Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 254 nm).

Column: A stainless steel column about 4 mm in inside diameter and 15 to 30 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 to 10 μm in particle diameter).

Column temperature: A constant temperature of about 25°C.

Mobile phase: Dissolve 1.1 g of sodium 1-octanesulfonate in 1000 mL of diluted acetic acid (100) (1 in 100). To 600 mL of this solution add 400 mL of a mixture of methanol and acetonitrile (3:2).

Flow rate: Adjust the flow rate so that the retention time of thiamine is about 12 minutes.

Selection of column: Proceed with 10 μL of the standard solution under the above operating conditions, and calculate the resolution. Use a column giving elution of thiamine and the internal standard in this order with the resolution between these peaks being not less than 6.

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

Thiamine Hydrochloride Injection

Vitamin B_1 Hydrochloride Injection

塩酸チアミン注射液

Thiamine Hydrochloride Injection is an aqueous solution for injection. It contains not less than 95% and not more than 115% of the labeled amount of thiamine hydrochloride ($C_{12}H_{17}ClN_4OS.HCl$: 337.27).

Method of preparation Prepare as directed under Injections, with Thiamine Hydrochloride.

Description Thiamine Hydrochloride Injection is a clear, colorless liquid.

pH: 2.5 - 4.5

Identification To a volume of Thiamine Hydrochloride Injection, equivalent to 0.05 g of Thiamine Hydrochloride according to the labeled amount, add water to make 25 mL. Proceed with 5 mL of this solution as directed in the Identification (1) under Thiamine Hydrochloride.

Assay Dilute with 0.001 mol/L hydrochloric acid TS if necessary, then measure exactly a volume of Thiamine Hydrochloride Injection, equivalent to about 0.02 g of thiamine hydrochloride ($C_{12}H_{17}ClN_4OS.HCl$), and add 20 mL of methanol and 0.001 mol/L hydrochloric acid TS to make 100 mL. To 25 mL of this solution, exactly measured, add exactly 5 mL of the internal standard solution, add 0.001 mol/L hydrochloric acid TS to make 50 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.1 g of Thiamine Hydrochloride Reference Standard (determine previously its water content), and dissolve in 0.001 mol/L hydrochloric acid TS to make exactly 50 mL. To 10 mL of this solution, exactly measured, add 20 mL of methanol and 0.001 mol/L hydrochloric acid TS to make exactly 100 mL. To 25 mL of this solution, exactly measured, add exactly 5 mL of the internal standard solution, add 0.001 mol/L hydrochloric acid TS to make 50 mL, and use this solution as the standard solution. Proceed as directed in the Assay under Thiamine Hydrochloride.

Amount (mg) of thiamine hydrochloride ($C_{12}H_{17}ClN_4OS.HCl$)

= amount (mg) of Thiamine Hydrochloride Reference Standard, calculated on the anhydrous basis

$$\times \frac{Q_T}{Q_S} \times \frac{1}{5}$$

Internal standard solution—A solution of methyl benzoate in methanol (1 in 200).

Containers and storage Containers—Hermetic containers. Storage—Light-resistant.

Thiamine Hydrochloride Powder

Vitamin B_1 Hydrochloride Powder

塩酸チアミン散

Thiamine Hydrochloride Powder contains not less than 95% and not more than 115% of the labeled amount of thiamine hydrochloride ($C_{12}H_{17}ClN_4OS.HCl$: 337.27).

Method of preparation Prepare as directed under Powders, with Thiamine Hydrochloride.

Identification To a portion of Thiamine Hydrochloride Powder, equivalent to 0.02 g of Thiamine Hydrochloride according to the labeled amount, add 50 mL of water and 10 mL of dilute acetic acid, shake, and filter. Proceed with 5 mL of the filtrate as directed in the Identification (1) under Thiamine Hydrochloride.

Purity Rancidity—Thiamine Hydrochloride Powder has no unpleasant or rancid odor. It is tasteless.

Assay Weigh accurately a quantity of Thiamine Hydrochloride Powder, equivalent to about 0.02 g of thiamine hydrochloride ($C_{12}H_{17}ClN_4OS.HCl$), add 60 mL of 0.01 mol/L hydrochloric acid TS, and heat on a water bath for 30 minutes. Shake vigorously for 10 minutes, cool, add methanol to make exactly 100 mL, and centrifuge. Pipet 25 mL of the supernatant, add exactly 5 mL of the internal standard solution, add water to make 50 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.1 g of Thiamine Hydrochloride Reference Standard (determine previously its water content), and dissolve in 0.01 mol/L hydrochloric acid TS to make exactly 50 mL. To 10 mL of this solution, exactly measured, add 50 mL of 0.01 mol/L hydrochloric acid TS, and add methanol to make exactly 100 mL. To 25 mL of this solution, exactly measured, add exactly 5 mL of the internal standard solution, add water to make 50 mL, and use this solution as the standard solution. Proceed as directed in the Assay under Thiamine Hydrochloride.

Amount (mg) of thiamine hydrochloride ($C_{12}H_{17}ClN_4OS.HCl$)
= amount (mg) of Thiamine Hydrochloride Reference Standard, calculated on the anhydrous basis

$$\times \frac{Q_T}{Q_S} \times \frac{1}{5}$$

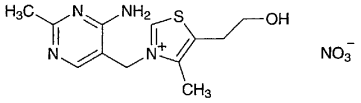
Internal standard solution—A solution of methyl benzoate in methanol (1 in 200).

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

Thiamine Nitrate

Vitamin B_1 Nitrate

硝酸チアミン



$C_{12}H_{17}N_5O_4S$: 327.36
3-(4-Amino-2-methylpyrimidin-5-ylmethyl)-5-(2-hydroxyethyl)-4-methylthiazolium nitrate [*532-43-4*]

Thiamine Nitrate, when dried, contains not less than 98.0% and not more than 102.0% of $C_{12}H_{17}N_5O_4S$.

Description Thiamine Nitrate occurs as white crystals or crystalline powder. It is odorless or a slight, characteristic odor.

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

Melting point: about 193°C (with decomposition).

Identification (1) Take 2-mL portions of a solution of Thiamine Nitrate (1 in 500), and add 2 to 3 drops of iodine TS: a red-brown precipitate or turbidity is produced. Upon further addition of 1 mL of 2,4,6-trinitrophenol TS, a yellow precipitate or turbidity is produced.

(2) To 1 mL of a solution of Thiamine Nitrate (1 in 500) add 1 mL of lead (II) acetate TS and 1 mL of a solution of sodium hydroxide (1 in 10), and warm: the color of the solution changes through yellow to brown, and on standing, a black-brown precipitate is produced.

(3) To 5 mL of a solution of Thiamine Nitrate (1 in 500) add 2.5 mL of sodium ;hydroxide TS and 0.5 mL of potassium hexacyanoferrate (III) TS. Then add 5 mL of 2-methyl-1-propanol, shake the mixture vigorously for 2 minutes, allow to stand, and examine under ultraviolet light (main wavelength: 365 nm): the 2-methyl-1-propanol layer shows a blue-purple fluorescence. This fluorescence disappears when the mixture is acidified, but reappears when it is again made alkaline.

(4) A solution of Thiamine Nitrate (1 in 50) responds to the Qualitative Tests (1) and (2) for nitrate.

pH Dissolve 1.0 g of Thiamine Nitrate in 100 mL of water: the pH of this solution is between 6.5 and 8.0.

Purity (1) Chloride—Perform the test with 0.20 g of Thiamine Nitrate. Prepare the control solution with 0.30 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.053%).

(2) Sulfate—Dissolve 1.5 g of Thiamine Nitrate in 30 mL of water and 2 mL of dilute hydrochloric acid, and add water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution with 0.35 mL of 0.005 mol/L sulfuric acid VS and 2 mL of dilute hydrochloric acid, and add water to make 50 mL (not more than 0.011%).

(3) Heavy metals—Dissolve 1.0 g of Thiamine Nitrate in 30 mL of water by warming, cool, and add 12 mL of 6 mol/L acetic acid (31) TS and water to make 50 mL. Perform the test with this solution as the test solution. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

Loss on drying Not more than 1.0% (0.5 g, 105°C, 2 hours).

Residue on ignition Not more than 0.20% (1 g).

Assay Weigh accurately about 0.1 g each of Thiamine Nitrate, previously dried, and Thiamine Hydrochloride Reference Standard (previously determine its water content in the same manner as directed under Thiamine Nitrate), and dissolve them in the mobile phase to make exactly 50 mL. To 10 mL each of the solutions, accurately measured, add exactly 5 mL each of the internal standard solution, add the mobile phase to make 50 mL, and use these solutions as the sample solution and the standard solution. Perform the test with 10 μL each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following operating conditions. and calculate the ratios, Q_T and Q_S, of the peak area of thiamine to that of the internal standard.

Amount (mg) of $C_{12}H_{17}N_5O_4S$
= amount (mg) of Thiamine Hydrochloride Reference Standard, Calculated on the anhydrous basis

$$\times \frac{Q_T}{Q_S} \times 0.9706$$

Internal standard solution—A solution of methyl benzoate in methanol (1 in 50).

Operating conditions—

Detector: An ultraviolet spectrophotometer (wavelength: 254 nm).

Column: A stainless steel column about 4 mm in inside diameter and 15 to 30 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 to 10 μm in particle diameter).

Column temperature: A constant temperature of 25 - 30°C.

Mobile phase: Dissolve 1.1 g of sodium l-octanesulfonate in 1000 mL of diluted acetic acid (100) (1 in 100). To 600 mL of this solution add 400 mL of a mixture of methanol and acetonitrile (3:2).

Flow rate: Adjust the flow rate so that the retention time of thiamine is about 12 minutes.

Selection of column: Proceed with 10 μL of the standard solution under the above operating conditions, and calculate the resolution. Use a column giving elution of thiamine and the internal standard in this order with the resolution between these peaks being not less than 6.

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