

thin-layer chromatography. Develop the plate with a mixture of chloroform, methanol, water, acetic acid (100) and ethyl acetate (5:4:1:1:1) to a distance of about 13 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** 3.5 – 5.0% (0.4 g, direct titration).

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

**Assay** Weigh accurately about 0.6 g of Timepidium Bromide, dissolve in 60 mL of a mixture of acetic anhydride and acetic acid (100) (2:1), and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

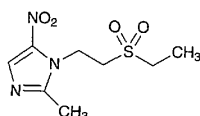
Each mL of 0.1 mol/L perchloric acid VS  
= 40.04 mg of  $C_{17}H_{22}BrNOS_2$

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

Storage—Light-resistant.

## Tinidazole

チニダゾール



$C_8H_{13}N_3O_4S$ : 247.27

Ethyl 2-(2-methyl-5-nitro-1*H*-imidazol-1-yl)ethyl sulfone  
[19387-91-8]

Tinidazole, when dried, contains not less than 98.5% of  $C_8H_{13}N_3O_4S$ .

**Description** Tinidazole occurs as a light yellow, crystalline powder. It is odorless or has a slight, characteristic odor. It has a bitter taste.

It is soluble in acetic anhydride and in acetone, sparingly soluble in methanol and in ethanol (95), and very slightly soluble in water and in diethyl ether.

**Identification** (1) Dissolve 0.01 g of Tinidazole in 2 mL of methanol, and add 1 mL of a solution of *N,N*-dimethylaniline in methanol (1 in 10): a yellow-green color develops.

(2) Determine the absorption spectrum of a solution of Tinidazole in methanol (1 in 50,000) 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 Tinidazole as directed in the paste 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** 125 – 129°C

**Purity** (1) Sulfate—To 2.0 g of Tinidazole add 100 mL

of water, boil for 5 minutes, cool, add water to make 100 mL, and filter. Take 25 mL of the filtrate, and add 1 mL of dilute hydrochloric acid and water to make 50 mL. Use this solution as the test solution, and perform the test. Prepare the control solution with 0.45 mL of 0.005 mol/L sulfuric acid VS (not more than 0.043%).

(2) Heavy metals—Proceed with 1.0 g of Tinidazole according to Method 4, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

(3) Arsenic—Prepare the test solution with 2.0 g of Tinidazole according to Method 3, and perform the test using Apparatus B (not more than 1 ppm).

(4) Related substances—Dissolve 0.050 g of Tinidazole in 2 mL of acetone, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add acetone 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  $\mu$ 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 ethyl acetate and diethylamine (19:1) to a distance of about 10 cm, air-dry the plate, heat at 100°C for 5 minute, and cool. 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 1.0% (1 g, 105°C, 2 hours).

**Residue on ignition** Not more than 0.1% (1 g).

**Assay** Weigh accurately about 0.35 g of Tinidazole, previously dried, dissolve in 50 mL of acetic anhydride, and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

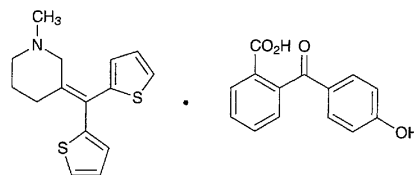
Each mL of 0.1 mol/L perchloric acid VS  
= 24.728 mg of  $C_8H_{13}N_3O_4S$

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

Storage—Light-resistant.

## Tipepidine Hibenazate

ヒベンズ酸チペピジン



$C_{15}H_{17}NS_2 \cdot C_{14}H_{10}O_4$ : 517.66

3-(Dithien-2-ylmethylene)-1-methylpiperidine mono[2-(4-hydroxybenzoyl)benzoate] [31139-87-4]

Tipepidine Hibenazate, when dried, contains not less than 98.5% of  $C_{15}H_{17}NS_2 \cdot C_{14}H_{10}O_4$ .