

Spot 2 μL each of the sample solution and the standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with a mixture of cyclohexane and isopropylamine (49:1) to a distance of about 10 cm, and air-dry the plate. Spray evenly Dragendorff's TS for spraying on the plate, dry the plate, and then spray evenly hydrogen peroxide TS: the spots other than the principal spot from the sample solution is not more intense than the spot from the standard solution.

Loss on drying Not more than 0.5% (1 g, 105°C, 3 hours).

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

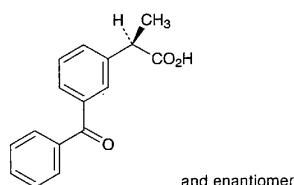
Assay Weigh accurately about 0.5 g of Ketamine Hydrochloride, previously dried, dissolve in 1 mL of formic acid, add 70 mL of a mixture of acetic anhydride and acetic acid (100) (6:1), and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

$$\begin{aligned} \text{Each mL of 0.1 mol/L perchloric acid VS} \\ = 27.149 \text{ mg of } \text{C}_{13}\text{H}_{16}\text{ClNO}\cdot\text{HCl} \end{aligned}$$

Containers and storage Containers—Tight containers.

Ketoprofen

ケトプロフェン



$\text{C}_{16}\text{H}_{14}\text{O}_3$: 254.28
(*RS*)-2-(3-Benzoylphenyl)propanoic acid [22071-15-4]

Ketoprofen, when dried, contains not less than 98.5% of $\text{C}_{16}\text{H}_{14}\text{O}_3$.

Description Ketoprofen occurs as a white, crystalline powder.

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

It is colored by light.

Identification (1) Dissolve 0.01 g of Ketoprofen in 1 mL of methanol, add 2 mL of 2,4-dinitrophenylhydrazine TS, and allow to stand for 30 minutes: an orange-yellow precipitate is formed.

(2) Determine the absorption spectrum of a solution of Ketoprofen in methanol (1 in 200,000) 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) Determine the infrared absorption spectrum of Ketoprofen, 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.

Melting point 94 – 97°C

Purity (1) Clarity and color of solution—Dissolve 0.5 g of Ketoprofen in 10 mL of methanol: the solution is clear and colorless.

(2) Chloride—Dissolve 2.0 g of Ketoprofen in 40 mL of methanol, and add 6 mL of dilute nitric acid and 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 VS add 40 mL of methanol, 6 mL of dilute nitric acid and water to make 50 mL (not more than 0.005%).

(3) Sulfate—Dissolve 2.0 g of Ketoprofen in 40 mL of methanol, and add 1 mL of dilute hydrochloric acid and water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution as follows: to 0.40 mL of 0.005 mol/L sulfuric acid VS add 40 mL of methanol, 1 mL of dilute hydrochloric acid and water to make 50 mL (not more than 0.010%).

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

(5) Arsenic—Prepare the test solution with 1.0 g of Ketoprofen according to Method 3, and perform the test using Apparatus B (not more than 2 ppm).

(6) Related substances—Conduct this procedure with a minimum of exposure to light, using light-resistant vessels. Dissolve 0.10 g of Ketoprofen in 10 mL of methanol, and use this solution as the sample solution. Pipet 2 mL of the sample solution, and add methanol to make exactly 100 mL. Pipet 5 mL of this solution, add methanol to make exactly 50 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 10 μL 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 carbon tetrachloride and acetic acid (100) (9:1) to a distance of about 10 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.

Loss on drying Not more than 0.5% (0.5 g, in vacuum, 60°C, 24 hours).

Residue on ignition Not more than 0.14% (1 g).

Assay Weigh accurately about 0.3 g of Ketoprofen, previously dried, dissolve in 25 mL of ethanol (95), add 25 mL of water, and titrate with 0.1 mol/L sodium hydroxide VS (indicator: 3 drops of phenolphthalein TS). Perform a blank determination, and make any necessary correction.

$$\begin{aligned} \text{Each mL of 0.1 mol/L sodium hydroxide VS} \\ = 25.429 \text{ mg of } \text{C}_{16}\text{H}_{14}\text{O}_3 \end{aligned}$$

Containers and storage Containers—Tight containers.

Storage—Light-resistant.