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Aspirin Suppositories

DEFINITION
Aspirin Suppositories contain NLT 90.0% and NMT 110.0% of the labeled amount of aspirin ($C_9H_8O_4$).

IDENTIFICATION

• **A.**

Sample: Transfer a portion of the melted Suppositories obtained in the *Assay*, equivalent to about 1 g of aspirin, to a 125-mL conical flask. Add 20 mL of alcohol and warm until completely disintegrated. Cool in an ice bath for 5 min, filter, and evaporate the filtrate to dryness. Use the residue.

Analysis: Heat the *Sample* with water for several minutes, cool, and add 1 or 2 drops of ferric chloride TS.

Acceptance criteria: A violet-red color is produced.

Change to read:

• **B. Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*:** ▲197A or ▲ (USP 1-May-2024) 197K

Sample: Prepare as directed in the *Sample* in *Identification A*.

Acceptance criteria: Meet the requirements

ASSAY

• **Procedure**

[Note—Use chloroform that recently was saturated with water.]

Chromatographic column: Prepare on the day of use. Insert a small pledget of glass wool above the stem constriction of a 20-cm × 2.5-cm chromatographic tube, and uniformly pack with a mixture of about 3 g of chromatographic siliceous earth and 2 mL of sodium bicarbonate solution (1 in 12).

Diluent: Chloroform and glacial acetic acid (99:1)

Standard stock solution: 1 mg/mL of USP Aspirin RS prepared as follows. Transfer 50 mg of USP Aspirin RS to a 50-mL volumetric flask, add 0.5 mL of glacial acetic acid, and dilute with chloroform to volume.

Standard solution: 0.05 mg/mL USP Aspirin RS from *Standard stock solution* in *Diluent*

Sample solution: Nominally 0.05 mg/mL of aspirin prepared as follows. Tare a small dish and glass rod, and then add NLT 5 Suppositories in the dish. Heat gently on a steam bath until the Suppositories are melted. Mix the melt by stirring with the rod, cool while stirring, and weigh. Transfer a portion of the cooled melt, equivalent to 50 mg of aspirin, to a 50-mL volumetric flask containing 1 mL of a solution of hydrochloric acid in methanol (1 in 50), add 40 mL of chloroform, mix, and dilute with chloroform to volume. Pipet 5 mL of the solution into the *Chromatographic column*, wash with 5 mL and then with 25 mL of chloroform, and discard the washings. Without delay, elute into a 100-mL volumetric flask with about 10 mL of a solution of glacial acetic acid in chloroform (1 in 10), and then with 85 mL of *Diluent*, and dilute with *Diluent* to volume.

Instrumental conditions

Mode: UV

Analytical wavelength: UV 280 nm

Cell: 1 cm

Blank: Chloroform

Analysis

Samples: *Standard solution* and *Sample solution*

Without delay, concomitantly determine the absorbances of the *Standard solution* and *Sample solution*.

Calculate the percentage of the labeled amount of aspirin ($C_9H_8O_4$) in the portion of Suppositories taken:

$$\text{Result} = (A_U/A_S) \times (C_S/C_U) \times 100$$

A_U = absorbance of the *Sample solution*

A_S = absorbance of the *Standard solution*

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

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

Acceptance criteria: 90.0%—110.0%

IMPURITIES

• **Limit of Free Salicylic Acid**

Ferric chloride–urea reagent: Prepare on the day of use. Add 60 g of urea to a mixture of 8 mL of ferric chloride solution (6 in 10) and 42 mL of 0.05 N hydrochloric acid. Dissolve the urea by swirling and without the aid of heat, and adjust the resulting solution, if necessary, by adding 6 N hydrochloric acid to a pH of 3.2.

Chromatographic column: Insert a small pledget of glass wool above the stem constriction of a 20-cm × 2.5-cm chromatographic tube, and uniformly pack with a mixture of about 1 g of chromatographic siliceous earth and 0.5 mL of 5 M phosphoric acid. Directly above this layer, pack a similar mixture of about 3 g of chromatographic siliceous earth and 2 mL of *Ferric chloride–urea reagent*.

Standard stock solution: 150 µg/mL of salicylic acid in chloroform

Standard solution: 15 µg/mL of salicylic acid prepared as follows. Pipet 5 mL of the *Standard stock solution* into a 50-mL volumetric flask containing 10 mL of methanol, 0.1 mL of hydrochloric acid, and 10 mL of a solution of glacial acetic acid in ether (1 in 10). Dilute with chloroform to volume.

Sample solution: Nominally 0.5 mg/mL of aspirin prepared as follows. Transfer a portion of the cooled mass from the previously melted Suppositories obtained in the *Assay*, equivalent to 50 mg of aspirin, to a small beaker. Add 10 mL of chloroform, warm slightly, and stir until dissolved. With the aid of 5 mL of chloroform, transfer the mixture to the *Chromatographic column*. Pass 50 mL of chloroform in several portions through the column, rinse the tip of the chromatographic tube with chloroform, and discard the eluate. If the purple zone reaches the bottom of the tube, discard the column, and repeat the test with a smaller quantity of melted Suppositories. Elute the adsorbed salicylic acid into a 100-mL volumetric flask containing 20 mL of methanol and 0.2 mL of hydrochloric acid by passing two 10-mL portions of a solution of glacial acetic acid in water-saturated ether (1 in 10) and then 30 mL of chloroform through the column, and dilute the eluate with chloroform to volume.

Instrumental conditions

Mode: UV

Analytical wavelength: UV 306 nm

Cell: 1 cm

Blank: Dilute a mixture of 10 mL of methanol, 0.1 mL of hydrochloric acid, and 10 mL of a solution of glacial acetic acid in ether (1 in 10) with chloroform to 50 mL.

Analysis

Samples: *Standard solution* and *Sample solution*

Concomitantly determine the absorbances of the *Standard solution* and *Sample solution*.

Acceptance criteria: NMT 3.0%; the absorbance of the *Sample solution* does not exceed that of the *Standard solution*.

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in well-closed containers and in a cool place.

• **USP Reference Standards** ⟨11⟩

USP Aspirin RS

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

Topic/Question	Contact	Expert Committee
ASPIRIN SUPPOSITORIES	Documentary Standards Support	SM22020 Small Molecules 2
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM22020 Small Molecules 2

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
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