

3.3.01

AOAC Official Method 928.03 Aluminum and Iron in Plants

Titrimetric Method

First Action 1928

Final Action

Take aliquot of *Solution I, 920.08* (see 3.1.05), containing enough Fe and Al to form ca 40 mg FePO_4 and AlPO_4 . Add few drops HNO_3 , $\text{Br}_2\text{-H}_2\text{O}$, or H_2O_2 to oxidize Fe. If solution does not already contain excess phosphate, add 0.5 g $(\text{NH}_4)_2\text{HPO}_4$, stir until dissolved, and dilute to 50 mL with H_2O . Add few drops thymol blue solution (0.1%: dissolve 0.1 g thymol blue in H_2O , add enough 0.1M NaOH to change color to blue, and dilute to 100 mL), and then add NH_4OH until solution just turns yellow. Add 0.5 mL HCl and 25 mL 25% $\text{CH}_3\text{COONH}_4$ solution, and stir. Let stand at room temperature until precipitate settles (ca 1 h). Filter, and wash 10 times with hot 5% NH_4NO_3 solution. Ignite at 500 –550°C and weigh as FePO_4 and AlPO_4 .

Fuse ignited residue in Pt crucible with ca 4 g $\text{Na}_2\text{CO}_3\text{-K}_2\text{CO}_3$ (1 + 1) mixture. When fusion is complete, let crucible cool, add 5 mL H_2SO_4 , and heat until copious fumes of SO_3 are evolved. Cool, transfer to flask, add H_2O , and digest until solution is clear. Reduce Fe with Zn, cool, and titrate with 0.02M KMnO_4 . Correct for blank and calculate as percent Fe or percent Fe_2O_3 . Calculate to FePO_4 and subtract from total FePO_4 and AlPO_4 to obtain AlPO_4 . Correct for blank and report as Al_2O_3 .

References: *JAOAC* **11**, 203(1928); **16**, 70(1933); **19**, 70(1936).

CAS-7784-30-7 (aluminum phosphate)

CAS-10045-86-0 (ferric phosphate)