

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

Column temperature: Room temperature.

Mobile phase: A mixture of water and acetonitrile (1:1).

Flow rate: Adjust the flow rate so that the retention time of styrene is between 7 minutes and 8 minutes.

Selection of column: Dissolve 0.02 g each of styrene and butyl paraoxybenzoate in 100 mL of acetone. To 5 mL of this solution add acetone to make 100 mL. Proceed with 20 μ L of this solution under the above operating conditions, and calculate the resolution. Use a column giving elution of butyl paraoxybenzoate and styrene 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 styrene obtained from 20 μ L of the standard solution is between 10 mm and 15 mm.

Water Not more than 10.0% (0.2 g, direct titration).

Assay (1) Sodium—Weigh accurately about 1 g of Sodium Polystyrene Sulfonate, calculated on the anhydrous basis, in a glass-stoppered flask, add exactly 50 mL of 3 mol/L hydrochloric acid TS, shake for 60 minutes, and filter. Discard the first 20 mL of the filtrate, pipet the subsequent 5 mL of the filtrate, and add water to make exactly 100 mL. Pipet 20 mL of this solution, add water to make exactly 1000 mL, and use this solution as the sample solution. Separately, pipet a suitable quantity of Standard Sodium Stock Solution, dilute exactly with water so that each mL of the solution contains 1 to 3 μ g of sodium (Na: 22.99), and use these solutions as the standard solution. Perform the test with the sample solution and the standard solution according to the Atomic Absorption Spectrophotometry under the following conditions, and determine the amount of sodium in the sample solution using the calibration curve obtained from the standard solutions.

Gas: Combustible gas—Acetylene

Supporting gas—Air

Lamp: A sodium hollow-cathode lamp

Wavelength: 589.0 nm

(2) Potassium exchange capacity—Weigh accurately about 1.5 g of Sodium Polystyrene Sulfonate, calculated on the anhydrous basis, in a glass-stoppered flask, add exactly 100 mL of Standard Potassium Stock Solution, shake for 15 minutes, and filter. Discard the first 20 mL of the filtrate, pipet the subsequent 10 mL of the filtrate, and add water to make exactly 100 mL. Pipet 10 mL of this solution, add water to make exactly 1000 mL, and use this solution as the sample solution. Separately, pipet a suitable quantity of Standard Potassium Stock Solution, dilute with water so that each mL of the solution contains 1 to 5 μ g of potassium (K: 39.10), and use the solution as the standard solution. Perform the test with these solutions as directed under the Atomic Absorption Spectrophotometry, and determine the amount *Y* (mg) of potassium in 1000 mL of the sample solution using the calibration curve obtained from the standard solution. The quantity of potassium absorbed on each g of Sodium Polystyrene Sulfonate, calculated on the anhydrous basis, is calculated from the following equation: it is between 0.110 g and 0.135 g.

Quantity (mg) of potassium (K) absorbed on 1 g of Sodium Polystyrene Sulfonate, calculated on the anhydrous basis

$$= \frac{(X - 100Y)}{W}$$

X: Amount (mg) of potassium in 100 mL of the Standard Potassium Stock Solution before exchange.

W: Mass (g) of Sodium Polystyrene Sulfonate taken, calculated on the anhydrous basis.

Gas: Combustible gas—Acetylene
Supporting gas—Air

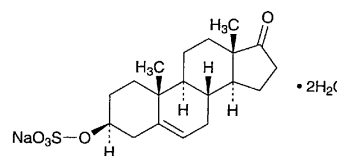
Lamp: A potassium hollow-cathode lamp

Wavelength: 766.5 nm

Containers and storage Containers—Tight containers.

Sodium Prasterone Sulfate

プラステロン硫酸ナトリウム



$C_{19}H_{27}NaO_5S \cdot 2H_2O$: 426.50

Monosodium 17-oxoandrost-5-en-3 β -yl sulfate dihydrate
[1099-87-2, anhydride]

Sodium Prasterone Sulfate contains not less than 98.0% of $C_{19}H_{27}NaO_5S$ (mol. wt.: 390.47), calculated on the dried basis.

Description Sodium Prasterone Sulfate occurs as white crystals or crystalline powder. It is odorless.

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

The pH of a solution of Sodium Prasterone Sulfate (1 in 200) is between 4.5 and 6.5.

Melting point: about 160°C (with decomposition, after drying).

Identification (1) Dissolve 0.01 g of Sodium Prasterone Sulfate in 4 mL of ethanol (95), add 2 mL of 1,3-dinitrobenzene TS and 2 mL of a solution of sodium hydroxide (1 in 8): a red-purple color develops, and gradually changes to brown.

(2) To 10 mL of a solution of Sodium Prasterone Sulfate (1 in 200) add 0.5 mL of bromine TS: the color of bromine TS immediately disappears.

(3) Determine the infrared absorption spectrum of Sodium Prasterone Sulfate 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.

(4) A solution of Sodium Prasterone Sulfate (1 in 200) responds to the Qualitative Tests for sodium salt.

Optical rotation $[\alpha]_D^{20}$: +10.7 – +12.1° (0.73 g, calculated on the dried basis, methanol, 20 mL, 100 mm).

Purity (1) Clarity and color of solution—Dissolve 0.25 g of Sodium Prasterone Sulfate in 50 mL of water: the solution is clear and colorless.

(2) Chloride—Dissolve 1.0 g of Sodium Prasterone Sulfate in 20 mL of acetone and 20 mL of water, 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 20 mL of acetone, 6 mL of dilute nitric acid and water to make 50 mL (not more than 0.011%).

(3) Sulfate—To 1.2 g of Sodium Prasterone Sulfate add 20 mL of water, shake vigorously for 5 minutes, and filter. To 10 mL of the filtrate add 20 mL of acetone, 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 20 mL of acetone, 1 mL of dilute hydrochloric acid and water to make 50 mL (not more than 0.032%).

(4) Heavy metals—Proceed with 2.0 g of Sodium Prasterone Sulfate 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) Other steroids—Dissolve 0.10 g of Sodium Prasterone Sulfate 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 200 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 with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of chloroform, methanol and water (75:22:3) to a distance of about 10 cm, and air-dry the plate. Spray evenly a mixture of sulfuric acid and ethanol (95) (1:1) on the plate, and heat 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 8.0 – 9.0% (0.5 g, in vacuum, phosphorus (V) oxide, 60°C, 3 hours).

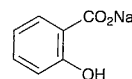
Assay Weigh accurately about 0.25 g of Sodium Prasterone Sulfate, dissolve in 30 mL of water. Apply this solution to a chromatographic column 10 mm in inside diameter, previously prepared by pouring 5 mL of strongly acidic ion-exchange resin (H type) for column chromatography, and elute at the rate of 4 mL per minute. Wash the chromatographic column with 100 mL of water, combine the washings with above effluent solution, and titrate with 0.05 mol/L sodium hydroxide VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

Each mL of 0.05 mol/L sodium hydroxide VS
= 19.524 mg of $C_{19}H_{27}NaO_5S$

Containers and storage Containers—Tight containers.

Sodium Salicylate

サリチル酸ナトリウム



$C_7H_5NaO_3$: 160.10

Monosodium 2-hydroxybenzoate [54-21-7]

Sodium Salicylate, when dried, contains not less than 99.5% of $C_7H_5NaO_3$.

Description Sodium Salicylate occurs as white, crystals or crystalline powder.

It is very soluble in water, freely soluble in acetic acid (100), and soluble in ethanol (95).

It is gradually colored by light.

Identification (1) Determine the infrared absorption spectrum of Sodium Salicylate, 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.

(2) A solution of Sodium Salicylate (1 in 20) responds to the Qualitative Tests for sodium salt.

pH The pH of a solution of 2.0 g of Sodium Salicylate in 20 mL of water is between 6.0 and 8.0.

Purity (1) Clarity of solution—Dissolve 1.0 g of Sodium Salicylate in 10 mL of water: the solution is clear, and its absorbance at 420 nm determined as directed under the Ultraviolet-visible Spectrophotometry is not more than 0.02.

(2) Chloride—Dissolve 0.5 g of Sodium Salicylate in 15 mL of water, add 6 mL of dilute nitric acid and ethanol (95) to make 50 mL, and perform the test using this solution as the test solution. Prepare the control solution with 0.30 mL of 0.01 mol/L hydrochloric acid VS, 28 mL of ethanol (95), 6 mL of dilute nitric acid and water to make 50 mL (not more than 0.021%).

(3) Sulfate—Dissolve 0.25 g of Sodium Salicylate in 5 mL of water, and add 0.5 mL of barium chloride TS: the solution shows no change.

(4) Sulfite and thiosulfate—Dissolve 1.0 g of Sodium Salicylate in 20 mL of water, add 1 mL of hydrochloric acid, and filter. Add 0.15 mL of 0.05 mol/L iodine VS to the filtrate: a yellow color develops.

(5) Heavy metals—Proceed with 1.0 g of Sodium Salicylate 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).

(6) Arsenic—To 1.0 g of Sodium Salicylate in a decomposition flask add 5 mL of nitric acid and 2 mL of sulfuric acid, and heat carefully until white fumes are evolved. After cooling, add 2 mL of nitric acid, and heat. After cooling, add several 2-mL portions of hydrogen peroxide (30), and heat until the solution is colorless to pale yellow. Repeat the procedure of adding nitric acid and hydrogen peroxide (30) and heating, if necessary. After cooling, add 2 mL of a saturated solution of ammonium oxalate monohydrate, and heat until white fumes are evolved. After cooling, add water