Aspirin Delayed-Release Tablets

DEFINITION

Aspirin Delayed-Release Tablets contain NLT 95.0% and NMT 105.0% of the labeled amount of aspirin ($C_9H_8O_4$).

IDENTIFICATION

Change to read:

• **A.** •The retention time of the aspirin peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.● (IRA 1-Jan-2017)

• **B. INFRARED ABSORPTION ⟨197K⟩**

Sample: Shake a quantity of finely powdered Tablets, equivalent to about 500 mg of aspirin, with 10 mL of alcohol for several minutes. Centrifuge the mixture. Pour off the clear supernatant, and evaporate it to dryness. Dry the residue under vacuum at 60° for 1 h.

Acceptance criteria: Meet the requirements

ASSAY

Change to read:

• **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: 0.5 mg/mL of USP Aspirin RS in *Diluent*

Sample stock solution: •Nominally 5 mg/mL of aspirin prepared as follows.● (IRA 1-Jan-2017) Transfer a quantity equivalent to about 100 mg of aspirin from NLT 20 finely powdered Tablets to a suitable container. Add 20.0 mL of *Diluent* and 10 glass beads. Shake vigorously for 10 min, and centrifuge.

Sample solution: •Nominally 0.5 mg/mL of aspirin in *Diluent* from *Sample stock solution*.● (IRA 1-Jan-2017)

Chromatographic system

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

Mode: LC

Detector: UV 280 nm

Column: 4.0-mm × 30-cm; packing L1

Flow rate: 2 mL/min

Injection volume: 10 µL

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 aspirin ($C_9H_8O_4$) in the portion of Tablets 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: 95.0%–105.0%

PERFORMANCE TESTS

Change to read:

• **DISSOLUTION ⟨711⟩,** *Procedure, Apparatus 1 and Apparatus 2, Delayed-Release Dosage Forms, Method B Procedure*

Apparatus 1: 100 rpm

Times

Acid stage: 2 h

Buffer stage: 90 min

Diluent: 0.1 N hydrochloric acid and 0.20 M tribasic sodium phosphate (3:1). Adjust, if necessary, with 2 N hydrochloric acid or 2 N sodium hydroxide to a pH of 6.8 ± 0.05.

Standard solution: •USP Aspirin RS of a known concentration in 0.1 N hydrochloric acid (for analyzing the *Acid stage*) and in *Diluent* (for analyzing the *Buffer stage*)● (IRA 1-Jan-2017)

Sample solution: Pass a portion of the solution under test through a suitable filter, diluted, if necessary, with 0.1 N hydrochloric acid (for analyzing the *Acid stage*) and with *Diluent* (for analyzing the *Buffer stage*).

Instrumental conditions

Mode: UV

Analytical wavelengths

Acid stage: 280 nm

Buffer stage: 265 nm

Analysis

Samples: *Standard solution* and *Sample solution*

Determine the percentage of the labeled amount of aspirin ($C_9H_8O_4$) dissolved by determining UV absorbances at the isosbestic point of aspirin and salicylic acid (about 280 nm in the *Acid stage*, and about 265 nm in the *Buffer stage*).

Tolerances

Acid stage: NMT 10% (*Q*) of the labeled amount of aspirin ($C_9H_8O_4$) is dissolved.

Buffer stage: NLT 75% (*Q*) of the labeled amount of aspirin ($C_9H_8O_4$) is dissolved.

• **UNIFORMITY OF DOSAGE UNITS ⟨905⟩:** Meet the requirements

IMPURITIES

Change to read:

• **LIMIT OF FREE SALICYLIC ACID**

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

•**System suitability solution:** 0.015 mg/mL of USP Salicylic Acid RS and 0.5 mg/mL of USP Aspirin RS in *Diluent*● (IRA 1-Jan-2017)

Standard solution: 0.015 mg/mL of USP Salicylic Acid RS •● (IRA 1-Jan-2017) in *Diluent*

Sample solution: Use the *Sample stock solution* from the *Assay*.

System suitability

Samples: •*System suitability solution*● (IRA 1-Jan-2017) and *Standard solution*

[NOTE—The relative retention times for salicylic acid and aspirin are about 0.7 and 1.0, respectively.]

Suitability requirements

Resolution: NLT 2.0 between salicylic acid and aspirin, •*System suitability solution*● (IRA 1-Jan-2017)

Relative standard deviation: NMT 4.0%, *Standard solution*

Analysis
Samples: *Standard solution* and *Sample solution*
Calculate the percentage of salicylic acid ($C_7H_6O_3$) in the portion of Tablets taken:

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

r_U = peak response of salicylic acid from the *Sample solution*
r_S = peak response of salicylic acid from the *Standard solution*
C_S = concentration of USP Salicylic Acid RS in the *Standard solution* (mg/mL)
C_U = nominal concentration of aspirin in the *Sample solution* (mg/mL)

Acceptance criteria: NMT 3.0%

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in tight containers.
- **USP Reference Standards** ⟨**11**⟩
  USP Aspirin RS
  USP Salicylic Acid RS