4.8.04

AOAC Official Method 935.13
Calcium in Animal Feed

Wet Ash Method

Final Action

A. Preparation of Solution

(*Caution: See Appendix B,* safety notes on nitric acid and perchloric acid.)

(**a**) Weigh 2.5 g sample into 500 or 800 mL Kjeldahl flask. Add 20–30 mL HNO_3 and boil gently 30–45 min to oxidize all easily oxidizable matter. Cool solution somewhat and add 10 mL 70–72% $HClO_4$. Boil very gently, adjusting flame as necessary, until solution is colorless or nearly so and dense white fumes appear. Use particular care not to boil to dryness (Danger!) at any time. Cool slightly, add 50 mL H_2O, and boil to drive out any remaining NO_2 fumes. Cool, dilute, filter into 250 mL volumetric flask, dilute to volume, and mix thoroughly.

(**b**) Weigh 2.5 g finely ground sample into SiO_2 or porcelain dish and ignite as in **942.05** (*see* 4.1.10). Add 40 mL HCl (1 + 3) and few drops HNO_3 to residue, boil, transfer to 250 mL volumetric flask, cool, dilute to volume, and mix thoroughly.

B. Determination

Pipet suitable aliquot of clear solution, **935.13A**(**a**) or (**b**), into beaker, dilute to 100 mL, and add 2 drops methyl red, **984.13B**(**c**) (*see* 4.2.09). Continue as in **927.02** (*see* 4.8.03), beginning "Add NH_4OH (1 + 1) dropwise . . ." except use 0.05*N* $KMnO_4$ for titration.

(100 mL is suitable aliquot of sample solution for grain feeds; for mineral feeds, 25 mL aliquot may be taken and titrated with 0.1*N* $KMnO_4$. For suitable precision, size of sample, aliquot, and concentration of $KMnO_4$ must be so adjusted that ≥20 mL standard $KMnO_4$ solution is used.)

References: Ind. Eng. Chem. Anal. Ed. **7,** 116, 167(1935). JAOAC **30,** 606(1947); **31,** 98(1948); **32,** 650(1949); **33,** 162(1950); **34,** 563(1951).

CAS-7440-70-2 (calcium)