

THE DETERMINATION OF FLUORIDE IN GLASS/ROCKS

This method involves the determination of fluoride in glass using an EDT directION Fluoride Ion Selective electrode.

Equipment Required:

1. EDT directION Model DR359TX pH/Ion analyzer
2. Fluoride Combination ISE Cat no 3221
3. Furnace and Platinum crucible.

Reagents:

1. Sodium carbonate A.R. (anhydrous)
2. Sodium Fluoride A.R.
3. Potassium Hydroxide
4. Potassium Chloride
5. Glacial Acetic acid
6. Hydrochloric acid

Preparation of Buffer Solution:

Dissolve 28g KOH in 300ml of distilled water in a 600ml beaker. When completely dissolved add 30ml glacial acetic acid. Add 37g KCl, dissolve completely. Cool to ambient temperature and dilute to one litre in a volumetric flask with distilled water.

Preparation of Standards:

Weigh 4.200g NaF and place in a 400 ml beaker. Add 300 ml distilled water and heat to dissolve. Cool, and pour into a one litre flask. Dilute to the mark, mix thoroughly and place in a dried plastic bottle. This is 10^{-1} M stock solution.

Prepare 10^{-3} Standard by diluting 5ml of stock solution to approximately 250ml with distilled water. Add 75ml buffer solution and 1.0g sodium carbonate and neutralise to pH 7.4 - 7.6 with hydrochloric acid.

The solution is transferred to a 500ml flask and diluted to the mark with water.

Prepare a 10^{-4} standard by diluting 50ml of the 10^{-3} standard to 250ml using distilled water. Add 67.5ml buffer solution and 0.9g sodium carbonate. Neutralise to pH 7.4 as before and make up to 500 ml.

Sample Preparation:

Fuse an accurately weighed, powdered sample with 2g of sodium carbonate in a platinum crucible. When a clear fused melt is obtained, continue heating for approximately 1 minute. Temperature estimate is 1000 degrees but this will vary with sample type. Remove the crucible from the burner and pour the contents onto a cooling plate. Place the crucible, lid and cooled melt into a 400ml beaker, add 75ml of buffer solution and dilute to 200ml using boiling water.

Place the beaker on a water bath for 30 minutes to loosen the melt from the crucible. Remove crucible and lid from beaker, and rinse the filter thoroughly with distilled water. Dilute to 300ml and adjust the pH to 7.4 - 7.6 with 1:3 hydrochloric acid, making sure the pH does not drift below 7.0.

Transfer the solution to a 1 litre flask and dilute to the mark using distilled water.

Method:

Immerse the electrodes in each of the standards, the least concentrated first, with the DR359TX in the Concentration mode. Immerse the electrodes in the sample solution and read off the concentration value.

Calculation:

$$\% \text{ Fluoride in sample} = \frac{\text{Contraction reading} \times 19 \times 100}{\text{Sample weight (g)}}$$

Al^{3+} , Fe^{3+} and La^{3+} may form interfering complexes while Ca^{2+} will cause precipitation of insoluble CaF_2 and so should not be present.