

15.3.01

AOAC Official Method 940.32 Sulfides in Depilatory Powders

Titrimetric Method

First Action 1940

Final Action

Pipet 50 mL 0.1M As_2O_3 solution, **939.12B** (*see* A.1.03), into 250 mL glass-stoppered volumetric flask. Weigh test sample containing <0.12 g sulfide calculated as H_2S and transfer to flask, washing down any material on sides of flask with H_2O . Add 20 mL HCl (1 + 1), stopper immediately, and shake vigorously until sample decomposes. (If sample contains CaCO_3 , slowly add the 20 mL acid through dropping funnel fitted with rubber stopper to fit flask. Shake gently, letting liberated CO_2 bubble up through acid. When reaction

subsides, drain remainder of acid into flask, remove funnel, stopper flask, and shake vigorously.)

Cool to room temperature and dilute to volume with H_2O . Filter through dry paper into dry flask. Pipet 100 mL filtrate (test solution) into 300 mL Erlenmeyer; add 5 mL starch solution (mix ca 1 g soluble starch with enough cold H_2O to make thin paste, and 100 mL boiling H_2O , and boil ca 1 min with stirring), and enough I_2 solution to form blue solution. Make alkaline with NaHCO_3 , adding 1–2 g excess. Titrate to permanent blue with 0.1M I , **939.13A** (*see* A.1.07). Subtract mL 0.1M I_2 consumed in alkaline titration from mL 0.1M As_2O_3 present in aliquot. 1 mL 0.1M As_2O_3 = 0.005411 g CaS or 0.01271 g BaS .

References: *JAOC* **23**, 437(1940); **25**, 113(1942); **27**, 112(1944).

CAS-21109-95-5 (barium sulfide)

CAS-20548-54-3 (calcium sulfide)