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Ergotamine Tartrate and Caffeine Tablets

DEFINITION
Ergotamine Tartrate and Caffeine Tablets contain NLT 90.0% and NMT 110.0% of the labeled amounts of ergotamine tartrate $[(C_{33}H_{35}N_5O_5)_2 \cdot C_4H_6O_6]$ and of caffeine $(C_8H_{10}N_4O_2)$.

[NOTE—Throughout the following procedures, protect samples, the Reference Standards, and solutions containing them by conducting the procedures without delay, under subdued light, or using low-actinic glassware.]

IDENTIFICATION

- A.** The retention times of the ergotamine and caffeine peaks of the *Sample solution* correspond to those of the *Standard solution*, as obtained in the Assay.

ASSAY

- PROCEDURE**
Buffer: Triethylamine, sulfuric acid, and water (4:0.5:2000) adjusted with sulfuric acid or 1 N sodium hydroxide to a pH of 3.0
Solution A: Acetonitrile and *Buffer* (15:85)
Solution B: Acetonitrile and *Buffer* (42:58)
Mobile phase: See [Table 1](#).

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	100	0
12	0	100
17	0	100
17.5	100	0
25	100	0

Diluent: 10 g/L of tartaric acid in water. Use *d*- or *l*-tartaric acid. Do not use a racemic mixture. Use a freshly prepared solution.

Ergotamine tartrate stock solution: 0.2 mg/mL of [USP Ergotamine Tartrate RS](#) in *Diluent* prepared as follows. Transfer a suitable quantity of [USP Ergotamine Tartrate RS](#) to an appropriate volumetric flask, and add 66% of the flask volume of *Diluent*. Sonicate for 5 min, and allow the solution to equilibrate to room temperature. Dilute with *Diluent* to volume.

Standard stock solution: 0.01 mg/mL of [USP Ergotamine Tartrate RS](#) and 1 mg/mL of [USP Caffeine RS](#) in *Diluent* prepared as follows. Transfer a suitable quantity of [USP Caffeine RS](#) to an appropriate volumetric flask, and add 66% of the flask volume of *Diluent*. Sonicate for 5 min, and allow the solution to equilibrate to room temperature. Add a suitable volume of *Ergotamine tartrate stock solution*, and dilute with *Diluent* to volume.

Standard solution: 0.001 mg/mL of [USP Ergotamine Tartrate RS](#) and 0.1 mg/mL of [USP Caffeine RS](#) from *Standard stock solution* in *Diluent*

System suitability solution: 0.001 mg/mL of [USP Ergotamine RS](#) in *Standard solution*

Sample stock solution: Transfer NLT 10 Tablets to a 1-L volumetric flask. Add *Diluent* to 66% of the flask volume. Shake mechanically for NLT 45 min to promote dissolution. It may be necessary to loosen and retighten the closure multiple times during the shaking to relieve pressure. Dilute with *Diluent* to volume. Use within 9 h.

Sample solution: Nominally 0.001 mg/mL of ergotamine tartrate and 0.1 mg/mL of caffeine from *Sample stock solution* in *Diluent*. Pass through a suitable filter, and use the filtrate within 9 h.

Chromatographic system

(See [Chromatography \(621\)](#), [System Suitability](#).)

Mode: LC

Detectors: UV 254 nm (for caffeine), in series with a fluorometric detector (for ergotamine and ergotamine), with excitation at 250 nm and detection at 435 nm

Column: 4.6-mm × 25-cm; 5-μm packing L7

Flow rate: 1.5 mL/min

Injection volume: 60 μL

System suitability

Samples: *Standard solution* and *System suitability solution*

[NOTE—The relative retention times for caffeine, ergotamine, and ergotamine are 1.0, 2.6, and 2.8, respectively.]

Suitability requirements

Resolution: NLT 1.5 for ergotamine and ergotamine, *System suitability solution*

Tailing factor: NMT 2.0 for ergotamine, *Standard solution*

Relative standard deviation: NMT 2.0% each for caffeine and ergotamine, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of caffeine ($C_8H_{10}N_4O_2$) in the portion of Tablets taken:

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

r_U = peak response of caffeine at 254 nm from the *Sample solution*

r_S = peak response of caffeine at 254 nm from the *Standard solution*

C_S = concentration of [USP Caffeine RS](#) in the *Standard solution* (mg/mL)

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

Calculate the percentage of the labeled amount of ergotamine tartrate [$(C_{33}H_{35}N_5O_9)_2 \cdot C_4H_6O_6$] in the portion of Tablets taken:

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

r_U = peak response of ergotamine at 435 nm from the *Sample solution*

r_S = peak response of ergotamine at 435 nm 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% each of the labeled amounts of caffeine and ergotamine tartrate

PERFORMANCE TESTS

• [DISSOLUTION \(711\)](#)

Medium: 10 g/L of tartaric acid in water. Use *d*- or *l*-tartaric acid. Do not use a racemic mixture. Use a freshly prepared solution; 900 mL.

Apparatus 2: 75 rpm

Time: 30 min

Buffer, Solution A, Solution B, Mobile phase, Diluent, Standard solution, System suitability solution, Chromatographic system, and System suitability: Proceed as directed in the Assay.

Sample solution: Pass a portion of the solution under test through a suitable filter, and discard the first portion of the filtrate.

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of caffeine ($C_8H_{10}N_4O_2$) dissolved:

$$\text{Result} = (r_U/r_S) \times C_S \times V \times (1/L) \times 100$$