3.4.11

AOAC Official Method 931.01
Phosphorus in Plants

Micro Method
First Action 1931
Final Action

A. Reagents

(**a**) *Phosphorus standard solution.*—0.025 mg P/mL. Dissolve 0.4394 g pure dry KH_2PO_4 in H_2O and dilute to 1 L. Dilute 50 mL of this solution to 200 mL.

(**b**) *Ammonium molybdate solution.*—Dissolve 25 g ammonium molybdate in 300 mL H_2O. Dilute 75 mL H_2SO_4 to 200 mL and add to ammonium molybdate solution.

(**c**) *Hydroquinone solution.*—Dissolve 0.5 g hydroquinone in 100 mL H_2O, and add one drop H_2SO_4 to retard oxidation.

(**d**) *Sodium sulfite solution.*—Dissolve 200 g Na_2SO_3 in H_2O, dilute to 1 L, and filter. Either keep this solution well stoppered or prepare fresh each time.

B. Preparation of Test Solution

To 1 or 2 g test portion in small porcelain crucible, add 1 mL $Mg(NO_3)_2$ solution [dissolve 950 g P-free $Mg(NO_3)_2$ $6H_2O$ in H_2O and dilute to 1 L], and place on steam bath. After few minutes, cautiously add few drops HCl, taking care that gas evolution does not push solids or test solution over edge of crucible. Make 2 or 3 further additions of few drops HCl while test solution is on bath so that as material approaches dryness it tends to char. If contents become too viscous for further drying on bath, complete drying on hot plate.

Cover crucible, transfer to cold furnace, and ignite 6 h at 500°C, or until even gray ash is obtained. (If necessary, cool crucible, dissolve ash in a little H_2O or alcohol–glycerol, evaporate to dryness, and return uncovered to furnace 4–5 h longer.) Cool, take up with HCl (1 + 4), and transfer to 100 mL beaker. Add 5 mL HCl and evaporate to dryness on steam bath to dehydrate SiO_2. Moisten residue with 2 mL HCl, add ca 50 mL H_2O, and heat few minutes on bath. Transfer to 100 mL volumetric flask, cool immediately, dilute to volume, mix, and filter, discarding first portion of filtrate.

C. Determination

To 5 mL aliquot filtrate in 10 mL volumetric flask, add 1 mL ammonium molybdate solution, rotate flask to mix, and let stand few seconds. Add 1 mL hydroquinone solution, again rotate flask, and add 1 mL Na_2SO_3 solution. (Last 3 additions may be made with Mohr pipet.) Dilute to volume with H_2O, stopper flask with thumb or forefinger, and shake to mix thoroughly. Let stand 30 min, and measure *A* with spectrophotometer set at 650 nm. Report as percent P.

References: *JAOAC* **14,** 216(1931).
J. Biol. Chem. **59,** 255(1924).

CAS-7723-14-0 (phosphorus)