isosbestic point of aspirin and salicylic acid at about 265 nm.

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

• **UNIFORMITY OF DOSAGE UNITS** ⟨905⟩, *Weight Variation* and *Content Uniformity*: Meet the requirements for weight variation with respect to aluminum hydroxide and to magnesium hydroxide. Meet the requirements for content uniformity with respect to aspirin.

IMPURITIES

• **LIMIT OF FREE SALICYLIC ACID**

Mobile phase, Diluent, Internal standard solution, Salicylic acid stock solution, Sample solution, and Chromatographic system: Proceed as directed in the *Assay* for *Aspirin*.

System suitability solution: Transfer about 325 mg of USP Aspirin RS to a 50-mL volumetric flask. Add 10.0 mL of *Salicylic acid stock solution* and 5.0 mL of *Internal standard solution*, dilute with *Diluent* to volume, and mix.

Standard solution: 0.2 mg/mL of USP Salicylic Acid RS prepared as follows. Transfer 10.0 mL of *Salicylic acid stock solution* and 5.0 mL of *Internal standard solution* to a 50-mL volumetric flask, dilute with *Diluent* to volume, and mix.

System suitability

Samples: *System suitability solution* and *Standard solution*

[NOTE—The relative retention times for salicylic acid, aspirin, and phenacetin are about 0.3, 0.6, and 1.0, respectively. Record each chromatogram until the chloroform peak appears at the relative retention time of about 1.8.]

Suitability requirements

Resolution: NLT 2.0 between two adjacent peaks for salicylic acid, aspirin, and phenacetin, *System suitability solution*

Tailing factor: NMT 2.0, *System suitability solution*

Relative standard deviation: NMT 3.0%, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of salicylic acid in the portion of Tablets taken:

$$\text{Result} = (R_U/R_S) \times (C_S/C_U) \times 100$$

R_U = peak response ratio of salicylic acid to phenacetin from the *Sample solution*

R_S = peak response ratio of salicylic acid to phenacetin 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

Aspirin, Alumina, and Magnesium Oxide Tablets

DEFINITION

Aspirin, Alumina, and Magnesium Oxide Tablets contain NLT 90.0% and NMT 110.0% of the labeled amount of aspirin ($C_9H_8O_4$), the equivalent of NLT 90.0% and NMT 110.0% of the labeled amount of aluminum hydroxide [$Al(OH)_3$], and NLT 90.0% and NMT 110.0% of the labeled amount of magnesium oxide (MgO).

IDENTIFICATION

Sample: The *Sample* is prepared as follows. To a 0.7-g portion of finely powdered Tablets, add 20 mL of 3 N hydrochloric acid and 5 drops of methyl red TS, heat to boiling, and add 6 N ammonium hydroxide until the color of the solution changes to deep yellow. Continue boiling for 2 min, and filter. The filtrate is used in *Identification* test *B*, and the precipitate is used in *Identification* test *C*.

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

• **B. IDENTIFICATION TESTS—GENERAL,** *Magnesium* ⟨191⟩

Sample solution: *Sample* filtrate

Acceptance criteria: Meet the requirements

• **C. IDENTIFICATION TESTS—GENERAL,** *Aluminum* ⟨191⟩

Sample solution: Wash the *Sample* precipitate with a hot solution of 20 mg/mL of ammonium chloride, and dissolve the precipitate in hydrochloric acid.

Acceptance criteria: Meet the requirements

• **D. PROCEDURE**

Analysis: Where the Tablets are composed of two layers, scrape a small amount of each layer into separate test tubes. Add 2 mL of water and 2 drops of methyl red TS to each tube, and shake for 15 s.

Acceptance criteria: The solution from the aspirin-containing layer is red, and the solution from the buffer-containing layer is yellow.

ASSAY

• **ASPIRIN**

Mobile phase: Methanol, phosphoric acid, and water (30:3:70)

Diluent: Dehydrated alcohol and hydrochloric acid (2000:20)

Aspirin standard stock solution: 5 mg/mL of USP Aspirin RS in *Diluent* prepared by blending at high speed for 1.5 min

Aspirin standard solution: 0.25 mg/mL of USP Aspirin RS prepared immediately from the *Aspirin standard stock solution* in dehydrated alcohol. Use these solutions within 1 h.

Salicylic acid standard stock solution: 5 mg/mL of USP Salicylic Acid RS in dehydrated alcohol. Transfer 3.0 mL of this solution to a 100-mL volumetric flask, and dilute with *Diluent* to volume.

Salicylic acid standard solution: 7.5 µg/mL of USP Salicyclic Acid RS from the *Salicylic acid standard stock solution* in dehydrated alcohol

System suitability solution: Transfer 5.0 mL of the *Aspirin standard stock solution* to a 100-mL volumetric flask, add 5.0 mL of the *Salicylic acid standard stock solution*, and dilute with dehydrated alcohol to volume.

Sample solution: Transfer a counted number of Tablets, equivalent to 2500 mg of aspirin, to a 120-mL blender