Carbon-Chain Length	Number of Double Bonds	Percentage (%)
18	3	5.0–11.0
20	0	≤1.0
20	1	≤1.0
22	0	≤1.0
22	1	≤0.3
24	0	≤0.5

Unsaponifiable matter ⟨401⟩**:** not more than 1.5%.

Peroxide value ⟨401⟩**—**

Mixed solvent—Mix 60 mL of glacial acetic acid with 40 mL of chloroform.

Potassium iodide solution—Prepare a saturated solution of potassium iodide in freshly boiled and cooled water, and store it protected from light. Discard it if it gives a color on addition of *Mixed solvent* and starch TS.

Procedure—Transfer about 10 g of Oil, accurately weighed, to a conical flask, add 30 mL of *Mixed solvent,* swirl to dissolve, add 0.5 mL of *Potassium iodide solution,* swirl the flask for 1 minute, accurately timed, add 30 mL of water, and titrate with 0.01 N sodium thiosulfate VS, with vigorous agitation, to a light yellow color. Add 0.5 mL of starch TS, and continue the titration until the blue color has disappeared. Perform a blank test, and make any necessary correction. Calculate the peroxide content, in mEq per kg, taken by the formula:

$$1000VN/W$$

in which V is the volume, in mL, of sodium thiosulfate required and N is its normality; and W is the weight, in g, of Oil taken. The limit is 10.0.

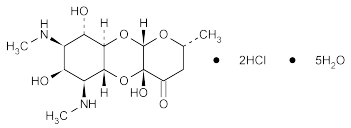
Water, *Method Ic* ⟨921⟩**:** not more than 0.1%.

Alkaline impurities—Mix 10 mL of acetone and 0.3 mL of water, and add 0.05 mL of bromophenol blue TS. If necessary, neutralize the solution to a green color with 0.01 N hydrochloric acid or 0.01 N sodium hydroxide. Add 10 mL of Soybean Oil, shake, and allow to stand. Titrate with 0.01 N hydrochloric acid VS to change the color of the upper layer to yellow: not more than 0.1 mL of 0.01 N hydrochloric acid is required.

Sterol composition—Proceed as directed in the section *Sterol Composition* under *Fats and Fixed Oils* ⟨401⟩: the sterol fraction of the Oil contains not more than 0.3% of brassicasterol.

Other requirements—For Soybean Oil intended for use in injectable dosage forms, which is specified in the *Labeling,* the requirements for *Acid Value, Peroxide Value, Unsaponifiable Matter,* and *Water* in the subsection *Other Vehicles* of the section *Ingredients* under *Injections* ⟨1⟩ must be met.

Spectinomycin Hydrochloride



$C_{14}H_{24}N_2O_7 \cdot 2HCl \cdot 5H_2O$ 495.35

4*H*-Pyrano[2,3-*b*][1,4]benzodioxin-4-one, decahydro-4a,7,9-trihydroxy-2-methyl-6,8-bis(methylamino)-, dihydrochloride, pentahydrate, [2*R*-(2α,4a*β*,5a*β*,6*β*,7*β*,8*β*,9α,9aα,10a*β*)]-.

(2*R*,4a*R*,5a*R*,6*S*,7*S*,8*R*,9*S*,9a*R*,10a*S*)-Decahydro-4a,7,9-trihydroxy-2-methyl-6,8-bis(methylamino)-4*H*-pyrano[2,3-*b*]1,4]benzodioxin-4-one dihydrochloride pentahydrate [22189-32-8].

Anhydrous 405.28 [21736-83-4].

» Spectinomycin Hydrochloride has a potency equivalent to not less than 603 µg of spectinomycin ($C_{14}H_{24}N_2O_7$) per mg.

Packaging and storage—Preserve in tight containers.

Labeling—Where it is intended for use in preparing injectable dosage forms, the label states that it is sterile or must be subjected to further processing during the preparation of injectable dosage forms.

USP Reference standards ⟨11⟩**—**
USP Spectinomycin Hydrochloride RS
USP Endotoxin RS

Identification, *Infrared Absorption* ⟨197M⟩**—**Do not dry specimen.

Crystallinity ⟨695⟩: meets the requirements.

Bacterial endotoxins ⟨85⟩**—**Where the label states that Spectinomycin Hydrochloride is sterile or that it must be subjected to further processing during the preparation of injectable dosage forms, it contains not more than 0.09 USP Endotoxin Unit per mg of spectinomycin.

Sterility ⟨71⟩**—**Where the label states that Spectinomycin Hydrochloride is sterile, it meets the requirements when tested as directed for *Membrane Filtration* under *Test for Sterility of the Product to be Examined*.

pH ⟨791⟩: between 3.8 and 5.6, in a solution containing 10 mg per mL.

Water, *Method I* ⟨921⟩: between 16.0% and 20.0%.

Residue on ignition ⟨281⟩: not more than 1.0%, the charred residue being moistened with 2 mL of nitric acid and 5 drops of sulfuric acid.

Assay—

Internal standard solution—Dissolve triphenylantimony in dimethylformamide to obtain a solution containing about 2 mg per mL.

Standard preparation—Transfer about 30 mg of USP Spectinomycin Hydrochloride RS, accurately weighed, to a glass-stoppered, 25-mL conical flask. Add 10.0 mL of *Internal standard solution* and 1.0 mL of hexamethyldisilazane, and shake intermittently for 1 hour.

Assay preparation—Proceed as directed under *Standard preparation* using Spectinomycin Hydrochloride.

Chromatographic system (see *Chromatography* ⟨621⟩)—The gas chromatograph is equipped with a flame-ionization detector and contains a 3-mm × 60-cm glass column packed with 5 percent phase G27 on 80- to 100-mesh support S1AB. The column and detector are maintained at about 190° and 220°, respectively, and the injection port at about 215°, and dry helium is used as the carrier gas at a flow rate of about 45 mL per minute. Chromatograph the *Standard preparation*, and record the chromatogram as directed for *Procedure:* the resolution, R, between the major peaks is not less than 2.0; and the relative standard deviation of the peak response ratios, R_S, from replicate injections of the *Standard preparation* is not more than 3.5%.

Procedure—Separately inject equal volumes (about 1 µL) of the *Standard preparation* and the *Assay preparation* into the chromatograph, record the chromatograms, and measure the responses for the major peaks. Calculate the ratio, R_U, of the response of the spectinomycin peak to the response of the internal standard peak in the chromatogram from the *Assay preparation*, and similarly calculate the ratio, R_S, in the chromatogram from the *Standard preparation.* Calculate the quantity, in µg, of $C_{14}H_{24}N_2O_7$ in the portion of Spectinomycin Hydrochloride taken to prepare the *Assay preparation* by the formula:

$$P(W_S)(R_U / R_S)$$

in which P is the potency of USP Spectinomycin Hydrochloride RS, in µg of spectinomycin per mg; and W_S is the weight, in

mg, of USP Spectinomycin Hydrochloride RS taken from the *Standard preparation;* and the other terms are as defined above.

Spectinomycin for Injectable Suspension

» Spectinomycin for Injectable Suspension contains an amount of Spectinomycin Hydrochloride equivalent to not less than 90.0 percent and not more than 120.0 percent of the labeled amount of spectinomycin ($C_{14}H_{24}N_2O_7$).

Packaging and storage—Preserve in *Containers for Sterile Solids* as described under *Injections* ⟨1⟩.

USP Reference standards ⟨11⟩**—**
USP Endotoxin RS
USP Spectinomycin Hydrochloride RS

Identification—*Infrared Absorption* ⟨197M⟩. Do not dry specimen.

pH ⟨791⟩: between 4.0 and 7.0, in the suspension constituted as directed in the labeling.

Other requirements—It conforms to the Definition, and meets the requirements for *Crystallinity, Bacterial endotoxins, Sterility, Water,* and *Residue on ignition* under *Spectinomycin Hydrochloride.* It meets also the requirements for *Uniformity of Dosage Units* ⟨905⟩ and *Labeling* under *Injections* ⟨1⟩.

Assay—

Internal standard solution, *Standard preparation,* and *Chromatographic system*—Prepare as directed in the *Assay* under *Spectinomycin Hydrochloride*.

Assay preparation 1—Suspend the contents of 1 container of Spectinomycin for Injectable Suspension in water, and dilute quantitatively with water to obtain a stock solution containing about 20 mg of spectinomycin per mL. Transfer 1.0 mL of this solution to a glass-stoppered, 25-mL conical flask, and freeze-dry. Add 10.0 mL of *Internal standard solution* and 1.0 mL of hexamethyldisilazane, and shake intermittently for 1 hour.

Assay preparation 2 (where the label states the quantity of spectinomycin in a given volume of constituted suspension)—Constitute 1 container of Spectinomycin for Injectable Suspension in a volume of water, accurately measured, corresponding to the volume of diluent specified in the labeling. Dilute an accurately measured portion of the constituted suspension quantitatively with water to obtain a stock solution containing about 20 mg of spectinomycin per mL. Transfer 1.0 mL of this solution to a glass-stoppered, 25-mL conical flask, and freeze-dry. Add 10.0 mL of *Internal standard solution* and 1.0 mL of hexamethyldisilazane, and shake intermittently for 1 hour.

Procedure—Proceed as directed in the *Assay* under *Spectinomycin Hydrochloride*. Calculate the quantity, in g, of $C_{14}H_{24}N_2O_7$ in the container of Spectinomycin for Injectable Suspension taken to prepare *Assay preparation 1* taken by the formula:

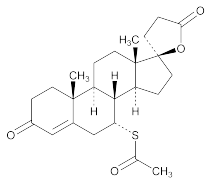
$$(L_1 / D_1)(P / 1000)(W_S)(R_U / R_S)$$

in which L_1 is the labeled quantity, in g, of $C_{14}H_{24}N_2O_7$ in the container, and D_1 is the concentration, in mg per mL, of spectinomycin in the stock solution used to prepare *Assay preparation 1*, on the basis of the labeled quantity in the container and the extent of dilution, and the other terms are as defined therein. Calculate the quantity, in mg, of $C_{14}H_{24}N_2O_7$ in each mL of constituted Injectable Suspension taken to prepare *Assay preparation 2* taken by the formula:

$$(L_2 / D_2)(P / 1000)(W_S)(R_U / R_S)$$

in which L_2 is the labeled quantity, in mg, of $C_{14}H_{24}N_2O_7$ in each mL of constituted suspension of Spectinomycin for Injectable Suspension, and D_2 is the concentration, in mg per mL, of spectinomycin in the stock solution used to prepare *Assay preparation 2*, on the basis of the labeled quantity in each mL of constituted suspension and the extent of dilution.

Spironolactone



$C_{24}H_{32}O_4S$ 416.57
Pregn-4-ene-21-carboxylic acid, 7-(acetylthio)-17-hydroxy-3-oxo-, γ-lactone, (7α,17α)-;
17-Hydroxy-7α-mercapto-3-oxo-17α-pregn-4-ene-21-carboxylic acid γ-lactone acetate [52-01-7].

DEFINITION
Spironolactone contains NLT 97.0% and NMT 103.0% of $C_{24}H_{32}O_4S$, calculated on the dried basis.

IDENTIFICATION

Change to read:

- **A. Infrared Absorption** ▲⟨197K⟩▲*USP35*
- **B. Ultraviolet Absorption** ⟨197U⟩
  Sample solution: 10 µg/mL in methanol
  Analytical wavelength: 238 nm
  Acceptance criteria: Absorptivities, calculated on the dried basis, do not differ by more than 3.0%.
- **C.**
  Sample solution: Add 100 mg to a mixture of 10 mL of water and 2 mL of 1 N sodium hydroxide.
  Analysis: Boil the mixture for 3 min, cool, and add 1 mL of glacial acetic acid and 1 mL of lead acetate TS.
  Acceptance criteria: A brown-to-black precipitate of lead sulfide is formed.

ASSAY
- **Procedure**
  Mobile phase: Methanol and water (60:40)
  Standard solution: 0.5 mg/mL of USP Spironolactone RS in a mixture of acetonitrile and water (1:1)
  Sample solution: 0.5 mg/mL of Spironolactone in a mixture of acetonitrile and water (1:1)
  Chromatographic system
  (See *Chromatography* ⟨621⟩, *System Suitability*.)
  Mode: LC
  Detector: UV 230 nm
  Column: 4.6-mm × 15-cm; packing L1
  Flow rate: 1 mL/min
  Injection size: 20 µL
  System suitability
  Sample: *Standard solution*
  Suitability requirements
  Tailing factor: NMT 2.0
  Relative standard deviation: NMT 1.5%
  Analysis
  Samples: *Standard solution* and *Sample solution*
  Calculate the percentage of spironolactone ($C_{24}H_{32}O_4S$) in the portion of sample taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak response from the *Sample solution*
r_S = peak response from the *Standard solution*