Aspirin Delayed-Release Capsules

DEFINITION

Aspirin Delayed-Release Capsules contain NLT 93.0% and NMT 107.0% 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 the contents of Capsules, 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) Remove, as completely as possible, the contents of NLT 20 Capsules. Mix the combined contents, and transfer a quantity equivalent to about 100 mg of aspirin to a suitable container. Add 20.0 mL of *Diluent* and about 10 glass beads. Shake vigorously for about 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 Capsules 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: 93.0%–107.0%

PERFORMANCE TESTS

Change to read:

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

Apparatus 1: 100 rpm

Time: 90 min, for *Buffer stage*; •2 h, for *Acid stage*• (IRA 1-Jan-2017)

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: A known concentration of USP Aspirin RS in a suitable medium

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

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 from 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:** The percentages of the labeled amount of aspirin ($C_9H_8O_4$) dissolved conform to *Dissolution* ⟨711⟩, *Acceptance Table 3* (*Acid stage*) and *Acceptance Table 4* (*Buffer stage*).• (IRA 1-Jan-2017)

• **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 in *Diluent*• (IRA 1-Jan-2017)

Sample solution: Use the *Sample stock solution* prepared as directed in 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 Capsules 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.
- **LABELING:** The label indicates that the Capsules or the contents thereof are enteric-coated.

- **USP REFERENCE STANDARDS** ⟨**11**⟩
  USP Aspirin RS
  USP Salicylic Acid RS