

(4) Heavy metals—Proceed with a volume of D-Sorbitol Solution, equivalent to 5.0 g of D-Sorbitol according to the labeled amount, and according to Method 1, perform the test. Prepare the control solution with 2.5 mL of Standard Lead Solution (not more than 5 ppm).

(5) Nickel—Take a volume of D-Sorbitol Solution, equivalent to 0.5 g of D-Sorbitol according to the labeled amount, add 3 drops of dimethylglyoxime TS and 3 drops of ammonia TS, and allow to stand for 5 minutes: no red color develops.

(6) Arsenic—Take a volume of D-Sorbitol Solution, equivalent to 1.5 g of D-Sorbitol according to the labeled amount, dilute with water or concentrate to 5 mL on a water bath, if necessary, cool, and perform the test with Apparatus B, using this solution as the test solution (not more than 1.3 ppm).

(7) Glucose—Take a volume of D-Sorbitol Solution, equivalent to 20.0 g of D-Sorbitol according to the labeled amount, dilute with water or concentrate to 40 mL on a water bath, if necessary, add 40 mL of Fehling's TS, and boil gently for 3 minutes. After cooling, filter the supernatant liquid cautiously through a glass filter (G4), leaving the precipitate in the flask as much as possible, wash the precipitate with hot water until the last washings no longer show alkalinity, and filter the washings through the glass filter. Dissolve the precipitate in the flask in 20 mL of iron (III) sulfate TS, filter through the glass filter, and wash the filter with water. Combine the filtrate and the washings, heat at 80°C, and titrate with 0.02 mol/L potassium permanganate VS: not more than 6.3 mL of 0.02 mol/L potassium permanganate VS is required.

(8) Sugars—Take a volume of D-Sorbitol Solution, equivalent to 20.0 g of D-Sorbitol according to the labeled amount, dilute with water or concentrate to 40 mL of a water bath, if necessary, add 8 mL of dilute hydrochloric acid, and heat under a reflux condenser in a water bath for 3 hours. After cooling, add 2 drops of methyl orange TS, followed by sodium hydroxide TS until an orange color develops, and add water to make 100 mL. Boil gently 10 mL of this solution with 10 mL of water and 40 mL of Fehling's TS for 3 minutes and proceed as directed in (7).

Residue on ignition Measure exactly a volume of D-Sorbitol Solution, equivalent to 5 g of D-Sorbitol according to the labeled amount, add 3 to 4 drops of sulfuric acid, and heat gently to evaporate. Ignite to burn, cool, and perform the test with the residue: not more than 1.0 mg.

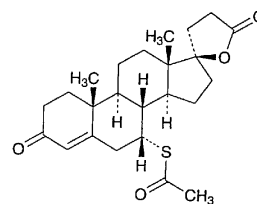
Assay Measure exactly a volume of D-Sorbitol Solution, equivalent to about 5 g of D-sorbitol (C₆H₁₄O₆) according to the labeled amount, and add water to make exactly 250 mL. Pipet 10 mL of this solution, add water to make exactly 100 mL. Pipet exactly 10 mL of the solution into an iodine flask, add exactly 50 mL of potassium periodate TS, and heat for 15 minutes in a water bath. Cool, add 2.5 g of potassium iodide, immediately stopper tightly, and shake well. Allow to stand for 5 minutes in a dark place, and titrate with 0.1 mol/L sodium thiosulfate VS (indicator: 3 mL of starch TS). Perform a blank determination.

Each mL of 0.1 mol/L sodium thiosulfate VS
= 1.8217 mg of C₆H₁₄O₆

Containers and storage Containers—Tight containers.

Spironolactone

スピロノラクトン



C₂₄H₃₂O₄S: 416.57

7 α -Acetylsulfanyl-3-oxo-17 α -pregn-4-ene-21,17 β -
carb lactone [52-01-7]

Spironolactone, when dried, contains not less than 97.0% and not more than 103.0% of C₂₄H₃₂O₄S.

Description Spironolactone occurs as a white to light yellow-brown, fine powder.

It is freely soluble in chloroform, soluble in ethanol (95), slightly soluble in methanol, and practically insoluble in water.

Melting point: 198 – 207°C (Insert the capillary tube into a bath at about 125°C, and continue the heating so that the temperature rises at a rate of about 10°C per minute in the range between 140°C and 185°C, and when the temperature is near the expected melting range, reduce the heating so that the temperature rises at a rate of about 3°C per minute.)

Identification (1) Determine the absorption spectrum of a solution of Spironolactone in methanol (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 Spironolactone Reference Standard prepared in the same manner as the sample solution: both spectra exhibit similar intensities of absorption at the same wavelengths.

(2) Determine the infrared absorption spectrum of Spironolactone, previously dried, as directed in the potassium bromide disk method under the Infrared Spectrophotometry, and compare the spectrum with the Reference Spectrum or the spectrum of Spironolactone Reference Standard: both spectra exhibit similar intensities of absorption at the same wave numbers. If any difference appears between the spectra, dissolve Spironolactone and Spironolactone Reference Standard in methanol, respectively, then evaporate methanol to dryness, and repeat the test on the residues.

Optical rotation [α]_D²⁰: –33 – –37° (after drying, 0.25 g, chloroform, 25 mL, 200 mm).

Purity (1) Mercapto compounds—Shake 2.0 g of Spironolactone with 20 mL of water, and filter. To 10 mL of the filtrate add 1 mL of starch TS and 0.05 mL of 0.01 mol/L iodine VS, and mix: a blue color develops.

(2) Related substances—Dissolve 0.20 g of Spironolactone in 10 mL of ethanol (95), and use this solution as the sample solution. Pipet 1 mL of this solution, add ethanol (95) to make exactly 100 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 for thin-layer chromatography. Develop the plate with *n*-butyl acetate to a distance of about 15 cm, and air-dry the plate. Spray evenly a solution of sulfuric acid in methanol (1 in 10) on the plate, and heat the plate at 105°C for 10 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 0.5% (1 g, 105°C, 2 hours).

Residue on ignition Not more than 0.1% (1 g).

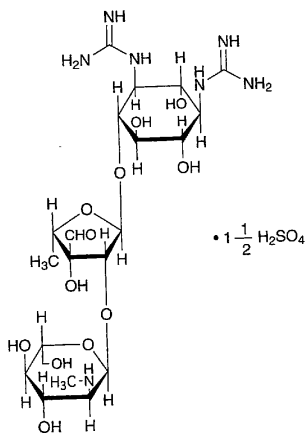
Assay Weigh accurately about 0.05 g each of Spironolactone and Spironolactone Reference Standard, previously dried at 105°C for 2 hours, dissolve in methanol to make exactly 250 mL. Pipet 5 mL each of these solutions, add methanol to make exactly 100 mL, and use these solutions as the sample solution and the standard solution, respectively. Perform the test with these solutions as directed under the Ultraviolet-visible Spectrophotometry, and determine the absorbances, A_T and A_S , of the sample solution and the standard solution at 238 nm.

$$\begin{aligned} & \text{Amount (mg) of } C_{24}H_{32}O_4S \\ &= \text{amount (mg) of Spironolactone Reference Standard} \\ & \quad \times \frac{A_T}{A_S} \end{aligned}$$

Containers and storage Containers—Tight containers.

Streptomycin Sulfate

硫酸ストレプトマイシン



$C_{21}O_{39}N_7O_{12} \cdot 1\frac{1}{2}H_2SO_4$: 728.69

O-2-Deoxy-2-methylamino- α -L-glucopyranosyl-(1 \rightarrow 2)-*O*-5-deoxy-3-*C*-formyl- α -L-lyxofuranosyl-(1 \rightarrow 4)-*N,N'*-diamidino-D-streptamine sesquisulfate [3810-74-0]

Streptomycin Sulfate conforms to the requirements of Streptomycin Sulfate in the Requirements for Antibiotic Products of Japan.

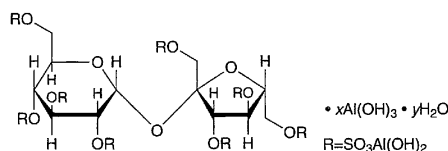
Description Streptomycin Sulfate occurs as a white to light yellowish white powder.

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

Sucralfate

Aluminum Sucrose Sulfate Ester

スクラルファート



$C_{12}H_{30}Al_8O_{51}S_8 \cdot xAl(OH)_3 \cdot yH_2O$
[54182-58-0]

Sucralfate contains not less than 17.0% and not more than 21.0% of aluminum (Al: 26.98) and not less than 34.0% and not more than 43.0% of sucrose octasulfate ester ($C_{12}H_{22}O_{35}S_8$: 982.80), calculated on the dried basis.

Description Sucralfate occurs as a white powder. It is odorless and tasteless.

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

It dissolves in dilute hydrochloric acid and in sulfuric acid-sodium hydroxide TS.

Identification (1) To 0.05 g of Sucralfate in a small test tube add 0.05 g of fresh pieces of sodium, and melt by careful heating. Immerse the test tube immediately in 100 mL of water, break the test tube, shake well, and filter. To 5 mL of the filtrate add 1 drop of sodium pentacyanonitrosylferrate (III) TS: a red-purple color develops.

(2) Dissolve 0.040 g of Sucralfate in 2 mL of dilute sulfuric acid, and add gently 2 mL of anthrone TS to make 2 layers: a blue color develops at the zone of contact, and gradually changes to blue-green.

(3) Dissolve 0.5 g of Sucralfate in 10 mL of dilute hydrochloric acid: the solution responds to the Qualitative Tests for aluminum.

Purity (1) Clarity and color of solution—Dissolve 1.0 g of Sucralfate in 10 mL of dilute sulfuric acid: the solution is clear and colorless.

(2) Chloride—Dissolve 0.5 g of Sucralfate in 30 mL of dilute nitric acid, and heat gently to boiling. After cooling, add water to make 100 mL, and to 10 mL of this solution add 3 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 with 0.70 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.50%).

(3) Heavy metals—Dissolve 1.0 g of Sucralfate in 20 mL of a solution of sodium chloride (1 in 5) and 1 mL of dilute hydrochloric acid, and to this solution add 2 mL of dilute acetic acid and water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution as follows: evaporate 1 mL of dilute hydrochloric acid on a water bath to dryness, and add 20 mL of a solution of