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Add the following:

▲ Mesalamine Suppositories

DEFINITION

Mesalamine Suppositories contain NLT 90.0% and NMT 110.0% of the labeled amount of mesalamine ($C_7H_7NO_3$).

IDENTIFICATION

- **A.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the Assay.
- **B.** The UV spectrum of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the Assay.

ASSAY

• PROCEDURE

Buffer: Dissolve 1.39 g of [potassium phosphate, monobasic](#) and 2.24 g of [octanesulfonic acid sodium salt, monohydrate](#) in 1 L of [water](#).

Adjust with [phosphoric acid](#) to a pH of 2.2.

Mobile phase: [Methanol](#), [acetonitrile](#), and *Buffer* (90:35:1000)

Standard solution: 0.1 mg/mL of [USP Mesalamine RS](#) in *Mobile phase*. Sonicate to dissolve, if necessary.

Sample stock solution: Nominally 5 mg/mL of mesalamine in 0.1 N [hydrochloric acid](#) prepared as follows. Transfer Suppositories (NLT 5) to a suitable volumetric flask. Melt the Suppositories completely at 60°. Add 80% of the flask volume of preheated 0.1 N [hydrochloric acid](#) at 60°. Stir vigorously to avoid lump formation. [NOTE—About 20 min stirring time is suggested.] Sonicate for NLT 15 min with intermittent shaking. Cool to room temperature and dilute with 0.1 N [hydrochloric acid](#) to volume. Allow to settle and pass through a suitable filter of 0.45-μm pore size.

Sample solution: Nominally 0.1 mg/mL of mesalamine from the *Sample stock solution* in *Mobile phase*

Chromatographic system

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

Mode: LC

Detector: UV 220 nm. For *Identification B*, use a diode array detector in the range of 200–400 nm.

Column: 4.6-mm × 15-cm; 5-μm packing [L7](#)

Column temperature: 30°

Flow rate: 2 mL/min

Injection volume: 20 μL

Run time: NLT 1.4 times the retention time of mesalamine

System suitability

Sample: *Standard solution*

Suitability requirements

Tailing factor: NMT 2.0

Relative standard deviation: NMT 1.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of mesalamine ($C_7H_7NO_3$) in the Suppositories taken:

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

r_U = peak response of mesalamine from the *Sample solution*

r_S = peak response of mesalamine from the *Standard solution*

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

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

Acceptance criteria: 90.0%–110.0%

PERFORMANCE TESTS

• [DISSOLUTION \(711\)](#)

Medium: Dissolve 27.22 g of [potassium phosphate, monobasic](#) and 6.5 g of [sodium hydroxide](#) in 1 L of [water](#). Adjust with 50% (w/v) [sodium hydroxide](#) solution to a pH of 7.5; 900 mL

Apparatus 2: 125 rpm, with suitable sinker

Temperature: 40°

Time: 60 min

Buffer: Transfer 7.1 g of [sodium phosphate, dibasic anhydrous](#) and 6.9 g of [sodium phosphate, monobasic](#) (monohydrate) in a 1000-mL volumetric flask, and dissolve in 800 mL of [water](#). Add 7.5 mL of [tetrabutylammonium hydroxide, 25 percent in methanol](#), mix, and dilute with [water](#) to volume.

Mobile phase: [Methanol](#) and *Buffer* (30:70)

Standard solution: 0.11 mg/mL of [USP Mesalamine RS](#) in *Medium*. Sonicate to dissolve, if necessary.

Sample solution: Pass a portion of the solution under test through a suitable filter of 0.45-μm pore size. Dilute 2.0 mL of filtrate with *Medium* to 20 mL.

Chromatographic system

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

Mode: LC

Detector: UV 330 nm

Column: 4.6-mm × 15-cm; 5-μm packing [L1](#)

Column temperature: 40°

Flow rate: 1 mL/min

Injection volume: 5 μL

Run time: NLT 2.4 times the retention time of mesalamine

System suitability

Sample: *Standard solution*

Suitability requirements

Tailing factor: NMT 2.0

Relative standard deviation: NMT 2.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of mesalamine ($C_7H_7NO_3$) dissolved:

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

r_U = peak response of mesalamine from the *Sample solution*

r_S = peak response of mesalamine from the *Standard solution*

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

V = volume of *Medium*, 900 mL

D = dilution factor for the *Sample solution*, 10

L = label claim of mesalamine (mg/Suppository)

Tolerances: NLT 80% (Q) of the labeled amount of mesalamine ($C_7H_7NO_3$) is dissolved.

• [UNIFORMITY OF DOSAGE UNITS \(905\)](#): Meets the requirements

IMPURITIES

• ORGANIC IMPURITIES

Buffer, Mobile phase, and Sample stock solution: Prepare as directed in the Assay.

System suitability solution: 2.5 μg/mL each of [USP Mesalamine RS](#) and [USP 3-Aminosalicylic Acid RS](#) in *Mobile phase*. Sonicate to dissolve, if necessary.

Standard solution: 1 μg/mL of [USP Mesalamine RS](#) in *Mobile phase*. Sonicate to dissolve, if necessary.



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