

10.10 Determination of manganese – Colorimetric method

Apparatus

- Photoelectric colorimeter – capable of measuring optical density of 520 nm.

Reagents

- Concentrated sulphuric acid – r.d 1.84.
- Sulphurous acid – saturated solution stored in an amber bottle in a cool place.
- Solution A – Mix 42 ml of water, 2 ml of sulphuric acid, 5 ml of sulphurous acid solution and 1 ml of phosphoric acid. This solution should be freshly prepared before use.
- Potassium periodate
- Sodium metabisulphite solution – 10 per cent (m/v), aqueous.
- Phosphoric acid – r.d 1.70
- Concentrated nitric Acid – r.d 1.42
- Standard manganese solution – Dissolve 0.5756 g of dry potassium permanganate in about 50 ml of water in a beaker of suitable size. Add 40 ml of concentrated sulphuric acid and reduce the permanganate by careful addition of sodium metabisulphite solution until the manganese solution just becomes colourless. Oxidize the excess sulphurous acid in the hot solution by the addition of a little nitric acid. Cool and transfer the solution quantitatively to a 2 litre graduated flask. Make up the volume and store the solution in a glass stoppered reagent bottle. This solution contains 0.1 mg of manganese per millilitre.

Procedure

1. Weigh accurately about 5 g of the material into a silica dish, char carefully and ash it in a muffle furnace at 600 to 700°C. Cool, extract the ash with 10 ml of solution A for 2 minutes and transfer to a 150 ml beaker. Rinse the dish first with 40 ml of solution A and then with distilled water, collecting the rinsing until the volume is 100 ml. Heat to the boil on a hot plate and evaporate the solution, using a boiling tube until the volume is reduced to 20 ml. Care should be taken not to allow the solution to bump. Allow the solution to stand overnight. Filter through a small disc of ash less filter paper under slight suction into a 150 ml beaker. Wash the filter paper and dilute and filtrate with water to about 100 ml. Add 2 ml of phosphoric acid and 0.3 g of potassium periodate. Boil to oxidize the manganese and continue boiling for about 15 minutes after the colour has been apparently fully developed. The final volume should not be less than 50 ml (If necessary, boiling water may be added to the solution while boiling). Cool and dilute to 100 ml. Measure the absorption of the solution at 52 nm by means of a suitable photo electric colorimeter.
2. Simultaneously carry out a control determination under the same conditions as above step 1, adding 5 ml of the standard manganese solution, 2 ml of sulphuric acid and 2 ml of phosphoric acid to 100 ml of water and oxidizing with potassium periodate as described in step 1.
3. Measure the absorption at 520 nm of a series of aliquots of the standard manganese solution treated in the same manner as the test solution. Plot a curve of these absorption values against concentration. From this curve, obtain the mass of manganese in the test solution and calculate the quantity of manganese 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.