

3.3.03

AOAC Official Method 921.01 Calcium in Plants

Titrimetric Micro Method First Action 1921 Final Action

Determination

Weigh 2 g test portion into small crucible and ignite in furnace at 500–550°C. Dissolve ash in HCl (1 + 4) and transfer to 100 mL beaker. Add 5 mL HCl and evaporate to dryness on steam bath to dehydrate SiO₂. Moisten residue with 5 mL HCl, add ca 50 mL H₂O, heat few minutes on steam bath, transfer to 100 mL volumetric flask, cool quickly to room temperature, dilute to volume, shake, and filter, discarding first portion of filtrate.

Pipet 15 mL aliquot into conical-tip centrifuge tube containing 2 mL saturated (NH₄)₂C₂O₄ solution and 2 drops methyl red (dissolve 1 g methyl red in 200 mL alcohol). Add 2 mL CH₃COOH (1 + 4), rotating tube to mix contents thoroughly. Add NH₄OH (1 + 4), while intermittently rotating tube, until solution is faintly alkaline; then add few drops of the CH₃COOH until color is faint pink (pH 5.0). (It is important at this point to rotate tube so that last bit of liquid in conical tip has required color.) Let stand 4 h; then centrifuge 15 min. (Precipitate should be in firm lump in tip of tube.) Remove supernate, using suction device, Figure 921.01, taking care not to disturb precipitate. Wash precipitate by adding 2 mL NH₄OH (1 + 49), rotating tube to break up precipitate. (It may be necessary to jar tube sharply.) Centrifuge 10 min, again remove supernate, and wash with reagent as before. Repeat washing of precipitate 3 times.

After removing last supernate, add 2 mL H₂SO₄ (1 + 4) to tube, break up precipitate as before, heat on steam bath to 80–90°C, and titrate in

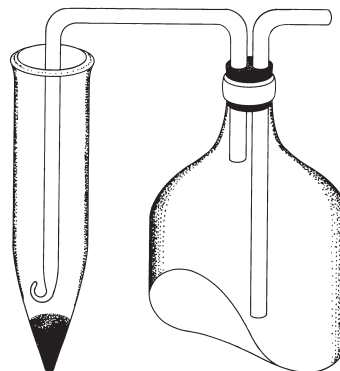


Figure 921.01. Suction device used in micro method for determining calcium.

tube with 0.004M KMnO₄, rotating liquid during titration to attain proper end point. If tube cools to <60°C during titration, as indicated by slow reduction of KMnO₄, reheat in steam bath few minutes and complete titration. Perform blank on identical volume H₂SO₄ in similar tube heated to same temperature to determine volume KMnO₄ solution necessary to give end point color. Subtract this value from buret reading.

$$1 \text{ mL } 0.004\text{N KMnO}_4 = 0.000400 \text{ g Ca}$$

Report as % Ca.

References: *J. Biol. Chem.* **47**, 475(1921); **50**, 527, 537(1922).
JAOAC **14**, 216(1931); **16**, 71(1933); **19**, 71(1936).

CAS-7440-70-2 (calcium)