4.8.14

AOAC Official Method 965.17
Phosphorus in Animal Feed and Pet Food

Photometric Method
First Action 1965
Final Action 1966

(Not applicable to mineral–mix feeds. Dry ashing procedure is not applicable to feeds, pet foods, or mineral mixes containing monobasic calcium phosphate.)

A. Apparatus

Spectrophotometer.—Capable of isolating 400 nm band and accepting 15 mm diameter cells.

B. Reagents

(**a**) *Molybdovanadate reagent*.—Dissolve 40 g ammonium molybdate $4H_2O$ in 400 mL hot H_2O and cool. Dissolve 2 g ammonium metavanadate in 250 mL hot H_2O and cool; add 250 mL 70% $HClO_4$. (*Caution: See* Appendix B, safety notes on perchloric acid.) Gradually add molybdate solution to vanadate solution with stirring, and dilute to 2 L.

(**b**) *Phosphorus standard solutions.*—(*1*) *Stock solution*.—2 mg P/mL. Dissolve 8.788 g KH_2PO_4 in H_2O and dilute to 1 L. (*2*) *Working solution*.—0.1 mg P/mL. Dilute 50 mL stock solution to 1 L.

C. Preparation of Standard Curve

Transfer aliquots of working standard solution containing 0.5, 0.8, 1.0, and 1.5 mg P to 100 mL volumetric flasks. Treat as in **D**, beginning "Add 20 mL molybdovanadate reagent, . . .". Prepare standard curve by plotting mg P against %*T* on semilog paper.

D. Determination

Ash 2 g test portion, in 150 mL beaker, 4 h at 600°C. Cool, add 40 mL HCl (1 + 3) and several drops HNO_3, and bring to bp. Cool, transfer to 200 mL volumetric flask, and dilute to volume with H_2O. Filter, and place aliquot containing 0.5–1.5 mg P in 100 mL volumetric flask. Add 20 mL molybdovanadate reagent, dilute to volume with H_2O, and mix well. Let stand 10 min; then read %*T* at 400 nm against 0.5 mg standard set at 100% *T*. (Use 15 mm diameter cells.) Determine mg P from standard curve.

$$\text{P, \%} = \frac{\text{mg P in aliquot}}{\text{g test portion in aliquot} \quad 10}$$

References: *JAOAC* **48,** 654(1965); **59,** 937(1976).

CAS-7723-14-0 (phosphorus)

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