AOAC Official Method 932.19
Bismuth Compounds in Drugs

Gravimetric Method
First Action 1932
Final Action 1965

(Applicable in absence of Pb.)

Caution: *See* Appendix B, safety notes on distillation, nitric acid, and hydrogen sulfide.

Thoroughly mix test sample and weigh 0.5 g into 500 mL Kjeldahl flask. Ignite gently over small flame, using wire gauze under flask, and increase heat towards end. Let cool, add 15–20 mL HNO_3, evaporate to dryness, and ignite as before until yellow or orange Bi_2O_3 is formed. Cool residue and dissolve in 10–15 mL warm HNO_3, using few mL 3% H_2O_2 if residue does not dissolve readily. Boil off excess H_2O_2 and wash into 400 mL beaker with H_2O, rinsing flask well. Dilute to ca 200 mL, make just neutral to litmus with NH_4OH, and add 5 mL HCl. Precipitate with H_2S completely.

Transfer precipitate to filter paper and wash once with HCl (5 + 200) and then several times with H_2O. Dissolve precipitate of Bi_2S_3 on filter with hot HNO_3 (1 + 2). Small residue of S (and HgS if Hg salts are present) usually remains. Neutralize filtrate with NH_4OH (2 + 3) and precipitate with 25 mL 20% $(NH_4)_2CO_3$ solution. Concentrate to ca 150 mL (by boiling, if desired) and let stand on steam bath 1–2 h. Collect precipitate in previously ignited, weighed Gooch, wash with small amount of H_2O, dry, ignite in furnace at ca 550°C, and weigh as Bi_2O_3.

Reference: *JAOAC* **15,** 422(1932).