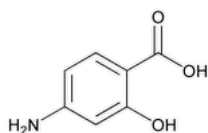


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## Aminosalicilic Acid



$C_7H_7NO_3$  153.14

Benzoic acid, 4-amino-2-hydroxy-

4-Aminosalicilic acid CAS RN®: 65-49-6; UNII: 5B2658E0N2.

### DEFINITION

Aminosalicilic Acid contains NLT 98.5% and NMT 100.5% of aminosalicilic acid ( $C_7H_7NO_3$ ), calculated on the anhydrous basis.

[CAUTION—Under no circumstances use a solution prepared from Aminosalicilic Acid if its color is darker than that of a freshly prepared solution.]

### IDENTIFICATION

#### • A.

**Sample stock solution:** Dissolve 250 mg in 3 mL of 1 N sodium hydroxide, transfer to a 500-mL volumetric flask, and dilute with water to volume.

**Sample solution:** Transfer a 5-mL aliquot of the *Sample stock solution* to a 250-mL volumetric flask containing 12.5 mL of pH 7 phosphate buffer (see *Reagents, Indicators, and Solutions—Buffer Solutions*), and dilute with water to volume.

**Analysis:** Compare the *Sample solution* in a suitable spectrometer against a blank of the same buffer in the same concentration.

**Acceptance criteria:** The *Sample solution* exhibits absorbance maxima at  $265 \pm 2$  and  $299 \pm 2$  nm, and the ratio  $A_{265}/A_{299}$  is 1.50–1.56.

#### • B.

**Sample:** 1 g

**Analysis:** Place the *Sample* in a small, round-bottom flask, and add 10 mL of acetic anhydride. Heat the flask on a steam bath for 30 min, add 40 mL of water, filter, cool, and allow to stand until the diacetyl derivative has crystallized. Collect the precipitate on a filter, wash well with water, and dry at  $105^\circ$  for 1 h.

**Acceptance criteria:** The diacetyl derivative melts at  $191^\circ$ – $197^\circ$ .

#### • C.

**Sample:** 0.1 g

**Analysis:** Shake the *Sample* with 10 mL of water, and filter. To 5 mL of the filtrate add 1 drop of ferric chloride TS.

**Acceptance criteria:** A violet color is produced.

### ASSAY

#### • PROCEDURE

**Solution A:** 12.7 mg/mL of tetrabutylammonium hydroxide in methanol

**Mobile phase:** *Solution A*, 0.05 M dibasic sodium phosphate, and 0.05 M monobasic sodium phosphate (150:425:425)

**Internal standard solution:** 5 mg/mL of acetaminophen in *Mobile phase*

**Standard solution:** 0.5 mg/mL of aminosalicilic acid prepared as follows. Transfer 12.5 mg of [USP Aminosalicilic Acid RS](#) to a 25-mL low-actinic volumetric flask, add 15 mL of *Mobile phase*, and swirl to dissolve. Add 2.5 mL of the *Internal standard solution*, and dilute with *Mobile phase* to volume.

**Sample solution:** 0.5 mg/mL of Aminosalicilic Acid prepared as follows. Transfer 12.5 mg of Aminosalicilic Acid to a 25-mL low-actinic volumetric flask, add 15 mL of *Mobile phase*, and swirl to dissolve. Add 2.5 mL of the *Internal standard solution*, and dilute with *Mobile phase* to volume.

#### Chromatographic system

(See [Chromatography \(621\), System Suitability](#).)

**Mode:** LC

**Detector:** UV 254 nm

**Column:** 4.6-mm  $\times$  25-cm; packing L1

**Flow rate:** 1.5 mL/min

**Injection volume:** 20  $\mu$ L

**System suitability****Sample:** *Standard solution*

[NOTE—The relative retention times for acetaminophen and aminosalicic acid are 0.83 and 1.0, respectively.]

**Suitability requirements****Resolution:** NLT 1.7 between aminosalicic acid and acetaminophen**Relative standard deviation:** NMT 1.0% for the peak response ratio of aminosalicic acid to acetaminophen**Analysis****Samples:** *Standard solution* and *Sample solution*

After use, wash the column for 30 min with a mixture of methanol, water, and phosphoric acid (77:23:0.6), and then wash for 30 min with a mixture of methanol and water (50:50).

Calculate the percentage of aminosalicic acid ( $C_7H_7NO_3$ ) in the portion of Aminosalicic Acid taken:

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

 $R_U$  = peak response ratio of aminosalicic acid to acetaminophen from the *Sample solution* $R_S$  = peak response ratio of aminosalicic acid to acetaminophen from the *Standard solution* $C_S$  = concentration of [USP Aminosalicic Acid RS](#) in the *Standard solution* (mg/mL) $C_U$  = concentration of Aminosalicic Acid in the *Sample solution* (mg/mL)**Acceptance criteria:** 98.5%–100.5% on the anhydrous basis**IMPURITIES**• **RESIDUE ON IGNITION (281):** NMT 0.2%• **CHLORIDE AND SULFATE, Chloride (221):****Sample solution:** 25 mg/mL in a mixture of nitric acid and water (5:15)**Acceptance criteria:** NMT 0.042%; the solution shows no more chloride than corresponds to 0.30 mL of 0.020 N hydrochloric acid.• **LIMIT OF *m*-AMINOPHENOL****Solution A:** 12.7 mg/mL of tetrabutylammonium hydroxide in methanol**Mobile phase:** *Solution A*, 0.05 M dibasic sodium phosphate, and 0.05 M monobasic sodium phosphate (150:425:425)**Internal standard solution:** 5 µg/mL of sulfanilamide in *Mobile phase***Standard stock solution:** 12 µg/mL of [USP \*m\*-Aminophenol RS](#) in *Mobile phase***Standard solution:** 1.2 µg/mL of [USP \*m\*-Aminophenol RS](#) prepared as follows. Transfer 10.0 mL of the *Standard stock solution* and 10.0 mL of the *Internal standard solution* to a 100-mL low-actinic volumetric flask, and dilute with *Mobile phase* to volume.**Sample solution:** 0.5 mg/mL of Aminosalicic Acid prepared as follows. Transfer 50 mg of Aminosalicic Acid to a 100-mL low-actinic volumetric flask, add 50 mL of *Mobile phase*, and swirl to dissolve. Add 10.0 mL of the *Internal standard solution*, and dilute with *Mobile phase* to volume.**Chromatographic system**(See [Chromatography \(621\), System Suitability](#).)**Mode:** LC**Detector:** UV 280 nm**Column:** 4.6-mm × 25-cm; 10-µm packing L1**Flow rate:** 1.5 mL/min**Injection volume:** 20 µL**System suitability****Sample:** *Standard solution*[NOTE—The relative retention times for sulfanilamide and *m*-aminophenol are about 0.66 and 1.0, respectively.]**Suitability requirements****Resolution:** NLT 2.5 between *m*-aminophenol and sulfanilamide**Relative standard deviation:** NMT 7%**Analysis****Samples:** *Standard solution* and *Sample solution*

After use, wash the column for 30 min with a mixture of methanol, water, and phosphoric acid (77:23:0.6), and then wash for 30 min with a mixture of methanol and water (50:50).

Calculate the percentage of *m*-aminophenol in the portion of Aminosalicic Acid taken:

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

 $R_U$  = peak response ratio of *m*-aminophenol to sulfanilamide from the *Sample solution* $R_S$  = peak response ratio of *m*-aminophenol to sulfanilamide from the *Standard solution*