

**Operating conditions—**

**Detector:** An ultraviolet absorption photometer (wavelength: 230 nm).

**Column:** A stainless steel column about 6 mm in inside diameter and about 15 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5  $\mu$ m in particle diameter).

**Column temperature:** A constant temperature of about 40°C.

**Mobile phase:** Adjust the pH of a mixture of water, methanol and 0.5 mol/L ammonium acetate TS (15:5:2) to 5.5 with acetic acid (31).

**Flow rate:** Adjust the flow rate so that the retention time of 3-(*p*-hydroxyphenyl)propionic acid is about 7 minutes.

**Selection of column:** Proceed with 10  $\mu$ L of the standard solution under the above operating conditions, and calculate the resolution. Use a column giving elution of 3-(*p*-hydroxyphenyl)propionic acid and the internal standard in this order with the resolution between these peaks being not less than 5.

**Detection sensitivity:** Adjust the detection sensitivity so that the peak height of 3-(*p*-hydroxyphenyl)propionic acid obtained from 10  $\mu$ L of the standard solution is not less than 30 mm.

(5) **Related substances—**Dissolve 0.10 g of Cetraxate Hydrochloride 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 100 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 5  $\mu$ L 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 chloroform, methanol and acetic acid (100) (20:4:3) to a distance of about 10 cm, and air-dry the plate. Spray evenly ninhydrin TS on the plate, and heat at 90°C for 10 minutes: 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, 105°C, 3 hours).

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

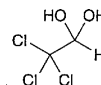
**Assay** Weigh accurately about 0.5 g of Cetraxate Hydrochloride, previously dried, dissolve in 100 mL of water, and adjust the pH of this solution to between 7.0 and 7.5 with dilute sodium hydroxide TS. To this solution add 10 mL of formaldehyde solution, stir for about 5 minutes, and titrate with 0.1 mol/L sodium hydroxide VS by taking over about 20 minutes (potentiometric titration). Perform a blank determination, and make any necessary correction.

$$\begin{aligned} \text{Each mL of 0.1 mol/L sodium hydroxide VS} \\ = 34.183 \text{ mg of } C_{17}H_{23}NO_4 \cdot HCl \end{aligned}$$

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

**Chloral Hydrate**

抱水クロラール



$C_2H_3Cl_3O_2$ : 165.40

2,2,2-Trichloroethane-1,1-diol [302-17-0]

Chloral Hydrate contains not less than 99.5% of  $C_2H_3Cl_3O_2$ .

**Description** Chloral Hydrate occurs as colorless crystals. It has a pungent odor and an acrid, slightly bitter taste.

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

It slowly volatilizes in air.

**Identification (1)** Dissolve 0.2 g of Chloral Hydrate in 2 mL of water, and add 2 mL of sodium hydroxide TS: the turbidity is produced, and it separates into two clear layers by warming.

(2) Heat 0.2 g of Chloral Hydrate with 3 drops of aniline and 3 drops of sodium hydroxide TS: the disagreeable odor of phenylisocyanide (poisonous) is perceptible.

**Purity (1)** Clarity and color of solution—Dissolve 1.0 g of Chloral Hydrate in 2 mL of water: the solution is clear and colorless.

(2) Acid—Dissolve 0.20 g of Chloral Hydrate in 2 mL of water, and add 1 drop of methyl orange TS: a yellow color develops.

(3) Chloride—Perform the test with 1.0 g of Chloral Hydrate. Prepare the control solution with 0.30 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.011%).

(4) Chloral alcoholate—Warm 1.0 g of Chloral Hydrate with 10 mL of sodium hydroxide TS, filter the upper layer, add iodine TS to the filtrate until a yellow color develops, and allow the solution to stand for 1 hour: no yellow precipitate is produced.

(5) Benzene—Warm the solution obtained in (1) with 3 mL of water: no odor of benzene is perceptible.

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

**Assay** Weigh accurately about 4 g of Chloral Hydrate in a glass-stoppered flask, add 10 mL of water and exactly 40 mL of 1 mol/L sodium hydroxide VS, and allow the mixture to stand for exactly 2 minutes. Titrate the excess sodium hydroxide immediately with 0.5 mol/L sulfuric acid VS (indicator: 2 drops of phenolphthalein TS). Perform a blank determination, and make any necessary correction.

$$\begin{aligned} \text{Each mL of 1 mol/L sodium hydroxide VS} \\ = 165.40 \text{ mg of } C_2H_3Cl_3O_2 \end{aligned}$$

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