

Assay of iodine(KI)

Reagents

1. Methyl orange indicator – Dissolve 0.50 g of methyl orange in water and dilute to one litre.
2. Dilute sulphuric acid – approximately 2 N.
3. Bromine water – Saturated aqueous solution. Determine the approximate concentration (mg/ml) by adding (from a burette) a measured volume to a flask containing 5 ml of 10 per cent potassium iodide solution, adding 5 ml of dilute sulphuric acid and titrating the liberated iodine with 0.1 N sodium thiosulphate solution.
4. Sodium sulphite solution – approximately one per cent (m/v).
5. Phenol solution – approximately 5 per cent (m/v).
6. Potassium iodide solution – approximately 10 per cent (m/v).
7. Standard sodium thiosulphate solution – 0.005 N (freshly standardized).
8. Starch solution – 1 per cent (freshly prepared) (m/v).
9. Sodium chloride solution – Dissolve 10 g of sodium chloride in water and make up the volume to 100 ml.
10. Potassium iodide control solution –

Dissolve 0.3280 g of potassium iodide in water and then make up the volume to 250 ml. Dilute 50 ml of this solution to 250 ml, and use 5 ml control (that is, 1.0 mg iodine or 0.308 mg potassium iodide).

11. Concentrated sulphuric acid – r.d. 1.84.

Preparation of sample solution -:Weigh accurately about 50 g of the material and suspend in 100 ml of water. Add 2 g of takadiastase and allow to stand at 37°C for 2 hours. Filter the solution and wash the residue with water. Collect the filtrate and washings and make up the volume to 250 ml in a graduated flask.

Procedure:-

Pipette 50 ml of the prepared same solution into a 200 ml Erlenmeyer flask. Neutralize to methyl orange indicator with dilute sulphuric acid. Add bromine water drop wise from burette in a quantity equivalent to 20 mg of bromine. After a few minutes, destroy most of the remaining free bromine by adding sodium sulphite solution drop wise with stirring. Wash down the neck and sides of the flask with water and completely remove free bromine by addition of a drop or two ml of potassium iodine solution and titrate the liberated iodine with standard sodium thiosulphate solution adding 1 ml of the starch indicator near the end of the titration. Carry out a blank determination of reagents and make one or more control determinations, using 50 ml of sodium

chloride solution to which have been added appropriate quantities of the potassium iodide control solution.

Calculation

$$\frac{1384 (V_1 - V_2) \times 1.2 \times N}{132 \times V} \text{ Iodine (as KI), on moisture free basis, per cent by mass} = \frac{m (100 - M)}{132 \times V}$$

Where, V_1 = Volume in ml of the standard sodium thiosulphate solution required for the test with the prepared sample solution, V_2 = Volume in ml of the standard sodium thiosulphate solution required for the blank determination N = Normality of the standard sodium thiosulphate solution. M = Mass in g of the material taken, and M = Per cent moisture content