

10.5 Determination of phosphorus – Precipitation method

Reagents

- Concentrated nitric acid
- Nitric acid (1:1) – A mixture of equal volumes of concentrated nitric acid and water.
- Ammonium molybdate stock solution – Take 200 g of powdered ammonium molybdate in a stoppered graduated cylinder of 1000 ml capacity, add to it 800 ml of water and shake well for 25 minutes to dissolve the ammonium molybdate. Add gradually 25 per cent ammonium hydroxide solution till the solution is clear (about 100 to 140 ml of ammonium hydroxide may be required). Avoid adding excess of ammonia. Make up the volume to one litre. If necessary, filter the solution through a fluted filter paper and stock this solution.
- Nitric acid solution – 2 per cent (m/v).
- Potassium nitrate solution – 3 per cent (m/v)
- Standard sodium hydroxide solution – 0.1 N.
- Standard nitric acid solution – 0.1 N.
- Phenolphthalein indicator solution – Dissolve 0.1 g of phenolphthalein in 100 ml of 60 per cent (m/v) rectified spirit.

Procedure

Precipitation

Take 10 ml aliquot of the prepared solution (As in ashing and extraction of calcium) in a 150 ml beaker. In a dry beaker, prepare ammonium molybdate solution by pouring into it, quickly and simultaneously 10 ml of the ammonium molybdate stock solution and 10 ml of concentrated nitric acid; or take 10 ml of concentrated nitric acid first in the beaker and into this pour quickly 10 ml of the ammonium molybdate stock solution, whirling the beaker during addition. Pour this freshly prepared clear liquid quickly into the beaker containing the aliquot and stir.

Filtration and washing

Allow the precipitate to stand overnight and then filter through a disc of Whatman filter paper No. 42 in a gooch crucible by suction or through a 9 cm Whatman filter paper No. 42 over an ordinary funnel. As far as possible only the supernatant liquid is passed through the filter paper, retaining the precipitate in the beaker. When the supernatant liquid is decanted off, the precipitate is washed twice with dilute nitric acid and then with potassium nitrate solution until the washings is free from acid. If ordinary funnel and filter paper are used, freedom from acidity may be tested by collecting sufficient filtrate in test tube to which a few drops of phenolphthalein indicator solution and one drop of the standard sodium hydroxide solution are added. If the pink color appears with one drop of the standard alkali, the precipitate is free from acid.

Titration

Transfer the precipitate with the filter paper back to the beaker in which precipitation was carried out. When gooch crucible is used for filtration, transfer the whole crucible along with the filter paper to the beaker in which precipitation was carried out. Add sufficient quantity of the standard sodium hydroxide solution from a burette just sufficient to dissolve the precipitate and then add 5 ml in excess. See that no yellow precipitate sticks to the filter paper. Note the total volume of the standard sodium hydroxide solution added. Add about 10 drops of phenolphthalein indicator solution and titrate the excess of alkali with the standard nitric acid.

Calculation

$$\text{Phosphorus (on moisture-free basis) per cent by mass} = \frac{336.75 (AN_1 - BN_2)}{m (100 - M)}$$

Where,

A = volume in ml of the standard sodium hydroxide solution used,

N₁ = normality of the standard sodium hydroxide solution,

B = volume in ml of the standard nitric acid used in to neutralize the excess alkali

N₂ = normality of the standard nitric acid,

m = mass in g of the material taken for the test and,

M = per cent moisture content

Reference: IS:7874 (part – II) – 1975. Methods for animal feeds and feeding stuffs. Part-II. Minerals and trace elements.