

ence Spectrum: both spectra exhibit similar intensities of absorption at the same wave numbers.

(4) To 5 mL of a solution of Thioridazine Hydrochloride (1 in 100) add 2 mL of ammonia TS, and heat on a water bath for 5 minutes. After cooling, filter, and acidify the filtrate with dilute nitric acid: the solution responds to the Qualitative Tests (2) for chloride.

Melting point 159 – 164°C

Purity (1) Heavy metals—Proceed with 1.0 g of Thioridazine Hydrochloride 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).

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

(3) **Related substances**—Conduct this procedure under the protection from the sunlight. Dissolve 0.10 g of Thioridazine Hydrochloride in 10 mL of methanol, and use this solution as the sample solution. Pipet 1 mL of the sample solution, and add methanol to make exactly 20 mL. Pipet 2 mL of this solution, add methanol to make exactly 10 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 5 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 chloroform, 2-propanol and ammonia solution (28) (74:25:1) to a distance of about 10 cm, and air-dry the plate. Examine the plate 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% (1 g, 105°C, 4 hours).

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

Assay Weigh accurately about 0.35 g of Thioridazine Hydrochloride, previously dried, dissolve in 80 mL of a mixture of acetic anhydride and acetic acid (100) (1:1), and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

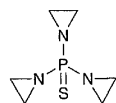
Each mL of 0.1 mol/L perchloric acid VS
= 40.70 mg of C₂₁H₂₆N₂S₂·HCl

Containers and storage Containers—Tight containers.

Storage—Light-resistant.

Thiotepa

チオテパ



C₆H₁₂N₃PS: 189.22

Tris(aziridin-1-yl)phosphine sulfate [52-24-4]

Thiotepa, when dried, contains not less than 98.0% of C₆H₁₂N₃PS.

Description Thiotepa occurs as colorless or white crystals, or white, crystalline powder. It is odorless.

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

A solution of Thiotepa (1 in 10) is neutral.

Identification (1) To 5 mL of a solution of Thiotepa (1 in 100) add 1 mL of hexaammonium heptamolybdate TS, and allow to stand: a dark blue color develops slowly when the solution is cold, or quickly when warm.

(2) To 5 mL of a solution of Thiotepa (1 in 100) add 1 mL of nitric acid: this solution responds to the Qualitative Tests (2) for phosphate.

(3) Dissolve 0.1 g of Thiotepa in a mixture of 1 mL of lead (II) acetate TS and 10 mL of sodium hydroxide TS, and boil: the gas evolved changes moistened red litmus paper to blue, and the solution shows a grayish red color.

Melting point 52 – 57°C

Purity (1) Clarity and color of solution—Dissolve 1.0 g of Thiotepa in 20 mL of water: the solution is clear and colorless.

(2) **Heavy metals**—Proceed with 1.0 g of Thiotepa in a platinum crucible 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**—Dissolve 0.20 g of Thiotepa in 5 mL of water, and add 1 mL of nitric acid and 1 mL of sulfuric acid. Take this solution, prepare the test solution according to Method 2, and perform the test using Apparatus B (not more than 10 ppm).

Loss on drying Not more than 0.20% (1 g, in vacuum, silica gel, 4 hours).

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

Assay Weigh accurately about 0.1 g of Thiotepa, previously dried, dissolve in 50 mL of a solution of potassium thiocyanate (3 in 20), add 25 mL of 0.05 mol/L sulfuric acid VS, exactly measured, and allow to stand for 20 minutes with occasional shaking. Titrate the excess sulfuric acid with 0.1 mol/L sodium hydroxide VS until the color of the solution changes from red to light yellow (indicator: 3 drops of methyl red TS). Perform a blank determination.

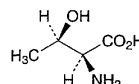
Each mL of 0.05 mol/L sulfuric acid VS
= 6.307 mg of C₆H₁₂N₃PS

Containers and storage Containers—Tight containers.

Storage—Light-resistant, and in a cold place.

L-Threonine

L-トレオニン



$C_4H_9NO_3$: 119.12
(2*S*,3*R*)-2-Amino-3-hydroxybutanoic acid [72-19-5]

L-Threonine, when dried, contains not less than 98.5% of $C_4H_9NO_3$.

Description L-Threonine occurs as white crystals or crystalline powder. It is odorless or has a slight, characteristic odor, and has a slightly sweet taste.

It is freely soluble in formic acid, soluble in water, and practically insoluble in ethanol (95).

Identification Determine the infrared absorption spectrum of L-Threonine, 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.

Optical rotation $[\alpha]_D^{20}$: $-26.0 - -29.0^\circ$ (after drying, 1.5 g, water, 25 mL, 100 mm).

pH Dissolve 0.20 g of L-Threonine in 20 mL of water: the pH of this solution is between 5.2 and 6.2.

Purity (1) Clarity and color of solution—Dissolve 1.0 g of L-Threonine in 20 mL of water: the solution is clear and colorless.

(2) Chloride—Perform the test with 0.5 g of L-Threonine. Prepare the control solution with 0.30 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.021%).

(3) Sulfate—Perform the test with 0.6 g of L-Threonine. Prepare the control solution with 0.35 mL of 0.005 mol/L sulfuric acid VS (not more than 0.028%).

(4) Ammonium—Perform the test with 0.25 g of L-Threonine. Prepare the control solution with 5.0 mL of Standard Ammonium Solution (not more than 0.02%).

(5) Heavy metals—Proceed with 1.0 g of L-Threonine according to Method 1, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

(6) Arsenic—Dissolve 1.0 g of L-Threonine in 5 mL of dilute hydrochloric acid, and perform the test with this solution as the test solution using Apparatus B (not more than 2 ppm).

(7) Other amino acids—Dissolve 0.30 g of L-Threonine in 50 mL of water, and use this solution as the sample solution. Pipet 1 mL of this solution, and add water to make exactly 50 mL. Pipet 5 mL of this solution, add water to make exactly 20 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 5 μ 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 1-butanol, water and acetic acid (100) (3:1:1) to a distance of about 10 cm, and dry the plate at 80°C for 30 minutes. Spray evenly the plate with a solution of ninhydrin in acetone (1 in 50), and heat the plate at 80°C for 5 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 0.20% (1 g, 105°C, 3 hours).

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

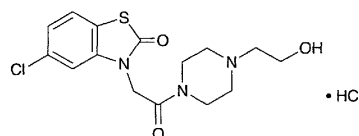
Assay Weigh accurately about 0.12 g of L-Threonine, previously dried, dissolve in 3 mL of formic acid, add 50 mL of acetic acid (100), and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 11.912 mg of $C_4H_9NO_3$

Containers and storage Containers—Tight containers.

Tiaramide Hydrochloride

塩酸チアラミド



$C_{15}H_{18}ClN_3O_3S \cdot HCl$: 392.30
5-Chloro-3-[2-[4-(2-hydroxyethyl)piperazin-1-yl]-2-oxoethyl]-3*H*-benzothiazol-2-one monohydrochloride
[35941-71-0]

Tiaramide Hydrochloride, when dried, contains not less than 98.5% of $C_{15}H_{18}ClN_3O_3S \cdot HCl$.

Description Tiaramide Hydrochloride occurs as a white, crystalline powder. It is odorless.

It is freely soluble in water, slightly soluble in ethanol (95) and in acetic acid (100), and practically insoluble in acetic anhydride and in diethyl ether.

The pH of a solution of Tiaramide Hydrochloride (1 in 20) is between 3.0 and 4.5.

Melting point: about 265°C (with decomposition).

Identification (1) Dissolve 5 mg of Tiaramide Hydrochloride in 5 mL of 0.1 mol/L hydrochloric acid TS, and add 3 drops of Dragendorff's TS: an orange precipitate is formed.

(2) Determine the infrared absorption spectrum of Tiaramide Hydrochloride, previously dried, as directed in the potassium chloride 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.

(3) A solution of Tiaramide Hydrochloride (1 in 50) responds to the Qualitative Tests for chloride.

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

(2) Heavy metals—Proceed with 2.0 g of Tiaramide Hydrochloride 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).

(3) Arsenic—Prepare the test solution with 1.0 g of Tiaramide Hydrochloride according to Method 1, and perform the test using Apparatus B. In the procedure, add 20 mL of diluted hydrochloric acid (1 in 2) (not more than 2 ppm).