Scope

This method is used for the determination of fluoride in drinking water, surface water, and domestic and industrial wastes according to USGS Method I-4327-85 and Standard Methods 4500-F ⁻ G and using an ion-selective electrode (ISE).

Summary

Fluoride is determined potentiometrically using a fluoride-specific ion-selective electrode with a sealed reference electrode in a double-junction configuration. The operation of the fluoride electrode is based upon the potential that develops across a crystal lanthanum fluoride membrane. This potential is proportional to the activity of fluoride ions in contact with the membrane. The fluoride ion activity is related to the free fluoride concentration. The activity coefficient is estimated from the total quantity of ions in solution or the ionic strength. A total ionic strength adjusting buffer (TISAB) is used to stabilize the ionic strengths of the samples at high levels, making their activity coefficients essentially the same. The quality of the analysis is assured through reproducible calibration and testing of the Flow Injection Analysis (FIA) system.

Interferences

Cations and most anions do not interfere with the response of the fluoride electrode to fluoride ions. Anions commonly associated with fluoride, such as chloride, bromide, iodide, sulfate, bicarbonate, nitrate, phosphate, and acetate, do not interfere with the electrode operation.

Polyvalent cations of silicon(IV), iron(II), and aluminum(III) interfere by forming complexes with fluoride. The amount of interference depends upon the concentrations of the complexing cations, the concentration of fluoride, and the pH of the sample. Eliminate this interference by adding 1,2-cyclohexylenedinitrilotetraacetic acid monohydrate (CDTA) to the TISAB to bind the complexing metal ions. In a sample containing 1-mg/L fluoride, CDTA binds approximately 3–5-mg/L aluminum or iron.

Hydroxide ion is an electrode interferant. Anions such as carbonate and phosphate make the sample more basic, increasing the hydroxide interference. Eliminate this interference by buffering the sample to pH 5.0–5.5 using the TISAB.

In solutions with pH <5, hydrogen ions complex with fluoride, forming a poorly ionized hydrogen fluoride complex (HF–HF). Buffer the sample to pH 5.0–5.5 to eliminate this interference.

Since electrode potentials are affected by temperature changes, samples and standards should be as close as possible to the same temperature. A 1 °C change in temperature can cause up to a 2% error in the fluoride results. The slope of the fluoride electrode also varies with temperature.

Performance Specifications

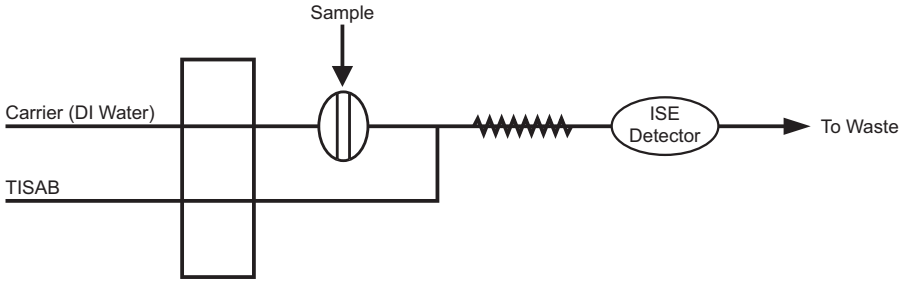
Range		0.10–4.0 mg/L
Throughput		24 samples/hour
Precision at	0.20 mg/L	<1% RSD
	2.0 mg/L	<0.5% RSD
Method Detection Limit (MDL)		0.05 mg/L
Accuracy*:		98%

* ERA (Environmental Resource Associates) WasteWatR Minerals Quality Control Sample.

Chemicals

Acetic Acid, glacial, $C_2H_4O_2$
1,2-Cyclohexylenedinitrilotetraacetic Acid Monohydrate (CDTA), $C_6H_{10}[N(CH_2CO_2H)_2]_2 \cdot H_2O$
Deionized Water (ASTM Type I or II)
Sodium Chloride, NaCl
Sodium Fluoride, NaF
Sodium Hydroxide, NaOH

Basic Flow Diagram



Selected References

Fluoride by ISE. *Methods for Chemical Analysis of Water and Wastewater*; EPA-600/4-79-020; U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory: Cincinnati, OH, 1984; Method 340.2.

Standard Methods for the Examination of Water and Wastewater, 21st ed.; American Public Health Association: Washington, D.C., 2005.

Methods for the Determination of Inorganic Substances in Water and Fluvial Sediments; Fluoride, Electrometric, Ion-Selective Electrode; I-4327-85; U.S. Geological Survey

Figures

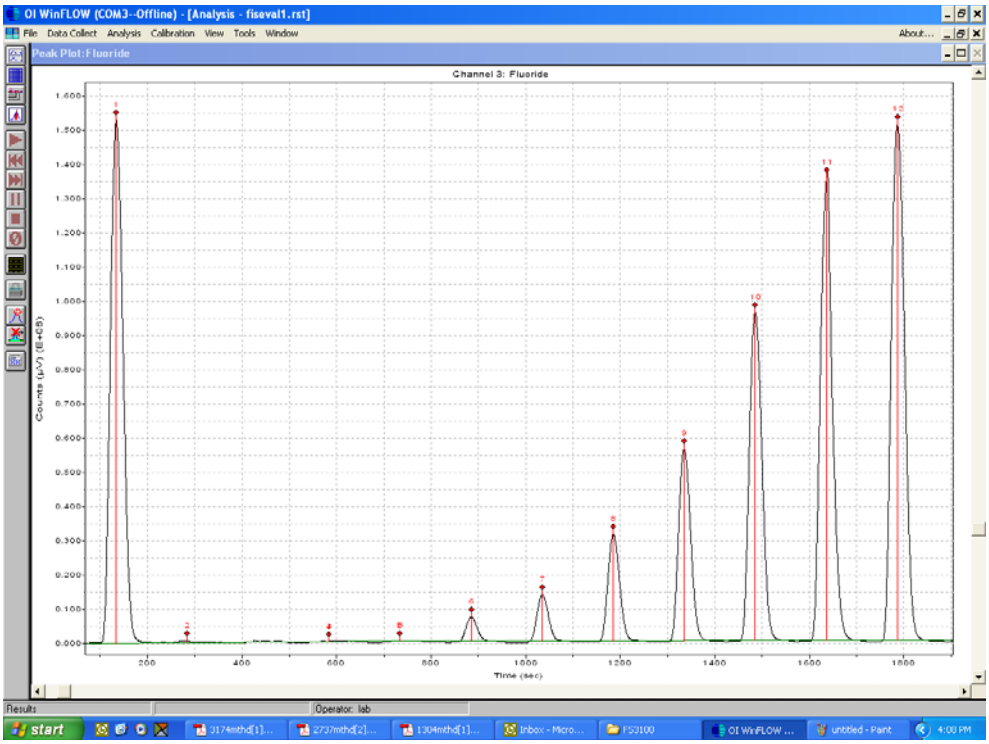


Figure 1. Fluoride Calibration (0.10–4.0 mg/L)

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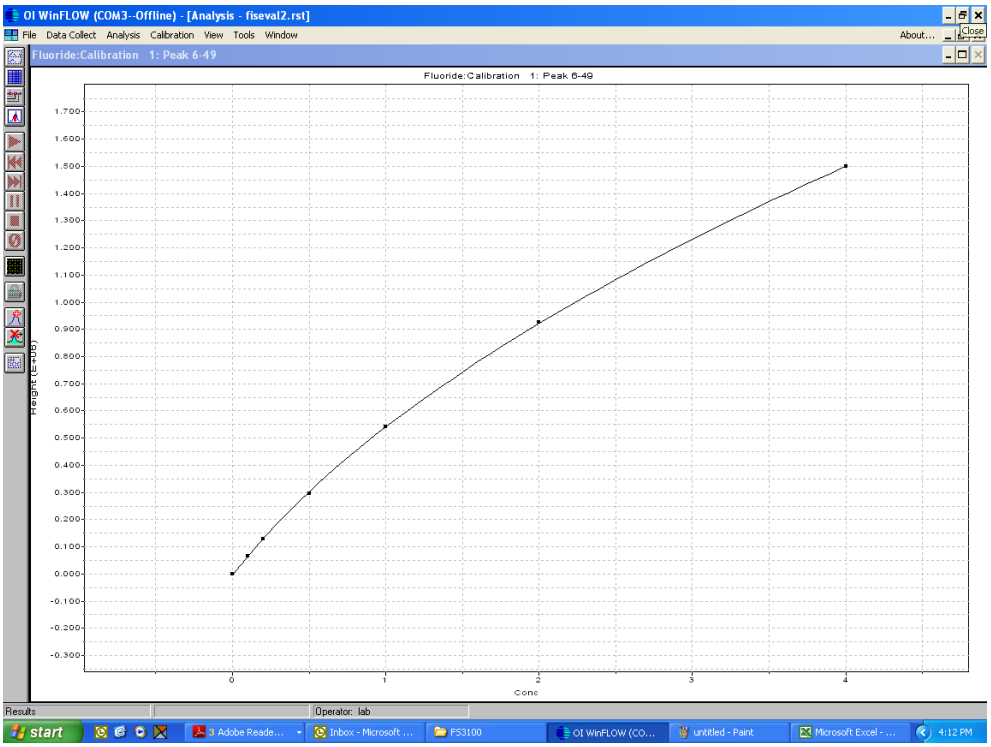

Figure 2. Fluoride Calibration Curve (0.10–4.0 mg/L)

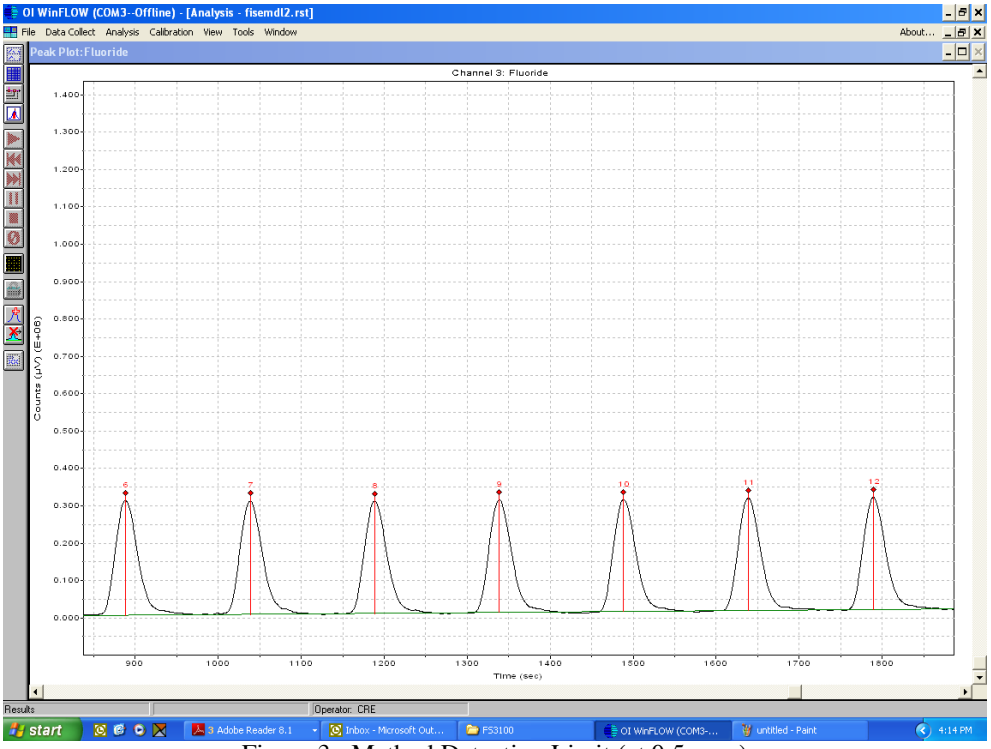

Figure 3. Method Detection Limit (at 0.5 ppm)

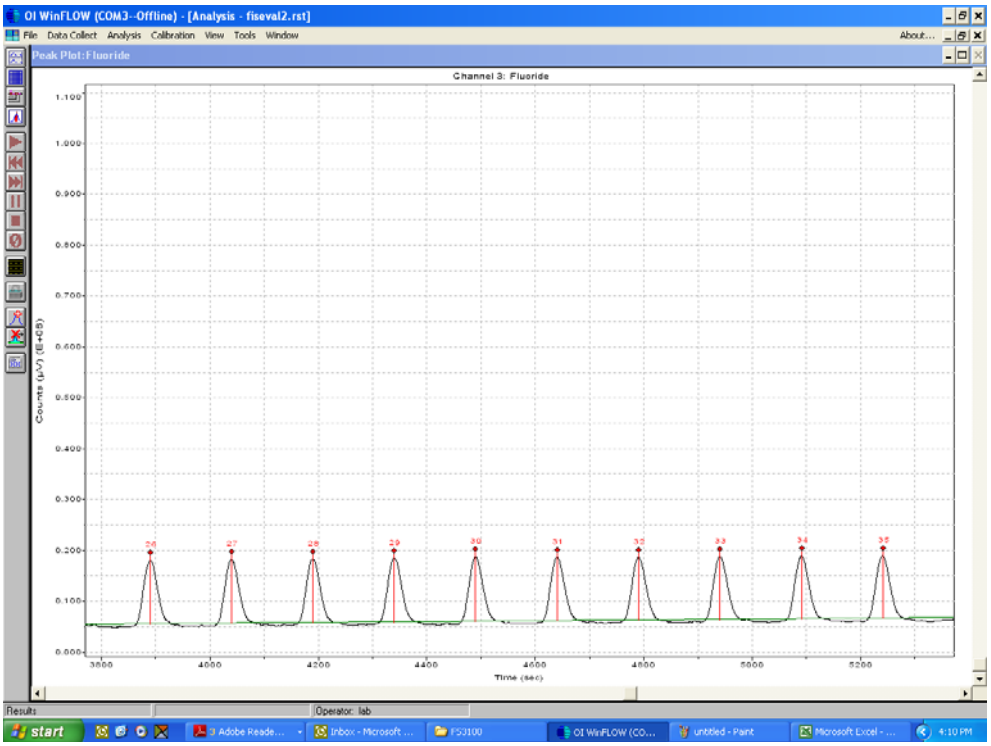

Figure 4. Fluoride Precision (at 0.2 ppm)

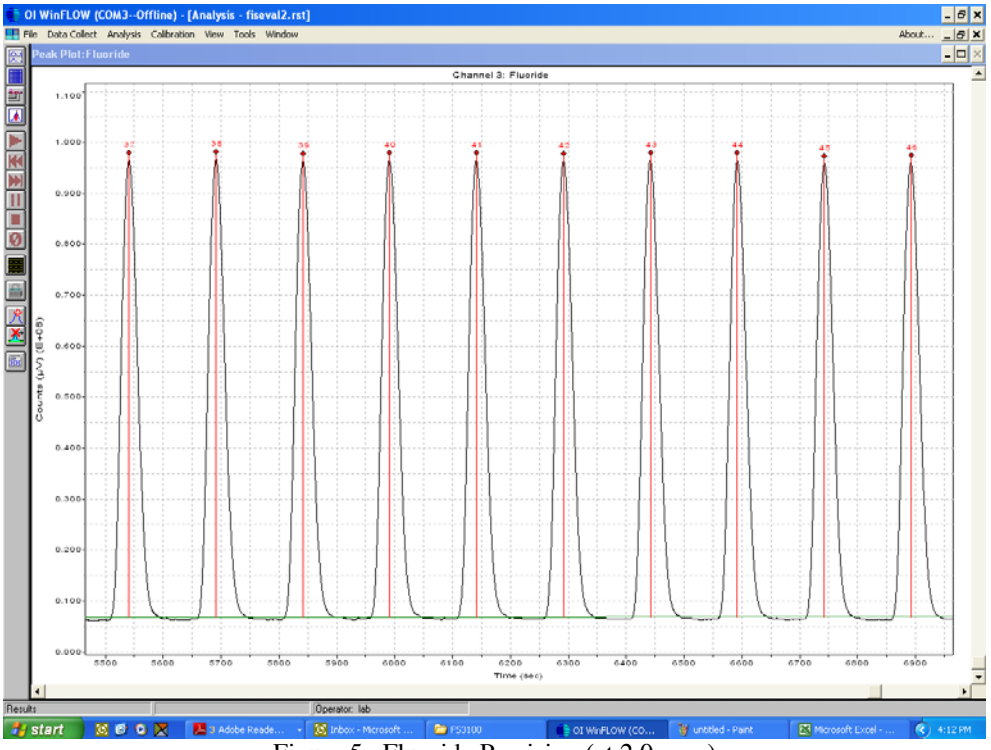

Figure 5. Fluoride Precision (at 2.0 ppm)

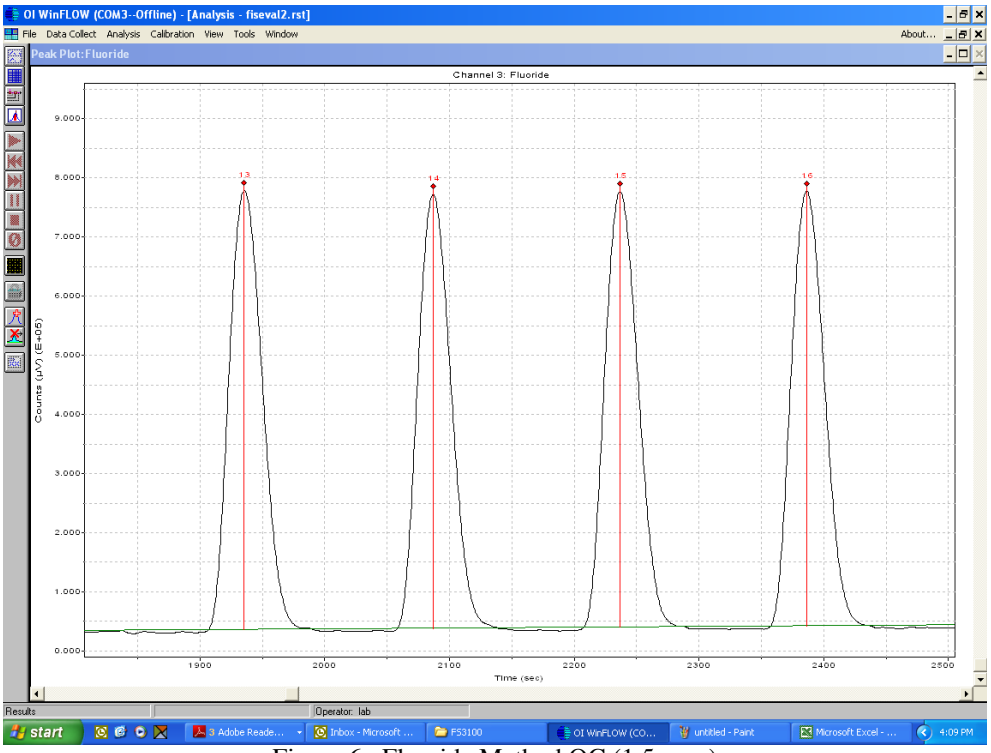

Figure 6. Fluoride Method QC (1.5 ppm)

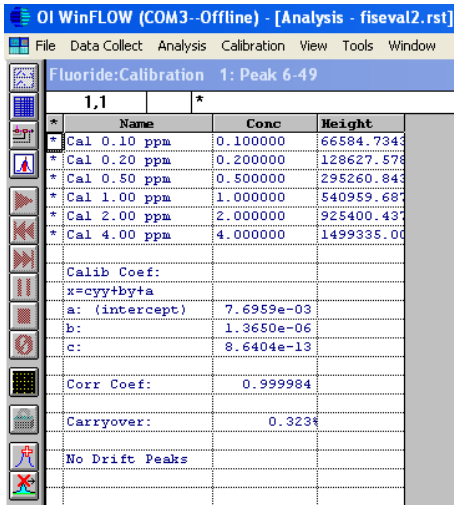

Figure 7. Fluoride Calibration Results (0.10–4.0 mg/L)

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