

## 10.4 Determination of calcium – Precipitation method

### Reagents

- Hydrochloric acid – 25 ml of concentrated hydrochloric acid diluted to 100 ml.
- Methyl red indicator – Dissolve 0.15 g of methyl red in 500 ml of water.
- Ammonium hydroxide solution – 50 per cent (v/v).
- Dilute ammonium hydroxide solution – 2 per cent (v/v).
- Ammonium oxalate solution – Saturated.
- Concentrated sulphuric acid
- Standard potassium permanganate solution – 0.1 N.

### Procedure

1. Ashing and extraction – Accurately weigh about 3 g of the material into a silica dish. Char carefully and continue the ashing in a muffle furnace at a temperature not above 450°C until the ash is white or almost so. Cool the ash, moisten with a few millilitres of distilled water and add 3 to 5 ml of concentrated hydrochloric acid drop by drop. Evaporate to dryness on a water-bath and continue heating on the water bath for one hour to render silica insoluble. Moisten the residue with 20 ml distilled water and add about 2 to 3 ml of concentrated hydrochloric acid. Heat on a water bath for a few minutes and filter through medium filter paper into a 250 ml volumetric flask. Wash the filter paper thoroughly with hot water, cool the filtrate and make it up to volume, shake thoroughly.
2. Transfer a 25 ml aliquot of the solution prepared as in (1) to a 400 ml beaker, dilute to about 100 ml with water and add two drops of methyl red indicator solution. Add ammonium hydroxide solution drop wise till a brownish – orange color is obtained (pH 5.6). Add two drops of hydrochloric acid so that the color of solution is pink (pH 2.5 to 3.0). Dilute to about 150 ml, bring to the boil and add slowly, with constant stirring, 10 ml of hot ammonium oxalate solution. If the red color of the solution changes to orange or yellow, add hydrochloric acid drop wide until the color again changes to pink. Leave overnight to allow the precipitate to settle. Filter the supernatant liquid through ash-less filter paper and wash the precipitate thoroughly with dilute ammonium hydroxide solution. Place the paper with the precipitate in the beaker which precipitation was carried out and add a mixture of 125 ml of water and 5 ml of concentrated sulphuric acid, heat to 70 to 90°C and titrate with the standard potassium permanganate solution until the first slight pink colour is obtained.

### Calculation

$$\text{Calcium (as Ca) (on moisture-free basis), per cent by mass} = \frac{2000 AN}{m (100-M)}$$

Where,

A = volume in ml of the standard potassium permanganate solution required in the titration

N = normality of the standard potassium permanganate solution

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.