

the gas evolved does not turn red litmus paper to blue.

(5) Cyanide—Dissolve 0.5 g of Potassium Iodide in 10 mL of water. To 5 mL of this solution add 1 drop of iron (II) sulfate TS and 2 mL of sodium hydroxide TS, warm, then add 4 mL of hydrochloric acid: no green color develops.

(6) Iodate—Dissolve 0.5 g of Potassium Iodide in 10 mL of freshly boiled and cooled water, and add 2 drops of dilute sulfuric acid and 1 drop of starch TS: no blue color develops immediately.

(7) Heavy metals—Proceed with 2.0 g of Potassium Iodide 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).

(8) Barium—Dissolve 0.5 g of Potassium Iodide in 10 mL of water, add 1 mL of dilute sulfuric acid, and allow to stand for 5 minutes: no turbidity is produced.

(9) Sodium—Dissolve 1.0 g of Potassium Iodide in 10 mL of water, and perform the Flame Coloration Test (1): a yellow color develops, but does not persist.

(10) Arsenic—Prepare the test solution with 0.40 g of Potassium Iodide according to Method 1, and perform the test using Apparatus B (not more than 5 ppm).

**Loss on drying** Not more than 1.0% (2 g, 105°C, 4 hours).

**Assay** Weigh accurately about 0.5 g of Potassium Iodide, previously dried, in an iodine flask, dissolve in 10 mL of water, add 35 mL of hydrochloric acid and 5 mL of chloroform, and titrate with 0.05 mol/L potassium iodate VS with shaking until the red-purple color of the chloroform layer disappears. The end point is reached when the red-purple color does not reappear in the chloroform layer within 5 minutes after the layer has been decolorized.

Each mL of 0.05 mol/L potassium iodate VS  
= 16.600 mg of KI

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

## Potassium Permanganate

過マンガン酸カリウム

KMnO<sub>4</sub>: 158.03

Potassium Permanganate, when dried, contains not less than 99.0% of KMnO<sub>4</sub>.

**Description** Potassium Permanganate occurs as dark purple crystals and has a metallic luster.

It is soluble in water.

A solution of Potassium Permanganate (1 in 1000) has a slightly sweet, astringent taste.

**Identification** A solution of Potassium Permanganate (1 in 100) responds to the Qualitative Tests for permanganate.

**Purity** (1) Water-insoluble substances—Dissolve 2.0 g of Potassium Permanganate, previously powdered, in 200 mL of water. Filter the insoluble substances through a tared glass filter (G4), wash with water until the last washing shows no color, and dry at 105°C for 2 hours: the mass of

the residue is not more than 4 mg.

(2) Arsenic—Dissolve 0.40 g of Potassium Permanganate in 10 mL of water, add 1 mL of sulfuric acid, add hydrogen peroxide (30) dropwise until the solution remains colorless, and evaporate on a sand bath nearly to dryness. Dissolve the residue in 5 mL of water, and perform the test with this solution as the test solution using Apparatus B: the color produced is not more intense than the following standard color.

**Standard color:** To 10 mL of water add 1 mL of sulfuric acid and the same volume of hydrogen peroxide (30) as used for the preparation of the test solution. Evaporate the solution on a sand bath nearly to dryness, add 2.0 mL of Standard Arsenic Solution and water to make 5 mL, and carry out the test with this solution in the same manner as the test solution (not more than 5 ppm).

**Loss on drying** Not more than 0.5% (1 g, silica gel, 18 hours).

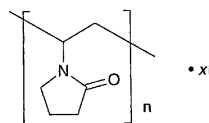
**Assay** Weigh accurately about 0.6 g of Potassium Permanganate, previously dried, dissolve in water to make exactly 200 mL, and use this solution as the sample solution. Pipet 25 mL of 0.05 mol/L oxalic acid VS into a 500-mL conical flask, add 200 mL of diluted sulfuric acid (1 in 20), and keep at a temperature between 30°C and 35°C. Transfer the sample solution to a buret. Add quickly 23 mL of the sample solution from the buret to the flask while shaking gently, and then allow the flask to stand until the red color disappears. Warm the mixture to a temperature between 55°C and 60°C, and continue the titration slowly until the red color persists for 30 seconds.

Each mL of 0.05 mol/L oxalic acid VS  
= 3.1607 mg of KMnO<sub>4</sub>

**Containers and storage** Containers—Tight containers.

## Povidone-Iodine

ポビドンヨード



(C<sub>6</sub>H<sub>9</sub>NO)<sub>n</sub>·xI

Poly[(2-oxopyrrolidin-1-yl)ethylene] iodine [25655-41-8]

Povidone-Iodine is a complex of iodine with 1-vinyl-2-pyrrolidone polymer.

It contains not less than 9.0% and not more than 12.0% of available iodine (I: 126.90), and not less than 9.5% and not more than 11.5% of nitrogen (N: 14.01), calculated on the dried basis.

**Description** Povidone-Iodine occurs as a dark red-brown powder. It has a faint, characteristic odor.

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

The pH of a solution of Povidone-Iodine (1 in 100) is between 1.5 and 3.5.

**Identification (1)** To 10 mL of diluted starch TS (1 in 10) add 1 drop of a solution of Povidone-Iodine (1 in 10): a deep blue color develops.

(2) To 1 mL of a solution of Povidone-Iodine (1 in 100) add 1 mL of sodium thiosulfate TS, and add 1 mL of ammonium thiocyanate-cobaltous nitrate TS and 2 drops of 1 mol/L hydrochloric acid TS: a blue color develops, and a blue precipitate is gradually formed.

**Purity (1)** Clarity and color of solution—Dissolve 0.30 g of Povidone-Iodine in 100 mL of water: the solution is clear and brown.

(2) Heavy metals—Proceed with 1.0 g of Povidone-Iodine 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) Arsenic—To 1.0 g of Povidone-Iodine in a crucible add 10 mL of a solution of magnesium nitrate hexahydrate in ethanol (95) (1 in 10), fire the ethanol to burn, and incinerate by gradual heating. If a carbonized substance remains, moisten it with a small quantity of nitric acid, and incinerate by ignition. After cooling, to the residue add 10 mL of dilute hydrochloric acid, dissolve by warming on a water bath, and perform the test with this solution using Apparatus B (not more than 2 ppm).

(4) Iodide ion—Weigh accurately about 0.5 g of Povidone-Iodine, dissolve in 100 mL of water, and add sodium hydrogensulfite TS until the color of iodine completely disappears. To this solution add exactly 25 mL of 0.1 mol/L silver nitrate VS, shake well with 10 mL of nitric acid, titrate the excess silver nitrate with 0.1 mol/L ammonium thiocyanate VS until the solution develops a red-brown color, and calculate the total amount of iodine (indicator: 1 mL of ammonium iron (III) sulfate TS). Perform a blank determination.

Each mL of 0.1 mol/L ammonium thiocyanate VS  
= 12.690 mg of I

Obtain the amount of iodide ion, calculated on the dried basis, by deducting the amount (%) of available iodine from the total amount (%) of iodine: it is not more than 6.6%.

**Loss on drying** Not more than 8.0% (1 g, 100°C, 3 hours).

**Residue on ignition** Not more than 0.05% (5 g).

**Assay (1)** Available iodine—Weigh accurately about 0.5 g of Povidone-Iodine, dissolve in 30 mL of water, and titrate with 0.02 mol/L sodium thiosulfate VS (indicator: 2 mL of starch TS).

Each mL of 0.02 mol/L sodium thiosulfate VS  
= 2.5381 mg of I

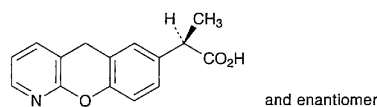
(2) Nitrogen—Weigh accurately about 1 g of Povidone-Iodine, dissolve in water to make exactly 50 mL, and use this solution as the sample solution. Pipet 1.0 mL of the sample solution, and perform the test as directed under the Nitrogen Determination.

Each mL of 0.005 mol/L sulfuric acid VS  
= 0.14007 mg of N

**Containers and storage** Containers—Tight containers.

## Pranoprofen

プラノプロフェン



$C_{15}H_{13}NO_3$ ; 255.27

(*RS*)-2-(10*H*-9-Oxa-1-azaanthracen-6-yl)-propanoic acid  
[52549-17-4]

Pranoprofen, when dried, contains not less than 98.5% of  $C_{15}H_{13}NO_3$ .

**Description** Pranoprofen occurs as a white to pale yellowish white crystalline powder.

It is freely soluble in *N,N*-dimethylformamide, soluble in acetic acid (100), sparingly soluble in methanol, slightly soluble in acetonitrile, in ethanol (95) and in acetic anhydride, very slightly soluble in diethyl ether, and practically insoluble in water.

A solution of Pranoprofen in *N,N*-dimethylformamide (1 in 30) shows no optical rotation.

**Identification (1)** Dissolve 0.02 g of Pranoprofen in 1 mol/L hydrochloric acid TS to make 100 mL, and dilute 10 mL of the solution with water to make 100 mL. 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.

(2) Determine the infrared absorption spectrum of Pranoprofen 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.

**Melting point** 186 – 190°C

**Purity (1)** Chloride—Dissolve 0.5 g of Pranoprofen in 40 mL of methanol, and 6 mL of dilute nitric acid, and add water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution as follows. To 0.30 mL of 0.01 mol/L hydrochloric acid add 40 mL of methanol, 6 mL of dilute nitric acid and water to make 50 mL (not more than 0.021%).

(2) Heavy metals—Proceed with 2.0 g of Pranoprofen according to Method 4, and perform the test. Prepare the control solution with 2.0 mL of the Standard Lead Solution (not more than 10 ppm).

(3) Related Substances—Dissolve 0.050 g of Pranoprofen in 50 mL of the mobile phase, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add the mobile phase to make exactly 200 mL, and use this solution as the standard solution. Perform the test with 10  $\mu$ L each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions. Determine each peak area from both solutions by the automatic integration method: the each area of the peaks other than the peak of pranoprofen from the sample solution is not larger than the peak area of