

water bath. After cooling, add water to make exactly 200 mL, and filter. Pipet 30 mL of the filtrate, transfer to an iodine flask, and proceed as directed in the Assay (1) under Calcium Para-aminosalicylate.

Each mL of 0.05 mol/L bromine VS
= 4.238 mg of $C_{14}H_{10}Ca_2N_2O_6 \cdot 7H_2O$

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

Calcium Polystyrene Sulfonate

ポリスチレンスルホン酸カルシウム

Calcium Polystyrene Sulfonate is a cation exchange resin prepared as the calcium form of the sulfonated styrene divinylbenzene copolymer. When dried, it contains not less than 7.0% and not more than 9.0% of calcium (Ca: 40.08).

Each g of Calcium Polystyrene Sulfonate, when dried, exchanges with 0.053 to 0.071 g of potassium (K: 39.10).

Description Calcium Polystyrene Sulfonate occurs as a pale yellowish white to light yellow powder. It is odorless and tasteless.

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

Identification (1) Determine the infrared absorption spectrum of Calcium Polystyrene Sulfonate, 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) Mix 0.5 g of Calcium Polystyrene Sulfonate with 10 mL of dilute hydrochloric acid, filter, and neutralize the filtrate with ammonia TS: the solution responds to the Qualitative Tests for calcium salt.

Purity (1) Ammonium—Place 1.0 g of Calcium 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 (not less than 5 ppm).

(2) Heavy metals—Proceed with 2.0 g of Calcium 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 1.0 g of Calcium Polystyrene Sulfonate according to Method 3, and perform the test using Apparatus B (not more than 2 ppm).

(4) Styrene—To 10.0 g of Calcium 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, dilute with acetone to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 5 μ L each of the sample solution and the standard solution as directed under the Gas Chromatography according to the following

conditions. Determine the peak heights, H_T and H_S , of styrene in each solution: H_T is not larger than H_S .

Operating conditions—

Detector: A hydrogen flame-ionization detector.

Column: A stainless steel column about 3 mm in inside diameter and about 2 m in length, having polyethylene glycol 20 mol/L coated at the ratio of 15% on siliceous earth for gas chromatography (150 to 180 μ m in particle diameter).

Column temperature: A constant temperature of about 90°C.

Carrier gas: Nitrogen.

Flow rate: Adjust the flow rate so that the retention time of styrene is about 9 minutes.

Selection of column: Proceed with 5 μ L of the standard solution under the above operating conditions. Use a column showing a clear peak of styrene.

Detection sensitivity: Adjust the sensitivity so that the peak height of styrene obtained from 5 μ L of the standard solution is between 8 mm and 12 mm.

(5) Sodium—Pipet 2 mL of the 50-mL solution obtained in Assay (1), add 0.02 mol/L hydrochloric acid TS to make exactly 500 mL, and use this solution as the sample solution. Separately, weigh accurately 0.2542 g of sodium chloride, previously dried at 130°C for 2 hours, and dissolve in 0.02 mol/L hydrochloric acid TS to make exactly 1000 mL. Pipet a suitable volume of this solution, and dilute with 0.02 mol/L hydrochloric acid TS to make a solution containing 1 to 3 μ g of sodium (Na: 22.99) per mL, and use these solutions as the standard solutions. Perform the test with the sample solution and the standard solutions 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: the amount of sodium is not more than 1%.

Gas: Combustible gas—Acetylene

Supporting gas—Air

Lamp: A sodium hollow-cathode lamp

Wavelength: 589.0 nm

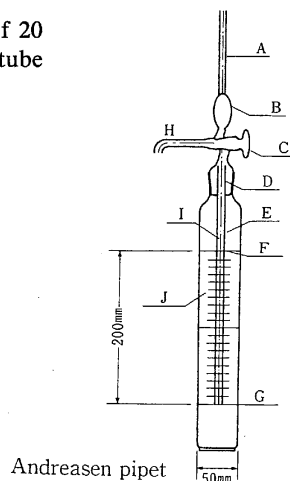
Loss on drying Not more than 10.0% (1 g, in vacuum, 80°C, 5 hours).

Microparticles (i) Apparatus: Use an apparatus as shown in the illustration.

Actual volume to the mark of 20 cm at which the sedimentation tube is inserted: 550 mL

Single suction volume: 10 mL

- A: Mark of pipet bulb
B: Pipet bulb for suction
C: Two-way stopcock
D: Vent-hole
E: Suction part of pipet
F: Mark of 20 cm
G: Base line of 0 cm
H: Outlet of pipet
I: Capillary tube of pipet
J: Sedimentation tube



(ii) Procedure: Weigh accurately about 5.5 g of Calcium Polystyrene Sulfonate, previously dried, add 300 mL of water of 25°C, and mix for 5 minutes. Transfer this turbid solution to the sedimentation tube J, keeping a temperature at 25°C, add water of 25°C to 2 mm below the mark F of 20 cm of the sedimentation tube J, and then insert the pipet. Open the two-way stopcock C, exhaust air, add exactly water from the vent-hole D to the mark F of 20 cm, and close the two-way stopcock C. Shake the apparatus well vertically and horizontally, disperse Calcium Polystyrene Sulfonate in water, and then open the two-way stopcock, and allow to stand at $25 \pm 1^\circ\text{C}$ for 5 hours and 15 minutes.

Then, draw exactly the meniscus of the turbid solution in sedimentation tube J up to the mark of pipet bulb A by suction, open the two-way stopcock C to the outlet of pipet H, and transfer exactly measured 20 mL of the turbid solution to a weighing bottle. Repeat the procedure, and combine exactly measured 20 mL of the turbid solution. Evaporate 20 mL of this turbid solution on a water bath to dryness, dry to constant mass at 105°C, and weigh the residue as W_S (g). Pipet 20 mL of used water, and weigh the residue in the same manner as W_B (g). Calculate the difference mi (g) between W_S and W_B , and calculate the amount of microparticles (S) by the following equation: the amount of microparticles is not more than 0.1%.

$$S (\%) = \frac{mi (\text{g}) \times V (\text{mL})}{20 (\text{mL}) \times \left(\begin{array}{l} \text{amount (g) of Calcium} \\ \text{Polystyrene Sulfonate} \end{array} \right)}$$

V : Actual volume (mL) to the mark of 20 cm at which the suction part of pipet is inserted.

Assay (1) Calcium—Weigh accurately about 1.0 g of Calcium Polystyrene Sulfonate, previously dried, and disperse in 5 mL of 3 mol/L hydrochloric acid TS. Transfer this mixture, and wash out completely with the aid of a small quantity of 3 mol/L hydrochloric acid TS to a column 12 mm in inside diameter and 70 mm in length, packed with a pledged of fine glass wool in the bottom of it, placing a 50-mL volumetric flask as a receiver under the column. Then collect about 45 mL of eluate, adding 3 mol/L hydrochloric acid TS to the column, and add water to make exactly 50 mL. Pipet 20 mL of this solution, adjust with ammonia TS to a pH of exactly 10. Titrate immediately with 0.05 mol/L disodium dihydrogen ethylenediamine tetraacetate VS until the red-purple color of the solution disappears, and a blue color develops (indicator: 0.04 g eriochrome black T-sodium chloride indicator). Perform a blank determination, and make any necessary correction.

Each mL of 0.05 mol/L disodium dihydrogen ethylenediamine tetraacetate VS
= 2.0039 mg of Ca

(2) Potassium exchange capacity—Pipet 50 mL of Standard Potassium Stock Solution into a glass-stoppered flask containing about 1.0 g of dried Calcium Polystyrene Sulfonate, accurately weighed, stir for 120 minutes, filter, and discard the first 20 mL of the filtrate. Pipet 5 mL of the subsequent filtrate, and add 0.02 mol/L hydrochloric acid TS to make exactly 100 mL. Pipet 10 mL of this solution, add 0.02 mol/L hydrochloric acid TS to make exactly 1000 mL, and use this solution as the sample solution. Separately, measure exactly a suitable volume of Standard Potassium

Stock Solution, dilute with 0.02 mol/L hydrochloric acid TS to make solutions containing 0.5 to 2.5 μg of potassium (K: 39.10) per mL, and use these solutions as the standard solutions. Perform the test with the sample solution and the standard solutions according to the Atomic Absorption Spectrophotometry under the following conditions, and determine the amount, Y (mg), of potassium in 1000 mL of the sample solution, using the calibration curve obtained from the standard solutions. The exchange quantity for potassium per g of dried Calcium Polystyrene Sulfonate is 0.053 to 0.071 g, calculating by the following equation.

$$\begin{aligned} &\text{Exchange quantity (mg) for potassium (K) per g of} \\ &\text{dried Calcium Polystyrene Sulfonate} \\ &= \frac{X - 100 Y}{W} \end{aligned}$$

X : The amount (mg) of potassium in 50 mL of Standard Potassium Stock Solution before exchange.

W : The amount (g) of dried Calcium Polystyrene Sulfonate taken.

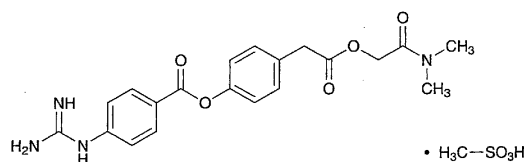
Gas: Combustible gas—Acetylene
Supporting gas—Air

Lamp: A potassium hollow-cathode lamp
Wavelength: 766.5 nm

Containers and storage Containers—Tight containers.

Camostat Mesilate

メシル酸カモスタット



$\text{C}_{20}\text{H}_{22}\text{N}_4\text{O}_5 \cdot \text{CH}_4\text{O}_3\text{S}$: 494.52
N,N-Dimethylcarbamoylmethyl 4-(4-guanidinobenzoyloxy)phenylacetate monomethanesulfonate [59721-29-8]

Camostat Mesilate, when dried, contains not less than 98.5% of $\text{C}_{20}\text{H}_{22}\text{N}_4\text{O}_5 \cdot \text{CH}_4\text{O}_3\text{S}$.

Description Camostat Mesilate occurs as white crystals or crystalline powder.

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

Identification (1) To 4 mL of a solution of Camostat Mesilate (1 in 2000) add 2 mL of 1-naphthol TS and 1 mL of diacetyl TS, and allow to stand for 10 minutes: a red color develops.

(2) Determine the absorption spectrum of a solution of Camostat Mesilate (1 in 100,000) as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum or the spectrum of a solution of Camostat Mesilate Reference Standard prepared in the same manner as the sample solution: both spectra exhibit similar intensities of absorption at the same wavelengths.