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Aspirin Boluses

DEFINITION

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

IDENTIFICATION

• **A. Procedure**

Analysis: Crush 1 Bolus. Boil a portion of the powder, equivalent to 300 mg of aspirin, with 50 mL of water. Cool and add a drop of ferric chloride TS.

Acceptance criteria: A violet-red color is produced.

• **B.** The retention time of the aspirin peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.

ASSAY

• **Procedure**

Mobile phase: 2 g/L of sodium 1-heptanesulfonate in a mixture of acetonitrile and water (15:85). Adjust with glacial acetic acid to a pH of 3.4.

Diluent: Acetonitrile and formic acid (99:1)

Standard solution: 0.4 mg/mL of USP Aspirin RS and 0.01 mg/mL of USP Salicylic Acid RS in *Diluent*

Sample stock solution: Nominally 4 mg/mL of aspirin prepared as follows. Finely powder NLT 10 Boluses. Transfer a portion of the powder to an appropriate volumetric flask and dilute with *Diluent* to volume. Stir the solution by mechanical means for 15 min.

Sample solution: Nominally 0.4 mg/mL of aspirin from *Sample stock solution* in *Diluent*. Pass a portion of this solution through a filter of 0.5-µm or finer pore size. Use the filtrate.

Chromatographic system

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

Mode: LC

Detector: UV 254 nm

Column: 4.6-mm × 25-cm; 5-µm packing L1

Flow rate: 1 mL/min

Injection volume: 20 µL

System suitability

Sample: *Standard solution*

[Note—The relative retention times for salicylic acid and aspirin are 0.6 and 1.0, respectively.]

Suitability requirements

Resolution: NLT 2.0 between salicylic acid and aspirin

Relative standard deviation: NMT 2.0% for aspirin

Analysis

Samples: *Standard solution* and *Sample solution*

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

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

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

r_S = peak response of aspirin from 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%

PERFORMANCE TESTS

• **Dissolution ⟨711⟩**

Medium: 0.5 M phosphate buffer (see *Reagents, Indicators, and Solutions—Buffer Solutions*), pH 7.4; 900 mL

Apparatus 2: 75 rpm

Time: 45 min

Diluent: Acetonitrile and formic acid (99:1)

Standard solution: USP Aspirin RS in *Diluent* at a suitable concentration. [Note—Prepare the solution at the time of use.]

Sample solution: Pass a portion of the solution under test through a suitable filter. Dilute with *Diluent*, if necessary.

Instrumental conditions

Mode: UV-Vis

Analytical wavelength: The isosbestic point of aspirin and salicylic acid at 265 ± 2 nm

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of aspirin ($C_9H_8O_4$) dissolved:

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

A_U = absorbance of the *Sample solution*

A_S = absorbance of the *Standard solution*

C_S = concentration of the *Standard solution* (mg/mL)

V = volume of *Medium*, 900 mL

D = dilution factor of the *Sample solution*, if necessary

L = label claim (mg/Bolus)

Tolerances: NLT 80% (Q) of the labeled amount of aspirin ($C_9H_8O_4$) is dissolved.

• **Uniformity of Dosage Units ⟨905⟩:** Meet the requirements

IMPURITIES

• **Limit of Salicylic Acid**

Mobile phase, Diluent, Sample solution, Chromatographic system, and **System suitability:** Proceed as directed in the *Assay*.

Salicylic acid standard solution: 0.01 mg/mL of USP Salicylic Acid RS in *Diluent*

Analysis

Samples: *Sample solution* and *Salicylic acid standard solution*

Calculate the actual concentration (C), in mg/mL, of aspirin ($C_9H_8O_4$) in the *Sample solution* taken:

$$\text{Result} = C_U \times (F/100)$$

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

F = percentage of the labeled amount of aspirin ($C_9H_8O_4$) in the portion of Boluses taken, as determined in the *Assay*

Calculate the percentage of salicylic acid ($C_7H_6O_3$) in the portion of Boluses taken:

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

r_U = peak response of salicylic acid from the *Sample solution*

r_S = peak response of salicylic acid from the *Salicylic acid standard solution*

C_S = concentration of USP Salicylic Acid RS in the *Salicylic acid standard solution* (mg/mL)

C = actual concentration of aspirin in the *Sample solution*

Acceptance criteria: NMT 0.3%

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight containers.

• **Labeling:** Label Boluses to indicate that they are for veterinary use only.

• **USP Reference Standards ⟨11⟩**

USP Aspirin RS

USP Salicylic Acid RS

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

Topic/Question	Contact	Expert Committee
ASPIRIN BOLUSES	Documentary Standards Support	SM32020 Small Molecules 3

Chromatographic Database Information: Chromatographic Database

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