10.11 Determination of cobalt – Colorimetric method

Apparatus

- Spectrophotometer or photoelectric colorimeter – of a suitable type.

Reagents

- Citric acid – 0.2 M. Prepare by dissolving 42 g of citric acid in 100 ml of water and standardize against standard sodium hydroxide solution using phenolphthalein as indicator.
- Bromophenol blue indicator solution – Dissolve 40 mg of bromophenol blue in 100 ml of water containing 5.7 ml sodium hydroxide (0.01 N).
- Methyl red indicator solution – Dissolve 25 mg of methyl red in 100 ml of ethyl alcohol (60 per cent v/v).
- Dithizone solution in chloroform – 0.2 per cent (m/v). Keep in a dark bottle in a refrigerator.
- Dithizone solution in carbon tetrachloride – 0.05 per cent (m/v). Keep in a dark bottle in a refrigerator.
- Phenolphthalein indicator solution – Dissolve 50 mg of phenolphthalein in 100 ml ethyl alcohol (50 per cent m/v).
- Buffer solution – Dissolve 6.184 g of boric acid and 25.62 g of Sorensen's salt (Na_2HPO_4).$2H_2O$) in 500 ml of standard sodium hydroxide (1.0 N) and make up the volume to one litre with distilled water.
- Concentrated nitric acid – 60 per cent (m/v).
- Perchloric acid – 60 per cent.
- Concentrated sulphuric acid – r.d. 1.84.
- Cresol red indicator solution – Dissolve 40 mg of cresol red in 100 ml of water containing 10.5 ml of sodium hydroxide (0.01N).
- Nitroso-R salt – 0.2 per cent (m/v) aqueous solution stored in dark.
- Standard cobalt solution – Dissolve 4.769.4 g of cobalt sulphate ($CoSO_4.7H_2O$) in distilled water, add one millilitre of concentrated sulphuric acid and make up the volume to one litre. Take one millilitre of this solution and make up volume to one litre with water in a graduated flask. This solution contains 1 microgram of cobalt per millilitre.

Procedure

1. Take an aliquot of 5 ml of the test solution as prepared in iron determination. Evaporate the water cautiously until all but a trace of sulphuric acid is removed. Add 7.5 ml of nitric acid to the residue and wash the solution into a 100 ml separating funnel. Dilute to about 30 ml with water.

2. Extraction with dithizone – Add 5 drops of bromophenol blue to the solution. Run in sodium hydroxide solution (1.0 N) until a distinct greenish blue colour appears through the yellowish tint due to the ferric citrate. The solution should still be acid to methylred. Dilute the solution to 50 ml. Extract the solution with successive 20 ml portions of dithizone solution in chloroform. Shake vigorously and run off the chloroform layer. When the chloroform layer retains the original green colour of the dithizone solution, the test solution is washed once with pure chloroform.

3. Adjust the pH of the aqueous phase to approximately 8.3 by adding a few drops of phenolphthalein and cautiously titrating with the buffer solution until the first sign of a purplish pink colour appears. Extract the cobalt with successive 10 ml portions of dithizone solution in carbon tetrachloride until the carbon tetrachloride phase retains the green colour of original dithizone solution. Boil off the carbon tetrachloride from a heat resistant boiling tube. Add to the residue 1 ml of nitric acid, 0.5 ml of perchloric

acid and 0.2 ml of sulphuric acid and heat till it becomes colourless. Heat the boiling tube for a few minutes in a muffle furnace at a temperature not above 350ºC to ensure complete removal of sulphuric acid.

4. Production of the cobalt – Nitroso-R salt complex – Dissolve the residue in 1 ml of citric acid and dilute with a little water so that the total volume is not more than 5 ml. Add accurately 1.2 ml of the buffer solution to adjust the pH. The pH is checked with cresol red by withdrawing a small drop of the solution. Develop the cobalt-nitroso-R salt complex by introducing 0.5 ml of the cobalt-nitroso-R salt solution while shaking. Boil for one minute. Cool and dilute to 10 ml. Measure the absorption of the solution in a suitable spectrophotometer or photoelectric colorimeter at 510 nm setting the reading of the blank at zero absorption. The blank is prepared simultaneously by using the same quantities of the reagents employed in the digestion and in the subsequent procedure. Make up the volume to 100 ml and develop the color in the same size aliquot and in the same manner as in the case of the test solution.

5. Prepare a series of standards by treating aliquots of the standard cobalt 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 cobalt present in the test solution and calculate the quantity of cobalt present in the test solution and calculate the quantity of cobalt 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.