tion, transfer to a graduated vessel, add sufficient Purified Water to make 1000 mL, and mix. NOTE—This method of preparation may be varied, provided the product meets the following requirements.

Packaging and storage—Preserve in tight containers, and protect from freezing.

Identification—

A: It responds to the tests for *Bismuth* 〈191〉 and for *Carbonate* 〈191〉.

B: Add 1 mL of 3 N hydrochloric acid to 1 mL of Milk of Bismuth: a clear solution is produced. Pour the clear solution into 10 volumes of water: a white precipitate is formed.

Microbial enumeration tests 〈61〉 **and Tests for specified microorganisms** 〈62〉—The total bacterial count does not exceed 100 cfu per mL and the test for *Escherichia coli* is negative.

Water-soluble substances—Boil 10 mL with 90 mL of water for 10 minutes, cool, add water to make the total volume 100 mL, mix, and filter. Evaporate 50 mL of the filtrate to dryness, and ignite it gently: the weight of the residue does not exceed 5 mg (0.1%).

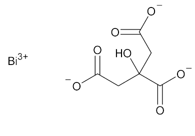
Arsenic, *Method I* 〈211〉—Evaporate 3.75 mL on a steam bath to dryness, add 2 mL of sulfuric acid, and heat until copious fumes of sulfur trioxide are evolved. The limit is 0.8 ppm.

Lead—To 5 mL add warm nitric acid, dropwise, until it is just dissolved, and pour the solution into 50 mL of water: a white precipitate may form. Filter, if necessary, evaporate the filtrate on a steam bath to 15 mL, again filter, and to 10 mL of the filtrate add an equal volume of 2 N sulfuric acid: no precipitate is formed.

Limit of alkalies and alkaline earths—Dissolve 2.0 mL in 5 mL of hydrochloric acid, dilute with water to 100 mL, add hydrogen sulfide to precipitate the bismuth completely, and filter. To 50 mL of the clear filtrate add 5 drops of sulfuric acid, evaporate to dryness, and ignite: the weight of the residue does not exceed 3 mg (0.3%).

Assay—Evaporate an accurately weighed quantity of Milk of Bismuth to dryness, and ignite the residue to constant weight. From the weight of the Bi_2O_3 so obtained determine the percentage in the assay specimen.

Bismuth Citrate



$BiC_6H_5O_7$ 398.08 [813-93-4].

» Bismuth Citrate contains not less than 49 per cent and not more than 54 per cent of bismuth (Bi).

Packaging and storage—Preserve in tight, light-resistant containers, store at controlled room temperature, and prevent exposure to excessive heat.

USP Reference standards 〈11〉—
USP Bismuth Citrate RS

Identification—

A: *Infrared Absorption* 〈197K〉: on the undried specimen.

B: When strongly heated, the salt chars, and on ignition leaves a more or less blackened residue having a yellow surface. The residue is soluble in warm nitric acid, and this solution, when dropped into a large excess of water, produces a white turbidity.

C: Dissolve 1 g in ammonia TS. When treated with hydrogen sulfide in excess, a black precipitate is obtained. Filter this mixture, drive off the excess hydrogen sulfide by heating, and allow to cool. To a portion of this cooled solution add an excess of calcium hydroxide TS, and boil: a white precipitate is formed. Reserve a second portion of the cooled solution for the test for *Limit of nitrate*.

Arsenic, *Method I* 〈211〉—Prepare the *Test Preparation* as follows. Triturate 300 mg with an equal weight of calcium hydroxide, and ignite. Dissolve the residue in 5 mL of 3 N hydrochloric acid: the limit is 10 µg per g.

Limit of nitrate—To the second portion of cooled solution reserved from *Identification* test *C*, add an equal volume of sulfuric acid, mix, and allow to cool. Into the liquid, drop a crystal of ferrous sulfate, and allow to stand for 30 minutes: no brown or brownish black color appears around the crystal.

Limit of copper, lead, and silver—

Standard solution—Prepare a solution containing 1000 µg of copper per mL, a solution containing 1000 µg of lead per mL, and a solution containing 1000 µg of silver per mL. Transfer 3.0 mL of each solution to a 2000-mL volumetric flask, dilute with 1 N nitric acid to volume, and mix. [NOTE—The concentrations of copper, lead, and silver in this solution may be modified by using a different quantity or by further dilution to bring the absorption responses within the working range of the atomic absorption spectrophotometer.]

Test solution—Ignite about 3 g of Bismuth Citrate, accurately weighed, in a porcelain crucible, cool, and cautiously add 6 N nitric acid to dissolve the residue. Add 100 mL of water, and mix. A white precipitate forms. Filter this mixture, evaporate on a steam bath to obtain about 15 mL of solution, and filter again. Dilute the filtrate with water to 20.0 mL.

Procedure—Concomitantly determine the absorbances of the *Standard solution* and the *Test solution* at the emission lines of 324.7 nm, 217 nm, and 328.1 nm for copper, lead, and silver, respectively, with an atomic absorption spectrophotometer (see *Spectrophotometry and Light-Scattering* 〈851〉) equipped with copper, lead, and silver hollow-cathode lamps and an oxidizing flame. The absorbances of the *Test solution* do not exceed those of the *Standard solution* for each element (10 µg per g).

Limit of soluble bismuth—

Standard solution—Transfer 242.0 mg of bismuth nitrate pentahydrate to a 100-mL volumetric flask. Add 3 mL of 1.5 N nitric acid, swirl to dissolve, dilute with water to volume, and mix. Transfer 1.0 mL of this solution to a 500-mL volumetric flask, add 250 mL of 1.5 N nitric acid, dilute with water to volume, and mix. This solution contains 2.0 µg of bismuth (Bi) per mL. [NOTE—The concentration of bismuth in this solution may be modified by using a different quantity or by further dilution to bring the absorption responses within the working range of the atomic absorption spectrophotometer.]

Test solution—Prepare a mixture of 5.0 g of Bismuth Citrate and 100 mL of water, and stir by mechanical means the suspension thus obtained for 2 hours. Pass through filter paper. Pass the filtrate thus obtained through a filter having a 0.1-µm or finer porosity. To 10.0 mL of the filtrate add 0.1 mL of nitric acid.

Procedure—Concomitantly determine the absorbances of the *Standard solution* and the *Test solution* at the emission line of 223.06 nm for bismuth with an atomic absorption spectrophotometer (see *Spectrophotometry and Light-Scattering* 〈851〉) equipped with a bismuth hollow-cathode lamp and an oxidizing flame. The absorbances of the *Test solution* do not exceed those of the *Standard solution* (40 µg per g).

Assay—Transfer about 300 mg of Bismuth Citrate, accurately weighed, to a porcelain crucible, and ignite. Allow to cool, add 2 mL of nitric acid to the residue, dropwise, and warm until complete solution has been effected. Add about 60 mL of water and 0.3 mL of xylenol orange TS, and titrate with 0.05 N edetate disodium VS to a yellow endpoint. Each mL of 0.05 N edetate disodium is equivalent to 10.45 mg of bismuth (Bi).

Bismuth Subcarbonate

» Bismuth Subcarbonate contains not less than 97.6 percent and not more than 100.7 per cent of $(BiO)_2CO_3$, calculated on the dried basis.

Packaging and storage—Preserve in well-closed containers, protected from light.

Identification—It responds to the tests for *Bismuth* ⟨191⟩ and for *Carbonate* ⟨191⟩.

Loss on drying ⟨731⟩**—**Dry it at 105° to constant weight: it loses not more than 1.0% of its weight.

Chloride ⟨221⟩**—**Mix 5.0 g of it with 10 mL of water, add 20 mL of nitric acid, warm to achieve dissolution, allow to cool, and dilute with water to obtain 100 mL of solution. To 6.6 mL of this stock solution add 4 mL of nitric acid, and dilute with water to obtain 50 mL of solution. A 15.0-mL portion of this test solution shows no more chloride than corresponds to 70 µL of 0.020 N hydrochloric acid (0.05%).

Limit of alkalies and alkaline earths—Boil 1.0 g of it with 20 mL of a mixture of acetic acid and water (1:1). After 2 minutes, cool and filter. Collect the filtrate, wash the residue with 20 mL of water, and add the washing to the filtrate. To this solution add 2 mL of 2 N hydrochloric acid and 20 mL of water. Heat to boiling and precipitate the bismuth by adding hydrogen sulfide. Cool the mixture, and filter. Collect the filtrate, wash the residue with water, and add the washing to the filtrate. Evaporate this solution to dryness on a water bath. To the residue add 0.5 mL of sulfuric acid, dry slowly, and cool: the weight of the residue does not exceed 10 mg (1.0%).

Limit of nitrate—

Indigo carmine titrant—Dissolve 4 g of indigo carmine in 900 mL of water, add 2 mL of sulfuric acid, and dilute with water to 1000 mL.

Standard solution—Prepare a solution of potassium nitrate in water containing 0.0815 mg per mL (equivalent to 0.05 mg of nitrate per mL). Add 20.0 mL of this solution to a 125-mL conical flask (*Standard solution*).

Test preparation—To 250 mg of Bismuth Subcarbonate in a 125-mL conical flask add 20 mL of water, and swirl to suspend.

Procedure—To the *Standard solution* and the *Test preparation* add 0.05 mL of *Indigo carmine titrant*. Carefully add 30 mL of sulfuric acid, and immediately titrate with *Indigo carmine titrant* to a stable blue endpoint. The volume of *Indigo carmine titrant* consumed by the *Test preparation* does not exceed that consumed by the *Standard solution* (0.4%).

Limit of silver—To 2.0 g of Bismuth Subcarbonate add 1 mL of water and 4 mL of nitric acid. Heat gently to achieve dissolution, add water to obtain 11 mL of solution, and cool. Add 2 mL of 1 N hydrochloric acid, and allow to stand in a dark place for 5 minutes. No more turbidity is produced than corresponds to that produced with 10 mL of a solution containing 7.87 µg of silver nitrate per mL concomitantly treated with 1 mL of nitric acid and 2 mL of 1 N hydrochloric acid (0.0025%).

Arsenic, *Method I* ⟨211⟩**—**Prepare the *Test Preparation* by dissolving 600 mg of it in 35 mL of 3 N hydrochloric acid. The limit is 5 µg per g.

Limit of copper—

Standard solution—To a 100-mL volumetric flask add 1.34 g of cupric chloride, 10 g of ammonium chloride, and 3 mL of sodium metabisulfite solution (275 mg per mL). Dilute with water to volume, and mix. This stock solution contains the equivalent of 5 mg of copper per mL. Dilute an accurately measured volume of this solution quantitatively and stepwise with 2 N nitric acid to obtain a solution containing the equivalent of 10 µg of copper per mL. Mix 0.25 mL of this solution and 9.75 mL of water (*Standard solution*).

Test solution—To 5 mL of the stock solution retained from the test for *Chloride* add 2 mL of 6 N ammonium hydroxide, dilute with water to 50 mL, mix, and filter. Use the filtrate as the *Test solution*.

Procedure—To 10 mL of the *Standard solution* and the *Test solution* add 1 mL of a solution of sodium diethyldithiocarbamate (1 in 1000): no more color is obtained from the *Test solution* than is obtained from the *Standard solution* (0.005%).

Limit of lead—

Diluent—Use 6 N nitric acid that is lead-free.

Standard solutions—Prepare a solution of lead nitrate in *Diluent* containing 0.1598 mg per mL. This solution contains 100 µg of lead per mL. Dilute an accurately measured volume of this solution, quantitatively and stepwise, with *Diluent* to obtain *Standard solutions* containing 1.0, 2.0, and 3.0 µg of lead per mL.

Test solution—Dissolve 12.5 g of Bismuth Subcarbonate in 75 mL of *Diluent*. Heat to boiling for 1 minute, cool, and dilute with water to 100 mL.

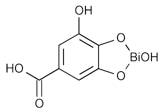
Procedure—Concomitantly determine the absorbances of the *Standard solutions* and the *Test solution* at the lead emission line of 283.3 nm with an atomic absorption spectrophotometer (see *Spectrophotometry and Light-scattering* ⟨851⟩) equipped with a lead hollow-cathode lamp and an air–acetylene flame, using a 1:5 dilution of the *Diluent* as the blank. Plot the absorbances of the *Standard solutions* versus concentration, in µg per mL, of lead, and draw the straight line best fitting the three plotted points. From the graph so obtained, determine the concentration, *C*, in µg per mL, of lead in the *Test solution*. Calculate the percentage of lead (Pb) in the portion of Bismuth Subcarbonate taken by the formula:

$$C / 1250.$$

The limit is 0.002%.

Assay—Dissolve about 500 mg of Bismuth Subcarbonate, accurately weighed, in 3 mL of nitric acid. Dilute with water to 250 mL, add 0.3 mL of xylenol orange TS, and titrate with 0.05 M edetate disodium VS to a yellow endpoint. Each mL of 0.05 M edetate disodium is equivalent to 12.75 mg of $(BiO)_2CO_3$.

Bismuth Subgallate



$C_7H_5BiO_6$ 394.09
Gallic acid bismuth basic salt [99-26-3].

» Bismuth Subgallate is a basic salt which, when dried at 105° for 3 hours, contains the equivalent of not less than 52.0 per cent and not more than 57.0 percent of Bi_2O_3.

Packaging and storage—Preserve in tight, light-resistant containers.

Identification—

A: When heated to redness, it at first chars, leaving finally a yellow residue. This residue responds to the tests for *Bismuth* ⟨191⟩.

B: Agitate thoroughly about 100 mg with an excess of hydrogen sulfide TS, filter, and boil the filtrate to expel the dissolved gas. Cool, and add 1 drop of ferric chloride TS: a purplish blue mixture is produced.