

10.13 Determination of fluorine - Distillation method

Apparatus

- Distillation flasks
- Nessler tubes – of 50 ml capacity
- Micro-burette

Reagents

1. Lime water free from fluorine – Dissolve lime in an excess of perchloric acid. Boil for 15 minutes. Dilute, cool and neutralize with fluorine-free sodium hydroxide. Filter through a Buchner funnel and wash. Make a saturated solution in distilled water using the lime thus free from fluorine.
2. Perchloric acid solution – 60 to 70 per cent (m/v). Heat some quantity for an hour or longer at 140 to 150°C.
3. Silver perchloric solution – 1 per cent (m/v). Prepare by adding sufficient sodium hydroxide solution to a solution of silver nitrate to cause precipitation. Filter and wash the precipitate with water. Dissolve the precipitate in perchloric acid and dilute.
4. Sodium hydroxide solution – 0.5 N.
5. Alizarin Indicator solution – Dissolve 0.02 g of sodium alizarin sulphonate in water and make up the volume to 100 ml.
6. Dilute hydrochloric acid – 0.05 N.
7. Buffer solution – Dissolve 0.1 g of hydroxylamine hydrochloride in water and make up the volume to 100 ml.
8. Thorium nitrate solution – Dissolve 0.5 g of hydrated thorium nitrate ($\text{Th}(\text{NO}_3)_4 \cdot 12\text{H}_2\text{O}$) in distilled water and make up the volume to one litre.
9. Standard fluorine solution – Dissolve 2.211 g of sodium fluoride in water and make up the volume to one litre in a graduated flask. Pipette out 10 ml of this solution into a one litre graduated flask and make up the volume. This solution contains 0.01 mg of fluorine per millilitre.

Procedure

1. Weigh accurately about 5 g of the material. Moisten with lime water. Dry on a water bath and ignite in a muffle furnace at about $550 \pm 20^\circ\text{C}$. When the ashing is complete, cool and transfer to distillation flask, washing it with water. Dissolve the residual ash in 10 to 15 ml of perchloric acid and transfer to the same distillation flask. Add sufficient silver perchlorate to bring about complete precipitation. Steam-distil at $132 \pm 3^\circ\text{C}$ into another flask containing 2 ml of the sodium hydroxide solution. Collect about 150 ml of the distillate. Transfer the distillate to a 200 ml graduated flask and make up the volume to the mark with water.
2. Transfer a suitable aliquot of the test solution containing 10 to 30 microgram of fluorine to a Nessler tube. Add 1 ml of alizarin indicator solution. Take the same quantity of the indicator solution in another Nessler tube. If necessary, the alkali in the test solution is neutralized with a drop or two of hydrochloric acid. To each tube, add 1 ml of the buffer solution 2.0 ml of hydrochloric acid and dilute to about 45 ml. The colour of the solutions should be straw-yellow. Add the thorium nitrate solution from a microburette to the test solution until a permanent slight pink colour appears (0.5 to 2.5 ml of thorium nitrate solution would be required). Add an equal volume of the thorium nitrate to the other Nessler tube. Adjust the colour of the solution in this Nessler tube by adding the standard fluorine solution from a micro-burette to the same intensity as the colour of the test solution. When the colour in both the tubes

matches, the amount of fluorine in the standard fluorine solution added is equal to the amount of fluorine present in the aliquot of the test solution. From this, calculate the amount of fluorine 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.