place 10 mg to 300 mg of Powdered Cellulose on top. Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. Place the boat inside the furnace and heat to about 900 °C with a current of air or oxygen at 0.5 L/minute to 1 L/minute. Elute the mercury and sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 ng to 200 ng.

Additive—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.

(3) ***Cadmium***—Weigh accurately 5.0 g of Powdered Cellulose 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 5 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 1.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Cadmium hollow cathode lamp
Wavelength: 228.8 nm

(4) ***Lead***—Use the test solution obtained in (3). Transfer 1.0 mL of standard lead 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 Atomic Absorption Spectrophotometery according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 2.0 ppm).

Gas: Acetylene or hydrogen – Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(5) ***Arsenic***—Proceed with 1.0 g of Microcrystalline Cellulose according to Method 3 and perform the test (not more than 2 ppm).

(6) ***Water-soluble substances***—Mix 6.0 g of Powdered Cellulose with 90 mL of freshly boiled and cooled water and allow to stand for 10 minutes with occasional stirring. Filter with the aid of vacuum, discard the first 10 mL of the filtrate and pass the subsequent filtrate through the same filter, if necessary, to obtain a clear filtrate. Evaporate a 15.0 mL volumes of the filtrate in an evaporation dish, previously weighed, to dryness without charring, dry at 105 °C for 1 hour and cool in a desiccators : the residue is not more than 15.0 mg. Perform a blank determination and make any necessary correction.

(7) ***Ether-soluble substances***—Place 10.0 g of Powdered Cellulose in a column, about 20 mm in inner diameter, and pass 50 mL of peroxide-free ether through the column. Evaporate the eluate to dryness in an evaporation dish, previously dried and weighed dry at 105 °C for 30 minutes and cool in a desiccator : the residue is not more than 15.0 mg. Perform a blank determination and make any necessary correction.

(8) ***Chloride***—Weigh accurately about 5 g of Powdered Cellulose, transfer to a 500 mL conical flask, add 250 mL of water, reflux for 1 hour and filter. To the filtrate, add 200 mL of water, reflux for 30 minutes and filter. Combine this filtrate with the preceding filtrate and the filtrate obtained by washing the residue with warm water. Add 1 mL of nitric acid and heat to boil. Add slowly 5 mL of 5 % silver nitrate solution and after the precipitate coagulates, filter with a glass filter. Wash with nitric acid (1 in 100) until silver nitrate is not detected, wash with water, dry at 130 °C and weigh. To determine the exact value of the precipitate, perform a blank determination and make any necessary correction. Convert 1 mg of the precipitate to 0.247 mg Cl: not more than 0.05 %.

Loss on Drying Not more than 6.5 % (1 g, 105 °C, 3 hours).

Residue on Ignition Not more than 0.3 % (1 g, cal-

culated on the dried basis).

Microbial Limit The total aerobic microbial count is not more than 1000 CFU/g, the total combined yeasts/mould count is not more than 100 CFU/g and *Escherichia coli*, *Salmonella* species, *Staphylococcus aureus* and *Pseudomonas aeruginosa* are not observed.

Containers and Storage ***Containers*—**Tight containers.

Cetanol

Cetanol is a mixture of solid alcohols and consists chiefly of cetanol ($C_{16}H_{34}O$: 242.44).

Description Cetanol appears as unctuous, white flakes, granules, or masses. Cetanol has a faint, characteristic odor and is tasteless.
Cetanol is very soluble in pyridine, freely soluble in ethanol, in dehydrated ethanol or in ether, very slightly soluble in acetic anhydride and practically insoluble in water.

Melting Point 47 ~ 53 °C. Proceed as directed in the Melting Point under Stearyl Alcohol.

Acid Value Not more than 1.0.

Hydroxyl Value 210 ~ 232.

Ester Value Not more than 2.0.

Iodine Value Not more than 2.0.

Purity Proceed as directed in the Purity under Stearyl Alcohol.

Residue on Ignition Not more than 0.05 % (2 g).

Containers and Storage ***Containers*—**Well-closed containers.

Chlorinated Lime

Chlorinated Lime contains not less than 30.0 % of available chlorine (Cl: 35.45).

Description Chlorinated Lime is a white powder and has a chlorine-like odor.
Chlorinated Lime dissolves partially in water. The solution changes red litmus paper to blue, then gradually decolorizes.

Identification (1) Add dilute hydrochloric acid to Chlorinated Lime: a gas, which has the odor of chlorine, evolves and the gas changes moistened starch-potassium iodide paper to blue.

(2) Shake 1 g of Chlorinated Lime with 10 mL of water and filter: the filtrate responds to the Qualitative Tests (2) and (3) for calcium salt.

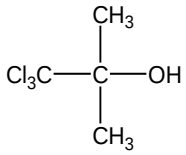
Assay Weigh accurately about 5.0 g of Chlorinated Lime, transfer to a mortar and triturate thoroughly with 50 mL of water. Transfer to a 500-mL volumetric flask with the aid of water and add water to make 500 mL. Add 10 mL of dilute hydrochloric acid and titrate the liberated iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 3 mL of starch TS). Perform a blank determination and make any necessary correction.

Each mL of 0.1 mol/L sodium thiosulfate VS
= 3.5453 mg of Cl

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light-resistant, and in a cold place.

Chlorobutanol



$C_4H_7Cl_3O$: 177.46

1,1,1-Trichloro-2-methylpropan-2-ol [*57-15-8*]

Chlorobutanol contains not less than 98.0 % and not more than 101.0 % of chlorobutanol ($C_4H_7Cl_3O$), calculated on the anhydrous basis.

Description Chlorobutanol appears as colorless or white crystals and has camphor-like odor.
Chlorobutanol is very soluble in methanol, in ethanol or in ether and slightly soluble in water.
Chlorobutanol slowly volatilize in air.

Melting point—Not lower than about 76 °C.

Identification (1) Take 5 mL of a solution of Chlorobutanol (1 in 200), add 1 mL of sodium hydroxide TS, then slowly add 3 mL of iodine TS: a yellow precipitate is produced and the odor of iodoform is perceptible.

(2) Take 0.1 g of Chlorobutanol, add 5 mL of sodium hydroxide TS, shake well the mixture, add 3 to 4 drops of aniline and warm gently: the disagreeable odor of phenyl isocyanide (poisonous) is perceptible.

Purity (1) ***Acid***—Shake thoroughly 0.10 g of the pulverized Chlorobutanol with 5 mL of water: 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. Perform the test. Pre-

pare the control solution with 1.0 mL of 0.01 mol/L hydrochloric acid VS by adding 25 mL of dilute ethanol, 6 mL of dilute nitric acid and add water to make 50 mL (not more than 0.071 %).

Water Not more than 6.0 % (0.2 g, volumetric titration, direct titration).

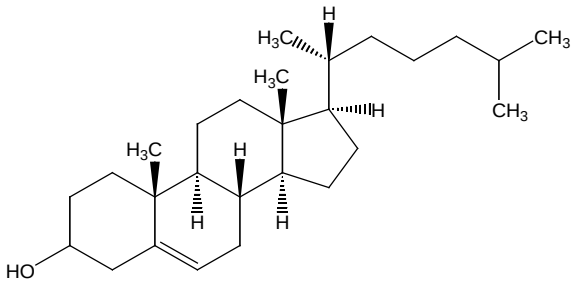
Residue on Ignition Not more than 0.1 % (1 g).

Assay Weigh accurately about 0.1 g of Chlorobutanol, transfer to a 200-mL Erlenmeyer flask and dissolve in 10 mL of ethanol. Add 10 mL of sodium hydroxide TS, boil under a reflux condenser for 10 minutes, cool, add 40 mL of dilute nitric acid and 25 mL of 0.1 mol/L silver nitrate VS and shake well. Add 3 mL of nitrobenzene and shake vigorously until the precipitate is coagulated. Titrate the excess silver nitrate with 0.1 mol/L ammonium thiocyanate VS (indicator: 2 mL of ferric ammonium sulfate TS). Perform a blank determination and make any necessary correction.

Each mL of 0.1 mol/L silver nitrate VS
= 5.915 mg of $C_4H_7Cl_3O$

Containers and Storage ***Containers*—**Tight containers.

Cholesterol



$C_{27}H_{46}O$: 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 appears as white to pale yellow crystals or granules, is odorless or has a slight odor and is tasteless.
Cholesterol is freely soluble in chloroform or in ether, soluble in dioxane, sparingly soluble in dehydrated ethanol and practically insoluble in water,
Cholesterol gradually changes to a yellow to pale yellow-brown color by light.

Identification (1) Dissolve 10 mg of Cholesterol in 1 mL of chloroform, add 1 mL of sulfuric acid and shake: a red color develops in the chloroform layer and the sulfuric acid layer shows a green fluorescence.

(2) Dissolve 5 mg of Cholesterol in 2 mL of chloroform, add 1 mL of acetic anhydride and 1 drop of sulfuric acid and shake: a red color is produced and it changes to green through blue.

Specific Optical Rotation $[\alpha]_D^{25}$: -34 ~ -38 ° (after drying, 0.2 g, dioxane, 10 mL, 100 mm)

Melting Point 147 ~ 150 °C.

Purity **(1)** ***Clarity of solution***—Place 0.5 g of Cholesterol in a glass-stoppered flask, dissolve in 50 mL of warm ethanol and allow to stand at room temperature for 2 hours: no turbidity or deposit is produced.

(2) ***Acid***—Place 1.0 g of Cholesterol in a flask, dissolve in 10 mL of ether, add 10.0 mL of 0.1 mol/L sodium hydroxide VS and shake for 1 minute. Evaporate the ether and boil for 5 minutes. Cool, add 10 mL of water and titrate with 0.05 mol/L sulfuric acid VS (indicator: 2 drops of phenolphthalein TS). Perform a blank determination and make any necessary correction: the volume of 0.1 mol/L sodium hydroxide VS consumed is not more than 0.30 mL.

Loss on Drying Not more than 0.3 % (1 g, in vacuum, 60 °C, 4 hours).

Residue on Ignition Not more than 0.1 % (1 g).

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light-resistant.

Cinnamon Oil

Cinnamon Oil is the essential oil distilled with steam from the leaves and twigs or bark of *Cinnamomum cassia* Blume or from the bark of *Cinnamomum zeylanicum* Nees (*Lauraceae*). Cinnamon Oil contains not less than 60 vol % of total aldehydes and not more than 4.0 % of coumarin ($C_9H_6O_2$: 146.14).

Description Cinnamon Oil is a yellow to brown liquid, has a characteristic, aromatic odor and a sweet, pungent taste.
Cinnamon Oil is clearly miscible with ethanol, ethanol or ether.
Cinnamon Oil is practically insoluble in water.
Upon aging or long exposure to air, Cinnamon Oil darkens and becomes viscous.

Specific gravity— d_{20}^{20} : 1.010 ~ 1.065.

Identification Shake 4 drops of Cinnamon Oil with 4 drops of nitric acid: the mixture forms white to pale yellow crystals at a temperature below 5 °C.

Purity **(1)** ***Rosin***—Mix 1.0 mL of Cinnamon Oil with 5 mL of ethanol, then add 3 mL of freshly prepared, saturated ethanol solution of lead acetate: no precipitate is produced.

(2) ***Heavy metals***—Proceed with 1.0 mL of Cinnamon Oil according to Method 2 and perform the test. Prepare the control solution with 4.0 mL of standard lead solution (not more than 40 ppm).

Assay (1) ***Total aldehydes***—Pipet 5.0 mL of Cinnamon Oil into a cassia flask, add 70 mL of sodium bisulfite TS and heat the mixture on a water-bath with frequent shaking to dissolve completely. To this solution, add sodium bisulfite TS to raise the lower level of the oily layer within the graduate volume of the neck. Allow to stand for 2 hours and measure the volume (mL) of the separated oily layer.

Total aldehydes (vol %)
= [5.0 – (volume of separated oily layer)] × 20

(2) ***Coumarin***—Take a suitable volume of Cinnamon Oil and use as the test solution. Separately, weigh 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 hydrogen flame ionization detector

Column: A capillary column about 0.25 mm in internal diameter and 60 m in length, coated with polyethylene glycol for gas chromatography 20M.

Column temperature: Maintain at 60 °C for 10 minutes, raise the temperature by 2 °C per minute until 190 °C and maintain at 190 °C for 85 minutes.

Injection port temperature: A constant temperature of about 200 °C

Detector temperature: A constant temperature of about 240 °C

Carrier gas: Helium

Flow rate: 1.5 mL/minute

Containers and Storage ***Containers***—Tight containers.

Storage—Light-resistant.

Clove Oil

Clove Oil is the volatile oil distilled with steam from the flower buds or leaves of Syzygium aromaticum Merrill et Perry (Myrtaceae). Clove Oil contains not less than 80.0 vol % of total eugenol.

Description Clove Oil is colorless or pale yellow-brown, clear liquid. It has a characteristic aroma and burning taste.
Clove Oil is miscible with ethanol or with ether.
Clove Oil is slightly soluble in water.
Clove Oil acquires a brown color upon aging or by air.

Identification (1) Take 5 drops of Clove Oil, add 10 mL of calcium hydroxide TS and shake vigorously: the oil forms a flocculent mass and a white to pale yellow color develops.

(2) Dissolve 2 drops of Clove Oil in 4 mL of ethanol and add 1 to 2 drops of ferric chloride TS: a green color is produced.

Refractive Index n_D^{20} : 1.527 ~ 1.537.

Specific Optical Rotation $[\alpha]_D^{20}$: 0 ~ -1.5° (100 mm)

Specific Gravity d_{20}^{20} : 1.040 ~ 1.068.

Purity **(1)** ***Clarity 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 phenol***—Take 1.0 mL of Clove Oil, add 20 mL of boiling water, shake vigorously, filter the aqueous layer after cooling and add 1 to 2 drops of ferric chloride TS: a yellow-green, but no blue to violet, color develops.

(3) ***Heavy metals***—Proceed with 1.0 mL of clove Oil according to Method 2, and perform the test. Prepare the control solution with 4.0 mL of Standard Lead Solution (not more than 40 ppm).

Assay Take 10.0 mL of Clove Oil in a Cassia flask, add 70 mL of sodium hydroxide TS, shake for 5 minutes, warm for 10 minutes on a water-bath with occasional shaking, add sodium hydroxide TS to the volume after cooling and allow to stand for 18 hours. Measure the volume (mL) of the separated oily layer.

Total eugenol (%)
= [10 - (volume of separated oily layer)] × 10

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light-resistant.

Coconut Oil

Coconut oil is the fixed oil obtained from the seeds of *Cocos nucifera* Linné (Palmae).

Description Coconut Oil is a white to pale yellow

mass, or colorless or pale yellow, clear oil, has a slight, characteristic odor and mild taste.
Coconut Oil is freely soluble in ether and in petroleum ether. Coconut Oil is practically insoluble in water.
At a temperature below 15 °C, Coconut Oil congeals to a hard and brittle solid.

Melting point—20 ~ 28 °C (Method 2).

Saponification Value 246 ~ 264.

Unsaponifiable Matter Not more than 1.0 %.

Acid Value Not more than 0.2.

Iodine Value 7 ~ 11.

Purity (1) ***Peroxide value***—Weigh accurately 5 g of Coconut Oil, transfer to a stoppered 250 mL conical 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 and add 30 mL of water. Titrate with 0.01 mol/L sodium thiosulfate VS until the blue color of the solution disappears after addition of 5 mL of starch TS near the end point where the solution is a pale yellow color. Perform a blank determination and make any necessary correction (not more than 0.1 mL of 0.01 mol/L sodium thiosulfate VS is consumed by the blank solution). Calculate the amount of peroxide by the following formula: not more than 5.

$$\text{Peroxide value (mEq/kg)} = [10 \times (V_1 - V_0)] / W$$

V_1: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed in the test
V_0: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed in the blank
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, allow to stand and titrate until the clear supernatant liquid becomes yellow in color: not more than 0.1 mL of 0.01 mol/L hydrochloric acid TS is consumed.

Containers and Storage ***Containers*—**Tight containers.

Corn Oil

Corn Oil is the fixed oil obtained from the embryo of *Zea mays* Linné (Gramineae).

Description Corn Oil is a clear, pale yellow oil, is odorless or has a slight odor and mild taste.
Corn Oil is miscible with ether or with petroleum ether. Corn Oil is slightly soluble in ethanol and practically insoluble in water.
At -7 °C, Corn Oil congeals to an unguentary mass.

Specific gravity—d_{25}^{25} : 0.915 ~ 0.921.

Saponification Value 187 ~ 195.

Unsaponifiable Matter Not more than 1.5 %.

Acid Value Not more than 0.2.

Iodine Value 103 ~ 130.

Purity ***Heavy metals***—Proceed with 1.0 g of Corn Oil according to Method 2 and perform the test. Prepare the control solution with 1.0 mL of standard lead solution (not more than 10 ppm).

Containers and Storage ***Containers***—Tight containers.

Corn Starch

Corn Starch is a starch granules obtained from the seeds of *Zea mays* Linné (Gramineae).

Description Corn Starch is a white to pale yellow mass or powder.
Corn Starch is practically insoluble in water or in dehydrated ethanol.

Identification (1) Under a microscope, Corn Starch, preserved in a mixture of water and glycerin (1 : 1), appears as irregularly polygonal simple grains of about 2 to 23 μm in diameter, or irregularly orbicular or spherical simple grains of about 25 to 35 μm in diameter. Hilum appears as distinct cave or 2 to 5 radial clefts; concentric striation absent. Corn Starch is observed a black cross, its intersection point on hilum when grains are put between two nicol prisms fixed at right angle to each other.

(2) To 1 g of Corn Starch add 50 mL of water, boil for 1 minute, and allow to cool: a subtle 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 deep blue color is formed and the color disappears by heating.

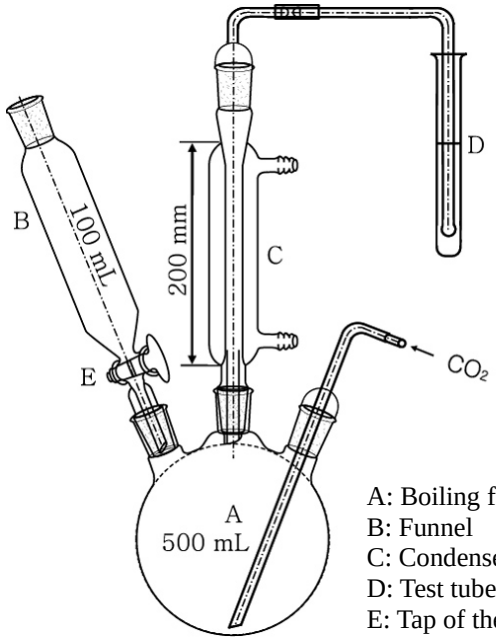
pH Put 5.0 g of Corn Starch in a non-metal vessel, add 25.0 mL of freshly boiled and cooled water, mix gently for 1 minute, and allow to stand for 15 minutes: the pH of the solution is between 4.0 and 7.0.

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 iron stand-

ard solution, add water to make 20 mL and use this solution as the control solution. Put 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 mercapto acetic acid and mix. Alkalize with strong ammonia water to litmus paper, 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 the solutions against a white background: the color of the test solution is not more intense than that of the control solution (not more than 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 liquid, add 1 mL of acetic acid (100) and 0.5 g to 1.0 g of potassium iodide, shake and allow 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 determination and make any necessary correction: not more than 1.4 mL of 0.002 mol/L sodium thiosulfate VS is consumed (not more than 20 ppm, calculated as hydrogen peroxide).

(3) ***Sulfur dioxide***—(i) Apparatus: Use apparatus shown in the figure.



A: Boiling flask
B: Funnel
C: Condenser
D: Test tube
E: Tap of the funnel

(ii) Procedure: Introduce 150 mL of water into the boiling flask, close the tap of the funnel, and pass carbon dioxide through the whole system at a rate of 100 ± 5 mL per minute. Pass cooling water through the condenser, and place 10 mL of hydrogen peroxide-sodium hydroxide TS (to a 9 : 1 mixture of water and hydrogen peroxide (hydrogen peroxide TS), add 3 drops of bromophenol blue TS and add 0.01 mol/L sodium hydroxide TS until the color changes to purple-blue; prepare before use) in the test-tube. After 15 minutes, remove the funnel without interrupting the stream of carbon dioxide, and introduce through the opening into the flask about 25 g of Corn Starch, accurately weighed, with the aid of 100 mL of water. Apply tap grease to the outside of the connection part of the funnel, and connect the funnel. Close the tap of the funnel, pour 80 mL of 2 mol/L hydrochloric acid TS into the funnel, open the tap to introduce the hydrochloric acid into the flask, and close the tap while several mL of the hydrochloric acid remains, in order to avoid losing sulfur dioxide. Place the flask in a water-bath, and heat the mixture for 1 hour. Transfer the contents of the test-tube with the aid of a little water to a wide-necked conical flask. Heat in a water-bath for 10 minutes, and cool. Add 0.1 mL of bromophenol blue TS, and titrate with 0.1 mol/L sodium hydroxide VS until the color changes from yellow to purple-blue lasting for at least 20 seconds. Perform a blank determination and make any necessary correction. Calculate the amount of sulfur dioxide by applying the following formula: it is not more than 50 ppm.

$$\text{Amount (ppm) of sulfur dioxide} = \frac{\text{Amount (mL) of 0.1 mol/L sodium hydroxide VS consumed}}{\text{Amount (g) of Corn Starch taken}} \times 1000 \times 3.203$$

(4) ***Foreign matter***—Under a 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 Not more than 15.0 % (1 g, 130 °C, 90 minutes).

Residue on Ignition Not more than 0.6 % (1 g)

Microbial Limit The total aerobic microbial count is not more than 1000 CFU/g, the total combined yeasts/mould count is not more than 100 CFU/g and *Escherichia coli*, *Salmonella* species, *Staphylococcus aureus* and *Pseudomonas aeruginosa* are not observed.

Containers and Storage ***Containers***—Well-closed containers.

Creosote

Creosote is a mixture of phenols obtained from wood tar derived from dry distillation of stems and branches of various plants of genus *Pinus* (*Pinaceae*), genus *Cryptomeria* (*Taxodiaceae*), genus *Fagus* (*Fagaceae*), genus *Afzelia* (genus *Intsia*) (*Leguminosae*), genus *Shorea* (*Dipterocarpaceae*) or genus *Tectona* (*Verbenaceae*), followed by distillation and collection at 180 to 230 °C, then further purification and redistillation. Creosote contains not less than 23 % and not more than 35 % of guaiacol ($C_7H_8O_2$: 124.14).

Description Creosote is colorless or pale yellow, clear liquid. Creosote has a characteristic odor.
Creosote is slightly soluble in water. Creosote is misci-

ble with methanol or with ethanol.
A saturated solution of Creosote is acidic
Cresote is highly refractive.
Creosote gradually changes in color by light or by air.

Identification Use the test solution obtained in the Assay as the test solution. Separately, dissolve 0.1 g of phenol, *p*-cresol, guaiacol and 2-methoxy-4-methylphenol in methanol, respectively, to make 100 mL. To 10 mL of each solution, add methanol to make 50 mL and use these solutions as the standard solution of phenol, *p*-cresol, guaiacol and 2-methoxy-4-methylphenol, respectively. Perform the test with 10 μL each of the test solution and the standard solutions as directed under Liquid Chromatography according to the following operating conditions: retention times of the main peaks obtained from the test solution are the same as those obtained from the standard solutions of phenol, *p*-cresol, guaiacol and 2-methoxy-4-methylphenol.

Operating conditions

Proceed as directed in the operating conditions in the Assay.

Specific Gravity d_{20}^{20} : Not less than 1.076.

Purity (1) ***Coal creosote***—Pipet 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, dissolve in a small quantity of ethyl acetate if necessary, 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 Gas Chromatography according to the following operating conditions: no peaks are detected with the test solution for the retention times corresponding to benzo[*α*]pyrene, benz[*α*]anthracene and dibenz[*α,h*] anthracene of the standard solution. If any peak is detected for retention times that correspond to benzo[*α*]pyrene, benz[*α*]anthracene or dibenz[*α,h*]anthracene, change these conditions to verify that such a peak does not belong to benzo[*α*]pyrene, benz[*α*]anthracene or dibenz[*α,h*]anthr-acene.

Operating conditions

Detector: A high performance mass spectrophotometer (EI)

Monitored ions:

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

Injection port temperature: A constant temperature of about 250 °C

Column: A quartz tube about 0.25 mm in internal diameter and about 30 m in length, with internal coating 0.25 μm to 0.5 μm in thickness made of 5 % diphenyl-95 % dimethyl polysiloxane for gas chromatography.

Column temperature: Inject the sample at a constant temperature of about 45 °C, raise the temperature to 240 °C at the rate of 40 °C per minute, maintain the temperature at 240 °C for 5 minutes, then raise the temperature to 300 °C at the rate of 4 °C per minute, then raise the temperature to 320 °C at the rate of 10 °C per minute and maintain the temperature 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

Test for required detectability: Pipet 1 mL of the standard solution, add methanol to make exactly 10 mL and use this solution as the system suitability solution. When the procedure is run with 1 μL of this solution under the above operating conditions, the signal-to-noise ratio of each peak is not less than 3.

System performance: When the procedure is run with 1 μL of the system suitability solution under the above operating conditions, benz[*α*]anthracene, benzo[*α*] pyrene and dibenz[*α,h*]anthracene are eluted in this order.

System repeatability: When the test is repeated 6 times with 1 μL each of the system suitability solution, the relative standard deviation of each peak area of benzo[*α*]pyrene, benz[*α*]anthracene and dibenz[*α,h*]anth-racene is not more than 10.0 %.

(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. To 5 mL of this solution, add methanol to make 20 mL. Pipet 2 mL of this solution, 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 Gas Chromatography according to the following operating conditions: no peaks are detected with the test solution for the retention time corresponding to acenaphthene of the standard 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 hydrogen flame ionization detector

Column: A fused silica tube about 0.25 mm in internal diameter and about 60 m in length, with internal coating 0.25 μm to 0.5 μm in thickness made of polymethy-lsiloxane for gas chromatography.

Column temperature: Inject the sample at a constant temperature of about 45 °C, raise the temperature to 160 °C at the rate of 11.5 °C per minute, raise the tem-

perature to 180 °C at the rate of 4 °C per minute, then raise the temperature to 270 °C at the rate of 8 °C per minute and maintain the temperature at 270 °C for 3 minutes.

Injection port temperature: About 250 °C

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

Test for required detectability: Pipet 1 mL of the standard solution, add methanol to make exactly 10 mL and use this solution as the system suitability solution. When the procedure is run with 1 μL of this solution under the above oprating conditions, the signal-to-noise ratio of the peak of acenaphthene is not less than 3.

System repeatability: When the test is repeated 6 times with 1 μL each of the system suitability solution under the above operating conditions, the relative standard deviation of the peak area of acenaphthene is not more than 6.0 %.

(3) ***Other impurities***—Take 1.0 mL of creosote, add 2 mL of petroleum benzin and 2 mL of barium hydroxide TS, shake and allow to stand: no blue or muddy brown color develops in the upper layer of the mixture and no red color develops in the lower layer.

Distilling Range 200 ~ 220 °C, not less than 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 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 Liquid Chromatography according to the following operating conditions and determine the peak areas, A_T and A_S, of guaiacol in each solution.

$$\text{Amount (mg) of guaiacol } (C_7H_8O_2) = \text{Amount (mg) of Guaiacol RS taken} \times \frac{A_T}{A_S}$$

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. When the procedure is run with 10 μL of this solution under the above operating conditions, phenol and guaiacol are eluted in this order with the resolution between their peaks being not less than 2.5.

System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions, the relative standard deviation of the peak area of guaiacol is not more than 1.5 %.

Containers and Storage ***Containers***—Tight containers.

Storage—Light-resistant.

Dextrin

Description Dextrin appears as white or pale yellow, amorphous powder or granules, has a slight, characteristic odor and a sweet taste, and does not irritate the tongue.

Dextrin is freely soluble in boiling water, soluble in water and practically insoluble in ethanol or in ether.

Identification Take 0.1 g of Dextrin, add 100 mL of water, shake and filter, if necessary. To 5 mL of the filtrate, add 1 drop of iodine TS: a pale red-brown or pale red-purple color develops.

Purity (1) ***Clarity and color of solution***—Take 2.0 g of Dextrin in a Nessler tube and 40 mL of water, dissolve by heating, cool and add water to make 50 mL: the solution is colorless or pale yellow, clear and even if turbid, the turbidity is not more than that of the following control solution.

Control solution—Take 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 before use.

(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: a red color develop.

(3) ***Chloride***—Take 2.0 g of Dextrin, add 80 mL of water, dissolve by heating, cool, add water to make 100 mL and filter. Take 40 mL of the filtrate and add 6 mL of dilute nitric acid and add water to make 50 mL. Perform the test. Prepare the control solution with 0.30 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.013 %).

(4) ***Sulfate***—Take 45 mL of the filtrate obtained in (3), add 1 mL of dilute hydrochloric acid and add water to make 50 mL and perform the test. Prepare the con-

trol solution with 0.35 mL of 0.005 mol/L sulfuric acid VS (not more than 0.019 %).

(5) ***Oxalate*—**Take 1.0 g of Dextrin, add 20 mL of water, dissolve by heating, cool, add 1 mL of acetic acid and filter. To 5 mL of the filtrate, add 5 drops of calcium chloride TS: no turbidity is produced immediately.

(6) ***Reducing sugars***—To 2.0 g of Dextrin, add 100 mL of water, mix for 30 minutes, add water to make 200 mL, filter and use the filtrate as test solution (A). To 10 mL of Fehling's TS, add 20 mL of the test solution, mix, heat to boil within 3 minutes, heat for 2 minutes and cool. Add 5 mL of potassium iodide (3 in 10) and 10 mL of 2 mol/L sulfuric acid, mix and titrate with 0.1 mol/L sodium thiosulfate (indicator: starch). Separately, proceed with 20 mL of anhydrous dextrose solution (1 in 1000) as test solution (B) in the same manner and perform a blank determination: it meets the following requirements (corresponds to 10 % dextrose).

$$(V_B - V_U) \leqq (V_B - V_S)$$

V_B: Volume (mL) of 0.1 mol/L sodium thiosulfate consumed in the blank determination

V_U: Volume (mL) of 0.1 mol/L sodium thiosulfate consumed with test solution (A)

V_S: Volume (mL) of 0.1 mol/L sodium thiosulfate consumed with test solution (B)

(7) ***Calcium*—**Take 5 mL of the filtrate obtained in (5), add 5 drops of ammonium oxalate TS: no turbidity is immediately produced.

(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 standard lead solution (not more than 20 ppm).

(9) ***Lead***—Weigh accurately 5.0 g of Dextrin 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 0.5 mL of standard lead 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 1.0 ppm).

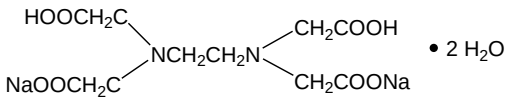
Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

Loss on Drying Not more than 10 % (0.5 g, 105 °C, 4 hours).

Residue on Ignition Not more than 0.5 % (0.5 g).

Containers and Storage ***Containers*—**Well-closed containers.

Disodium Edetate Hydrate



Disodium Ethylenediaminetetraacetate
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 contains not less than 99.0 % and not more than 101.0 % of disodium edetate hydrate ($C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O$).

Description Disodium Edetate Hydrate appears as white crystals or crystalline powder, is odorless and has a slightly acidic taste.
Disodium Edetate Hydrate is soluble in water and practically insoluble in ethanol or in ether.

Identification (1) Dissolve 10 mg of Disodium Edetate Hydrate in 5 mL of water, add 2 mL of a solution of potassium chromate (1 in 200) and 2 mL of arsenic trioxide TS and heat on a water-bath for 2 minutes: a purple color develops.

(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. Collect the precipitate, wash with 50 mL of water and dry at 105 °C for 1 hour: the precipitate melts between 240 °C and 244 °C (with decomposition).

(3) A solution of Disodium Edetate Hydrate (1 in 20) responds to the Qualitative Tests (1) for sodium salt.

pH Dissolve 1.0 g of Disodium Edetate Hydrate in 100 mL of water: the pH of this solution is between 4.3 and 4.7.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Disodium Edetate Hydrate in 50 mL of water:

the solution is clear and colorless.

(2) ***Cyanide***—Transfer 1.0 g of Disodium Edetate Hydrate to a round-bottomed flask, dissolve in 100 mL of water, add 10 mL of phosphoric acid and distil. Place 15 mL of 0.5 mol/L sodium hydroxide VS in a 100-mL measuring cylinder, which is used as a receiver and immerse the top end of the condenser into the solution. Distil the mixture until the distillate measures 100 mL and use this solution as the test solution. Transfer 20 mL of the test solution to a glass-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 to stand for a few minutes. Mix well with 5 mL of pyridine-pyrazolone TS and allow to stand between 20°C and 30 °C for 50 minutes: the solution is not more intense than the following control solution.

Control solution—Pipet exactly 1.0 mL of standard cyanide solution, add 15 mL of 0.5 mol/L sodium hydroxide VS and add water to make exactly 1000 mL, transfer 20 mL of this solution to a glass-stoppered test tube and proceed as directed for the test solution.

(3) ***Heavy metals***—Proceed with 2.0 g of Disodium Edetate Hydrate according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 10 ppm).

(4) ***Lead***—Weigh accurately 5.0 g of Disodium Edetate Hydrate 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 mL 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 supplement, 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, 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 solution as directed under Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 2.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(5) ***Arsenic***—Prepare the test solution with 1.0 g of Disodium Edetate Hydrate according to Method 1 and perform the test (not more than 2 ppm).

(6) ***Nitrilotriacetic acid***—Weigh accurately 1 g of Disodium Edetate Hydrate, dissolve in copper (II) nitrate to make exactly 100 mL and use this solution as the test solution. Separately, weigh accurately 0.1 g of Nitrilo-triacetic Acid RS, add 0.5 mL of ammonia water, add water to make exactly 10 mL and use this solution as the standard stock solution. Weigh accurately 1.0 g of disodium ethylenediaminetetraacetate, add 100 μL of the standard stock solution, add copper (II) nitrate 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 Liquid Chromatography according to the following operating conditions and determine each peak area by the automatic integration method: the peak area of nitrilotriacetic acid in the test solution is not larger than that of nitrilotriacetic acid in the standard solution (not more than 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 octasilyl 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/minute

System suitability

System performance: Weigh 10 mg of disodium ethylenediaminetetraacetate, add 100 μL of the standard solution and add copper (II) nitrate to make exactly 100 mL. When the procedure is run with 50 μL of this solution, the resolution between the peaks of nitrilotriacetic acid and copper is not less than 3. The relative retention times of nitrilotriacetic acid and copper with respect to the retention time of disodium ethylenediaminetetra-acetate are 0.35 and 0.65, respectively.

Residue on Ignition 37.0 ~ 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 until the color of the solution changes from blue to red. (Indicator: 40 mg of eriochrome black T-sodium chloride indicator)

Each mL of 0.1mol/L zinc VS
= 37.224 mg of $C_{10}H_{14}N_2Na_2O_8.2H_2O$

Containers and Storage ***Containers*—**Well-closed containers.

Ethanol

CH_3CH_2OH

Alcohol C_2H_6O: 46.07

Ethanol [*64-17-5*]

Ethanol contains not less than 95.1 vol % and not more than 96.9 vol % (by specific gravity) of ethanol (C_2H_6O) at 15 °C.

Description Ethanol is clear, colorless liquid, has a characteristic odor and burning taste.
Ethanol is miscible with water.
Ethanol is flammable and burns with a pale blue flame on ignition.
Ethanol is volatile.

Identification Determine the infrared spectra of Ethanol and Ethanol RS, previously dried, as directed in the liquid film method disk method under the Infrared Spectrophotometry, respectively: both spectra exhibit similar intensities of absorption at the same wavenumbers.

Specific Gravity d_{15}^{15} : 0.809 ~ 0.816.

Purity (1) ***Clarity of solution***—Ethanol is clear and colorless. To 1.0 mL of Ethanol add water to make 20 mL, and allow to stand for 5 minutes: the resulting liquid is clear.

(2) ***Acid or alkali*** –To 20 mL of Ethanol add 20 mL of freshly boiled and cooled water and 0.1 mL of a solution prepared by addition of 7.0 mL of ethanol and 20 mL of water to 1.0 mL of phenolphthalein: it is colorless. Add 1.0 mL of 0.01 mol/L sodium hydroxide VS to this solution: a pale red color develops.

(3) ***Volatile impurities***—Pipet exactly 500 mL of Ethanol, add 150 μL of 4-methylpentan-2-ol, and use this solution as the sample solution. Seperately, to 100 μL of purified methanol add Ethanol to make exactly 50 mL. Pipet exactly 5 mL of this solution, add Ethanol to make exactly 50 mL, and use this solution as the standard solution (1). Separately, to exactly 50 μL each of purified methanol and acetaldehyde add Ethanol to make exactly 50 mL. To exactly 100 μL of this solution add Ethanol to make exactly 10 mL, and use this solution as the standard solution (2). Separately, to exactly 150 μL of acetal add Ethanol to make exactly 50 mL. To exactly 100 μL of this solution add Ethanol to make exactly 10 mL, and use this solution as the standard solution (3). Separately, to exactly 100 μL of benzene add Ethanol to make exactly 100 mL, pipet 100 μL of this solution, add Ethanol to make exactly 50 mL and use this solution as the standard solution (4). Perform the test with exactly 1μL each of the sample solution and the standard solutions (1), (2), (3) and (4) as directed under Gas Chromatography according to the following conditions, and determine the peak areas of acetaldehyde, A_E, benzene, B_E and acetal, C_E obtained with Ethanol, and the peak area of methanol with the standard solution (1), the peak area of aldehyde, A_T with the standard solution (2), the peak area of acetal, C_T with the standard solution (3) and the peak area of benzene, B_T: the peak area of methanol is not more than 1/2 times the peak area of methanol with the standard solution (1). When calculate the amounts of the volatile impurities by the following equation, the sum of acetaldehyde and acetal as acetaldehyde is not more than 10 vol ppm, and the amount of benzene is not more than 2 vol ppm. The total area of the peaks other than the peak mentioned above and the peak having the area not more than 3 % of 4-methylpentan-2-ol is not more than the peak area of 4-methylpentan-2-ol.

Total amount (vol ppm) of acetaldehyde and acetal

$$= \frac{10A_E}{A_T - A_E} + \frac{30C_E}{C_T - C_E}$$

Amount (vol ppm) of benzene = $\frac{2B_E}{B_T - B_E}$

If necessary, identify the peak of benzene by using a different stationary liquid phase and suitable chromatographic conditions.

Operation conditions

Detector: A hydrogen flame-ionization detector

Column: A fused silica tube 0.32 mm in internal diameter and 30 m in length, coated with 6 % cyanopropyl phenyl-94 % dimethyl silicone polymer for gas chromatography in 1.8 μm thickness.

Column temperature: Inject at a constant temperature of about 40 °C, maintain the temperature for 12 minutes, then raise up to 240 °C at the rate of 10 °C per minute, and maintain at a constant temperature of about 240 °C for 10 minutes.

Carrier gas: Helium

Flow rate: 35 cm/sec

Split ratio: about 1 : 20

System suitability

System performance : When the procedure is run with 1 μL of the standard solution (2) under the above operating conditions, acetaldehyde and methanol are eluted in this order with the resolution between these peaks being not less than 1.5.

(4) ***Other impurities*** **(absorbance)—**Perform the test with Ethanol as directed under Ultraviolet-visible Spectrophotometry 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, between 250 nm and 260 nm and between 270 nm and

340 nm are not more than 0.40, 0.30 and 0.10, respectively, and the absorption curve is flat.
(5) ***Residue on evaporation***—Pipet exactly 100 mL of Ethanol, evaporate in a tared dish on a water bath, and dry for 1 hour at 105 °C: the mass of the residue does not exceed 2.5 mg.

Containers and Storage *Containers*—Tight containers. ***Storage*—**Light-resistant, and remote from fire.

Anhydrous Ethanol

CH_3CH_2OH

Dehydrated Ethanol
Dehydrated Alcohol C_2H_6O: 46.07

Ethanol [*64-17-5*]

Anhydrous Ethanol contains not less than 99.5 vol % (by specific gravity) of ethanol (C_2H_6O) at 15 °C.

Description Anhydrous Ethanol is a clear, colorless liquid.
Anhydrous Ethanol is miscible with water.
Anhydrous Ethanol is flammable and burns with a pale blue flame on ignition.
Anhydrous Ethanol is volatile.
Boiling point—78 ~ 79 °C.

Identification Proceed as directed in the Identification under Ethanol.

Specific Gravity d_{15}^{15} : 0.794 ~ 0.797

Purity Proceed as directed in the Purity under Ethanol

Containers and Storage *Containers*—Tight containers. ***Storage*—**Light-resistant, and remote from fire.

Ether

$CH_3CH_2OCH_2CH_3$

Diethyl ether $C_4H_{10}O$: 74.12
Ethoxyethane [*60-29-7*]

Ether contains not less than 96.0 % and not more than 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 is a colorless, clear, mobile liquid, has a characteristic odor.
Ether is miscible with ethanol.
Ether is soluble in water.
Ether is highly volatile and flammable.
Ether is slowly oxidized by the action of air and light, with the formation of peroxides.
Vapor of Ether, when mixed with air and ignited, may explode violently.
Boiling point—35 ~ 37 °C.

Specific Gravity d_{20}^{20} : 0.718 ~ 0.721.

Purity (1) ***Foreign odor***—Place 10 mL of Ether in an evaporation dish, and allow to evaporate spontaneously to a volume of about 1 mL: no foreign odor is perceptible. Drop this residue onto a piece of clean, odorless filter paper to evaporate the ether: no foreign odor is perceptible.
(2) ***Acid*—**Place 10 mL of diluted ethanol (4 in 5) and 0.5 mL of phenolphthalein TS in a glass-stoppered flask, and add 0.02 mol/L sodium hydroxide VS dropwise to produce a red color which persists after shaking for 30 seconds. Add 25 mL of Ether, stopper the flask, shake gently, and add 0.40 mL of 0.02 mol/L sodium hydroxide VS with shaking: a red color develops.
(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, protecting from light, with occasional shaking: no color is produced in the ether layer and the aqueous layer.
(4) ***Peroxide*—**Place 10 mL of Ether in a Nessler tube, add 1 mL of freshly prepared solution of potassium iodide (1 in 10), shake for 1 minute, then add 1 mL of starch TS, and shake well: no color is produced in the ether layer and in the aqueous layer.
(5) ***Residue on evaporation*—**Evaporate 140 mL of Ether, and dry the residue at 105 °C for 1 hour: the residue is not more than 1.0 mg.

Containers and Storage *Containers*—Tight containers. ***Storage*—**Light-resistant, without fill up, remote from fire, and not exceeding 25 °C.

Ethylenediamine

$H_2NCH_2CH_2NH_2$

$C_2H_8N_2$: 60.10

Ethylenediamine contains not less than 97.0 % and not more than 101.0 % of ethylenediamine ($C_2H_8N_2$).

Description Ethylenediamine is clear, colorless to pale yellow liquid, has an ammonia-like odor.
Ethylenediamine is miscible with water, with ethanol or with ether.
Ethylenediamine has a caustic nature and an irritating property.
Ethylenediamine is gradually affected by air.

Specific gravity—d_{20}^{20} : About 0.898.

Identification (1) A solution of Ethylenediamine (1 in 500) is alkaline.

(2) Take 2 mL of cupric sulfate TS and add 2 drops of Ethylenediamine: a blue-purple color develops.

(3) Take 40 mg of Ethylenediamine, add 6 drops of benzoyl chloride and 2 mL of a solution of sodium hydroxide (1 in 10), warm for 2 to 3 minutes with occasional shaking, collect 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 is between 247 °C and 251 °C.

Purity (1) ***Heavy metals***—Place 1.0 g of Ethylenediamine in a porcelain crucible, evaporate to dryness on a water-bath, cover loosely, ignite at a low temperature until charred, proceed according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 20 ppm).

(2) ***Residue on evaporation***—Pipet exactly 5 mL of Ethylenediamine, heat on a water-bath to dryness and dry to a constant weight at 105 °C: the residue is not more than 3.0 mg.

Distilling Range 114 ~ 119 °C, not less than 95 vol %.

Assay Weigh accurately about 0.7 g of Ethylenediamine in a glass-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 $C_2H_8N_2$

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light-resistant, and almost well-filled.

Eucalyptus Oil

Eucalyptus Oil is the essential oil distilled with steam from the leaves of *Eucalyptus globulus* Labillardiere or allied plants (Myrtaceae). Eucalyptus Oil contains not less than 70.0 % of cineole ($C_{10}H_{18}O$: 154.25).

Description Eucalyptus Oil is clear, colorless or pale yellow liquid, has a characteristic, aromatic odor and pungent taste.
Eucalyptus Oil is neutral.

Identification Shake 1 mL of Eucalyptus Oil vigorously with 1 mL of phosphoric acid and allow to stand: the solution congeals within 30 minutes.

Refractive Index n_D^{20} : 1.458 ~ 1.470.

Specific Gravity d_{20}^{20} : 0.907 ~ 0.927.

Purity (1) ***Clarity 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*—**Proceed with 1.0 mL of Eucalyptus Oil according to Method 2, and perform test. Prepare the control solution with 4.0 mL of Standard Lead Solution (not more than 40 ppm).

(3) ***Phenol***—To 5 mL of Eucalyptus Oil, add 5 mL of sodium hydroxide TS and shake: the volume of eucalyptus oil does not decrease.
To 1 mL of Eucalyptus Oil, add 20 mL of water, shake well and allow to stand to separate the layers. Take 10 mL of the water layer and add 1 drop of iron (II) chloride: the solution does not exhibit a purple color.

(4) ***Aldehyde***—Put 10 mL of Eucalyptus Oil in 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 (60 v/v % ethanol) until the color of the solution changes from red to yellow and the pale yellow color of the indicator persists in the lower layer even after 2 minutes of vigorous shaking. Add 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, not more than 2.0 mL of a 0.5 mol/L solution of potassium hydroxide in ethanol (60 v/v % ethanol) is consumed.

Assay Weigh accurately about 0.1 g of Eucalyptus Oil and dissolve in hexane to make exactly 25 mL. Pipet exactly 5 mL of this solution, add exactly 5 mL of the internal standard solution, then add hexane to make exactly 100 mL and use this solution as the test solution. Separately, weigh accurately about 0.1 g of Cineol RS, proceed as directed in the test solution 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 Gas Chromatography according to the following operating conditions. Calculate the ratios, Q_T and Q_S, of the peak area of cienol to that of the internal standard of each solutions, for the test solution and the standard solution, respectively.

$$\text{Amount (mg) of Cienol } (C_{10}H_{18}O) = \text{amount (mg) of Cienol RS} \times \frac{Q_T}{Q_S}$$

Internal standard solution—A solution of anisol in hexane (1 in 250).

Operating conditions

Detector: A hydrogen flame-ionization detector.

Column: A glass column, about 3 mm in internal diameter and about 5 m in length, having alkylene glycol phthalate ester for gas chromatography coated at the ratio of 10 % on silanized siliceous earth 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 cineol is about 11 minutes.

System suitability

System performance: Dissolve 0.1 g each of cineol and limonene in 25 mL of hexane. To 1 mL of this solution, add hexane to make 20 mL. When the procedure is run with 2 μL of the standard solution under the above operating conditions, limonene and cineol are eluted in this order with a resolution between their peaks being not less than 1.5.

Containers and Storage ***Containers***—Tight containers.

Storage—Light-resistant.

Fennel Oil

Oleum Foeniculi

Funnel Oil is the essential oil distilled with steam from the fruit of *Foeniculum vulgare* Miller (Umbelliferae) or of *Illicium verum* Hooker fil. (Illiciaceae).

Description Fennel Oil is colorless to pale yellow liquid. Fennel Oil has a characteristic, aromatic odor and sweet taste with slightly bitter aftertaste.
Fennel Oil is miscible with ethanol or with ether.
Fennel Oil is practically insoluble in water.
When cold, white crystals or crystalline masses may often separate from the 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. Perform the test with the test solution as directed under Thin-layer Chromatography. Spot 5 μL of the test solution on a plate of silicagel with a fluoroscenti indicator for thin-layer chromatography. 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 under ultraviolet light (mail wavelength : 254 nm) : a main spot with a dark purple color appears at the R_f value of about 0.4.

Refractive Index n_D^{20} : 1.528 ~ 1.560.

Specific Gravity d_{20}^{20} : 0.955 ~ 0.995.

Purity (1) ***Clarity of solution***—Take 1.0 mL of Fennel Oil and add 3 mL of ethanol: the solution is clear. To this solution, add 7 mL of ethanol: the solution remains clear.

(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 standard lead solution (not more than 40 ppm).

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light-resistant

Formalin

Formalin contains not less than 35.0 % and not more than 38.0 % of formaldehyde (CH_2O: 30.03). Formalin contains 5 % to 13 % of methanol to prevent polymerization.

Description Formalin is a clear and colorless liquid. Vapor is irritating to the mucous membrane.
Formalin is miscible with water or with ethanol.
When stored for a long time, especially in a cold place, Formalin may become cloudy.

Identification (1) Dilute 2 mL of Formalin with 10 mL of water in a test tube and add 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 droops of Formalin to 5 mL of sulfuric acid in which 0.1 g of salicylic acid has been dissolved, and warm the solution: a persistent, dark red color develops.

Purity (1) ***Acid***—Dilute 20 mL of Formalin with 20 mL of water and add 5.0 mL of 0.1 mol/L sodium hydroxide VS and 2 drops of bromothymol blue TS: a blue color develops.

(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 Gas Chromatography according to the following operating conditions and determine the ratios, Q_T and Q_S, of the peak area of methanol with respect to that of the internal standard (between 5 % and 13 %).

$$\text{Content (\%) of methanol} = \frac{\text{Amount of methanol} \times \dfrac{Q_T}{Q_S}}{\text{Amount of Formalin taken}} \times 100$$

Internal standard solution—Dilute 10 mL of anhydrus ethanol with water to make 100 mL.

Operating conditions

Detector: A hydrogen 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 ethylben-zene-divinylbenzene copolymer for gas chromatography (150 μm to 180 μm in particle diameter).

Column temperature: 120 °C

Injection port temperature: 150 °C

Carrier gas: Nitrogen

Flow rate: 30 ~ 40 mL/minute

System suitability

System performance: When the procedure is run with 1 μL of the standard solution under the above operating conditions, the resolution between the peaks of methanol and ethanol is not less than 2.0.

Residue on Ignition Not more than 0.06 % (5 mL, after evaporation)

Assay Weigh accurately 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 exactly 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 determination and make any necessary correction.

Each mL of 0.05 mol/L iodine VS
= 1.5013 mg of CH_2O

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light-resistant.

Gelatin

Gelatin is a product prepared from aqueous extract of raw collagen by heating. The raw collagen is obtained by acid or alkali treatment of the bone, skin, ligament or tendon of animals.

Description Gelatin appears as colorless or white to pale yellow-brown sheets, shreds, granules or powder, is odorless and tasteless.

Gelatin is very soluble in hot water and practically insoluble in ethanol or in ether.

Gelatin does not dissolve in water, but slowly swells and softens when immersed in it, gradually absorbing water, 5 to 10 times its own mass.

Gelatin derived from an acid-treated collagen exhibits an isoelectric point between pH 7.0 and 9.0 and Gelatin derived from an alkali-treated collagen exhibits an isoelectric point between pH 4.5 and 5.0.

Identification (1) Take 5 mL of a solution of Gelatin (1 in 100) and add chromium trioxide TS or picric acid TS drop-wise: a precipitate is produced.

(2) Take 5 mL of a solution of Gelatin (1 in 5000) and add tannic acid TS drop-wise: the solution becomes turbid.

Purity (1) ***Foreign odor and water-insoluble substances*—**Dissolve 1.0 g of Gelatin in 40 mL of water by heating: the solution has no disagreeable odor, is clear, or only slightly opalescent and has no more color than Color Matching Fluid A.

(2) ***Sulfite*—**Take 20.0 g of Gelatin in a round-bottomed 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. Attach a condenser, immediately distil the solution, immersing the end of the condenser into a receiver containing 50 mL of iodine TS and continue the distillation until 50 mL of distillate is obtained. Acidify the distillate with hydrochloric acid dropwise, add 2 mL of barium chloride TS and heat on a water-bath until the color of iodine TS is discharged. Collect the precipitates, wash with water and ignite: the residue is not more than 4.5 mg, but the residue obtained from Gelatin for use in the preparation of capsules and tablets is not more than 75 mg. Perform a blank determination and make any necessary correction.

(3) ***Heavy metals*—**Proceed with 0.5 g of Gelatin according to Method 2 and perform the test. Prepare the control solution with 2.5 mL of standard lead solution (not more than 50 ppm).

(4) ***Mercury*—**Place 2.0 g of Gelatin in a decomposition flask, add 20 mL of diluted sulfuric acid (1 in 2) and 100 mL of a solution of potassium permanganate (3 in 50), heat gently under a reflux condenser and boil for 2 hours. If the solution becomes clear during boiling, reduce the temperature of the solution to about 60 °C, add further 5 mL of a solution of potassium permanganate (3 in 50), boil again and repeat the above-mentioned procedure until the precipitate of manganese dioxide remains for about 20 minutes. Cool, add a solution of hydroxylamine hydrochloride (1 in 5) until the precipitate of manganese dioxide disappears, add water to make exactly 150 mL and use the solution as the test solution. Perform the test as directed under the Atomic Absorption Spectrophotometry (cold vapor type). Place the test solution in a test bottle of the atomic absorption spectrophotometer, add 10 mL of stannous chloride-sulfuric acid TS, connect the bottle immediately to the atomic absorption spectrophotometer and circulate air. Determine the absorbance, A_T, of the test solution at 253.7 nm when the indication of the recorder has risen rapidly and become constant. Separately, place 2.0 mL of standard mercury solution a decomposition flask, add 20 mL of diluted sulfuric acid

(1 in 2) and 100 mL of a solution of potassium permanganate (3 in 50) and proceed in the same manner as for the test solution. Determine the absorbance, A_S, of the standard solution: A_T is not more than A_S (not more than 0.1 ppm).

(5) ***Lead***—Weigh accurately 5.0 g of Gelatin 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 mL 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 supplement, 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, 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 0.75 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 1.5 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(6) ***Chromium***—Place 5 g of Gelatin in a decomposition flask, add 50 mL of water and 10 mL of nitric acid, mix and allow to stand. Heat gently until the vigorous reaction subsides, cool, add 5 mL of sulfuric acid and heat gently. When the contents of the flask start to darken, add 2 to 3 mL volumes of nitric acid and continue heating. Decomposition is complete when the contents of the flask are pale yellow to colorless. Cool the decomposed liquid, add water to make 50 mL and use this solution as the test solution. Prepare the blank solution by repeating the same procedure. Separately, to 20 mL of chromium standard stock solution (1000 ppm), add 0.2 % nitric acid to make 200 mL. Pipet 20 mL of this solution and make 200 mL (10 μg/mL), and pipet 1 mL and 5 mL and make 10 mL each with 0.2 % nitric acid, and use these solutions as the standard solutions (1, 5, 10 ppm). Perform the test with the test solution and each standard solution as directed under electrothermal type Atomic Absorption Spectrophotometry: not more than 10 ppm.

(7) ***Arsenic***—Take 15.0 g of Gelatin in a flask, add 60 mL of diluted hydrochloric acid (1 in 5) and heat until solution is dissolved. Add 15 mL of bromine TS, heat until the excess of bromine is expelled, neutralize with ammonia TS, add 1.5 g of dibasic sodium phosphate and allow to cool. To this solution, add 30 mL of magnesia TS, allow to stand for 1 hour and collect the precipitates. Wash the precipitates five times with 10 mL volumes of diluted ammonia TS (1 in 4) and dissolve in diluted hydrochloric acid (1 in 4) to make exactly 50 mL. Perform the test with 5 mL of this solution: the solution is not more intense than the following standard stain.

Standard stain—Proceed with 15 mL of Standard Arsenic Solution, instead of Gelatin, in the same manner (not more than 1 ppm).

Loss on Drying Not more than 15.0 %. Take about 1 g of Gelatin, accurately weighed, in a weighed 200 mL beaked containing 10 g of sea sand (No.1), previously dried at 110 °C for 3 hours. Add 20 mL of water, allow to stand for 30 minutes with occasional shaking, evaporate to dryness on a water-bath with occasional shaking and dry the residue at 110 °C for 3 hours.

Residue on Ignition Not more than 2.0 % (0.5 g).

Microbial Limit The total aerobic microbial count is not more than 1000 CFU/g, the total combined yeasts/mould count is not more than 100 CFU/g and *Escherichia coli*, *Salmonella* species, *Staphylococcus aureus* and *Pseudomonas aeruginosa* are not observed.

Containers and Storage ***Containers***—Tight containers.

Purified Gelatin

Purified Gelatin is a product prepared from aqueous extract of raw collagen by heating. The raw collagen is obtained by acid or alkali treatment of the bone, skin, ligament, or tendon of animals.

Description Purified Gelatin appears as colorless to pale yellow sheets, shreds, pellets or powder and is odorless and tasteless.
Purified Gelatin is very soluble in hot water and practically insoluble in ethanol or in ether.
Purified Gelatin does not dissolve in water, but slowly swells and softens when immersed in water and absorbs water, 5 to 10 times its own mass.
Purified Gelatin derived from an acid-treated collagen exhibits an isoelectric point between pH 7.0 and 9.0 and Purified Gelatin derived from an alkali-treated collagen has an isoelectric point at pH 4.5 to 5.0.

Identification Proceed as directed in the Identification under Gelatin.

Purity (1) ***Foreign odor and Water-insoluble substances***—Dissolve 1.0 g of Purified Gelatin in 40 mL of water by heating: the solution is clear, colorless and

free from any disagreeable odor when the layer of the solution is 20 mm in depth.

(2) ***Sulfite***—Take 20.0 g of Purified Gelatin in a round-bottomed 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. Attach a condenser, immediately distil the solution, immersing the end of the condenser into a receiver containing 50 mL of iodine TS and continue the distillation until 50 mL of distillate is obtained, Acidify the distillate by dropwise addition of hydrochloric acid, add 2 mL of barium chloride TS and heat on a water-bath until the color of iodine TS is discharged. Collect the precipitates, wash with water and ignite: the residue is not more than 1.5 mg. Perform a blank determination and make any necessary correction.

(3) ***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 standard lead solution (not more than 20 ppm).

(4) ***Arsenic***—Proceed as directed in the Purity Arsenic under Gelatin.

(5) ***Mercury***—Proceed as directed in the Purity Mercury under Gelatin.

Loss on Drying Not more than 15.0 %. Proceed as directed in the Loss on drying under Gelatin.

Residue on Ignition Not more than 2.0 % (0.5 g).

Microbial Limit The total aerobic microbial count is not more than 1000 CFU/g, the total combined yeasts/mould count is not more than 100 CFU/g and *Escherichia coli*, *Salmonella* species, *Staphylococcus aureus* and *Pseudomonas aeruginosa* are not observed.

Containers and Storage ***Containers*—**Tight containers.

Glyceryl Monostearate

Glyceryl Monostearate is a mixture of α- and β-glyceryl monostearate and other fatty acid esters of glycerin.

Description Glyceryl Monostearate appears as white to pale yellow, waxy masses, thin flakes, or granules, has a characteristic odor and taste.
Glyceryl Monostearate is very soluble in hot ethanol, soluble in chloroform sparingly soluble in ether and practically insoluble in water or ethanol.
Glyceryl Monostearate is slowly affected by light.

Identification (1) Heat 0.2 g of Glyceryl Monostearat with 0.5 g of potassium bisulfate until thoroughly charred: 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. This separated solid dissolves when shaken with 3 mL of ether.

Saponification Value 157 ~ 170.

Acid Value Not more than 15.

Iodine Value Not more than 3.0. Use chloroform instead of cyclohexane.

Melting Point Not less than 55 °C (Method 2).

Purity (1) ***Acidity or alkalinity***—Take 1.0 g of Glyceryl Monostearate, add 20 mL of boiling water and cool with swirling: the solution is neutral.

(2) ***Heavy metals***—Proceed with 2.0 g of Glyceryl Monostearate according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 10 ppm).

Residue on Ignition Not more than 0.10 % (1 g).

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light-resistant.

Glycine

H_2NCH_2COOH

Aminoacetic Acid $C_2H_5NO_2$: 75.07

[*56-40-6*]

Glycine, when dried, contains not less than 98.5 % and not more than 101.0 % of glycine ($C_2H_5NO_2$).

Description Glycine, appears as white crystals or crystalline powder, is odorless and has a sweet taste.
Glycine is freely soluble in water or in 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 Infrared Spectrophotometry: both spectra exhibit similar intensities of absorption at the same wavenumbers. If any difference appears between the spectra, dissolve each with 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 between 5.6 and 6.6.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Glycine in 20 mL of water: the solution is clear and colorless.

(2) ***Chloride***—Proceed with 0.5 g of Glycine and perform the test. Prepare the control solution with 0.30 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.021 %).

(3) ***Sulfate***—Proceed with 0.6 g of Glycine and perform the test, Prepare the control solution with 0.35 mL of 0.005 mol/L sulfuric acid VS (not more than 0.028 %).

(4) ***Ammonium***— Proceed with 0.25 g of Glycine and perform the test. Prepare the control solution with 5.0 mL of standard ammonium solution (not more than 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 standard lead solution (not more than 20 ppm).

(6) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat, place 10 mg to 300 mg of Glycine on top then 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, place only the test specimen in a nickel boat without the additives. Place the boat inside the combustion furnace and heat to about 900 °C with a current of air or oxygen at 0.5 L/minute to 1 L/minute. Elute the mercury and sample in a sampling tube. Transfer the mercury vapor to a cold vapor atomic absorption spectrometer by heating the sampling 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 plot a calibration curve from the absorbances. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 ng to 200 ng.

Additive—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) ***Lead***—Weigh accurately 5.0 g of Glycine 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 2.5 mL of standard lead 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 5.0 ppm).

Gas: Acetylene or hydrogen – Air
Lamp: 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 (not more than 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 the test 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. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 5 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 n-butanol, water and acetic acid (100) (3 : 1 : 1) to a distance of about 10 cm and dry the plate at 80 °C for 30 minutes. Spray evenly a solution of ninhydrin in acetone (1 in 50) and heat at 80 °C for 5 minutes: the spots other than the principal spot from the test solution are not more intense than the spot from the standard solution.

Loss on Drying Not more than 0.3 % (1 g, 105 °C, 3 hours).

Residue on Ignition Not more than 0.1 % (1 g).

Assay Weigh accurately about 0.15 g of Glycine, previously dried, 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, Endpoint Detection Method in Titrimetry). Perform a blank de-

termination and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 7.507 mg of $C_2H_5NO_2$

Containers and Storage ***Containers*—**Well-closed containers.

Exsiccated Gypsum

Exsiccated Gypsum possibly corresponds to the formula $CaSO_4 \cdot 1/2H_2O$.

Description Exsiccated Gypsum is a white to grayish white powder and is odorless and tasteless.
Exsiccated Gypsum is slightly soluble in water and practically insoluble in ethanol.
Exsiccated Gypsum 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, Exsiccated Gypsum loses its solidifying property.

Identification Shake 1 g of Exsiccated Gypsum with 20 mL of water for 5 minutes and filter: the filtrate responds to the Qualitative tests (2) and (3) for calcium salt and to the Qualitative Tests for sulfate.

Purity ***Alkali***—Take 3.0 g of Exsiccated Gypsum in a glass-stoppered test tube, add 10 mL of water and 1 drop of phenolphthalein TS and shake vigorously: no red color develops.

Solidification Take 10.0 g of Exsiccated Gypsum, add 10 mL of water, stir immediately for 3 minutes and allow to stand: the period necessary for water no longer to separate, upon pressing with a finger, is not more than 10 minutes from the time when water was first added.

Containers and Storage ***Containers*—**Tight containers.

Honey

Mel

Honey is the saccharine substances obtained from the honeycomb of *Apis mellifera* Linné or *Apis indica* Radoszkowski (Apidae).

Description Honey is a pale yellow to pale yellow-brown, syrupy liquid. Usually Honey is transparent, but often opaque with separated crystals.
Honey has a characteristic odor and a sweet taste.

Specific Gravity Mix 50.0 g of Honey with 100 mL of water: d_{20}^{20}, is not less than 1.111.

Purity (1) ***Acid***—Weigh 10 g of Honey, dissolve in 50 mL of water and neutralize with l mol/L potassium hydroxide TS (indicator: 2 drops of phenolphthalein TS): not more than 0.5 mL is required.

(2) ***Chloride***—Proceed with 1.0 g of Honey and perform the test. Prepare the control solution with 0.5 mL of 0.02 mol/L hydrochloric acid (not more than 0.035 %).

(3) ***Sulfate***—Proceed with 1.0 g of Honey and perform the test. Prepare the control solution with 0.5 mL of 0.005 mol/L sulfuric acid (not more than 0.024 %).

(4) ***Ammonia-coloring substances*—**Mix 1 g of Honey with 2.0 mL of water and filter. To the filtrate, add 2 mL of ammonia TS: the solution does not change immediately.

(5) ***Resorcin-coloring substances*—**Weigh 5 g of Honey, add 15 mL of ether, mix by shaking, filter and evaporate the ether solution at ordinary temperature. To the residue, add l to 2 drops of resorcin TS: a yellow-red color may develop in the solution of resorcin and in the residue and a red to red-purple color which does not persist more than l hour.

(6) ***Starch or dextrin*—**(i) Shake 7.5 g of Honey with 15 mL of water, warm the mixture on a water-bath and add 0.5 mL of tannic acid TS. After cooling, filter and to 1.0 mL of the filtrate, add 1.0 mL of dehydrated ethanol containing 2 drops of hydrochloric acid: no turbidity is produced.

(ii) Weigh 2 g of Honey, add 10 mL of water, warm in a water-bath, mix and allow to coo1. Shake 1.0 mL of the mixture with l drop of iodine TS: no blue, green or red-brown color develops.

(7) ***Foreign matter*—**Mix 1 g of Honey with 2.0 mL of water, centrifuge the mixture and examine the precipitate microscopically: no foreign substance except pollen grains is observable.

(8) ***5-Hydroxymethylfurfural*—**Weigh accurately about 5 g of the sample, dissolve in 25 mL of water and transfer to a 50 mL volumetric flask. Add 0.5 mL of 15 % potassium ferrocyanide, mix, add 0.5 mL of 30 % zinc acetate, mix, add water to make exactly 50 mL (add 1 drop of ethanol if foam develops). Filter, discard the first 10 mL of the filtrate and use the subsequent filtrate as the sample solution. Transfer 5 mL each of the sample solution to 2 test tubes and use one as the test solution and the other as the blank solution. Add 5 mL of water to the test solution, add 5 mL of 0.2 % sodium hydrogen sulfite to the blank solution and mix well. Determine the absorbances at 284 nm and 336 nm of the test solution and the blank solution, using water and 0.1 % sodium hydrogen sulfite as the control solutions, respectively (not more than 80 ppm).

$$\text{Amount (ppm) of hydroxymethylfurfural} = \frac{(A_{284} - A_{336}) \times 149.7 \times 5}{S}$$

A_{284} and A_{336}: Absorbances at 284 nm and 336 nm (test solution – blank solution)

S: Amount (g) of the sample taken

Ash Not more than 0.4 %.

Microbial Limit The total aerobic microbial count is not more than 1000 CFU/g, the total combined yeasts/mould count is not more than 100 CFU/g, and *Escherichia coli*, *Salmonella*, *Pseudomonas aeruginosa* and *Staphylococcus aureus* are not observed.

Containers and Storage ***Containers*—**Tight containers.

Hydrogenated Oil

Hydrogenated Oil is the fat obtained by hydrogenation of fish oil or of other oils originating from animal or vegetable.

Description Hydrogenated Oil is a white mass or powder, has a characteristic odor and a mild taste.
Hydrogenated Oil is freely soluble in ether, very slightly soluble in ethanol and practically insoluble in water. Only the oil obtained by hydrogenation of castor oil is slightly soluble in ether, very slightly soluble in ethanol and practically insoluble in water.

Acid Value Not more than 2.0.

Purity (1) ***Moisture and coloration***—Proceed as directed in the Purity (1) under Beef Tallow.

(2) ***Alkali***—Proceed as directed in the Purity (2) under Beef Tallow.

(3) ***Chloride***—Proceed as directed in the Purity (3) under Beef Tallow.

(4) ***Heavy metals***—Take 2.0 g of Hydrogenated Oil, add 5 mL of dilute hydrochloric acid and 10 ml of water, heat on a water-bath for 5 minutes with occasionally shaking. Filter after cooling, make slightly alkaline with 5 ml of ammonia TS to the filtrate and add 3 drops of sodium sulfide: the solution does not change.

(5) ***Nickel***—Place 5.0 g of Hydrogenated Oil in a quartz or porcelain crucible, heat slightly with caution at the beginning and after carbonization, incinerate by strong heating (500 ± 20 °C). 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 a solution of citric acid (1 in 5), make alkaline with 5 mL of ammonia TS and cool in running water. To this solution, add 1 mL of dimethyl-glyoxime TS, add water to make 20 mL and use this solution as the test solution. Allow to stand for 5 minutes: the solution has no more color than the following control solution.

Control solution—Evaporate 1 mL of hydrochloric acid on a water-bath to dryness, add 1 mL of standard nickel solution and 3 mL of dilute hydrochloric acid and add 6 mL of water. Proceed as directed in 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 to a stoppered 250 mL conical 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 and add 30 mL of water. Titrate with 0.01 mol/L sodium thiosulfate VS until the blue color of the solution disappears after addition of 5 mL of starch TS near the end point where the solution is a pale yellow color. Perform a blank determination and make any necessary correction (not more than 0.1 mL of 0.01 mol/L sodium thiosulfate VS is consumed by the blank solution). Calculate the amount of peroxide by the following formula: not more than 3.

$$\text{Amount (mEq/kg) of peroxide} = \frac{[10 \times (V_1 - V_0)]}{W}$$

V_1: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed in the test

V_0: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed in the blank

W: Amount (g) of Hydrogenated Oil taken

Residue on Ignition Not more than 0.1 % (5 g).

Containers and Storage ***Containers*—**Well-closed containers.

Hydroxypropylcellulose

[*9004-64-2*]

Hydroxypropylcellulose is a hydroxypropyl ether of cellulose. Hydroxypropylcellulose, when dried, contains not less than 53.4 % and not more than 77.5 % of hydroxypropoxyl group ($-OC_3H_6OH$: 75.09).

Description Hydroxypropylcellulose is a white to yellowish white powder and is odorless.
Hydroxypropylcellulose practically insoluble in ether.
Hydroxypropylcellulose forms a viscous liquid upon addition of water or ethanol.

Identification (1) Take 1 g of Hydroxypropylcellulose, add 100 mL of water, heat on a water-bath at 70 °C for 5 minutes with stirring and cool while shaking. Allow to stand at room temperature until it becomes more homogeneous and viscous and use this solution as the test solution. To 2 mL of the test solution, add 1 mL of anthrone TS gently: a blue to

green color develops at the zone of contact.

(2) Heat the test solution obtained in (1): white turbidity or white precipitate is produced and the turbidity or the precipitate disappears when cooled.

(3) Take 1 g of Hyddroxypropylcellulose, add 100 mL of ethanol and allow to stand after stirring: a homogeneous and viscous liquid is produced.

pH Dissolve 1.0 g of Hydroxypropylcellulose in 50 mL of freshly boiled and cooled water: the pH of the solution is between 5.0 and 7.5.

Purity (1) ***Clarity and color of solution***—Use an outer glass cylinder, about 25 cm in height, 25 mm in internal diameter and 2 mm in thickness, with a high-quality glass plate, 2 mm in thickness at the bottom and inner glass cylinder, about 30 cm in height, 15 mm in internal diameter and 2 mm in thickness, with high-quality glass plate, 2 mm in thickness at the bottom. In the outer cylinder place a solution prepared by adding 1.0 g of Hydroxypropylcellulose to 100 mL of water, heat while stirring on a water-bath at 70 °C and then cool to room temperature. Place this cylinder in a sheet of white paper on which 15 parallel, black, 1-mm lines in width are drawn at 1-mm intervals. Place the inner cylinder and move it up and down while viewing downward through the bottom of the inner cylinder and measure the minimum height of the solution between the bottom of the outer cylinder and the lower end of the inner cylinder at the time when the lines on the paper cannot be differentiated: the average value obtained from three repeated procedures is greater than that obtained form the following control solution treated in the same manner.

Control solution—5.50 mL of 0.005 mol/L sulfuric acid VS, add 1 mL of dilute hydrochloric acid, 5 mL of ethanol and water to make 50 mL. To this solution, add 2 mL of barium chloride TS, mix, allow to stand for 10 minutes and shake well before use.

(2) ***Chloride***—Add 1.0 g of Hydroxypropylcellulose to 30 mL of water, heat on a water-bath with stirring for 30 minutes and filter while being hot. Wash the residue with three 15 mL volumes of hot water, combine the washings with the filtrate and add water to make 100 mL after cooling. To 10 mL of the test solution, add 6 mL of dilute nitric acid and water to make 50 mL and perform the test using this solution as the test solution. Prepare the contrrol solution with 0.40 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.142 %).

(3) ***Sulfate***—To 20 mL of the test solution obtained in (2), add 1 mL of dilute hydrochloric acid and water to make 50 mL and perform the test using this solution as the test solution. Prepare the control solution with 0.40 mL of 0.05 mol/L sulfuric acid VS (not more than 0.048 %).

(4) ***Heavy metals***—Proceed with 1.0 g of Hydroxypropylcellulose according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 20 ppm).

(5) ***Arsenic***—Prepare the test solution with 1.0 g of Hydroxypropylcellulose according to Method 3 and perform the test (not more than 2 ppm).

(6) ***Lead***—Weigh accurately about 5 g of Hydroxypropylcellulose, transfer to a platinum or quartz crucible, moisten with a small amount of sulfuric acid, and heat slowly to pre-incinerate at a temperature as low as possible. Add 1 mL of sulfuric acid, heat slowly, and ignite at 450 °C to 550 °C to incinerate. After incineration, dissolve the residue in a small amount of nitric acid (1 in 150), add nitric acid (1 in 150) to make 10 mL and use this solution as the test solution. Separately, transfer 1.0 mL of standard lead 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 2.0 ppm).

Gas: Acetylene or hydrogen – Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(7) ***Propylene chlorohydrin***—Weigh accurately 1 g of Hydroxypropylcellulose, add 5 mL of diethyl ether, stopper and sonicate for 10 minutes. Centrifuge this extract and use the clear supernatant liquid as the test solution. Separately, weigh accurately 0.1 g of propylene chlorohydrin [Aldrich 292087 (a mixture of 1-Chloro-2- propanol 70 % and 2-Chloro-1- propanol 25 %) or equivalent] and add diethyl ether to make 100 mL. To an amount of this solution, add diethyl ether to make solutions containing 6 ng to 25 ng of propylene chlorohydrin per 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 Gas Chromatography according to the following operating conditions. Determine the peak area of each standard solution with respect to the concentration (ng/mL) and plot a calibration curve. Determine the peak area of propylene chlorohydrin in the test solution and calculate the amount of propylene chlorohydrin in the test solution from the calibration curve: not more than 0.1 ppm.

Operating conditions

Column: A fused silica column 0.53 mm in internal diameter and 30 m in length, with internal coating 1 μm in thickness made of polyethylene glycol 20M for gas chromatography. If necessary, use a guard column.

Detector: Electron capture detector (ECD)

Injection port temperature: 200 °C

Column temperature: Maintain at 35 °C for 7 minutes, raise the temperature to 200 °C at the rate of 8 °C per minute and maintain at 200 °C for 5 minutes.

Detector temperature: 230 °C
Carrier gas: Nitrogen or helium
Flow rate: Adjust the flow rate so that the retention times of 1-chloro-2-propanol and 2-chloro-1-propanol are about 11.7 minutes and about 12.5 minutes, respectively.

Loss on Drying Not more than 5.0 % (1 g, 105°C, 4 hours).

Residue on Ignition Not more than 0.5 % (1 g).

Assay (1) ***Apparatus***—Reaction flask: A 5-mL screw-cap pressure-tight glass bottle, having an inverted conical bottom inside, 20 mm in outside diameter, 50 mm in height up to the neck and 2 mL in capacity up to a height of about 30 mm, equipped with a pressure-tight septum of heat-resisting resin and also with an inside stopper or sealer of fluoroplastic.

Heater: A square aluminum block, 60 mm to 80 mm in thickness, having holes, 20.6 mm in diameter and 32 mm in depth, capable of maintaining the inside temperature within ± 1 °C.

(2) ***Procedure***—Weigh accurately about 65 mg of Hydroxypropylcellulose, previously dried, transfer to the reaction flask, add 65 mg of adipic acid, 2.0 mL of the internal standard solution and 2.0 mL of hydroiodic acid, stopper the flask tightly and weigh accurately. Shake the flask for 30 seconds, heat at 150 °C on the heater for 30 minutes with repeated shaking at 5 minute intervals and continue heating for an additional 30 minutes. Allow the flask to cool and again weigh acurately. If the mass loss is less than 10 mg, use the upper layer of the mixture as the test solution. Separately, take 65 mg of adipic acid, 2.0 mL of hydroiodic acid in another reaction flask, stopper tightly and weigh accurately. Add 50 μL of iospropyl iodide RS and again weigh accurately. Shake the reaction flask for 30 seconds and use the upper layer of the content as the standard solution. Perform the test as directd under the Gas Chromatography with 1 μL each of the test solution and the standard solution according to the following operating conditions and, calculate the ratios, Q_T and Q_S, of the peak area of isopropyl iodide to that of the internal standard for the test solution and the standard solution, respectively.

$$\text{Amount (\%) of hydroxypropoxyl group } (C_3H_7O_2) = \frac{Q_T}{Q_S} \times \frac{W_S}{\text{amount (mg) of the sample}} \times 44.17$$

W_S: Amount(mg) of isopropyl iodide in the standard solution.

Internal standard solution—A solution of *n*-octane in *o*-xylene (4 in 100).

Operating conditions
Detector: A thermal conductivity detector or hydrogen flame-ionization detector.
Column: A column, about 3 mm in internal diameter and about 3 m in length, packed with siliceous earth for gas chromatography (180 μm to 250 μm in particle diameter), coated with methyl silicone polymer for gas chromatography at the ratio of 20 %.
Column temperature: A constant temperature of about 100 °C.
Carrier gas: Helium (for thermal-conductivity detector), helium or nitrogen (for hydrogen flame-ionization detector).
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 operating conditions, use a column gining elution of isopropyl iodide and the internal standard in this order with complete separation of each peak.

Containers and Storage ***Containers*—**Well-closed containers.

Low Substituted Hydroxypropylcellulose

[*9004-64-2*, Hydroxypropylcellulose]

Low Substituted Hydorxypropylcellulose is a low substituted hydroxypropylether of cellulose. Low Substituted Hydroxypropylcellulose, when dried, contains not less than 5.0 % and not more than 16.0 % of hydroxypropoxyl group ($-OC_3H_6OH$: 75.09).

Description Low Substituted Hydroxypropylcellulose appears as white to yellowish white powder or granules, is tasteless, odorless or has a slight, characteristic odor.
Low Substituted Hydroxypropylcellulose is practically insoluble in ethanol or in ether.
Low Substituted Hydroxypropylcellulose dissolves in a solution of sodium hydroxide (1 in 10) and becomes viscous solution.
Low Substituted Hydroxypropylcellulose swells in water, in sodium carbonate TS or in 2 mol/L hydrochloric acid TS.

Identification (1) Take 20 mg of Low Substituted Hyd-roxypropylcellulose, add 2 mL of water, shake and produce a turbid solution. Add 1 mL of anthrone TS gently: a blue to blue-green color develops at the zone of contact.

(2) Take 0.1 g of Low Substituted Hydroxypropylcellulose, add 10 mL of water, stir and produce a turbid solution. Add 1 g of sodium hydroxide, shake until it becomes homogeneous and use this solution as the

test solution. To 0.1 mL of the test solution, add 9 mL of diluted sulfuric acid (9 in 10), shake well, heat in a water-bath for exactly 3 minutes, immediately cool in an ice-bath, add carefully 0.6 mL of ninhydrin TS, shake well and allow to stand at 25 °C : a red color develops at first and it changes to purple within 100 minutes.

(3) Take 5 mL of the test solution obtained in (2), add 10 mL of a mixture of acetone and methanol (4 : 1) and shake: a white, flocculent precipitate is produced.

pH Take 1.0 g of Low Substituted Hydroxypropylcellulose, add 100 mL of freshly boiled and cooled water and shake: the pH of the solution is between 5.0 and 7.5.

Purity (1) ***Chloride***—Take 0.5 g of Low Substituted Hydroxypropylcellulose, add 30 mL of hot water, stir well, heat on a water-bath for 10 minutes and filter the supernatant liquid by decantation while being hot. Wash the residue thoroughly with 50 mL of hot water, combine the washings with the filtrate and add water to make 100 mL after cooling. To 5 mL of this solution, add 6 mL of dilute nitric acid and water to make 50 mL and perform the test using this solution as the test solution. Prepare the control solution with 0.25 mL of 0.01 mol/L hydrochloric acid (not more than 0.335 %).

(2) ***Heavy metals***—Prceed with 2.0 g of Low Substituted Hydroxypropylcellulose according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 10 ppm).

(3) ***Arsenic***—Prepare the test solution with 1.0 g of Low Substituted Hydroxypropylcellulose, according to Method 3 and perform the test (not more than 2 ppm).

Loss on Drying Not more than 6.0 % (1 g, 105 °C, 1 hour).

Residue on Ignition Not more than 1.0 % (1 g).

Assay Proceed as directed in the Assay under Hydroxypropylcellulose, but add 15 μL of iospropyl iodide RS instead of 50 μL of iospropyl iodide RS, and perform the test with 2 μL each of the test solution and the standard solution instead of 1 μL each and use a solution of *n*-octane in *o*-xylene (1 in 50) as the internal standard solution.

$$\text{Amount (\%) of hydroxypropoxyl group } (C_3H_7O_2) = \frac{Q_T}{Q_S} \times \frac{W_S}{\text{amount (mg) of the sample}} \times 44.17$$

Containers and Storage ***Containers***—Tight containers.

Hypromellose

Hydroxypropylmethylcellulose

[*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 (-OCH_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, together 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 appears as white to yellowish white, powder or granules.
Hypromellose is practically insoluble in dehydrated ethanol.
Hypromellose swells with water and becomes a clear or slightly turbid, viscous solution.

Identification (1) Disperse evely 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 allown the beaker to stand: 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 °C, and stir: the resulting liquid is a clear or a slightly cloudy, 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, heat in a water-bath for exactly 3 minutes, immediately cool in an ice-bath, add carefully 0.6 mL of ninhydrin TS, shake and allow to stand at 25 °C : a red color develops at first and it changes to purple within 100 minutes.

(4) Pour and spread out 2 to 3 mL of the viscous fluid obtained in (2) on to a glass plate and allow the water to evaporate : a transparent film results.

(5) Take exactly 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 not less than 50 °C.

Viscosity **Method 1**: Apply to Hypromellose having a labeled viscosity of less than 600 mPa·s. Put exactly Hypromellose, equivalent to 4.000 g, calculated on the dried basis, in a tared, wide-mouth bottle, add hot water to make 200.0 g, stopper the bottle, stir by mechanical means at 350- to 450-revolutions per minute for 10 to 20 minutes to get a homogeneous dispersion. If necessary, take off the sample attached on the walls of the bottle, put them in the dispersed solution, and dissolve by continuing the stirring in a water bath not exceeding 10 °C for 20 to 40 minutes while stirring. Add cooled water, if necessary, to make 200.0 g, and use this solution as the test solution. Centrifuge the test solution if necessary to expel any entrapped air bubbles. Perform the test with the test solution at at 20 ± 1 °C as directed in Method 1 under the Viscosity Determination: not less than 80 % and not more than 120 % of the labeled viscosity.

Method 2: Apply to Hypromellose having a labeled viscosity of not less than 600 mPa·s. Put exactly Hypromellose, equivalent to 10.00 g, calculated on the dried basis, in a tared, wide-mouth bottle, add hot water to make 500.0 g, and prepare the test solution in the same manner as directed in Method 1. Perform the test with the test solution at at 20 ± 1 °C as directed in Method 2 under the Viscosity Determination, using a single cylinder-type rotational viscometer, according to the following operating conditions: not less than 75 % and not more than 140 % of the labeled viscosity.

Operating conditions

Apparatus: Brookfield type viscometer LV model

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
Not less than 600 and less than 1400	3	60	20
Not less than 1400 and less than 3500	3	12	100
Not less than 3500 and less than 9500	4	60	100
Not less than 9500 and less than 99500	4	6	1000
Not less than 99500	4	3	2000

Operation of apparatus: Read value after 2 minutes of rotation, and stop the rotation for 2 minutes. Repeat this operation 2 times more, and average three observed values.

pH Allow the test sample obtained in the Viscosity to stand at 20 ± 2 °C for 5 minutes: the pH of the solution thus obtained is between 5.0 and 8.0.

Purity (1) ***Heavy metals***—Put 1.0 g of Hypromellose a 100-mL Kjeldahl flask, add a sufficient amount of a mixture of nitric acid and sulfuric acid (5:4) to wet the sample, and heat gently. Repeat this procedure until to use total 18 mL of the mixture of nitric acid and sulfuric acid (5 : 4), and heat until the solution changes to black. After cooling, add 2 mL of nitric acid, and heat until the solution changes to black. Repeat this procesure until the solution no longer changes to black, and heat strongly until dense white fumes are evolved. After cooling, add 5 mL of water, boil gently until dense white fumes are evolved, then heat until the volume of the solution becomes 2 to 3 mL. After cooling, if the solution reveals yellow color by addition of 5 mL of water, add 1 mL of hydrogen peroxide(30), and heat until the volume of the solution becomes 2 to 3 mL. After cooling, dilute the solution with 2 to 3 mL of water, transfer to a Nessler tube, add water to make 25 mL, and use this solution as the test solution. Separately, put 2.0 mL of standard lead solution in a 100-mL Kjeldahl flask, add 18 mL of the mixture of nitric acid and sulfuric acid (5 : 4) and an amount of nitric acid equal to that used for the preparation of the test solution, and heat until dense white fumes are evolved. After cooling, add 10 mL of water. In the case where hydrogen peroxide (30) is added for the preparation of the test solution, add the same amount of hydrogen peroxide (3), then proceed in the same manner for the preparation of the test solution, and use so obtained solution as the control solution. Adjust the test solution and the control solution to pH 3.0 to 4.0 with ammonia solution (28), and add water to make 40 mL, respectively. To this solutions add 1.2 mL of thioacetamide-alkaline glycerin TS, 2 mL of acetate buffer solution, pH 3.5 and water to make 50 mL, separately. After allowing to stand for 5 minutes, oserve vertically both tubes on a white background: the color obtained with the test solution is not more intense than that with the control solution (not more than 20 ppm).

(2) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat and place 10 mg to 300 mg of Hypromellose on top. Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. Place the boat inside the furnace and heat to about 900 °C with a current of air or oxygen at 0.5 L/minute to 1 L/minute. Elute the mercury and sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from

specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 ng to 200 ng.

Additive—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.

(3) ***Cadmium***—Weigh accurately 5.0 g of Hypromel-lose 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 5 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 1.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Cadmium hollow cathode lamp
Wavelength: 228.8 nm

(4) ***Lead***—Weigh accurately 5.0 g of Hypromellose 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 solution as directed under Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 2.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(5) ***Propylene chlorohydrin***—Weigh accurately 1 g of Hypromellose, add 5 mL of diethyl ether, stopper and sonicate for 10 minutes. Centrifuge this extract and use the clear supernatant liquid as the test solution. Separately, weigh accurately 0.1 g of propylene chlorohydrin [Aldrich 292087(a mixture of 1-Chloro-2-propanol 70 % and 2-Chloro-1- propanol 25 %) or equivalent] and add diethyl ether to make 100 mL. To an amount of this solution, add diethyl ether to make solutions containing 6 ng to 25 ng of propylene chlorohydrin per 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 Gas Chromatography according to the following operating conditions. Determine the peak area of each standard solution with respect to the concentration (ng/mL) and plot a calibration curve. Determine the peak area of propylene chlorohydrin in the test solution and calculate the amount of propylene chlorohydrin in the test solution from the calibration curve: not more than 0.1 ppm.

Operating conditions

Column: A fused silica column 0.53 mm in internal diameter and 30 m in length, with internal coating 1 μm in thickness made of polyethylene glycol 20M for gas chromatography. If necessary, use a guard column.

Detector: Electron capture detector (ECD)

Injection port temperature: 200 °C

Column temperature: Maintain at 35 °C for 7 minutes, raise the temperature to 200 °C at the rate of 8 °C per minute and maintain at 200 °C for 5 minutes.

Detector temperature: 230 °C

Carrier gas: Nitrogen or helium

Flow rate: Adjust the flow rate so that the retention times of 1-chloro-2-propanol and 2-chloro-1-propanol are about 11.7 minutes and about 12.5 minutes, respectively.

Loss on Drying Not more than 5.0 % (1 g, 105 °C, 1

hours).

Residue on Ignition Not more than 1.5 % (1.0 g).

Assay (1) ***Apparatus***—Reaction bottle : A 5-mL screw-cap pressure-tight glass vial, about 20 mm in outside diameter, about 50 mm in height, having the neck 20 mm in outside diameter and 13 mm in internal diameter, 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 equivalent.

Heater: A square-shaped aluminum block, having holes 20 mm in diameter and 32 mm in depth, adopted to the reaction bottles, and capable of stirring the content of the reaction bottle by means of magnetic stirrer or of reciprocal shaker about 100 times per minute..

(2) ***Procedure***—Weigh accurately about 65 mg of Hypromellulose, transfer to the 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, stopper the bottle tightly, immediately and weigh accurately. Stir or shake for 60 minutes while heating so that the temperature of the bottle content is 130 ± 2 °C. In the case when the stirrer or shaker is not available, heat for 30 minutes with repeated shaking at 5-minute intervals by hand, and continue heating for an additional 30 minutes. Allow the flask to cool and again weigh accurately. If the mass loss is less than 0.50 % or there is no evidence of a leak, use the upper layer of the content as the test solution. Separately, put 60 to 100 mg of adipic acid, 2.0 mL of the internal standard solution and 2.0 mL of hydroiodic acid in a reaction bottle, stopper immediately and weigh accurately. 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 thoroughly the reaction bottle and use the upper layer of the content 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 ratios, Q_{Ta} and Q_{Tb}, of the peak area of methyl iodide from the test solution to that of the internal standard and Q_{Sa} and Q_{Sb}, of the peak area of methyl iodide and isopropyl iodide, respectively, from the standard solution to that of the internal standard from the standard solution.

$$\text{Content (\%) of methoxyl group } (CH_3O) = \frac{Q_{Ta}}{Q_{Sa}} \times \frac{W_{Sa}}{W} \times 21.864$$

$$\text{Content (\%) of hydroxypropoxyl group } (C_3H_7O_2) = \frac{Q_{Tb}}{Q_{Sb}} \times \frac{W_{Sb}}{W} \times 44.17$$

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 (1 in 25).

Operating conditions

Detector: A thermal conductivity detector or hydrogen flame-ionization detector.

Column: A glass column, 3 to 4 mm in internal diameter and 1.8 to 3 m in length, peakd with siliceous earth for gas chromatography (125 μm to 150 μm in diameter), coated with methyl silicone polymer at the ratio of 10 to 20 %.

Column temperature: A constant temperature of about 100 °C.

Carrier gas: Helium for thermal conductivity detector, or helium or nitrogen for hydrogen flame ionization detector.

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

System suitability

System performance: When the procedure is run with 1 to 2 μL of the standard solution under the above operating conditions, methyl iodide, isopropyl iodide and the internal standard are eluted in this order, with complete separation of these peaks.

Containers and Storage ***Containers*—**Well-closed containers.

Hypromellose 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 (-OC_3H_6OH: 75.09) and carboxybenzoyl group (-COC_6H_4COOH: 149.12).

Hypromellose Phthalate contains not less than 21.0 % and not more than 35.0 % of carboxybenzoyl group, calculated on the anhydrous basis.

Substitution type	Carboxybenzoyl group (%)	
	Minimum	Maximum
200731	27.0	35.0
220824	21.0	27.0

The substitution type of Hypromellulose Phthalate is shown together with its viscosity in millipascal-

seconds (mPa . s), on the label.

Description Hypromellulose Phthalate appears as white powder or granules, is odorless and tasteless.
Hypromellulose Phthalate is practically insoluble in water, in acenitrile, in dehydrated ethanol, and in hexane.
Hypromellulose Phthalate becomes a viscous liquid when a mixture of methanol and dichloromethane (1 : 1) or a mixture of dehydrated ethanol and acetone (1 : 1) is added.
Hypromellulose Phthalate dissolves in sodium hydroxide TS.

Identification Determine the infrared absorption spectra of Hypromellulose Phthalate and Hypromellulose Phthalate RS, as directed in the potassium bromide disk method under Infrared Spectrophotometry: both spectra exhibit similar intensities of absorption at the same wavenumbers.

Viscosity Dissolve 10 g of Hypromellulose Phthalate previously dried at 105 °C for 1 hour, in 90 g of a mixture of methanol and dichloromethane in equal mass ratio by mixing and shaking. Determine the viscosity at 20 ± 0.1 °C according to Method 1 under the Viscosity Determination: not less than 80 % and not more than 120 % of the labeled unit.

Purity (1) ***Chloride***—Dissolve 1.0 g of Hypromellulose Phthalate in 40 mL of 0.2 mol/L sodium hydroxide, add 1 drop of phenolphthalein TS and add dilute nitric acid drop-wise, with vigorous stirring, until the red color is discharged. Add an additional 20 mL of dilute nitric acid with stirring, and heat on a water-bath, with stirring, until the gel-like precipitate formed becomes granular. After cooling, centrifuge and separate the clear supernatant liquid and wash the residue with three 20 mL portions of water by centrifuging each time, combine the clear supernatant liquid and the washings, add water to make 200 mL, and filter. Perform the test with 50 mL of the filtrate. Prepare the control solution as follows: take 0.50 mL of 0.01 mol/L hydrochloric acid, add 10 mL of 0.2 mol/L sodium hydroxide TS and 7 mL of dilute nitric acid, and water to make 50 mL (not more than 0.07 %).

(2) ***Heavy metals***—Proceed with 2.0 g of Hypromellulose Phthalate according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 10 ppm)

(3) ***Phthalic acid***—Weigh accurately about 2.0 g of Hypromellulose Phthalate, add about 50 mL of acetonitrile, sonicate for dissolving partially, add 10 mL of water, sonicate again to dissolve further, after cooling, 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 operating conditions and determine the peak areas, A_T and A_S, of phthalic acid for the test solution and the standard solution, respectively: the content of phthalic acid ($C_8H_6O_4$: 166.13) is not more than 1.0 %.

$$\text{Content (\%) of phthalic acid } (C_8H_6O_4) = \frac{C}{W} \times \frac{A_T}{A_S} \times 10$$

C : Concentration(μg/mL) of phthalic acid in the standard solution.
W : 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, 4 mm in internal diameter and about 25 cm in length, packed with octadecylsilylated silica gel (3 μm to l0 μm in particle diameter).
Column temperature: A constant temperature of about 20 °C.
Mobile phase: A mixture of 0.1 mol/L cyanoacetic acid and acetonitrile (17 : 3).
Flow rate: 2.0 mL/minute.
System suitability
System performance: When the procedure is run with 10 μL of the standard solution under the above operating conditions, the number of theoretical plates and the symmetry factor of the peak of phthalic acid is not less than 2500 and not more than 1.5, respectively.
System repeatability: When the test is repeated 6 times with 10 μL each of the standard solution under the above operating conditions, the relative standard deviation of the peak area of phthalic acid is not more than 1.0 %.

Water Not more than 5.0 % (1g, direct titration, using a mixture of dehydrated ethanol and dichloromethane (3 : 2) instead of methanol for Karl Fischer method).

Residue on Ignition Not more than 0.2 % (1 g)

Assay Weigh accurately about 1.0g of Hypromellulose Phthalate, dissolve in 50 mL of a mixture of acetone, ethanol and water (2 : 2 : 1), titrate with 0.1 mol/L of sodium hydroxide VS (indicator: 2 drops of phenolphthalein TS). Perform a blank determination and make necessary correction.

$$\text{Amount (\%) of carboxybenzoyl group } (C_8H_5O_3) = \frac{0.01 \times 149.1 \times V}{W} - \frac{2 \times 149.1 \times P}{166.1}$$

P : Content (%) of phthalic acid under Purity (3) Phthalic acid.

V : Volume (mL) of 0.1 mol/L sodium hydroxide used in titration.

W : Amount (g) of the sample, calculrated on anhydrous basis.

Containers and Storage ***Containers*—**Tight containers.

Kaolin

Kaolin is a native, hydrous aluminum silicate.

Description Kaolin is white or nearly white, fragmentary mass or powder and has a slightly clay-like odor.
Kaolin is practically insoluble in water, in dehydrated ethanol or in ether.
Kaolin is insoluble in dilute hydrochloric acid or in sodium hydroxide TS.
When moistended with water, Kaolin darkens and becomes plastic.

Identification (1) Heat 1 g of Kaolin with 10 mL of water and 5 mL of sulfuric acid in a porcelain dish and evaporate the mixture nearly to dryness. Cool, add 20 mL of water, boil for 2 to 3 minutes and filter: the color of the residue is gray.

(2) The filtrate obtained in (1), responds to the Qualitative Tests (1), (2) and (4) for aluminum salt.

Purity (1) ***Acidity or alkalinity***—Add 25 mL of water to 1.0 g of Kaolin, agitate thoroughly and filter: the pH of the filtrate is between 4.0 and 7.5.

(2) ***Acid-soluble substances***—Add 20 mL of dilute hydrochloric acid to 1.0 g of Kaolin, agitate for 15 minutes and filter. Evaporate 10 mL of the filtrate to dryness and ignite between 450 and 550 °C to a constant weight: the ignited residue is not more than 10 mg.

(3) ***Carbonate***—Stir 1.0 g of Kaolin with 5 mL of water and add 10 mL of diluted sulfuric acid (1 in 2): no foam is produced.

(4) ***Heavy metals***—Boil 1.5 g of Kaolin gently with 50 mL of water and 5 mL of hydrochloric acid for 20 minutes with frequent agitation, cool, centrifuge and separate the clear supernatant liquid. Wash the precipitate twice with 10 mL of water, conetrifuge each time and combine the clear supernatant liquid and the washings. Add drop-wise strong ammonia water to this solution until a slight precipitate occurs, then add dilute hydrochloric acid drop-wise while agitating strongly to complete solution. Add 0.45 g of hydroxylamine hydrochloride and heat. Cool, add 0.45 g of sodium acetate and 6 mL of dilute acetic acid, filter, if necessary and 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. Prepare the control solution as follows: To 2.5 mL of standard lead solution add 0.15 g of hydroxylamine hydrochloride, 0.15 g of sodium acetate, 2 mL of acetic acid and add water to make 50 mL (not more than 50 ppm).

(5) ***Iron***—Add 10 mL of dilute hydrochloric acid to 40 mg of Kaolin and heat for 10 minutes with shaking in a water-bath. After cooling, add 0.5 g of tartaric acid, dissolve with shaking, prepare the test solution with this solution according to Method 2 and perform the test according to Method B. Prepare the control solution with 2.0 mL of standard iron solution (not more than 500 ppm).

(6) ***Arsenic***—Add 5 mL of water and 1 mL of sulfuric acid to 1.0 g of Kaolin and heat on a water-bath until white fumes begin to evolve. Add water to make 5 mL after cooling and perform the test (not more than 2 ppm).

(7) ***Foreign matter***—Place 5 g of Kaolin in a breaker, add 100 mL of water, stir and decant to leave sand. Repeat this procedure several times with 100 mL volumes of water: no sandy residue remains.

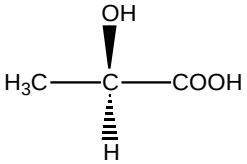
Loss on Ignition Not more than 15.0 % (1 g, 600 °C, 5 hours).

Plasticity Add 7.5 mL of water to 5 g of Kaolin and agitate thoroughly: the resultant mass has no remarkable fluidity.

Microbial Limit The total aerobic microbial count is not more than 1000 CFU/g, the total combined yeasts/mould count is not more than 100 CFU/g and *Escherichia coli*, *Salmonella* species, *Staphylococcus aureus* and *Pseudomonas aeruginosa* are not observed.

Containers and Storage ***Containers*—**Well-closed containers.

Lactic Acid



and enantiomer

2-Hydroxypropionic acid $C_3H_6O_3$: 90.08

(2*RS*)-2-Hydroxypropanoic acid [*50-21-5*]

Lactic Acid is a mixture of lactic acid and lactic anhydride. Lactic Acid contains not less than 85.0 % and not more than 92.0 % of lactic acid ($C_3H_6O_3$).

Description Lactic Acid is a clear, colorless or pale yellow, viscous liquid, odorless or has faint, unpleasant

odor.
Lactic Acid is miscible with water, ethanol or ether.
Lactic Acid is hygroscopic.

Specific gravity— d_{20}^{20} : About 1.20.

Identification A solution of Lactic Acid in water (1 in 50) changes blue litmus paper to red and responds to the Qualitative Tests 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 VS (not more than 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 sulfuric acid VS (not more than 0.010 %).

(3) ***Heavy metals*—**Take 2.0 g of Lactic Acid, add 10 mL of water and 1 drop of phenolphthalein TS, and add ammonia TS dropwise until a pale red color appears. Add 2 mL of dilute acetic acid and water to make 50 mL, and perform the test using this solution as the test solution. Prepare the control solution with 2.0 mL of standard lead solution and 2 mL of dilute acetic acid, and dilute with water to make 50 mL (not more than 10 ppm).

(4) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat, place 10 mg to 300 mg of Lactic Acid on top then 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, place only the test specimen in a nickel boat without the additives. Place the boat inside the furnace and heat to about 900 °C with a current of air or oxygen at 0.5 L/minute to 1 L/minute. Elute the mercury and sample in a sampling tube. Transfer the mercury vapor to a cold vapor atomic absorption spectrometer by heating the sampling 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 plot a calibration curve from the absorbances. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 ng to 200 ng.

Additive—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 Lactic Acid 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 standard lead 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 2.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(6) ***Iron*—**Prepare the test solution with 4.0 g of Lactic Acid according to Method 1, and perform the test according to Method A. Prepare the control solution with 2.0 mL of standard iron solution (not more than 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 add water to make 10 mL. Take 5 mL of this solution as the test solution and perform the test (not more than 4 ppm).

(8) ***Sugars*—**Take 1.0 g of Lactic Acid, add 10 mL of water, and neutralize with sodium hydroxide TS. Boil the mixture with 10 mL of Fehling's TS for 5 minutes: no red precipitate is produced.

(9) ***Citric acid, oxalic acid , phosphoric acid and tartaric acid*—**Take 1.0 g of Lactic Acid, add 1.0 mL of water, followed by 40 mL of calcium hydroxide TS. Boil the mixture for 2 minutes: no change occurs.

(10) ***Glycerin or mannitol*—**Shake 10 mL of Lactic Acid with 12 mL of ether: no turbidity is produced.

(11) ***Volatile fatty acids*—**Warm Lactic Acid: any acetic acid-like or butyric acid-like odor is not pro-

duced.

(12) ***Cyanide*—**Transfer 1.0 g of Lactic Acid to a Nessler tube, add 10 mL of water and 1 drop of phenolphthalein TS, add dropwisely a solution of sodium hydroxide (1 in 10) with shaking until a pale red color develops, add 1.5 mL of a solution of sodium hydroxide (1 in 10) and water to make 20 mL, and heat in a water-bath for 10 minutes. Cool, add dropwisely dilute acetic acid until a red color of the solution disappears, add 1 drop of dilute acetic acid, add 10 mL of phosphate buffer solution, pH 6.8, and 0.25 mL of chloramine TS, stopper immediately, mix gently, and allow to stand for 5 minutes. To the solution, add 15 mL of pyridine-pyrazolone TS and water to make 50 mL, and allow to stand at 25 °C for 30 minutes: the solution is not more intense than the following control solution.

Control solution—Pipet exactly 1.0 mL of standard cyanide solution, and add water to make exactly 20 mL. Transfer 1.0 mL of this solution to a Nessler tube, add 10 mL of water and 1 drop of phenolphthalein TS, and proceed as directed in the test solution.

(13) ***Readily carbonizable substances*—** Superimpose slowly 5 mL of Lactic Acid, previously kept at 15 °C, upon 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 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, take 5 mL of the first distillate, add water to make 100 mL and use this solution as the test solution. To 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, add 0.4 mL of anhydrous sodium sulfite solution (1 in 5) and 3 mL of sulfuric acid and add 0.2 mL of chromotropic acid TS. The color thus produced is not more intense than the color produced by 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.

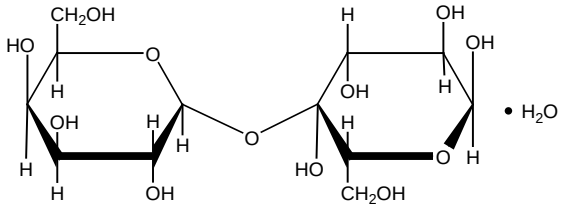
Residue on Ignition Not more than 0.10 %(1 g).

Assay Weigh accurately about 3 g of Lactic Acid, transfer in a Erlenmeyer flask, add accurately measured 40 mL of 1 mol/L sodium hydroxide VS, invert a watch glass over the flask, and heat in a water-bath for 10 minutes. Titrate the excess sodium hydroxide with 0.5 mol/L sulfuric acid VS immediately (indicator: 2 drops of phenolphthalein TS). Perform a blank determination, any make any necessary correction.

Each mL of 1 mol/L sodium hydroxide VS
= 90.08 mg of $C_3H_6O_3$

Containers and Storage ***Containers*—**Tight containers.

Lactose Hydrate



$C_{12}H_{22}O_{11}\cdot H_2O$: 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 disaccharide obtained from milk, consist of one unit of glucose and one unit of galactose. The label states that the granulated powder is Lactose Hydrate.

Description Lactose Hydrate appears as white crystals, powder or granulated powder, is odorless.
Lactose Hydrate is freely soluble in water and practically insoluble in ethanol.

Identification Determine the infrared spectra of Lactose Hydrate and Lactose Hydrate RS, previously dried, as directed in the potassium bromide disk method under the Infrared Spectrophotometry : both spectra exhibit similar intensities of absorption at the same wavenumbers.

Specific Optical Rotation $[\alpha]_D^{20}$: +54.4 ~ +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, allow to cool and add 0.2 mL of ammonia TS. After standing for 30 minutes, add water to make exactly 100 mL and determine the optical rotation of this solution in a 100 mm cell.

Purity Proceed as directed in the Purity under Anhydrous Lactose.

Microbial Limit The total aerobic microbial count is not more than 100 CFU/g, the total combined yeasts/mould count is not more than 50 CFU/g and *Salmonella* spices and *Escherichia coli* are not be observed.

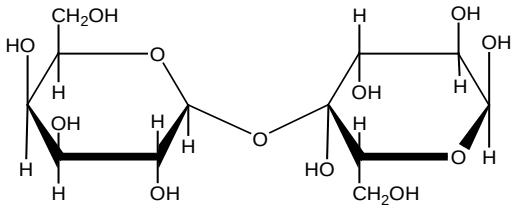
Loss on Drying Not more than 0.5 % (1 g, 80 °C, 2 hours) (not more than 1.0 % for the granulated powder).

Residue on Ignition Not more than 0.10 % (1 g).

Water 4.5 ~ 5.5 % (1 g, volumetric titration, direct titration) Use a mixture of methanol for Water Determination and formamide for Water Determination (2 : 1) instead of methanol for Water Determination (between 4.0 and 5.5 % for the granulated powder).

Containers and Storage ***Containers***—Well-closed containers.

Anhydrous Lactose



$C_{12}H_{22}O_{11}$: 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 mixture of β-lactose and α-lactose.
The relative quantities of α-lactose and β-lactose in Anhydrous Lactose are indicated as the isomer ratio.

Description Anhydrous Lactose appears as white crystals or powder.
Anhydrous Lactose is freely soluble in water and practically insoluble in ethanol.

Identification Determine the infrared spectra of Anhydrous Lactose and Anhydrous Lactose RS, previously dried, as directed in the potassium bromide disk method under the Infrared Spectrophotometry: both spectra exhibit similar intensities of absotption at the same wave numbers.

Specific Optical Rotation $[\alpha]_D^{20}$: +54.4 ~ +55.9°. Weigh accurately about 10 g of Anhydrous Lactose, dissolve in 80 mL of water warmed to 50 °C, allow to cool and add 0.2 mL of ammonia TS. After standing for 30 minutes and water to make exactly 100 mL and determine the optical rotation of this solution in a 100 mm cell.

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. Determine the absorbance at 400 nm of this solution as directed under the Ultraviolet-visible Spectrophotometry, using water as the blank: not more than 0.04.

(2) ***Acid or alkali***—Dissolve 6 g of Anhydrous Lactose in 25 mL of freshly boiled and cooled water by heating, cool, add 0.3 mL of phenolphthalein TS: the solution is colorless. To this solution, add 0.1 mol/L sodium hydroxide TS until the solution changes from colorless to red: not more than 0.4 mL is consumed.

(3) ***Heavy metals***—Proceed with 4.0 g of Anhydrous Lactose according to Method 2 and perform the test. Prepare the control solution with 2 mL of standard lead solution (not more than 5 ppm).

(4) ***Proteins and light absorbing substances***—Dissolve 1.0 g of Anhydrous Lactose in water to make 100 mL and use this solution as the test solution. Determine the absorbances, as directed under the Ultraviolet-visible Spectrophotometry, using water as the blank: not more than 0.25 at between 210 nm and 220 nm and not more than 0.07 at between 270 nm and 300 nm.

Isomer Ratio Place 1 mg of Anhydrous Lactose in an about 5 mL screw capped reaction vial for gas chromatography, add 0.45 mL of dimethylsulfoxide, stopper, and shake well. Add 1.8 mL of a mixture of pyridine and trimethylsilylimidazole (18 : 7), mix, allow to stand for 20 minutes and use this solution as the test solution. Perform the test with 2 μL of the test solution as directed under the Gas Chromatography according to the following operating conditions and determine peak areas A_a and A_b, of α-lactose and β-lactose, respectively and calculate the content (%) of α-lactose and the content (%) of β-lactose in Anhydrous Lactose by the following equations.

$$\text{Content (\%) of } \alpha\text{-lactose} = \frac{A_a}{A_a + A_b} \times 100$$

$$\text{Content (\%) of } \beta\text{-lactose} = \frac{A_b}{A_a + A_b} \times 100$$

Operating conditions

Detector: A hydrogen flame-ionization detector.

Test injection port temperature: About 275 °C.

Column: A column, about 4 mm in internal diameter and about 0.9 m in length, packed with siliceous earth for gas chromatography coated at the ratio of 3 % with 25 % phenyl-25 % cyanopropyl-methylsilicone polymer for gas chromatography.

Column temperature: A constant temperature of about 215 °C.

Carrier gas: Helium.

Flow rate: A constant flow rate of about 40 mL per minute.

System suitability

System performance: Prepare a solution with 1 mg of the mixture of α-lactose and β-lactose (1 : 1) in the same manner as for preparing the test solution. When the procedure is run with 2 μL of this solution under the above operating conditions, a ratio of the

retenion time of α-lactose to that of β-lactose is about 0.7 with the resolution between their peaks being not less than 3.0.

Microbial Limit The total aerobic microbial count is not more than 100 CFU/g, the total combined yeasts/mould count is not more than 50 CFU/g and *Salmonella* species and *Escherichia coli* are not observed.

Loss on Drying Not more than 0.5 % (1 g, 80 °C, 2 hours).

Water Not more than 1.0 % (1 g, volumetric titration, direct titration, using a mixture of methanol for Karl Fischer Method and formamide for Karl Fischer Method (2 : 1) instead of methanol for Karl Fischer Method).

Residue on Ignition Not more than 0.1 % (1 g).

Containers and Storage ***Containers***—Well-closed containers.

Hydrous Lanolin

Hydrous Lanolin is Purified Lanolin to which water is added. Hydrous Lanolin contains not less than 70.0 % and not more than 75.0 % of Purified Lanolin (as determined by the test for Residue on evaporation).

Description Hydrous Lanolin is a pale yellow, ointment like substance and has a slight, characteristic odor, which is not rancid.
Hydrous Lanolin is soluble in ether or in cyclohexane, with the separation of water.
When melted by heating on a water-bath, it separates into a clear oily layer and a clear water layer.

Melting Point About 39 °C.

Identification Dissolve 1 g of Hydrous Lanolin in 1 mL of cyclohexane and remove the separated water. Proceed with 1 mL of the cyclohexane solution in the Identification under Purified Lanolin.

Acid Value Not more than 1.0.

Iodine Value 18 ~ 36. Heat a suitable amount of Hydrous Lanolin on a water-bath to remove its almost moisture, then weigh accurately about 0.8 g of the treated Hydrous Lanolin in a glass-stoppered 500 mL flask and proceed as directed in the Iodine value under Purified Lanolin.

Purity (1) ***Acid or alkali, Chloride, Ammonia and Water-soluble organic substances***—Proceed as directed in Purity (1), (2), (3) and (4) under Purified Lanolin.

(2) ***Petrolatum***—Dry the residue after evaporation and proceed as directed in Purity (5) under Purified Lanolin.

(3) ***Butylhydroxytoluene***—Dry the residue after evaporation and proceed as directed in Purity (6) under Purified Lanolin (not more than 200 ppm.).

Residue on Evaporation Weigh accurately about 12.5 g of Hydrous Lanolin, dissolve in 50 mL of ether, place in a separatory funnel, transfer the separated aqueous layer to another separatory funnel, add 10 mL of ether, shake and combine the ether layer and ether in the first separatory funnel. Shake the ether layer with 3 g of anhydrous sodium sulfate and filter through dry filter paper. Wash the separatory funnel and the filter paper twice with 20 mL volumes of ether, combine the washings with the filtrate, evaporate on a water-bath until the odor of ether is no longer perceptible, dry in a dessicator (in vacuum, silica gel) for 24 hours and weigh.

Containers and Storage ***Containers—***Well-closed containers.

Storage—Not exceeding 30 °C.

Purified Lanolin

Purified Lanolin is the purified product of the fat-like substance obtained from the wool of *Ovis aries* Linné (Bovidae).

Description Purified Lanolin is a pale yellow to yellowish brown, viscous, ointment-like substance and has a faint, characteristic but not rancid odor.
It is very soluble in ether or in cyclohexane, freely soluble in tetrahydrofuran or in toluene, very slightly soluble in ethanol.
Purified Lanolin is partially insoluble in water, but miscible without separation with about twice volumes of water and retaining ointment-like viscosity.

Melting point—37 ~ 43 °C.

Identification Superimpose carefully 1 mL of a solution of Purified Lanolin in cyclohexane solution (1 in 50) on 2 mL of sulfuric acid: a red-brown color develops at the zone of contact and the sulfuric acid layer shows a green fluorescence.

Acid Value Not more than 1.0.

Iodine Value 18 ~ 36. Weigh accurately about 0.8 g of Purified Lanolin in a glass-stoppered 500 mL flask and add 10 mL of cyclohexane to dissolve and add 25.0 mL of Hanus's TS and mix well. If a clear solution is not obtained, add more cyclohexane to make clear and allow the mixture to stand for 1 hour between 20 °C and 30 °C in a light-resistant, well-closed containers while occasional shaking. Add 20 mL of a solution of

potassium iodide (1 in 10) and 100 mL of water, shake and titrate the liberated iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 1 mL of starch TS). Perform a blank determination in the same manner and make any necessary correction.

$$\text{Iodine value} = \frac{(a - b) \times 1.269}{\text{amount (g) of sample}}$$

a: Volume (mL) of 0.1 mol/L sodium thiosulfate VS consumed in the blank determination.

b: Volume (mL) of 0.1 mol/L sodium thiosulfate VS consumed in the titration.

Purity (1) ***Acidity or alkality***—Take 5 g of Purified Lanolin, add 25 mL of water, boil for 10 minutes and cool. Add water to restore the previous mass and separate the aqueous layer: the aqueous layer is neutral.

(2) ***Chloride***—Take 2.0 g of Purified Lanolin, add 40 mL of water, boil for 10 minutes and cool. Add water to restore the previous 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 VS (not more than 0.036 %).

(3) ***Ammonia***—Take 10 mL of the aqueous layer obtained in (1), add 1 mL of sodium hydroxide TS and boil: the gas evolved does not turn moistened red litmus paper to blue.

(4) ***Water-soluble organic substance***—Take 5 mL of the aqueous layer obtained in (1), add 0.25 mL of 0.002 mol/L potassium permanganate VS and allow to stand for 5 minutes: the red color of the solution does not disappear.

(5) ***Petrolatum***—Dissolve 1.0 g of Purified Lanolin in 10 mL of a mixture of tetrahydrofuran and isooctane (1 : 1) and use this solution as the test solution. Add dissolve 20 mg of vaseline in 10 mL of a mixture of tetrahydrofuran and isooctane (1 : 1) and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 25 μL each of the test solution and the standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with isooctanol to a distance of about 10 cm and air-dry the plate. Spray evenly diluted sulfuric acid (1 in 2) on the plate, heat the plate at 80 °C for 5 minutes, cool and examine under ultraviolet light (main wavelength: 365 nm): no fluorescent spot is observed in the same level with the spot of standard solution. For this test use a thin-layer plate previously developed with isooctanol to the upper end, dried in air and heated at 110 °C for 60 minutes.

(6) ***Butylhydroxytoluene***—Weigh accurately 1.0 g of Purified Lanolin, dissolve in carbon disulfide, add exactly 1.0 mL of the internal standard solution, add carbon sulfide to make exactly 10 mL and use this solution as the test solution. Separately, weigh accurately about 0.2 g of Butylhydroxytoluene 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 each of this solution and the internal standard solution, 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 Gas Chromatography according to the following operating conditions and determine the ratios, Q_T and Q_S, of the peak area of butylhydroxytoluene with respect to that of the internal standard in the test solution and the standard solution (not more than 200 ppm).

Operating conditions

Detector: A hydrogen flame ionization detector

Column: A column 4 mm in internal diameter and 1.5 m in length, packed with silanized diatomaceous earth for gas chromatography coated with poly(dimethyl)siloxane for gas chromatography at the mass ratio of 10 %.

Column temperature: A constant temperature of about 150 °C

Injection port temperature: 180 °C

Detector temperature: 300 °C

Carrier gas: Nitrogen

Flow rate: 40 mL/minute

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 Not more than 0.5 % (1 g, 105 °C, 2 hours).

Ash Not more than 0.1 % (proceed as directed in the Ash under the Test for Herbal Drugs)

Containers and Storage ***Containers*—**Well-closed containers.

***Storage*—**Not exceeding 30 °C.

Lard

Lard is the fat obtained from *Sus scrofa* Linné var. *domesticus* Gray (Suidae).

Description Lard is a white, soft, unctuous mass and has a faint, characteristic odor and a bland taste.
Lard is freely soluble in ether or in petroleum ether, very slightly soluble in ethanol and practically insoluble in water.

Melting Point 36 ~ 42 °C (Method 2).

Congealing Point of Fatty Acids 36 ~ 42 °C.

Saponificatioin Value 195 ~ 203.

Acid Value Not more than 2.0.

Iodine Value 46 ~ 70.

Purity (1) ***Moisture and coloration***—Melt 5 g of Lard by heating on a water-bath: it forms a clear liquid, from which no water separates. Observe the liquid in a layer, 10 mm in thickness: the liquid is colorless to slightly yellow.

(2) ***Alkali***—Take 2.0 g of Lard, add 10 mL of water, melt by warming on a water-bath and shake vigorously. Cool and add 1 drop of phenolphthalein TS to the separated water layer: the layer is colorless.

(3) ***Chloride***—Take 1.5 g of Lard, add 30 mL of ethanol, boil for 10 minutes under a reflux condenser and 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 mixture is not more intense than that of the following control solution.

Control solution—Take 1.0 mL of 0.01 mol/L hydrochloric acid VS, add ethanol to make 20 mL and add 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, stopper lightly with absorbent cotton and allow to stand at 20 °C for 18 hours. Collect the separated crystals, moisten with ethanol and examine under a microscope of 200 magnification: the crystals are in the form of rhomboidal plates grouped irregularly and do not contain prisms or needles grouped in fan-shaped clusters.

Containers and Storage ***Containers*—**Well-closed containers.

***Storage*—**Not exceeding 30 °C.

Lauromacrogol

Polyoxyethylene Lauryl Alcohol Ether

Lauromacrogol is a polyoxyethylene ether prepared by the polymerization of ethylene oxide with lauryl alcohol.

Description Lauromacrogol is a colorless or pale yellow, clear liquid or white, petrolatum-like or waxy solid, has a characteristic odor and a somewhat bitter and slightly irritative taste.
Lauromacrogol is very soluble in ethanol, in ether or in carbon tetrachloride.
Lauromacrogol is freely soluble or dispersed as fine oily drops in water.

Identification (1) Shake well 0.5 g of Lauromacrogol with 10 mL of water and 5 mL of ammonium thiocyanate-cobalt nitrate TS, then shake with 5 mL of chloroform and allow to stand: the chloroform layer becomes blue.

(2) Dissolve 0.35 g of Lauromacrogol in 10 mL of carbon tetrachloride and perform the test as directed in the Solution method under the Infrared Spectrophotometry 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}.

Purify (1) ***Acid***—Transfer 10.0 g of Lauromacrogol into a flask and add 50 mL of neutralized ethanol. Heat on a water nearly to boil, shaking once or twice while heating. Cool and add 5.3 mL of 0.1 mol/L sodium hydroxide VS and 5 drops of phenolphthalein TS: a red color develops.

(2) ***Unsaturated compound***—Shake 0.5 g of Lauromacrogol with 10 mL of water and 5 drops of bromine TS: the color of the solution does not disappear.

(3) ***Ethylene oxide and dioxane***—Weigh accurately 1.00 g (M_T) of Lauromacrogol, transfer to a 10 mL vial, add 1.0 mL of water and mix to obtain a homogeneous solution. 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 dioxane standard solution. 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 of a solution (this solution is stable for 3 monhts when kept at -20 °C sealed with a Teflon-coated silicon membrane and crimp stopper) containing 50 μg of ethylene oxide per mL. 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 ethylene oxide standard solution. Prepare this solution before use. Separately, transfer about 1.00 g (M_R) of Lauromacrogol to an identical 10 mL vial, add 0.5 mL of the ethylene oxide standard solution and 0.5 mL of the dioxane standard solution and mix to obtain a homogeneous solution. Allow to stand at 70 °C for 45 minutes and use this solution as standard solution (1). Transfer 0.5 mL of the ethylene oxide standard solution to a 10 mL vial, add 0.1 mL of a freshly prepared 10 mg/L acetaldehyde standard solution and 0.1 mL of the dioxane standard solution, and mix to obtain a homogeneous solution. 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 standard solution (1) as directed under Gas Chromatography according to the following operating conditions. Determine the peak area and calculate the amount of ethylene oxide and dioxane in each solution: not more than 1 ppm of ethylene oxide and not more than 10

ppm of dioxane.

$$\text{Amount (ppm) of ethylene oxide} = \frac{A_T \times C}{(A_R \times M_T) - (A_T \times M_R)}$$

A_T = Peak area of ethylene oxide in the test solution

A_R = Peak area of ethylene oxide in standard solution (1)

M_T= Amount (g) of the test specimen in the test solution

M_R = Amount (g) of the test specimen in standard solution (1)

C = Amount (μg) of ethylene oxide added to 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 standard solution (1)

C = Amount (μg) of dioxane added to standard solution (1)

Operating conditions

Detector: A hydrogen flame ionization detector

Column: A glass or quartz capillary tube 0.32 mm in internal diameter and about 30 m in length, the inner surface of which is coated with a 1.0 μm thick layer of poly(dimethyl)siloxane.

Column temperature: Maintain at 50 °C for 5 minutes, increase the temperature at a rate of 5 °C per minute to 180 °C, increase the temperature at a rate of 30 °C per minute to 230 °C and maintain for 5 minutes.

Injection port temperature: A constant temperature of about 150 °C

Head-space sample temperature: 70 °C

Detector temperature: A constant temperature of about 250 °C

Carrier gas: Helium

Split ratio: About 1 : 20

System suitability

System performance: When the procedure is run with 1.0 mL of standard solution (2) under the above operating conditions, the resolution between the peaks of acetaldehyde and ethylene oxide is not less than 2.0 and the signal-to-noise ratio is not less than 5.

Residue on Ignition Not more than 0.2 % (1 g).

Containers and Storage ***Containers*—**Tight containers.

Magnesium Stearate

Magnesium Stearate consists chiefly of magnesium salts of stearic acid ($C_{18}H_{36}O_2$: 284.48) and palmitic acid ($C_{16}H_{32}O_2$: 256.42). Magnesium Stearate, when dried, contains not less than 4.0 % and not more than 5.0 % of magnesium (Mg: 24.31).

Description Magnesium Stearate is a white, light, bulky powder, is smooth to the touch and sticky to the skin, and has no odor or a faint, characteristic odor.
Magnesium Stearate is practically insoluble in water or in ethanol.

Identification (1) Mix 5.0 g of Magnesium Stearate with 50 mL of peroxide-free ether, 20 mL of dilute nitric acid and 20 mL of water in a round-bottom flask and heat to dissolve completely under a reflux condenser. After cooling, transfer the contents of the flask to a separatory funnel, shake, allow the layers to separate and transfer the aqueous layer to a flask. Extract the ether layer with two 4 mL volumes of water and combine these extracts to the main aqueous extract. After washing the combined aqueous extract with 15 mL of peroxide-free ether, transfer to a 50-mL volumetric flask, add water to make exactly 50 mL, mix and use this solution as the test solution (keep this solution for the tests of chloride and sulfate.): the test solution responds to the Qualitative Tests for magnesium salt.

(2) The retention times of the peaks corresponding to stearic acid and palmitic acid in the chromatogram of the test solution correspond to those in the chromatogram of the system suitability solution, as obtained in the Purity (5).

Purity (1) ***Acid or alkali***—Heat 1.0 g of Magnesium Stearate in 20 mL of freshly boiled and cooled water on a water-bath for 1 minute while shaking and filter after cooling. To 10 mL of the filtrate, add 0.05 mL of bromothymol blue TS and add exactly 0.05 mL of 0.1 mol/L hydrochloric acid or 0.1 mol/L sodium hydroxide VS: the color of the solution changes.

(2) ***Chloride***—Perform the test with 10.0 mL of the test solution obtained in the Identification (1). Prepare the control solution with 1.40 mL of 0.02 mol/L hydrochloric acid (not more than 0.10 %).

(3) ***Sulfate***—Perform the test with 10.0 mL of the test solution obtained in the Identification (1). Prepare the control solution with 10.2 mL of 0.01 mol/L sulfuric acid (not more than 1.0 %).

(4) ***Heavy metals***—Heat 1.0 g of Magnesium Stearate weakly at first, then incinerate at about 500 ± 25 °C. After cooling, add 2 mL of hydrochloric acid, evaporate on a water-bath to dryness, add 20 mL of water and 2 mL of dilute acetic acid to the residue and heat for 2 minutes. After cooling, filter this solution through a filter paper, wash the filter paper with 15 mL of water and combine the washing with the filtrate. To the filtrate, add water to make 50 mL and perform the test. Prepare the control solution as follows: evaporate 2 mL of hydrochloric acid on a water-bath to dryness, add 2 mL of dilute acetic acid, 2.0 mL of standard lead solution and water to make 50 mL (not more than 20 ppm).

(5) ***Relative content of stearic acid and palmitic***

acid—Transfer about 0.1 g of Magnesium Stearate, accurately weighed, to a small Erlenmeyer flask fitted with a reflux condenser. Add 5.0 mL of boron trifluoride-methanol TS, mix and reflux for about 10 minutes to dissolve the solids. Add 4.0 mL of heptane through the condenser and reflux for about 10 minutes. After cooling, add 20 mL of saturated sodium chloride solution, shake and allow the layers to separate. Transfer the heptane layer through 0.1 g of anhydrous sodium sulfate, previously washed with heptane, to another flask. Transfer 1.0 mL of this solution to a 10-mL volumetric flask, dilute with heptane to volume, 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 operating conditions and determine the area, *A*, of the methyl stearate peak and the total of the areas, *B*, of all of fatty acid ester peaks. Calculate the % of stearic acid in the fatty acid fraction of Magnesium Stearate by the following equation.

$$\text{Content (\%) of stearic acid} = \frac{A}{B} \times 100$$

Similarly, calculate the % of palmitic acid in Magnesium Stearate. The methyl stearate peak and the total of the methyl stearate and methyl palmitate peaks are not less than 40.0 % and not less than 90.0 % of the total area of all fatty acid ester peaks, respectively, in the chromatogram.

Operating conditions

Detector: A hydrogen flame-ionization detector.

Column: A fused silica capillary column about 0.32 mm in internal diameter and about 30 m in length, the inside coated with a 0.5 μm layer of polyethylene glycol 15000-diepoxide for gas chromatography.

Column temperature: Maintain at 70 °C for about 2 minutes after injection, then program to increase the temperature at the rate of 5 °C per minute to 240 °C and to maintain this temperature for 5 minutes.

Injection port temperature: A constant temperature of about 220 °C.

Detector temperature: A constant temperature of about 260 °C.

Carrier gas: Helium.

Flow rate: Adjust the flow rate so that the retention time of methyl stearate is about 32 minutes.

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, mix and proceed in the test manner as directed for the preparation of the test solution. Use this 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 equivalent to 5 to 15 % of that from the system suitability solution.

System performance: When the procedure is run with 1 μL of the system suitability solution according to the above operating conditions, methyl palmitate and methyl stearate are eluted in this order, with the relative retention time of methyl palmitate to methyl stearate being about 0.86, and with the resolution between these peaks of not less than 5.0.

System reproducibility: When the test is repeated 6 times with 1 μL each of the system suitability solution under the above operating conditions: the relative deviations of the peak area of methyl palmitate and methyl stearate are not more than 6.0 % and the relative deviations of the peak area ratios of methyl palmitate to methyl stearate is not more than 1.0 %.

Loss on Drying Not more than 6.0 % (2 g, 105 °C, a constant weight).

Microbial Limit The total aerobic microbial count is not more than 1000 CFU/g, the total combined yeasts/mould count is not more than 500 CFU/g and *Salmonella* species and *Escherichia coli* are not observed.

Assay Transfer about 0.5 g of previously dried Magnesium Stearate, accurately weighed, to a 250 mL flask, add 50 mL of a mixture of *n*-butanol and dehydrated ethanol (1 : 1), 5 mL of strong ammonia water, 3 mL of ammonium chloride buffer solution, pH 10, 30.0 mL of 0.1 mol/L disodium ethylenediamine tetraacetate VS and 1 to 2 drops of eriochrome black T TS and mix. Heat at 45 °C to 50 °C to make the solution clear and after cooling, titrate the excess disodium ethylenediamine tetraacetate with 0.1 mol/L zinc sulfate VS until the solution changes from blue to purple. Perform a blank determination and make any necessary correction.

Each mL of 0.1 mol/L disodium ethylenediamine tetraacetate = 2.4305 mg of Mg

Containers and Storage ***Containers*—**Tight containers.

Medicinal Soap

Medicinal Soap is the sodium salts of fatty acids.

Description Medicinal Soap appears as white to pale yellow powder or granules, and has a characteristic odor free from rancidity.

Medicinal Soap 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, add 60 mL of dilute sulfuric acid slowly and warm in a water-bath for 20 minutes. After cooling, filter off the precipitate and wash with warm water until the washing no longer shows acidity to methyl orange TS. Transfer the precipitate to a small beaker and heat on a water-bath to complete separation of water and 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 between 18 °C and 28 °C. Acid value is between 185 and 205 and Iodine value is between 82 and 92.

Purity (1) ***Acid or alkali***—Dissolve 5.0 g of Medicinal Soap in 85 mL of neutralized ethanol by warming on a water-bath, filter while hot through absorbent cotton and wash the filter and the residue three times with 5 mL volumes of hot neutralized ethanol. Combine the filtrate and the washings, add hot neutralized ethanol to make exactly 100 mL and perform the following tests quickly using this solution as the test solution at 70 °C.

(i) Add 3 drops of phenolphthalein TS and 0.20 mL of 0.1 mol/L sodium hydroxide VS to 40 mL of the test solution: a red color develops.

(ii) Add 3 drops of phenolphthalein TS and 0.20 mL of 0.05 mol/L sulfuric acid VS to 40 mL of the test solution: no red color develops.

(2) ***Heavy metals***—Proceed with 1.0 g of Medicinal Soap according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 20 ppm).

(3) ***Ethanol-insoluble substances***—Weigh accurately about 2 g of Medicinal Soap, dissolve by warming in 100 mL of neutralized ethanol, filter the solution through a glass filter, wash the residue with 100 mL of hot neutralized ethanol and dry at 105 °C for 4 hours: the residue is not more than 1.0 %.

(4) ***Water-insoluble substances***—Wash thoroughly the dried substances obtained in (3) with 200 mL of water and dry at 105 °C for 4 hours: the residue is not more than 0.15 %.

(5) ***Alkali carbonates***—Take the washings obtained in (4), add 3 drops of methyl orange TS and 2 mL of 0.05 mol/L sulfuric acid VS: a red color develops.

Loss on Drying Not more than 5.0 % in the case of the powder and not more than 10.0 % in the case of the granules. Weigh accurately about 0.5 g of Medicinal Soap in a tared beaker, add 10 g of sea sand, previously dried at 105 °C for 1 hour, and again weigh the beaker. Add 10 mL of ethanol, evaporate on a water-bath to dryness with thorough stirring and dry at 105 °C for 3 hours.

Containers and Storage ***Containers—***Well-closed containers.

Mentha Oil

Mentha Oil is the essential oil which is distilled with steam from the aerial parts of *Mentha arvensis* Linné var. *piperascens* Malinvaud (Labiatae) and from which solids are removed after cooling. Mentha Oil contains not less than 30.0 % of menthol ($C_{10}H_{20}O$: 156.27).

Description Mentha Oil is a colorless or pale yellow, clear liquid, has a characteristic, pleasant aroma and a pungent taste, followed by a cool aftertaste. Mentha Oil is miscible with ethanol, with dehydrated ethanol, with warm ethanol, or with ether.
Mentha Oil is practically insoluble in water.

Refractive Index n_D^{20} : 1.455 ~ 1.467.

Specific Optical Rotation $[\alpha]_D^{20}$: -17.0 ~ -36.0 ° (100 mm).

Specific Gravity $[\alpha]_D^{20}$: 0.885 ~ 0.910.

Acid Value Not more than 1.0.

Purity (1) ***Clarity of solution***—Take 1.0 mL of Mentha Oil, add 3.5 mL of diluted ethanol (7 in 10) and shake: Mentha Oil dissolves clearly. To the solution, add 10 mL of ethanol: the solution is clear or has no more turbidity, if any, than the following control solution.

Control solution—Take 0.70 mL of 0.01 mol/L hydrochloric acid VS, 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.

(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 standard lead solution (not more than 40 ppm).

(3) ***Dimethyl sulfide***—To 1 mL of the distillate obtained by distilling about 25 mL of Mentha Oil, add carefully 5 mL of mercury (II) chloride TS: no white film is produced within 1 minute at the interface of the two liquids.

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 operating conditions. Calculate the ratios,

Q_T and Q_S, of the peak area of menthol to that of the internal standard, for the test solution and the standard solution, respectively.

$$\text{Amount (g) of menthol } (C_{10}H_{20}O) = \text{amount (g) of } l\text{-Menthol RS} \times \frac{Q_T}{Q_S}$$

Internal standard solution—A solution of *n*-ethyl caprylate in ethanol (1 in 25).

Operating conditions

Detector: A hydrogen flame-ionization detector.

Column: A column about 3 mm in internal diameter and about 2 m in length, packed with 25 % of polyethylene glycol 6000 for gas chromatography supported on acid washed 180 μm to 250 μm siliceous earth for gas chromatography.

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 under the above operating conditions and use a column giving elution of the internal standard and *l*-menthol in this order with a resolution between the two peaks being not less than 5.

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light-resistant.

Methylcellulose

[*9004-67-5*]

Methylcellulose is a methyl ether of cellulose.
Methylcellulose contains not less than 26.0 % and not more than 33.0 % of methoxyl group (-OCH_3: 31.03), calculated on the dried basis.
The kinematic viscosity of Methylcellulose is shown in millipascal second (mPa·s) on the label.

Description Methylcellulose appears as a white to yellowish white, powder or granules.
Methylcellulose is practically insoluble in dehydrated ethanol.
Methylcellulose swells, when water is added and forms a clear or slightly turbid, viscous liquid.

Identification (1) Disperse evenly 1 g of Methylcellulose over the surface of 100 mL of water in a beaker, while gently tapping the top of the container, if necessary, and allow the beaker to stand: it aggregates on the surface of water.

(2) Add 1.0 g of Methylcellulose to 100 mL of hot water, and stir: it becomes a suspension. Cool the suspension to 5 °C, and stir: the resulting liquid is a clear or a slightly cloudy, viscous fluid.

(3) Take 0.1 mL of the test solution obtained in (2), add 9 mL of diluted sulfuric acid (9 in 10), shake, heat in a water-bath for exactly 3 minutes, immediately cool in an ice-bath, add carefully 0.6 mL of ninhydrin TS, shake and allow to stand at 25 °C: a red color develops immediately and it does not change to purple within 100 minutes.

(4) Pour and spread out 2 to 3 mL of the viscous fluid obtained in (2) onto a glass plate, and allow the water to evaporate: a transparent film results.

(5) Pipet 50 mL of water, add exactly 50 mL of the viscous fluid obtained in (2), and warm to rise the temperature at a rate of 2 to 5 °C per minute while stirring: the temperature, when a white turbidity of the solution starts to increase, is not less than 50 °C.

Viscosity ***Method I***: Apply to Methylcellulose having a labeled viscosity of less than 600 mPa·s. Place an exact portion of Methylcellulose, equivalent to 4.000 g on the dried basis, in a tared, wide-mouth bottle, add hot water to make 200.0 g, stopper the bottle, mix with a stirrer 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 dispersed solution, and dissolve by a continuous stirring in a water-bath not exceeding 5 °C for 20 to 40 minutes. Add cooled water, if necessary, to make 200.0 g, and centrifuge the solution if necessary to expel any entrapped air bubbles. Use the so obtained solution as the test solution. Perform the test with the test solution at 20±0.1 °C as directed in the Method I under the Viscosity Determination: not less than 80 % and not more than 120 % of the labeled viscosity.

Method II: Apply to Methylcellulose having a labeled viscosity of not less than 600 mPa·s. Place an exact portion of Methylcellulose, equivalent to 10.00 g on the dried basis, in a tared, wide-mouth bottle, add hot water to make 500.0 g, stopper the bottle, and prepare the test solution in the same manner as directed in Method I . Perform the test with the test solution at 20±0.1 °C as directed in the Method II (2) under the Viscosity Determination, using a single cylinder –type rotational viscometer, according to the following operating conditions: not less than 75 % and not more than 140 % of the labeled viscosity.

Operating conditions

Apparatus: Brookfield type viscometer LV model
Rotor No., rotation frequency, and conversion factor : Use the conditions as directed in the following table, depending on the labeled viscosity.

Labeled viscosity (mPa·s)	Rotor No.	Rotation frequency /min	Conversion factor
Not less than 600 and less	3	60	20

than 1400			
Not less than 1400 and less than 3500	3	12	100
Not less than 3500 and less than 9500	4	60	100
Not less than 9500 and less than 99500	4	6	1000
Not less than 99500	4	3	2000

Procedure of apparatus: Read value after 2 minutes of rotation, and stop the rotation for 2 minutes. Repeat this procedure two more times, and average three observed values.

pH Allow the test solution obtained in the Viscosity to stand at 20±2 °C for 5 minutes: the pH of the so obtained solution is between 5.0 and 8.0.

Purity (1) ***Chloride***—Transfer 0.5 g of Methylcellulose to a beaker, add 30 mL of boiling water, mix well and filter while hot with an insulated funnel. Wash the residue in the beaker and on the filter paper with three 15 mL volumes of boiling water. Combine the washings with the filtrate, add water to make 100 mL and use this solution as solution A. To 5 mL of solution A, add 6 mL of dilute nitric acid and use this solution as the test solution. Perform the test with the test solution as directed under Chloride Test: not more than the amount equivalent to 0.4 mL of 0.01 mol/L hydrochloric acid.

(2) ***Sulfate***—To 40 mL of solution A prepared under Chloride, add 1 mL of dilute hydrochloric acid and use this solution as the test solution. Perform the test with the test solution as directed under Sulfate Test: not more than the amount equivalent to 0.4 mL of 0.01 mol/L sulfuric acid.

(3) ***Heavy metals***—Put 1.0 g of Methylcellulose in a 100-mL Kjeldahl flask, add a sufficient amount of a mixture of nitric acid and sulfuric acid (5 : 4) to wet the test specimen, and heat gently. Repeat this procedure until to use totally 18 mL of the mixture of nitric acid and sulfuric acid (5 : 4). Then boil gently until the solution changes to black. After cooling, add 2 mL of nitric acid, and heat until the solution changes to black. Repeat this procedure until the solution no longer changes to black, and heat strongly until dense white fumes are evolved. After cooling, add 5 mL of water, boil gently until dense white fumes are evolved, then heat until the volume of the solution becomes to 2 to 3 mL. After cooling, if the solution reveals yellow color by addition of 5 mL of water, add 1 mL of hydrogen peroxide, and heat until the volume of the solution becomes to 2 to 3 mL. After cooling, dilute the solution with 2 to 3 mL of water, transfer to a Nessler tube, add water to make 25 mL, and use this solution as the test solution. Separately, put 2.0 mL of standard lead solution in a 100-mL Kjeldahl flask, add 18 mL of the mixture of nitric acid and sulfuric acid (5 : 4) and an amount of nitric acid equal to that used for preparation of the test solution, and heat until white fumes are evolved . After cooling, add 10 mL of water. In the case where hydrogen peroxide is added for the preparation of the test solution, add the same amount of hydrogen peroxide, then proceed in the same manner for preparation of the test solution, and use so obtained solution as the control solution. Adjust the pHs of the test solution and the control solution to 3.0 - 4.0 with strong ammonia solution water, and add water to make 40 mL each. To these solutions add 1.2 mL each of thioacetamide-alkaline glycerin TS, 2 mL each of acetate buffer solution, pH 3.5 and water to make 50 mL each. After allowing to stand for 5 minutes, observe vertically both tubes on a white background: the color obtained from the test solution is not more intense than that from the control solution (not more than 20 ppm).

(4) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat, place 10 mg to 300 mg of Methylcellulose on top then 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, place only the test specimen in a nickel boat without the additives. Place the boat inside the furnace and heat to about 900 °C with a current of air or oxygen at 0.5 L/minute to 1 L/minute. Elute the mercury and sample in a sampling tube. Transfer the mercury vapor to a cold vapor atomic absorption spectrometer by heating the sampling 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 plot a calibration curve from the absorbances. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 ng to 200 ng.

Additive—Use (a) aluminum oxide or (b) a mixture of calcium hydroxide and sodium carbonate (1:1) and activate at 950 °C for 30 minutes before use.

(5) ***Cadmium***—Weigh accurately 5.0 g of Methylcellulose 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 5 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 1.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Cadmium hollow cathode lamp
Wavelength: 228.8 nm

(6) ***Lead***—Weigh accurately 5.0 g of Methylcellulose 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 standard lead 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 2.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(7) ***Arsenic***—Proceed with 0.5 g of Methylcellulose according to Method 3 and perform the test (not more than 4 ppm).

Loss on Drying Not more than 5.0 % (1 g, 105 °C, 1 hour).

Residue on Ignition Not more than 1.5 % (1 g).

Assay (1) ***Apparatus***—Reaction bottle: A 5-mL pressure-tight glass vial, having 20 mm in outside diameter and 50 mm in height, the neck 20 mm in outside diameter and 13 mm in internal diameter, equipped with a septum of butyl –rubber processed having the surface with fluoroplastics, which can be fixed tightly to vial with aluminum seal, or equivalent.
Heater: A square-shaped aluminum block, having holes 20 mm in diameter and 32 mm in depth, so that the reaction bottle can be fitted. Use a heater capable of stirring the content of the reaction bottle by means of magnetic stirrer or by connecting the reaction bottle to a reciprocal shaker of about 100 times per minute.

(2) ***Procedure***—Weigh accurately about 65 mg of Methylcellulose, transfer to the reaction bottle, add 60 mg to 100 mg of adipic acid, 2.0 mL of the internal standard solution and 2.0 mL of hydroiodic acid, stopper the bottle immediately, and weigh accurately. Stir or shake for 60 minutes while heating so that the temperature of the bottle content is 130±2 °C. In the case when the stirrer or shaker is not available, heat for 30 minutes with repeated shaking at 5-minute intervals by hand, and continue heating for an additional 30 minutes. Allow the bottle to cool, and again weigh accurately. If the mass loss is less than 0.50 % or there is no evidence of a leak, use the upper layer of the mixture as the test solution. Separately, put 60 to 100 mg of adipic acid in a reaction bottle, 2.0 mL of the internal standard solution and 2.0 mL of hydroiodic acid, stopper the bottle immediately, and weigh accurately. Add 45 µL of iodomethane for assay through the septum using a micro-syringe, weigh accurately, stir 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 ratios , Q_T and Q_S, of the peak area of iodomethane to that of the internal standard for the test solution and the standard solution, respectively.

$$\text{Content (\%) of methoxyl group (-CH}_3\text{O)} = \frac{Q_T}{Q_S} \times \frac{W_S}{W} \times 21.86$$

W_S: Amount (mg) of iodomethane in the standard solution

W : Amount (mg) of Methylcellose taken, 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 hydrogen flame-ionization detector.

Column: A glass column 3 to 4 mm in internal diameter and 1.8–3 m in length, packed with siliceous earth for gas chromatography, 125 to 150 μm in diameter, coated with methyl silicone polymer at the ratio of 10 to 20 %.

Column temperature: A constant temperature of about 100 °C.

Carrier gas: Helium for thermal conductivity detector, or Helium or Nitrogen for hydrogen flame-ionization detector.

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

System suitability

System performance: When the procedure is run with 1 to 2 μL of the standard solution under the above operating conditions, iodomethane and the internal standard are eluted in this order, with complete separations among the three peaks.

Containers and Storage ***Containers*—**Well-closed containers.

Nitrogen

N_2: 28.01

Nitrogen contains not less than 99.5 % and not more than 101.0 % of nitrogen (N_2).

Description Nitrogen is colorless gas and is odorless. 1 mL of Nitrogen dissolves in 65 mL of water and in 9 mL of ethanol at 20 °C and at a pressure of 101.3 kPa. 1000 mL of Nitrogen at 0 °C and at a pressure of 101.3 kPa weighs about 1.251 g.
Nitrogen is inert and does not support combustion.

Identification The flame of a burning wood splinter is extinguished immediately in an atmosphere of Nitrogen.

Purity (1) ***Carbon dioxide***—Maintain the containers of Nitrogen at a temperature between 18 and 22 °C for more than 6 hours before the test and correct the volume to be at 20 °C and 101.3 kPa. Pass 1000 mL of Nitrogen into 50 mL of barium hydroxide TS in a Nessler tube during 15 minutes through a delivery tube with an orifice, approximately 1 mm in diameter, keeping the end of the tube at a distance of 2 mm from the bottom of the Nessler tube: any turbidity produced is not more than that produced in the following control solution.

Control solution—Take 50 mL of barium hydroxide TS in a Nessler tube, add 1 mL of a solution of 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 in the test solution and the standard solution as obtained in the Assay: the amount of oxygen is not more than 0.5 %.

$$\text{Amount (vol\%) of oxygen} = \frac{A_T}{A_S}$$

Assay Collect the sample as directed under the Purity. Introduce 1.0 mL of Nitrogen into a gas-measuring tube or syringe for gas chromatography from a metal cylinder with a vaccum valve, through a directly connected polyvinyl chloride tube. Perform the test with this solution as directed under the Gas Chromatography according to the following operating conditions. Measure the peak area, A_T of oxygen. Separately, introduce 1.0 mL of oxygen into the gas mixer, add carrier gas to make exactly 100 mL , mix thoroughly and use this mixture as the standard gas mixture. Proceed with 1.0 mL of this mixture in the same manner under Nitrogen and measure the peak area, A_S of oxygen.

$$\text{Amount (\%) of Nitrogen } (N_2) = 100 - \frac{A_T}{A_S}$$

Operating conditions

Detector: A thermal-conductivity detector.

Column: A column, about 3 mm in internal diameter and about 3 m in length, packed with zeolite for Gas Chromatography (250 to 350 μm in particle diameter).

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: Introduce 1.0 mL of oxygen into the gas mixer, add Nitrogen to make 100 mL and mix thoroughly. When the procedure is run with 1.0 mL of this mixture according to the above operating conditions, oxygen and nitrogen are elutied in this order and clearly resoluted each other.

System reproducibility: When the test is repeated 5 times according to the above conditions with the standard gas mixture: the relative standard deviation of peak area of oxygen is not more than 2.0 %.

Containers and Storage ***Containers*—**Metal cylinders.

***Storage*—**Not exceeding 40 °C.

Olive Oil

Olive Oil is the fixed oil obtained by expression from the ripe fruit of *Olea europaea* Linné (Oleaceae).

Description Olive oil is pale yellow oil, has faint odor which is not rancid, and has bland taste.
Olive oil is miscible with ether or with petroleum ether.
Olive oil is slightly soluble in ethanol.
The whole or a part of it congeals at between 0 °C and 6 °C.

Congealing point of the fatty acids—17 ~ 26 °C.

Saponification Value 186 ~ 194.

Unsaponifiable Matters Not more than 1.5 %.

Specific Gravity d_{25}^{25} : 0.908 ~ 0.914.

Acid Value Not more than 1.0.

Iodine Value 79 ~ 88.

Purity (1) ***Drying oil***—Mix 2 mL of Olive Oil with 10 mL of diluted nitric acid (1 in 4), add 1 g of powdered sodiun nitrite little by little with thorough shaking and allow to stand in a cold place for 4 to 10 hours: the mixture congeals to a white solid.

(2) ***Peanut oil***—Weigh exactly about 1.0 g of Olive Oil, dissolve in 60 mL of sulfuric acid-hexane-methanol TS, boil for 2.5 hours on a water-bath under a reflux condenser, cool, transfer to a separatory funnel and add 100 mL of water. Wash the flask with 50 mL of petroleum ether, add the washing to the separatory funnel, shake, allow to stand and separate 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 volumes of water until the washings show no more acidity to methyl orange TS. Then add 5 g of anhydrous sodium sulfate, shake, filter, wash the anhydrous sodium sulfate twice with 10 mL volumes of petroleum ether, filter the washings using the former funnel, combine the filtrates, and distil the petroleum ether on a water-bath passing 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 exactly 2 mL of this solution, add acetone to make exactly 20 mL and use this solution as the standard solution. Perform the test with exactly 2 μL each of the test solution and the standard solution as directed under the Gas Chromatography according to the following operating conditions. Measure the peak heights, H_T and H_S, of methyl behenate for the test solution and the standard solution, respectively: H_T is not higher than H_S.

Operating conditions

Detector: A hydrogen flame-ionization detector.

Column: A glass column, about 3 mm in internal diameter and about 2 m in length, packed with silanized siliceous earth for gas chromatography (150 μm to 180 μm in particle diameter), coated with polyethylene glycol 20 M in a ratio of 5 %.

Column temperature: A constant temperature of about 220 °C.

Carrier gas: Nitrogen.

Flow rate: Adjust the flow rate so that the retention time of methyl benhenate is about 18 minutes.

Detection sensitivity: Adjust the detection sensitivity so that the peak height of methyl behenate obtained from 2 μL of the standard solution is 5 mm to 10 mm.

(3) ***Heavy metals***—Proceed with 1.0 g of Olive Oil according to Method 2 and perform the test. Prepare the control solution with 1.0 mL of standard lead solution (not more than 10 ppm).

Containers and Storage ***Containers***—Tight containers.

Orange Oil

Orange Oil is the essential oil obtained by expression from the peel of the edible fruit of *Citrus* species (Rutaceae).

Description Orange Oil is a yellow to yellow-brown liquid, and has a characteristic, aromatic odor and slightly bitter taste.
Orange Oil is miscible with an equal volume of ethanol with turbidity.

Refractive Index n_D^{20} : 1.472 ~ 1.474.

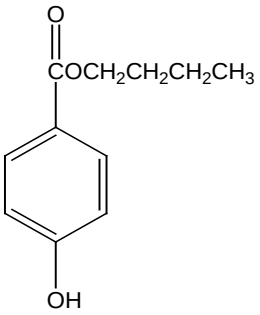
Specific Optical Rotation $[\alpha]_D^{20}$: +85 ~ +99° (100 mm).

Specific Gravity d_{20}^{20} : 0.842 ~ 0.848.

Purity ***Heavy metals***—Proceed with 1.0 mL of Orange Oil according to Method 2 and perform the test. Prepare the control solution with 4.0 mL of standard lead solution (not more than 40 ppm).

Containers and Storage *Containers*—Tight containers.
***Storage*—**Light-resistant.

Butylparaben



Butyl Parahydroxybenzoate $C_{11}H_{14}O_3$: 194.23

Butyl 4-hydroxybenzoate [*94-26-8*]

Butylparaben, when dried, contains not less than 98.0 % and not more than 102.0 % of butylparaben ($C_{11}H_{14}O_3$).

Description Butylparaben appears as colorless crystals or white, crystalline powder.
Butylparaben is freely soluble in ethanol or in acetone, and practically insoluble in water.

Identification Determie the infrared spctra of Butylparaben and Butylparaben RS as directed in the potassium bromide disk method under Infrared Spectrophotometry : both spectra exhibit similar intensities of absorption at the same wavenumbers.

Melting Point 68 ~ 71 °C.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Butylparaben in 100 mL of dehydrated ethanol : the solution is clear and not more intensely colored than the following control solution.

Control solution—To 5.0 mL of cobalt (II) chloride colorimetric stock solution, 12.0 mL of iron (III) chloride colorimetric stock solution and 2.0 mL of cupric sulfate colorimetric stock solution add water to make 1000 mL.

(2) ***Acidity***—Dissolve 0.20 g of Butylparaben in 5 mL of dehydrated ethanol, add 5 mL of freshly boiled and cooled water and 0.1 mL of bromocresol green-sodium hydroxide-ethanol TS, then add 0.1 mL of 0.1 mol/L sodium hydroxide VS : the solution shows a blue color.

(3) ***Heavy metals***—Dissolve 1.0 g of Butylparaben in 25 mL of acetone, add 2 mL of dilute acetic acid and water to make 50 mL, and perform the test using this solution as the test solution. Prepare the control solution as follows : to 2.0 mL of standard lead solution add 25 mL of acetone, 2 mL of dilute acetic acid, and water to make 50 mL (not more than 20 ppm).

(4) ***Related substances***—Dissolve 0.10 g of Butylparaben in 10 mL of acetone, and use this solution as the test solution. Pipet 0.5 mL of the test solution, add acetone to make exactly 100 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 2 μL each of the test solution and standard solution on a plate of silica gel with a fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of methanol, water and acetic acid (100) (70 : 30 : 1) to a distance of about 15 cm, and air-dry the plate. Examine under ultraviolet light (principal wavelength : 254 nm) : the spot other than the principal spot is not more intense than the spot obtained with the standard solution.

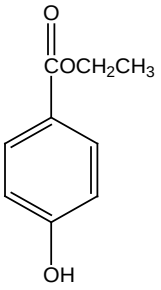
Residue on Ignition Not more than 0.10 % (1 g).

Assay Weigh accurately about 1 g of Butylparaben, add exactly 20 mL of 1 mol/L sodium hydroxide VS, heat at about 70 °C for 1 hour, and immediately cool in ice. Titrate the excess sodium hydroxide with 0.5 mol/L sulfuric acid VS up to the second equivalent point(potentiometric titration, Endpoint Detection Method in Titrimetry). Perform a blank determination.

Each mL of 1 mol/L sodium hydroxide VS
= 194.2 mg of $C_{11}H_{14}O_3$

Containers and Storage ***Containers*—**Well-closed containers.

Ethylparaben



Ethyl Parahydroxybenzoate $C_9H_{10}O_3$: 166.17

Ethyl 4-hydroxybenzoate [*120-47-8*]

Ethylparaben, when dried, contains not less than 98.0 % and not more than 102.0 % of ethylparaben ($C_9H_{10}O_3$).

Description Ethylparaben appears as colorless crystals or white, crystalline powder.
Ethylparaben is freely soluble in ethanol or in acetone, and very slightly soluble in water.

Identification Determie the infrared spctra of Ethylparaben and Ethylparaben RS as directed in the potassium bromide disk method under Infrared Spec-

trophotometry : both spectra exhibit similar intensities of absorption at the same wavenumbers.

Melting Point 115 ~ 118 °C.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Ethylparaben in 100 mL of dehydrated ethanol : the solution is clear and not more intensely colored than the following control solution.

Control solution—To 5.0 mL of cobalt (II) chloride colorimetric stock solution, 12.0 mL of iron (III) chloride colorimetric stock solution and 2.0 mL of cupric sulfate colorimetric stock solution add water to make 1000 mL.

(2) ***Acidity***—Dissolve 0.20 g of Ethylparaben in 5 mL of dehydrated ethanol, add 5 mL of freshly boiled and cooled water and 0.1 mL of bromocresol green-sodium hydroxide-ethanol TS, then add 0.1 mL of 0.1 mol/L sodium hydroxide VS : the solution shows a blue color.

(3) ***Heavy metals***—Dissolve 1.0 g of Ethylparaben in 25 mL of acetone, add 2 mL of dilute acetic acid and water to make 50 mL, and perform the test using this solution as the test solution. Prepare the control solution as follows : to 2.0 mL of Standard Lead Solution add 25 mL of acetone, 2 mL of dilute acetic acid, and water to make 50 mL(not more than 20 ppm)

(4) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat and place 10 mg to 300 mg of Ethylparaben on top. Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. Place the boat inside the furnace and heat to about 900 °C with a current of air or oxygen at 0.5 L/minute to 1 L/minute. Elute the mercury and sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 ng to 200 ng.

Additive—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 Ethylparaben 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 solution as directed under Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 2.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(6) ***Arsenic***—Proceed with 0.5 g of Ethylparaben according to Method 3 and perform the test (not more than 4 ppm).

(7) ***Related substances***—Dissolve 0.10 g of Ethylparaben in 10 mL of acetone, and use this solution as the test solution. Pipet 0.5 mL of the test solution, add acetone to make exactly 100 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 2 μL each of the test solution and standard solution on a plate of silica gel with a fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of methanol, water and acetic acid (100) (70 : 30 : 1) to a distance of about 15 cm, and air-dry the plate. Examine under ultraviolet light(principal wavelength : 254 nm) : the spot other than the principal spot is not more intense than the spot obtained with the standard solution.

Loss on Drying Not more than 0.5 % (1 g, silica gel, 5 hours).

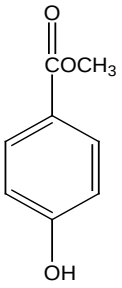
Residue on Ignition Not more than 0.10 % (1 g).

Assay Weigh accurately about 1.0 g of Ethylparaben, add exactly 20 mL of 1 mol/L sodium hydroxide VS, heat at about 70 °C for 1 hour, and immediately cool in ice. Titrate the excess sodium hydroxide with 0.5 mol/L sulfuric acid VS up to the second equivalent point(potentiometric titration, Endpoint Detection Method in Titrimetry). Perform a blank determination.

Each mL of 1mol/L sodium hydroxide VS
= 166.2 mg of $C_9H_{10}O_3$

Containers and Storage ***Containers*—**Well-closed containers.

Methylparaben



Methyl Parahydroxybenzoate $C_8H_8O_3$: 152.15

Methyl 4-hydroxybenzoate [*98-76-3*]

Methylparaben, when dried, contains not less than 98.0 % and not more than 102.0 % of methylparaben ($C_8H_8O_3$).

Description Methylparaben appears as colorless crystals or white, crystalline powder.
Methylparaben is freely soluble in ethanol or in acetone, and slightly soluble in water.

Identification Determie the infrared spctra of Methylparaben and Methylparaben RS as directed in the potassium bromide disk method under Infrared Spectrophotometry : both spectra exhibit similar intensities of absorption at the same wavenumbers.

Melting Point 125 ~ 128 °C.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Methylparaben in 100 mL of dehydrated ethanol : the solution is clear and not more intensely colored than the following control solution.

Control solution—To 5.0 mL of cobalt (II) chloride colorimetric stock solution, 12.0 mL of iron (III) chloride colorimetric stock solution and 2.0 mL of cupric sulfate colorimetric stock solution add water to make 1000 mL.

(2) ***Acidity***—Dissolve .020 g of Methylparaben in 5 mL of dehydrated ethanol, add 5 mL of freshly boiled and cooled water and 0.1 mL of bromocresol green-sodium hydroxide-ethanol TS, then add 0.1 mL of 0.1 mol/L sodium hydroxide VS : the solution shows a blue color.

(3) ***Heavy metals***—Dissolve 1.0 g of Methylparaben in 25 mL of acetone, add 2 mL of dilute acetic acid and water to make 50 mL, and perform the test using this solution as the test solution. Prepare the control solution as follows : to 2.0 mL of standard lead solution add 25 mL of acetone, 2 mL of dilute acetic acid, and water to make 50 mL(not more than 20 ppm)

(4) ***Lead*—**Weigh accurately 5.0 g of Methylparaben 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 solution as directed under Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 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 Methylparaben according to Method 3 and perform the test (not more than 4 ppm).

(6) ***Related substances***—Dissolve 0.10 g of Methylparaben in 10 mL of acetone, and use this solution as the test solution. Pipet 0.5 mL of the test solution, add acetone to make exactly 100 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 2 μL each of the test solution and the

standard solution on a plate of silica gel with a fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of methanol, water and acetic acid (100) (70 : 30 : 1) to a distance of about 15 cm, and air-dry the plate. Examine under ultraviolet light(principal wavelength : 254 nm) : the spot other than the principal spot is not more intense than the spot obtained with the standard solution.

Loss on Drying Not more than 0.5 % (1 g, silica gel, 5 hours).

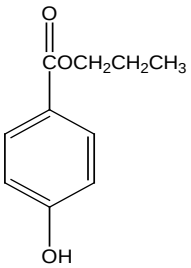
Residue on Ignition Not more than 0.10 % (1 g).

Assay Weigh accurately about 1 g of Methylparaben, add exactly 20 mL of 1 mol/L sodium hydroxide VS, heat at about 70 °C for 1 hour, and immediately cool in ice. Titrate the excess sodium hydroxide with 0.5 mol/L sulfuric acid VS up to the second equivalent point(potentiom-etric titration, Endpoint Detection Method in Titrimetry). Perform a blank determination.

Each mL of 1 mol/L sodium hydroxide VS
= 152.1 mg of $C_8H_8O_3$

Containers and Storage ***Containers*—**Well-closed containers.

Propylparaben



Propyl Parahydroxybenzoate $C_{10}H_{12}O_3$: 180.20

Propyl 4-hydroxybenzoate [*94-13-3*]

Propylparaben, when dried, contains not less than 98.0 % and not more than 102.0 % of propylparaben ($C_{10}H_{12}O_3$).

Description Propylparaben appears as colorless crystals or white, crystalline powder.
Propylparaben is freely soluble in ethanol or in acetone, and very slightly soluble in water.

Identification Determie the infrared spctra of Propyl-paraben and Propylparaben RS as directed in the potassium bromide disk method under Infrared Spectrophotometry : both spectra exhibit similar intensities of absorption at the same wavenumbers.

Melting Point 96 ~ 99 °C.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Propylparaben in 10 mL of dehydrated ethanol : the solution is clear and not more intensely colored than the following control solution.

Control solution—To 5.0 mL of cobalt (II) chloride colorimetric stock solution, 12.0 mL of iron (III) chloride colorimetric stock solution and 2.0 mL of cupric sulfate colorimetric stock solution add water to make 1000 mL.

(2) ***Acidity***—Dissolve .020 g of Propylparaben in 5 mL of dehydrated ethanol, add 5 mL of freshly boiled and cooled water and 0.1 mL of bromocresol green-sodium hydroxide-ethanol TS, then add 0.1 mL of 0.1 mol/L sodium hydroxide VS : the solution shows a blue color.

(3) ***Heavy metals***—Dissolve 1.0 g of Propylparaben in 25 mL of acetone, add 2 mL of dilute acetic acid and water to make 50 mL, and perform the test using this solution as the test solution. Prepare the control solution as follows : to 2.0 mL of standard lead solution add 25 mL of acetone, 2 mL of dilute acetic acid, and water to make 50 mL(not more than 20 ppm)

(4) ***Related substances***—Dissolve 0.10 g of Propyl-paraben in 10 mL of acetone, and use this solution as the sample solution. Pipet 0.5 mL of the sample solution, add acetone to make exactly 100 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography. Spot 2 μL each of the sample solution and standard solution on a plate of silica gel with a fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of methanol, water and acetic acid (100) (70 : 30 : 1) to a distance of about 15 cm, and air-dry the plate. Examine under ultraviolet light(principal wavelength : 254 nm) : the spot other than the principal spot is not more intense than the spot obtained with the standard solution.

Residue on Ignition Not more than 0.10 % (1 g).

Assay Weigh accurately about 1.0 g of Propylparaben, add exactly 20 mL of 1 mol/L sodium hydroxide VS, heat at about 70 °C for 1 hour, and immediately cool in ice. Titrate the excess sodium hydroxide with 0.5 mol/L sulfuric acid VS up to the second equivalent point(potentiom-etric titration, Endpoint Detection Method in Titrimetry). Perform a blank determination.

Each mL of 1 mol/L sodium hydroxide VS
= 180.2 mg of $C_{10}H_{12}O_3$

Containers and Storage ***Containers*—**Well-closed containers.

Paraffin

Paraffin is a mixture of solid hydrocarbons obtained from petroleum.

Description Paraffin is colorless or white, slightly clear, crystalline mass, odorless and tasteless.
Paraffin is sparingly soluble in ether and practically insoluble in water, in ethanol or in dehydrated 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 ignore: 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 with shaking carefully: the odor of hydrogen sulfide is perceptible.

Melting Point 50 ~ 75 °C (Method 2).

Purity (1) ***Acid or alkali***—Boil 10.0 g of Paraffin with 10 mL of hot water and 1 drop of phenolphthalein TS in a water-bath for 5 minutes and shake vigorously: a red color is not produced. Add 0.20 mL of 0.02mol/L sodium hydroxide VS to this solution and shake: a red color is produced.

(2) ***Heavy metals***—Ignite 2.0 g of Paraffin in a crucible, first moderately until chared, then between 450 °C and 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 and perform the test. Prepare the control solution as follows: to 2.0 mL of standard lead solution, add 2 mL of dilute acetic acid and water to make 50 mL (not more than 10 ppm).

(3) ***Arsenic***—Prepare the test solution with 1.0 g of Paraffin according to Method 3 and perform the test (not more than 2 ppm).

(4) ***Sulfur compounds***—Take 4.0 g of Paraffin, add 2 mL of dehydrated 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 aqueous 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 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 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 Ultraviolet-visible Spectrophotometry, using as the blank the clear lower liquid obtained by shaking vigorously for 1 minute 25 mL of *n*-heptane and 5.0 mL of dimethylsulfoxide. Separately, dissolve Naphthalene RS in dimethylsul-foxide 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 Ultraviolet-visible Spectrophotometry, using dimethylsulfoxide as the blank. The absorbance of the test solution between 265 nm and 420 nm is not more than one-third of the absorbance of the standard solution at 278 nm.

(6) ***Readily carbonizable substances***—Melts 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 test of readily carbonizable substances and warm at 70 °C for 5 minutes in a water-bath. Remove the tube from the water-bath, immediately shake vigorously and vertically for 3 seconds and warm for 1 minutes in a water-bath at 70 °C. Repeat this procedure five times: the color of the sulfuric acid layer is not more intense than that of the following control solution.

Control solution—Add 1.5 mL of cobaltous chloride colorimetric stock solution, 0.5 mL of cupric sulfate colorimetric stock solution and 5 mL of liquid paraffin to 3.0 mL of ferric chloride colorimetric stock solution and shake vigorously.

Containers and Storage ***Containers—***Well-closed containers.

Liquid Paraffin

Liquid Paraffin is a mixture of liquid hydrocarbons obtained from petrolatum. Tocopherol of a suitable from may be added at a concentration not exceeding 0.001 % as a stabilizer.

Description Liquid Paraffin is colorless, transparent, oily liquid, nearly free from fluorescence, and is odorless and tasteless.
Liquid Paraffin is freely soluble in ether, very slightly soluble in dehydrated ethanol and practically insoluble in water or in ethanol.

Boiling point—Above 300 °C.

Identification Proceed as directed in the Identification under Paraffin.

Specific Gravity d_{20}^{20} : 0.860 ~ 0.890.

Viscosity Not less than 37mm^2/s (Method 1, 37.8 °C).

Purity (1) ***Odor***—Transfer a suitable amount of Liquid Paraffin to a small beaker and heat on a water-bath: a foreign odor is not perceptible.

(2) ***Acid or alkali***—Shake vigorously 10 mL of Liquid Paraffin with 10 mL of hot water and 1 drop of phenolphthalein TS: no red color develops. Shake this solution with 0.20 mL/L of 0.02 mol/L sodium hydrox-

ide: a red color develops.

(3) ***Heavy metals***—Proceed as directed in the 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 and moisten with 2 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 0.5 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 solution as directed under Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 1.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: 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 an ethanol solution of magnesium nitrate (1 in 50), add 1.5 mL of strong hydrogen peroxide water and fire to burn. (not more than 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 ice-water for 4 hours: the turbidity produced, if any, is not more intense than that of the following control solution.

Control solution—Take 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***— Take 4.0 g of Paraffin, add 2 mL of dehydrated 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 aqueous layer.

(8) ***Polcyclic aromatic hydrocarbons***—Take 25 mL of Liquid Paraffin by a 25-mL cylinder, transfer to a 100-mL separatory funnel and wash out the cylinder with 25 mL of *n*-hexane and shake vigorously. Shake this solution vigorously for 2 minutes with 5.0 mL of dimethylsufoxide and allow to stand for 15 minutes. Transfer the lower layer to a 10-mL glass-stopered centrifuge tube and centrifuge between 2500 revolutions per minute and 3000 revolutions per minute for about 10 miutes and use the clear solution 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 glass-stoppered centrifuge tube, centrifuge between 2500 and 3000 revolutions per minute for about 10 minutes and use the clear solution as a control solution. Immediately determine the absorbance of the test solution using the control solution as the blank: not more than 0.10 at the wavelength region between 260 nm and 350 nm.

(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 in 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 and the sulfuric acid layer is not more intense than the following control solution.

Control solution—Mix 3.0 mL of ferric chloride colorimetric stock solution with 1.5 mL of cobaltous chloride colorimetric stock solution and 0.50 mL of cupric sulfate colorimetric stock solution.

Containers and Storage ***Containers***—Tight containers.

Light Liquid Paraffin

Light Liquid Paraffin is a mixture of hydrocarbons obtained from petroleum. Tocopherol of a suitable form may be added at a concentration not exceeding 0.001 % as a stabilizer.

Description Light Liquid Paraffin is a clear, colorless oily liquid, nearly free from fluorescence, and is odorless and tasteless.
Light Liquid Paraffin is freely soluble in ether and practically insoluble in water or in ethanol.

Boiling point—Above 300 °C.

Identification Proceed as directed in the Identification under Paraffin.

Specific Gravity d_{20}^{20} : 0.830 ~ 0.870.

Viscosity Less than 37 mm^2/s (Method 1, 37.8 °C).

Purity (1) ***Odor, Acid or alkali, 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

Containers and Storage ***Containers*—**Tight containers.

Peanut Oil

Peanut Oil is the fixed oil obtained from the seeds of *Arachis hypogaea* Linné (Leguminosae).

Description Peanut Oil is a pale yellow, clear oil, odorless or has a slight odor, and a mild taste.
Peanut Oil is miscible with ether or petroleum ether.
Peanut Oil is slightly soluble in ethanol.

Specific gravity— d_{25}^{25} : 0.909 ~ 0.916.

Congealing point of the fatty acids—22 ~ 33 °C.

Identification Saponify 5 g of Peanut Oil by boiling with 2.5 mL of sodium hydroxide solution (3 in 10) and 12.5 mL of ethanol. Evaporate the ethanol, dissolve the residue in 50 mL of hot water and add dilute hydrochloric acid in excess until the free fatty acids separate as an oily layer. Cool the mixture, remove the separated fatty acids and dissolve them in 75 mL of ether. To the ether solution, add a solution of 1 g of lead acetate in 40 mL of ethanol and allow the mixture to stand for 18 hours. Filter the supernatant liquid, transfer the precipitate to the filter with the aid of ether and filter by suction. Place the precipitate in a beaker, heat it with 40 mL of dilute hydrochloric acid and 20 mL of water until the oily layer is entirely clear, cool and decant the water layer. Boil the fatty acids with 50 mL of diluted hydrochloric acid (1 in 100). When the solution prepared by dissolving 0.1 g of the fatty acids in 10 mL of ethanol is not darkened by the addition of 2 drops of sodium sulfide TS, allow the fatty acids to solidify and press them between dry filter papers to exclude moisture. Dissolve the solid fatty acid in 25 mL of diluted ethanol (9 in 10) with the aid of gentle heat and then cool to 15 °C to crystallize the fatty acids. Recrystallize them from diluted ethanol (9 in 10) 20 mL and dry in a dessicator (P_2O_5, in vacuum) for 4 hours: the melting point of the dried crystals is between 73 °C and 76 °C.

Saponification Value 188 ~ 196.

Unsaponifiable Matters Not more than 1.5 %.

Acid Value Not more than 0.2.

Iodine Value 84 ~ 103.

Containers and Storage ***Containers*—**Tight containers.

White Petrolatum

White Petrolatum is a decolorized and purified mixture of hydrocarbons obtained from petroleum.

Description White Petrolatum is a white to pale yellow, homogeneous, unctuous mass, is odorless and tasteless.
White Petrolatum is practically insoluble in water, in ethanol or in dehydrated ethanol.
White Petrolatum dissolves in ether making a clear liquid or producing slight insoluble substances. White Petrolatum becomes a clear liquid when warmed.

Melting Point 38 ~ 60 °C (Method 3).

Purity (1) ***Color***—Melt White Petrolatum by warming and pour 5 mL into a test tube and keep the content in a liquid condition: the liquid is not more intense than the following control solution, when observed transversely from side against a white background.

Control solution—Add 3.4 mL of water to 1.6 mL of ferric chloride colorimetric stock solution.

(2) ***Acid or alkali***—Take 35.0 g of White Petrolatum, add 100 mL of hot water, shake vigorously for 5 minutes and then draw off the aqueous layer. Treat twice the White Petrolatum layer in the same manner using 50 mL volumes of hot water. To the combined aqueous layer, add 1 drop of phenolphthalein TS and boil: no red color is produced. Further add 2 drops of methyl orange TS: no red color is produced.

(3) ***Heavy metals***—Proceed with 1.0 g of White Petrolatum according to Method 2 and perform the test. Prepare the control solution with 3.0 mL of standard lead solution (not more than 30 ppm).

(4) ***Arsenic***—Prepare the test solution with 1.0 g of White Petrolatum, according to Method 3 and perform the test. Add 10 mL of an ethanol solution of magnesium nitrate (1 in 50), then add 1.5 mL of strong hydrogen peroxide water and ale to burn (not more than 2 ppm).

(5) ***Sulfur compound***—Take 4.0 g of White Petrolatum, add 2 mL of dehydrated ethanol and 2 drops of sodium hydroxide solution (1 in 5) saturated with lead monoxide, warm the mixture for 10 minutes at about 70 °C with frequent shaking and allow to cool: no dark color is produced.

(6) ***Organic acids***—Take 100 mL of dilute ethanol, add 1 drop of phenolphthalein TS and titrate with 0.01 mol/L sodium hydroxide VS, until the color of the solution changes to pale red. Mix this solution with 20.0 g of White Petrolatum and boil for 10 minutes under a reflux condenser. Add 2 to 3 drops of phenolphthalein TS to the mixture and 0.40 mL of 0.1 mol/L sodium hydroxide VS with vigorous shaking: the color of the

solution remains red.

(7) ***Fats and fatty oils or rosins***—Take 10.0 g of White Petrolatum, add 50 mL of sodium hydroxide solution (1 in 5) and boil for 30 minutes under a reflux condenser. Cool the mixture, separate the aqueous layer and filter, if necessary. To the aqueous layer, add 200 mL of dilute sulfuric acid: neither oily matter nor precipitate is produced.

(8) ***Polycyclic aromatic hydrocarbons***—Weigh accurately 1.0 g of White Petrolatum, dissolve in 50 mL of hexane, previously shaken twice with 10 mL of dimethyl sulfoxide. Transfer this solution to a stoppered 125 mL separatory funnel. Add 20 mL of dimethyl sulfoxide, shake vigorously for 1 minute and allow to stand until two layers are formed. Transfer the lower layer to a second separatory funnel and repeat the extraction with 20 mL of dimethyl sulfoxide. Add 20 mL of hexane, shake vigorously for 1 minute and allow to stand until two layers are formed. Separate the lower layer, dilute with 50.0 mL of dimethyl sulfoxide and use this solution as the test solution. Determine the absorbance at 260 nm to 420 nm as directed under Ultraviolet-visible Spectrophotometry, using as the control the clear lower liquid obtained by vigorously shaking 25 mL of hexane with 10 mL of dimethyl sulfoxide for 1 minute. Separately, dissolve Naphthalene RS in dimethyl sulfoxide to obtain a solution containing 6.0 mg in 1000 mL, and use this solution as the standard solution. Determine the absorbance at 278 nm as directed under Ultraviolet-visible Spectrophotometry, using dimethylsulfoxide as the control. The absorbance at 260 nm to 420 nm of the test solution is not greater than the absorbance at 278 nm of the standard solution (not more than 300 ppm).

Residue on Ignition Not more than 0.05 % (2 g).

Containers and Storage ***Containers*—**Tight containers.

Yellow Petrolatum

Yellow Petrolatum is a purified mixture of hydrocarbons obtained from petroleum.

Description Yellow Petrolatum is a yellow, homogeneous, unctuous mass, odorless and tasteless.
Yellow Petrolatum is slightly soluble in ethanol and practically insoluble in water.
Yellow Petrolatum dissolves in ether, in petroleum benzine or in turpentine oil, making a clear liquid or producing slight insoluble substances.
Yellow Petrolatum becomes a yellow, homogeneous unctuous mass with no odor or taste when warmed.

Melting Point 38 ~ 60 °C (Method 3).

Purity (1) ***Color***—Proceed as directed in the Purity (1) under White Petroleum. Use following control solution.

Control solution—Take 3.8 mL of ferric chloride colorimetric stock solution and add 1.2 mL of cobaltous chloride colorimetric stock solution.

(2) ***Acid or alkali, Heavy metals, Arsenic, Sulfur compound, Organic acids, Fats and fatty oils or resins***—Proceed as directed in the Purity (2), (3), (4), (5), (6) and (7) under White Petroleum.

(3) ***Polycyclic aromatic hydrocarbons***—Weigh accurately 1.0 g of Yellow Petrolatum, dissolve in 50 mL of hexane, previously shaken twice with 10 mL of dimethyl sulfoxide. Transfer this solution to a stoppered 125 mL separatory funnel. Add 20 mL of dimethyl sulfoxide, shake vigorously for 1 minute and allow to stand until two layers are formed. Transfer the lower layer to a second separatory funnel and repeat the extraction with 20 mL of dimethyl sulfoxide. Add 20 mL of hexane, shake vigorously for 1 minute and allow to stand until two layers are formed. Separate the lower layer, dilute with 50.0 mL of dimethyl sulfoxide and use this solution as the test solution. Determine the absorbance at 260 nm to 420 nm as directed under Ultraviolet-visible Spectrophotometry, using as the control the clear lower liquid obtained by vigorously shaking 25 mL of hexane with 10 mL of dimethyl sulfoxide for 1 minute. Separately, dissolve Naphthalene RS in dimethyl sulfoxide to obtain a solution containing 9.0 mg in 1000 mL, and use this solution as the standard solution. Determine the absorbance at 278 nm as directed under Ultraviolet-visible Spectrophotometry, using dimethylsulfoxide as the control. The absorbance at 260 nm to 420 nm of the test solution is not greater than the absorbance at 278 nm of the standard solution (not more than 300 ppm).

Residue on Ignition Not more than 0.05 % (2 g).

Containers and Storage ***Containers*—**Tight containers.

Petroleum Benzin

Petroleum Benzin is a mixture of low boiling point hydrocarbons from petroleum.

Description Petroleum Benzin is a colorless, clear, volatile liquid. Petroleum Benzin shows no fluorescence, and has a characteristic odor.

Petroleum Benzin is miscible with dehydrated ethanol or ether.

Petroleum Benzin is practically insoluble in water. Petroleum Benzin is very flammable.

Specific gravity—d_{20}^{20} : 0.65 ~ 0.71.

Purity (1) ***Acid***—Shake vigorously 10 mL of Petroleum Benzin with 5 mL of water for 2 minutes and allow to stand: the separated aqueous layer does not change moistened blue litmus paper to red.

(2) ***Sulfur compounds and reducing substances***—Take 10 mL of Petroleum Benzin, add 2.5 mL of ammonia-ethanol TS 2 to 3 drops of silver nitrate TS and warm the mixture at about 50 °C for 5 minutes and protected from light: no brown color develops.

(3) ***Fatty oil and sulfur compounds***—Drop and evaporate 10 mL of Petroleum Benzin in small volumes on odorless filter paper spread on a previously warmed glass plate: 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 heat the residue at 105 °C to constant weight: not more than 1 mg.

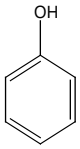
(6) ***Readily carbonizable substances***—Shake vigorously 5 mL of Petroleum Benzin with 5 mL of sulfuric acid for readily carbonizable substances for 5 minutes in a Nessler tube and allow to stand: the sulfuric acid layer is not more intense than Color Matching Fluid A.

Distilling Range 50 ~ 80 °C, not less than 90 %.

Containers and Storage ***Containers—***Tight containers.

Storage—Remote from fire, and not exceeding 30°C.

Phenol



Carbolic Acid C_6H_6O: 94.11

Phenol [*108-95-2*]

Phenol contains not less than 98.0 % and not more than 101.0 % of phenol (C_6H_6O).

Description Phenol appears as colorless to slightly red crystals or crystalline mass, and has a characteristic odor.
Phenol is very soluble in ethanol or in ether and soluble in water.
Phenol (10 g) is liquefied by addition of 1 mL of water.
The color changes gradually through red to dark red by light or air.
Phenol cauterizes the skin, turning it white.

Congealing point—About 40 °C.

Identification (1) Add 1 drop of ferric chloride TS to 10 mL of a solution of Phenol (1 in 100): a blue-purple color develops.

(2) Add bromine TS dropwise to 5 mL of a solution of Phenol (1 in 10,000): a white precipitate is produced, which at first dissolves with shaking, but becomes permanent as excess of the reagent is added.

Purity (1) ***Clarity and color of solution and acidity or alkalinity***—Dissolve 1.0 g of Phenol in 15 mL of water: the solution is clear and neutral or only faintly acid. Add 2 drops of methyl orange TS: no red color develops.

(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: not more than 0.05 %.

Assay Weigh accurately about 1.5 g of Phenol and dissolve in water to make exactly 1000 mL. Transfer exactly 25 mL of this solution to an iodine flask, add exactly 30 mL of 0.05 mol/L bromine VS, then 5 mL of hydrochloric acid and stopper the flask immediately. Shake the flask repeatedly for 30 minutes, allow to stand for 15 minutes, then add 7 mL of potassium iodide TS, stopper the flask immediately and shake well. Add 1 mL of chloroform, stopper the flask and shake thoroughly. Titrate the liberated iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 1 mL of starch TS). Perform a blank determination and make any necessary correction.

Each mL of 0.05 mol/L bromine VS
= 1.5686 mg of C_6H_6O

Containers and Storage ***Containers—***Tight containers.

Storage—Light-resistant.

Phenol for Disinfection

Carbolic Acid for Disinfection

Phenol for Disinfection contains not less than 95.0 % and not more than 101.0 % of phenol (C_6H_6O: 94.11).

Description Phenol for Disinfection appears as colorless to slightly red crystals, crystalline mass, or liquid containing the crystal and has a characteristic odor.
Phenol for Disinfection is very soluble in ethanol or in ether and freely soluble in water.
10 g of Phenol for Disinfection is liquefied by addition of 1 mL of water.
Phenol for Disinfection cauterizes the skin, turning it white.

Congealing point—About 30 °C.

Identification (1) Take 10 mL of a solution of Phenol for Disinfection (1 in 100) and add 1 drop of ferric

chloride TS: a blue-purple color is produced.

(2) Take 5 mL of a solution of Phenol for Disinfection (1 in 10000) and add bromine TS drop-wise: a white precipitate is formed and it dissolves at first upon shaking but becomes permanent as excess of the reagent is added.

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 5.0 g of Phenol for Disinfection, evaporate on a water-bath and dry the residue at 105 °C for 1 hour: not more than 0.10 %.

Assay Weigh accurately about 1 g of Phenol for Disinfection, and dissolve in water to make exactly 1000 mL. Pipet exactly 25 mL of the solution into an iodine flask, add exactly 30 mL of 0.05 mol/L bromine VS and 5 mL of hydrochloric acid, stopper immediately, shake for 30 minutes and allow to stand for 15 minutes. Add 7 mL of potassium iodide TS, stopper immediately, shake well and titrate the liberated iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 1 mL of starch TS). Perform a blank determination and make any necessary correction.

Each mL of 0.05 mol/L bromine VS
= 1.5685 mg of C_6H_6O

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light-resistant.

Liquefied Phenol

Liquefied Carbolic Acid

Liquefied Phenol is Phenol maintained in a liquid condition by the presence of 10 % of Water or Purified Water. Liquefied Phenol contains not less than 88.0 % of phenol (C_6H_6O: 94.11).

Description Liquefied Phenol is a colorless or slightly reddish liquid, and has a characteristic odor.
Liquefied Phenol is miscible with ethanol, with ether or with glycerin.
A mixture of equal volumes of Liquefied Phenol and glycerin is miscible with water.
The color changes gradually to dark red on exposure to light or air.
Liquefied Phenol cauterizes the skin, turning it white.

Specific gravity— d_{20}^{20} : About 1.065.

Identification Proceed as directed in the Identification under Phenol.

Boiling Point Not more than 182 °C.

Purity Proceed as directed in the Purity under Phenol.

Assay Dissolve about 1.7 g of Liquefied Phenol, accurately weighed, proceed as directed in the Assay under Phenol.

Each mL of 0.05 mol/L bromine VS
= 1.5685 mg of C_6H_6O

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light-resistant.

Polyethylene Glycol 1500

Macrogol 1500

Polyethylene Glycol 1500 is a mixture containing equal amounts of lower and higher polymers of ethylene oxide and water, represented by the formula $[HOCH_2(CH_2OCH_2)_nCH_2OH]$, in which the value of *n* is 5 or 6 for the lower polymer and from 28 to 36 for the higher.

Description Polyethylene Glycol 1500 is a white, smooth petrolatum-like solid, is odorless or has a faint, characteristic odor.
Polyethylene Glycol 1500 is very soluble in water, in pyridine or in diphenyl ether, freely soluble in methanol, sparingly soluble in ethanol, very slightly soluble in dehydrated ethanol and practically insoluble in ether.

Congealing point—37 ~ 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 and filter, if necessary. To the filtrate, add 1 mL of a solution of phosphomolybdic acid (1 in 10): a yellow-green precipitate is formed.

pH Dissolve 1.0 g of Polyethylene Glycol 1500 in 20 mL of water: the pH of the solution is between 4.0 and 7.0.

Purity (1) ***Clarity and color of solution***—Dissolve 5.0 g of Polyethylene Glycol 1500 in 50 mL of water: the 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 VS: the solution is red.

(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 standard lead solution (not more than 20 ppm).

(4) ***Ehtylene glycol and diethylene glycol***—Place 50.0 g of Polyethylene Glycol 1500 in distilling flask, add 75 mL of diphenyl ether, warm to dissolve, if nec-

essary, distil slowly in vaccum of 0.13 to 0.27 kPa and take 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, cool in ice-water, congeal the diphenyl ether and filtrate into a 25-mL volumetric flask. Wash the residue with 5 mL of ice-cold water, combine the washing 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. Take 10.0 mL each of the test solution and the standard solution and add to each 15.0 mL of cerium (IV) diammonium nitrate TS. Perform the test with these solutions as directed under the Ultraviolet-visible Spectrophotometry within 2 to 5 minutes: the absorbance of the solution from the test solution at the wavelength of maximum absorption at about 450 nm is not larger than the absorbance of the solution 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 dioxane standard 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 ethylene oxide standard solution. Prepare 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 ethylene oxide standard solution and 0.5 mL of the dioxane standard 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 ethylene oxide standard solution into a 10 mL vial, add 0.1 mL of a freshly prepared 10 mg/L acetaldehyde standard solution and 0.1 mL of the dioxane standard 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 of the test solution and standard solution (1) as directed under Gas Chromatography according to the following operating conditions. Determine the peak areas of ethylene oxide and dioxane in each solution and calculate their amounts: not more than 1 ppm of ethylene oxide and not more than 10 ppm of dioxane.

$$\text{Amount (ppm) of ethylene oxide} = \frac{A_T \times C}{(A_R \times M_T) - (A_T \times M_R)}$$

A_T = Peak area of ethylene oxide in the test solution

A_R = Peak area of ethylene oxide in standard solution (1)

M_T = Amount (g) of Polyethylene Glycol 1500 in the test solution

M_R = Amount (g) of Polyethylene Glycol 1500 in standard solution (1)

C = Amount (μg) of ethylene oxide added to 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 standard solution (1)

C = Amount (μg) of dioxane added to standard solution (1)

Operating conditions

Detector: A hydrogen flame ionization detector

Column: A glass or quartz capillary tube 0.32 mm in internal diameter and about 30 m in length, with internal coating 1.0 μm in thickness made of poly(dimethyl) siloxane.

Column temperature: 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 at 230 °C for 5 minutes.

Injection port temperature: A constant temperature of about 150 °C

Headspace sample temperature: 70 °C

Detector temperature: A constant temperature of about 250 °C

Carrier gas: Helium

Split ratio: About 1 : 20

System suitability: When the procedure is run with 1.0 mL of standard solution (2) under the above operating conditions, the resolution between the peaks of acetaldehyde and ethylene oxide is not less than 2.0 and the signal-to-noise ratio of dioxane is not less than 5.

Water Not more than 1.0 % (2 g, volumetric titration, direct titration).

Residue on Ignition Not more than 0.10 % (1 g).

Containers and Storage ***Containers*—**Tight containers.

Polyethylene Glycol 20000

Macrogol 20000

Polyethylene Glycol 20000 is a polymer of ethylene

oxide and water, represented by the formula [$HOCH_2(CH_2OCH_2)_n CH_2OH$], in which the value of *n* ranges from 340 to 570.

Description Polyethylene Glycol 20000 is a white, paraffin-like flake or powder, is ordorless or has a faint, characteristic odor.
Polyethylene Glycol 20000 is freely soluble in water and in pyridine and practically insoluble in methanol, in ethanol, in anhydrous ether, in petroleum benzine and in Polyethylene Glycol 400.

Congealing point—56 ~ 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 and filter, if necessary. To the filtrate, add 1 mL of a solution of phosphomolybdic acid (1 in 10): a yellow-green precipitate is produced.

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 solution is clear an colorless.

(2) ***Acid***—Dissolve 5.0 g of Polyethylene Glycol 20000 in 20 mL of neutralized ethanol by warming, cool and add 0.20 mL of 0.1 mol/L sodium hydroxide VS and 1 drop of phenolphthalein TS: the color of the solution is red.

(3) ***Heavy metals***—Proceed with 1.0 g of Polyethylene Glycol 2000 according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 20 ppm).

(4) ***Ethylene oxide and dioxane***—Weigh accurately 1.00 g (M_T) of Polyethylene Glycol 2000, 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 dioxane standard 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 ethylene oxide standard solution. Prepare before use. Separately, put 1.00 g (M_R) of Polyethylene Glycol 2000 into an identical 10 mL vial, add 0.5 mL of the ethylene oxide standard solution and 0.5 mL of the dioxane standard 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 ethylene oxide standard solution into a 10 mL vial, add 0.1 mL of a freshly prepared 10 mg/L acetaldehyde standard solution and 0.1 mL of the dioxane standard 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 of the test solution and standard solution (1) as directed under Gas Chromatography according to the following operating conditions. Determine the peak areas of ethylene oxide and dioxane in each solution and calculate their amounts: not more than 1 ppm of ethylene oxide and not more than 10 ppm of dioxane.

$$\text{Amount (ppm) of ethylene oxide} = \frac{A_T \times C}{(A_R \times M_T) - (A_T \times M_R)}$$

A_T = Peak area of ethylene oxide in the test solution
A_R = Peak area of ethylene oxide in standard solution (1)
M_T = Amount (g) of Polyethylene Glycol 2000 in the test solution
M_R = Amount (g) of Polyethylene Glycol 2000 in standard solution (1)
C = Amount (μg) of ethylene oxide added to 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 standard solution (1)
C = Amount (μg) of dioxane added to standard solution (1)

Operating conditions
Detector: A hydrogen flame ionization detector
Column: A glass or quartz capillary tube 0.32 mm in internal diameter and about 30 m in length, with internal coating 1.0 μm in thickness made of poly(dimethyl) siloxane.
Column temperature: 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 at 230 °C for 5 minutes.
Injection port temperature: A constant temperature of about 150 °C
Headspace sample temperature: 70 °C
Detector temperature: A constant temperature of about 250 °C
Carrier gas: Helium
Split ratio: About 1 : 20
System suitability: When the procedure is run with 1.0 mL of standard solution (2) under the above operating conditions, the resolution between the peaks of acetaldehyde and ethylene oxide is not less than 2.0 and the signal-to-noise ratio of dioxane is not less than 5.

Average Molecular Mass Weigh accurately about 15.0 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, pipet exactly 300 mL of freshly distilled pyridine into a 1000 mL light-resistant stoppered bottle, add 42 g of phthalic anhydride, dissolve with vigorous shaking and allow to stand for 16 hours or more. Pipet exactly 25 mL of this solution, transfer to the former stoppered pressure bottle, stopper the bottle tightly, wrap it securely with strong cloth and immerse in a water-bath, having a temperature of 98 ± 2 °C, to the same depth as the mixture in the bottle. Maintain the temperature of the bath at 98 ± 2 °C for 60 minutes. Remove the bottle from the bath and allow to cool in 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). Titrate with 0.5 mol/L sodium hydroxide VS until a pale red color remains for not less than 15 seconds. Perform a blank determination and make any necessary correction: average molecular mass is between 15000 and 25000.

$$\text{Average molecular mass} = \frac{\text{Amount (g) of sample} \times 4000}{a - b}$$

a : Volume(mL) of 0.5 mol/L sodium hydroxide VS used in the blank determination,

b : Volume(mL) of 0.5 mol/L sodium hydroxide VS used in the test.

Water Not more than 1.0 % (2 g, volumetric titration, direct titration).

Residue on Ignition Not more than 0.2 % (1 g).

Containers and Storage ***Containers*—**Well-closed containers.

Polyethylene Glycol 400

Macrogol 400

Polyethylene Glycol 400 is a polymer of ethylene oxide and water, represented by the formula $[HOCH_2(CH_2OCH_2)_n\ CH_2OH]$, in which the value of n ranges from 7 to 9.

Description Polyethylene Glycol 400 is a clear, colorless and viscous liquid, has no odor or a slight, characteristic odor.

Polyethylene Glycol 400 is miscible with water, with methanol, with ethanol or with pyridine.

Polyethylene Glycol 400 is soluble in ether.

Polyethylene Glycol 400 is slightly hygroscopic.

Congealing point—4 ~ 8 °C.

Specific gravity—d_{20}^{20} : 1.110 ~ 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 and filter, if necessary. To the filtrate, add 1 mL of a solution of phosphomolybdic acid (1 in 10): a yellow-green precipitate is formed.

pH Dissolve 1.0 g of Polyethylene Glycol 400 in 20 mL of water: the pH of this solution is between 4.0 and 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 VS: the solution is red.

(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 standard lead solution (not more than 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. Weigh accurately about 50 mg each of 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 the standard solution as directed under the Gas Chromatography according to the following operating conditions. Determine the peak heights, H_{Ta} and H_{As}, of ethylene glycol, for the test solution and the standard solution, respectively and the peak heights, H_{Tb} and H_{Sb}, of diethylene glycol, for the test solution and the standard solution, respectively and calculate the amount of ethylene glycol and diethylene glycol: the sum of the contents of ethylene glycol and diehtylene glycol is not more than 0.25 %.

$$\text{Amount (mg) of Ethylene Glycol} = \text{amount (mg) of ethylene glycol RS} \times \frac{H_{Ta}}{H_{Sa}} \times \frac{1}{10}$$

$$\text{Amount (mg) of Ethylene Glycol} = \text{amount (mg) of diethylene glycol RS} \times \frac{H_{Tb}}{H_{Sb}} \times \frac{1}{10}$$

Operating conditions

Detector: A hydrogen flame-ionization detector.

Column: A column, about 3 mm in internal diameter and about 1.5 m in length, packed with siliceous earth for gas chromatography, 150 μm to 180 μm in particle diameter, coated with D-sorbitol at the 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: When the procedure is run with 2 μL of the standard solution under the above operating conditions, ethylene glycol and diethylene glycol are eluted in this order.

Detection sensitivity: Adjust the detection sensitivity so that the peak height of diethylene glycol obtained from 2 μL of the standard solution composes 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 dioxane standard 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 ethylene oxide standard solution. Prepare 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 ethylene oxide standard solution and 0.5 mL of the dioxane standard 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 ethylene oxide standard solution into a 10 mL vial, add 0.1 mL of a freshly prepared 10 mg/L acetaldehyde standard solution and 0.1 mL of the dioxane standard 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 of the test solution and standard solution (1) as directed under Gas Chromatography according to the following operating conditions. Determine the peak areas of ethylene oxide and dioxane in each solution and calculate their amounts: not more than 1 ppm of ethylene oxide and not more than 10 ppm of dioxane.

$$\text{Amount (ppm) of ethylene oxide} = \frac{A_T \times C}{(A_R \times M_T) - (A_T \times M_R)}$$

A_T = Peak area of ethylene oxide in the test solution

A_R = Peak area of ethylene oxide in standard solution (1)

M_T = Amount (g) of Polyethylene Glycol 400 in the test solution

M_R = Amount (g) of Polyethylene Glycol 400 in standard solution (1)

C = Amount (μg) of ethylene oxide added to 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 standard solution (1)

C = Amount (μg) of dioxane added to standard solution (1)

Operating conditions

Detector: A hydrogen flame ionization detector

Column: A glass or quartz capillary tube 0.32 mm in internal diameter and about 30 m in length, with internal coating 1.0 μm in thickness made of poly(dimethyl) siloxane.

Column temperature: 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 at 230 °C for 5 minutes.

Injection port temperature: A constant temperature of about 150 °C

Headspace sample temperature: 70 °C

Detector temperature: A constant temperature of about 250 °C

Carrier gas: Helium

Split ratio: About 1 : 20

System suitability: When the procedure is run with 1.0 mL of standard solution (2) under the above operating conditions, the resolution between the peaks of acetaldehyde and ethylene oxide is not less than 2.0 and the signal-to-noise ratio of dioxane is not less than 5.

Average Molecular Mass Add 42 g of phthalic anhydride to 300 mL of freshly distilled pyridine, exactly measured, in a 1000 mL light-resistant stoppered bottle. Shake the bottle vigorously to dissolve the solid and allow to stand for 16 hours or more. Pipet exactly 25 mL of this solution into an about 200 mL stoppered pressure bottle, add about 1.5 g of Polyethylene Glycol 400, accurately weighed, stopper the bottles wrap it securely with strong cloth and immerse in a water-bath, having a temperature of 98 ± 2 °C, to the level so that the mixture in the bottle soaks completely in water. Maintain the temperature of the bath at 98 ± 2 °C for 30 minutes. Remove the bottle from the bath and allow to cool in air to room temperature. Add 50.0 mL of 0.5 mol/L sodium hydroxide VS and 5 drops of a solution of phenolphtalein in pyridine (1 in 100). Titrate with 0.5 mol/L sodium hydroxide VS until a pale red color remains for not less than 15 seconds. Perform a blank determination and make any necessary correction: Average molecular mass is between 380 and 420.

$$\text{Average molecular mass} = \frac{\text{Amount (g) of sample} \times 400}{a - b}$$

a : Volume (mL) of 0.5 mol/L sodium hydroxide

VS used in the blank determination,

b: Volume (mL) of 0.5 mol//L sodium hydroxide VS used in the test.

Water Not more than 1.0 % (2 g, volumetric titration, direct titration).

Residue on Ignition Not more than 0.10 % (1 g).

Containers and Storage ***Containers*—**Tight containers.

Polyethylene Glycol 4000

Macrogol 4000

Polyethylene Glycol 4000 is a polymer of ethylene oxide and water, represented by the formula $[HOCH_2(CH_2OCH_2)_n\ CH_2OH]$, in which the value of n ranges from 59 to 84.

Description Polyethylene Glycol 4000 is a white, paraffin-like solid, flakes or power, is colorless or has a faint, characteristic odor.
Polyethylene Glycol 4000 is very soluble in water, freely soluble in methanol or in pyridine and practically insoluble in dehydrate ethanol or in ether.

Congealing point—53 ~ 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 and filter, if necessary. To the filtrate, add 1 mL of a solution of phosphomolybdic acid (1 in 10): a yellow-green precipitate is produced.

pH Dissolve 1.0 g of Polyethylene Glycol 4000 in 20 mL of water: the pH of this solution is between 4.0 and 7.5.

Purity (1) ***Clarity and color of solution***—A solution of 5.0 g of Polyethylene Glycol 4000 in 50 mL of water is clear and colorless.

(2) ***Acid***—Dissolve 5.0 g of Polyethylene Glycol 4000 in 20 mL of neutralized ethanol by warrning, cool and add 0.20 mL of 0.1 mol/L sodium hydroxide VS and 1 drop of phenolphthalein TS: the color of the solution is red.

(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 standard lead solution (not more than 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 dioxane standard 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 ethylene oxide standard solution. Prepare 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 ethylene oxide standard solution and 0.5 mL of the dioxane standard 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 ethylene oxide standard solution into a 10 mL vial, add 0.1 mL of a freshly prepared 10 mg/L acetaldehyde standard solution and 0.1 mL of the dioxane standard 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 of the test solution and standard solution (1) as directed under Gas Chromatography according to the following operating conditions. Determine the peak areas of ethylene oxide and dioxane in each solution and calculate their amounts: not more than 1 ppm of ethylene oxide and not more than 10 ppm of dioxane.

$$\text{Amount (ppm) of ethylene oxide} = \frac{A_T \times C}{(A_R \times M_T) - (A_T \times M_R)}$$

A_T = Peak area of ethylene oxide in the test solution

A_R = Peak area of ethylene oxide in standard solution (1)

M_T = Amount (g) of Polyethylene Glycol 4000 in the test solution

M_R = Amount (g) of Polyethylene Glycol 4000 in standard solution (1)

C = Amount (μg) of ethylene oxide added to 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 standard solution (1)

C = Amount (μg) of dioxane added to standard solution (1)

Operating conditions

Detector: A hydrogen flame ionization detector

Column: A glass or quartz capillary tube 0.32 mm in internal diameter and about 30 m in length, with internal coating 1.0 μm in thickness made of poly(dimethyl) siloxane.

Column temperature: 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 at 230 °C for 5 minutes.

Injection port temperature: A constant temperature of about 150 °C

Headspace sample temperature: 70 °C

Detector temperature: A constant temperature of about 250 °C

Carrier gas: Helium

Split ratio: About 1 : 20

System suitability: When the procedure is run with 1.0 mL of standard solution (2) under the above operating conditions, the resolution between the peaks of acetaldehyde and ethylene oxide is not less than 2.0 and the signal-to-noise ratio of dioxane is not less than 5.

Average Molecular Mass 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, pipet exactly 300 mL of freshly distilled pyridine into a 1000-mL light-resistant, stoppered bottle, add 42 g of phthalic anhydride, dissolve with vigorous shaking and allow to stand for 16 hours or more. Pipet exactly 25 mL of this solution, transfer to the former stoppered pressure bottle, stopper the bottle tightly, wrap it securely with strong cloth and perform the test, Average molecular mass under Polyethyleneglycol 400: average molecular mass is between 2600 and 3800.

Water Not more than 1.0 % (2 g, volumetric titration, direct titration).

Residue on Ignition Not more than 0.25 % (1 g).

Containers and Storage ***Containers*—**Well-closed containers.

Polyethylene Glycol 6000

Macrogol 6000

Polyethylene Glycol 6000 is a polymer of ethylene oxide and water, represented by the formula $[HOCH_2(CH_2OCH_2)_n\ CH_2OH]$, in which the value of *n* ranges from 165 to 210.

Description Polyethylene Glycol 6000 is a white, paraffin-like solid, flake or powder, is odorless or has a faint, characteristic odor.

Polyethylene Glycol 6000 is very soluble in water, freely soluble in pyridine and practically insoluble in ethanol, in dehydrated ethanol or in ether.

Congealing point—56 ~ 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 and filter, if necessary, To the filtrate add 1 mL of a solution of phosphomolybdic acid (1 in 10): a yellow-green precipitate is produced.

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 solution is clear and colorless.

(2) ***Acid***—Dissolve 5.0 g of Polyethylene Glycol 6000 in 20 mL of neutralized ethanol by warming, cool and add 0.20 mL of 0.1 mol/L sodium hydroxide VS 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 standard lead solution (not more than 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 dioxane standard 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 ethylene oxide standard solution. Prepare 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 ethylene oxide standard solution and 0.5 mL of the dioxane standard 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 ethylene oxide standard solution into a 10 mL vial, add 0.1 mL of a freshly prepared 10 mg/L acetaldehyde standard solution and 0.1 mL of the dioxane standard 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 of the test solution and standard solution (1) as directed under Gas Chromatography according to the following operating conditions. Determine the peak areas of ethylene oxide and dioxane in each solution and calculate their amounts: not more than 1 ppm of ethylene oxide and not more than 10 ppm of dioxane.

$$\text{Amount (ppm) of ethylene oxide} = \frac{A_T \times C}{(A_R \times M_T) - (A_T \times M_R)}$$

A_T = Peak area of ethylene oxide in the test solution

A_R = Peak area of ethylene oxide in standard solution (1)

M_T = Amount (g) of Polyethylene Glycol 6000 in the test solution

M_R = Amount (g) of Polyethylene Glycol 6000 in standard solution (1)

C = Amount (μg) of ethylene oxide added to 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 standard solution (1)

C = Amount (μg) of dioxane added to standard solution (1)

Operating conditions

Detector: A hydrogen flame ionization detector

Column: A glass or quartz capillary tube 0.32 mm in internal diameter and about 30 m in length, with internal coating 1.0 μm in thickness made of poly(dimethyl) siloxane.

Column temperature: 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 at 230 °C for 5 minutes.

Injection port temperature: A constant temperature of about 150 °C

Headspace sample temperature: 70 °C

Detector temperature: A constant temperature of about 250 °C

Carrier gas: Helium

Split ratio: About 1 : 20

System suitability: When the procedure is run with 1.0 mL of standard solution (2) under the above operating conditions, the resolution between the peaks of acetaldehyde and ethylene oxide is not less than 2.0 and the signal-to-noise ratio of dioxane is not less than 5.

Average Molecular Mass 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, pipet exactly 300 mL of freshly distilled pyridine into a 1000-mL light-resistant, stoppered bottle, add 42 g of phthalic anhydride, dissolve with vigorous shaking and allow to stand for 16 hours or more. Pipet exactly 25 mL of this solution, transfer to the former stoppered pressure bottle, stopper the bottle tightly, wrap it securely with strong cloth and perform the test, Average molecular mass under Polyethyleneglycol 400: average molecular mass is between 7300 and 9300.

Water Not more than 1.0 % (2 g, volumetric titration, direct titration).

Residue on Ignition Not more than 0.2 % (1 g).

Containers and Storage ***Containers*—**Well-closed containers.

Polyoxyl 40 Stearate

Polyoxyl 40 Stearate is the monostearate of condensation polymers of ethylene oxide represented by the formula $[H(OCH_2CH_2)_nOOCC_{17}H_{35}]$, in which n is approximately 40.

Description Polyoxyl 40 Stearate is a white to pale yellow, waxy solid or powder, is odorless or has a faint fat-like odor.

Polyoxyl 40 Stearate is soluble in water, in ethanol or in ether.

Saponification Value 25 ~ 35.

Acid Value Not more than 1.

Congealing Point 39.0 ~ 44.0 °C.

Congealing Point of the Fatty Acid Not less than 53°C.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Polyoxyl 40 Stearate in 20 mL of water: the solution is clear and colorless.

(2) ***Heavy metals***—Proceed with 2.0 g of Polyoxyl 40 Stearate according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 10 ppm).

(3) ***Arsenic***—Prepare the test solution with 0.67 g of Polyoxyl 40 Stearate, according to Method 3 and perform the test (not more than 3 ppm).

Residue on Ignition Not more than 0.1 % (1 g).

Containers and Storage ***Containers*—**Tight containers.

Polysorbate 80

Polysorbate 80 is a polyoxyethylene ether of anhydrous sorbitol, partially esterified with oleic acid.

Description Polysorbate 80 is a colorless or orange viscous liquid, having a faint, characteristic odor and a warm, slightly bitter taste.

Polysorbate 80 is freely soluble in water and slightly soluble in ether.

Polysorbate 80 is miscible with methanol, with ethanol, with warm ethanol, with pyridine and with chloroform.

pH—The pH of a solution of Polysorbate 80 (1 in 20) is between 5.5 and 7.5.

Identification (1) Take 5 mL of a solution of Polysorbate 80 (1 in 20), add 5 mL of sodium hydroxide TS, boil for 5 minutes, cool and acidify with dilute hydrochloric acid: the solution is opalescent.

(2) Take 5 mL of a solution of Polysorbate 80 (1 in 20) and add 2 to 3 drops of bromine TS: the color of the test solution is discharged.

(3) Mix 6 mL of Polysorbate 80 with 4 mL of water at an ordinary temperature or the lower tempreature: a jelly-like mass is produced.

(4) Take 10 mL of a solution of Polysorbate 80 (1 in 20), add 5 mL of ammonium thiocyanate-cobaltous nitrate TS, shake well, add 5 mL of chloroform, shake and allow to stand: a blue color develops in the chloroform layer.

Saponification Value 45 ~ 55.

Specific Gravity d_{20}^{20}: 1.065 ~ 1.095.

Acid Value Not more than 2.0.

Oleic Acid Weigh accurately about 25 g of Polysorbate 80, transfer to a stoppered 500 mL flask, add 250 mL of ethanol and 7.5 g of potassium hydroxide and mix. Immediately attach to a condenser and reflux on a water bath for 1 to 2 hours. Transfer to a 800 mL beaker, wash the flask with 100 mL of water and add the washing to the beaker. Remove the alcohol completely by evaporation, occasionally adding water to supplement the amount of alcohol evaporated in the water bath. Neutralize with sulfuric acid (1 in 2) and add a further 10 % of the amount consumed. Heat this solution while stirring until the fatty acid layer is separated. Transfer the fatty acid layer to a 500 mL separatory funnel, wash with three to four 20 mL volumes of hot water and add the washings to the original water-soluble layer at saponification. Extract the combined solution with three 50 mL volumes of petroleum ether, add to the fatty acid layer, evaporate to dryness in a previously weighed container and weigh the residue: not less than 22 % and not more than 24 %. For the oleic acid thus obtained, the acid value is between 196 and 206 when the test is performed as directed in the Acid Value under Fats and Fatty Oils, and the iodine value is between 80 and 92.

Hydroxyl Value 65 ~ 80

Iodine Value 19 ~ 24. Use chloroform instead of carbon tetrachloride and titrate without using an indicator, until the yellow color of iodine disappears.

Viscosity 345 ~ 445 mm^2/s (Method 1, 25 °C).

Purity (1) ***Heavy metals***—Proceed with 2.0 g of Polysorbate 80 according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 10 ppm).

(2) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat and place 10 mg to 300 mg of Polysorbate 80 on top. Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. Place the boat inside the furnace and heat to about 900 °C with a current of air or oxygen at 0.5 L/minute to 1 L/minute. Elute the mercury and sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 ng to 200 ng.

Additive—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.

(3) ***Cadmium***—Weigh accurately 5.0 g of Polysorbate 80 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 5 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 1.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Cadmium hollow cathode lamp
Wavelength: 228.8 nm

(4) ***Lead***—Weigh accurately 5.0 g of Polysorbate 80 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 solution as directed under Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 2.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(5) ***Arsenic***—Prepare the test solution with 1.0 g of Polysorbate 80 according to Method 3 and perform the test (not more than 2 ppm).

(6) ***Ethylene oxide and dioxane***—Put 1.0 g of Polysorbate 80 into a 10 mL headspace vial, dissolve in 2.0 mL of water, seal immediately with a Teflon-coated silicon membrane and an aluminum cap, shake lightly and use this solution as test solution (1). Separately, put 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 test solution (1) and use this solution as 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 ethylene oxide standard solution. Pipet 1.0 mL of Dioxane RS, dilute 20000-fold with water and use this solution as the dioxane standard solution. Mix 6.0 mL of the ethylene oxide standard solution and 2.5 mL of the dioxane standard 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 acetaldehyde standard solution. Put 2.0 mL of the acetaldehyde standard solution and 2.0 mL of the ethylene oxide standard solution into a 10 mL headspace vial, seal immediately with a Teflon-coated silicon membrane and an aluminum cap, shake lightly and use this solution as the control solution. Perform the test with 1 mL of test solution (1) and test solution (2) as directed under Gas Chromatography according to the following operating conditions. Determine the peak area of ethylene oxide and dioxane in each solution and calculate their amounts: not more than 1 ppm of ethylene oxide and not more than 10 ppm of dioxane.

$$\text{Amount (ppm) of ethylene oxide} = \frac{2C_{EO} \times A_a}{A_b - A_a}$$

C_{EO} = Concentration (μg/mL) of ethylene oxide in test solution (1)
A_a = Peak area of ethylene oxide in test solution (1)
A_b = Peak area of ethylene oxide in test solution (2)

$$\text{Amount (ppm) of dioxane} = \frac{2 \times 1.03 \times C_D \times A_{a'}}{A_{b'} - A_{a'}}$$

C_D = Concentration (μg/mL) of dioxane in test solution (1)
1.03 = Density (g/mL) of dioxane
$A_{a'}$ = Peak area of dioxane in test solution (1)
$A_{b'}$ = Peak area of dioxane in test solution (2)

Operating conditions

Detector: A hydrogen flame ionization detector

Column: A fused silica tube 0.53 mm in internal diameter and about 50 m in length, with internal coating 5 μm in thickness made of poly(dimethyl)(diphenyl) siloxane.

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.

Injection port temperature: A constant temperature of about 85 °C

Headspace sample temperature: 80 °C

Detector temperature: A constant temperature of about 250 °C

Carrier gas: Helium

Flow rate: 4.0 mL/minute

Split ratio: About 1 : 3.5

Retention time: The relative retention times of acetaldehyde and dioxane with respect to the retention time

of ethylene oxide (about 6.5 minutes) are 0.9 and 1.9, respectively.

System suitability

System performance: When the procedure is run with 1.0 mL of the control solution under the above operating conditions, the resolution between the peaks of acetaldehyde and ethylene oxide is not less than 2.0.

Water Not more than 3.0 % (1 g, volumetric titration, back titration).

Residue on Ignition Not more than 0.15 % (2 g).

Containers and Storage ***Containers*—**Tight containers.

Potassium Carbonate

K_2CO_3: 138.21

Potassium Carbonate, when dried, contains not less than 99.0 % and not more than 101.0 % of K_2CO_3.

Description Potassium Carbonate appears as white granules or powder, is odorless.
Potassium Carbonate is very soluble in water and practically insoluble in ethanol.
A solution of Potassium Carbonate (1 in 10) is alkaline.
Potassium Carbonate is hygroscopic.

Identification A solution of Potassium Carbonate (1 in 10) responds to the Qualitative Tests for potassium salt and for carbonate.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Potassium Carbonate in 20 mL of water: the solution is clear and colorless.

(2) ***Chloride***—To 0.2 g of Potassium Carbonate, add 6 mL of dilute nitric acid, heat, cool, add 6 mL of dilute nitric acid and use this solution as the test solution. Perform the test with the test solution as directed under Chloride Limit Test: not more than the amount corresponding to 0.3 mL of 0.01 mol/L hydrochloric acid.

(3) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat and place 10 mg to 300 mg of Potassium Carbonate on top. Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. Place the boat inside the furnace and heat to about 900 °C with a current of air or oxygen at 0.5 L/minute to 1 L/minute. Elute the mercury and sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 ng to 200 ng.

Additive—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 Potassium Carbonate, transfer to a 150 mL beaker and add 30 mL of water. Add hydrochloric acid in small amounts until the test specimen is completely dissolved 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 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 solution (2 in 100) and shake. Extract this solution with two 20 mL volumes of chloroform and evaporate the extract to dryness in 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 1.0 mL of standard lead 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 2.0 ppm).

Gas: Acetylene or hydrogen – Air
Lamp: 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 hydro-

chloric acid and evaporate to dryness on a water-bath. Dissolve the residue in 35 mL of water and 2 mL of dilute acetic acid, dilute with water to make 50 mL and perform the test. Prepare the control solution as follows: evaporate 6 mL of dilute hydrochloric acid on a water-bath to dryness, add 2 mL of dilute acetic acid and 2.0 mL of standard lead solution to dryness and dilute with water to make 50 mL (not more than 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 Test (1): no persisting yellow color is produced.

(7) ***Arsenic***—Prepare the test solution with 0.5 g of Potassium Carbonate, according to Method 1 and perform the test (not more than 4 ppm).

Loss on Drying Not more than 1.0 % (3 g, 180 °C, 4 hours).

Assay Dissolve about 1.5 g of Potassium Carbonate, previously dried and accurately weighed, in 25 mL of water, titrate with 0.5 mol/L sulfuric acid until the blue color of the solution changes to yellow-green, boil cautiously, then cool and titrate until a greenish yellow color develops (indicator: 2 drops of bromocresol green TS).

Each mL of 0.5 mol/L sulfuric acid
= 69.10 mg of K_2CO_3

Containers and Storage ***Containers***—Tight containers.

Potassium Hydroxide

KOH: 56.11

Potassium Hydroxide contains not less than 85.0 % and not more than 101.0 % of potassium hydroxide (KOH).

Description Potassium Hydroxide appears as white fused masses, in small pellets, in flasks, in sticks and in other forms. Potassium Hydroxide is hard and brittle and shows a crystalline fracture.
Potassium Hydroxide is freely soluble in water or in ethanol and practically insoluble in ether.
Potassium Hydroxide rapidly absorbs carbon dioxide in air.
Potassium Hydroxide deliquesces in the presence of moisture.

Identification (1) A solution of Potassium Hydroxide (1 in 500) is alkaline.

(2) A solution of Potassium Hydroxide (1 in 25) responds to the Qualitative Test for potassium salt.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Potassium Hydroxide in 20 mL of water: the solution is clear and colorless.

(2) ***Chloride***—Dissolve 2.0 g of Potassium Hydroxide in water and add water to make 100 mL. To 25 mL of the solution, add 8 mL of dilute nitric acid and water to make 50 mL. Perform the test. Prepare the control solution with 0.7 mL of 0.01 mol/L hydrochloric acid (not more than 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. Dissolve the residue in 35 mL of water, 2 mL of dilute acetic acid and 1 drop of ammonia TS, add water to make 50 mL and perform the test. Prepare the control solution as follows: evaporate 7 mL of dilute hydrochloric acid on a water-bath to dryness, dissolve the residue in 2 mL of dilute acetic acid and 3.0 mL of standard lead solution and add water to make 50 mL (not more than 30 ppm).

(4) ***Mercury***—Dissolve 2 g of Potassium Hydroxide in 10 mL of water. Add 1 mL of potassium permanganate (3 in 50) and about 30 mL of water and shake. Neutralize by gradually 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 (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 to the test bottle of an atomic absorption spectrophotometer. Add 10 mL of stannous chloride, connect the bottle immediately to the atomic absorption spectrophotometer and circulate air by operating the diaphragm pump. Read the absorbance of the test solution when the indication of the recorder rises rapidly and becomes constant. The absorbance is not more than that of the following solution: to 2 mL of mercury standard solution, add 1 mL of potassium permanganate (3 in 50), about 30 mL of water and the amount of hydrochloric acid used to treat the test solution and proceed in the same manner as the test specimen (not more than 0.1 ppm).

(5) ***Lead***—Weigh accurately 5.0 g of Potassium Hydroxide, transfer to a 150 mL beaker and add 30 mL of water. Add hydrochloric acid in small amounts until the test specimen is completely dissolved 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 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 solution (2 in 100) and shake. Extract this solution with two 20 mL volumes of chloroform and evaporate the extract to dryness in 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 1.0 mL of standard lead 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 2.0 ppm).

Gas: Acetylene or hydrogen – Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(6) ***Arsenic***—Dissolve 0.5 g of Potassium Hydroxide in 5 mL of water. Neutralize by gradually adding hydrochloric acid and use this solution as the test solution. Perform the test with the test solution as directed under Arsenic Test (not more than 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 Test (1): no persistent yellow color develops.

(8) ***Potassium carbonate***—The amount of potassium carbonate (K_2CO_3: 138.21) is not more than 2.0 % when calculated by the following equation using *B* (mL) obtained in the Assay.

$$\text{Amount of potassium carbonate (mg)} = 138.21 \times B$$

Assay Weigh accurately about 1.5 g of Potassium Hydroxide and dissolve in 40 mL of freshly boiled and cooled water. Cool the solution to 15 °C, add 2 drops of phenolphthalein TS and titrate with 0.5 mol/L sulfuric acid until the red color of the solution disappears. Record the amount *A* (mL) of 0.5 mol/L sulfuric acid consumed, then add 2 drops of methyl orange TS and titrate again with 0.5 mol/L sulfuric acid until the solution changes to a persistent pale red color. Record the amount *B* (mL) of 0.5 mol/L sulfuric acid consumed. Calculate the amount of KOH from the amount, *A* (mL) - *B* (mL).

Each mL of 0.5 mol/L sulfuric acid = 56.11 mg of KOH

Containers and Storage ***Containers*—**Tight containers.

Potassium Sulfate

K_2SO_4:174.26

Potassium Sulfate, when dried, contains not less than 99.0 % and not more than 101.0 % of potassium sulfate (K_2SO_4).

Description Potassium Sulfate appears as colorless crystals or a white, crystalline powder, has a slightly saline and somewhat bitter taste.
Potassium Sulfate is soluble in water and practically insoluble in ethanol.

Identification A solution of Potassium Sulfate (1 in 20) responds to the Qualitative Tests for potassium salt and for sulfate.

Purity (1) ***Clarity and color of solution and acidity or alkality***—Dissolve 1.0 g of potassium Sulfate in 20 mL of water: the solution is clear, colorless 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 (not more than 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 standard lead solution (not more than 10 ppm).

(4) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat and place 10 mg to 300 mg of Potassium Sulfate on top. Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. Place the boat inside the furnace and heat to about 900 °C with a current of air or oxygen at 0.5 L/minute to 1 L/minute. Elute the mercury and sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 ng to 200 ng.

Additive—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

test specimen is completely dissolved 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 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 solution (2 in 100) and shake. Extract this solution with two 20 mL volumes of chloroform and evaporate the extract to dryness in 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 1.0 mL of standard lead 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 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 electrothermal type Atomic Absorption Spectrophotometry. The absorbance of the test solution is not more than that of the following solution: to 3 mL of selenium standard solution, add water to make 100 mL and proceed in the same manner as the test solution (not more than 30 ppm).

(7) ***Sodium***—Dissolve 1.0 g of Potassium Sulfate in 20 mL of water and perform the test as directed under the Flame Coloration Test (1): no persistent yellow color develops.

(8) ***Arsenic***—Prepare the test solution with 4.0 g of Potassium Sulfate according to Method 1 and perform the test (not more than 5 ppm).

Loss on Drying Not more than 1.0 % (1 g, 110 °C, 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 with water until the last washing shows no opalescence on the addition of silver nitrate TS. Dry, ignite to constant weight between 500 °C and 600 °C by rising the temperature gradually and weigh as barium sulfate ($BaSO_4$: 233.39).

Amount (mg) of Potassium Sulfate (K_2SO_4)
= amount (mg) of barium sulfate ($BaSO_4$) × 0.7466

Containers and Storage ***Containers*—**Well-closed containers.

Potato Starch

Amylum Solani

Potato Starch consists of starch granules derived from the tuber of *Solanum tuberosum* Linné (*Solanaceace*).

Description Potato Starch is a white powder.
Potato Starch is practically insoluble in water or in dehydrated ethanol.

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 2- to 4-compound grains; spherical simple grains with non -centric or slightly eccentric hilum; striation distinct in all grains; a black cross, its intersection point on hilum, is observed when grains are put between two nicol prisms fixed at right angle to each other.
(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 pasty liquid obtained in (2), add 0.05 mL of diluted iodine TS (1 in 10): an orange to dark blue-purple color develops, and the color disappears by heating.

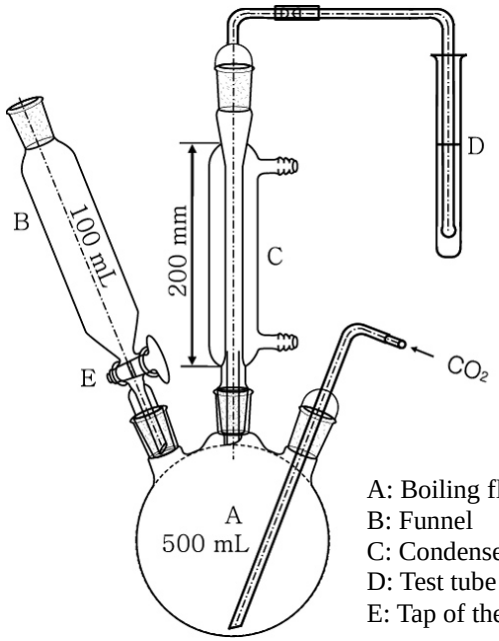
pH Place 5.0 g of Potato Starch in a non -metal vessel, add 25.0 mL of freshly boiled and cooled water, mix gently for 1 minute to form a suspension, and allow to stand for 15 minutes: the pH of this solution is between 5.0 and 8.0.

Purity (1) ***Iron***—To 1.5 g of Potato Starch, add 15 mL of 2 mol/L hydrochloric acid TS, mix, filter, and use the filtrate as the test solution. To 2.0 mL of standard iron solution, add water to make 20 mL, and use this solution as the control solution. Transfer 10 mL each of the test solution and the control solution in test tubes, add 2 mL each of a solution of citric acid (1 in 5) and 0.1 mL each of mercaptoacetic acid, and mix. Make the each solution alkaline to litmus paper with strong ammonia water, add water to make 20 mL each, 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 darker than that of the control solution (not more than 10 ppm).

(2) ***Oxidizing substances***—To 4.0 g of Potato Starch, add 50.0 mL of water, mix by shaking for 5 minutes and centrifuge. To 30.0 mL of the supernatant liquid, add 1 mL of acetic acid (100) and 0.5 to 1.0 g of

potassium iodide, mix by shaking and allow to stand for 20 to 25 minutes in a dark place. Add 1 mL of starch TS and titrate the solution with 0.002 mol/L sodium thiosulfate VS until the color of the solution disappears. Perform a blank determination and make any necessary correction: the volume of 0.002 mol/L sodium thiosulfate VS consumed is not more than 1.4 mL (not more than 20 ppm, calculated as hydrogen peroxide).

(3) ***Sulfur dioxide***—(i) Apparatus: Use apparatus shown in the figure.



A: Boiling flask
B: Funnel
C: Condenser
D: Test tube
E: Tap of the funnel

(ii) Procedure: Introduce 150 mL of water into the boiling flask, close the tap of the funnel, and pass carbon dioxide through the whole system at a rate of 100 ± 5 mL per minute. Pass cooling water through the condenser, and place 10 mL of hydrogen peroxide-sodium hydroxide TS (to a 9 : 1 mixture of water and hydrogen peroxide (hydrogen peroxide TS), add 3 drops of bromophenol blue TS and add 0.01 mol/L sodium hydroxide TS until the color changes to purple-blue; prepare before use) in the test-tube. After 15 minutes, remove the funnel without interrupting the stream of carbon dioxide, and introduce through the opening into the flask about 25 g of Potato Starch, accurately weighed, with the aid of 100 mL of water. Apply tap grease to the outside of the connection part of the funnel, and connect the funnel. Close the tap of the funnel, pour 80 mL of 2 mol/L hydrochloric acid TS into the funnel, open the tap to introduce the hydrochloric acid into the flask, and close the tap while several mL of the hydrochloric acid remains, in order to avoid losing sulfur dioxide. Place the flask in a water-bath, and heat the mixture for 1 hour. Transfer the contents of the test-tube with the aid of a little water to a wide-necked conical flask. Heat in a water-bath for 15 minutes, and cool. Add 0.1 mL of bromophenol blue TS, and titrate with 0.1 mol/L sodium hydroxide VS until the color changes from yellow to purple-blue lasting for at least 20 seconds. Perform a blank determination and make any necessary correction. Calculate the amount of sulfur dioxide by applying the following formula: it is not more than 50 ppm.

$$\text{Amount (ppm) of sulfur dioxide} = \frac{\text{Amount (mL) of 0.1 mol/L sodium hydroxide VS consumed}}{\text{Amount (g) of Potato Starch taken}} \times 1000 \times 3.203$$

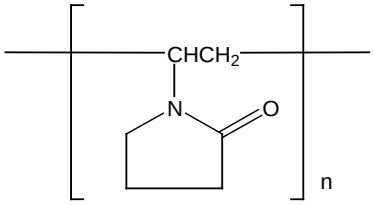
Loss on Drying Not more than 20.0 % (1 g, 130 °C, 90 minutes).

Residue on Ignition Not more than 0.6 % (1 g).

Microbial Limit The total aerobic microbial count is not more than 1000 CFU/g, the total combined yeasts/mould count is not more than 100 CFU/g, and Escherichia coli, Salmonella, Pseudomonas aeruginosa and Staphylococcus aureus are not observed.

Containers and Storage ***Containers*—**Well-closed containers.

Povidone



Polyvidone
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 not less than 11.5 % and not more than 12.8 % of nitrogen (N: 14.01), calculated on the anhydrous basis.
Povidone has a nominal K-value of not less than 25 and not more than 90. The nominal K-value is shown on the label.

Description Povidone is a white to pale yellow fine powder, is odorless or has a faint, characteristic odor.
Povidone is freely soluble in water, in methanol or in ethanol, slightly soluble in acetone and practically insoluble in ether.
Povidone is hygroscopic.

Identification 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 Infrared Spectrophotometry: 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 between 3.0 and 5.0 for Povidone having the nominal K-value of 30 or less and between 4.0 and 7.0 for Povidone having the nominal K-value exceeding 30.

Purity (1) ***Clarity and color of solution*** —Dissolve 1.0 g of Povidone in 20 mL of water: the solution is clear and colorless to pale yellow, or pale red.

(2) ***Heavy metals***—Proceed with 2.0 g of Povidone according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 10 ppm).

(3) ***Lead***—Weigh accurately 5.0 g of Povidone 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 30 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 60 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(4) ***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, heat at 60 °C for 60 minutes, allow to cool to room temperature and use this solution as the test solution. Separately, dissolve 0.10 g of freshly distilled acetaldehyde in water previously cooled to 4 °C to make exactly 100 mL. Allow to stand at 4 °C for about 20 hours, pipet 1.0 mL of this solution, add 0.05 mol/L pyrophosphate buffer solution, pH 9.0, to make exactly 100 mL and use this solution as the standard solution. Measure 0.5 mL each of the test solution, the standard solution and water (for blank test), transfer to separate cells, add 2.5 mL of 0.3 mol/L pyrophosphate buffer solution, pH 9.0 and 0.2 mL of β-nicotinamide adenine dinucleotide TS to each of these cells, mix and stopper tightly. Allow stand for 2 to 3 minutes at 22 ± 2 °C and perform the test with these solutions as directed under the Ultraviolet-visible Spectrophotometry using water as the control solution. Determine the absorbances, A_{T1}, A_{S1} and A_{B1} of the subsequent solution of the test solution, the standard solution and water at 340 nm, respectively. Add 0.05 mL of aldehyde dehydrogenase solution to each of the cells, mix and stopper tightly. Allow to stand for 5 minutes at 22 ± 2 °C. Determine the absorbances, A_{T2}, A_{S2} and A_{B2} of aldehyde of these solutions in the same manner as above: the content of aldehydes is not more than 500 ppm expressed as acetaldehyde.

$$\text{Content (ppm) of aldehydes} = \frac{(A_{T2} - A_{T1}) - (A_{B2} - A_{B1})}{(A_{S2} - A_{S1}) - (A_{B2} - A_{B1})} \times \frac{1000}{W}$$

W : Weighed amount (g) of Povidone, calculated on the anhydrous basis.

(5) ***1-Vinyl-2-pyrrolidone***—Weigh accurately about 0.25 g of Povidone, dissolve in diluted methanol (1 in 5) to make exactly 10 mL and use this solution as the test solution. Separately, dissolve 50 mg of 1-vinyl-2-pyrrolidone in methanol to make exactly 100 mL. Pipet 1.0 mL of this solution and add methanol to make exactly 100 mL. Pipet 5.0 mL of this solution, add diluted methanol (1 in 5) 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 operating conditions and determine the peak areas, A_T and A_S of 1-vinyl-2-pyrrolidone for the test solution and the standard solution, respectively: the content of 1-vinyl-2pyrrolidone is not more than 10 ppm.

$$\text{Content (ppm) of 1 - vinyl - 2 - pyrrolidone} = \frac{A_T}{A_S} \times \frac{2.5}{W}$$

W : Weighed amount (g) of Povidone, calculated on the anhydrous basis.

Operating conditions

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

Column: Stainless steel columns, about 4 mm in internal diameter and about 2.5 cm in length and about 4 mm in internal diameter and about 25 cm in length, packed with octylsilanized silica gel for liquid chromatography (5 μm in particle diameter) and use them as a guard column and a separation column, respectively.

Column temperature: A constant temperature of about 40 °C.

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

Flow rate: Adjust the flow rate so that the retention time of 1-vinyl-2-pyrrolidone is about 10 minutes.

System suitability

Selection of column : Dissolve 10 mg of 1-vinyl-2-pyrrolidone and 0.5 g of vinyl acetate in 100 mL of methanol. To 1 mL of this solution, add diluted methanol (1 in 5) to make 100 mL. Proceed with 50 μL of this solution according to the above operating conditions and calculate the resolution. Use a column giving elution of 1-vinyl-2-pyrrolidone and vinyl acetate in this order with the resolution between their peaks being not less than 2.0.

System repeatability : When the test is repeated 6 times with the standard solution under the above operating conditions, the relative standard deviation of obtained peak areas of 1-vinyl-2-pyrrolidone is not more than 2.0 %.

Detection sensitivity: Adjust the detection sensitivity so that the peak height of 1-vinyl-2-pyrrolidone obtained from 50 μL of the standard solution is between 10 mm and 15 mm.

Washing of the guard column: After each test with the test solution, wash away the polymeric material of Povidone from the guard column by passing the mobile phase through the column backwards for about 30 minutes at the same flow rate as applied in the test.

(6) ***2-Pyrrolidone***—Dissolve 0.1 g of Povidone in water to make 50 mL and use this solution as the test solution. Separately, dissolve 0.1 g of 2-Pyrrolidone RS in water to make 100 mL, pipet 3.0 mL of this solution, add water 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. When detection of 2-pyrrolidone in the pre-column is confirmed at detector 1 (about 1.2 minutes), switch the flow of the mobile phase directly from the pump to the analytical column. Determine each peak area of each solution by the automatic integration method: the peak area of 2-pyrrolidone obtained from the test solution is not more than that of 2-pyrrolidone from the standard solution (not more than 3.0 %).

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength 205 m). Detector 1 is placed between the pre-column and the analytical column, and detector 2 is placed after the analytical column.

Column: A stainless steel column about 4 mm in internal diameter and about 2.5 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter) as the pre-column, and a stainless steel column about 4 mm in internal diameter and about 25 cm in length, packed with aminohexadecylsilyl silica gel for liquid chromatography (5 μm in particle diameter) as the analytical column.

Column temperature: A constant temperature of about 30 °C

Mobile phase: Adjust the pH of water to 2.4 with phosphoric acid.

Flow rate: 1 mL/minute

System suitability

System performance: When the procedure is run with 10 μL of the standard solution under the above operating conditions, the symmetry factor is not more than 2.0.

Washing of the pre-column: After each test with the test solution or the standard solution, wash away the polymeric material of Povidone from the pre-column by passing the mobile phase through the column backwards for about 30 minutes at the same flow rate as applied in the test.

(7) ***Peroxides***—Weigh exactly an amount of Povidone, equivalent to 4.0 g 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 the test solution, add 2 mL of titanium trichloride-sulfuric acid TS and mix. Allow to stand for 30 minutes and perform the test with this solution as directed under the Ultraviolet-visible Spectrophotometry, using a solution prepared by adding 2 mL of 13 % sulfuric acid to 25 mL of the test solution as a blank : the absorbance of the subsequent solution of the test solution at 405 nm is not more than 0.35 (not more than 400 ppm, expressed as hydrogen peroxide).

(8) ***Hydrazine***—Transfer 2.5 g of Povidone to a 50-mL centrifuge tube, add 25 mL of water and stir to dissolve. Add 500 μL of a solution of salicyladehyde in methanol (1 in 20), stir and warm at 60 °C for 15 minutes in a water-bath. Allow to cool, add 2.0 mL of toluene, stopper tightly, shake vigorously for 2 minutes, centrifuge and use the upper layer of the mixture as the test solution. Separately, dissolve 90 mg of salicylaldazine in toluene to make exactly 100 mL. Pipet exactly 1 mL of this solution, add toluene to make exactly 100 mL and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under the Thin-layer Chromatography. Spot 10 μL each of the test solution and the standard solution on a plate coated with dimethylsilanzed silica gel with a fluorescent indicator for thin-layer chromatography. Develop the plate with diluted methanol (2 in 3) to a distance of about 15 cm and air-dry the plate. Examine under ultraviolet light (main wavelength: 365 nm): the R_f value of the fluorescent spot from the standard solution is about 0.3 and the fluorescence of the spot from the test solution corresponding to the spot from the standard solution is not more intense than that of the spot from the standard solution (not more than 1 ppm).

Water Not more than 5.0 % (0.5 g, volumetric titra-

tion, direct titration).

Residue on Ignition Not more than 0.1 % (1 g)

K-Value Weigh accurately an amount of Povidone, equivalent to 1.00 g calculated on the anhydrous basis 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 as directed in Method 1 under the Viscosity Determination and calculate the K-value by the following formula: the K-value of Povidone is not less than 90.0 % and not more than 108.0 % of the nominal K-value.

$$K = \frac{1.5\log\eta_{rel} - 1}{0.15 + 0.003c} + \frac{\sqrt{300c\log\eta_{rel} + (c + 1.5c\log\eta_{rel})^2}}{0.15c + 0.003c^2}$$

c : Mass (g) of Povidone in 100 mL of the solution, calculated on the anhydrous basis,

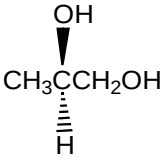
η_{rel} : Kinematic viscosity of the test solution relative to that of water.

Assay Weigh accurately about 0.1 g of Povidone and place in a Kjeldahl flask. Add 5 g of a powdered mixture of 33 g of potassium sulfate, 1 g of cupric sulfate and 1 g of titanium dioxide and wash down any adhering sample from the neck of the flask with a small amount of water. Add 7 mL of sulfuric acid allowing to flow down the inside wall of the flask. Heat the flask on an asbestos wire gauze over a free flame until the solution has a clear, yellow-green color and the inside wall of the flask is free from a carbonaceous material and then heat for further 45 minutes. After cooling, add cautiously 20 mL of water, cool the solution and connect the flask to the distillation apparatus previously washed by passing steam through it. To the absorption flask, add 30 mL of a solution of boric acid (1 in 25), 3 drops of bromocresol green-methyl red TS and sufficient water to immerse the lower end of the condenser tube. Add 30 mL of a solution of sodium hydroxide (2 in 5) through the funnel, rinse cautiously the funnel with 10 mL of water, immediately close the clamp attached to the rubber tube, then start the distillation with steam to get 80 mL to 100 mL of the distillate. Remove the absorption flask from the lower end of the condenser tube, rinsing the end part with a small quantity of water and titrate the distillate with 0.025 mol/L sulfuric acid until the color of the solution changes from green through pale grayish blue to pale grayish red-purple. Perform a blank determination and make any necessary correction.

Each mL of 0.025 mol/L sulfuric acid = 0.7003 mg of N

Containers and Storage ***Containers*—**Tight containers.

Propylene Glycol



and enantiomer

$C_3H_8O_2$: 76.09

(2*RS*)-Propane-1,2-diol [*57-55-6*]

Description Propylene Glycol is a clear, colorless, viscous liquid, is odorless and has a slightly bitter taste. Propylene Glycol is miscible with water, with methanol, with ethanol and with pyridine.
Propylene Glycol is freely soluble in ether.
Propylene Glycol 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 under a reflux condenser on a water-bath 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 and dry in a desiccator (silica gel) for 4 hours: the crystals melt between 174 °C and 178 °C.

(2) Heat gently 1 mL of Propylene Glycol with 0.5 g of potassium bisulfate: a characteristic odor is evolved.

(3) The retention time of the principal peak obtained from the test solution of Purity (8) is the same as that of the principal peak from the standard solution.

Specific Gravity d_{20}^{20} : 1.035 ~ 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 VS: the solution has 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 (not more than 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 sulfuric acid (not more than 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 standard lead solution (not more than 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 solution as directed under Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 2.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(6) ***Arsenic***—Prepare the test solution with 1.0 g of Propylene Glycol according to Method 1 and perform the test (not more than 2 ppm).

(7) ***Glycerin***—Heat 1.0 g of Propylene Glycol with 0.5 g of potassium bisulfate and evaporate to dryness: no odor of acrolein is perceptible.

(8) ***Ethylene glycol and diethylene glycol***—Weigh accurately a suitable amount of Propylene Glycol and the internal standard, dissolve in methanol to make 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 accurately an amount 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 Gas Chromatography according to the following operating conditions. Determine the peak areas of each solution by the automatic integration method: the ratio of the peak area of diethylene glycol to that of the internal standard obtained from the test solution is not more than the ratio of the peak area of diethylene glycol to that of the internal standard from the standard solution (not more than 0.10 %), and the ratio of the peak area of ethylene glycol to that of the internal standard obtained from the test solution is not more than the ratio of the peak area of ethylene glycol to that of the internal standard from the standard solution (not more than 0.10 %).

Internal standard—2,2,2-Trichloroethanol

Operating conditions

Detector: A hydrogen flame ionization detector

Column: A quartz glass tube 0.53 mm in internal diameter and about 30 m in length, with internal coating 3.0 μm in thickness made of cyanopropylphenyldimethylpolysiloxane (6:94) for gas chromatography.

Column temperature: Maintain at 100 °C for 4 minutes, raise the temperature to 120 °C at the rate of 50 °C per minute, maintain for 10 minutes, raise the temperature to 220 °C at the rate of 50 °C per minute and maintain at 220 °C for 6 minutes.

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/minute

Split ratio: About 1 : 10

System suitability

System performance: When the procedure is run with 1 μL of the standard solution under the above operating conditions, the resolution between the peaks of ethylene glycol and propylene glycol is not less than 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 Not more than 0.5 % (2 g, volumetric titration, direct titration).

Residue on Ignition Weigh accurately about 20 g of Propylene Glycol in a weighed crucible and heat to boiling. Stop heating and immediately ignite to burn. Cool, moisten the residue with 0.2 mL of sulfuric acid and heat strongly with care to constant weight: the residue is not more than 0.005 %.

Distilling Range 184 ~ 189 °C, not less than 95 vol %

Containers and Storage ***Containers*—**Tight containers.

Pyroxylin

Pyroxylin is a nitric acid ester of cellulose. Pyroxylin is usually moistened with isopropanol or some appropriate solvent.

Description Pyroxylin appears as white cotton-like substance or white flaskes.
Pyroxylin is freely soluble in acetone and very slightly soluble in ether.
Upon heating or exposure to light, Pyroxylin is decomposed with the evolution of nitrous acid vapors.

Identification Ignite Pyroxylin: it burns very rapidly with a luminous 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***—Shake 1.0 g of Pyroxylin, previously dried at 80 °C for 2 hours, with 20 mL of water for 10 minutes: 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 residue is not more than 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 gelatinize the sample. Ignite the contents to carbonize the test sample, ignite at about 500 °C for 2 hours and allow to cool in a dessicator (silica gel): the residue is not more than 0.30 %.

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light-resistant, packed loosely, remote from fire, and preferably in a cold place.

Rape Seed Oil

Rape Seed Oil is the fixed oil obtained from the seed of *Brassica campestris* Linné subsp. *napus* Hooker fil. et Anderson var. *nippo-oleifera* Makino (Cruciferae).

Description Rape Seed Oil is clear, pale yellow, slightly viscous oil, is odorless or has a slight odor and mild taste.
Rape Seed Oil is miscible with ether, with chloroform or with petroleum benzine.
Rape Seed Oil is slightly soluble in ethanol.

Specific gravity— d_{25}^{25} : 0.906 ~ 0.920.

Saponification Value 169 ~ 195.

Unsaponifiable Matters Not more than 1.5 %.

Acid Value Not more than 0.2.

Iodine Value 95 ~ 127.

Containers and Storage ***Containers*—**Tight containers.

Rice Starch

Rice Starch consists of the starch granules obtained from the seeds of *Oryza sativa* Linné (Gramineae).

Description Rice Starch is a white mass or powder, odorless and tasteless.
Under a microscope, Rice Starch appears as polyhedral, simple grains, 3 μm to 10 μm, mostly 4 μm to 6 μm, in size. These simple grains often gather in ellipsoidal, compound grains, 50 μm to 100 μm in diameter. Hilum and striation are not observable.
Rice Starch is practically insoluble in water or in ethanol.

Identification (1) To 1 g of Rice Starch add 50 mL of water, boil, and allow to cool: a turbid, neutral and pasty liquid is formed.

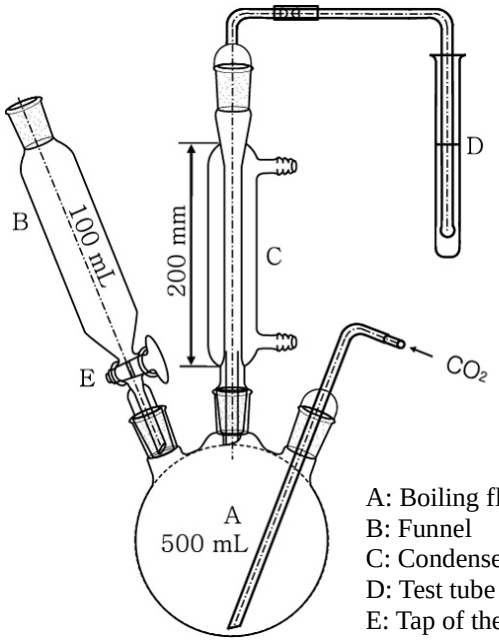
(2) To a portion of Rice Starch add iodine TS: a dark blue-purple color is produced.

Purity (1) ***Iron***—To 1.5 g of Rice 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 standard lead solution, add water to make 20 mL and use this solution as the control solution. Put 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 mercapto acetic acid and mix. To these solutions, add strong ammonia water until the color of litmus paper changes from red to blue then add water to make 20 mL and mix. Put 10 mL each of these solutions into test tubes, allow 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 (not more than 10 ppm).

(2) ***Oxidizing substances***—To 4.0 g of Rice Starch, add 50.0 mL of water, shake for 5 minutes and centrifuge. To 30.0 mL of the clear supernatant liquid, add 1 mL of acetic acid (100) and 0.5 g to 1.0 g of potassium iodide, shake and allow 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 determination and make any necessary correction.

Each mL of 0.002 mol/L sodium thiosulfate VS
= 34 μg g of oxidizing substances,
calculated as hydrogen peroxide

(3) ***Sulfur dioxide***—(i) Apparatus: Use apparatus shown in the figure.



A: Boiling flask
B: Funnel
C: Condenser
D: Test tube
E: Tap of the funnel

(ii) Procedure: Introduce 150 mL of water into the boiling flask, close the tap of the funnel, and pass carbon dioxide through the whole system at a rate of 100 ± 5 mL per minute. Pass cooling water through the condenser, and place 10 mL of hydrogen peroxide-sodium hydroxide TS (to a 9 : 1 mixture of water and hydrogen peroxide (hydrogen peroxide TS), add 3 drops of bromophenol blue TS and add 0.01 mol/L sodium hydroxide TS until the color changes to purple-blue; prepare before use) in the test-tube. After 15 minutes, remove the funnel without interrupting the stream of carbon dioxide, and introduce through the opening into the flask about 25 g of Rice Starch, accurately weighed, with the aid of 100 mL of water. Apply tap grease to the outside of the connection part of the funnel, and connect the funnel. Close the tap of the funnel, pour 80 mL of 2 mol/L hydrochloric acid TS into the funnel, open the tap to introduce the hydrochloric acid into the flask, and close the tap while several mL of the hydrochloric acid remains, in order to avoid losing sulfur dioxide. Place the flask in a water-bath, and heat the mixture for 1 hour. Transfer the contents of the test-tube with the aid of a little water to a wide-necked conical flask. Heat in a water-bath for 15 minutes, and cool. Add 0.1 mL of bromophenol blue TS, and titrate with 0.1 mol/L sodium hydroxide VS until the color changes from yellow to purple-blue lasting for at least 20 seconds. Perform a blank determination and make any necessary correction. Calculate the amount of sulfur dioxide by applying the following formula: it is not more than 50 ppm.

$$\text{Amount (ppm) of sulfur dioxide} = \frac{\text{Amount (mL) of 0.1 mol/L sodium hydroxide VS consumed}}{\text{Amount (g) of Rice Starch taken}} \times 1000 \times 3.203$$

Loss on Drying Not more than 15.0 % (1 g, 130 °C, 90 minutes).

Ash Not more than 0.6 %.

Microbial Limit The total aerobic microbial count is not more than 1000 CFU/g, the total combined yeasts/mould count is not more than 100 CFU/g and *Escherichia coli*, *Salmonella* species, *Staphylococcus aureus* and *Pseudomonas aeruginosa* are not observed.

Rosin

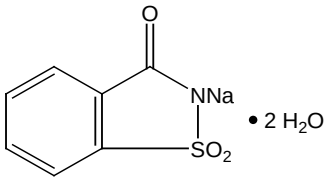
Rosin is the resin obtained from the exudation of plants of *Pinus* species (Pinaceae), from which essential oils have been removed.

Description Rosin is a pale yellow to pale brown, glassily transparent, brittle mass and its surface is often covered with a yellow powder. The fractured surface is shell-like and lustrous.
Rosin has a slight odor.
Rosin melts easily and burns with a yellow-brown flame.
Rosin is freely soluble in ethanol, in acetic acid (100) or in ether.
A solution of Rosin in ethanol is acidic.

Acid Value 150 ~ 177.

Ash Not more than 0.1 %.

Saccharin Sodium Hydrate



$C_7H_4NNaO_3S \cdot 2H_2O$: 241.20

Sodium 1,2-benzothiazol-3-olate 1,1-dioxide
[*6155-57-3*]

Saccharin Sodium Hydrate, when dried, contains not less than 98.0 % and not more than 101.0 % of saccharin sodium ($C_7H_4NNaO_3S$: 205.17).

Description Saccharin Sodium Hydrate appears as colorless crystals or white, crystalline powder, and has intensely sweet taste, even in 10000 dilutions.
Saccharin Sodium Hydrate is freely soluble in water or in methanol and sparingly soluble in ethanol or in acetic acid (100).
Saccharin Sodium Hydrate effloresces slowly and loses

about half the amount of water of crystallization in air.

Identification (1) Determine the infrared spectra of Saccharin Sodium Hydrate and Saccharin Sodium Hydrate RS as directed in the potassium bromide disk method under the Infrared Spectrophotometry: Both spectra exhibit similar intensities of absorption at the same wavenumbers.

(2) A solution of Saccharin Sodium Hydrate (1 in 10) responds to the Qualitative Tests for sodium salt.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Saccharin Sodium Hydrate in 1.5 mL of water or in 50 mL of ethanol: the solution is clear and colorless.

(2) ***Acid or alkali***—Dissolve 1.0 g of Saccharin Sodium Hydrate in 10 mL of water and add 1 drop of phenolphthalein TS: the solution is colorless. Add 1 drop of 0.1 mol/L sodium hydroxide VS to the solution: the color changes to 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, dilute with water to make 50 mL and rub the inner wall of the vessel with a glass rod until crystallization begins. Allow the solution to stand for 1 hour after the beginning of crystallization and then filter through dry filter paper. Discard the first 10 mL of the filtrate and take 25 mL of the subsequent filtrate. Add 2 mL of dilute acetic acid and water to make 50 mL and perform the test. Prepare the control solution as follows: to 2.0 mL of the standard lead solution, add 2 mL of dilute acetic acid and water to make 50 mL and use this solution as the control solution (not more than 20 ppm).

(4) ***Selenium***—Dissolve 1 g of Saccharin Sodium Hydrate in 100 mL of water and use this solution as the test solution. Determine the absorbance as directed under flameless type Atomic Absorption Spectrophotometry: the absorbance of the test solution is not more than that of selenium standard solution (take 30 mL and make 100 mL) (not more than 30 ppm).

(5) ***Lead***—Weigh accurately 5.0 g of Saccharin Sodium Hydrate 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 0.5 mL of standard lead 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 1.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(6) ***Arsenic***—Transfer 1.25 g of Saccharin Sodium Hydrate to a decomposition flask, add 10 mL of nitric acid and 5 mL of sulfuric acid and heat. Repeat this procedure until the solution becomes colorless to pale yellow and heat until white fumes are evolved. Cool, add 10 mL of water and 15 mL of saturated ammonium oxalate solution and heat until white fumes are evolved again. Cool, add water to make 25 mL and use 5 mL of the solution as the test solution. Perform the test with the test solution as directed under Arsenic Test: it meets the requirement. Transfer 5 mL of standard arsenic solution to a decomposition flask, add 10 mL of nitric acid and 5 mL of sulfuric acid, proceed in the same manner as the test specimen and use this solution as the standard stain (not more than 4 ppm).

(7) ***Benzoate and salicylate***—Dissolve 0.5 g of Saccharin Sodium Hydrate in 10 mL of water, add 5 drops of acetic acid and 3 drops of ferric chloride TS: no turbidity is produced and no red-purple to purple color develops.

(8) ***Orthotoluene sulfonamide***—Dissolve 10 g of Saccharin Sodium Hydrate in 50 mL of water and extract three times with 30 mL volumes of ethyl acetate. Combine all the ethyl acetate extracts, wash with 30 mL of a solution of sodium chloride (1 in 4), dehydrate with 5 g of anhydrous sodium sulfate and evaporate the ethyl acetate. Dissolve the residue in 5.0 mL of the internal standard solution and use this solution as the test solution. Separately, dissolve 0.10 g of orthotoluene sulfonamide in ethyl acetate to make exactly 100 mL. Pipet 1.0 mL of this solution, evaporate on a water-bath to dryness, dissolve the residue in 5.0 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 operating conditions and calculate the ratios, Q_T and Q_S, of the peak height of orthotoluene sulfonamide to that of the internal standard for the test solution and the standard solution, respectively: Q_T is not more than Q_S.

Internal standard solution—A solution of caffeine in ethyl acetate (1 in 500).

Operating conditions

Detector: A hydrogen flame-ionization detector.
Column: A column, about 3 mm in internal diame-

ter and about 1 m in length, packed with siliceous earth for gas chromatography (180 μm to 250 μm in diameter), coated with diethyleneglycol polyester for gas chromatography succinate at the ratio of 3 %.

Column temperature: A constant temperature of about 200 °C.

Injection port temperature: A constant temperature of about 250 °C.

Carrier gas: Nitrogen.

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

System suitability

System performance: When the procedure is run with 1 μL of the standard solution according to the above operating conditions, the internal standard and orthotoluene sulfonamide are eluted in this order with a resolution between their peaks being not less than 2.0.

System repeatability: When the test is repeated 6 times with 1 μL each of the standard solution according to the above operating conditions, the relative standard deviation of the ratio of the peak height of orthotoluene sulfonamide to that of the internal standard is not more than 2.0 %.

(9) ***Readily carbonizable substances***—Perform the test with 0.20 g of Saccharin Sodium Hydrate. Allow the solution to stand between 48 °C and 50 °C for 10 minutes: the solution is not more intense than Color Matching Fluid A.

Water Not more than 15.0 % (0.1 g, volumetric titration, direct titration).

Loss on Drying Not more than 15.0 % (1 g, 120 °C, 4 hours).

Assay Weigh accurately about 0.5 g of Saccharin Sodium Hydrate, dissolve in 50 mL of acetic acid (100), heat slightly if necessary, and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration, Endpoint Detection Method in Titrimetry). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 20.52 mg of $C_7H_4NNaO_3S$

Containers and Storage ***Containers*—**Well-closed containers.

Sesame Oil

Oleum Sesami

Sesame Oil is the fixed oil obtained from seeds of *Sesamum indicum* Linné (Pedaliaceae).

Description Sesame Oil is clear, pale yellow oil, odorless or has a faint, characteristic odor and has bland taste.

Sesame Oil is miscible with ether, with chloroform, with petroleum ether or with carbon disulfide.

Sesame Oil is slightly soluble in ethanol.

Sesame Oil congeals between 0 °C and -5 °C.

Congealing point of the fatty acids—20 ~ 25 °C.

Identification Take 1 mL of Sesame Oil, add 0.1 g of sucrose and 10 mL of hydrochloric acid and shake for 30 seconds: the acid layer becomes pale red and changes to red on standing.

Saponification Value 187 ~ 194.

Unsaponifiable Matters Not more than 2.0 %.

Specific Gravity d_{25}^{25} : 0.914 ~ 0.921.

Acid Value Not more than 0.2.

Iodine Value 103 ~ 118.

Cottonseed Oil Place 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. Warm carefully until the carbon disulfide is expelled and immerse the test tube to one-third of its length in a boiling, saturated solution of sodium chloride: no red color develops within 15 minutes.

Triglyceride Composition Weigh accurately about 0.2 g of Sesame Oil, transfer to a 10 mL volumetric flask, dissolve in the mobile phase and use this solution as the test solution. Perform the test with 20 μL of the test solution as directed under Liquid Chromatography according to the following operating conditions and determine the areas of the 8 glyceride major peaks and calculate the amounts by the area percentage method: 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 is 5.0 to 14.0 %, 1-linoleoyl-2-oleoyl-3-stearoyl-rac-glycerol is 2.0 to 8.0 %, and 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/minute

Time span of measurement: 40 minues beginning 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. When the procedure is run with 20 μL of this solution under the above operating conditions, the relative retention times of 1,2-dioleoyl-3-linoleoyl-rac-glycerol and 1,2-dilinoleoyl-3-palmitoyl-rac-glycerol are 0.93 and 1.0, respectively, with the resolution between these peaks being not less than 1.8.

System repeatability: When the test is repeated 6 times with 20 μL each of the test solution under the above operating conditions, the relative standard deviation of the peak area is not more than 1.5 % and the peak area ratio of 1,2-dioleoyl-3-linoleoyl-rac-glycerol with respect to the peak area of 1,2-dilinoleoyl-3-palmitoyl-rac-glycerol is not more than 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-glyerol, triolein and 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 and 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 standard lead solution (not more than 10 ppm).

Containers and Storage ***Containers***—Tight containers.

Purified Shellac

Purified Shellac is a resin-like substance obtained from a purified secretion of *Laccifer lacca* Kerr (Coccidae).

Description Purified Shellac is a pale yellow-brown to brown, lustrous, hard, brittle scutella, and has no odor or has a faint, characteristic odor.
Purified Shellac is freely soluble in ethanol or in dehydrated ethanol and practically insoluble in water or in ether.
Purified Shellac dissolves in sodium hydroxide TS.

Acid Value 60 ~ 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).

Purity (1) ***Heavy metals***—Proceed with 2.0 g of Purified Shellac according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 10 ppm).

(2) ***Lead***—Weigh accurately 5.0 g of Purified Shellac 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 solution as directed under Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 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 an ethanol solution of magnesium nitrate (1 in 50), then add 1.5 mL of strong hydrogen peroxide water and fire to burn (not more than 5 ppm).

(4) ***Ethanol-insoluble substances***—Dissolve about 5 g of Purified Shellac, accurately weighed, in 50 mL of ethanol on a water-bath while shaking. Pour the ethanol solution into a tared extraction thimble, previously dried at 105 °C for 2 hours, in a Soxhlet extractor and extract with ethanol for 3 hours: the residue is not more than 2.0 %. Use a cylindrical weighing bottle for taring the extraction thimble.

(5) ***Rosin***—Dissolve 2.0 g of Purified Shellac in 10 mL of dehydrated ethanol with thorough shaking, add gradually 50 mL of petroleum ether while shaking and filter, if necessary. Wash the solution twice with 50 mL volumes of water, filter the upper layer and evaporate the filtrate on a water-bath to dryness. Dissolve the residue in 2 mL of a mixture of carbon tetrachloride and phenol (2 : 1), transfer the solution to a depression of a spot plate and fill the neighboring depression with a mixture of carbon tetrachloride and bromine (4 : 1). Immediately cover both depressions with a watch glass and allow to stand: the solution of the residue exhibits no purple or blue color within 1 minute.

(6) ***Wax***—Dissolve 10.0 g of Purified Shellac in

150 mL of a solution of sodium carbonate (9 in 200) with shaking on a water-bath and continue the heating for 2 hours. After cooling, collect the floating wax by filtration, wash the wax and the filter paper with water, transfer to a beaker and dry at 65 °C until the water is almost evaporated. Transfer the wax together with the filter paper to an extraction thimble in a Soxhlet extractor. Dissolve the wax remaining in the beaker with a suitable quantity of chloroform by warming. Pour the solution into the thimble and extract with chloroform for 2 hours. Evaporate the chloroform solution to dryness and dry the residue at 105 °C for 3 hours: the residue is not more than 20 mg.

Loss on Drying Not more than 2.0 %. Weigh accurately about 1 g of medium powder of Purified Shellac and dry at 40 °C for 4 hours, then for 15 hours in a desiccator (calcium chloride for drying).

Ash Not more than 1.0 % (1 g, proceed as directed in the Ash under the Test for Herbal Drugs).

Containers and Storage ***Containers***—Well-closed containers.

White Shellac

White Shellac is a resin-like substance obtained from a bleached secretion of *Laccifer lacca* Kerr (Coccidae).

Description White Shellac appears as yellowish white to pale yellow, hard, brittle granules and is odorless or has faint, characteristic odor.
White Shellac is sparingly soluble in ethanol, very slightly soluble in petroleum ether and practically insoluble in water.
White Shellac dissolves in sodium hydroxide TS.

Acid Value 65 ~ 90. Weigh accurately about 0.5 g of White Shellac, add 50 mL of neutralized ethanol as a solvent and dissolve by warming. After cooling, perform the test.

Purity (1) ***Chloride***—Shake and dissolve 0.40 g of White Shellac in 5 mL of ethanol while warming, add 40 mL of water and cool. Add 12 mL of dilute nitric acid and water to make 100 mL and filter. Perform the test using 50 mL of the filtrate as the test solution. Prepare the control solution as follows: to 0.80 mL of 0.01 mol/L hydrochloric acid VS, add 2.5 mL of ethanol, 6 mL of dilute nitric acid and water to make 50 mL (not more than 0.140 %).

(2) ***Sulfate***—Shake and dissolve 0.40 g of White Shellac in 5 mL of ethanol by warming, add 40 mL of water and cool. Add 2 mL of dilute hydrochloric acid and water to make 100 mL and filter. Perform the test using 50 mL of the filtrate as the test solution. Prepare the control solution as follows: to 0.45 mL of 0.005 mol/L sulfuric acid, add 2.5 mL of ethanol, 1 mL of dilute hydrochloric acid and water to make 50 mL (not more than 0.110 %).

(3) ***Heavy metals***—Proceed as directed in the Purity (1) under Purified Shellac.

(4) ***Lead***—Weigh accurately 5.0 g of White Shellac 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 solution as directed under Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 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 Not more than 6.0 %. Weigh accurately about 1 g of medium powder of White Shellac and dry at 40°C for 4 hours, then for 15 hours in a desiccator (calcium chloride for drying).

Ash Not more than 1.0 % (1 g, proceed as directed in the Ash under the Test for Herbal Drugs).

Containers and Storage ***Containers***—Well-closed containers.

Storage—In a cold place.

Light Anhydrous Silicic Acid

Light Anhydrous Silicic Acid contains not less than 98.0 % and not more than 101.0 % of silicon dioxide (SiO_2: 60.08), calculated on the incinerated basis.

Description Light Anhydrous Silicic Acid is a white to bluish white, light, fine power, is odorless, tasteless and smooth to the touch.
Light Anhydrous Silicic Acid is practically insoluble in water, in ethanol, or in ether.
Light Anhydrous Silicic Acid dissolves in hydrofluoric acid, in potassium hydroxide TS or in hot sodium hydroxide TS and does not dissolve in dilute hydrochloric acid.

Identification (1) Dissolve 0.1 g of Light Anhydrous Silicic Acid in 20 mL of sodium hydroxide TS by boiling and add 12 mL of ammonium chloride TS: a white, gelatinous precipitate is produced and the precipitate does not dissolve in dilute hydrochloric acid.

(2) Take the precipitate obtained in (1), add 10 mL of a solution of methylene blue (1 in 10000) and wash with water: the precipitate has a blue color.

(3) Prepare a bead by fusing dibasic sodium ammonium phosphate on a platinum loop. Bring the hot, transparent bead into contact with Light Anhydrous Silicic Acid and fuse again: an insoluble matter is perceptible in the bead. The resulting bead, upon cooling, becomes opaque and acquires a reticulated appearance.

Purity (1) ***Chloride***—Dissolve 0.5 g of Light Anhydrous Silicic Acid in 20 mL of sodium hydroxide TS by boiling, cool, filter, if necessary and wash with 10 mL of water. Combine the filtrate and washings, add 18 mL of dilute nitric acid, shake and add water to make 50 mL. Perform the test. Prepare the control solution as follows: to 0.15 mL of 0.01 mol/L hydrochloric acid VS, add 20 mL of sodium hydroxide TS, 18 mL of dilute nitric acid and add water to make 50 mL (not more than 0.011 %).

(2) ***Heavy metals***—Dissolve 0.5 g of Light Anhydrous Siliclc Acid in 20 mL of sodium hydroxide TS by boiling, cool, add 15 mL of acetic acid, shake, filter, if necessary, wash with 10 mL of water, combine the filtrate and washings and add water to make 50 mL. 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 standard lead solution, 2 mL of dilute acetic acid and water to make 50 mL and use this solution as the control solution (not more than 40 ppm).

(3) ***Aluminum***—Dissolve 0.5 g of Light Anhydrous Silicic Acid in 40 mL of sodium hydroxide TS by boiling, cool, 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 potassium aluminum sulfate in water and add 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***—Take 40 mg of Light Anhydrous Silicic Acid, add 10 mL of dilute hydrochloric acid and heat for 10 minutes in a water-bath while shaking. After cooling, add 0.5 g of L-tartaric acid to dissolve with shaking. Prepare the test solution with this solution according to Method 2 and perform the test according to Method B. Prepare the control solution with 2.0 mL of standard iron solution (not more than 500 ppm).

(5) ***Calcium***—Dissolve 1.0 g of Light Anhydrous Silicic Acid in 30 mL of sodium hydroxide TS by boiling, cool, add 20 mL of water, 1 drop of phenolphthalein TS and dilute nitric acid until the color of this solution disappears, immediately add 5 mL of dilute acetic acid, shake, add water to make 100 mL and obtain a clear liquid by centrifugation or filtration. To 25 mL of this liquid, add 1 mL of oxalic acid TS and ethanol to make 50 mL, immediately shake 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 °C 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. To 25 mL of this solution, add 1 mL of oxalic acid TS and ethanol to make 50 mL and shake.

(6) ***Arsenic***—Dissolve 0.40 g of Light Anhydrous Silicic Acid in 10 mL of sodium hydroxide TS by boiling in a porcelain crucible, cool, add 5 mL of water and 5 mL of dilute hydrochloric acid, shake and perform the test (not more than 5 ppm).

Loss on Drying Not more than 7.0 % (1 g, 105 °C, 4 hours).

Loss on Ignition Not more than 12.0 % (1 g, 850 °C to 900 °C, constant weight).

Volume Test Weigh 5.0 g of Light Anhydrous Silicic Acid, transfer gradually to a 200 mL measuring cylinder and allow to stand: the volume is not less than 70 mL.

Assay Weigh accurately about 1.0 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 and heat between 110 °C and 120 °C for 2 hours. 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, filter rapidly and wash the residue with warm water until the last washing becomes negative to the Qualitative Tests (2) for chloride. Transfer the residue together with the filter paper to a platinum crucible, ignite to ash and continue the ignition for 30 minutes. Cool, weigh the crucible and designate the mass as *a* (g). Moisten the residue in the crucible with water, add 6 mL of hydrofluoric acid and 3 drops of sulfuric acid and evaporate to dryness. Heat strongly for 5 minutes, cool, weigh the crucible and designate the mass as *b* (g).

Amount (g) of silicon dioxide (SiO_2) = $a - b$

Containers and Storage ***Containers*—**Tight containers.

Sodium Acetate Hydrate

$C_2H_3NaO_2 \cdot 3H_2O$: l36.08

Monosodium acetate trihydrate, [*6131-90-4*]

Sodium Acetate Hydrate, when dried, contains not less than 99.5 % and not more than 101.0 % of sodium acetate ($C_2H_3NaO_2$: 82.03), calculated on the anhydrous basis.

Description Sodium Acetate Hydrate appears as colorless crystals or white, crystalline powder, odorless or has a slight, acetous odor and has cool, saline and slight bitter taste.
Sodium Acetate Hydrate is very soluble in water, freely soluble in acetic acid (100), soluble in ethanol and practically insoluble in ether.
Sodium Acetate Hydrate is efflorescent in warm, dry air.

Identification A solution of Sodium Acetate Hydrate (1 in 10) responds to the Qualitative Tests 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 solution is clear and colorless.

(2) ***Acid or alkali***—Dissolve 1.0 g of Sodium Acetate Hydrate in 20 mL of freshly boiled and cooled water and add 3 drops of phenolphthalein TS: a red color develops. When cooled to 10 °C, or 1.0 mL of 0.01 mol/L hydrochloric acid VS is added after cooling to 10 °C, the red color disappears.

(3) ***Chloride***—Perform the test with 1.0 g of Sodium Acetate Hydrate. Prepare the control solution with 0.30 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.011 %).

(4) ***Sulfate***—Perform the test with 1.0 g of Sodium Acetate Hydrate. Prepare the control solution with 0.35 mL of 0.005 mol/L sulfuric acid VS (not more than 0.017 %).

(5) ***Heavy metals***—Proceed with 2.0 g of Sodium Acetate Hydrate according to Method 1 and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 10 ppm).

(6) ***Calcium and magnesium***—Dissolve 4.0 g of Sodium Acetate Hydrate in 25 mL of water, add 6 g of ammonium chloride, 20 mL of strong ammonia water and 0.25 mL of a solution of sodium bisulfite (1 in 10) and titrate with 0.01 mol/L disodium ethylenediamine tetraacetate VS until the blue color changes to grayish blue (indicator: 0.1 g of methylthymol blue-potassium nitrate indicator): the amount of 0.01 mol/L disodium ethylenediamine tetraacetate VS consumed is not more than 0.5 mL.

(7) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat and place 10 mg to 300 mg of Sodium Acetate Hydrate on top. Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. Place the boat inside the furnace and heat to about 900 °C with a current of air or oxygen at 0.5 L/minute to 1 L/minute. Elute the mercury and sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 ng to 200 ng.

Additive—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.

(8) ***Lead*—**Weigh accurately 5.0 g of Sodium Acetate Hydrate 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 solution as directed under Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 2.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(9) ***Arsenic***—Prepare the test solution with 1.0 g of Sodium Acetate Hydrate, according to Method 1 and perform the test (not more than 2 ppm).

(10) ***Potassium permanganate-reducing substance***—Dissolve 1.0 g of Sodium Acetate Hydrate in 100 mL of water, add 5 mL of dilute sulfuric acid, boil, add 0.50 mL of 0.002 mol/L potassium permanganate VS and further boil for 5 minutes: the red color of the solution does not disappear.

Loss on Drying Not less than 39.0 % and not more than 40.5 % (1 g, first at 80 °C for 2 hours and then at 130 °C for 2 hours).

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 until the color of the solution changes from yellow to green (indicator: 1 mL of α-naphtholbenzeine TS). Perform a blank determination and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid
= 8.203 mg of $C_2H_3NaO_2$

Containers and Storage ***Containers*—**Tight containers.

Sodium Bisulfite

Sodium Hydrogen Sulfite $NaHSO_3$: 104.06

Sodium Bisulfite is a mixture of sodium bisulfite and sodium pyrosulfite. Sodium Bisulfite contains not less than 64.0 % and not more than 67.4 % of sulfur dioxide (SO_2: 64.06).

Description Sodium Bisulfite appears as white granules or powder, and has the odor of sulfur dioxide.
Sodium Bisulfite is freely soluble in water and practically insoluble in ethanol and in ether.
A solution of Sodium Bisulfite (1 in 20) is acid.
Sodium Bisulfite is slowly affected by air or by light.

Identification A solution of Sodium Bisulfite (1 in 20) responds to the Qualitative Tests for sodium salt and for bisulfite.

Purity (1) ***Clarity and color of solution*—**Dissolve 1.0 g of Sodium Bisulfite in 10 mL of water: the 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 and allow to stand for 5 minutes: no turbidity is produced.

(3) ***Heavy metals*—**Dissolve 1.0 g of Sodium Bisulfite in 10 mL of water, add 5 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 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 and 2.0 mL of standard lead solution and dilute with water to make 50 mL (not more than 20 ppm).

(4) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat. In the case of a solid test specimen, take 10 to 300 mg of the test specimen, cut and homogenized. In the case of a liquid sample, allow 0.1 to 0.5 mL to completely permeate additive (a). Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. 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 sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen

(not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 to 200 ng.

Additive—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 Sodium Bisulfite, transfer to a 150 mL beaker and add 30 mL of water. Add hydrochloric acid in small amounts until the test specimen is completely dissolved 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 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 solution (2 in 100) and shake. Extract this solution with two 20 mL volumes of chloroform and evaporate the extract to dryness in 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, transfer 1.0 mL of standard lead 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 2.0 ppm).

Gas: Acetylene or hydrogen – Air
Lamp: 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 with 2.0 mL of standard iron solution (not more than 20 ppm).

(7) ***Selenium***—Weigh accurately 2.0 g of Sodium Bisulfite and transfer to a 50 mL beaker. Add 10 mL of water and 5 mL of hydrochloric acid, boil to remove sulfur dioxide and use this solution as the test solution. Separately, put 1.0 g of Sodium Bisulfite and 0.5 mL of selenium standard solution into a beaker, proceed in the same manner as the test solution and use this solution as the control solution. To each of the test solution and the control solution, add 2 g of hydrazine sulfate, dissolve by warming, allow to stand for 5 minutes, 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 (not more than 5 ppm).

(8) ***Arsenic***—Dissolve 0.5 g of Sodium Bisulfite in 10 mL of water. Add 1 mL of sulfuric acid, heat on a sand-bath until white fumes are evolved, add water to make 5 mL and perform the test (not more than 4 ppm).

Assay Weigh accurately about 0.15 g of Sodium Bisulfite and transfer immediately to an iodine flask containing 50 mL of 0.05 mol/L iodine VS, stopper, shake and allow to stand for 5 minutes in a dark place. Add 1 mL of hydrochloric acid and titrate the excess iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 1 mL of starch TS). Perform a blank determination and make any necessary correction.

Each mL of 0.05 mol/L iodine VS
= 3.2032 mg of SO_2

Containers and Storage ***Containers***—Tight containers.

Storage—Lght-resistant, preferably well-filled, and not exceeding 30 °C.

Sodium Carbonate Hydrate

$Na_2CO_3 \cdot 10H_2O$: 286.14

Sodium Carbonate Hydrate contains not less than 99.0 % and not more than 103.0 % of sodium carbonate hydrate ($Na_2CO_3 \cdot 10H_2O$).

Description Sodium Carbonate Hydrate appears as colorless or white crystals.
Sodium Carbonate Hydrate is freely soluble in water and practically insoluble in ethanol or in ether.
A solution of Sodium Carbonate Hydrate (1 in 10) is alkaline.
Sodium Carbonate Hydrate is efflorescent in air.
Sodium Carbonate Hydrate liquefies in its water of crystallization at 34 °C and becomes anhydrous at above 100 °C.

Identification A solution of Sodium Carbonate Hydrate (1 in 20) responds to the Qualitative Tests for sodium salt and for carbonate.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Sodium Carbonate Hydrate in 5 mL of water:

the solution is clear and colorless

(2) ***Chloride*—**Dissolve 0.5 g of Sodium Carobonate Hydrate in 10 mL of water, add 7 mL of dilute nitric acid, dilute with water to make 50 mL and perform the test. Prepare the control solution with 1.0 mL of 0.01 mol/L hydrochloric acid (not more than 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, dilute with water to make 50 mL and perform the test. Prepare the control solution as follows: evaporate 8 mL of dilute hydrochloric acid on a water-bath to dryness, add 2 mL of dilute acetic acid and 2.0 mL of standard lead solution and dilute with water to make 50 mL (not more than 10 ppm).

(4) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat and place 10 to 300 mg of Sodium Carbonate Hydrate on top. Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. 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 sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 to 200 ng.

Additive—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 Sodium Carbonate Hydrate, transfer to a 150 mL beaker and add 30 mL of water. Add hydrochloric acid in small amounts until the test specimen is completely dissolved 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 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 solution (2 in 100) and shake. Extract this solution with two 20 mL volumes of chloroform and evaporate the extract to dryness in 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, transfer 2.0 mL of standard lead 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 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 (not more than 3.1 ppm).

Loss on Drying 61.0 ~ 63.0 % (1 g, 105 °C, 4 hours).

Assay Dissolve about 3 g of Sodium Carbonate Hydrate, weighed accurately, in 25 mL of water and titrate with 0.5 mol/L sulfuric acid until the color of the solution changes from blue to yellow-green. Boil carefully, cool and further titrate until a greenish yellow color appears (indicator: 2 drops of bromocresol green TS).

Each mL of 0.5 mol/L sulfuric acid
= 143.07 mg of $Na_2CO_3 \cdot 10H_2O$

Containers and Storage ***Containers*—**Tight containers.

Dried Sodium Carbonate

Anhydrous Sodium Carbonate
Sodium Carbonate Na_2CO_3: 105.99

Dried Sodium Carbonate, when dried, contains not less than 99.0 % and not more than 101.0 % of sodium carbonate (Na_2CO_3).

Description Dried Sodium Carbonate appears as

white crystals or crystalline powder.
Dried Sodium Carbonate is freely soluble in water and practically insoluble in ethanol or in ether.
A solution of Dried Sodium Carbonate (1 in 10) is alkaline.
Dried Sodium Carbonate is hygroscopic.

Identification A solution of Dried Sodium Carbonate (1 in 20) responds to the Qualitative Tests for sodium salt and for carbonate.

Purity (1) ***Clarity and color of solution*** —Dissolve 1.0 g of Dried Sodium Carbonate in 10 mL of water: the 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, dilute with water to make 50 mL and perform the test. Prepare the control solution with 1.0 mL of 0.01 mol/L hydrochloric acid (not more than 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 on a water-bath to dryness. Dissolve the residue in 35 mL of water and 2 mL of dilute acetic acid, dilute with water to make 50 mL and perform the test. Prepare the control solution as follows: evaporate 7.5 mL of dilute hydrochloric acid on a water-bath to dryness, add 2 mL of dilute acetic acid and 2.0 mL of standard lead solution and dilute with water to make 50 mL (not more than 20 ppm).

(4) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat and place 10 to 300 mg of Dried Sodium Carbonate on top. Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. 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 sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 to 200 ng.

Additive—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 Dried Sodium Carbonate, transfer to a 150 mL beaker and add 30 mL of water. Add hydrochloric acid in small amounts until the test specimen is completely dissolved 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 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 solution (2 in 100) and shake. Extract this solution with two 20 mL volumes of chloroform and evaporate the extract to dryness in 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, transfer 1.0 mL of standard lead 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 2.0 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 Dried Sodium Carbonate according to Method 1 and perform the test (not more than 3.1 ppm).

Loss on Drying Not more than 2.0 % (2 g, 105 °C, 4 hours).

Assay Dissolve about 1.2 g of Dried Sodium Carbonte, weighed accurately, in 25 mL of water, and titrate with 0.5 mol/L sulfuric acid until the color of solution changes from blue to yellow-green. Then boil cautiously, cool, titrate until to a greenish yellow color develops (Indicator: bromcresol green TS, 2 drops).

Each mL of 0.5 mol/L sulfuric acid
= 52.99 mg of Na_2CO_3.

Containers and Storage ***Containers***—Tight containers.

Sodium Hydroxide

NaOH: 40.00

Sodium Hydroxide contains not less than 95.0 % and not more than 101.0 % of sodium hydroxide (NaOH).

Description Sodium Hydroxide appears as white fused masses, small pellets, flakes, sticks and other forms. Sodium Hydroxide is hard and brittle and shows a crystalline fracture.
Sodium Hydroxide is freely soluble in water or in ethanol and practically insoluble in ether.
Sodium Hydroxide rapidly absorbs carbon dioxide in air.
Sodium Hydroxide deliquesces in moist air.

Identification (1) A solution of Sodium Hydroxide (1 in 500) is alkaline.

(2) A solution of Sodium Hydroxide (1 in 25) responds to the Qualitative Tests for sodium salt.

Purity (1) ***Clarity and color of solution***—Dissolve 1.0 g of Sodium Hydroxide in 20 mL of water: the solution is clear and colorless.

(2) ***Chloride***—Dissolve 2.0 g of Sodium Hydroxide in water and add water to make 100 mL. To 25 mL of the solution, add 10 mL of dilute nitric acid and water to make 50 mL and perform the test. Prepare the control solution with 0.7 mL of 0.01 mol/L hydrochloric acid (not more than 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 on a water-bath to dryness. Dissolve the residue in 35 mL of water, add 2 mL of dilute acetic acid and 1 drop of ammonia TS, add water to make 50 mL and perform the test. Prepare the control solution as follows: evaporate 11 mL of dilute hydrochloric acid on a water-bath to dryness, dissolve the residue in 2 mL of dilute acetic acid and 3.0 mL of standard lead solution, add water to make 50 mL (not more than 30 ppm).

(4) ***Potassium***—Dissolve 0.10 g of Sodium Hydroxide in water and dilute with water to make 40 mL. Add 1.0 mL of dilute acetic acid to 4.0 mL of this solution and shake. Add 5.0 mL of a solution of sodium tetraphenylboron (1 in 30), shake immediately and allow to stand for 10 minutes: the solution has no more turbidity than the following control solution.

Control solution—Dissolve 9.5 mg of potassium chloride in water and dilute with water to make 1000 mL. Add 1.0 mL of dilute acetic acid to 4.0 mL of this solution, shake and proceed as directed above.

(5) ***Sodium carbonate***—The amount of sodium carbonate (Na_2CO_3: 105.99) is not more than 2.0 %, when calculated by the following equation using B (mL) which is obtained in the Assay.

Amount (mg) of sodium carbonate = $105.99 \times B$

(6) ***Mercury***—Dissolve 2.0 g of Sodium Hydroxide in 1 mL of a solution of potassium permanganate (3 in 50) and 30 mL of water, neutralize gradually with purified hydrochloric acid and add 5 mL of diluted sulfuric acid (1 in 2). To this solution, add a solution of hydroxylamine hydrochloride (1 in 5) until the precipitate of manganese dioxide disappears, add water to make exactly 100 mL and use this solution as the test solution. Perform the tests according to the Atomic Absorption Spectrophotometry (Cold vapor type) with the test solution. Place the test solution in the test bottle of an atomic absorption spectrophotometer, add 10 mL of stannous chloride-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 indication of the recorder rises rapidly and becomes constant at the wavelength of 253.7 nm. On the other hand, to 2.0 mL of standard mercury solution add 1 mL of a solution of potassium permanganate (3 in 50), 30 mL of water and a volume of purified hydrochloric acid equal to that used in the preparation of the test solution and read the absorbance, A_S of the solution obtained by the same procedure as used for the test solution: A_T is smaller A_S.

(7) ***Lead***—Weigh accurately 5.0 g of Sodium Hydroxide 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 0.25 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 0.5 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp

Wavelength: 228.8 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 gradually adding hydrochloric acid and use this solution as the test solution. Perform the test with the test solution as directed under Arsenic Test (not more than 4 ppm).

Assay Weigh accurately about 1.5 g of Sodium Hydroxide and dissolve in 40 mL of freshly boiled and cooled water. Cool the solution to 15 °C, add 2 drops of phenolphthalein TS and titrate with 0.5 mol/L sulfuric acid until the red color of the solution disappears. Record the amount, *A* (mL), of 0.5 mol/L sulfuric acid consumed. Then add 2 drops of methyl orange TS to the solution and further titrate with 0.5 mol/L sulfuric acid until the solution shows a persistent pale red color. Record the amount, *B* (mL), of 0.5 mol/L sulfuric acid consumed. Calculate the amount of NaOH from the difference, *A* (mL) -*B* (mL).

Each mL of 0.5 mol/L sulfuric acid
= 40.00 mg of NaOH

Containers and Storage ***Containers*—**Tight containers.

Sodium Lauryl Sulfate

Sodium Lauryl Sulfate is a mixture of sodium alkyl sulfate consisting chiefly of sodium lauryl sulfate ($C_{12}H_{25}NaO_4S$: 289.38).

Description Sodium Lauryl Sulfate appears as white to pale yellow crystals or powder, and has a slight, characteristic odor.
Sodium Lauryl Sulfate is sparingly soluble in methanol and in ethanol.
1 g of Sodium Lauryl Sulfate dissolves in 10 mL of water, forming a clear or an opalescent solution, which foams on agitation.

Identification (1) Take 0.2 g of the residue obtained in Total alcohol content, add 4 mL of bromine-cyclohexane TS with vigorous shaking, add 0.3 g of *N*-bromosuccinimide and heat in a water-bath at 80 °C for 5 minutes: a red color develops.

(2) A solution of Sodium Lauryl Sulfate (1 in 10) responds to the Qualitative Tests (1) for sodium salt.

(3) Take a solution of Sodium Lauryl Sulfate (1 in 10), add dilute hydrochloric acid to make acid, boil gently and cool: the solution responds to the Qualitative Tests for sulfate,

Purity (1) ***Alkali***—Dissolve 1.0 g of Sodium Lauryl Sulfate in 100 mL of water, add 2 drops of phenol red TS and 0.60 mL of 0.1 mol/L hydrochloric acid VS: the solution remains yellow.

(2) ***Sodium chloride***—Dissolve about 5 g of Sodium Lauryl Sulfate, accurately weighed, in 50 mL of water, neutralize the solution with dilute nitric acid, if necessary, add 5.0 mL of 0.1 mol/L sodium chloride TS and titrate with 0.1 mol/L silver nitrate VS (indicator: 2 drops of fluorescein sodium TS). Perform a blank determination and make any necessary correction.

Each mL of 0.1 mol/L silver nitrate VS
= 5.844 mg of NaCl

The combined content of sodium chloride (NaCl: 58.44) and sodium sulfate (Na_2SO_4: 142.04) obtained in the next (3) is not more than 8.0 %.

(3) ***Sodium sulfate***—Dissolve about 1 g of Sodium Lauryl Sulfate, accurately weighed, in 10 mL of water, add 100 mL of ethanol and heat at a temperature just below the boiling point for 2 hours. Filter through a glass filter (G4) while hot and wash with 100 mL of boiling ethanol. Dissolve the precipitate by washing with 150 mL of water, collecting the washings in a beaker. Add 10 mL of hydrochloric acid, heat to boiling, add 25 mL of barium chloride TS and allow to stand overnight. Collect the precipitate and wash with water until the last washing shows no opalescence with silver nitrate TS. Dry the precipitate, ignite to a constant weight between 500 °C and 600 °C by raising the temperature gradually and weigh as barium sulfate ($BaSO_4$: 233.39).

Amount (mg) of sodium sulfate (Na_2SO_4)
= amount (mg) of barium sulfate ($BaSO_4$) × 0.6086

(4) ***Unsulfated alcohols***—Dissolve about 10 g of Sodium Lauryl Sulfate, accurately weighed, in 100 mL of water, add 100 mL of ethanol and transfer to a separatory funnel. Extract the solution with three 50 mL volume of petroleum benzin. If an emulsion forms, sodium chloride may be added to promote separation of the two layers. Combined the petroleum benzin extracts and wash with three 50 mL volume of water. Evaporate the petroleum benzin on a water-bath and dry the residue at 105 °C for 30 minutes. The mass of the dried residue is not more than 4.0 % of the mass of the Sodium Lauryl Sulfate taken.

(5) ***Heavy metals***—Proceed with 1.0 g of Sodium Lauryl Sulfate according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 20 ppm).

Water Not more than 5.0 % (0.5 g, volumetric titaration, direct titration).

Total alcohol content Dissolve about 5.0 g of Sodium Lauryl Sulfate, accurately weighed, in 150 mL of water and 50 mL of hydrochloric acid and boil under a reflux condenser for 4 hours. Cool, extract twice with 75 mL volumes of ether and evaporate the combined ether extracts on a water-bath. Dry the residue at 105

°C for 30 minutes: the residue is not less than 59.0 %.

Containers and Storage ***Containers*—**Well-closed containers.

Dibasic Sodium Phosphate Hydrate

$Na_2HPO_4 \cdot 12H_2O$: 358.14

Dibasic Sodium Phosphate Hydrate, when dried, contains not less than 98.0 % and not more than 101.0 % of dibasic sodium phosphate (Na_2HPO_4: 141.96)

Description Dibasic Sodium Phosphate Hydrate appears as colorless or white crystals and is odorless.
Dibasic Sodium Phosphate Hydrate is freely soluble in water and practically insoluble in ethanol or in ether.
Dibasic Sodium Phosphate Hydrate effloresces in warm, dry air.

Identification A solution of Dibasic Sodium Phosphate Hydrate (1 in 10) responds to the Qualitative Tests (1) and (2) for sodium salt and the Qualitative Tests for phosphate.

pH Dissolve 1.0 g of Dibasic Sodium Phosphate Hydrate in 50 mL of water: the pH of this solution is between 9.0 and 9.4.

Purity (1) ***Clarity and color of solution*—**Dissolve 1.0 g of Dibasic Sodium Phosphate Hydrate in 20 mL of water: the 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: not more than 0.2 %.

(3) ***Chloride***—Dissolve 1.0 g of Dibasic Sodium Phosphate Hydrate in 7 mL of dilute nitric acid, add water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution with 0.40 mL of 0.01 mol/L hydrochloric acid (not more than 0.014 %).

(4) ***Sulfate***—Dissolve 0.5 g of Dibasic Sodium Phosphate Hydrate in 2 mL of dilute hydrochloric acid and add water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution with 0.40 mL of 0.005 mol/L sulfuric acid (not more than 0.038 %)

(5) ***Carbonate***—Take 2.0 g of Dibasic Sodium Phosphate Hydrate, add 5 mL of water, boil, cool, and add 2 mL of hydrochloric acid: no foam is produced.

(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. Perform the test using this solution as the test solution. Prepare the control solution with 2.0 mL of standard lead solution by adding 2 mL of dilute acetic acid and water to make 50 mL (not more than 10 ppm).

(7) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat and place 10 to 300 mg of Dibasic Sodium Phosphate Hydrate on top. Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. 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 sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 to 200 ng.

Additive—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.

(8) ***Cadmium***—Weigh accurately 5.0 g of Dibasic Sodium Phosphate Hydrate and transfer to a platinum crucible. Dry, carbonize and incinerate at 450 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 1.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Cadmium hollow cathode lamp
Wavelength: 228.8 nm

(9) ***Lead***—Weigh accurately 5.0 g of Dibasic Sodium Phosphate Hydrate, transfer to a 150 mL beaker and add 30 mL of water. Add hydrochloric acid in small amounts until the test specimen is completely dissolved 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 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 solution (2 in 100) and shake. Extract this solution with two 20 mL volumes of chloroform and evaporate the extract to dryness in 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, transfer 2.0 mL of standard lead 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 Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 4.0 ppm).

Gas: Acetylene or hydrogen – Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(10) ***Arsenic***—Prepare the test solution with 1.0 g of Dibasic Sodium Phosphate Hydrate according to Method 1 and perform the test (not more than 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 (1 in 4) and 10 mL of disodium ethylenediaminetetraacetate (1 in 40) and shake. 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, determine the potential using a fluoride electrode and determine the amount of fluoride from the calibration curve: not more than 10 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 1000 mL 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 (1 in 4) and 10 mL of disodium ethylene-diaminetetraacetate (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), add water to make 100 mL each and use these solutions as the standard solutions. Pipet 50 mL of each standard solution into polyethylene containers. Determine the potential using a fluoride electrode and plot a calibration curve with the log values of the fluoride concentrations.

Loss on drying 57.0 ~ 61.0 % (10 g, at 40 °C for 3 hours at first, then at 105 °C for 5 hours).

Assay Dissolve about 3 g of Dibasic Sodium Phosphate Hydrate, previously dried and accurately weighed, in 50 mL of water. Titrate with 0.5 mol/L sulfuric acid VS keeping at 15°C until the color of the solution changes from green to dark-greenish red-purple (indicator: 3 to 4 drops of methyl orange-xylenecyanol FF TS).

Each mL of 0.5 mol/L sulfuric acid VS
= 141.96 mg of Na_2HPO_4

Containers and Storage ***Containers***—Tight containers.

Dried Sodium Sulfite

Na_2SO_3: 126.04

Dried Sodium Sulfite contains not less than 97.0 % and not more than 101.0 % of sodium sulfite (Na_2SO_3).

Description Dried Sodium Sulfite appears as white crystals or powder and odorless.
Dried Sodium Sulfite is freely soluble in water and practically insoluble in ethanol or in ether.
Dried Sodium Sulfite gradually changes in moist air.

pH—The pH of a solution of Dried Sodium Sulfite (1 in 10) is about 10.

Identification An aqueous solution of Dried Sodium Sulfite (1 in 20) responds to the Qualitative Tests 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 clear and colorless.

(2) ***Thiosulfate***—Dissolve 1.0 g of Dried Sodium Sulfite in 15 mL of water, add gradually 5 mL of hydrochloric acid, shake and allow to stand for 5 minutes: no turbidity is produced.

(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 and perform the test. Prepare the control solution as follows: evaporate 3 mL of hydrochloric acid to dryness and add 2 mL of dilute acetic acid, 1.0 mL of standard lead solution and water to make 50 mL (not more than 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. Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. 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 sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 to 200 ng.

Additive—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 Dried Sodium Sulfite and transfer to a platinum crucible. Dry, carbonize and incinerate at 450 to 550 °C. If incineration is not achieved, 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 (dissolve 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 if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 solution as directed under Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 2.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(6) ***Iron***—To 10.0 g of Dried Sodium Sulfite, add 25 mL of water, shake until almost dissolved, add slowly and carefully 15 mL of hydrochloric acid TS and boil. Cool, add water to make 100 mL and use this solution as the test stock solution. Use 10.0 mL of this solution as the test solution. Separately, to 1 mL of iron standard solution, add water to make 10 mL and use this solution as the standard solution. Prepare before use. To the test solution and the standard solution, add 2 mL of citric acid (2 in 10) and 0.1 mL of thioglycolic acid and 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 (not more than 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, to 1.0 g of Dried Sodium Sulfite, add 20 mL of selenium standard solution and 10 mL of formaldehyde, 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 in a water bath for 20 minutes: the color of the test solution is not more intense than that of the

standard solution (not more than 10 ppm).

(8) ***Zinc***—To 10.0 g of Dried Sodium Sulfite, add 25 mL of water, shake until almost dissolved, add slowly and carefully 15 mL of hydrochloric acid TS and boil. Cool, add water to make 100 mL, pipet 2.0 mL of this solution, add water to make 10 mL and use this solution as the test solution. Separately, dissolve 0.440 g of zinc sulfate ($ZnSO_4 . 7H_2O$) and 1 mL of acetic acid in water to make 100 mL and use this solution as the zinc standard stock solution. Pipet a suitable amount of this solution and dissolve in water to make a solution containing 25 μg per mL. Pipet 1.0 mL, 2.0 mL and 4.0 mL of this solution, add water to make exactly 100 mL so that each mL contains 0.25 μg, 0.5 μg and 1.0 μg of zinc, respectively, and use these solutions as the standard solutions. Perform the test with the test solution and the standard solutions as directed under Atomic Absorption Spectrometry. Determine the absorbances at 213.9 nm of the test solution and the standard solutions and calculate the concentration of zinc according to the Calibration Method (not more than 25 ppm).

(9) ***Arsenic***—Dissolve 0.5 g of Dried Sodium Sulfite in 5 mL of water, add 1 mL of sulfuric acid and evaporate on a sand-bath until white fumes are evolved. Add water to make 5 mL, take this solution as the test solution and perform the test (not more than 4 ppm).

Assay Weigh accurately about 0.2 g of Dried Sodium Sulfite, transfer immediately to an iodine flask containing 50.0 mL of 0.05 mol/L iodine VS, stopper, shake and allow to stand for 5 minutes in a dark place. Add 1 mL of hydrochloric acid and titrate the excess iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 1 mL of starch TS). Perform a blank determination and make any necessary correction.

Each mL of 0.05 mol/L iodine VS
= 6.302 mg of Na_2SO_3.

Containers and Storage ***Containers***—Tight containers.

Sorbitan Sesquioleate

Sorbitan Sesquioleate is a mixture of monoester and diester of sorbitol anhydride, partially esterified with oleic acid.

Description Sorbitan Sesquioleate is pale yellow to pale yellow-brown, viscous oily liquid, has faint, characteristic odor and slightly bitter taste.
Sorbitan Sesquioleate is freely soluble in ether, slightly soluble in ethanol and very slightly soluble in methanol.
Sesquioleate is dispersed as fine oily drops in water.

Identification (1) Take 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. Cool, shake with 5 mL of petroleum ether, allow to stand and separate the upper layer and the lower layer. Shake 2 mL of the lower layer with 2 mL of freshly prepared catechol solution (1 in 10), then with 5 mL of sulfuric acid: a red to red-brown color develops.

(2) Heat the upper layer obtained in (1) on a water-bath and 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 between 30 °C and 35 °C with stirring: the solution develops an opalescence and, when cooled, crystals are formed.

Saponification Value 150 ~ 168.

Specific Gravity d_{25}^{25} : 0.960 ~ 1.020.

Purity (1) ***Acid***—Take 2.0 g of Sorbitan Sesquioleate, add 50 mL of neutralized ethanol and heat on a water-bath nearly to boiling with stirring once or twice. Cool, add 4.3 mL of 0.1 mol/L sodium hydroxide VS and 5 drops of phenolphthalein TS: a red color develops.

(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 standard lead solution (not more than 10 ppm).

(3) ***Arsenic***—Prepare the test solution with 1.0 g of Sorbitan Sesquioleate according to Method 2 and perform the test (not more than 2 ppm).

Water Not more than 3.0 % (1 g, volumetric titration, direct titration, stir for 30 minutes).

Residue on Ignition Not more than 1.0 % (1 g).

Containers and Storage ***Containers***—Tight containers.

Soybean Oil

Oleum Sojae

Soybean Oil is the fixed oil obtained from the seeds of *Glycine max* Merrill (Leguminosae).

Description Soybean Oil is clear, pale yellow oil, odorless or has a slight odor and bland taste.
Soybean Oil is miscible with ether or with carbon tetrachloride.
Soybean Oil is slightly soluble in ethanol and practically insoluble in water.
Soybean Oil congeals between -10 and -17 °C.

Congealing point of the fatty acids—22 ~ 27 °C.

Saponification Value 188 ~ 195.

Unsaponifiable Matter Not more than 1.0 %.

Specific Gravity d_{25}^{25} : 0.916 ~ 0.922.

Acid Value Not more than 0.2.

Iodine Value 126 ~ 140.

Purity (1) ***Free fatty acid***—The free fatty acids in 10 g of Soybean Oil require for neutralization not more than 2.5 mL of 0.020 mol/L sodium hydroxide.

(2) ***Peroxide value***—Weigh accurately 10 g of Soybean Oil, transfer to a stoppered conical 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, shake for exactly for 1 minute and add 30 mL of water. Titrate with 0.01 mol/L sodium thiosulfate VS until the blue color of the solution disappears after addition of 0.5 mL of starch TS near the end point where the solution is a pale yellow color. Perform a blank determination and make any necessary correction. Calculate the amount of peroxide by the following formula: not more than 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 test

V_0: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed in the blank

W: Amount (g) of Soybean Oil taken

(3) ***Heavy metals***—Proceed with 2.0 g of Soybean Oil according to Method 2 under Heavy Metals Limit Test and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 10 ppm).

Containers and Storage ***Containers*—**Tight containers.

Stearic Acid

Stearic Acid is a solid fatty acid obtained from fats and consists chiefly of stearic acid ($C_{18}H_{36}O_2$: 248.48) and palmitic acid ($C_{16}H_{32}O_2$: 256.42).

Description Stearic Acid appears as white, unctuous or crystalline masses or powder and has a faint, fatty odor.

Stearic Acid is freely soluble in ether, soluble in ethanol and practically insoluble in water.

Melting point—56 ~ 72 °C (Method 2).

Acid Value 194 ~ 210.

Iodine Value Not more than 4.0.

Saponification Value 197 ~ 212. Weigh accurately 3 g of Stearic Acid, transfer to a 250 mL flask and perform the test as directed in the Saponification value under Fats and Fatty Oils.

Unsaponifiable Matter Not more than 1.5 %.

Water Not more than 0.2 % (volumetric titration, direct titration)

Purity (1) ***Mineral acid*—**Melt 5 g of Stearic Acid by warming, shake with 5 mL of boiling water for 2 minutes, filter after cooling and add 1 drop of methyl orange TS to the filtrate: no red color develops.

(2) ***Heavy metals*—**Proceed with 1.0 g of Stearic Acid according to Method 2 and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 20 ppm).

(3) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat and place 10 mg to 300 mg of Stearic Acid on top. Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. Place the boat inside the furnace and heat to about 900 °C with a current of air or oxygen at 0.5 L/minute to 1 L/minute. Elute the mercury and sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 ng to 200 ng.

Additive—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 Stearic Acid 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 mL 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 supplement, dry and continue with incineration. If incineration is incomplete, repeat the above procedure once and if necessary, add a final 2 mL to 5 mL of nitric acid (1 in 2) 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 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 0.5 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 solution as directed under Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 1.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(5) ***Arsenic***—Proceed with 0.5 g of Stearic Acid according to Method 3 and perform the test (not more than 4 ppm).

(6) ***Fat and paraffin—***Boil 1.0 g of Stearic Acid with 0.5 g of anhydrous sodium carbonate and 30 mL of water: the solution, while hot, is clear or not more turbid than the following control solution.

Control solution—Take 0.70 mL of 0.01 mol/L hydrochloric acid, add 6 mL of dilute nitric acid and water to make 30 mL and add 1 mL of silver nitrate TS.

Residue on Ignition Not more than 0.1 % (1 g).

Containers and Storage ***Containers—***Well-closed containers

Stearyl Alcohol

Stearyl Alcohol is a mixture of solid alcohols and consists chiefly of stearyl alcohol ($C_{18}H_{38}O$: 270.49).

Description Stearyl Alcohol is a white, unctuous matter and has a faint, characteristic odor and is tasteless.
Stearyl Alcohol is freely soluble in ethanol, in dehydrated ethanol or in ether and practically insoluble in water.

Melting Point 56 ~ 62 °C. Prepare the sample according to Method 2, then attach tightly a capillary tube to the bottom of the thermometer by means of a rubber band or by any suitable means, and make the bottom of the capillary tube equal in position to 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 until the temperature rises to 5°C below the expected melting point. Then regulate the rate of increase to 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 Not more than 1.0.

Hydroxyl Value 200 ~ 220.

Ester Value Not more than 3.0.

Iodine Value Not more than 2.0.

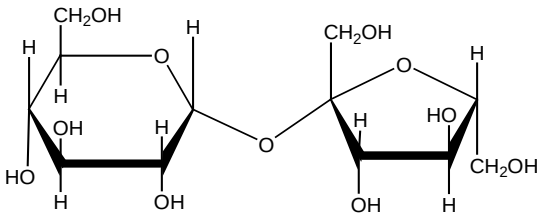
Purity (1) ***Clarity and color of solution—***Dissolve 3.0 g of Stearyl Alcohol in 25 mL of dehydrated ethanol by warming: the solution is clear.

(2) ***Alkali—***Take the solution obtained in (1) and add 2 drops of phenolphthalein TS: no red color develops.

Residue on Ignition Not more than 0.05 % (2 g).

Containers and Storage ***Containers—***Well-closed containers.

Sucrose



$C_{12}H_{22}O_{11}$: 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-(hydroxymethyl)oxane-3,4,5-triol [*57-50-1*]

Description Sucrose appears as white crystalline powder, or lustrous colorless or white crystals, is odorless and has a sweet taste.
Sucrose is very soluble in water, very slightly soluble in ethanol and practically insoluble in ether.
A solution of Sucrose (1 in 10) is neutral.

Identification (1) When 1 g of Sucrose is ignited, it melts and swells and decomposes, emitting an odor of caramel, to bulky charcoal.
(2) Take 0.1 g of Sucrose, 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: red to dark red precipitate is produced.

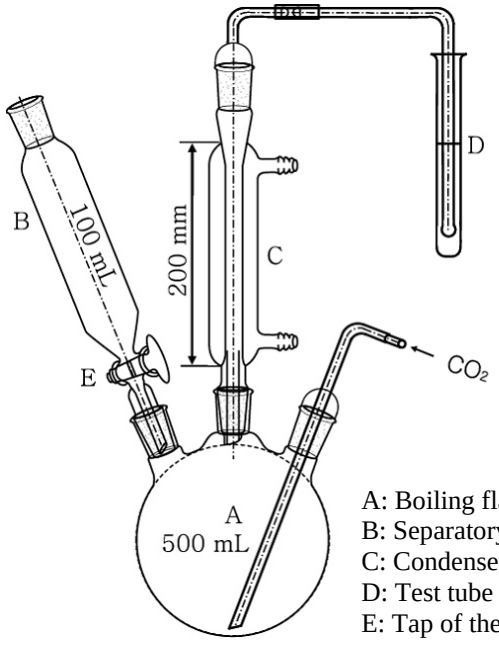
Specific Optical Rotation $[\alpha]_D^{20}$: +65.0 ~ +67.0° (after drying, 13 g, 50 mL of water, 100 mm).

Purity (1) ***Clarity and color of solution***—Dissolve 100 g of Sucrose in 100 mL of water, take 50 mL of this solution in a Nessler tube and view transversely the Nessler tube against a white background: the solution is colorless or slightly yellow and has blue color. Fill the solution in the Nessler tube, stopper and allow to stand for 2 days: no precipitate is produced.

(2) ***Chloride***—Take 10.0 g of sucrose and add 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. Perform the. Prepare the control solution with 0.30 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.005 %).

(3) ***Sulfate***—Take 40 mL of the test solution obtained in (2), add 1 mL of dilute hydrochloric acid and water to make 50 mL. Perform the test. Prepare the control solution with 0.50 mL of 0.005 mol/L sulfuric acid VS (not more than 0.006 %).

(4) ***Sulfur dioxide***—(i) Apparatus: Use apparatus shown in the figure.



A: Boiling flask
B: Separatory funnel
C: Condenser
D: Test tube
E: Tap of the funnel

(ii) Procedure: Introduce 150 mL of water into the boiling flask, close the tap of the funnel, and pass carbon dioxide through the whole system at a rate of 100 ± 5 mL per minute. Pass cooling water through the condenser, and place 10 mL of hydrogen peroxide-sodium hydroxide TS (to a 9 : 1 mixture of water and hydrogen peroxide (hydrogen peroxide TS), add 3 drops of bromophenol blue TS and add 0.01 mol/L sodium hydroxide TS until the color changes to purple-blue; prepare before use) in the test-tube. After 15 minutes, remove the funnel without interrupting the stream of carbon dioxide, and introduce through the opening into the flask about 25 g of Sucrose, accurately weighed, with the aid of 100 mL of water. Apply tap grease to the outside of the connection part of the funnel, and connect the funnel. Close the tap of the funnel, pour 80 mL of 2 mol/L hydrochloric acid TS into the funnel, open the tap to introduce the hydrochloric acid into the flask, and close the tap while several mL of the hydrochloric acid remains, in order to avoid losing sulfur dioxide. Place the flask in a water-bath, and heat the mixture for 1 hour. Transfer the contents of the test-tube with the aid of a little water to a wide-necked conical flask. Heat in a water-bath for 10 minutes, and cool. Add 0.1 mL of bromophenol blue TS, and titrate with 0.1 mol/L sodium hydroxide VS until the color changes from yellow to purple-blue lasting for at least 20 seconds. Perform a blank determination and make any necessary correction. Calculate the amount of sulfur dioxide by applying the following formula: it is not more than 20 ppm.

$$\text{Sulfur dioxide (ppm)} = \frac{\text{Amount (mL) of 0.1 mol/L sodium hydroxide consumed}}{\text{Amount (g) of Sucrose taken}} \times 1000 \times 3.203$$

(5) ***Calcium***—Take 10 mL of the test solution obtained in (2) and add 1 mL of ammonium oxalate TS: this solution shows immediately no change.

(6) ***Heavy metals***—Proceed with 5.0 g of Sucrose according to Method 1 and perform the test. Prepare the control solution with 2.5 mL of standard lead solution (not more than 5 ppm).

(7) ***Lead***——Put exactly 50 mg of Sucrose in a polytetrafuruoroethylene decomposition-vessel, add 0.5 mL of nitric acid to dissolve, seal up the vessel, and heat a 150 °C for 5 hours. After cooling, add water to make exactly 5 mL, and use this solution as the test solution. Perform the test with more than 3 parts of the test solution as directed in the standard addition method under Atomic Absorption Spectrophotometry (electrothermal type) according to the following conditions. The standard solution is prepared by adding water to a suitable volume of Standard Lead Solution exactly volumed, and perform a blank determination with a solution prepared by adding water to 10.0 mL of nitric acid to make exactly 100 mL, and make any necessary correction (not more than 0.5 ppm).

Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm
Temperature for drying: 110 °C
Temperature for incineration: 600 °C

Temperature for atomization: 2100 °C

(8) ***Arsenic*—**Prepare the test solution with 1.0 g of Sucrose according to Method 1 and perform the test (not more than 2 ppm).

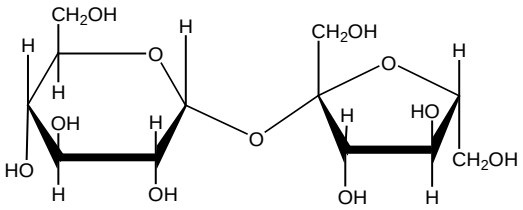
(9) ***Invert sugar*—**Dissolve 5.0 g of Sucrose in water to make 100 mL and filter, if necessary and use this solution as the test solution. Separately, place 100 mL of alkaline cupric sulfate TS in a 300 mL beaker, cover the beaker with a watch glass and boil. Immediately add 50 mL of the test solution, boil the mixture exactly for 5 minutes, add at once 50 mL of freshly boiled and cooled water, dip in a water-bath of a temperature below 10 °C for 5 minutes and collect the precipitate in a tared glass filter (G4). Wash the residue on the filter with water until the last washing is neutral, then wash with 10 mL of ethanol, add 10 mL of ether and dry at 105 °C for 30 minutes: the residual precipitate is not more than 0.120 g.

Loss on Drying Not more than 1.30 % (15 g, 105 °C, 2 hours).

Residue on Ignition Not more than 0.10 % (2 g).

Containers and Storage ***Containers*—**Well-closed containers.

Purified Sucrose



$C_{12}H_{22}O_{11}$: 342.30

Purified Sucrose contains no additives. For Purified Sucrose used for preparation of the large volume infusions, the label states the purpose.

Description Purified Sucrose appears as white crystalline powder, or lustrous colorless or white crystals.
Purified Sucrose is very soluble in water and slightly soluble in ethanol.

Identification (1) Take 10 mg each of Purified Sucrose and white soft sugar, add diluted methanol (3 in 5) to make 20 mL each and use these solutions as the test solution and the standard solution (1), respectively. Separately, to 10 mg each of glucose, lactose, fructose and white soft sugar, add methanol (3 in 5) to make 20 mL and use this solution as the standard solutions (1) and (2). Perform the test with the test solution and the solutions as directed under the Thin-layer Chromatography. Spot 2 μL each of the test solution and the standard solutions (1) and (2) on a plate of silica gel for thin-layer chromatography and dry the plate completely. Develop the plate with a mixture of 1,2-dichloroethane, acetic acid (100), methanol and water (10 : 5 : 3 : 2) to a distance of about 15 cm and dry the plate with a hot air. And immediately repeat the development with replaced developing mixture and dry the plate in the same way. Spray evenly a solution of 0.5 g of thymol in 100 mL off mixture of ethanol and sulfuric acid (19 : 1), heat at 130 °C for 10 minutes: the principal spot from the test solution is the same with the principal spot from the standard solution (1) in the R_f, color and size and four spots from the standard solution (2) are apparently distinguishable.

(2) Dissolve 50.0 g of Purified Sucrose in recently boiled and cooled water to make 100 mL and use this solution as the test solution. To 1 mL of the test solution, add water to make 100 mL, then to 5 mL of this solution add 0.15 mL of freshly prepared cupric sulfate TS and 2 mL of freshly prepared 2 mol/L sodium hydroxide TS: the solution is clear and blue and not changes on boiling. Then to this solution, add 4 mL of dilute hydrochloric acid, boil and add 4 mL of 2 mol/L sodium hydroxide TS: orange precipitates are immediately produced.

Specific Optical Rotation $[\alpha]_D^{20}$: +66.3 ~ +67.0 ° (26.0 g, water, 100 mL, 100 mm).

Purity (1) ***Clarity and color of solution***—The test solution obtained in the Identification (2) is clear and is not more intense than the following control solution.

Control solution—Take 2.4 mL of ferric chloride colorimetric stock solution and 0.6 mL of cobaltous chloride colorimetric stock solution add 7.0 mL of diluted hydrochloric acid (7 in 250). To 5.0 mL of this solution, add 95.0 mL of diluted hydrochloric acid (7 in 250).

(2) ***Acid or alkali***—Take 10 mL of the test solution obtained in the Identification (2), add 0.3 mL of phenolphthalein TS: the solution is colorless and develops a red color on addition of 0.3 mL of 0.01 mol/ 1 sodium hydroxide VS.

(3) ***Sulfite***—Dissolve 5.0 g of Sucrose in 40 mL of water, add 2.0 mL of dilute sodium hydroxide TS and water to make exactly 50 mL and use this solution as the test solution. Separately, dissolve 76 mg of sodium metabisulfate in water to make exactly 50 mL, then pipet 5.0 mL of this solution, add water to make exactly 100 mL. Pipet 3.0 mL of this solution, add 4.0 mL of dilute sodium hydroxide TS and water to make exactly 100 mL and use this solution as the standard solution. Immediately, pipet 10.0 mL each of the test solution and the standard solution, add 1.0 mL of 3 mol/L hydrochloric acid, 2.0 mL of decolorized fuchsin TS and 2.0 mL of formalin TS and allow to stand for 30 minutes. Determine the absorbance at 583 nm of the test solution and the standard solution as directed under

the Ultraviolet-visible Spectrophotometry using the control solution obtained by proceeding with 10.0 mL of water in the same manner as above, the absorbance of the test solution is not larger than that of the standard solution (not more than 15 ppm as SO_2). When the standard solution does not show a red color, result of the left is invalid.

(4) ***Lead***—Take 50.0 mg of Purified Sucrose in a polytetrafuruoroethylene decomposition-vessel, add 0.5 mL of nitric acid to dissolve, seal up the vessel 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. Perform the test with more than 3 parts of the test solution as directed in the standard addition method under the Atomic Absorption Spectrophotometry (electrothermal type) according to the following operating conditions. The standard solution is prepared by adding water to a suitable volume of standard lead solution exactly and perform a blank determination with a solution prepared by adding water to 10.0 mL of nitric acid to make exactly 100 mL and make any necessary correction (not more than 0.5 ppm).

Lamp: A hollow cathode lamp.
Wavelength: 283.3 nm.
Temperature for drying: 110 °C.
Temperature for incineration: 600 °C.
Temperature for atomization: 2100 °C.

(5) ***Invert sugar***—Transfer 5 mL of the test solution obtained in the Identification (2) to a test-tube, about 150 mm in length and about 16 mm in diameter, add 5 mL of water, 1.0 mL of 1 mol/L sodium hydroxide TS and 1.0 mL of methylene blue TS, mix and place in a water-bath. After exactly 2 minutes, take the tube out of the bath and examine the solution immediately: the blue color does not disappear completely (0.04 %). Ignore any blue color at the air and solution interface.

Conductivity (1) ***Potassium chloride conductivity calibration standard solution***—Dissolve powdered potassium chloride, previously dried at 500 °C to 600 °C for 4 hours, in newly distillated water having less conductivity than 2 $\mu S \cdot cm^{-1}$ to get three kinds of the standard solution containing 0.7455 g, 0.0746 g and 0.0149 g of potassium chloride in 1000.0 g, respectively. The conductivities of these solutions at 20 °C are shown in the following table.

Standard solution (g/1000.0 g)	Conductivity ($\mu S \cdot cm^{-1}$)	Resistivity ($\Omega \cdot cm$)
0.7455	1330	752
0.0746	133.0	7519
0.0149	26.0	37594

(2) ***Apparatus***—Use an appropriate conductivity meter. The conductivity is determined to measure the electrical resistance of the column of liquid between the electrodes of the immersed measuring device (conductivity cell). The apparatus is supplied with alternative current to avoid the effects of electrode polarization. It is usually equipped with a temperature compensation device. The conductivity cell contains of two parallel platinum electrodes coated with platinum black and both electrodes are generally protected by a glass tube which allows good exchange between the solution and the electrodes. Use a cell giving the cell constant of 0.01 cm^{-1} to 1 cm^{-1}.

(3) ***Procedure***—Use the suitable potassium chloride conductivity calibration standard solution to the measurement. After washing the well with water, rinse 2 to 3 times with the calibration standard solution, kept up the cell with the calibration standard solution and determine the conductivity of the calibration standard solution kept at 20 ± 0.1 °C. Repeat the determination and measure the conductivity of the calibration standard solution, G_{x_0} (μS), after a stable reading of ± 3 % is obtained. The cell constant, J, is calculated by the following equation.

$$J = \frac{x_{\mathrm{KCl}}}{G_{x_0}}$$

J : Cell constant (cm^{-1}),
x_{KCl} : Conductivity constant of the potassium chloride conductivity calibration standard solution ($\mu S \cdot cm^{-1}$) (20 °C),
G_{x_0} : Conductivity measured (μS).

Dissolve 31.3 g of Purified Sucrose in newly distillated water to make exactly 100 mL and use this solution as the test solution. After washing well the cell with water, rinse the cell with the test solution 2 to 3 times, fill up with the test solution and determine the conductivity of the test solution, G_T (μS), kept at 20 ± 0.1 °C, while stirring. Determine the conductivity of the water used for preparation of the test solution, G_0 (μS), in the same manner as above and calculate the conductivity, x_T ($\mu S \cdot cm^{-1}$) and x_0 ($\mu S \cdot cm^{-1}$), by the following expressions.

$$x_T\,(\mu S \cdot cm^{-1}) = JG_T$$
$$x_0\,(\mu S \cdot cm^{-1}) = JG_0$$

Determine the corrected conductivity, x_C, of the test solution by the following equation: not more than 35 $\mu S \cdot cm^{-1}$.

$$x_C\,(\mu S \cdot cm^{-1}) = x_T - 0.35\,x_0$$

Loss on Drying Not more than 0.1 % (2 g, 105 °C, 3 hours).

Dextrins For Sucrose used to prepare large volume aqueous infusions, to 2 mL of the test solution obtained

in the Identification (2), add 8 mL of water, 0.05 mL of dilute hydrochloric acid and 0.05 mL of iodine TS: the solution remains yellow.

Bacterial Endotoxins Less than 0.25 EU/mg of Purified Sucrose when Purified Sucrose is used to prepare large volume aqueous infusions.

Containers and Storage ***Containers*—**Well-closed containers.

Talc

Talc is a native, hydrous magnesium silicate, sometimes containing a small volume of aluminum silicate.

Description Talc is white to grayish white, fine, crystalline powder, is odorless and tasteless.
Talc is unctuous and adheres readily to the skin.
Talc is practically insoluble in water, in ethanol or in ether.

Identification (1) Mix 0.2 g of Talc with 0.9 g of anhydrous sodium carbonate and 1.3 g of anhydrous potassium carbonate and heat the mixture in a platinum or nickel crucible until fusion is complete. Cool and transfer the fused mixture to a beaker with the aid of 50 mL of hot water. Add hydrochloric acid until it ceases to cause effervescence, add 10 mL of hydrochloric acid and evaporate the mixture on a water-bath to dryness. Cool, add 20 mL of water, boil and filter. Add 10 mL of a solution of methylene blue (1 in 1000) to the residue and wash with water: the precipitate is blue.

(2) Dissolve 2 g of ammonium chloride and 5 mL of ammonia TS in the filtrate obtained in (1), filter, if necessary and add disodium hydrogen phosphate TS: a white, crystalline precipitate is produced.

(3) Determine the infrared spectrum of Talc as directed in the potassium bromide disk method under Infrared Spectrophotometry: it exhibits absorption at the wavenumbers between 3675 cm^{-1} and 3679 cm^{-1}, 1016 cm^{-1} and 1020 cm^{-1} and between 667 cm^{-1} and 671 cm^{-1}.

Purity (1) ***Acid-soluble substances*—**Weigh accurately about 1 g of Talc, heat with 20 mL of dilute hydrochloric acid at 50 °C for 15 minutes with stirring. Cool, add water to make exactly 50 mL and filter. Centrifuge, if necessary, until the filtrate becomes clear. To 25 mL of this filtrate, add 1 mL of dilute sulfuric acid, evaporate to dryness and ignite to a constant weight at 800 ± 25 °C: the residue is not more than 2.0 %.

(2) ***Acid or alkali and water-soluble substances*—**Take 10.0 g of Talc, add 50 mL of water, weigh and boil for 30minutes, supplying water lost by evaporation. Cool, add water to restore the original mass and filter. Centrifuge, if necessary, until the filtrate becomes clear: the filtrate is neutral. Evaporate 20 mL of the filtrate to dryness and dry the residue at 105 °C for 1 hours: the residue is not more than 4.0 mg

(3) ***Lead***—Weigh 10.0 g of Talc, add slowly 50 mL of 0.5 mol/L hydrochloric acid TS while stirring and heat with a reflux condenser on a water bath for 30 minutes. After cooling, transfer the solution to a beaker and allow to settle. Filter the clear supernatant liquid, retaining as much as possible of the settled precipitate in the beaker. Wash the beaker and precipitate with three 10 mL volumes of hot water, filter and wash the filter paper with 15 mL of hot water. Cool the filtrate, add 100 mL of water and use this solution as the test solution. Separately, put 5.0 mL, 7.5 mL, 10.0 mL and 12.5 mL of standard lead solution into 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 Atomic Absorption Spectrophotometry according to the following operating conditions and determine the content of lead in the test solution: not more than 0.001 %.

Gas: Acetylene – Air
Lamp: Lead hollow cathode lamp
Wavelength: 217.0 nm

(4) ***Aluminum***—[Perchlorates mixed with heavy metals are explosive. Take proper precautions while performing this procedure.] Weigh 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. Stir gently, add 35 mL of hydrofluoric acid and slowly evaporate to dryness until about 0.5 mL remains. To the residue, add 5 mL of hydrochloric acid, cover with a watch glass, heat to dissolve and cool. Transfer to a 50 mL volumetric flask, wash the polytetrafluoroethylene dish and the watch glass with water, combine the washings and add water to make 50 mL. To 5 mL of this solution, add 10 mL of cesium chloride TS and 10 mL of hydrochloric acid, 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. Immediately before use, take 10 mL of this solution, add water to make 100 mL and use this solution as the aluminum standard stock solution. This solution contains 100 μg of aluminum per mL. Put 5.0 mL, 10.0 mL, 15.0 mL and 20.0 mL of the aluminum standard stock solution into volumetric flasks, each containing 10 mL of hydrochloric acid and 10 mL of cesium chloride, 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 Atomic Absorption Spectrophotometry according to the following operating conditions and determine the content of aluminum in the test solution: not more than 2.0 %.

Gas: Acetylene – Nitrous oxide

Lamp: Aluminum hollow cathode lamp
Wavelength: 309.3 nm

Cesium chloride TS—To 2.53 g of cesium chloride, add water to make 100 mL.

(5) ***Iron***—Weigh 10.0 g of Talc, add slowly 50 mL of 0.5 mol/L hydrochloric acid TS while stirring and heat with a reflux condenser on a water bath for 30 minutes. After cooling, transfer the solution to a beaker and allow to settle. Filter the clear supernatant liquid, retaining as much as possible of the settled precipitate in the beaker. Wash the beaker and precipitate with three 10 mL volumes 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. To 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 iron (II) chloride in hydrochloric acid (150 in 1000) so that each mL contains 250 μg of iron (Fe) and use this solution as the iron standard stock solution. Prepare before use. Put 2.0 mL, 2.5 mL, 3.0 mL and 4.0 mL of the iron standard stock solution into 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 Atomic Absorption Spectrophotometry and determine the content of iron in the test solution: not more than 0.25 %.

Gas: Acetylene – Air
Lamp: Iron hollow cathode lamp
Wavelength: 248.3 nm

(6) ***Calcium***—[Perchlorates mixed with heavy metals are explosive. Take proper precautions while performing this procedure.] Weigh 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. Stir gently, add 35 mL of hydrofluoric acid and slowly evaporate to dryness until about 0.5 mL remains. To the residue, add 5 mL of hydrochloric acid, cover with a watch glass, heat to dissolve and cool. Transfer to a 50 mL volumetric flask, wash the polytetrafluoroethylene dish and the watch glass with water, combine the washings and add water to make 50 mL. To 5.0 mL of this solution, add 10 mL of hydrochloric acid and 10 mL of lanthanum chloride TS, add water to make 100 mL and use this solution as the test solution. Separately, dissolve 3.67 g of calcium chloride dihydrate in dilute hydrochloric acid to make 1000 mL. Immediately before use, pipet 10 mL of this solution, add water to make 100 mL and use this solution as the calcium standard stock solution. This solution contains 100 μg of calcium per mL. Put 1.0 mL, 2.0 mL, 3.0 mL and 4.0 mL of the calcium standard stock solution into 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 Atomic Absorption Spectrophotometry according to the following operating conditions and determine the content of calcium in the test solution: not more than 0.9 %.

Gas: Acetylene – nitrous oxide
Lamp: Calcium hollow cathode lamp
Wavelength: 422.7 nm

Lanthanum chloride TS—To 5.9 g of lanthanum oxide, add slowly 10 mL of hydrochloric acid and heat. After cooling, add water to make 100 mL.

(7) ***Arsenic***—Take 0.5 g of Talc, add 5 mL of dilute sulfuric acid and heat gently to boiling with shaking. Cool immediately, filter and wash the residue with 5 mL of dilute sulfuric acid, then with 10 mL of water. Combine the filtrate and the washing, evaporate to 5 mL on a water-bath and perform the test (not more than 4 ppm).

(8) ***Asbestos***—Proceed as directed in the following (i) and (ii): no asbestos is detected. If either test is positive, proceed as directed in (i): no asbestos is detected.

(i) Determine the infrared spectrum of Talc as directed in the potassium bromide disk method under Infrared Spectrophotometry: Check absorption at the wavenumbers between 757 cm^{-1} and 759 cm^{-1} (amphibole asbestos) or between 600 cm^{-1} and 650 cm^{-1} (serpentine asbestos). If an absorption peak occurs between 757 cm^{-1} and 759 cm^{-1}, weigh an amount of the test specimen, ignite at 850 °C for not less than 30 minutes and cool. Determine the infrared spectrum again and check the absorption peak at the wavenumbers between between 757 cm^{-1} and 759 cm^{-1}, which indicates the presence of tremolite among amphibole asbestos.

(ii) Measure the powder diffraction of Talc as directed under X-Ray Powder Diffraction Determination according to the following operating conditions: check the diffraction peaks where the diffraction angle 2θ is 10.4 ~ 10.6 ° (amphibole asbestos), and 24.2 ~ 24.4 ° and 12.0 ~ 12.2 ° (serpentine white asbestos).

Operating conditions
X-ray light source: Cu Kα monochrometer
Tube current and voltage: 24 ~ 30 mA, 40 kV
Incident angle: 1 °
Detection slit: 0.2 °
Scan speed: 0.1 °/minute
Scan range (diffraction angle 2θ): 10 ~ 13 °, 24 ~ 26 °

(iii) Examine Talc under an optical microscope for the shape, color, etc. of asbestos. The following characteristics indicate the presence of asbestos.
① Length to width ratio of fibers is within a range of 20 : 1 to 100 : 1, or not less than 100 : 1 for fibers longer than 5 μm.

② There is a capability of splitting into very thin fibrils.
③ There are two or more of the following four characteristics.

(i) Parallel fibers occurring in bundles
(ii) Fiber bundles displaying frayed ends
(iii) Fibers in the form of thin needles
(iv) Matted masses of individual fibers and/or fibers showing curvature

Loss on Ignition Not more than 5.0 % (1 g, between 450 °C and 550 °C, 3 hours).

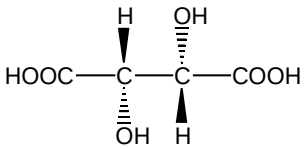
Microbial Limit The total aerobic microbial count is not more than 1000 CFU/g, the total combined yeasts/mould count is not more than 100 CFU/g and *Escherichia coli*, *Salmonella* species, *Staphylococcus aureus* and *Pseudomonas aeruginosa* are not observed.

Magnesium Content [Perchlorates mixed with heavy metals are explosive. Take proper precautions while performing this procedure.] Weigh 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. Stir gently, add 35 mL of hydrofluoric acid and slowly evaporate to dryness until about 0.5 mL remains. To the residue, add 5 mL of hydrochloric acid, cover with a watch glass, heat to dissolve and cool. Transfer to a 50 mL volumetric flask, wash the polytetrafluoroethylene dish and the watch glass with water, combine the washings and add water to make 50 mL. To 0.5 mL of this solution, add water to make 100 mL. To 4.0 mL of this solution, add 10 mL of hydrochloric acid and 10 mL of lanthanum chloride TS, add water to make 100 mL and use this solution as the test solution. Separately, dissolve 8.365 g of magnesium chloride in dilute hydrochloric acid to make 1000 mL. To 5 mL of this solution, add water to make 500 mL and use this solution as the magnesium standard stock solution. This solution contains 10 μg of magnesium per mL. Put 2.5 mL, 3.0 mL, 4.0 mL and 5.0 mL of the magnesium standard solution into volumetric flasks, each containing 10 mL of hydrochloric acid and 10 mL of lanthanum chloride, 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 Atomic Absorption Spectrophotometry according to the following operating conditions and determine the content of magnesium in the test solution: not more than 17.0 % and not more than 19.5 %.

Gas: Acetylene – Air
Lamp: Magnesium hollow cathode lamp
Wavelength: 285.2 nm

Containers and Storage ***Containers*—**Well-closed containers.

Tartaric Acid



$C_4H_6O_6$: 150.09

(2*R*,3*R*)-2,3-Dihydroxybutanedioic acid [*87-69-4*]

Tartaric Acid, when dried, contains not less than 99.7 % and not more than 101.0 % of tartaric acid ($C_4H_6O_6$).

Description Tartaric Acid appears as colorless crystals or white, crystalline powder, is odorless and has strong acid taste.
Tartaric Acid is very soluble in water, freely soluble in ethanol and slightly soluble in ether.
A solution of Tartaric Acid (1 in 10) is dextrorotatory.

Identification (1) Ignite Tartaric Acid gradually: it decomposes and an odor of burning sugar is perceptible.

(2) A solution of Tartaric Acid (1 in 10) changes blue litmus paper to red and responds to the Qualitative Tests 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 (not more than 0.048 %).

(2) ***Oxalate***—Dissolve 1.0 g of Tartaric Acid in 10 mL of water and 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 standard lead solution (not more than 10 ppm).

(4) ***Mercury***—Spread evenly about 1 g of additive (a) into a ceramic boat and place 10 to 300 mg of Tartaric Acid on top. Spread evenly about 0.5 g of additive (a) and 1 g of additive (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 test specimen in a nickel boat without the additives. 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 sample 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 additives in a ceramic 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. Substitute the value of A – Ab into the calibration curve to calculate the amount of mercury in the test specimen (not more than 1.0 ppm).

Operating conditions

Analyzer: Use a mercury analyzer automated from specimen combustion to gold amalgam sampling and cold vapor atomic absorption spectrometry. A mercury analyzer equipped with a separate catalyst in the combustion chamber may be used.

Mercury standard 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 mercury standard stock solution with 0.001 % L-cysteine so that each mL contains 0 to 200 ng.

Additive—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 Tartaric Acid and transfer to a platinum crucible. Dry, carbonize and incinerate at 450 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 supplement, 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, 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 solution as directed under Atomic Absorption Spectrophotometry according to the following operating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 2.0 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp
Wavelength: 283.3 nm

(6) ***Calcium***—Neutralize a solution of 1.0 g of Tartaric Acid in 10 mL of water with ammonia TS and add 1 mL of ammonium oxalate TS: no turbidity is produced.

(7) ***Arsenic***—Prepare the test solution with 2.0 g of Tartaric Acid according to Method 1 and perform the test (not more than 1 ppm).

Specific Optical Rotation $[\alpha]_D^{20}$: +12 ~ +13° (2 g, water, 10 mL, 100 mm)

Loss on Drying Not more than 0.5 % (3 g, silica gel, 3 hours).

Residue on Ignition Not more than 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 $C_4H_6O_6$

Containers and Storage ***Containers*—**Well-closed containers.

Titanium Oxide

TiO_2: 79.87

Titanium Oxide, when dried, contains not less than 98.5 % and not more than 101.0 % of titanium oxide (TiO_2).

Description Titanium Oxide is a white powder, odorless and tasteless.
Titanium Oxide is practically insoluble in water, in dehydrated ethanol or in ether.
Titanium Oxide dissolves in hot sulfuric acid or in hydrofluoric acid and does not dissolve in hydrochloric acid, in nitric acid or in dilute sulfuric acid.
When fused by heating with potassium bisulfate, with potassium hydroxide, or with potassium carbonate, it changes to soluble salts.
Shake 1 g of Titanium Oxide with 10 mL of water: the mixture is neutral.

Identification Heat 0.5 g of Titanium Oxide with 5 mL of sulfuric acid until white fumes are evolved, cool, add cautiously water to make 100 mL and filter. To 5 mL of the filtrate, add 2 to 3 drops of hydrogen peroxide TS: a yellow-red color develops.

Purity (1) ***Lead***—Place 1.0 g of Titanium Oxide in a platinum crucible, add 10.0 g of potassium bisulfate, heat gently with caution at the beginning, then raise the temperature gradually and heat strongly with occasional shaking until the contents fuse to yield a clear liquid. Cool, add 30 mL of a solution of ammonium citrate (9 in 20) and 50 mL of water, dissolve by heating on a water-bath, cool, add water to make 100 mL and use this solution as the test stock solution. Take 25 mL of the solution to a separatory funnel, add 10 mL of a solution of ammonium sulfate (2 in 5) and 5 drops of thymol blue TS, neutralize with ammonia TS and add

2.5 mL of ammonia TS. To this solution, add 20.0 mL of a solution of dithizone in n-butyl acetate (1 in 500), shake for 10 minutes and use this n-butyl acetate solution as the test solution. Separately, place 1.0 mL of standard lead solution in a platinum crucible, proceed as directed in 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 Spectrophotometry according to the following operating conditions: the absorbance of the test solution is smaller than that of the standard solution (not more than 10 ppm).

Gas: Acetylene or hydrogen - Air
Lamp: Lead hollow cathode lamp.
Wavelength: 283.3 nm.

(2) ***Arsenic***—Perform the test with 20 mL of the test stock solution obtained in (1) as the test solution: the stain is not more intense than the following standard stain.

Standard stain—Proceed in the same manner without Titanium Oxide and use this solution as the control solution. Transfer 0.2 mL of standard arsenic solution to a generator bottle, add the control solution to make 20 mL and proceed in the same manner as the test solution (not more than 1 ppm).

(3) ***Water-soluble substances***—Shake thoroughly 4.0 g of Titanium Oxide with 50 mL of water and allow to stand overnight. Shake thoroughly with 2 mL of ammonium chloride TS, add further 2 mL of ammonium chloride TS, if necessary and allow titanium oxide to settle. Add water to make 200 mL, shake thoroughly and filter through double filter paper. Discard the first 10 mL of the filtrate, evaporate 100 mL of the clear filtrate on a water-bath and heat strongly at 650 °C to constant weight: the mass of the residue is not more than 5.0 mg.

(4) ***Antimony***—Perform the test with 25 mL of the test stock solution obtained in (1) and use this solution as the test solution. Separately, proceed with 2 mL of standard antimony solution in the same manner and use this solution as the standard solution. Perform the test with the test solution and the standard solution as directed under Atomic Absorption Spectrophotometry according to the following goperating conditions: the absorbance of the test solution is not more than that of the standard solution (not more than 2 ppm).

Gas: Acetylene or hydrogen – Air
Lamp: 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. Add slowly purified hydrochloric acid to neutralize, add 5 mL of diluted sulfuric acid (1 in 2), add hydroxylamine hydrochloride solution (1 in 5) until the manganese dioxide precipitate disappears, 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 Atomic Absorption Spectrophotometry (cold vapor type). Place the test solution in a sample bottle of the atomic absorption spectrophotometer, add 10 mL of stannous chloride-sulfuric acid TS, connect the bottle immediately to the spectrophotometer, circulate air and read the absorbance, A_T, of the test solution after the recorder reading has risen rapidly and become constant at a wavelength of 253.7 nm. Separately, weigh accurately 13.5 mg of mercury (II) chloride, previously dried in a desiccator (silica gel) for 6 hours), add 10 mL of dilute nitric acid and add water to make exactly 1000 mL. Pipet 10 mL of this solution, add 1 mL of dilute nitric acid and add water to make exactly 100 mL. To 2.0 mL of this solution, add 1 mL of potassium permanganate (3 in 50), 30 mL of water and the amount of purified hydrochloric acid used to prepare the test solution, proceed in the same manner as the test solution and determine the absorbance A_S, of this solution: A_T is not greater than A_S (not more than 1 ppm).

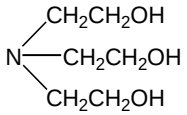
Loss on Drying Not more than 0.5 % (1 g, 105 °C, 3 hours).

Assay Weigh accurately about 0.2 g of Titanium Oxide, previously dried, transfer to a crucible and add 3 g of potassium pyrosulfate. Cover and heat gently at first, gradually raise the temperature and then heat the fused contents for 30 minutes. Continue heating for 30 minutes at a higher temperature to make the fused mixture a deep yellow-red, almost clear liquid. Cool, transfer the contents of 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 into the beaker and heat on a water-bath until the solution becomes almost clear. Dissolve 2 g of L-tartaric acid in the solution, add 2 to 3 drops of bromothymol blue TS, neutralize with ammonia TS and acidify with 1 mL to 2 mL of diluted sulfuric acid (1 in 2). Pass hydrogen sulfide sufficiently through the solution, add 30 mL of ammonia TS, again saturate the solution with hydrogen sulfide, allow to stand for 10 minutes and filter. Wash the precipitate on the filter paper with ten 25 mL volumes of a solution of ammonium tartrate (1 in 100), containing 2.5 mL of ammonium sulfide TS. When the precipitate is filtered and washed, prevent ferrous sulfide from oxidation by filling the solution on the filter paper. Combine the filtrate and the washings, add 40 mL of diluted sulfuric acid (1 in 2) and boil to expel hydrogen sulfide. Cool and dilute with water to make 400 mL. Add gradually 40 mL of cupferron TS to the solution with stirring and allow to stand. After sedimentation of a yellow precipitate, add again cupferron TS until a white precipitate is produced. Filter by slight suction using quantitative filter paper, wash twenty times with diluted hydrochloric acid (1 in 10) and remove water by stronger suction at the last washing. Dry the precipitate together with

the filter paper at 70 °C, transfer to a tared crucible and heat very gently at first and raise the temperature gradually after smoke stops evolving. Heat strongly between 900 °C and 950 °C to constant weight, cool and weigh as titanium oxide (TiO_2).

Containers and Storage ***Containers*—**Well-closed containers.

Trolamine



$C_6H_{15}NO_3$: 149.19

Triethanolamine

2,2′,2″-Nitrilotriethanol [*102-71-6*]

Trolamine is a mixture of alkanolamines consisting largely of triethanolamine containing some diethanolamine and monoethanolamine. Trolamine contains not less than 99.0 % and not more than 107.4 % of alkanolamines, calculated on the basis as triethanolamine ($C_6H_{15}NO_3$: 149.19).

Description Triethanolamine is colorless or pale yellow viscose liquid with a slight odor of ammonia.
Triethanolamine is miscible with water, with ethanol or with chloroform.

Identification (1) Take 1 mL of Trolamine, add 0.1 mL of cupric sulfate TS: a deep blue color is developed. 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) Take 1 mL of Trolamine, add 0.3 mL of cobaltous chloride TS : a carmine-red color is developed.

(3) Heat 1 mL of Trolamine gently in a test tube: the vapors turn moistened red litmus paper blue.

Refractive Index n_D^{20} : 1.481 ~ 1.486.

Specific Gravity d_{20}^{20} : 1.120 ~ 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 standard lead 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 solution. To 12 mL each of the test solution, the control solution and the blank solution, add 2 mL of pH 3.5 acetate buffer, 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 the control solution (not more than 10 ppm).

Water Not more than 0.5 % (1.0 g, volumetric titration, direct titration, using a mixture of 5.0 mL of acetic acid (100) and 20 mL of methanol instead of methanol for Water Determination).

Residue on Ignition Not more than 0.05 % (2 g).

Assay Dissolve about 2 g of Trolamine, accurately weighed, in 75 mL of water, titrate with 1 mol/L of hydrochloric acid (indicator: methylet TS 2 drops).

Each mL of 1 mol/L hydrochloric acid
= 149.19 of $C_6H_{15}NO_3$

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light-resistant.

Turpentine Oil

Oleum Terebinthinae

Turpentine Oil is the essential oil distilled with steam from the wood or balsam of *Pinus* species (Pinaceae).

Description Turpentine Oil is 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} : 1.465 ~ 1.478.

Specific Gravity d_{20}^{20} : 0.860 ~ 0.875.

Purity (1) ***Foreign matter*—**Turpentine Oil has no offensive odor. Shake 5 mL of Turpentine Oil with 5 mL of a solution of potassium hydroxide (1 in 6): the aqueous layer does not show a yellow-brown to dark brown color.

(2) ***Hydrochloric acid-coloring substances*—**Shake 5 mL of Turpentine Oil with 5 mL of hydrochloric acid and allow to stand for 5 minutes: the hydrochloric acid layer is pale yellow and not brown.

(3) ***Mineral oil*—**Place 5 mL of Turpentine Oil in a cassia flask, cool to a temperature not exceeding 15 °C, add drop-wise 25 mL of fuming sulfuric acid while shaking, warm between 60 °C and 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: not more than 0.1 mL of oil separates.

(4) ***Peroxide value***—Weigh accurately 2 g of Turpentine Oil, transfer to a stoppered conical 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 solution of potassium iodide, stopper the flask, allow to stand for 1 minute, shake continuously and add 30 mL of water. Titrate with 0.01 mol/L sodium thiosulfate VS until the blue color of the solution disappears after addition of 0.5 mL of starch TS near the end point where the solution is a pale yellow color. Perform a blank determination and make any necessary correction (not more than 0.1 mL of 0.01 mol/L sodium thiosulfate VS is consumed by the blank solution). Calculate the amount of peroxide by the following formula: not more than 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 test

V_0: Volume (mL) of 0.01 mol/L sodium thiosulfate VS consumed in the blank

W: Amount (g) of Turpentine Oil taken

Distilling Range 150 ~ 170 °C, not less than 90 %

Containers and Storage ***Containers***—Tight containers.

Storage—Light-resistant.

Urea

H_2NCONH_2

CH_4N_2O: 60.06

[*57-13-6*]

Urea contains not less than 99.0 % and not more than 101.0 % of urea (CH_4N_2O).

Description Urea appears as a colorless or white crystals or crystalline powder, is odorless and has a fresh salty taste.
Urea is very soluble in water, freely soluble in boiling ethanol, soluble in ethanol and slightly soluble in ether.
A solution of Urea (1 in 100) is neutral.

Identification (1) Heat about 0.5 g of Urea in a test tube: it liquefies and ammonia is evolved. Continue the heating until the liquid becomes turbid, then cool. Dissolve the fused mass in a mixture of 10 mL of water and 2 mL of sodium hydroxide TS (1 in 10) and add 1 drop of cupric sulfate TS: the solution acquires a reddish violet color.

(2) Dissolve 0.1 g of Urea in 1 mL of water and add 1mL of nitric acid: a white crystalline precipitate of urea nitrate is formed.

Melting Point 132.5 ~ 134.5 °C.

Purity (1) ***Chloride***—Perform the test with 2.0 g of Urea. Prepare the control solution with 0.40 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.007 %).

(2) ***Sulfate***— Perform the test with 2.0 g of Urea. Prepare the control solution with 0.40 mL of 0.005 mol/L sulfuric acid VS (not more than 0.010 %).

(3) ***Heavy metals***—Proceed with 1.0 g of Urea according to Method 1 and perform the test. Prepare the control solution with 2.0 mL of standard lead solution (not more than 20 ppm)

(4) ***Ethanol-insoluble matter***—Dissolve 5.0 g of Urea in 50 mL of warm ethanol and if any insoluble residue remains, filter the solution through a glass filter, wash the residue with 20 mL of warm ethanol and dry at 105 °C for 1 hour: the residue is not more than 2.0 mg.

Residue on Ignition Not more than 0.1 % (1 g).

Assay Transfer about 0.2 g of Urea, accurately weighed, dissolve in water and add water to make exactly 200 mL. Pipet exactly 5 mL of this solution into a Kjeldahl flask and proceed as directed under Nitrogen Determination.

Each mL of 0.005 mol/L sulfuric acid VS
= 0.30028 mg of CH_4N_2O

Containers and Storage ***Containers***—Well-closed containers.

Water

H_2O : 18.02

Water meets the quality standards for drinking water in the Management of Drinking Water Act. Water meets the 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 as the test solution. Prepare the control solution with 0.15 mL of standard ammonium solution, dilute with purified water for Ammonium Limit Test to make 30 mL and proceed in the same manner as the test solution (not more than 0.05 mg/L).

(2) ***Nitrogen from nitrites***—Place 50 mL of Water in a Nessler tube, add 0.3 g of griess-romijin's nitrous acid TS, dissolve with shaking, and allow to stand for 10 minutes: it does not exhibit a pale red color.

Water for Injection

Water for Injection is prepared by distillation or ultrafiltration, either from Water after applying appropriate pretreatments such as ion exchange or reverse osmosis, or from Purified Water in Bulk. When Water for Injection is prepared by ultrafiltration (methods for refining water by using a reverse osmosis membrane module, an ultrafiltration membrane module capable of removing substances having molecular masses 6000 and above, or a module using both types of membranes), care must be taken to avoid microbial contamination of the water processing system, and to provide water with equivalent quality to that prepared by distillation. Water for Injection must be used immediately after preparation. However, it may be stored temporarily, if adequate systems to prevent microbial proliferation, such as high temperature circulation, are established.

Description Water for Injection appears as clear and colorless liquid and is odorless.

Purity ***Total organic carbon*—**Not more than 0.50 mg/L.

Bacterial Endotoxins Less than 0.25 EU/mL.

Conductivity When the test is performed according to the following method, the conductivity at 25 °C of Water for Injection is not more than 2.1 μS/cm. Transfer a suitable amount of Water for Injection to a beaker and shake. Adjust the temperature to 25 ± 1 °C and measure the conductivity periodically while shaking vigorously. When the change in conductivity becomes not more than 0.1 μS/cm per 5 minutes, adopt the observed value as the conductivity (25 °C) of Water for Injection.

Purified Water in Bulk

Purified Water in Bulk is prepared from Water by ion exchange, distillation, reverse osmosis or ultrafiltration, or by a combination of these processes. Purified Water in Bulk must be used immediately after preparation. However, it may be stored temporarily, if microbial proliferation is suppressed.

Description Purified Water in Bulk appears as clear and colorless liquid and is odorless.

Purity ***Total organic carbon***—Not more than 0.50 mg/L.

Conductivity When the test is performed according to the following method, the conductivity at 25 °C of Purified Water in Bulk is not more than 2.1 μS/cm. Transfer a suitable amount of Purified Water in Bulk to a beaker and shake. Adjust the temperature to 25 ± 1 °C and measure the conductivity periodically while shaking vigorously. When the change in conductivity becomes not more than 0.1 μS/cm per 5 minutes, adopt the observed value as the conductivity (25 °C) of Purified Water in Bulk.

Purified Water in Containers

Purified Water in Containers is prepared from Purified Water in Bulk by introducing it into tight containers.

Description Purified Water in Containers appears as clear and colorless 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 VS and boil again for 10 minutes: the red color of the solution does not disappear.

Conductivity When the test is performed according to the following method, the conductivity at 25 °C of Purified Water in Containers is not more than 25 μS/cm for containers with a nominal volume of 10 mL or less, and not more than 5 μS/cm for containers with a nominal volume greater than 10 mL.
Transfer a suitable amount of Purified Water in Containers to a beaker and shake. Adjust the temperature to 25 ± 1 °C and measure the conductivity periodically while shaking vigorously. When the change in conductivity becomes not more than 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 total aerobic microbial count is not more than 100 CFU/mL. Perform the test using soybean-casein digest agar medium.

Containers and Storage ***Containers*—**Tight containers.

Sterile Water for Injection

Sterile Water for Injection is prepared from Water for Injection by introducing it into a hermetic container then sterilizing the product, or introducing previously sterilized Water for Injection into a sterile container by applying aseptic manipulation, then sealing up the container. When Sterile Water for Injection is prepared using Water for Injection prepared by distillation, it may be labeled as Distilled Water for Injection as a commonly used name. When Sterile Water for Injection is prepared by the distillation and packed in containers may be labeled as Distilled Water for Injection as a commonly used name.

Description Sterile Water for Injection appears as clear and colorless 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 and boil again for 10 minutes: the red color of the solution does not disappear.

Sterility Test It meets the requirement.

Conductivity When the test is performed according to the following method, the conductivity at 25 °C of Sterile Water for Injection is not more than 25 μS/cm for containers with a nominal volume of 10 mL or less, and not more than 5 μS/cm for containers with a nominal volume greater than 10 mL.
Transfer a suitable amount of Sterile Water for Injection to a beaker and shake. Adjust the temperature to 25 ± 1 °C and measure the conductivity periodically while shaking vigorously. When the change in conductivity becomes not more than 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.

Foreign Insoluble Matter Test for Injections It meets the requirement according to Method 1.

Determination of Volume of Injection in Containers It meets the requirement.

Insoluble Particulate Matter Test for Injections It meets the requirement.

Containers and Storage ***Containers*—**Hermetic containers. Plastic containers for aqueous infusions may be used.

Sterile Purified Water

Sterile Purified Water is prepared from Purified Water in Bulk by introducing it into a hermetic container then sterilizing the product, or by introducing previously sterilized Purified Water in Bulk into a sterile container by applying aseptic manipulation, then sealing up the container.

Description Sterile Purified Water is clear, colorless 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 and boil again for 10 minutes: the red color of the solution does not disappear.

Conductivity When the test is performed according to the following method, the conductivity at 25 °C of Sterile Purified Water is not more than 25 μS/cm for containers with a nominal volume of 10 mL or less, and not more than 5 μS/cm for containers with a nominal volume greater than 10 mL.
Transfer a suitable amount of Sterile Purified Water to a beaker and shake. Adjust the temperature to 25 ± 1 °C and measure the conductivity periodically while shaking vigorously. When the change in conductivity becomes not more than 0.1 μS/cm per 5 minutes, adopt the observed value as the conductivity (25 °C) of Sterile Purified Water.

Sterility Test It meets the requirement.

Containers and Storage ***Containers*—**Hermetic containers. Plastic containers for aqueous injections may be used.

Wheat Starch

Wheat Starch is the starch obtained from the caryopsis of wheat, *Triticum aestivum* Linné (*Gramineae*).

Description Wheat Starch appears as white masses or powder.
Wheat Starch is practically insoluble in water or in anhydrous ethanol.

Identification (1) Examine under a microscope using a mixture of water and glycerinol (1 : 1): Wheat Starch presents large and small granules and, very rarely, intermediate sizes. The large granules, 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 granules are elliptical or fusiform and the hilum appears as a slit along the main axis. The small granules, 2 to 10 μm in diameter, are rounded or polyhedral. Between crossed polarizing prisms, the granules show 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 subtle, white-turbid pasty liquid is formed.

(3) To 1 mL of the liquid obtained in (2), add 0.05 mL of diluted iodine TS (1 in 10): a dark blue-purple color is produced, and the color disappears by heating.

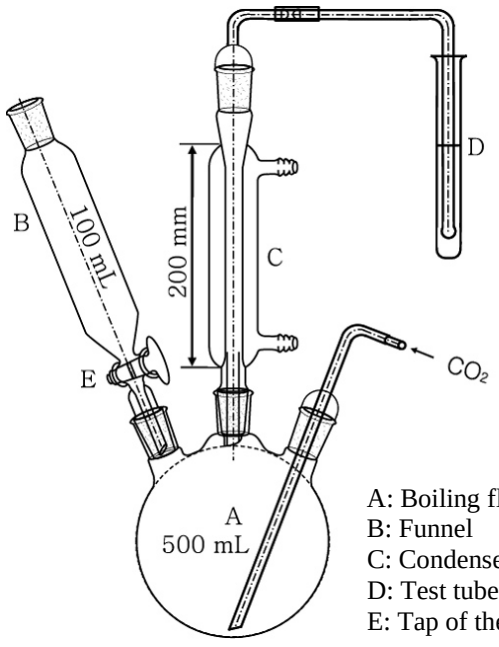
pH Put 5.0 g of Wheat Starch in a non-metal vessel, add 25.0 mL of freshly boiled and cooled water, mix gently for 1 minute and allow to stand for 15 minutes: the pH of the solution is between 4.5 and 7.0.

Purity (1) ***Iron***—To 1.5 g of Wheat 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 standard iron solution, add water to make 20 mL and use this solution as the control solution. Put 10 mL each of the test solution and the control solution in test tubes, add 2 mL of a solution of citric acid (1 in 5) and 0.1 mL of mercapto acetic acid and mix. Alkalize with ammonia solution (28) to litmus paper, 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 the solutions against a white background: the color of the test solution is not more intense than that of the control solution (not more than 10 ppm).

(2) ***Oxidizing substances***—To 4.0 g of Wheat Starch, add 50.0 mL of water, shake for 5 minutes and centrifuge. To 30.0 mL of the clear supernatant liquid, add 1 mL of acetic acid (100) and 0.5 g to 1.0 g of potassium iodide, shake and allow 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 determination and make any necessary correction: the volume of 0.002 mol/L sodium thiosulfate VS consumed is not more than 1.4 mL (not more than 20 ppm, calculated as hydrogen peroxide).

(3) ***Sulfur dioxide***—(i) Apparatus: Use apparatus shown in the figure.



A: Boiling flask
B: Funnel
C: Condenser
D: Test tube
E: Tap of the funnel

(ii) Procedure: Introduce 150 mL of water into the boiling flask, close the tap of the funnel, and pass carbon dioxide through the whole system at a rate of 100 ± 5 mL per minute. Pass cooling water through the condenser, and place 10 mL of hydrogen peroxide-sodium hydroxide TS (to a 9 : 1 mixture of water and hydrogen peroxide (hydrogen peroxide TS), add 3 drops of bromophenol blue TS and add 0.01 mol/L sodium hydroxide TS until the color changes to purple-blue; prepare before use) in the test-tube. After 15 minutes, remove the funnel without interrupting the stream of carbon dioxide, and introduce through the opening into the flask about 25 g of Wheat Starch, accurately weighed, with the aid of 100 mL of water. Apply tap grease to the outside of the connection part of the funnel, and connect the funnel. Close the tap of the funnel, pour 80 mL of 2 mol/L hydrochloric acid TS into the funnel, open the tap to introduce the hydrochloric acid into the flask, and close the tap while several mL of the hydrochloric acid remains, in order to avoid losing sulfur dioxide. Place the flask in a water-bath, and heat the mixture for 1 hour. Transfer the contents of the test-tube with the aid of a little water to a wide-necked conical flask. Heat in a water-bath for 15 minutes, and cool. Add 0.1 mL of bromophenol blue TS, and titrate with 0.1 mol/L sodium hydroxide VS until the color changes from yellow to purple-blue lasting for at least 20 seconds. Perform a blank determination and make any necessary correction. Calculate the amount of sulfur dioxide by applying the following formula: it is not more than 50 ppm.

$$\text{Amount (ppm) of sulfur dioxide} = \frac{\text{Amount (mL) of 0.1 mol/L sodium hydroxide VS consumed}}{\text{Amount (g) of Wheat Starch taken}} \times 1000 \times 3.203$$

(4) ***Total protein***—Weigh accurately 6 g of Wheat Starch containing 2 mg of nitrogen, transfer to a combustion flask, add 4 g of a powdered mixture consisting of 100 g of potassium sulfate, 5 g of cupric sulfate and 2.5 g of selenium, and add 3 glass beads. Wash any adhering particles from the neck into the flask with 5 mL of sulfuric acid, allowing it to tun down the inside wall of the flask, and mix by turning. Close the mouth of the flask, heat gradually at first, then increase the temperature until boiling. Continue the heating for 30 minutes, taking care to prevent the upper part of the flask from being overheated. Cool, dissolve the solid by carefully adding 25 mL of water, cool again and place in a steam distillation apparatus. Add 30 mL of sodium hydroxide solution (42 in 100) and distill immediately by passing steam through the mixture. Collect about 40 mL of distillate in 20 mL of 0.01 mol/L hydrochloric acid and enough 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 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 distillate with 0.01 mol/L sodium hydroxide TS, using methyl purple TS as the indicator. Determine the nitrogen content according to the following equation, where *a* is the volume (mL) of 0.01 mol/L sodium hydroxide TS consumed in the above titration and *b* is the volume (mL) of 0.01 mol/L sodium hydroxide TS consumed using 50 mg of glucose titrated in the same manner as the test solution, and multiply by the conversion factor 6.25 to calculate the total protein: not more than 0.3 %.

$$\text{Nitrogen content} = \frac{0.01401(b - a)}{W}$$

W: Amount (mg) of sample specimen taken

Loss on Drying Not more than 15.0 % (1 g, 130 °C, 90 minutes).

Residue on Ignition Not more than 0.6 % (1 g).

Microbial Limit The total aerobic microbial count is not more than 1000 CFU/g, the total combined yeasts/mould count is not more than 100 CFU/g, and Escherichia coli, Salmonella, Pseudomonas aeruginosa and Staphylococcus aureus are not observed.

Containers and Storage ***Containers*—**Well-closed containers.

5) Quasi Drugs

Absorbent Cotton

Absorbent Cotton is the hair of the seed of *Gossypium herbaceum* Linné, or that of other species of the same genus (Malvaceae), deprived of fatty matter and bleached.

Description Absorbent Cotton is white, soft, fine filament-like hair, is odorless and tasteless.
Under a microscope, Absorbent Cotton is hollow, flattened and twisted bans, striate and slightly thickened at the edges.
Absorbent Cotton dissolves in ammonia copper TS and does not dissolve in ordinary solvents.

Purity Obtain certain amount of Absorbent Cotton from different 10 parts of the same package and combine them to get the required amount.

(1) ***Acid or alkali***—Add 100 mL of freshly boiled and cooled water to 10 g of Absorbent Cotton, digest and add 3 drops of phenolphthalein TS to make 25 mL of the extracts: no red color develops. Add 1 drop of methyl orange TS to 25 mL of the extracts: no red color develops.

(2) ***Water-soluble substances***—To 5 g of Absorbent Cotton, add 500 mL of water and boil gently for 30 minutes, while adding water to maintain the original volume. Pour the extract through a funnel into another vessel, transfer the cotton to the funnel and press out the water absorbed therein with a glass rod. Wash the cotton with two 150 mL volumes of hot water and pressing the cotton after each washing. Filter the combined extracts and washings. Evaporate the filtrate to concentrate, transfer to a weighing bottle and dry at 105 °C to constant weight: the amount of the residue is not more than 14.0 mg. Perform a blank determination and make any necessary correction.

(3) ***Dyes***—Digest 10 g of Absorbent Cotton with 100 mL of ethanol, press out and transfer 50 mL of the extracts to a Nessler tube. Observe downward: a yellow color develops, but neither a blue nor a green color develops.

(4) ***Fluorescent whitening agents***—Irradiate Absorbent Cotton with ultraviolet light in a dark place: no fluorescence is perceptible on the surface.

(5) ***Submersion rate***—Prepare a test basket, 3.0 g in mass, made of copper wire, about 0.4 mm in diameter in the form of a cylinder 50 mm in diameter and 80 mm in depth, both spaces of 20 mm between the wires. Place 5 g of Absorbent Cotton in the basket, hold the basket on its side, 12 mm above the surface of water between 24 °C and 26 °C and drop the basket gently into the water, which is 200 mm deep: the time required for complete submersion is not more than 8 seconds.

(6) ***Absorbency***—Leave the submerged basket at the bottom of the water in (5) as it is for 3 minutes. Lift the basket gently from the water, keeping its side horizontal and allow to drain for 1 minute on the wire gauze of a sieve No. 10 in the same horizontal position. Then place in a beaker and weigh: the mass of water absorbed is not less than 100.0 g.

(7) ***Other filaments***—Dip 1.0 g of Absorbent Cotton in 0.5 mol/L iodine TS for 1 minute and wash well with water: no colored filament is found.

(8) ***Neps and adhering impurities***—Spread evenly about 1 g of Absorbent Cotton between two 10 cm-square, colorless, transparent plates and examine neps and adhering impurities (fragments of rinds and seeds): the total number of the fragments more than 2.5 mm in diameter is not more than 5.

Ash Not more than 0.25 % (5 g, proceed as directed in the Ash under Test for Herbal Drugs).

Containers and Storage ***Containers*—**Well-closed containers.

Purified Absorbent Cotton

Purified Absorbent Cotton is the hair of the seed of *Gossypium herbaceum* Linné, or that of other species of the same genus (Malvaceae), carefully selected, free from adhering impurities, deprived of fatty matter and bleached.

Description Purified Absorbent Cotton is white, soft, fine filament-like hair, is odorless and tasteless.
Under a microscope, Purified Absorbent Cotton is hollow, flattened and twisted band, striate and slightly thickened at the edges.
Purified Absorbent Cotton dissolves in ammonia copper TS and does not dissolve in ordinary solvents.

Purity (1) ***Acid or alkali, Water-soluble substances, Dyes, Fluorescent whitening agents, Absorbency, Other filaments, Neps and adhering impurities***—Proceed as directed in the Purity (1), (2), (3), (4), (5), (6), (7) and (8) under Absorbent Cotton.

(2) ***Short fibers***—Take 0.10 g of Purified Absorbent Cotton, separate the fibers into two groups, one consisting of fibers not exceeding 6.0 mm in length (short fibers) and the other consisting of fibers exceeding 6.0 mm in length, weigh both groups and determine the content of the short fibers: not more than 10 %.

$$\text{Content (\%) of the short fibers} = \frac{W_2}{W_1 + W_2} \times 100$$

W_1: Mass of the group of fibers exceeding 6.0 mm in length.

W_2: Mass of the group of fibers not exceeding 6.0 mm in length.

Ash Not more than 0.25 % (5 g, proceed as directed in the Ash under Test for Herbal Drugs).

Containers and Storage ***Containers*—**Well-closed containers.

Sterile Absorbent Cotton

Sterile Absorbent Cotton is sterilized Absorbent Cotton.

Description Sterile Absorbent Cotton is white soft, fine, filamentary hair, is odorless and tasteless.
Under a microscope, Sterile Absorbent Cotton is hollow, flattened and twisted band, striate and slightly thickened at the edges.
Sterile Absorbent Cotton dissolves in ammonia copper TS and does not dissolve in ordinary solvents.

Purity Proceed as directed in the Purity under Absorbent Cotton.

Ash Not more than 0.25 % (5 g, proceed as directed in the Ash under the Test for Herbal Drugs).

Sterility Test Proceed with about 0.5 g of Sterile Absorbent Cotton (whole amount if not more than 0.5 g) as directed in the Sterility test under Sterile Absorbent Gauze.

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Impervious to any microbe.

Sterile Purified Absorbent Cotton

Sterile Purified Absorbent Cotton is sterilized Purified Absorbent Cotton.

Description Sterile Purified Absorbent Cotton is white, soft, fine, filamentary hair, is odorless and tasteless.
Under a microscope, Sterile Purified Absorbent Cotton is hollow, flattened and twisted band, striate and slightly thickened at the edges.
Sterile Purified Absorbent Cotton dissolves in ammonia copper TS and does not dissolve in ordinary solvents.

Purity Proceed as directed in the Purity under Purified Absorbent Cotton.

Ash Not more than 0.25 % (5 g, proceed as directed in the Ash under the Test for Herbal Drugs).

Sterility Test Proceed with about 0.5 g of Sterile Purified Absorbent Cotton (whole amount if not more than 0.5 g) as directed in the Sterility test under Sterile Absorbent Gauze.

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Impervious to any microbe.

Absorbent Gauze

Absorbent Gauze consists of non-fatty and well-bleached cotton cloth of plain weave using pure cotton threads obtained from hairs of the seed of *Gossypium hirsutum* Linné or other species of the same genus (Malvaceae).
The amount of Absorbent Gauze is expressed in terms of its type, length and width. The Absorbent Gauze folded twice or more for special purposes may be expressed in terms of its folded type, length and width.

Description Absorbent Gauze is a white cotton cloth. Absorbent Gauze is odorless and tasteless.

Purity (1) ***Water-soluble substances*—**Place 20 g of Absorbent Gauze in 500 mL of water and boil gently 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 Absorbent Gauze with two 250 mL volumes of boiling water, pressing after each washing. Combine the extract and the washing, filter and add water to make 1000 mL. Transfer 400 mL of the filtrate to a beaker, evaporate to concentrate and place the residue in a weighing bottle. Wash the beaker with a small amount of water, combine the washings with the residue in the weighing bottle and dry at 105 °C to constant weight: the residue is not more than 20.0 mg. Perform a blank determination 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: no red color develops. Take 200 mL of the test solution and add 2 drops of methyl orange TS: no red color develops.

(3) ***Dextrin or starch*—**Take 200 mL of the extract obtained in (1), add 2 drops of iodine TS: no red-purple to blue color develops.

(4) ***Dyes*—**Digest 10 g of Absorbent Gauze with 80 mL of ethanol, press out and transfer 50 mL of the extracts to a Nessler tube. Observe downward: a yellow color develops, but neither a blue nor a green color develops.

(5) ***Fluorescent whitening agents*—**Irradiate Absorbent Gauze with ultraviolet light in a dark place: no fluorescence is perceptible on the surface.

(6) ***Submersion rate*—**Prepare a test basket, 3.0 g in mass, made of copper wire, about 0.4 mm in diameter

in the form of a cylinder 50 mm in diameter and 80 mm in depth, both spaces of 20 mm between the wires. Place 5 g of Absorbent Gauze in the basket, hold the basket on its side, 12 mm above the surface of water between 24 °C and 26 °C and drop the basket gently into the water, which is 200 mm deep: the time required for complete submersion is not more than 8 seconds.

(7) ***Other filaments***—Dip 1.0 g of Absorbent Gauze in 0.5 mol/L iodine TS for 1 minute and wash well with water: no colored filament is found.

Texture The texture requirements of Absorbent Gauze are as followings.

(1) ***Number of threads***—Prepare a frame of 2.54 cm × 2.54 cm and set the thread of to the edge of the frame. Count the integral number of the threads in 6.45 cm^2 frame and average the results of more than 3 counts. Except the closely woven parts.

(2) ***Mass***—Weigh a piece of Absorbent Gauze, 1 m in length, stated size in width and express the mass in terms of g per square meter. When it has closely woven parts at both edges in the length or width directions, measure full length and full width. When it has no closely woven parts in the length and width directions, measure the length and the width of the net. Fold Absorbent Gauze into about a 10 cm^2, allow to stand at ordinary temperature for 4 hours in a desiccator, previously saturated with the vapor above a saturated solution of sodium nitrate and weigh.

(3) ***Length***—Place Absorbent Gauze on a flat plate, eliminate the unnatural creases or tensions and measure the full length at the center line. When it has closely woven parts at both edges in the direction of the length, measure the full length. When it has no closely woven parts, measure only the net. For Absorbent Gauze being used as folded twice or more for special purpose, measure the length of folded state.

(4) ***Width***—Place Absorbent Gauze on a plate, smooth out the unnatural creases or tensions and measure the full width at more than 3 different locations. Take the average of these measurements. When it has closely woven parts at both edges in the direction of the width, measure the full width. When it has no closely woven parts, measure only the net. For Absorbent Gauze being used as folded twice or more for special purposes, measure the width of folded state.

(5) ***Fold***—Count the folding number of Absorbent Gauze folded twice and more for special purpose.

Ash Not more than 0.25 % (5 g, proceed as directed in the Ash under Test for Herbal Drugs).

Containers and Storage ***Containers***—Well-closed containers.

<table>
<tr><th rowspan="3">Type</th><th colspan="2">Number of threads per 2.54 cm</th><th rowspan="3">Number of threads per 6.45 cm^2</th><th colspan="2">Weight</th><th rowspan="3">Length</th><th rowspan="3">Allowable range of widths</th><th colspan="2">Folded twice or more</th></tr>
<tr><th rowspan="2">Warp</th><th rowspan="2">Filling</th><th rowspan="2">g/m^2</th><th rowspan="2">Allowable range</th><th rowspan="2">Length and width</th><th rowspan="2">Folds</th></tr>
<tr></tr>
<tr><td rowspan="2">1</td><td>*41-47</td><td>33-39</td><td>75-85</td><td>49.8</td><td rowspan="8">± 12 % for those not more than 90 cm in width

± 8 % for those exceeding 90 cm in width</td><td>Not less than 98 %</td><td>Not less than 1.6 mm less than the labeled width</td><td rowspan="9">Not less than 98 %</td><td rowspan="9">Not less than the labeled number of folds</td></tr>
<tr><td>41-47</td><td>33-39</td><td>76-84</td><td>49.8</td><td rowspan="8">Not less than 95 %</td><td rowspan="8">For those not more than 5 cm, not less than 20 % less than the labeled width

For those exceeding 5 cm and not more than 30 cm, not less than 1.0 cm less than the labeled width

For those exceeding 30 cm, not less than 1.5 cm less than the labeled width</td></tr>
<tr><td>2</td><td>30-34</td><td>26-30</td><td>57-63</td><td>37.4</td></tr>
<tr><td>3</td><td>26-30</td><td>22-26</td><td>49-55</td><td>32.3</td></tr>
<tr><td>4</td><td>22-26</td><td>18-22</td><td>41-47</td><td>27.8</td></tr>
<tr><td>5</td><td>20-24</td><td>16-20</td><td>37-43</td><td>25.7</td></tr>
<tr><td>6</td><td>18-22</td><td>14-18</td><td>33-39</td><td>22.5</td></tr>
<tr><td>7</td><td>18-22</td><td>10-14</td><td>29-35</td><td>20.6</td></tr>
<tr><td>8</td><td>1-16</td><td>8-12</td><td>21-27</td><td>13.8</td><td>± 12 %</td></tr>
</table>

Sterile Absorbent Gauze

Sterile Absorbent Gauze is the sterilized Absorbent Gauze.

Description Sterile Absorbent Gauze is a white cotton cloth and is odorless and tasteless.

Purity Proceed as directed in the Purity under Absorbent Gauze.

Texture Proceed as directed in the Texture under Absorbent Gauze

Ash Not more than 0.25 % (5 g, proceed as directed in the Ash under the Test for Herbal Drugs).

Sterility Take Sterile Absorbent Gauze from package aseptically under an aseptic circumstances, take about 1.0 g of it (whole content in the case of less than 1 g) evenly from 5 different parts around the center volume, put into a test tube of 25 mm × 200 mm containing 60 mL each of fluid thioglycollate medium using an appropriate utensil and perform the test as directed under the Sterility Test. In the case of the test for the growth of fungi, a 200-mL Erlenmeyer flask can also be used. When performing an efficient test of the medium under a condition without Sterile Absorbent Gauze, the medium supports the substantial growth of the incubated microorganisms.

Test number used in the Sterility Test is indicated in the following table.

The number of the same products sterilized simultaneously	Number of products used for test
Less than 100	4
Not less than 100 and less than 500	10
Not less than 500	20

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Impervious to any microbe.

Adhesive Plaster

Method of Preparation Adhesive Plaster consists of a mixture of carefully selected rubber, resins, zinc oxide and other substances, resulting in an adhesive mass. This adhesive mass is spreaded evenly on a fabric.

Description The surface of Adhesive Plaster is milky white and adheres well to the skin.

Purity ***Adhesive mass***—The back of the fabric is free from adhesive mass. When unrolled, no large amount of the adhesive mass moves to the back of the next fabric. When removed from the skin, no large amount of the adhesive mass remains on the skin.

Texture Adhesive Plaster is usually rectangular: the length is not less than 98 % of the labeled length. Measure the width at 5 suitable separate locations of the plaster: the average of the 5 measurements is not less than 94 % of the labeled width.

Tensile Strength Cut strip parallel with the warp, 12 mm in standard width, about 200 mm in length, allow to stand at ordinary temperature for 4 hours in a desiccator, previously saturated with the vapor over a saturated sodium nitrite solution. Using a pendulum-type testing machine, set the target distance to 150 mm and nip firmly with a clamp whose width is between 25 mm and 50 mm. Pull at the rate of 300 mm per minute and measure the maximum breaking load: the average of 10 measurements is not less than 7.5 kg. When the width is narrower than the standard width, calculate with the necessary correction.

Adhesive Strength Cut a strip parallel with the warp, 12 mm in standard width, about 250 mm in length and apply 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 a thermostat at 37 °C for 30 minutes. At once, pass a rubber roller, 850 g in mass, twice over Adhesive Plaster at a rate of 300 mm per minute. Leave in a thermostat at 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 edge of the test plate. Using a pendulum-type testing machine, nip firmly the free edge with the upper clamp and the test plate with the lower clamp. Peel off successively at a rate of 300 mm per minute and measure the load in 4 trials at intervals of about 20 mm: the average is not less than 150 g. When the width is narrower than the standard width, calculate with the necessary correction.

Containers and Storage ***Containers*—**Well-closed containers.

***Storage*—**Light-resistant.

Bandage

Gauze Bandage

Bandage is made of Type 1, 2 or 3 Absorbent Gauze. The length and width of Bandage are stated on the package.

Description The Bandage is in various length and width and threads per 2.54 cm^2 and threads per 6.45 cm^2 are followed by the texture, number of threads under Absorbent Gauze. Bandage is cut by the length

and width declared on the label.

Purity Proceed as directed in the Purity under Absorbent Gauze.

Texture Its length is not less than 98 % of that declared on the label and its average width measured 5 points in an appropriate interval is not more than 1.6 mm less than the declared width.

Containers and Storage ***Containers*—**Well-closed containers.

***Storage*—**Each cut.

Calamine Lotion

Method of Preparation

Calamine	80 g
Zinc Oxide	80 g
Glycerin	20 mL
Bentonite Magma	250 mL
Calcium Hydroxide Solution	a sufficient quantity
	To make 1000 mL

Dilute the Bentonite Magma with an equal volume of Calcium Hydroxide Solution. To 3 g of Calcium Hydroxide, add 1000 mL of cold purified water, mix with occasional shaking for 1 hour, allow to stand and use the clear supernatant liquid as the Calcium Hydroxide Solution. Mix the Calamine and Zinc Oxide intimately with Glycerin and about 100 mL of the diluted Bentonite Magma, triturating until a smooth, uniform paste is formed. Gradually incorporate the remainder of the diluted Magma. Finally add enough Calcium Hydroxide Solution to make 1000 mL and shake well. If a more viscous consistency in the Lotion is desired, the quantity of Bentonite Magma may be increased to not more than 400 mL.

Microbial Limit When perform the test, *pseudomonas* and *staphylococcus* are not observed.

Containers and Storage ***Containers*—**Tight containers.

Cresol

C_7H_8O: 108.14

Cresol is a mixture of isomeric cresols.

Description Cresol is a clear, colorless or yellow to yellow-brown liquid, has a phenol-like odor.
Cresol is miscible with ethanol or with ether.
Cresol is sparingly soluble in water.
Cresol dissolves in sodium hydroxide TS.
A saturated solution of Cresol is neutral to bromocresol purple TS.
Cresol is a highly refractive liquid.
Cresol becomes dark brown by light or on aging.

Identification Take 5 mL of a saturated solution of Cresol and add 1 to 2 drops of dilute ferric chloride TS: a blue-purple color develops.

Specific Gravity d_{20}^{20} : 1.032 ~ 1.041.

Purity (1) ***Hydrocarbons*—**Dissolve 1.0 mL of Cresol in 60 mL of water: the solution shows no more turbidty than that produced in the following control solution.

Control solution—Take 54 mL of water, add 6.0 mL of 0.005 mol/L sulfuric acid and 1.0 mL of barium chloride TS and after thorough shaking, 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 develop yellow, but neither brown nor dark tint.

(3) ***Phenol***—Weigh accurately about 2.5 g of Cresol, add 10 mL 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, neutralize with 20 vol % nitric acid added dropwise, 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. Pipet 5 mL each of the test solution into two test tubes with the 25 mL graduation line, and pipet 5 mL each of the standard solution into another two test tubes. To each test tube, add 5 mL of Millon's TS, allowing it to flow down the inner wall of the test tube, mix, heat in a boiling water bath for 30 minutes, cool in a cold water bath for not less than 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 one of each pair of test tubes, add water to all test tubes to make 25 mL and allow to stand for 16 hours. The test tubes with formaldehyde show a yellow color and the test tubes without formaldehyde show 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 marked B_1 and B_2, representing the solution not treated and the solution treated with formaldehyde, respectively. Pipet 10 mL from the test tube containing formaldehyde and Cresol into a test tube marked N_1, and pipet 10 mL from the test tube containing only Cresol without formaldehyde into a test tube marked N_2. Observe the colors of test tubes N_1 and N_2 in a colorimeter. Add to tube N_1 the orange to red colored solution from buret B_1, and add to tube N_2 an equal volume of the yellow colored solution from

buret B_2, until the colors in tubes N_1 and N_2 match (not more than 5.0 %).

Content (%) of phenol in Cresol = $5V/W$

V: Amount (mL) of standard phenol solution consumed from buret B1
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 insert the stopper. Shake for 30 minutes, allow to stand for 15 minutes, add 5 mL of potassium iodide (1 in 5) and insert the stopper, taking precautions to prevent the escape of bromine vapor. Mix by shaking thoroughly, remove the stopper, and rinse the stopper and the neck of the flask with a small amount of water and combine the 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 determination and make any necessary correction.

Each mL of 0.05 mol/L bromine VS
= 1.5685 mg of C_6H_6O

Distilling Range 196 ~ 206 °C, not less than 90 %.

Containers and Storage ***Containers*—**Tight containers.
***Storage*—**Light-resistant.

Cresol Solution

Cresol Solution contains not less than 1.25 vol % and not more than 1.60 vol % of cresol.

Method of Preparation

Saponated Cresol Solution	30 mL
Water or Purified Water	a sufficient quantity
	To make 1000 mL

Prepare by mixing the above ingredients.

Description Cresol Solution is a clear or slightly turbid, yellow solution, has the odor of cresol.

Identification Take 0.5 mL of the oily layer obtained in the Assay, add 30 mL of water, mix with shaking, filter and perform the following tests using the filtrate as the test solution.

(i) Take 5 mL of the test solution and add 1 to 2 drops of ferric chloride TS: a blue-purple color develops.

(ii) Take 5 mL of the test solution and add 1 to 2 drops of bromine TS: a pale yellow, flocculent precipitate is produced.

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. Distil into a Cassia flask which contains 30 g of powdered sodium chloride and 3 mL of kerosene, exactly measured, until the distillate measures 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. Shake often the Cassia flask in warm water to dissolve the sodium chloride and allow to stand for 15 minutes. After cooling to 15 °C, add a saturated solution of sodium chloride and allow to stand for more than 3 hours with occasional shaking. Allow to stand for 1 to 2 minutes with gentle shaking to combine the separated oil drops with the oil layer. The volume (mL) subtracted 3 (mL) from the oil layer measured represents the amount (mL) of Cresol.

Containers and Storage ***Containers*—**Tight containers.

Saponated Cresol Solution

Saponated Cresol Solution contains not less than 42 vol % and not more than 52 vol % of cresol.

Method of Preparation

Cresol	500 mL
Vegetable oil	300 mL
Potassium Hydroxide	a suitable quantity
Water or Purified Water	a sufficient quantity
	To make 1000 mL

Dissolve Potassium Hydroxide, in required quantity for saponification, in a sufficient quantity of Water or Purified Water, add this solution to vegetable oil, previously warmed, add a sufficient quantity of Ethanol, if necessary, heat on a water-bath by thorough stirring and continue the saponification. After complete saponification, add Cresol, stir thoroughly until the mixture becomes clear and add sufficient Water or Purified water to make 1000 mL. A corresponding amount of Sodium Hydroxide may be used in place of Potassium Hydroxide.

Description Saponated Cresol Solution is yellow-brown to red-brown, viscous liquid, has the odor of cresol.
Saponated Cresol Solution is miscible with water, with ethanol or with glycerin.
Saponated Cresol Solution is alkaline.

Identification Proceed as directed in the Identification under Cresol, using the distillate in the Purity (3).

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: no red color is observed.

(2) ***Unsaponifiable matter*—**Take 1.0 mL of Saponated Cresol Solution, add 5 mL of water and shake: the solution is clear.

(3) ***Cresol fraction*—**Transfer 180 mL of Saponated Cresol Solution to a 2000-mL distilling flask, add 300 mL of water and 100 mL of dilute sulfuric acid and distil with steam until the distillate becomes clear. Draw off the water from the condenser and continue the distillation until water vapor begins to come out of the tip of the condenser. Cool the condenser again and continue distillation for 5 minutes. Dissolve 20 g of sodium chloride per 100 mL of the distillate, allow to stand and collect the separated clear oil layer. After adding about 15 g of powdered calcium chloride for drying in small volumes with frequent shaking, allow to stand for 4 hours. Filter and distil exactly 50 mL of the filtrate: the distillate is not less than 43 mL at between 196 °C and 206 °C.

Assay Transfer exactly 5 mL of Saponated Cresol Solution, to a 500-mL distilling flask, holding the pipet vertically for 15 minutes to draw off the solution into the flask. 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 distil into a Cassia flask which contains 30 g of powdered sodium chloride and exactly 3 mL of kerosene, until 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. Allow the Cassia flask to stand in warm water for 15 minutes to dissolve the sodium chloride with frequent shaking. Cool to 15 °C, add a saturated solution of sodium chloride and allow to stand for more than 3 hours with occasional shaking. Allow to stand for 1 to 2 minutes with gentle shaking and combine the separated oil drops with the oil layer. The volume (mL) subtracted 3 (mL) from the oil layer measured represents the amount (mL) of cresol.

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light-resistant.

Dental Iodine Glycerin

Dental Iodine Glycerin contains not less than 9.0 % and not more than 11.0 % of iodine (I: 126.90), not less than 7.2 % and not more than 8.8 % of potassium iodide (KI: 166.00) and not less than 0.9 % and not more than 1.1 % of zinc sulfate ($ZnSO_4 \cdot 7H_2O$: 287.58).

Method of Preparation

Iodine	10 g
Potassium Iodide	8 g
Zinc Sulfate Hydrate	1 g
Glycerin	35 mL
Purified Water	a sufficient quantity
	To make 100 mL

Dissolve and mix the above ingredients.

Description Dental Iodine Glycerin is a dark red-brown liquid. Dental Iodine Glycerin has odor of iodine.

Identification (1) The colored solution obtained in the Assay (1) has a red color. Determine the absorption spectrum of this solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits maxima between 510 nm and 514 nm (iodine).

(2) The colored solution obtained in the Assay (2) has a red color. Determine the absorption spectrum of this solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits maxima 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 and mix. Then add 2 mL of sodium hydroxide TS, add 1 mL of a solution of cupric chloride in ethanol (1 in 10) and shake: a blue color is observed (glycerin).

(4) The colored solution obtained in the Assay (3) has a red-purple to purple color. Determine the absorption spectrum of this solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits maxima 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 5.0 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 about 0.4 g of Potassium Iodide RS, previously dried at 105 °C for 4 hours and dissolve in diluted ethanol (3 in 10) to make exactly 50 mL. Pipet 5.0 mL of this solution, add water to make exactly 200 mL and use this solution as the standard solution. Pipet 10.0 mL each of the test solution and the standard solution, to each add exactly 20 mL of a mixture of chloroform and hexane (2 : 1), shake immediately and separate the chloroform-hexane layer [use the water layer in (2)]. 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 Spectrophotometry, using a mixture of chloroform and hexane (2 : 1) as the blank and make any necessary corrections.

$$\text{Amount (mg) of iodine (I)} = \text{amount (mg) of Iodine RS} \times \frac{A_T}{A_S}$$

(2) ***Potassium iodide***—Separate the water layers of the test solution and the standard solution obtained in (1), pipet 7 mL each of the water layers and to each add 1.0 mL of diluted hydrochloric acid (1 in 2), 1 mL of sodium nitrite TS and 10 mL of a mixture of chloroform and hexane (2 : 1) and shake immediately. Separate the chloroformhexane 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 of directed under the Ultraviolet-visible Spectrophotometry, using a mixture of chloroform and hexane (2 : 1) as the blank and make any necessary corrections.

$$\text{Amount (mg) of potassium iodide (KI)} = \text{amount (mg) of Potassium Iodide RS} \times \frac{A_T}{A_S}$$

(3) ***Zinc sulfate hydrate***—Pipet 5 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, to each add 10 mL of a mixture of chloroform and hexane (2 : 1), shake and allow to stand. Pipet 3 mL each of tile water layers, add to each add 2 mL of boric acid-potassium chloride-sodium hydroxide buffer solution, pH 10.0, and 2 mL of zincon TS and water to make exactly 25 mL. Determine the absorbances, A_T and A_S, obtained from the test solution and the standard solution, respectively, at 620 nm as directed under the Ultraviolet-visible Spectrophotometry, using the solution prepared in the same manner with 3 mL of water as the blank and make any necessary corrections.

$$\text{Amount (mg) of zinc sulfate hydrate } (ZnSO_4 \cdot 7H_20)_3 = \text{amount (mg) of zinc in 10 mL of standard zinc stock solution} \times \frac{A_T}{A_S} \times 4.397$$

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light-resistant.

Dental Phenol with Camphor

Method of Preparation

Phenol	35g
d-or dl-Camphor	65g
	100g

Melt Phenol by warming, add *d*-Camphor or *dl*-Camphor and mix.

Description Dental Phenol with Camphor is colorless or pale red liquid, and has a characteristic odor.

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light-resistant.

Ethanol for Disinfection

Alcohol for disinfection

Ethanol for Disinfection contains not less than 76.9vol % and not more than 81.4vol % (by specific gravity) of ethanol (C_2H_6O: 46.07) at 15 °C.

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 is clear, colorless liquid.
Ethanol for Disinfection is miscible with water.
Ethanol for Disinfection burns with a pale blue flame on ignition.
Ethanol for Disinfection is volatile.

Identification (1) Mix 1 mL of Ethanol with 2 mL of iodine TS and 1mL of sodium hydroxide TS: a pale yellow precipitate is produced.

(2) Heat 1 mL of Ethanol with 1 mL of acetic acid (100) and 3 drops of sulfuric acid: the odor of ethyl acetate is perceptible.

Specific Gravity d_{15}^{15} : 0.860 ~ 0.873.

Purity Proceed as directed in the Purity under Ethanol.

Containers and Storage ***Containers*—**Tight containers.

***Storage*—**Light-resistant.

Zinc Oxide Ointment

Zinc Oxide Ointment contains not less than 18.5 % and not more than 21.5 % of zinc oxide (ZnO: 81.41).

Method of Preparation

Zinc Oxide	200 g
Liquid Paraffin	30 g

White Ointment	a sufficient quantity
	To make 1000 g

Prepare as directed under Ointments, with the above ingredients.

Description Zinc Oxide Ointment is white.

Identification Place 1 g of Zinc Oxide Ointment in a crucible, melt by warming, heat gradually raising the temperature until the mass is thoroughly charred and then ignite: a yellow color is observed and disappears on cooling. To the residue, add 10 mL of water and 5 mL of dilute hydrochloric acid, shake well and filter. To the filtrate, add 2 to 3 drops of potassium ferrocyanide TS: a white precipitate is produced (zinc oxide).

Purity ***Calcium, magnesium and other foreign inorganic matters***—Place 2.0 g of Zinc Oxide Ointment in a crucible, melt by warming and heat gradually raising the temperature, until the mass is thoroughly charred. Ignite the mass strongly until the residue becomes uniformly yellow and cool. Add 6 mL of dilute hydrochloric acid and heat on a water-bath for 5 to 10 minutes: the solution is colorless and clear. Filter the solution, add 10 mL of water to the filtrate and add ammonia TS until the precipitate first formed redissolves. Add 2 mL each of ammonium oxalate TS and dibasic sodium phosphate TS to this solution: the solution remains unchanged or becomes very slightly turbid within 5 minutes.

Assay Weigh accurately about 2 g of Zinc Oxide Ointment, place in a crucible, melt by warming, heat gradually raising the temperature until the mass is thoroughly charred and then ignite until the residue becomes uniformly yellow and cool. Dissolve the residue in 1 mL of water and 1.5 mL of hydrochloric acid and add water to make exactly 100 mL. Pipet exactly 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 begin to form in the solution. Add 5 mL of ammonia-ammonium chloride buffer solution, pH 10.7, and titrate with 0.05 mol/L disodium ethylene diaminetetraacetate VS (indicator: 40 mg of eriochrome black T-sodium chloride indicator)

Each mL of 0.05 mol/L
disodium ethylenediaminetetraacetate VS
= 4.071 mg of ZnO

Containers and Storage ***Containers*—**Tight containers.