

(3) *pH*. Proceed as directed in § 436.202 of this subchapter, using the drug reconstituted as directed in the labeling.

[39 FR 19161, May 30, 1974, as amended at 50 FR 19921, May 13, 1985]

§ 453.130 Lincomycin hydrochloride oral dosage forms.

§ 453.130a Lincomycin hydrochloride monohydrate capsules.

(a) *Requirements for certification—(1) Standards of identity, strength, quality, and purity.* Lincomycin hydrochloride monohydrate capsules are composed of lincomycin hydrochloride monohydrate and suitable diluents, enclosed in a gelatin capsule. Each capsule contains 250 milligrams of lincomycin or 500 milligrams of lincomycin. The lincomycin content is satisfactory if it is not less than 90 percent and not more than 120 percent of the number of milligrams of lincomycin that it is represented to contain. Its moisture content is not more than 7.0 percent. The lincomycin hydrochloride monohydrate used conforms to the standards prescribed by § 453.30(a)(1).

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

(a) The lincomycin hydrochloride monohydrate used in making the batch for potency, moisture, pH, specific rotation, infrared absorption spectrum, and identity.

(b) The batch for potency and moisture.

(ii) Samples required:

(a) The lincomycin hydrochloride monohydrate used in making the batch: 10 packages, each containing approximately 300 milligrams.

(b) The batch: A minimum of 30 capsules.

(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: Place a representative number of capsules into a high-speed glass blender jar with sufficient sterile distilled water to obtain a stock solution of convenient concentration. Blend for 3 to 5 minutes. Remove an aliquot of the stock solution and further dilute 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, except prepare the sample for assay as follows: Place the contents of 5 capsules in a 100-milliliter volumetric flask and add about 60 milliliters of methanol. Place on a steam bath and allow to boil gently for 5 minutes. Remove from the steam bath, add more methanol, and adjust to mark after cooling to ambient temperature. Dilute an aliquot equivalent to 50 milligrams of lincomycin to 25 milliliters with methanol. Transfer 2 milliliters to a centrifuge tube and evaporate to dryness on a steam bath with a stream of dry air. Dissolve the residue in 1 milliliter of dry pyridine. Calculate the lincomycin content of the capsules as follows:

$$\text{Lincomycin content in} \\ \text{milligrams per capsule} = \frac{R_u \times W_s \times d \times f}{R_s \times N}$$

where:

R_u =Area of lincomycin sample peak/Area of internal standard;

R_s =Area of lincomycin standard peak/Area of internal standard;

W_s =Weight of lincomycin working standard in milligrams;

d =Dilution factor;

f =Potency of lincomycin working standard in milligrams of lincomycin per milligram;

N =Number of capsules used.

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

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

§ 453.130b Lincomycin hydrochloride syrup.

(a) *Requirements for certification—(1) Standards of identity, strength, quality, and purity.* Lincomycin hydrochloride syrup is a syrup containing lincomycin hydrochloride monohydrate, one or