

iopanoic acid ($C_{11}H_{12}I_3NO_2$; 570.93).

Method of preparation Prepare as directed under Tablets, with Iopanoic Acid.

Identification (1) Weigh a quantity of powdered Iopanoic Acid Tablets, equivalent to 0.25 g of Iopanoic Acid according to the labeled amount, shake with 10 mL of acetone for 5 minutes, and centrifuge. Evaporate 5 mL of the supernatant liquid on a water bath to dryness, dry the residue at $105^\circ C$ for 1 hour, dissolve 1 mg of the dried matter in 1 mL of acetone, and add 3 mL of dilute hydrochloric acid: the solution responds to the Qualitative Tests for primary aromatic amines, and a red color develops.

(2) Heat 0.1 g of the dried matter obtained in (1) over a flame: a purple gas is evolved.

(3) Determine the infrared absorption spectrum of the dried matter obtained in (1) as directed in the potassium bromide disk method under the Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 3435 cm^{-1} , 3345 cm^{-1} , 1290 cm^{-1} , 1229 cm^{-1} , 939 cm^{-1} and 872 cm^{-1} .

Purity Soluble halides—To a quantity of powdered Iopanoic Acid Tablets, equivalent to 2.5 g of Iopanoic Acid according to the labeled amount, add 20 mL of water and 2.5 mL of ammonia TS, and add 20 mL of dilute nitric acid and water to make 100 mL. Proceed as directed in the purity (1) under Iopanoic Acid.

Assay Weigh accurately not less than 20 Iopanoic Acid Tablets, and powder. Weigh accurately a portion of the powder, equivalent to about 0.4 g of iopanoic acid ($C_{11}H_{12}I_3NO_2$), add 20 mL of acetone, centrifuge after thorough shaking, and filter the supernatant liquid. Repeat 4 times the same procedures with each 10-mL portion of acetone in each case, filter through the same filter paper, combine the filtrates, and evaporate on a water bath to dryness. Cool, add 1 g of zinc powder and 10 mL of acetic acid (100) to the residue, and proceed as directed in the Assay under Iopanoic Acid.

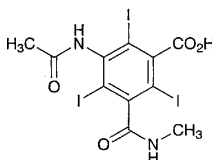
Each mL of 0.1 mol/L silver nitrate VS
= 19.031 mg of $C_{11}H_{12}I_3NO_2$

Containers and storage Containers—Tight containers.

Storage—Light-resistant.

Iotalamic Acid

イオタラム酸



$C_{11}H_9I_3N_2O_4$: 613.91

3-Acetylamino-2,4,6-triiodo-5-(methylaminocarbonyl)-benzoic acid [2276-90-6]

Iotalamic Acid, when dried, contains not less than

99.0% of $C_{11}H_9I_3N_2O_4$.

Description Iotalamic Acid occurs as a white powder. It is odorless.

It is sparingly soluble in ethanol (95), very slightly soluble in water, and practically insoluble in diethyl ether.

It dissolves in sodium hydroxide TS.

It gradually colored by light.

Identification (1) Heat 0.1 g of Iotalamic Acid over a flame: a purple gas is evolved.

(2) Determine the infrared spectrum of Iotalamic Acid, previously dried, as directed in the potassium bromide disk method under the Infrared Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wave numbers.

Purity (1) Clarity and color of solution—Dissolve 2.0 g of Iotalamic Acid in 10 mL of sodium hydroxide TS: the solution is clear and colorless.

(2) Primary aromatic amines—To 0.50 g of Iotalamic Acid add 15 mL of water, and dissolve it in 1 mL of sodium hydroxide TS while ice-cooling. Add 4 mL of a solution of sodium nitrite (1 in 100) to the solution, immediately add 12 mL of 1 mol/L hydrochloric acid TS, and shake gently. Then allow the mixture to stand for exactly 2 minutes, add 8 mL of ammonium amidosulfate TS, and shake occasionally for 5 minutes. Add 3 drops of a solution of 1-naphthol in ethanol (95) (1 in 10), allow to stand for 1 minute, add 3.5 mL of ammonia-ammonium chloride buffer solution, pH 10.7, mix, and immediately add water to make 50 mL. Determine within 20 minutes the absorbance of this solution at 485 nm as directed under the Ultraviolet-visible Spectrophotometry, using a solution, prepared in the same manner, as the blank: the absorbance is not more than 0.25.

(3) Soluble halides—Dissolve 0.5 g of Iotalamic Acid in 20 mL of diluted ammonia TS (1 in 40), add 6 mL of dilute nitric acid, shake, allow to stand for 5 minutes, and filter. Transfer the filtrate to a Nessler tube, wash the residue with 20 mL of water, combine the filtrate and the washings, and add water to make 50 mL. Proceed as directed for the Chloride Limit Test using this solution as the test solution. Prepare the control solution as follows: to 0.10 mL of 0.01 mol/L hydrochloric acid VS and add 20 mL of diluted ammonia TS (1 in 40), 6 mL of dilute nitric acid and water to make 50 mL.

(4) Iodine—Dissolve 0.20 g of Iotalamic Acid in 2.0 mL of sodium hydroxide TS, add 2.5 mL of 0.5 mol/L sulfuric acid TS, and allow to stand for 10 minutes with occasional shaking. Add 5 mL of chloroform, shake well, and allow to stand: the chloroform layer remains colorless.

(5) Heavy metals—Proceed with 1.0 g of Iotalamic 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).

(6) Arsenic—Prepare the test solution with 0.6 g of Iotalamic Acid according to Method 3, and perform the test using Apparatus B (not more than 3.3 ppm).

Loss on drying Not more than 0.5% (1 g, $105^\circ C$, 4 hours).

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

Assay Weigh accurately about 0.4 g of Iotalamic Acid, previously dried, place it in a saponification flask, dissolve

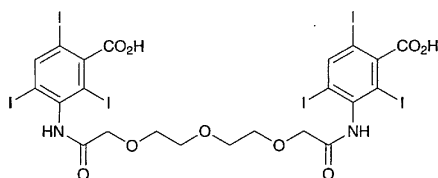
in 40 mL of sodium hydroxide TS, add 1 g of zinc powder, and heat for 30 minutes under a reflux condenser. Cool, filter, wash the flask and the filter paper with 50 mL of water, and combine the washings and the filtrate. Add 5 mL of acetic acid (100) to this solution, and titrate with 0.1 mol/L silver nitrate VS, until the color of the precipitate changes from yellow to green (indicator: 1 mL of tetrabromophenolphthalein ethyl ester TS).

Each mL of 0.1 mol/L silver nitrate VS
= 20.464 mg of $C_{11}H_9I_3N_2O_4$

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

Iotroxic Acid

イオトロクス酸



$C_{22}H_{18}I_6N_2O_9$; 1215.81
3,3'-(3,6,9-Trioxaundecanedioyl)diiminobis-(2,4,6-triiodobenzoic acid) [51022-74-3]

Iotroxic Acid contains not less than 98.5% of $C_{22}H_{18}I_6N_2O_9$, calculated on the anhydrous basis.

Description Iotroxic Acid occurs as a white crystalline powder.

It is soluble in methanol, slightly soluble in ethanol (95), and practically insoluble in water and in diethyl ether.

It is gradually colored by light.

Identification (1) Heat 0.1 g of Iotroxic Acid over a flame: a purple gas evolves.

(2) Dissolve a suitable amount of Iotroxic Acid in a suitable amount of methanol, evaporate the methanol under reduced pressure, and determine the infrared absorption spectrum of the residue so obtained as directed in the potassium bromide disk method under the Infrared Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wave numbers.

Purity (1) Clarity and color of solution—Dissolve 1.0 g of Iotroxic Acid in 10 mL of diluted sodium hydroxide TS (1 in 5): the solution is clear and colorless.

(2) Primary aromatic amines—Dissolve 0.20 g of Iotroxic Acid in 5 mL of water and 1 mL of sodium hydroxide TS, add 4 mL of a solution of sodium nitrite (1 in 100) and 10 mL of 1 mol/L hydrochloric acid TS, mix, and allow to stand for 2 minutes. Add 5 mL of ammonium amidosulfate TS, shake well, allow to stand for 1 minute, then add 0.4 mL of a solution of α -naphthol in ethanol (95) (1 in 10), 15 mL of sodium hydroxide TS and water to make exactly 50 mL. Read the absorbance of this solution at 485 nm as directed under the Ultraviolet-visible Spectrophotometry,

using a blank solution obtained in the same manner as above: the absorbance is not more than 0.22.

(3) Iodine—Dissolve 0.20 g of Iotroxic Acid in 2.0 mL of sodium hydrogen carbonate TS, add 5 mL of toluene, mix well, and allow to stand: the toluene layer is colorless.

(4) Free iodine ion—Weigh accurately about 5.0 g of Iotroxic Acid, dissolve in 12 mL of a solution of meglumine (3 in 20), add water to make 70 mL, and adjust the pH to about 4.5 with acetic acid (100). To this solution add 2 mL of 0.1 mol/L sodium chloride TS, and titrate with 0.001 mol/L silver nitrate VS (potentiometric titration).

Each mL of 0.001 mol/L silver nitrate
= 0.12690 mg of I

Content of iodine ion in Iotroxic Acid, calculated on the anhydrous basis, is not more than 0.004%.

(5) Heavy metals—Heat strongly 1.0 g of Iotroxic Acid as directed under the Residue on Ignition Test, then proceed 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).

(6) Related substances—Dissolve 0.15 g of Iotroxic Acid in 10 mL of methanol, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add methanol to make exactly 200 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 10 μ L each of the sample 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, acetone and formic acid (6:4:1) to a distance of about 15 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): the spots other than the principal spot from the sample solution are not more intense than the spot from the standard solution.

Water 1.0 – 2.0% (0.5 g, direct titration).

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

Assay Weigh accurately about 0.5 g of Iotroxic Acid, dissolve in 40 mL of sodium hydroxide TS in a saponification flask, add 1 g of zinc powder, and boil for 30 minutes under a reflux condenser. After cooling, filter, wash the flask and the filter paper with 50 mL of water, and combine the washings to the filtrate. To this solution add 5 mL of acetic acid (100), and titrate with 0.1 mol/L silver nitrate VS (potentiometric titration).

Each mL of 0.1 mol/L silver nitrate VS
= 20.264 mg of $C_{22}H_{18}I_6N_2O_9$

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

Ipratropium Bromide

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