Buffered Aspirin Tablets

DEFINITION

Buffered Aspirin Tablets contain Aspirin and suitable buffering agents. Tablets contain NLT 90.0% and NMT 110.0% 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 chloroform for several min. Centrifuge the mixture. Pour off the clear supernatant, and evaporate it to dryness.

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: 90.0%–110.0%

PERFORMANCE TESTS

• **DISSOLUTION** ⟨711⟩

Medium: 0.05 M acetate buffer, prepared by mixing 2.99 g of sodium acetate trihydrate and 1.66 mL of glacial acetic acid with water to obtain a total of 1000 mL of solution with a pH of 4.50 ± 0.05; 500 mL

Apparatus 2: 75 rpm. [NOTE—Where the Tablet is composed of multiple layers, a stainless steel wire helix may be used, if needed, to hold the Tablet in proper orientation in the apparatus.]

Time: 30 min

Standard solution: A known concentration of USP Aspirin RS in *Medium*. Prepare the *Standard solution* at the time of use. [NOTE—A quantity of methanol not to exceed 1% of the total volume of the *Standard solution* may be used to dissolve the Reference Standard prior to dilution with *Medium*.]

Sample solution: Pass a portion of the solution under test through a suitable filter, and dilute with *Medium*, if necessary.

Instrumental conditions

Mode: UV

Analytical wavelength: 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 at about 265 nm.

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

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 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%

SPECIFIC TESTS

- **ACID-NEUTRALIZING CAPACITY ⟨301⟩:** NLT 1.9 mEq of acid is consumed for each 325 mg of aspirin in the Tablets.

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in tight containers.
- **USP REFERENCE STANDARDS ⟨11⟩**
  USP Aspirin RS
  USP Salicylic Acid RS