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Bismuth Subsalicylate Magma

DEFINITION

Bismuth Subsalicylate Magma is a suspension of Bismuth Subsalicylate in water that contains NLT 90.0% and NMT 110.0% of the labeled amount of bismuth subsalicylate ($C_7H_5BiO_4$). Bismuth subsalicylate is a basic salt that when dried at 105° for 3 h contains NLT 56.0% and NMT 59.4% bismuth (Bi) and NLT 36.5% and NMT 39.3% of total salicylates.

Dry at 105° for 3 h to determine the solids content and, after determining the solids content, perform all tests on a portion of the dried Magma.

IDENTIFICATION

Change to read:

• **A.** ▲**Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*: 197M**▲ (CN 1-May-2020)

• **B.** **Identification Tests—General, *Bismuth*⟨191⟩:** Meets the requirements

ASSAY

• **Bismuth**

Sample solution: Transfer an equivalent to 300 mg of bismuth subsalicylate, previously dried at 105° for 3 h, to a porcelain crucible, and ignite. Allow it to cool, and add about 2 mL of nitric acid to the residue, dropwise, warming until dissolved. Add about 60 mL of water and 0.3 mL of xylenol orange TS.

Titrimetric system

Mode: Direct titration

Titrant: 0.05 M edetate disodium VS

Endpoint detection: Visual

Analysis: Titrate the *Sample solution* with *Titrant* to a yellow endpoint. Each mL of *Titrant* is equivalent to 10.45 mg of bismuth (Bi).

Acceptance criteria: 56.0%–59.4% of bismuth on the previously dried basis

• **Total Salicylates**

Solution A: Ferric ammonium sulfate TS, 1 N hydrochloric acid, and water (4:1:15)

Standard stock solution: 0.2 mg/mL of USP Salicylic Acid RS in water

Standard solution: 0.05 mg/mL of USP Salicylic Acid RS in water, prepared by adding 25.0 mL of *Standard stock solution* and 70 mL of water to a 100-mL volumetric flask. Adjust with 0.5 N sodium hydroxide or 1 N hydrochloric acid to a pH of 4.5, before dilution with water to volume.

Reacted standard solution: To 25.0 mL of *Standard solution* add 1.0 mL of *Solution A*.

Unreacted standard solution: To 25.0 mL of the *Standard solution* add 1.0 mL of 0.05 N hydrochloric acid.

Sample solution: Transfer an equivalent to 52 mg of bismuth subsalicylate from previously dried Magma at 105° for 3 h to a 200-mL volumetric flask. Add 10 mL of 0.5 N sodium hydroxide, heat on a steam bath for 15 min, allow to cool, and dilute with water to volume. Centrifuge 70 mL, and then transfer 50.0 mL of the clear supernatant to a beaker. Add about 40 mL of water, and adjust with 0.5 N sodium hydroxide or 1 N hydrochloric acid to a pH of 4.5. Transfer this solution to a 100-mL volumetric flask with the aid of water, and dilute with water to volume.

Reacted sample solution: To 25.0 mL of *Sample solution* add 1.0 mL of *Solution A*.

Unreacted sample solution: To 25.0 mL of the *Sample solution* add 1.0 mL of 0.05 N hydrochloric acid.

Blank: Water, adjusted with 0.5 N sodium hydroxide or 1 N hydrochloric acid to a pH of 4.5

Reacted blank solution: To 25.0 mL of *Blank* add 1.0 mL of *Solution A*.

Unreacted blank: To 25.0 mL of *Blank* add 1.0 mL of 0.05 N hydrochloric acid.

Instrumental conditions

Mode: UV

Analytical wavelength: 525 nm

Analysis

Samples: *Reacted standard solution*, *Unreacted standard solution*, *Reacted sample solution*, *Unreacted sample solution*, *Reacted blank solution*, and *Unreacted blank*

Concomitantly determine the absorbances of the *Samples*.

Calculate the percentage of total salicylates in the portion of dried Magma taken:

$$\text{Result} = [(A_{UR} - A_{UU} - B)/(A_{SR} - A_{SU} - B)] \times (C_S/C_U) \times 100$$

A_{UR} = absorbance of the *Reacted sample solution*

A_{UU} = absorbance of the *Unreacted sample solution*

B = difference in the absorption of the *Reacted blank solution* and the absorption of the *Unreacted blank*

A_{SR} = absorbance of the *Reacted standard solution*

A_{SU} = absorbance of the *Unreacted standard solution*

C_S = concentration of USP Salicylic Acid RS in the *Standard solution* (mg/mL)

C_U = concentration of bismuth subsalicylate in the *Sample solution* (mg/mL)

Acceptance criteria: 36.5%–39.3% of total salicylates on the previously dried basis

IMPURITIES

- **Limit of Copper, Lead, and Silver**

Standard stock solution: Add 3.0 mL each of 1000-µg/mL solutions of copper, lead, and silver, respectively, to a 2000-mL flask, and dilute with 1 M nitric acid to volume.

Standard solution: 1.5 µg/mL of copper, 1.5 µg/mL of lead, and 1.5 µg/mL of silver, in 1 M nitric acid from the *Standard stock solution*. The concentrations of copper, lead, and silver may be modified by using different volumes or concentrations to bring the absorption response within the working range of the atomic absorption spectrophotometer.

Sample solution: Ignite 3 g of sample in a porcelain crucible, cool, cautiously add 6 M nitric acid to dissolve the residue, and evaporate on a steam bath. Ignite the residue, cool, transfer the residue to a tared conical flask, and wash the flask with about 5 mL of 6 M nitric acid, adding the wash to the conical flask. Dissolve the residue with the aid of heat, and add water to obtain a solution weighing 20.0 g. The concentrate of bismuth subsalicyclate may be modified by using the same proportions used for modifying the *Standard solution*, by using a different quantity, or by further dilution.

Instrumental conditions

(See *Atomic Absorption Spectroscopy* ⟨852⟩.)

Mode: Atomic absorption spectrophotometry

Analytical wavelength: 324.7 nm for copper; 217 nm for lead; 328.1 nm for silver

Lamps: Copper, lead and silver hollow-cathode, and oxidizing flames

Analysis

Samples: *Standard solution* and *Sample solution*

Concomitantly determine the absorbances of the *Standard solution* and the *Sample solution*

Acceptance criteria: 10 ppm; the absorbances of the *Sample solutions* do not exceed those of the *Standard solutions* for each element.

- **Limit of Soluble Bismuth**

Standard solution: 2 µg/mL of bismuth (Bi), prepared as follows. Add 242.0 mg of bismuth nitrate pentahydrate to a 100-mL volumetric flask, add 3 mL of 1.5 M nitric acid, swirl to dissolve, and dilute with water to volume. Add 1.0 mL of this solution to a 500-mL volumetric flask, add 250 mL of 1.5 M nitric acid, and dilute with water to volume. The concentration of bismuth in this solution may be modified by using a lesser dilution or by further dilution to bring the absorption response within the working range of the atomic absorption spectrophotometer.

Sample solution: 5.0 g of bismuth subsalicylate from dried Magma in 100 mL of water, and stir the suspension thus obtained for 2 h at 20°–23°. Pass through filter paper. Pass the filtrate thus obtained through a filter of 0.1-µm or less pore size. Add 0.1 mL of nitric acid to 10.0 mL of the filtrate. The concentrate of bismuth subsalicyclate may be modified by using the same proportions used for modifying the *Standard solution*, by using a different quantity, or by further dilution.

Instrumental conditions

(See *Atomic Absorption Spectroscopy* ⟨852⟩.)

Mode: Atomic absorption spectrophotometry

Analytical wavelength: 223.06 nm for bismuth

Lamp: Bismuth hollow-cathode and an oxidizing flame

Analysis

Samples: *Standard solution* and *Sample solution*

Concomitantly determine the absorbances of the *Standard solution* and the *Sample solution*.

Acceptance criteria: 40 ppm; the absorbances of the *Sample solution* do not exceed those of the *Standard solution*.

- **Limit of Nitrate**

Standard solution: To 0.1 g of salicylic acid add 6 mL of water, 4.0 mL of a solution containing 100 µg of nitrate per mL, and 20 mL of sulfuric acid. Prepare concomitantly with the *Sample solution*.

Sample solution: Add 10 mL of water to 0.1 g of Magma. Carefully add 20 mL of sulfuric acid, and mix.

Acceptance criteria: 0.4%; the *Sample solution* should not be more yellow than the *Standard solution*.

- **Limit of Free Salicylic Acid**

Mobile phase: Methanol and 0.06 M acetic acid (550:450)

Diluent: Acetonitrile and water (1:1)

Standard solution: 0.02 mg/mL of USP Salicylic Acid RS in *Diluent*

Sample solution: Add 260 mg of bismuth subsalicylate from dried Magma to a glass centrifuge tube, add about 12 mL of acetonitrile, shake by mechanical means for 20 min, and centrifuge. Decant the supernatant into a suitable container. Repeat the acetonitrile addition, shaking, centrifuging, and decanting, combining the decanted liquid with the first decantate. Pass the combined liquid through a filter of 0.5-µm pore size, collecting the filtrate in a 50-mL volumetric flask. Wash the container with 5 mL of acetonitrile, and filter the wash, collecting the filtrate in the volumetric flask. Dilute with water to volume.

Chromatographic system

(See *Chromatography ⟨621⟩, System Suitability*.)

Mode: LC

Detector: UV 300 nm

Columns

Guard: 3.2-mm × 1.5-cm; 5-µm packing L1

Analytical: 4.6-mm × 30-cm; 5-µm packing L1

Flow rate: 1 mL/min

Injection volume: 20 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 free salicylic acid in the portion of Magma taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak area of salicylic acid from the *Sample solution*

r_S = peak area of salicylic acid from of the *Standard solution*

C_S = concentration of USP Salicylic Acid RS in the *Standard solution* (mg/mL)

C_U = concentration of the bismuth subsalicylate in the *Sample solution* (mg/mL)

Acceptance criteria: NMT 0.2%

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in tight, light-resistant containers.
- **Labeling:** The label states that this article is not intended for direct administration to humans or animals.
- **USP Reference Standards ⟨11⟩**

USP Bismuth Subsalicylate RS

USP Salicylic Acid RS

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
BISMUTH SUBSALICYLATE MAGMA	Documentary Standards Support	SM22020 Small Molecules 2
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM22020 Small Molecules 2

Chromatographic Database Information: Chromatographic Database

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