10.14 Determination of fluorine - Ion selective electrode (ISE) method

Apparatus

- Ion analyzer (Fig. 10.8)
- Single junction reference electrode
- Solid state fluoride electrode
- Magnetic stirrer & stir bars
- Plastic lab ware

Required solutions

- Distilled or deionised water
- 100 ppm fluoride standard solution
- Reference electrode filling solution
- Total Ionic Strength Adjuster Buffer (TISAB-III). To provide constant background ionic strength, decomplex fluoride and adjust solution pH.



Fig. 10.8 lon analyzer

Preparation of sample

Weigh accurately about 0.3 g sample into 100 ml plastic beaker. Add 5 ml of 5 M hydrochloric acid and mix it well to dissolve all the material. Transfer the solution into 100 ml volumetric flask and make the volume 100 ml with deionised water. Take 25 ml aliquot in a plastic beaker and add 2.5 ml TISAB-III and measure the concentration on Ion analyzer.

Preparation of standard

Stock solution (500 ppm): Accurately weigh 1.105 g NaF (reagent grade, dried 4 hours at 100°C) into 1 litre volumetric flask. Dissolve and dilute to volume with H_2O and mix thoroughly. Store in plastic bottle at room temperature.

Preparation of working standard from readily available 100 ppm fluoride solution:

- 1) 1 ppm fluoride standard solution: Take 1 ml stock solution (100 ppm) into 100 ml volumetric flask and dilute to volume with H_2O and mix. It gives 1 ppm fluoride concentration.
- 2) 10 ppm fluoride standard solution: Take 10 ml stock solution (100 ppm) into 100 ml volumetric flask and dilute to volume with H₂O and mix. It gives 10 ppm fluoride concentration.

Take 25 ml aliquot and add 2.5 ml TISAB-III and use for meter calibration.

Note: Use plastic lab ware for fluorine estimation.

Determination of fluoride concentration

Connect fluoride and single junction reference electrodes to ISE meter, place electrodes in standards for calibration of ISE meter. After calibration of meter with two or three standards, place electrodes in sample and stir the solution at constant rate, read concentration of standard and unknown solution directly from the meter.

Calculation

% Fluorine = $\frac{\text{Meter reading (ppm) x 100 x 10}^{-4}}{\text{Weight of sample (g)}}$

Reference: AOAC (1995) Official Methods of Analysis. 975.08.