Prepare the control solution with 0.40 mL of 0.005 mol/L sulfuric acid VS (not more than 0.096%).

(4) Heavy metals—Proceed with 2.0 g of Dimorpholamine 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).

Loss on drying Not more than 0.5% (1 g, in vacuum, phosphorus (V) oxide, 8 hours).

Residue on ignition Not more than 0.10% (1 g).

Assay Weigh accurately about 0.6 g of Dimorpholamine, previously dried, and dissolve in 10 mL of acetic anhydride and 40 mL of nitrobenzene. Titrate with 0.1 mol/L perchloric acid VS until the color of the solution changes from red through purple to blue-purple (indicator: 5 drops of neutral red TS). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 39.855 mg of $C_{20}H_{38}N_4O_4$

Containers and storage Containers—Tight containers.
Storage—Light-resistant.

Dimorpholamine Injection

ジモルホラミン注射液

Dimorpholamine Injection is an aqueous solution for injection. It contains not less than 95% and not more than 105% of the labeled amount of dimorpholamine ($C_{20}H_{38}N_4O_4$: 398.54).

Method of preparation Prepare as directed under Injections, with Dimorpholamine.

Description Dimorpholamine Injection is a clear, colorless liquid.

Identification (1) To a volume of Dimorpholamine Injection, equivalent to 0.1 g of Dimorpholamine according to the labeled amount, add 3 drops of Dragendorff's TS: an orange color develops.

(2) To a volume of Dimorpholamine Injection, equivalent to 0.05 g of Dimorpholamine according to the labeled amount, add 1 mL of dilute hydrochloric acid, and evaporate on a water bath to dryness. Dissolve this residue in 2 mL of hydrochloric acid, and proceed as directed in the Identification (3) under Dimorpholamine.

Assay Measure exactly a volume of Dimorpholamine Injection, equivalent to about 0.03 g of dimorpholamine ($C_{20}H_{38}N_4O_4$), and add water to make exactly 200 mL. Pipet 1 mL of this solution, shake with exactly 4 mL of the internal standard solution for 5 minutes, and use this solution as the sample solution. Separately, weigh accurately about 0.15 g of dimorpholamine for assay, previously dried in a desiccator (in vacuum, phosphorus (V) oxide) for 8 hours, and dissolve in water to make exactly 1000 mL. Pipet 1 mL of this solution, shake with exactly 4 mL of the internal standard solution for 5 minutes, and use this solution as the standard solution. Perform the test with 10 μm each of the sample 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 area of dimorpholamine to that of the internal standard, respectively.

Amount (mg) of dimorpholamine ($C_{20}H_{38}N_4O_4$)
= amount (mg) of dimorpholamine for assay
$\times \frac{Q_T}{Q_S} \times \frac{1}{5}$

Internal standard solution—A solution of butyl parahydroxybenzoate in acetonitrile (1 in 25,000).

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 216 nm).

Column: A stainless steel column about 4 mm in inside 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 (1:1).

Flow rate: Adjust the flow rate so that the retention time of dimorpholamine is about 4 minutes.

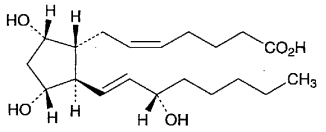
Selection of column: Proceed with 10 μL of the standard solution under the above operating conditions, and calculate the resolution. Use a column giving elution of dimorpholamine and the internal standard in this order with the resolution between these peaks being not less than 2.0.

Containers and storage Containers—Hermetic containers.

Dinoprost

Prostaglandin $F_{2\alpha}$

ジノプロスト



$C_{20}H_{34}O_5$: 354.48
(5*Z*)-7-{(1*R*,2*R*,3*R*,5*S*)-3,5-Dihydroxy-2-[(1*E*,3*S*)-3-hydroxyoct-1-en-1-yl]cyclopentyl}hept-5-enoic acid
[*551-11-1*]

Dinoprost contains not less than 98.5% of $C_{20}H_{34}O_5$, calculated on the anhydrous basis.

Description Dinoprost occurs as white, waxy masses or powder, or a clear, colorless to light yellow and viscous liquid. It is odorless.

It is very soluble in *N,N*-dimethylformamide, freely soluble in methanol, in ethanol (99.5) and in diethyl ether, and very slightly soluble in water.

Identification (1) To 5 mg of Dinoprost add 2 mL of sulfuric acid, and dissolve by shaking for 5 minutes: a dark red color develops. To this solution add 30 mL of sulfuric acid: an orange color develops with a green fluorescence.

(2) Dissolve 1 mg of Dinoprost in 50 mL of diluted sul-

furic acid (7 in 10), and warm in a water bath heated at 50°C for 40 minutes. After cooling, determine the absorption spectrum of the solution as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wavelengths.

(3) Warm Dinoprost at 40°C to effect a liquid, and determine the infrared absorption spectrum of the liquid as directed in the liquid film method under the Infrared Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibits similar intensities of absorption at the same wave numbers.

Optical rotation $[\alpha]_D^{20}$: +24 - +31° (0.2 g, ethanol (99.5), 10 mL, 100 mm).

Purity **(1)** Clarity and color of solution—Dissolve 0.20 g of Dinoprost in 5 mL of ethanol (99.5): the solution is clear and colorless to pale yellow.

(2) Related substances—Dissolve 0.010 g of Dinoprost in 2 mL of methanol, add water to make 10 mL, and use this solution as the sample solution. Pipet 3 mL of the sample solution, add diluted methanol (1 in 5) to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 10 μL each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions. Determine each peak area of these solutions by the automatic integration method: the total area of the peaks other than the peak of dinoprost from the sample solution is not larger than the peak area of dinoprost from the standard solution.

Operating conditions—

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

Column: A stainless steel column about 5 mm in inside 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 25°C.

Mobile phase: A mixture of 0.02 mol/L potassium dihydrogenphosphate TS and acetonitrile (5:2).

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

Selection of column: Dissolve 0.01 g each of isopropyl parahydroxybenzoate and propyl parahydroxybenzoate in 2 mL of methanol, and andd water to make 10 mL. To 1 mL of this solution add diluted methanol (1 in 5) to make 30 mL, proceed with 10 μL of this solution under the above operating conditions, and calculate the resolution. Use a column giving elution of isopropyl parahydroxybenzoate and propyl parahydroxybenzoate in this order with the resolution between these peaks being not less than 2.5.

Detection sensitivity: Adjust the detection sensitivity so that the peak height of dinoprost from the standard solution composes 5% to 15% of the full scale.

Time span of measurement: About 1.5 times as long as the retention time of dinoprost after the solvent peak.

Water Not more than 0.5% (0.3 g, direct titration).

Assay Weigh accurately about 0.05 g of Dinoprost, dissolve in 30 mL of *N,N*-dimethylformamide, and titrate with 0.02 mol/L tetramethylammonium hydroxide VS under a stream of nitrogen (potentiometric titration). Perform a blank determination, and make any necessary correction.

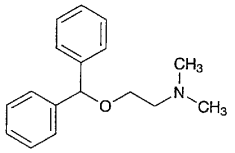
Each mL of 0.02 mol/L tetramethylammonium hydroxide VS
= 7.090 mg of $C_{20}H_{34}O_5$

Containers and storage Containers—Tight containers.

Storage—Light-resistant, and in a place not exceeding 5°C.

Diphenhydramine

ジフェンヒドラミン



$C_{17}H_{21}NO$: 255.35

N-(2-Benzhydryloxyethyl)-*N,N*-dimethylamine [*58-73-1*]

Diphenhydramine contains not less than 96.0% of $C_{17}H_{21}NO$.

Description Diphenhydramine is a clear, light yellow to yellow liquid. It has a characteristic odor, and has a burning taste at first, followed by a slight sensation of numbness on the tongue.

It is miscible with acetic anhydride, with acetic acid (100), with ethanol (95) and with diethyl ether.

It is very slightly soluble in water.

Boiling point: about 162°C (in vacuum, 0.67 kPa).

Refractive index n_D^{20}: about 1.55

It is gradually affected by light.

Identification **(1)** To 0.05 g of Diphenhydramine add 2 mL of sulfuric acid: an orange-red precipitate is produced immediately, and its color changes to red-brown on standing. Add carefully 2 mL of water to this solution: the intensity of the color changes, but the color tone does not change.

(2) Dissolve 0.1 g of Diphenhydramine in 10 mL of dilute ethanol, add an excess of a saturated solution of 2,4,6-trinitrophenol in dilute ethanol with stirring, and cool in ice. Collect the produced crystals, recrystallize from dilute ethanol, and dry at 105°C for 30 minutes: the crystals melt between 128°C and 133°C.

Specific gravity d_{20}^{20}: 1.013 - 1.020

Purity **(1)** β-Dimethylaminoethanol—Dissolve 1.0 g of Diphenhydramine in 20 mL of diethyl ether, and extract with two 10-mL portions of water with thorough shaking. Combine the water extracts, and add 2 drops of phenolphthalein TS and 1.0 mL of 0.05 mol/L sulfuric acid VS: no red color develops.

(2) Benzohydrol—Transfer 1.0 g of Diphenhydramine to a separator, dissolve in 20 mL of diethyl ether, and extract with two 25-mL portions of diluted hydrochloric acid (1 in 15) with thorough shaking. Separate the diethyl ether layer, evaporate slowly on a water bath, and dry in a desiccator (in vacuum, silica gel) for 2 hours: the mass of the residue is not more than 0.020 g.

(3) Heavy metals—Proceed with 1.0 g of Diphenhydramine according to Method 2, and perform the test. Prepare