

200 Units per mg. One unit indicates an amount of the enzyme which produces 1  $\mu\text{mol}$  of D-glucono- $\delta$ -lactone in 1 minute at 25°C and pH 7.0 from glucose used as the substrate.

**Glucose-pepton medium** See the Sterility Test under the General Tests, Processes and Apparatus.

**Glucose TS** Dissolve 30 g of glucose in water to make 100 mL. Prepare as directed under Injections.

**L-Glutamic acid**  $\text{HOOC}(\text{CH}_2)_2\text{CH}(\text{NH}_2)\text{COOH}$   
[K 9047, Special class]

**L-Glutamine**  $\text{H}_2\text{NCO}(\text{CH}_2)_2\text{CH}(\text{NH}_2)\text{COOH}$   
[K 9103, Special class]

**7-(Glutarylglucyl-L-arginylamino)-4-methylcoumarin**  
 $\text{C}_{23}\text{H}_{30}\text{N}_6\text{O}_7$  White powder. It is freely soluble in acetic acid (100), sparingly soluble in dimethylsulfoxide, and practically insoluble in water.

**Absorbance**  $E_{1\text{cm}}^{1\%}$  (325 nm): 310 - 350 [2 mg, diluted acetic acid (100) (1 in 500), 200 mL].

**Optical rotation**  $[\alpha]_{\text{D}}^{20}$ : -50 - -60° [0.1 g, diluted acetic acid (100) (1 in 2), 10 mL, 100 mm].

**Purity** Related substances—Prepare the sample solution by dissolving 5 mg of 7-(glutarylglucyl-L-arginylamino)-4-methylcoumarin in 0.5 mL of acetic acid (100), and perform the test as directed under the Thin-layer Chromatography. Spot 5  $\mu\text{L}$  of the sample 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 (100) (15:12:10:3) to a distance of about 10 cm, air-dry the plate, and dry more at 80°C for 30 minutes. After cooling, allow the plate to stand for 30 minutes in a box filled with iodine vapors: any observable spot other than the principal spot at the Rf value of about 0.6 does not appear.

**7-(Glutarylglucyl-L-arginylamino)-4-methylcoumarin TS**  
Dissolve 5 mg of 7-(glutarylglucyl-L-arginylamino)-4-methylcoumarin in 0.5 to 1 mL of acetic acid (100), lyophilize, dissolve this in 1 mL of dimethylsulfoxide, and use this solution as solution A. Dissolve 30.0 g of 2-amino-2-hydroxymethyl-1,3-propanediol and 14.6 g of sodium chloride in 400 mL of water, adjust the pH to 8.5 with dilute hydrochloric acid, add water to make 500 mL, and use this solution as solution B. Mix 1 mL of the solution A and 500 mL of the solution B before use.

**Glycerin**  $\text{C}_3\text{H}_8\text{O}_3$  [K 8295, Glycerol, Special class. Same as the monograph Concentrated Glycerin]

**Glycine**  $\text{H}_2\text{NCH}_2\text{COOH}$  [K 8291, Special class]

**Glycyrrhizic acid for thin-layer chromatography**  
 $\text{C}_{42}\text{H}_{62}\text{O}_{16} \cdot x\text{H}_2\text{O}$  Colorless or white, sweet, crystalline powder. Freely soluble in hot water and in ethanol (95), and practically insoluble in diethyl ether.

**Melting point:** 213 - 218°C (with decomposition).

**Purity** Related substances—Dissolve 0.010 g of glycyrrhizic acid for thin-layer chromatography in 5 mL of dilute ethanol, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add dilute ethanol to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 10  $\mu\text{L}$  each of the sample solution and the standard solution as directed in the Identification under Glycyrrhiza: the spots other than the principal spot at the Rf value of about 0.3 from the sample solution

are not more intense than the spot from the standard solution.

**Graphite carbon for gas chromatography** Prepared for gas chromatography.

**Griess-Romijn's nitric acid reagent** Triturate thoroughly 1 g of 1-naphthylamine, 10 g of sulfanilic acid and 1.5 g of zinc dust in a mortar.

**Storage**—Preserve in tight, light-resistant containers.

**Griess-Romijn's nitrous acid reagent** Triturate thoroughly 1 g of 1-naphthylamine, 10 g of sulfanilic acid and 89 g of tartaric acid in a mortar.

**Storage**—Preserve in tight, light-resistant containers.

**Guaiacol**  $\text{CH}_3\text{OC}_6\text{H}_4\text{OH}$  Clear, colorless to yellow liquid or colorless crystals, having a characteristic aroma. Sparingly soluble in water, and miscible with ethanol (95), with diethyl ether and with chloroform. Melting point: about 28°C

**Purity**—Perform the test with 0.5  $\mu\text{L}$  of guaiacol as directed under the Gas Chromatography according to the following conditions. Measure each peak area by the automatic integration method, and calculate the amount of guaiacol by the area percentage method: It showed the purity of not less than 99.0%.

**Operating conditions**

**Detector:** Hydrogen flame-ionization detector.

**Column:** A glass column about 3 mm in inside diameter and about 2 m in length, packed with siliceous earth for gas chromatography, 150- to 180- $\mu\text{m}$  in particle diameter, coated with polyethylene glycol 20 M at the ratio of 20%.

**Column temperature:** A constant temperature of about 200°C.

**Carrier gas:** Nitrogen

**Flow rate:** Adjust the flow rate so that the retention time of guaiacol is 4 to 6 minutes.

**Detection sensitivity:** Adjust the detection sensitivity so that the peak height of guaiacol obtained from 0.5  $\mu\text{L}$  of guaiacol is about 90% of the full scale.

**Time span of measurement:** About 3 times as long as the retention time of guaiacol after the solvent peak.

**Hanus' TS** Dissolve 20 g of iodine monobromide in 1000 mL of acetic acid (100). Preserve in light-resistant, glass-stoppered bottles, in a cold place.

**Heavy hydrogenated solvent for nuclear magnetic resonance spectroscopy** Prepared for nuclear magnetic resonance spectroscopy. Heavy hydrogenated chloroform ( $\text{CDCl}_3$ ), heavy hydrogenated dimethyl sulfoxide [ $(\text{CD}_3)_2\text{SO}$ ], heavy water ( $\text{D}_2\text{O}$ ), and heavy hydrogenated pyridine ( $\text{C}_5\text{D}_5\text{N}$ ) are available.

**Heavy water for nuclear magnetic resonance spectroscopy**  $\text{D}_2\text{O}$  Prepared for nuclear magnetic resonance spectroscopy.

**Helium** He Not less than 99.995 vol%.

**Hematoxylin**  $\text{C}_{16}\text{H}_{14}\text{O}_6 \cdot n\text{H}_2\text{O}$  White or light yellow to brownish crystals or crystalline powder. It is soluble in hot water and in ethanol (95), and sparingly soluble in cold water.

**Residue on ignition:** not more than 0.1% (1 g).

**Hematoxylin TS** Dissolve 1 g of hematoxylin in 12 mL of ethanol (99.5). Dissolve 20 g of aluminum potassium sul-

fate 12-water in 200 mL of warm water, cool, and filter. After 24 hours, mix these two prepared solutions. Allow to stand for 8 hours in a wide-mouthed bottle without using a stopper, and filter.

**Heparin sodium** [Same as the namesake monograph]

**HEPES buffer solution, pH 7.5** Dissolve 2.38 g of *N*-2-hydroxyethylpiperazine-*N'*-2-ethanesulfonic acid in 90 mL of water, adjust to pH 7.5 with 5 mol/L sodium hydroxide TS, and add water to make 100 mL.

**Heptane**  $\text{CH}_3(\text{CH}_2)_5\text{CH}_3$  [K 9701, Special class]

**Hexaammonium heptamolybdate tetrahydrate**  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$  [K 8905, Special class]

**Hexamethylenetetramine**  $(\text{CH}_2)_6\text{N}_4$  [K 8847, Special class]

**Hexamine** See hexamethylenetetramine.

**Hexane**  $\text{C}_6\text{H}_{14}$  [K 8848, Special class]

**Hexane for liquid chromatography**  $\text{CH}_3(\text{CH}_2)_4\text{CH}_3$  Colorless, clear liquid. Miscible with ethanol (95), with diethyl ether, with chloroform and with benzene.

**Boiling point:** about 69°C

**Purity** (1) Ultraviolet absorptive substances—Read the absorbances of hexane for liquid chromatography as directed under the Ultraviolet-visible Spectrophotometry, using water as the blank: not more than 0.3 at the wavelength of 210 nm, and not more than 0.01 between 250 nm and 400 nm.

(2) Peroxide—To a mixture of 100 mL of water and 25 mL of dilute sulfuric acid add 25 mL of a solution of potassium iodide (1 in 10) and 20 g of hexane for liquid chromatography. Stopper tightly, shake, and allow to stand in a dark place for 15 minutes. Titrate this solution, while shaking well, with 0.01 mol/L sodium thiosulfate (indicator: 1 mL of starch TS). Perform a blank determination in the same manner.

***n*-Hexane for liquid chromatography** See hexane for liquid chromatography.

**Hexane for purity of crude drug** [K 8848, Special class] Use hexane meeting the following additional specification. Evaporate 300.0 mL of hexane for purity of crude drug in vacuum at a temperature not higher than 40°C, add the hexane to make exactly 1 mL, and use this solution as the sample solution. Separately, dissolve 2.0 mg of  $\gamma$ -BHC in hexane to make exactly 100 mL. Pipet 1 mL of this solution, and add hexane to make exactly 100 mL. Further pipet 2 mL of this solution, add hexane to make exactly 100 mL, and use this solution as the standard solution I. Perform the test with 1  $\mu\text{L}$  each of the sample solution and the standard solution I as directed under the Gas Chromatography according to the following operating conditions, and determine each peak area by the automatic integration method: the total area of peak other than the solvent peak from the sample solution is not larger than the peak area of  $\gamma$ -BHC from the standard solution I.

**Operating conditions**

Proceed the operating conditions in the Purity (3) under Powdered Ginseng except detection sensitivity and time span of measurement.

**Detection sensitivity:** Pipet 1 mL of the standard solution I, add hexane to make exactly 20 mL, and use this solution as the standard solution II. Adjust the detection sensitivity

so that the peak area of  $\gamma$ -BHC obtained from 1  $\mu\text{L}$  of the standard solution II can be measured by the automatic integration method, and the peak height of  $\gamma$ -BHC from 1  $\mu\text{L}$  of the standard solution I is about 20% of the full scale.

**Time span of measurement:** About three times as long as the retention time of  $\gamma$ -BHC after the solvent peak.

**Hexane for ultraviolet-visible spectrophotometry** [K 8848, Special class]. When determining the absorbance of hexane for ultraviolet-visible spectrophotometry as directed under the Ultraviolet-visible Spectrophotometry, using water as the blank solution, its value is not more than 0.10 at 220 nm and not more than 0.02 at 260 nm, and it has no characteristic absorption between 260 nm and 350 nm.

***n*-Hexane for ultraviolet-visible spectrophotometry** See hexane for ultraviolet-visible spectrophotometry.

**Hexasilanized silica gel for liquid chromatography** Prepared for liquid chromatography.

**L-Histidine hydrochloride** See L-histidine hydrochloride monohydrate.

**L-Histidine hydrochloride monohydrate**  $\text{C}_6\text{H}_9\text{N}_3\text{O}_2\cdot\text{HCl}\cdot\text{H}_2\text{O}$  [K 9050, Special class].

**Homatropine hydrobromide**  $\text{C}_{16}\text{H}_{21}\text{NO}_3\cdot\text{HBr}$  [Same as the namesake monograph].

**Honokiol**  $\text{C}_{18}\text{H}_{18}\text{O}_2\cdot x\text{H}_2\text{O}$  Odorless white, crystals or crystalline powder.

**Purity**—Dissolve 1 mg of honokiol in the mobile phase to make exactly 10 mL, and use this solution as the sample solution. Perform the liquid chromatography with 10  $\mu\text{L}$  of the sample solution as directed in the Component determination under Magnolia Bark: total area of peaks other than honokiol from the sample solution is not larger than 1/10 of total area of the peaks other than the solvent peak.

**Horse serum** Collect the blood from horse in a flask, coagulate, and allow to stand at room temperature until the serum is separated. Transfer the separated serum in glass containers, and preserve at  $-20^\circ\text{C}$ .

**Human insulin desamide substance-containing TS** Dissolve 1.5 mg of Insulin Human (Genetical Recombination) in 1 mL of 0.01 mol/L hydrochloric acid TS, allow to stand at 25°C for 3 days, and when the procedure is run with this solution according to the conditions as directed in Purity (1) Related substances under Insulin Human (Genetical Recombination), the solution contains about 5% of the desamide substance.

**Human insulin dimer-containing TS** Allow to stand Insulin Human (Genetical Recombination) at 25°C for 10 days or more, and dissolve 4 mg of this in 1 mL of 0.01 mol/L hydrochloric acid TS.

**Hydralazine hydrochloride**  $\text{C}_8\text{H}_8\text{N}_4\cdot\text{HCl}$  [Same as the namesake monograph]

**Hydralazine hydrochloride for assay**  $\text{C}_8\text{H}_8\text{N}_4\cdot\text{HCl}$  [Same as the monograph Hydralazine Hydrochloride. When dried, it contains not less than 99.0% of hydralazine hydrochloride ( $\text{C}_8\text{H}_8\text{N}_4\cdot\text{HCl}$ ).]

**Hydrazine hydrate** See hydrazine monohydrate.

**Hydrazine monohydrate**  $\text{NH}_2\text{NH}_2\cdot\text{H}_2\text{O}$  [K 8871: 1980, Special class]

**Hydrazine sulfate** See hydrazinum sulfate.

**Hydrazinum sulfate**  $N_4H_6SO_4$  [K 8992, Special class]

**Hydrazinum sulfate TS** Dissolve 1.0 g of hydrazinum sulfate in water to make 100 mL.

**Hydrobromic acid** HBr [K 8509, Special class]

**Hydrochloric acid** HCl [K 8180, Special class]

**Hydrochloric acid-ammonium acetate buffer solution, pH 3.5** Dissolve 25 g of ammonium acetate in 45 mL of 6 mol/L hydrochloric acid TS, and add water to make 100 mL.

**Hydrochloric acid, dilute** Dilute 23.6 mL of hydrochloric acid with water to make 100 mL (10%).

**Hydrochloric acid-ethanol TS** See hydrochloric acid-ethanol (95) TS.

**Hydrochloric acid-ethanol (95) TS** Dilute 23.6 mL of hydrochloric acid with ethanol to make 100 mL.

**Hydrochloric acid-potassium chloride buffer solution, pH 2.0** To 10.0 mL of 0.2 mol/L hydrochloric acid VS add 88.0 mL of 0.2 mol/L potassium chloride TS, adjust the pH to  $2.0 \pm 0.1$  by adding 0.2 mol/L hydrochloric acid VS, then add water to make 200 mL.

**Hydrochloric acid, purified** Add 0.3 g of potassium permanganate to 1000 mL of diluted hydrochloric acid (1 in 2), distil, discard the first 250 mL of the distillate, and collect the following 500 mL of the distillate.

**0.001 mol/L Hydrochloric acid TS** Dilute 10 mL of 0.1 mol/L hydrochloric acid TS with water to make 1000 mL.

**0.01 mol/L Hydrochloric acid TS** Dilute 100 mL of 0.1 mol/L hydrochloric acid TS with water to make 1000 mL.

**0.02 mol/L Hydrochloric acid TS** Dilute 100 mL of 0.2 mol/L hydrochloric acid TS with water to make 1000 mL.

**0.05 mol/L Hydrochloric acid TS** Dilute 100 mL of 0.5 mol/L hydrochloric acid TS with water to make 1000 mL.

**0.1 mol/L Hydrochloric acid TS** Dilute 100 mL of 1 mol/L hydrochloric acid TS with water to make 1000 mL.

**0.2 mol/L Hydrochloric acid TS** Dilute 18 mL of hydrochloric acid with water to make 1000 mL.

**0.5 mol/L Hydrochloric acid TS** Dilute 45 mL of hydrochloric acid with water to make 1000 mL.

**1 mol/L Hydrochloric acid TS** Dilute 90 mL of hydrochloric acid with water to make 1000 mL.

**2 mol/L Hydrochloric acid TS** Dilute 180 mL of hydrochloric acid with water to make 1000 mL.

**3 mol/L Hydrochloric acid TS** Dilute 270 mL of hydrochloric acid with water to make 1000 mL.

**5 mol/L Hydrochloric acid TS** Dilute 450 mL of hydrochloric acid with water to make 1000 mL.

**6 mol/L Hydrochloric acid TS** Dilute 540 mL of hydrochloric acid with water to make 1000 mL.

**7.5 mol/L Hydrochloric acid TS** Dilute 675 mL of hydrochloric acid with water to make 1000 mL.

**10 mol/L Hydrochloric acid TS** Dilute 900 mL of hydrochloric acid with water to make 1000 mL.

**Hydrocortisone**  $C_{21}H_{30}O_5$  [Same as the namesake monograph]

**Hydrocortisone acetate**  $C_{23}H_{32}O_6$  [Same as the namesake monograph]

**Hydrocotarnine hydrochloride for assay**  
 $C_{12}H_{15}NO_3 \cdot HCl \cdot H_2O$  [Same as the monograph Hydrocotarnine Hydrochloride. When dried, it contains not less than 99.0% of hydrocotarnine hydrochloride ( $C_{12}H_{15}NO_3 \cdot HCl \cdot H_2O$ ).]

**Hydrofluoric acid** HF [K 8819, Special class] It contains not less than 46.0% of HF.

**Hydrogen**  $H_2$  [K 0512, Standard substance, Third class] It contains not less than 99.99% of  $H_2$ .

**Hydrogen chloride-ethanol TS** See hydrogen chloride-ethanol (99.5) TS.

**Hydrogen chloride-ethanol (99.5) TS** Pass dry hydrogen chloride, which is generated by slowly adding 100 mL of sulfuric acid dropwise to 100 mL of hydrochloric acid and dried by washing with sulfuric acid, through 75 g of ethanol (99.5) cooled in an ice bath until the increase in mass has reached 25 g. Prepare before use.

**Hydrogen hexachloroplatinate (IV) hexahydrate**  
 $H_2PtCl_6 \cdot 6H_2O$  [K 8153, Special class]

**Hydrogen hexachloroplatinate (IV)-potassium iodide TS** To 3 mL of hydrogen hexachloroplatinate (IV) TS add 97 mL of water and 100 mL of a solution of potassium iodide (3 in 50). Prepare before use.

**Hydrogen hexachloroplatinate (IV) TS** Dissolve 2.6 g of chloroplatinic acid in water to make 20 mL (0.25 mol/L).

**Hydrogen peroxide TS** Dilute 1 volume of hydrogen peroxide (30) with 9 volumes of water. Prepare before use (3%).

**Hydrogen peroxide TS, dilute** Dilute 1 mL of hydrogen peroxide (30) with 500 mL of water, and dilute 5 mL of this solution with water to make 100 mL. Prepare before use.

**Hydrogen peroxide water, strong** See hydrogen peroxide (30).

**Hydrogen peroxide (30)**  $H_2O_2$  [K 8230, Hydrogen peroxide, Special class, Concentration: 30.0 - 35.5%].

**Hydrogen sulfide**  $H_2S$  Colorless, poisonous gas, heavier than air. It dissolves in water. Prepare by treating iron (II) sulfide heptahydrate with dilute sulfuric acid or dilute hydrochloric acid. Other sulfides yielding hydrogen sulfide with dilute acids may be used.

**Hydrogen sulfide TS** A saturated solution of hydrogen sulfide. Prepare by passing hydrogen sulfide into cold water. Preserve in well-filled, light-resistant bottles, in a dark, cold place.

**Hydrogen tetrachloroaurate (III) tetrahydrate**  
 $HAuCl_4 \cdot 4H_2O$  [K 8127, Special class]

**Hydrogen tetrachloroaurate (III) tetrahydrate TS** Dissolve 1 g of hydrogen tetrachloroaurate (III) tetrahydrate in 35 mL of water (0.2 mol/L).

**Hydroiodic acid** HI [K 8917, Special class]

**Hydrophilic silica gel for liquid chromatography** Dio-

lized porous silica gel prepared for liquid chromatography (5–10  $\mu\text{m}$  in particle diameter).

**Hydroquinone**  $\text{C}_6\text{H}_4(\text{OH})_2$  [K 8738, Special class]

**Hydroxocobalamin acetate**  $\text{C}_{62}\text{H}_{89}\text{CoN}_{13}\text{O}_{15}\text{P}\cdot\text{C}_2\text{H}_4\text{O}_2$   
Dark red crystals or powder.

*Loss on drying:* not more than 12% (0.05 g, in vacuum not exceeding 0.67 kPa, phosphorus (V) oxide, 100°C, 6 hours).

*Content:* not less than 98.0%. *Assay*—Proceed as directed in the Assay under Hydroxocobalamin Acetate.

***m*-Hydroxyacetophenone**  $\text{C}_8\text{H}_8\text{O}_2$  White to light yellowish white crystalline powder.

*Melting point:* about 96°C

*Purity* Related substances—Perform the test with 10  $\mu\text{L}$  of a solution of *m*-hydroxyacetophenone in 0.1 mol/L phosphate buffer solution, pH 4.5 (1 in 15,000) as directed in the Assay under Cefalexin: Any obstructive peaks for determination of Cefalexin are not observed.

***p*-Hydroxyacetophenone**  $\text{C}_8\text{H}_8\text{O}_2$  White to pale yellow crystals or crystalline powder. It is freely soluble in methanol.

*Melting point:* 107 — 111°C

*Purity*—Weigh 1 mg of *p*-hydroxyacetophenone, add methanol and dissolve to make exactly 10 mL, and use this solution as the sample solution. Perform the test with 20  $\mu\text{L}$  of the sample solution as directed under the Liquid Chromatography according to the Component determination under Peony Root: the total area of the peaks other than the peak of *p*-hydroxyacetophenone from the sample solution is not larger than 3/100 of the total area of the peaks other than the solvent peak.

**3-Hydroxybenzoic acid**  $\text{HOC}_6\text{H}_4\text{COOH}$  White, crystals or crystalline powder.

*Identification*—Determine the infrared absorption spectrum according to the paste method under the Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 3300  $\text{cm}^{-1}$ , 1690  $\text{cm}^{-1}$ , 1600  $\text{cm}^{-1}$ , 1307  $\text{cm}^{-1}$ , 1232  $\text{cm}^{-1}$  and 760  $\text{cm}^{-1}$ .

*Melting point:* 203 – 206°C

*Purity* Clarity—Dissolve 1 g of 3-hydroxybenzoic acid in 20 mL of methanol: the solution is clear.

*Content:* not less than 99.0%. *Assay*—Weigh accurately about 0.2 g of 3-hydroxybenzoic acid, dissolve in 20 mL of diluted ethanol (95) (1 in 2), and titrate with 0.1 mol/L sodium hydroxide VS (indicator: 3 drops of cresol red TS) until the color of the solution changes from yellow to dark orange-red. Perform a blank determination and make any necessary correction.

Each mL of 0.1 mol/L sodium hydroxide VS  
= 13.812 mg of  $\text{C}_7\text{H}_6\text{O}_3$

***p*-Hydroxybenzoic acid** See parahydroxybenzoic acid.

***N*-2-Hydroxyethylpiperazine-*N'*-2-ethanesulfonic acid**  $\text{C}_8\text{H}_{18}\text{N}_2\text{O}_4\text{S}$  White crystalline powder.

*Purity* Clarity and color of solution—Dissolve 11.9 g of *N*-2-hydroxyethylpiperazine-*N'*-2-ethanesulfonic acid in 50 mL of water: the solution is clear and colorless.

*Content:* not less than 99.0%. *Assay*—Weigh accurately about 1 g of *N*-2-hydroxyethylpiperazine-*N'*-2-ethanesulfonic acid, dissolve in 60 mL of water, and titrate with 0.5 mol/L sodium hydroxide VS (Potentiometric titration).

Each mL of 0.5 mol/L sodium hydroxide VS  
= 119.15 mg of  $\text{C}_8\text{H}_{18}\text{N}_2\text{O}_4\text{S}$

***d*-3-Hydroxy-*cis*-2,3-dihydro-5-[2-(dimethylamino)ethyl]-2-(4-methoxyphenyl)-1,5-benzothiazepine-4(5*H*)-one hydrochloride**  $\text{C}_{20}\text{H}_{24}\text{N}_2\text{O}_3\text{S}\cdot\text{HCl}$  To 9 g of diltiazem hydrochloride add 50 mL of ethanol (99.5), and dissolve by heating at 80°C. To this solution add slowly 50 mL of a solution of potassium hydroxide in ethanol (99.5) (33 in 500) dropwise, and heat for 4 hours with stirring. Cool in an ice bath, filter, and evaporate the filtrate to dryness. Dissolve the residue in ethanol (99.5), add slowly a solution of hydrochloric acid in ethanol (99.5) (59 in 250) to make acidic, and filter. Add diethyl ether slowly to the filtrate, and filter the crystals produced. To the crystals add ethanol (99.5), heat to dissolve, add 0.5 g of activated charcoal, allow to stand, and filter. After cooling the filtrate in an ice-methanol bath, filter the crystals formed, and wash with diethyl ether. Further, add ethanol (99.5) to the crystals, and heat to dissolve. After cooling, filter the crystals produced, and dry under reduced pressure. White crystals or crystalline powder, having a slight, characteristic odor.

*Purity*—Dissolve 0.050 g of *d*-3-hydroxy-*cis*-2,3-dihydro-5-[2-(dimethylamino)ethyl]-2-(*p*-methoxyphenyl)-1,5-benzothiazepine-4(5*H*)-one hydrochloride in chloroform to make exactly 10 mL, and use this solution as the sample solution. Perform the test with the sample solution as directed under the Thin-layer Chromatography. Spot 20  $\mu\text{L}$  of the sample solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of ethanol (99.5), chloroform, water and acetic acid (100) (12:10:3:1) to a distance of about 13 cm, and air-dry the plate. Spray evenly iodine TS on the plate: any spot other than the principal spot does not appear.

*Water:* not more than 1.0% (0.5 g).

*Content:* not less than 99.0%, calculated on the anhydrous basis. *Assay*—Weigh accurately about 0.5 g of *d*-3-hydroxy-*cis*-2,3-dihydro-5-[2-(dimethylamino)ethyl]-2-(*p*-methoxyphenyl)-1,5-benzothiazepine-4(5*H*)-one hydrochloride, dissolve in 2.0 mL of formic acid, add 60 mL of acetic anhydride, and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination in the same manner.

Each mL of 0.1 mol/L perchloric acid VS  
= 40.89 mg of  $\text{C}_{20}\text{H}_{24}\text{N}_2\text{O}_3\text{S}\cdot\text{HCl}$

***d*-3-Hydroxy-*cis*-2,3-dihydro-5-[2-(dimethylamino)ethyl]-2-(*p*-methoxyphenyl)-1,5-benzothiazepine-4(5*H*)-one hydrochloride** See *d*-3-hydroxy-*cis*-2,3-dihydro-5-[2-(dimethylamino)ethyl]-2-(4-methoxyphenyl)-1,5-benzothiazepine-4(5*H*)-one hydrochloride.

**2-Hydroxy-1-(2-hydroxy-4-sulfo-1-naphthylazo)-3-naphthoic acid**  $\text{C}_{21}\text{H}_{14}\text{N}_2\text{O}_7\text{S}$  [K 8776, Special class]

**Hydroxylamine hydrochloride** See hydroxylammonium chloride.

**Hydroxylamine hydrochloride-ferric chloride TS** See hydroxylammonium chloride-iron (III) chloride TS.

**Hydroxylamine hydrochloride TS** See hydroxylammonium chloride TS.

**Hydroxylamine perchlorate**  $\text{NH}_2\text{OH}\cdot\text{HClO}_4$  Hygroscopic, white crystals. Dissolves in water and in ethanol (95).