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Ergoloid Mesylates Tablets

» Ergoloid Mesylates Tablets contain not less than 90.0 percent and not more than 110.0 percent of the labeled amount of ergoloid mesylates, consisting of not less than 30.3 percent and not more than 36.3 percent of the methanesulfonate salt of each of the individual alkaloids (dihydroergocristine, dihydroergocornine, and dihydroergocryptine). The ratio of *alpha*- to *beta*-dihydroergocryptine mesylate is not less than 1.5:1.0 and not more than 2.5:1.0.

Packaging and storage—Preserve in tight, light-resistant containers. Store at 25°, excursions permitted between 15° and 30°.

Labeling—Label the Tablets to indicate that they are intended for swallowing intact.

USP Reference standards ⟨11⟩—
USP Ergoloid Mesylates RS

Identification—Mix a small amount of powdered Tablets, equivalent to about 5 mg of ergoloid mesylates, with 5 mL of water and 5 mL of a mixture of equal volumes of glacial acetic acid and sulfuric acid, and add 1 drop of freshly prepared ferric chloride solution (1 in 20): a violet-blue color develops within 5 minutes.

Dissolution ⟨711⟩—

Medium: water; 500 mL.

Apparatus 2: 50 rpm, the distance between the paddle blade and the inside bottom of the vessel being maintained at 4.5 ± 0.2 cm during the test.

Time: 30 minutes.

Determine the amount of ergoloid mesylates dissolved using the following procedure.

Mobile phase—Prepare as directed in the *Assay.*

Standard solution—Dissolve an accurately weighed quantity of USP Ergoloid Mesylates RS in water to obtain a solution having a known concentration of about 50 µg per mL. Transfer 4 mL of this solution for every 0.5 mg of ergoloid mesylates contained in the Tablets to a 200-mL volumetric flask, add 1 mL of 0.1 N hydrochloric acid and 100 mL of water, mix, and dilute with water to volume.

Test solution—Transfer a 20-mL portion of the solution under test to a suitable container, add 100 µL of 0.1 N hydrochloric acid, and mix.

Chromatographic system—Proceed as directed in the *Assay* except that the sum of the relative standard deviation values for the four ergoloid mesylates peaks for replicate injections is not more than 3.0%.

Procedure—Separately inject equal volumes (about 500 µL) of the *Standard solution* and the *Test solution* into the chromatograph, record the chromatograms, and measure the responses for the major peaks. Calculate the quantity of ergoloid mesylates as directed in the *Assay.*

Tolerances—Not less than 75% *(Q)* of the labeled amount of ergoloid mesylates is dissolved in 30 minutes.

Uniformity of dosage units ⟨905⟩: meet the requirements.

Assay—

Mobile phase—Prepare a filtered and degassed solution containing a mixture of water, acetonitrile, and triethylamine (700:300:9). Make adjustments if necessary (see *System Suitability* under *Chromatography ⟨621⟩*).

Internal standard solution—Transfer about 113 mg of papaverine hydrochloride to a 1-L flask. Add a mixture of 0.01 M tartaric acid and acetonitrile (2:1) to volume, and mix.

Standard preparation—Transfer about 33 mg of USP Ergoloid Mesylates RS, accurately weighed, to a 100-mL volumetric flask. Dissolve in and dilute with *Internal standard solution* to volume, and mix. Use a freshly prepared solution.

Assay preparation—Weigh and finely powder not fewer than 20 Tablets. Transfer an accurately weighed portion of the powder, equivalent to about 5 mg of ergoloid mesylates, to a 50-mL centrifuge tube. Add 15.0 mL of *Internal standard solution,* insert the stopper into the tube, and shake for about 15 minutes. Centrifuge, filter if necessary, and use the clear supernatant.

Chromatographic system (see *Chromatography ⟨621⟩*)—The liquid chromatograph is equipped with a 280-nm detector and a 4.6-mm × 15-cm column that contains packing L1. [Note—Use an L1 column capable of handling pH values greater than 11.] The flow rate is about 1.5 mL per minute. Chromatograph the *Standard preparation,* and record the peak responses as directed for *Procedure:* the column efficiency determined for the dihydro-β-ergocryptine mesylate peak is not less than 1000 theoretical plates; the tailing factor for the dihydro-β-ergocryptine mesylate peak is not more than 2.0; the resolution, *R,* between the dihydro-α-ergocryptine mesylate and dihydroergocristine mesylate is not less than

2.0, and between dihydroergocristine and dihydro-β-ergocryptine peaks is not less than 2.0; and the relative standard deviation of the ratio of the sum of the four peaks to the internal standard for replicate injections is not more than 1.5%.

Procedure—Separately inject equal volumes (about 20 µL) of the *Standard preparation* and the *Assay preparation* into the chromatograph, record the chromatograms, and measure the responses for the major peaks. The order of elution is papaverine, dihydroergocornine, dihydro-α-ergocryptine, dihydroergocristine, and dihydro-β-ergocryptine. Calculate the quantity, in mg, of ergoloid mesylates in the portion of Tablets taken by the formula:

$$15C(R_U/R_S)$$

in which C is the concentration, in mg per mL, of USP Ergoloid Mesylates RS in the *Standard preparation;* and R_U and R_S are the sums of the ratios of responses of the four major peaks to the response of the internal standard peak obtained from the *Assay preparation* and the *Standard preparation,* respectively. Calculate the percentage of each of the individual alkaloids taken by the formula:

$$100R_i(MW)y_i/\Sigma[R_i(MW)_i]$$

in which R_i is the peak response ratio of the individual alkaloid to the internal standard; $(MW)_i$ is the molecular weight of the individual alkaloid; and $\Sigma[R_i(MW)_i]$ is the summation of the products of peak response ratios and molecular weights for the four alkaloids.

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
ERGOLOID MESYLATES TABLETS	Documentary Standards Support	SM42020 Small Molecules 4

Chromatographic Database Information: Chromatographic Database

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