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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 Salicylic 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 jar containing 100.0 mL of *Diluent*, and blend at high speed for 1.5 min. Immediately filter a portion of the mixture thus obtained, and transfer 1.0 mL of the filtrate to a 100-mL volumetric flask. Immediately dilute with dehydrated alcohol to volume. Promptly inject this *Sample solution* into the chromatograph as directed for *Analysis*.

Chromatographic system

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

Mode: LC

Detector: UV 205 nm

Column: 4.6-mm × 3-cm; 5-µm packing L7

Flow rate: 3.5 mL/min

Injection volume: 10 µL

System suitability

Samples: *Aspirin standard solution*, *Salicylic acid standard solution*, and *System suitability solution*

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

Suitability requirements

Resolution: NLT 2.0 between the aspirin peak and the salicylic acid peak, *System suitability solution*



Tailing factor: NMT 2.0 for the aspirin and salicylic acid peaks, *Aspirin standard solution* and *Salicylic acid standard solution*

Relative standard deviation: NMT 2.0% for the aspirin and salicylic acid peaks, *Aspirin standard solution* and *Salicylic acid standard solution*

Analysis

Samples: *Aspirin standard solution*, *Salicylic acid standard solution*, and *Sample solution*

Calculate the percentage of aspirin ($C_9H_8O_4$) in each Tablet 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 *Aspirin standard solution* (mg/mL)

C_U = nominal concentration of aspirin in the *Sample solution* (mg/mL)

Acceptance criteria: 90.0%–110.0%

• ALUMINUM HYDROXIDE

Edetate disodium titrant: Dissolve 18.6 g of edetate disodium in water to make 1000 mL, and standardize the solution as follows. Weigh 2 g of aluminum wire, transfer to a 1000-mL volumetric flask, and add 50 mL of a mixture of hydrochloric acid and water (1:1). Swirl the flask to ensure contact of the aluminum and the acid, and allow the reaction to proceed until all of the aluminum has dissolved. Dilute with water to volume. Pipet 10 mL of this solution into a 250-mL beaker; add, in the order named and with continuous stirring, 25.0 mL of edetate disodium titrant and 20 mL of acetic acid–ammonium acetate buffer TS; and boil gently for 5 min. Cool, and add 50 mL of alcohol and 2 mL of dithizone TS. Titrate with 0.05 M zinc sulfate VS to a bright rose-pink color. Perform a blank determination, substituting 10 mL of water for the aluminum solution, and make any necessary correction.

Calculate the molarity of the solution taken:

$$\text{Result} = (W/A_r) \times V$$

W = weight of aluminum in the portion of the solution taken (mg)

A_r = atomic weight of aluminum, 26.98

V = volume of *Edetate disodium titrant* consumed (mL)

Sample solution: To a portion of the powdered Tablets (NLT 20) equivalent to 600 mg of aluminum hydroxide, add 20 mL of water, stir, and slowly add 30 mL of 3 N hydrochloric acid. Heat gently, if necessary, to aid solution, cool, and transfer to a 200-mL volumetric flask. Wash the beaker with water, adding the washings to the flask, and add water to volume.

Analysis: To 20 mL of the *Sample solution* in a 250 mL-beaker, add 20 mL of water; then add, in the order named and with continuous stirring, 25.0 mL of *Edetate disodium titrant* and 20 mL of acetic acid–ammonium acetate buffer TS, and heat the solution near the boiling temperature for 5 min. Cool, and add 50 mL of alcohol and 2 mL of dithizone TS. Titrate with 0.05 M zinc sulfate VS until the color changes from green-violet to rose-pink. Perform a blank determination, substituting 10 mL of water for the *Sample solution*, and make any necessary correction. Each mL of 0.05 M *Edetate disodium titrant* is equivalent to 3.900 mg of aluminum hydroxide $[Al(OH)_3]$.

Acceptance criteria: 90.0%–110.0%

• MAGNESIUM OXIDE

Sample solution: Prepare as directed in the Assay for *Aluminum Hydroxide*.

Eriochrome black indicator solution: Dissolve 200 mg of eriochrome black T in a mixture of 15 mL of triethanolamine and 5 mL of dehydrated alcohol.

Titrant: 0.05 M edetate disodium VS

Analysis: To a volume of the *Sample solution* equivalent to 40 mg of magnesium oxide in a 400 mL beaker, add, while mixing, 20 mL of triethanolamine and 200 mL of water. Cool the solution for 10 min, while stirring, by immersion in an ice bath. Remove the beaker from the ice bath, and add 15 mL of ammonia–ammonium chloride buffer TS and 2 drops of *Eriochrome black indicator solution*. Titrate with *Titrant* to a blue endpoint, allowing 60 s between drops of *Titrant* as the endpoint is approached (after first color change is observed). The titration should be completed within 10 min after the addition of the buffer and indicator. If any precipitate is observed prior to titration, the solution should be discarded, and a new solution prepared. Perform a blank determination, substituting an equivalent volume of water for the volume of *Sample solution* used, and make any necessary correction. Each mL of *Titrant* consumed is equivalent to 2.015 mg of magnesium oxide (MgO).

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 1000 mL of solution having a pH of 4.50 ± 0.05 ; 900 mL

Apparatus 1 (10-mesh screen): 100 rpm