10.8 Determination of iron – Colorimetric method

Apparatus

- Heat resistant glass tube – of 50 ml capacity, marked at 30 ml.
- Centrifuge – suitable for clarifying the isoamyl alcohol phase.
- Photoelectric colorimeter – capable of measuring optical density at 495 nm. (Fig. 10.6)

Fig. 10.6 Photoelectric colorimeter

Reagents

- Distilled water – redistilled.
- Concentrated sulphuric acid – r.d. 1.84
- Perchloric acid – 60 per cent (m/m) solution.
- Concentrated nitric acid – 60 per cent (m/m).
- Ammonium hydroxide solution – 25 per cent (m/m).
- Concentrated hydrochloric acid – 35 per cent (m/m)
- Hydrogen peroxide solution – 0.1 per cent (m/m) solution in water stored in a brown bottle in a refrigerator.
- Isoamyl alcohol – of boiling point 129 to 132ºC.
- Potassium thiocyanate solution – Dissolve 50 g of potassium thiocyanate (KSCN) in 100 ml of water.
- Standard iron solution – Dissolve 0.7022 g of ferrous ammonium sulphate ($FeSO_4(NH_4)_2SO_4.6H_2O$) in 100 ml of water, add 5 ml of concentrated sulphuric acid, warm slightly and add potassium permanganate solution (approximately 0.1 N) drop by drop until the solution shows a slight pink coloration. Make up the volume to one litre in a graduated flask. Pipette 10 ml of this solution into a one litre graduated flask, add 10 ml of hydrogen peroxide solution and make up the volume with water. This solution contains 1 microgram of iron per millilitre.

Procedure

Preparation of the test solution

1. Weigh accurately about 2.0 g of the material and transfer to a 200 ml Erlenmeyer flask. Add 2 ml of concentrated sulphuric acid, 3 ml of perchloric acid and 5 ml of concentrated nitric acid. Digest until a clear solution is obtained and while fumes of sulphuric acid are evolved. Dilute with 10 ml of water and make up the volume to 200 ml with water in a graduated flask. Preserve this solution for the determination of copper and cobalt.

2. Take a suitable aliquot of the test solution containing about 10 microgram of iron and transfer to the heat resistant glass tube. Add ammonium hydroxide solution until the solution is just alkaline to phenolphthalein. Add 1 ml of concentrated hydrochloric acid and 1 ml of hydrogen peroxide solution and make up the volume in the tube to 30 ml with distilled water. Add 10 ml of isoamyl alcohol, accurately measured, and 2 ml of potassium thiocyanate solution, stopper the tube and shake for 20 seconds. Transfer enough of the isoamyl alcohol phase meant for colour measurement to the centrifuge tubes, and centrifuge for 5 minutes at about 3000 rev/min. Measure the absorption of the solution in a suitable photo-electric colorimeter at 495 nm setting the reading of the blank at zero absorption. The blank is prepared simultaneously by

using the same quantities of acid employed in the digestion, making up the volume and developing the colour in the same size aliquot and in the same manner as in the case of the test solution.

3. Prepare a series of standards by treating aliquots of the standard iron solution in the same manner as the test solution. From the absorption of the standard solutions, prepare a standard curve plotting absorption values against concentrations. From this curve, obtain the mass of iron present in the test solution and calculate the quantity of iron present in 100 g of the material on moisture free basis.

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