tate 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. T o 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. T o 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 dr yness on a water bath. T o the residue add 0.5 mL of sulfuric acid, dr y 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.

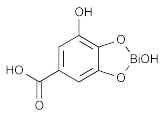
Procedure—Concomitantly determine the absorbances of the *Standard solutions* and the *Test solution* at the lead emission line of 283.3 nm with an atomic absorption spectrophotometer (see *Spectrophotometry and Light-scattering* ⟨851⟩) equipped with a lead hollow-cathode lamp and an air–acetylene flame, 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.