O·I·Analytical

Methodology

Fluoride by ISE and Combined Flow Injection Analysis (FIA) and Segmented Flow Analysis (SFA)

(Cartridge Part #A002832)

1.0 Scope and Application

1.1 This method is used for the determination of fluoride in drinking water, surface water, and domestic and industrial wastes using an ion-selective electrode (ISE) (Reference 15.2).

1.2 The Method Detection Limit (MDL) of this method is 0.02 mg/L fluoride. The applicable range of the method is 0.20–8.0 mg/L fluoride. The range may be extended to analyze higher concentrations by sample dilution.

2.0 Summary of Method

2.1 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 ISE 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 per Equation 1.

EQUATION 1

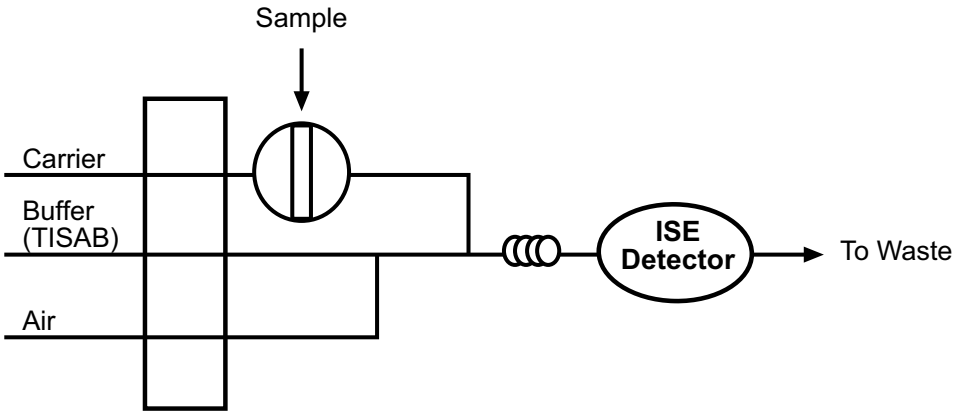
$$A = (\gamma) \times (c)$$

Where:
A = Activity
γ = Activity coefficient
c = 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.

2.2 The quality of the analysis is assured through reproducible calibration and testing of the Flow Injection Analysis (FIA)/Segmented Flow Analysis (SFA) system.

2.3 A general flow diagram of the FIA/SFA system is shown below (see Section 17.0 for a detailed flow diagram).



3.0 Definitions

Definitions for terms used in this method are provided in Section 16.0, “Glossary of Definitions and Purposes.”

4.0 Interferences

4.1 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.

4.2 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 and iron.

4.3 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 (Reference 15.4).

4.4 In solutions with pH <5, hydrogen ions combine with fluoride, forming a poorly ionized hydrogen fluoride complex (HF–HF). Buffer the sample to pH 5.0–5.5 to eliminate this interference (Reference 15.4).

4.5 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.

5.0 Safety

5.1 The toxicity or carcinogenicity of each compound or reagent used in this method has not been fully established. Each chemical should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level.

5.2 For reference purposes, a file of Material Safety Data Sheets (MSDS) for each chemical used in this method should be available to all personnel involved in this chemical analysis. The preparation of a formal safety plan is also advisable.

5.3 The following chemicals used in this method may be highly toxic or hazardous and should be handled with extreme caution at all times. Consult the appropriate MSDS before handling.

5.3.1 Acetic Acid, glacial, $C_2H_4O_2$ (FW 60.05)

5.3.2 1,2-Cyclohexylenedinitrilotetraacetic Acid Monohydrate (CDTA), $C_6H_{10}[N(CH_2CO_2H)_2]_2 \cdot H_2O$ (FW 364.36)

5.3.3 Kleenflow™ Basic (Part #A002294)

5.3.4 Sodium Chloride, NaCl (FW 58.44)

5.3.5 Sodium Fluoride, NaF (FW 41.99)

5.3.6 Sodium Hydroxide, NaOH (FW 40.00)

5.4 Unknown samples may be potentially hazardous and should be handled with extreme caution at all times.

5.5 Proper personal protective equipment (PPE) should be used when handling or working in the presence of chemicals.

5.6 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

6.0 Apparatus, Equipment, and Supplies

6.1 Flow Injection Analysis (FIA)/Segmented Flow Analysis (SFA) System (OI Analytical Flow Solution® 3000) consisting of the following:

6.1.1 120-Place Autosampler

6.1.2 Data Acquisition System (PC or Notebook PC) with WinFLOW™ software

6.1.3 Fluoride by ISE Cartridge (Part #A002832), which includes:

6.1.3.1 Expanded Range (ER) Potentiometric Detector

6.1.3.2 Combination Fluoride ISE

6.2 Sampling equipment—Sample bottle, high density polyethylene (HDPE), with polytetrafluoroethylene (PTFE)-lined cap. Clean by washing with detergent and water, rinsing with two aliquots of reagent water, and air drying on a rack.

6.3 Standard laboratory equipment including volumetric flasks, pipettes, syringes, etc. should all be cleaned, rinsed, and dried per bottle cleaning procedure in Section 6.2.

7.0 Reagents and Calibrants

7.1 Raw Materials

7.1.1 Acetic Acid, glacial, $C_2H_4O_2$ (FW 60.05)

7.1.2 Brij®-35, 30% w/v (Part #A21-0110-33)

7.1.3 1,2-Cyclohexylenedinitrilotetraacetic Acid Monohydrate (CDTA), $C_6H_{10}[N(CH_2CO_2H)_2]_2 \cdot H_2O$ (FW 364.36)

7.1.4 Deionized Water (ASTM Type I or II)

7.1.5 Kleenflow Basic (Part #A002294)

7.1.6 Sodium Chloride, NaCl (FW 58.44)

7.1.7 Sodium Fluoride, NaF (FW 41.99)

7.1.8 Sodium Hydroxide, NaOH (FW 40.00)

7.2 Reagent Preparation

Note: For best results, filter and degas all reagents prior to use. Avoid the use of glass-distilled water. Immediately after preparation, transfer all reagents and calibrant solutions to polyethylene containers.

7.2.1 Reagent Water

7.2.1.1 Degassed and deionized reagent water can be prepared in one of the following manners:

7.2.1.1.1 Place distilled/deionized water under a strong vacuum for 15–20 minutes. Magnetic stirring or sonification will aid in the degassing process.

7.2.1.1.2 Purge distilled/deionized water with a stream of nitrogen gas (or other inert gas) through a frit for approximately 5 minutes.

7.2.1.2 After preparing the degassed reagent water, store the reagent water in a tightly sealed container to protect it from reabsorption of atmospheric gases. For best results, store degassed reagent water under a slight vacuum when not in use.

7.2.2 Start-up Solution (1 L)

7.2.2.1 Add 0.25 mL of Brij-35 to approximately 800 mL of reagent water (Section 7.2.1) in a 1-L volumetric flask.

7.2.2.2 Dilute to 1,000 mL with reagent water and mix gently.

7.2.3 1 N Sodium Hydroxide (100 mL)

7.2.3.1 Dissolve 4 g of sodium hydroxide in approximately 50 mL of reagent water in a 100-mL volumetric flask.

7.2.3.2 Dilute to 100 mL with reagent water and mix well.

Warning: Mixing sodium hydroxide and water produces a great amount of heat. Take appropriate precautions.

7.2.4 Stock Total Ionic Strength Adjusting Buffer (Stock TISAB) (2 L)

7.2.4.1 While stirring, carefully add 115 mL of glacial acetic acid to approximately 1,000 mL of reagent water in a 2-L beaker.

7.2.4.2 Add 116 g of sodium chloride, 75 g of sodium hydroxide, and 8 g of CDTA. Stir until dissolved.

7.2.4.3 Allow the solution to cool to room temperature. Adjust the pH to 5.25 with 1 N sodium hydroxide (Section 7.2.3).

7.2.4.4 Add 2 mL of stock 1,000 mg/L fluoride calibrant (Section 7.3.1) for a final fluoride concentration of 1 mg/L.

7.2.4.5 Quantitatively transfer the solution to a 2-L volumetric flask and dilute to 2,000 mL with reagent water and mix well.

Warning: Mixing glacial acetic acid, sodium hydroxide, and water produces a great amount of heat. Take appropriate precautions.

Note: Store in a polyethylene container at 4°C. If stored properly, this solution is stable for 4–6 weeks.

7.2.5 Working TISAB (1 L)

7.2.5.1 Add 2 mL of Brij-35 to 1,000 mL of stock TISAB (Section 7.2.4) and mix gently.

Note: Prepare this solution fresh daily.

7.2.6 Carrier/Sampler Wash Solution—Reagent Water

7.2.7 Electrode Storage Solution (50% v/v TISAB, 5 mg/L Fluoride) (100 mL)

7.2.7.1 Add 50 mL of stock TISAB (Section 7.2.4) and 0.5 mL of stock 1,000 mg/L fluoride calibrant (Section 7.3.1) to approximately 30 mL of reagent water in a 100-mL volumetric flask.

7.2.7.2 Add 50 µL of Brij-35.

7.2.7.3 Dilute to 100 mL with reagent water and mix well.

7.3 Calibrant Preparation

7.3.1 Stock 1,000 mg/L Fluoride Calibrant (500 mL)

7.3.1.1 Dissolve 1.1051 g of dry sodium fluoride in approximately 400 mL of reagent water in a 500-mL volumetric flask.

7.3.1.2 Dilute to 500 mL with reagent water and mix well.

Note: Store in a tightly sealed polyethylene container at 4°C. If stored properly, this solution is stable for 4–6 weeks.

7.3.2 Intermediate 100 mg/L Fluoride Calibrant (100 mL)

7.3.2.1 Use a volumetric pipet to add 10 mL of stock calibrant (Section 7.3.1) to approximately 80 mL of reagent water in a 100-mL volumetric flask.

7.3.2.2 Dilute to 100 mL with reagent water and mix well.

Note: Store in a tightly sealed polyethylene container. Prepare this solution fresh daily.

7.3.3 Intermediate 10 mg/L Fluoride Calibrant (100 mL)

7.3.3.1 Use a volumetric pipet to add 1 mL of stock calibrant (Section 7.3.1) to approximately 80 mL of reagent water in a 100-mL volumetric flask.

7.3.3.2 Dilute to 100 mL with reagent water and mix well.

Note: Store in a tightly sealed polyethylene container. Prepare this solution fresh daily.

7.3.4 Working Calibrants (100 mL)

7.3.4.1 Add the designated volumes of stock calibrant (see Equation 2) to the required number of 100-mL volumetric flasks that each contain approximately 80 mL of reagent water.

7.3.4.2 Dilute each solution to the mark with reagent water and mix well.

Note: Prepare working calibrants fresh daily.

EQUATION 2

$$C_1V_1 = C_2V_2$$

Where:
C_1 = Concentration (in mg/L) of stock solution (or calibrant)
V_1 = Volume (in L) of stock solution (or calibrant) to be used
C_2 = Desired concentration (in mg/L) of working calibrant to be prepared
V_2 = Final volume (in L) of working calibrant to be prepared

By solving this equation for the volume of stock solution to be used (V_1), the following equation is obtained:

$$V_1 = \frac{C_2V_2}{C_1}$$

Since the desired concentration (C_2), the final volume (V_2), and the concentration of the stock solution (C_1) are all known for any given calibrant concentration in a defined volume, the volume of stock solution to be used (V_1) is easily calculated.

7.3.4.3 Calibrants covering the entire range of this analysis can be prepared from the following table using 100-mL volumetric flasks.

Working Calibrant Final Concentration (mg/L)	Volume of Secondary Calibrant Solution (mL)		
	Fluoride 10 mg/L	Fluoride 100 mg/L	Fluoride 1,000 mg/L
0.20	2.0	0.2	0.02
0.50	5.0	0.5	0.05
1.0	10	1.0	0.1
2.0	20	2.0	0.2
4.0		4.0	0.4
8.0		8.0	0.8

8.0 Sample Collection, Preservation, and Storage

8.1 Samples should be collected in polyethylene bottles that have been thoroughly cleaned and rinsed with reagent water (Section 7.2.1).

8.2 The volume of sample collected should be sufficient to ensure that a representative sample is obtained, replicate analysis is possible, and waste disposal is minimized.

8.3 Store samples in tightly closed polyethylene containers and refrigerate at 4°C. Determine fluoride in unpreserved samples as soon as possible to eliminate loss of analyte. Special preservation techniques are not required.

8.4 Holding time for samples is 14 days from the time of collection (Reference 15.3).

9.0 Quality Control

Note: The following QC procedures are provided for reference purposes only and are not a substitute for any QC procedures that may be required for regulatory compliance.

9.1 It is recommended that each laboratory that uses this method operate a formal quality control program. The minimum requirements of such a program should consist of an initial demonstration of laboratory capability and the periodic analysis of Laboratory Control Samples (LCSs) and Matrix Spike/Matrix Spike Duplicates (MS/MSDs) as a continuing check on performance. Laboratory performance should be compared to established performance criteria to determine if the results of the analyses meet the performance characteristics of the method.

9.2 Method Detection Limit (MDL)—To establish the ability to detect fluoride at low levels, the analyst should determine the MDL using the apparatus, reagents, and calibrants that will be used in the practice of this method. An MDL less than or equal to the MDL listed in Section 1.2 should be achieved prior to practice of this method.

9.2.1 An MDL is calculated by analyzing a matrix spike at a concentration of two to three times the expected detection limit of the analyzer. Seven consecutive replicate analyses of this matrix spike should be analyzed, and the MDL should be calculated using Equation 3.

EQUATION 3

$$MDL = (t) \times (S)$$

Where:

t = Student's t value for a 99% confidence level and a standard deviation estimate with n–1 degrees of freedom (t = 3.14 for seven replicates)

S = Standard deviation of the replicate analyses

9.2.2 It is recommended that the MDL be calculated after every six months of operation, when a new operator begins work, or whenever there is any significant change in the instrument response.

9.3 Analyses of MS/MSD samples are required to demonstrate method accuracy and precision and to monitor matrix interferences (interferences caused by the sample matrix).

9.3.1 Matrix Spike/Matrix Spike Duplicate (MS/MSD)—The laboratory should spike, in duplicate, a minimum of 10% of all samples (one sample in duplicate in each batch of 10 samples) from a given sampling site.

9.3.2 The concentration of the spike in the sample shall be determined as follows:

9.3.2.1 If, as in compliance monitoring, the concentration of fluoride in the sample is being checked against a regulatory concentration limit, the spiking level shall be at that limit.

9.3.2.2 If the concentration of fluoride in a sample is not being checked against a limit, the spike shall be at the concentration of the LCS or at least four times greater than the MDL.

9.4 Analyses of Laboratory Reagent Blanks (LRBs) are required to demonstrate freedom from contamination and that the compounds of interest and interfering compounds have not been carried over from a previous analysis.

9.5 As part of the QC program for the laboratory, method precision and accuracy for samples should be assessed and records should be maintained.

9.5.1 An LCS should be analyzed with every sample batch, and the mean (m) and the standard deviation (S) should be recorded. After multiple analyses, the mean should be plotted with limits of $m+2S$ and $m–2S$. The mean and the limits should be recalculated after every 5–10 new measurements.

9.5.2 If the LCS measurement falls outside the range calculated in Section 9.5.1, then the problem should be addressed, and that sample batch should be reanalyzed if necessary.

9.6 Reference Sample—To demonstrate that the analytical system is in control, the laboratory may wish to periodically test an external reference sample, such as a Standard Reference Material (SRM) available from the National Institute of Standards and Technology (NIST). Corrective action should be taken if the measured concentration significantly differs from the stated concentration.

10.0 Configuration and Start-up

10.1 Instrument Configuration

10.1.1 Configure the OI Analytical Flow Solution 3000 Analyzer according to the Operator's Manual and verify that each module is properly powered on.

10.1.2 Verify that the Fluoride by ISE Cartridge (Part #A002832) is configured as illustrated in the flow diagram shown in Section 17.0.

10.1.3 Connect the appropriate pump tubes to the cartridge and to their appropriate reagent containers according to the flow diagram.

10.2 Instrument Stabilization

10.2.1 Connect the reagent pump tubes to a reagent bottle containing the start-up solution (Section 7.2.2). Start the pump at low speed, allowing the start-up solution to flow through the entire system.

10.2.2 Verify that the flowcell of each detector is purged of all bubbles and that the flow is stable and free from surging before proceeding.

10.3 Baseline Verification

10.3.1 Create and save a Method in WinFLOW. Refer to the WinFLOW Operator's Manual (Reference 15.5) for help on creating a Method.

10.3.2 Create and save a Sample Table in WinFLOW that will be used to generate a calibration curve using at least three calibrants that cover the full range of expected concentrations in the samples to be analyzed. This Sample Table should also be used to analyze all necessary QC samples as well as the analytical batch of samples to be analyzed. For help on creating a Sample Table, refer to the WinFLOW Operator's Manual (Reference 15.5).

10.3.3 Select **Collect Data** in the WinFLOW main window, enter the user's identification, select the appropriate Method and Sample Table, and begin to collect baseline data. Very sharp fluctuations in the baseline and/or consistent drifting are typically signs of bubbles in the flowcell. The flowcell must be free of bubbles prior to beginning analysis.

10.4 Calibration and Standardization

10.4.1 Prepare a series of at least three working calibrants using the stock solutions (Section 7.3) according to Equation 2, covering the desired analysis range.

10.4.2 Place the calibrants in the autosampler in order of increasing concentration. Each calibrant should be analyzed according to the analytical procedures in Section 11.0. A calibration curve will be calculated by the WinFLOW software.

10.4.3 Acceptance or control limits for the calibration results should be established using the difference between the measured value of each calibrant and the corresponding "true" concentration.

10.4.4 Each calibration curve should be verified by analysis of a Laboratory Control Sample (LCS, Section 9.5). Using WinFLOW software, calibration, verification, and sample analysis may be performed in one continuous analysis.

11.0 Procedure

11.1 Analysis

11.1.1 Remove the rubber cap covering the fluoride electrode tip. Configure the cartridge according to the flow diagram (Section 17.0).

11.1.2 Insert the fluoride electrode.

11.1.2.1 The seal is made by the O-ring on the locking collar. The collar should be loosened a few turns when the electrode is inserted. Very gentle pressure may be necessary to push the electrode past the O-ring.

11.1.2.2 Locate the exit ports of the flowcell. Align the bottom of the electrode with the tops of the exit ports.

11.1.2.3 Once the electrode is in place, gently tighten the collar by hand. Since some fluid may spill during this step, surround the flowcell with absorbent material and clean any fluid that remains outside the flowcell.

11.1.2.4 Monitor the flowcell briefly, ensuring that the seal is intact and leak-free.

11.1.2.5 Connect the fluoride electrode connector to the BNC plug on the detector chassis.

11.1.3 After the baseline has been verified according to Section 10.3, place all reagents on-line and allow to pump at least 15–30 minutes. Verify there are no bubbles in the flowcell or in the exit tubes from the flowcell (Section 11.2.1). Autozero the baseline before beginning the analysis.

11.1.4 Load the sampler tray with calibrants, blanks, samples, and QC samples.

Note: The matrix of the working standards, blanks, and QC samples should match that of the samples being analyzed.

11.1.5 Using the Method and Sample Table created for the analytical batch to be analyzed and with the baseline verified to be stable, begin the analysis by selecting the "Fast Forward" button on the left side of the Data Analysis window in WinFLOW. This will initiate the sequential analysis of samples as defined in the Sample Table.

11.1.6 When the analysis is complete, stop the pump, release the tension on all pump tubes, and power off the system. See Section 11.2.5 for detailed information on electrode storage.

11.2 Operating Notes

11.2.1 Remove all bubbles from the flowcell and from both exit tubes. Block the flow from one side until all of the bubbles are removed from the other exit tube. Repeat as necessary.

11.2.2 Be sure to select ISE in the Method editor. The response will follow a second or third order equation.

11.2.3 Allow the system to stabilize for approximately 15–30 minutes before beginning the analysis. Stabilize the reference electrode by several injections of high concentration calibrants.

11.2.4 At the beginning of the analysis, check the detector offset. Open the Serial Communications window during the "Baseline View" mode. Set the offset to 50% ±5% by turning the adjustment knob on the front panel of the detector.

11.2.5 Proper storage of the electrode after the analysis is complete depends upon the amount of expected downtime.

11.2.5.1 For overnight storage, store the electrode in the flowcell without flushing the cartridge. The electrode will remain submerged in working TISAB (Section 7.2.5), which is adequate for overnight storage.

11.2.5.2 For short-term storage (2–10 days), remove the electrode from the flowcell and store it in the electrode storage solution (Section 7.2.7). Flush the cartridge with reagent water for 15–30 minutes.

11.2.5.3 For long-term storage (>10 days), Flush the cartridge, flowcell, and electrode with reagent water for 15–30 minutes. Remove the electrode from the flowcell and gently dry it with a nonabrasive material. Store the electrode dry with the plastic cover reinstalled to protect the electrode face.

11.2.6 Clean the cartridge and the flowcell periodically (approximately every 1–2 weeks) with Kleenflow Basic.

11.2.6.1 Remove the electrode from the flowcell. Disconnect the tubing from the bottom of the flowcell.

Caution: Never allow Kleenflow Basic (or any other strong base or acid) to contact the electrode surface. The electrode may become fouled and its usable lifetime will be reduced.

11.2.6.2 Pump Kleenflow Basic through the cartridge and the debubbler for 15–30 minutes. Pump reagent water through the system to remove the Kleenflow Basic.

11.2.6.3 Clean the flowcell by submerging it in Kleenflow Basic for 15–30 minutes. Rinse the flowcell thoroughly with reagent water. Wipe the inner surface of the flowcell with a lint-free material.

11.2.7 If sensitivity deteriorates, clean the electrode face with liquid soap and water to remove organic deposits. Alternatively, use fluoride toothpaste on a soft towel to clean the electrode. In both cases, rinse well with reagent water and soak for several hours in electrode storage solution (Section 7.2.7).

11.2.8 Run blanks ("read baseline" samples) about every 10 samples to compensate for drift inherent in the assay.

11.2.9 The fluoride electrode used in this method is a sealed reference type and is rated for six months of use. The actual lifetime may differ depending upon usage and storage. Refer to the electrode manual for details.

12.0 Data Analysis and Calculations

12.1 The calibration curve allows for accurate quantitation of the concentration in each sample.

12.2 WinFLOW software reports the concentration of each sample relative to the calibration curve.

13.0 Method Performance

Range:	0.20–8.0 mg/L
Throughput:	24 samples/hour
Precision:	
0.20 mg/L	<5% RSD
1.0 mg/L	<2% RSD
8.0 mg/L	<1% RSD
Method Detection Limit (MDL):	0.02 mg/L

14.0 Pollution Prevention and Waste Management

14.1 It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land-disposal restrictions. In addition, it is the laboratory's responsibility to protect air, water, and land resources by minimizing and controlling all releases from fume hoods and bench operations. Also, compliance is required with any sewage discharge permits and regulations.

14.2 For further information on waste management, consult Section 13.6 of *Less is Better: Laboratory Chemical Management for Waste Reduction* (Reference 15.1).

15.0 References

15.1 *Less is Better: Laboratory Chemical Management for Waste Reduction.* Available from the American Chemical Society, Department of Government Regulations and Science Policy, 1155 16th Street, NW, Washington, DC, 20036.

15.2 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.

15.3 Sample Preservation. *Methods for Chemical Analysis of Water and Wastes*; EPA-600/4-79-020; U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory: Cincinnati, OH, 1984; xvii.

15.4 *Standard Methods for the Examination of Water and Wastewater*, 20th ed.; American Public Health Association: Washington, D.C., 1998.

15.5 WinFLOW Software and Operator's Manual (Part #A002877). Available from OI Analytical, P.O. Box 9010, College Station, TX, 77842-9010.

16.0 Glossary of Definitions and Purposes

The definitions and purposes are specific to this method but have been conformed to common usage as much as possible.

16.1 Units of weights and measures and their abbreviations

16.1.1 Symbols

°C	degrees Celsius
%	percent
±	plus or minus
≥	greater than or equal to
≤	less than or equal to

16.1.2 Alphabetical characters

g	gram
L	liter
mg	milligram
mg/L	milligram per liter
µg	microgram
µg/L	microgram per liter
mL	milliliter

ppm	parts per million
ppb	parts per billion
M	molar solution
N	normal solution

16.2 Definitions

16.2.1 Laboratory Control Sample (LCS)—An aliquot of LRB to which a quantity of the analyte of interest is added in the laboratory. The LCS is analyzed like a sample. Its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

16.2.2 Laboratory Reagent Blank (LRB)—An aliquot of reagent water and other blank matrix that is treated like a sample, including exposure to all glassware, equipment, and reagents that are used with other samples. The LRB is used to determine if the method analyte or other interferences are present in the laboratory environment, reagents, or apparatus.

16.2.3 Matrix Spike/Matrix Spike Duplicate (MS/MSD)—An aliquot of an environmental sample to which a quantity of the method analyte is added in the laboratory. The MS/MSD is analyzed like a sample. Its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentration of the analyte in the sample matrix must be determined in a separate aliquot, and the measured values in the MS/MSD must be corrected for the background concentration.

16.2.4 Method Detection Limit (MDL)—The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.

17.0 Figures

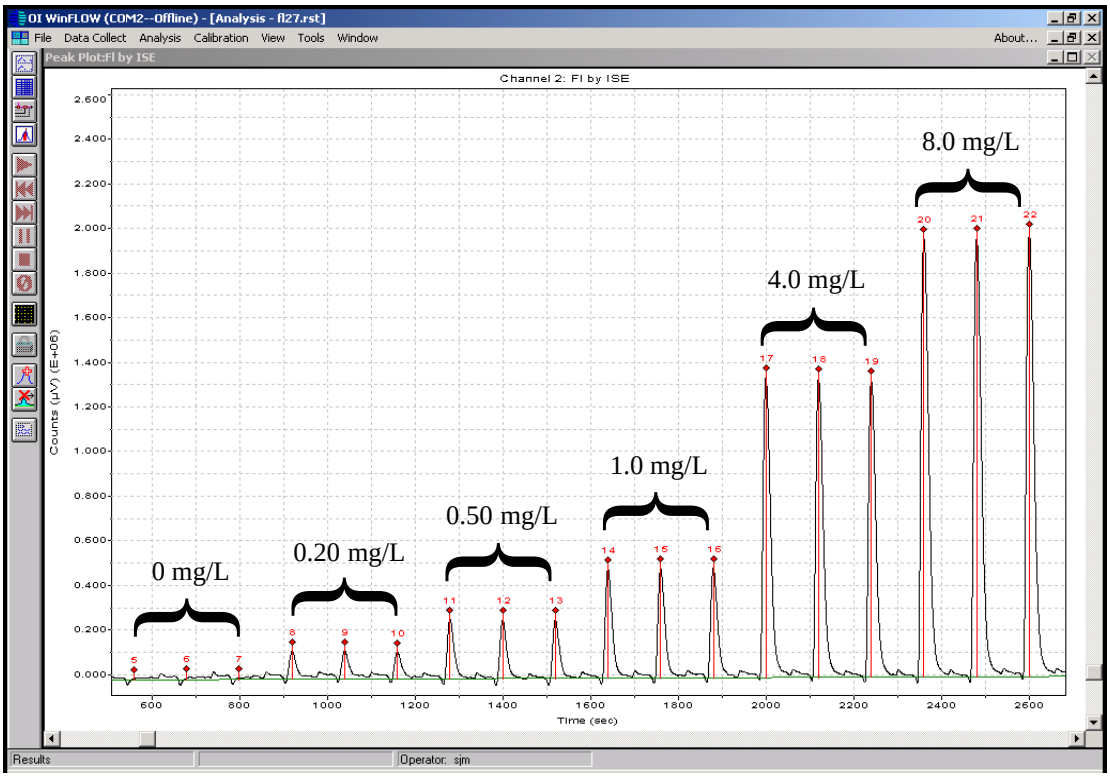


Figure 1. Calibration (0.20–8.0 mg/L)

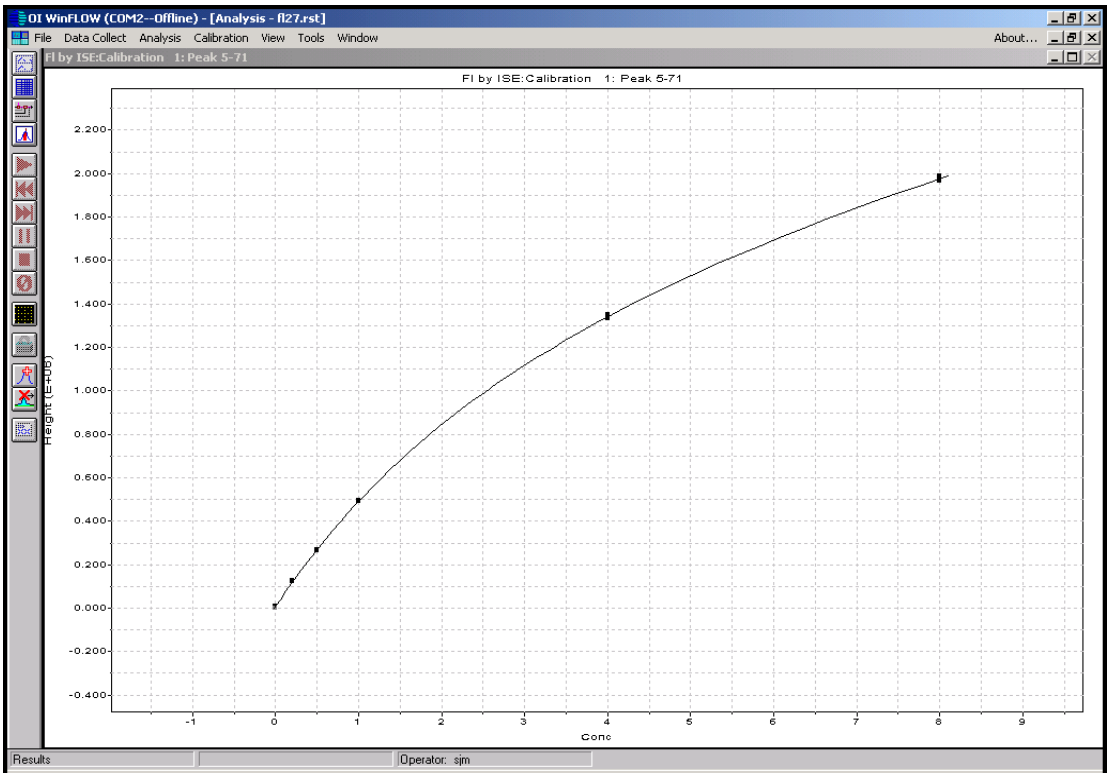


Figure 2. Calibration Curve (0.20–8.0 mg/L)

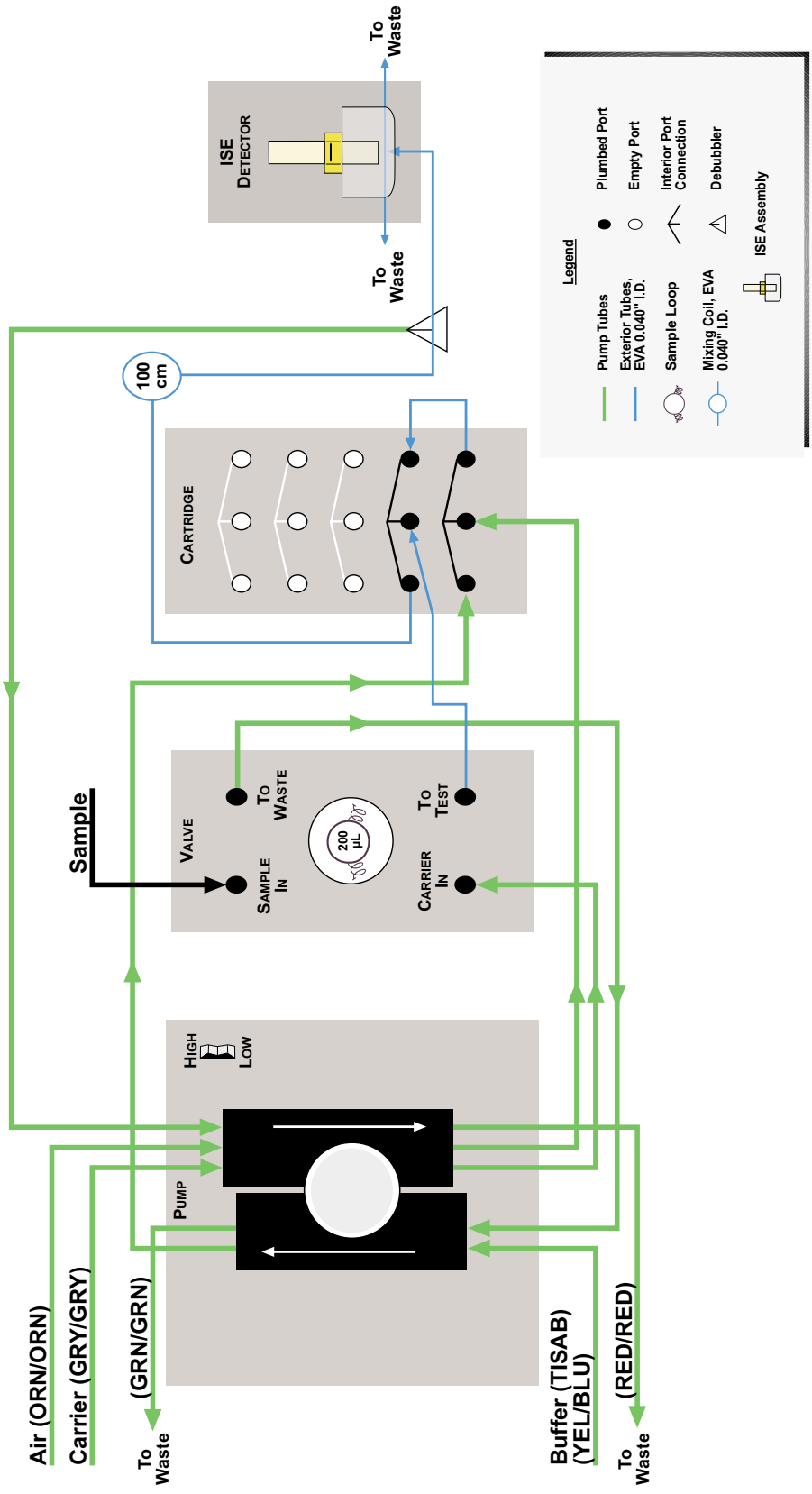


Figure 3. Detailed Flow Diagram for Fluoride by ISE and FIA/SFA on a Flow Solution 3000, Cartridge Part #A002832

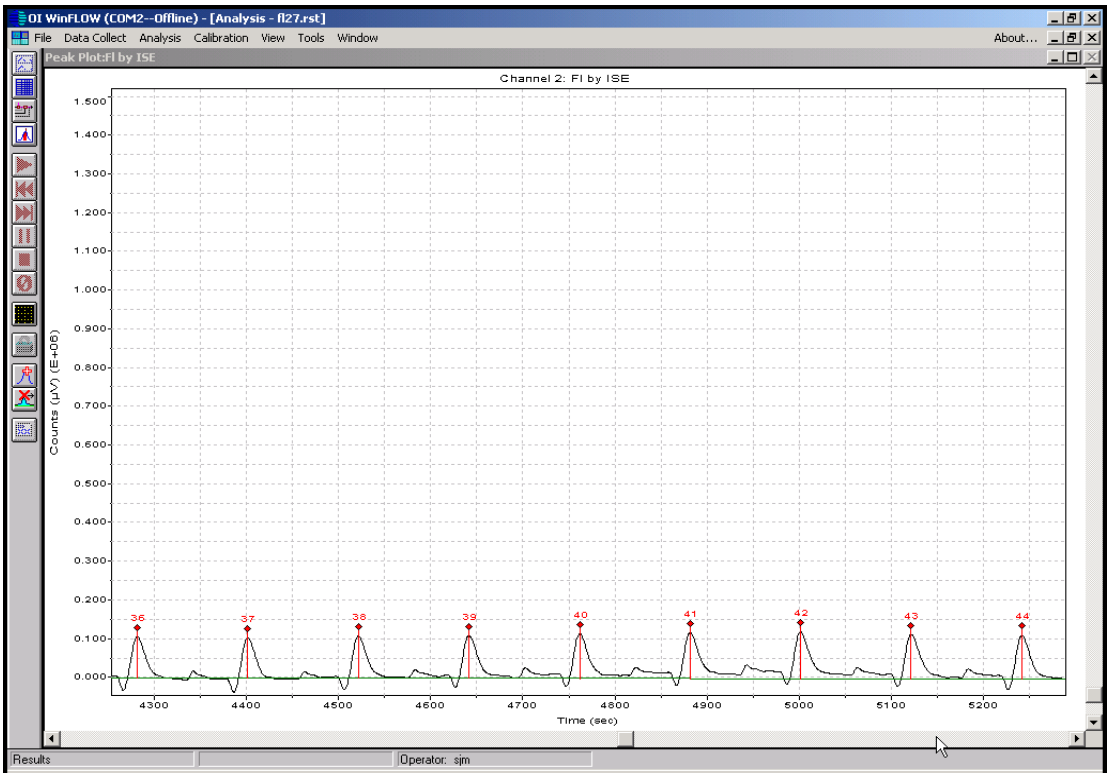


Figure 4. Precision at 0.20 mg/L (<5% RSD)

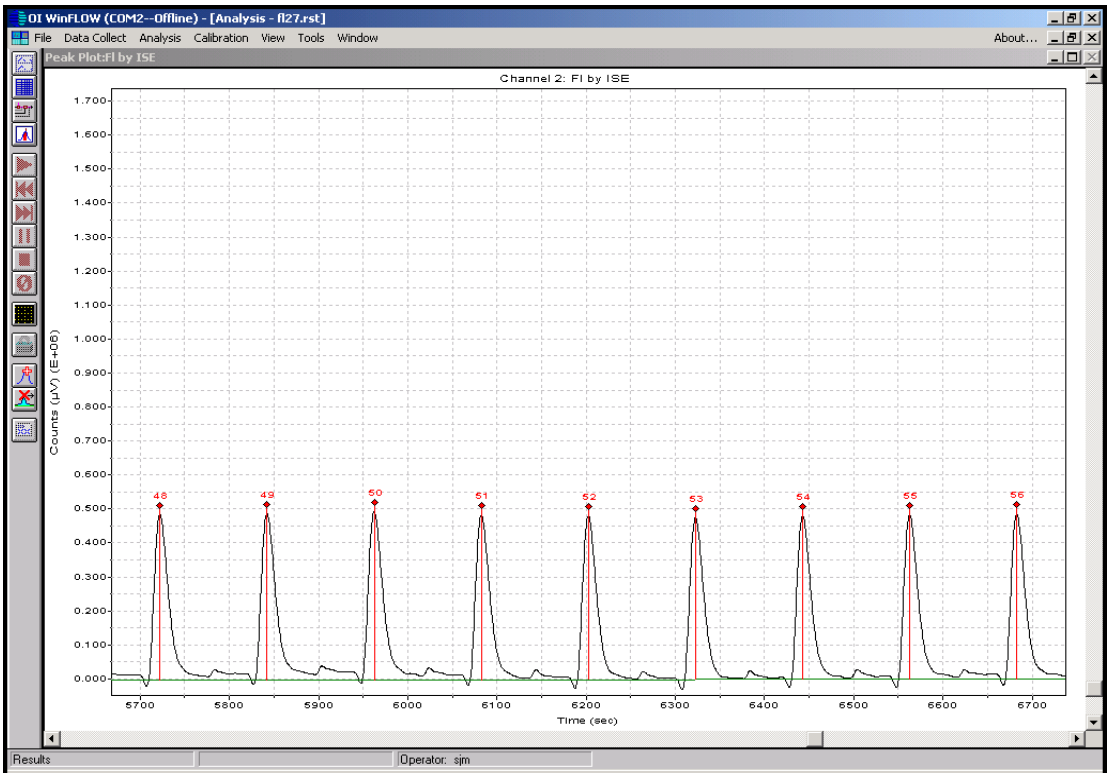


Figure 5. Precision at 1.0 mg/L (<2% RSD)

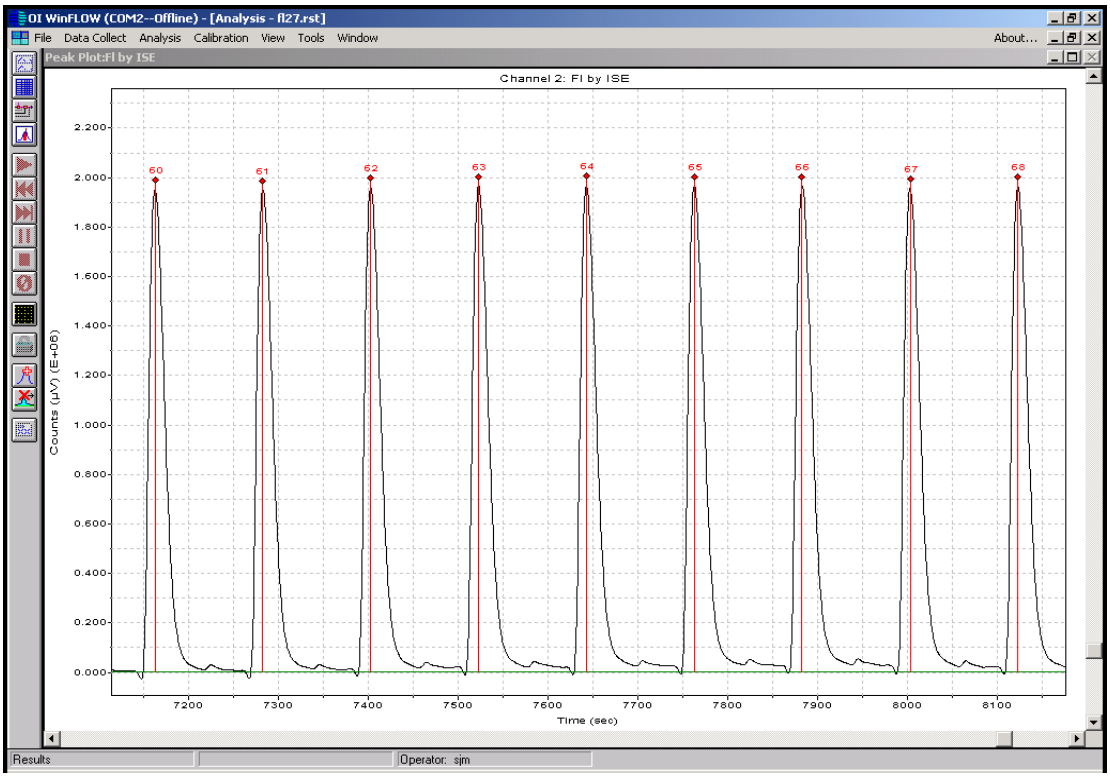


Figure 6. Precision at 8.0 mg/L (<1% RSD)

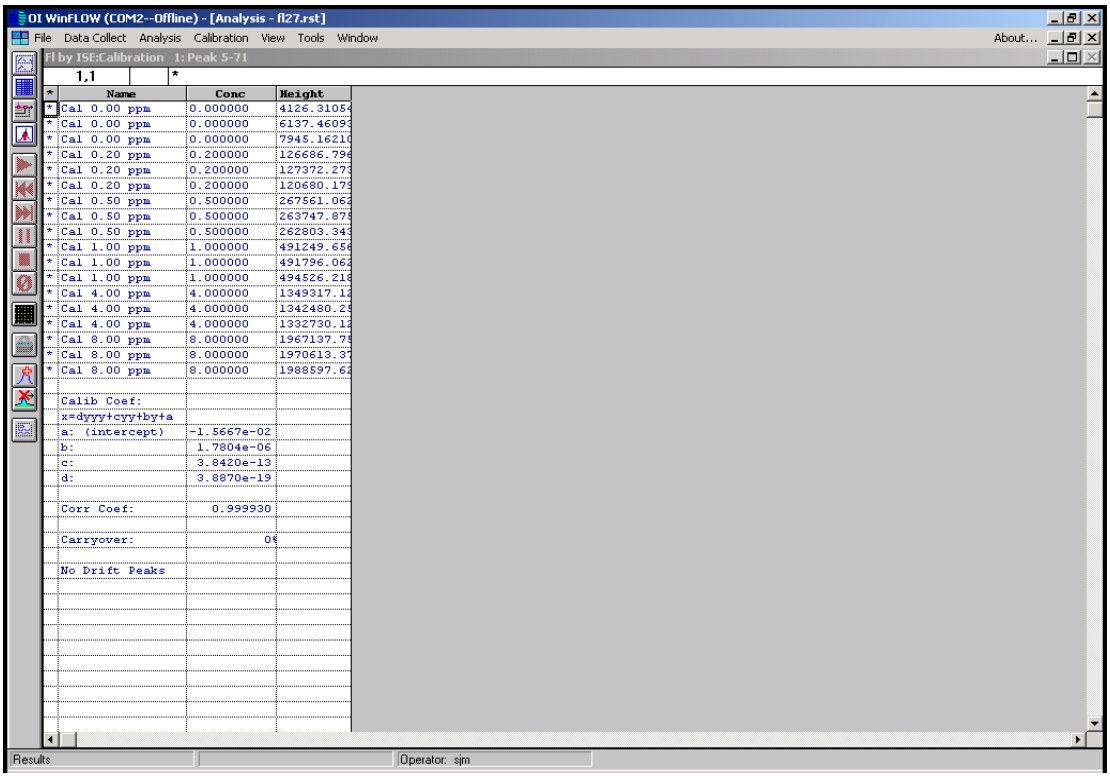


Figure 7. Calibration Results (0.20–8.0 mg/L)

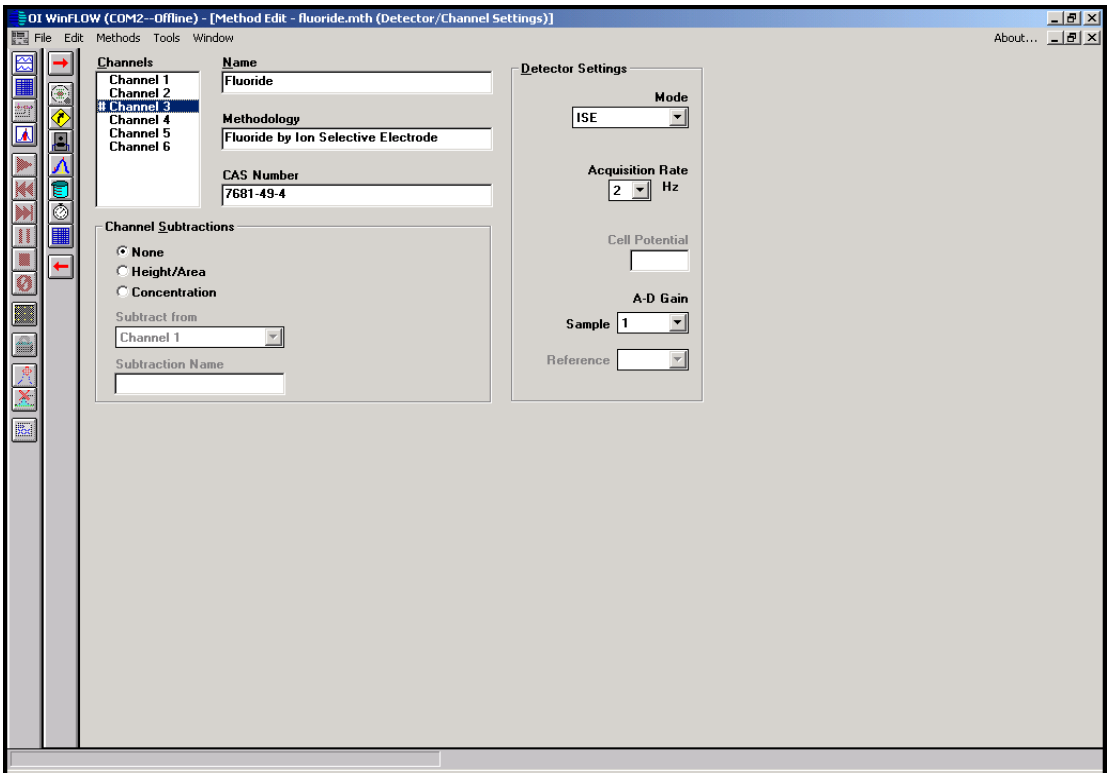


Figure 8. WinFLOW Method Editor—Detector/Channel Settings

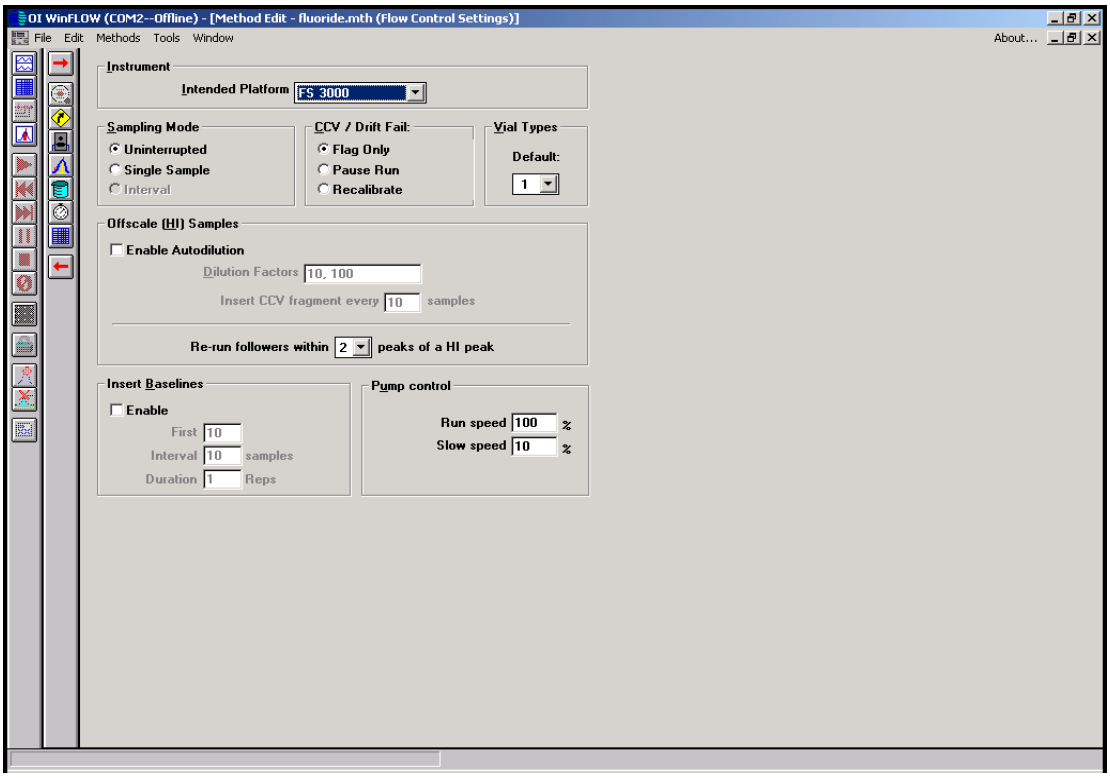


Figure 9. WinFLOW Method Editor—Flow Control Settings

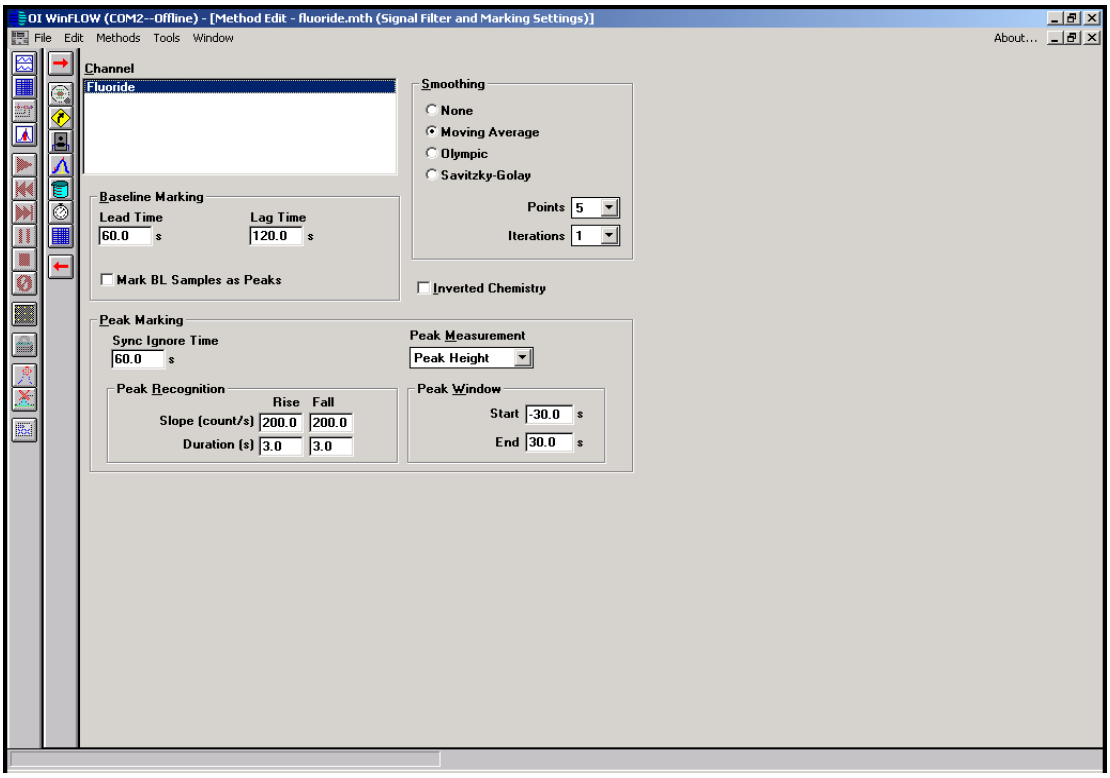


Figure 10. WinFLOW Method Editor—Signal Filter and Marking Settings

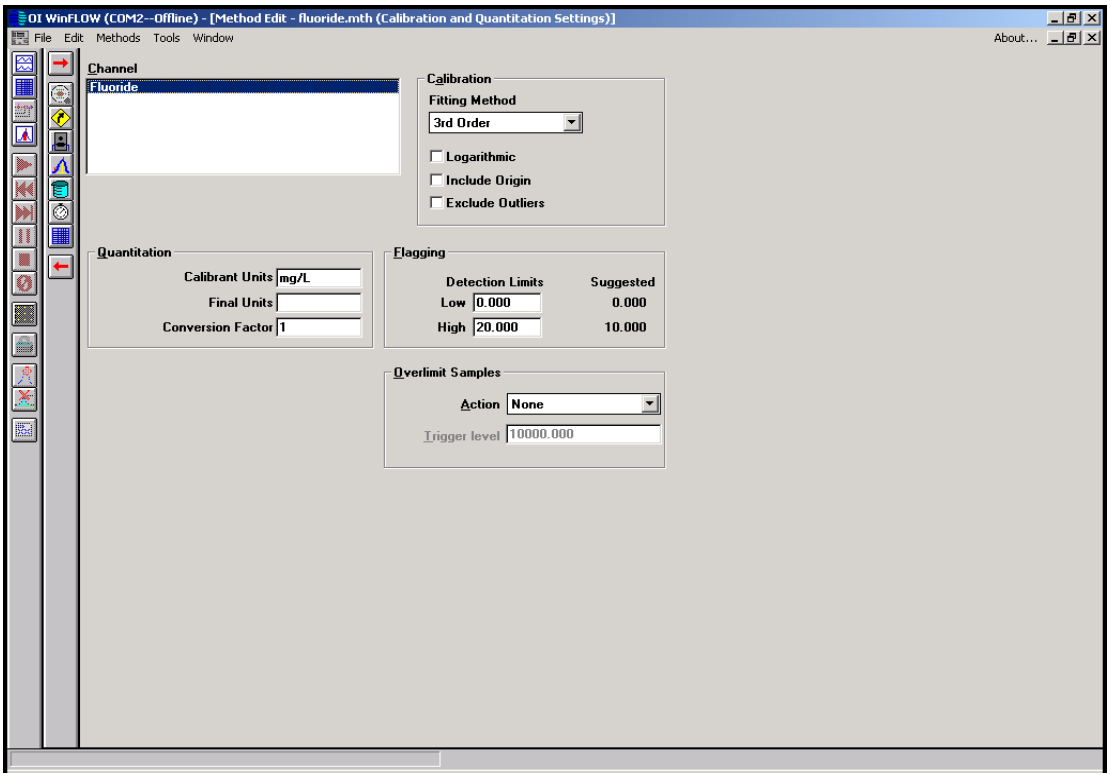


Figure 11. WinFLOW Method Editor—Calibration and Quantitation Settings

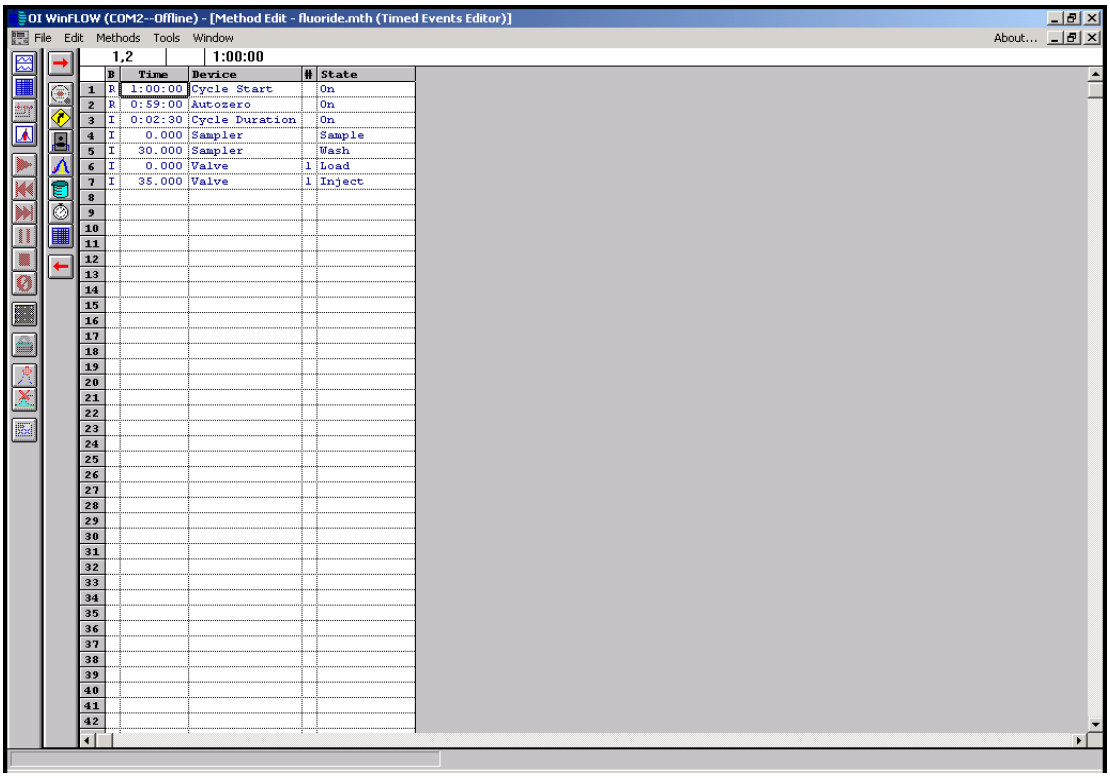


Figure 12. WinFLOW Method Editor—Timed Events Editor

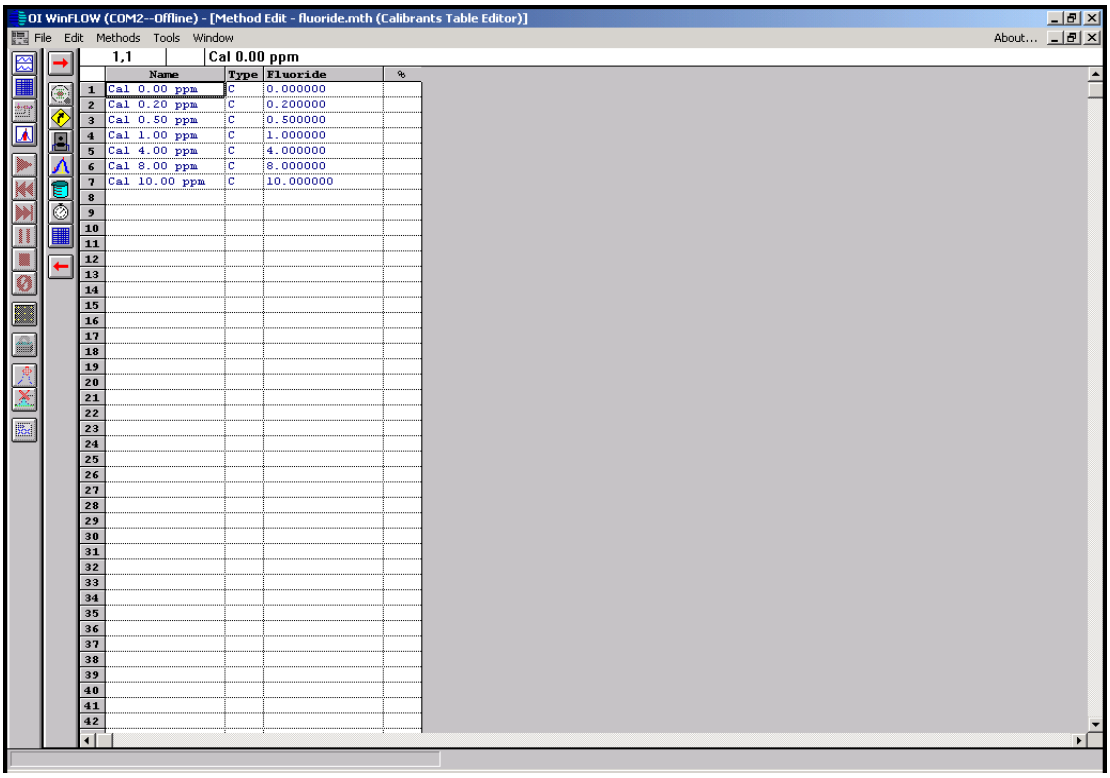


Figure 13. WinFLOW Method Editor—Calibrants Table Editor

Results were obtained under optimal operating conditions. Actual results may vary depending on sample introduction, cleanliness of sample containers, reagent purity, operator skill, and maintenance of instruments.

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