

Determination of Fluoride in Water

The ion-selective electrode method has been selected for the determination of fluoride in water because of its practical convenience and reliability. Others also available include the lanthanum alizarin complexone colorimetric method but some involve appreciable practical experience in, for example, special distillation techniques.

1. SCOPE

Waters containing soluble uncomplexed ionic fluoride at levels down to 0.1mg per litre. The total soluble fluoride can be obtained by use of a citrate total ionic strength buffer (8.2).

2. PRINCIPLE OF METHOD

Direct reading of response of a fluoride ion-selective electrode immersed in the sample, without need for preliminary distillation.

3. APPARATUS

Fluoride ion-selective electrode. Available commercially and normally consisting of an electrode made from a single crystal of lanthanum fluoride (in the form of a disc or plug) cemented into the end of a polyvinyl chloride or other suitable plastic tube, in combination with a reference calomel electrode. Expanded-scale pH meter.

4. REAGENTS

Stock fluoride solution, 1ml = 100 micrograms F. Dissolve 221mg anhydrous sodium fluoride in water and make up to 1,000ml. Standard fluoride solution, 1ml = 10 micrograms F. Dilute 100ml of stock fluoride solution to 1,000ml. Total ionic strength adjustment buffer solution.

Place approximately 500ml distilled water in a 1-litre beaker. Add 57ml glacial acetic acid, 58g sodium chloride and 12g sodium citrate dihydrate,

dissolve. Cool, insert a calibrated pH electrode and reference electrode into the solution and slowly add approximately 6 N sodium hydroxide (about 125ml) until the pH is between 5.0 and 5.5. Cool to room temperature, transfer to a 1 litre volumetric flask and add distilled water to the mark.

5. PROCEDURE

Follow the instructions provided by the electrode supplier. Normally the electrode should be inserted into the sample and the sample agitated for 3min until a steady meter reading is obtained, using an ionic strength adjustment buffer where necessary. A typical procedure is as follows: Calibrate the instrument using a series of standard solutions containing respectively 0, 0.2, 0.4, 0.6, 0.8, 1.0, 1.2, 1.6 and 2.0mg per litre fluoride (prepared by adding 0, 1.0, 2.0 . . . 10.0ml of standard fluoride into a series of 100ml volumetric flasks, adding to each 50ml of total ionic strength adjustment buffer solution and making each up to 100ml). Place 50ml of sample in a 100ml volumetric flask, dilute to 100ml with total ionic strength adjustment buffer solution.

Transfer each standard and sample to a series of 150ml beakers. Immerse the electrode and measure the developed potential while agitating. Allow the electrode to remain in the solution 3min before taking a final reading. Rinse the electrode with distilled water and blot dry between each reading. When using an expanded-scale pH meter (or specific-ion meter) calibrate the electrode frequently by checking the potential reading of the 1.0mg/l (50µgF) standard and adjust the calibration control (if necessary) until the meter reads as before. Confirm the calibration after reading each unknown and also after reading each standard when preparing the standard curve. Using the potential measurement for each unknown sample, read the corresponding fluoride concentration from the standard curve.

6. EXPRESSION OF RESULTS

Results should be expressed in mg F per litre of sample.

7. SPECIAL CASES

Special buffer solutions, to be added to the sample, may be necessary in the presence of aluminium in amounts up to 0.5 (8.1) or 5.0mg per litre (8.2). Interference may also arise from the presence of other polyvalent cations which form complexes with fluoride ions such as Fe^{+3} , Th^{+4} . The addition of a polyvalent anion such as citrate or phosphate has been used to avoid such interferences.

8. REFERENCES

- 8.1 Patterson, S. J., Bunton, N. G. and Crosby, N. T. *Water Treatment & Exam*, **18**, 182-191, 1969.
- 8.2 Harwood, J. E. *Water Res.*, **3**, 273-280, 1969.
- 8.3 Standard Methods for the Examination of Water and Wastewater, 13th Edition, pp. 172-174. American Public Health Association, 1970.