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Ergotamine Tartrate Sublingual Tablets

DEFINITION

Ergotamine Tartrate Sublingual Tablets contain NLT 90.0% and NMT 110.0% of the labeled amount of ergotamine tartrate [$(C_{33}H_{35}N_5O_5)_2 \cdot C_4H_6O_6$].

IDENTIFICATION

• **A.**

Solution A: Shake chloroform with ammonium hydroxide, draw off the chloroform layer, and use the chloroform saturated with ammonia.

Solution B: Combine equal volumes of glacial acetic acid and ethyl acetate.

Solution C: Combine equal volumes of ferric chloride TS and water.

Sample solution: Triturate a portion of finely powdered Tablets, equivalent to 5 mg of ergotamine tartrate, with 10 mL of solvent hexane for a few min. Allow to settle, and discard the solvent hexane extract. To the residue add 10 mL of *Solution A*, triturate for a few min, filter, and evaporate the filtrate on a steam bath to dryness. Dissolve the residue in 8 mL of *Solution B*.

Analysis

Sample: *Sample solution*

Part 1: To 1 mL of *Sample solution* add slowly, with continuous agitation and cooling, 1 mL of sulfuric acid.

Part 2: To the resulting solution from *Part 1,* add 0.1 mL of *Solution C*.

Acceptance criteria: Both criteria for *Part 1* and *Part 2* must be met.

Part 1: A blue color with a red tinge develops.

Part 2: The red tinge becomes less apparent and the blue color more pronounced.

ASSAY

• **Procedure**

Mobile phase: Acetonitrile and 0.01 M monobasic potassium phosphate (55:45)

Diluent: Acetonitrile and water (55:45)

Internal standard solution: 0.16 mg/mL of ergonovine maleate in *Diluent*

Standard stock solution: 0.2 mg/mL of USP Ergotamine Tartrate RS in *Diluent*

Standard solution: 0.02 mg/mL of USP Ergotamine Tartrate RS from *Standard stock solution* and 0.016 mg/mL of ergonovine maleate from *Internal standard solution* prepared as follows. Transfer 5.0 mL of *Standard stock solution* to a 50-mL volumetric flask, add 5.0 mL of *Internal standard solution,* and dilute with *Diluent* to volume.

Sample solution: Nominally 0.02 mg/mL of ergotamine tartrate from Tablets prepared as follows. Transfer a number of whole Tablets, equivalent to 10 mg of ergotamine tartrate, to a 500-mL volumetric flask. Add 50.0 mL of *Internal standard solution* and 300 mL of *Diluent.* Sonicate for 10 min, and dilute with *Diluent* to volume. Pass through a membrane disk of 0.45-µm pore size, discarding the first 25 mL of the filtrate.

Chromatographic system

(See *Chromatography, System Suitability* ⟨621⟩.)

Mode: LC

Detector: UV 254 nm

Column: 3.9-mm × 30-cm; packing L1

Flow rate: 1 mL/min

Injection volume: 20 µL

System suitability

Sample: *Standard solution*

[Note—The relative retention times for ergonovine maleate and ergotamine tartrate are about 0.7 and 1.0, respectively.]

Suitability requirements

Resolution: NLT 3.0 between the ergotamine tartrate and ergonovine maleate

Column efficiency: NLT 3000 theoretical plates for ergotamine tartrate

Tailing factor: NMT 2.0 for ergotamine tartrate

Relative standard deviation: NMT 2.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of ergotamine tartrate $[(C_{33}H_{35}N_5O_5)_2 \cdot C_4H_6O_6]$ in the portion of Tablets taken:

$$\text{Result} = (R_U/R_S) \times (C_S/C_U) \times 100$$

R_U = peak response ratio of ergotamine tartrate to the internal standard from the *Sample solution*

R_S = peak response ratio of ergotamine tartrate to the internal standard from the *Standard solution*

C_S = concentration of USP Ergotamine Tartrate RS in the *Standard solution* (mg/mL)

C_U = nominal concentration of ergotamine tartrate in the *Sample solution* (mg/mL)

Acceptance criteria: 90.0%–110.0%

PERFORMANCE TESTS

- **Disintegration ⟨701⟩:** 5 min
- **Uniformity of Dosage Units ⟨905⟩:** Meet the requirements

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in well-closed, light-resistant containers. Store at 25°, excursions permitted between 15° and 30°.
- **Labeling:** Label Tablets to indicate that they are intended for sublingual administration.
- **USP Reference Standards ⟨11⟩**
  USP Ergotamine Tartrate RS

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

Topic/Question	Contact	Expert Committee
ERGOTAMINE TARTRATE SUBLINGUAL TABLETS	Documentary Standards Support	SM42020 Small Molecules 4

Chromatographic Database Information: Chromatographic Database

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