

0.1M potassium phosphate buffer, pH 8.0 (solution 3), to the reference concentration of 1.0 microgram of clindamycin per milliliter (estimated).

(3) *Sterility*. Proceed as directed in § 436.20 of this chapter, using the method described in paragraph (e)(1) of that section.

(4) *Pyrogens*. Proceed as directed in § 436.32(a) of this chapter, using a solution containing 24 milligrams of clindamycin per milliliter.

(5) [Reserved]

(6) *Depressor substances*. Proceed as directed in § 436.35 of this chapter.

(7) *Moisture*. Proceed as directed in § 436.201 of this chapter.

(8) *pH*. Proceed as directed in § 436.202 of this chapter, using an aqueous solution containing 10 milligrams per milliliter.

(9) *Crystallinity*. Proceed as directed in § 436.203(a) of this chapter.

(10) *Identity*. Proceed as directed in § 436.211 of this chapter, using the sample preparation method described in paragraph (b)(2) of that section, except dry the sample for 2 hours at 100° C. and allow to equilibrate with the atmosphere for 1 hour.

[39 FR 19161, May 30, 1974, as amended at 46 FR 60568, Dec. 11, 1981; 50 FR 19921, May 13, 1985]

#### § 453.30 Lincomycin hydrochloride monohydrate.

(a) *Requirements for certification—(1) Standards of identity, strength, quality, and purity*. Lincomycin hydrochloride monohydrate is the monohydrated hydrochloride salt of lincomycin. It is freely soluble in water and soluble in acetone and dimethylformamide. It is so purified and dried that:

(i) Its potency is not less than 790 micrograms of lincomycin per milligram.

(ii) [Reserved]

(iii) Its moisture content is not less than 3.0 percent and is not more than 6.0 percent.

(iv) Its pH in an aqueous solution containing 100 milligrams per milliliter is not less than 3.0 and not more than 5.5.

(v) Its specific rotation in an aqueous solution at 25° C. is not less than +135° and not more than +150°.

(vi) It passes the infrared identity test.

(vii) Its content of lincomycin B is not more than 5 percent.

(viii) It passes the identity test if the elution pattern of the lincomycin sample compares quantitatively to that of the lincomycin working standard under identical conditions of gas liquid chromatography.

(ix) It is crystalline.

(2) *Labeling*. It shall be labeled in accordance with the requirements of § 432.5(b) of this chapter.

(3) *Requests for certification; samples*. In addition to complying with the requirements of § 431.1 of this chapter, each such request shall contain:

(i) Results of tests and assays on the batch for potency, moisture, pH, specific rotation, infrared absorption spectrum, lincomycin B content, crystallinity, and identity.

(ii) Samples of the batch: 10 packages, each containing approximately 300 milligrams.

(b) *Tests and methods of assay—(1) Potency*. Use either of the following methods; however, the results obtained from the gas liquid chromatography assay shall be conclusive.

(i) *Microbiological turbidimetric assay*. Proceed as directed in § 436.106 of this chapter, preparing the sample for assay as follows: Dissolve an accurately weighed sample in sufficient sterile distilled water to obtain a stock solution of convenient concentration. Further dilute an aliquot of the stock solution with sterile distilled water to the reference concentration of 0.5 microgram of lincomycin per milliliter (estimated).

(ii) *Gas liquid chromatography assay*. Proceed as directed in § 436.306 of this chapter.

(2) [Reserved]

(3) *Moisture*. Proceed as directed in § 436.201 of this chapter.

(4) *pH*. Proceed as directed in § 436.202 of this chapter, using an aqueous solution containing 100 milligrams per milliliter.

(5) *Specific rotation*. Accurately weigh 500 milligrams of lincomycin hydrochloride monohydrate in a 25-milliliter, glass stoppered volumetric flask and fill to volume with distilled water. Proceed as directed in § 436.210 of this

chapter, using a 2.0-decimeter polarimeter tube and calculate the specific rotation on an anhydrous basis.

(6) *Infrared absorption spectrum.* Proceed as directed in § 436.211 of this chapter, using the sample preparation method described in paragraph (b)(2) of that section.

(7) *Lincomycin B content.* Proceed as directed in § 436.306 of this chapter.

(8) *Identity.* Proceed as described in § 436.306 of this chapter.

(9) *Crystallinity.* Proceed as directed in § 436.203(a) of this chapter.

[39 FR 19161, May 30, 1974, as amended at 46 FR 3839, Jan. 16, 1981; 50 FR 19921, May 13, 1985]

**§ 453.30a Sterile lincomycin hydrochloride monohydrate.**

(a) *Requirements for certification—(1) Standards of identity, strength, quality, and purity.* Lincomycin hydrochloride monohydrate is the monohydrated hydrochloride salt of lincomycin. It is freely soluble in water and soluble in acetone and dimethylformamide. It is so purified and dried that:

(i) Its potency is not less than 790 micrograms of lincomycin per milligram.

(ii) It is sterile.

(iii) [Reserved]

(iv) It is nonpyrogenic.

(v) It contains no depressor substances.

(vi) Its moisture content is not less than 3.0 percent and not more than 6.0 percent.

(vii) Its pH in an aqueous solution containing 100 milligrams per milliliter is not less than 3.0 and not more than 5.5.

(viii) Its specific rotation in an aqueous solution at 25° C. is not less than +135° and not more than +150°.

(ix) It passes the infrared identity test.

(x) Its content of lincomycin B is not more than 5 percent.

(xi) It passes the identity test if the elution pattern of the lincomycin sample compares quantitatively to that of the lincomycin working standard under identical conditions of gas liquid chromatography.

(xii) It is crystalline.

(2) *Labeling.* It shall be labeled in accordance with the requirements of § 432.5(b) of this chapter.

(3) *Requests for certification; samples.* In addition to complying with the requirements of § 431.1 of this chapter, each such request shall contain:

(i) Results of tests and assays on the batch for potency, sterility, pyrogens, depressor substances, moisture, pH, specific rotation, infrared absorption spectrum, lincomycin B content, identity, and crystallinity.

(ii) Samples required:

(a) For all tests except sterility: 10 packages, each containing approximately 300 milligrams.

(b) For sterility testing: 20 packages, each containing approximately 300 milligrams.

(b) *Tests and methods of assay—(1) Potency.* Use either of the following methods; however, the results obtained from the gas liquid chromatography assay shall be conclusive.

(i) *Microbiological turbidimetric assay.* Proceed as directed in § 436.106 of this chapter, preparing the sample for assay as follows: Dissolve an accurately weighed sample in sufficient sterile distilled water to obtain a stock solution of convenient concentration. Further dilute an aliquot of the stock solution with sterile distilled water to the reference concentration of 0.5 microgram of lincomycin per milliliter (estimated).

(ii) *Gas liquid chromatography assay.* Proceed as directed in § 436.306 of this chapter.

(2) *Sterility.* Proceed as directed in § 436.20 of this chapter, using the method described in paragraph (e)(1) of that section.

(3) [Reserved]

(4) *Pyrogens.* Proceed as directed in § 436.32(a) of this chapter, using a solution containing 0.5 milligram of lincomycin per milliliter.

(5) *Depressor substances.* Proceed as directed in § 436.35 of this chapter.

(6) *Moisture.* Proceed as directed in § 436.201 of this chapter.

(7) *pH.* Proceed as directed in § 436.202 of this chapter, using an aqueous solution containing 100 milligrams per milliliter.