

It is gradually colored by light.

The pH of a solution of Sodium Picosulfate (1 in 20) is between 7.4 and 9.4.

Identification (1) Mix 5 mg of Sodium Picosulfate with 0.01 g of 1-chloro-2,4-dinitrobenzene, and melt by gentle heating for 5 to 6 seconds. After cooling, add 4 mL of potassium hydroxide-ethanol TS: an orange-red color develops.

(2) To 0.2 g of Sodium Picosulfate add 5 mL of dilute hydrochloric acid, boil for 5 minutes, cool, and add 1 mL of barium chloride TS: a white precipitate is formed.

(3) Determine the absorption spectrum of a solution of Sodium Picosulfate (1 in 25,000) 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.

(4) Determine the infrared absorption spectrum of Sodium Picosulfate, previously dried at 105°C in vacuum for 4 hours, 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.

(5) A solution of Sodium Picosulfate (1 in 10) responds to the Qualitative Tests for sodium salt.

Absorbance $E_{1\text{cm}}^{1\%}$ (263 nm): 120 – 130 (calculated on the anhydrous basis, 4 mg, water, 100 mL).

Purity (1) Clarity and color of solution—Dissolve 1.0 g of Sodium Picosulfate in 10 mL of water: the solution is clear and colorless to pale yellow.

(2) Chloride—Perform the test with 0.5 g of Sodium Picosulfate. Prepare the control solution with 0.40 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.028%).

(3) Sulfate—Perform the test with 0.40 g of Sodium Picosulfate. Prepare the control solution with 0.35 mL of 0.005 mol/L sulfuric acid VS (not more than 0.042%).

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

(6) Related substances—Dissolve 0.25 g of Sodium Picosulfate in 5 mL of methanol, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add methanol to make exactly 500 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 1-butanol, water and acetic acid (100) (74:20:19) to a distance of about 10 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 3.0 – 4.5% (0.5 g, direct titration).

Assay Weigh accurately about 0.4 g of Sodium Picosulfate, dissolve in 50 mL of methanol, add 7 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
= 48.14 mg of $\text{C}_{18}\text{H}_{13}\text{NNa}_2\text{O}_8\text{S}_2$

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

Sodium Polystyrene Sulfonate

ポリスチレンスルホン酸ナトリウム

Sodium Polystyrene Sulfonate is a cation exchange resin prepared as the sodium form of the sulfonated styrene divinylbenzene copolymer. It contains not less than 9.4% and not more than 11.0% of sodium (Na: 22.99), calculated on the anhydrous basis.

Each g of Sodium Polystyrene Sulfonate, calculated on the anhydrous basis, exchanges with not less than 0.110 g and not more than 0.135 g of potassium (K: 39.10).

Description Sodium Polystyrene Sulfonate occurs as a yellow-brown powder. It is odorless and tasteless.

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

Identification (1) Determine the infrared absorption spectrum of Sodium Polystyrene Sulfonate 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) To 1 g of Sodium Polystyrene Sulfonate add 10 mL of dilute hydrochloric acid, stir, and filter. Add ammonia TS to the filtrate to neutralize: the solution responds to the Qualitative Tests for sodium salt.

Purity (1) Ammonium—Place 1.0 g of Sodium Polystyrene Sulfonate in a flask, add 5 mL of sodium hydroxide TS, cover the flask with a watch glass having a moistened strip of red litmus paper on the underside, and boil for 15 minutes: the gas evolved does not change the red litmus paper to blue.

(2) Heavy metals—Proceed with 2.0 g of Sodium Polystyrene Sulfonate 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 2.0 g of Sodium Polystyrene Sulfonate according to Method 3, and perform the test using Apparatus B (not more than 1 ppm).

(4) Styrene—To 10.0 g of Sodium Polystyrene Sulfonate add 10 mL of acetone, shake for 30 minutes, centrifuge, and use the supernatant liquid as the sample solution. Separately, dissolve 0.010 g of styrene in acetone to make exactly 100 mL. Pipet 1 mL of this solution, add acetone to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 20 μL each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and determine peak areas, A_T and A_S , of styrene in each solution: A_T is not larger than A_S .

Operating conditions—

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

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

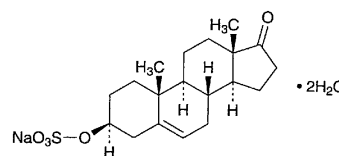
Lamp: A potassium hollow-cathode lamp

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).