1.0 Scope and Application

1.1 This method is used for the determination of bromide in natural water and wastewater.

1.2 The Method Detection Limit (MDL) of this method is 0.020 mg/L bromide. The applicable ranges of the method are 0.20–50 mg/L bromide using a 200–µL sample loop, 0.10–10 mg/L using a 200–µL sample loop, and 0.50–100 mg/L using a 30–µL sample loop. The range may be extended to analyze higher concentrations by sample dilution or injection of different sample volumes.

2.0 Summary of Method

2.1 The sample, buffered to pH 5.6, reacts with chloramine-T trihydrate to oxidize bromide to hypobromous acid. Hypobromous acid reacts with fluorescein to form pink eosin (tetrabromofluorescein). The absorbance is measured at 520 nm (References 15.1–15.4, 15.7–15.9).

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

2.3 A general flow diagram of the FIA 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 Iodine interferes quantitatively. In most water samples, iodine concentration is negligible. For best results, the iodine concentration can be determined separately and subtracted from the apparent bromide concentration determined by this method.

4.2 The presence of less than 0.50 mg/L cyanide or less than 500 mg/L chloride does not interfere. Reduce chloride interference by adding sodium thiosulfate.

4.3 Thiocyanate interferes linearly.

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, CH$_3$COOH (FW 60.05)

5.3.2 Ammonium Chloride, NH$_4$Cl (FW 53.49)

5.3.3 Chloramine-T Trihydrate, CH$_3$C$_6$H$_4$SO$_2$NNaCl$_3$H$_2$O (FW 281.69)

5.3.4 Fluorescein, C$_{20}$H$_{10}$O$_5$Na$_2$ (FW 376.28)

5.3.5 Kleenflow™ Basic (Part #A002294)

5.3.6 Nitric Acid, concentrated, HNO$_3$ (FW 63.01)

5.3.7 Potassium Bromide, KBr (FW 119.01)

5.3.8 Potassium Hydroxide, KOH (FW 56.11)

5.3.9 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) System (OI Analytical Flow Solution® 3000) consisting of the following:

6.1.1 120-Place Autosampler

6.1.3 Expanded Range (ER) Photometric Detector with 5-mm path length flowcell and 520-nm optical filter

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

6.1.5 Bromide Cartridge (Part #A002763)

6.2 Sampling equipment—Sample bottle, amber glass, with polytetrafluoroethylene (PTFE)-lined cap. Clean by washing with detergent and water, rinsing with two aliquots of reagent water, and drying by baking at 110°–150°C for a minimum of one hour.

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, CH₃COOH (FW 60.05)

7.1.2 Ammonium Chloride, NH₄Cl (FW 53.49)

7.1.3 Chloramine-T Trihydrate, CH₃C₆H₄SO₂NNaCl³H₂O (FW 281.69)

7.1.4 Deionized Water (ASTM Type I or II)

7.1.5 Fluorescein, C₂₀H₁₀O₅Na₂ (FW 376.28)

7.1.6 Kleenflow Basic (Part #A002294)
7.1.7 Nitric Acid, concentrated, HNO₃ (FW 63.01)

7.1.8 Potassium Bromide, KBr (FW 119.01)

7.1.9 Potassium Hydroxide, KOH (FW 56.11)

7.1.10 Sodium Hydroxide, NaOH (FW 40.00)

7.2 Reagent Preparation

Note: For best results, filter and degas all reagents prior to use.

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 glass frit for approximately 5 minutes.

7.2.1.1.3 Boil distilled/deionized water in an Erlenmeyer flask for 15–20 minutes. Remove the flask from the heat source, cover it with an inverted beaker, and allow it to cool to room temperature.

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, Carrier, and Sampler Wash Solution—Reagent Water

7.2.3 5 N Potassium Hydroxide (1 L)

7.2.3.1 While stirring, carefully add 280 g of potassium hydroxide to approximately 400 mL of reagent water in a 1-L volumetric flask.

7.2.3.2 Allow the solution to cool to room temperature. Dilute to 1,000 mL with reagent water and mix well.

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

7.2.4 Ammonium Chloride Buffer, pH 5.6 (1 L)

7.2.4.1 Dissolve 9.426 g of ammonium chloride in approximately 500 mL of reagent water in a 1-L beaker.
7.2.4.2 While stirring, carefully add 57 mL of glacial acetic acid.

7.2.4.3 Adjust the pH to 5.6 by adding approximately 150 mL of 5 N potassium hydroxide (Section 7.2.3).

7.2.4.4 Quantitatively transfer the solution to a 1-L volumetric flask. Dilute to 1,000 mL with reagent water and mix well.

**Warning:** Mixing acetic acid and water produces a great amount of heat. Take appropriate precautions.

**Note:** This solution is stable for 4–6 weeks if stored at 4°C.

7.2.5 Chloramine-T Reagent (100 mL)

7.2.5.1 Dissolve 0.5 g of chloramine-T trihydrate in 80 mL of reagent water in a 100-mL volumetric flask.

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

**Note:** Prepare this solution fresh weekly.

7.2.6 0.1 N Sodium Hydroxide (1 L)

7.2.6.1 While stirring, carefully add 4 g of sodium hydroxide to approximately 700 mL of reagent water in a 1-L volumetric flask.

7.2.6.2 Dilute to 1,000 mL with reagent water and mix well.

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

7.2.7 Stock Fluorescein Reagent (100 mL)

7.2.7.1 Dissolve 0.125 g of fluorescein in 25 mL of 0.1 N sodium hydroxide (Section 7.2.6) in a 100-mL volumetric flask.

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

**Note:** Store in an amber bottle. This solution is stable for 4–6 weeks if stored at 4°C.

7.2.8 Working Fluorescein Reagent (100 mL)

7.2.8.1 Add 5 mL of stock fluorescein reagent (Section 7.2.7) to approximately 80 mL of reagent water in a 100-mL volumetric flask.

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

**Note:** Prepare this solution fresh daily.
7.2.9 0.1 N Nitric Acid (1 L)

7.2.9.1 While stirring, carefully add 6.25 mL of concentrated nitric acid to approximately 800 mL of reagent water in a 1-L volumetric flask.

7.2.9.2 Dilute to 1,000 mL with reagent water and mix well.

**Warning:** Mixing nitric acid and water produces a great amount of heat. Take appropriate precautions.

7.3 Calibrant Preparation

7.3.1 Stock Calibrant 1,000 mg/L Bromide (1 L)

7.3.1.1 Dissolve 1.489 g of potassium bromide (dried overnight) in approximately 800 mL of reagent water in a 1-L volumetric flask.

7.3.1.2 Dilute to 1,000 mL with reagent water and mix well.

**Note:** This solution is stable for 4–6 weeks if stored at 4°C.

7.3.2 Intermediate Calibrant 100 mg/L Bromide (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:** Prepare the intermediate calibrant fresh daily.

7.3.3 Working Calibrants (100 mL)

7.3.3.1 Add the designated volumes of stock calibrant (see Equation 1) to the required number of 100-mL volumetric flasks that each contain approximately 80 mL of reagent water.

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

**Note:** Prepare working calibrants fresh daily.
EQUATION 1

\[ C_1 V_1 = C_2 V_2 \]

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:

\[ V_1 = \frac{C_2 V_2}{C_1} \]

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.

### 7.3.3.3 Calibrants covering the entire range of this analysis can be prepared from the following tables.

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<table>
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<td>1,000</td>
<td>100</td>
</tr>
</tbody>
</table>

### 8.0 Sample Collection, Preservation, and Storage

8.1 Samples should be collected in plastic or glass 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 Determine bromide in unpreserved samples as soon as possible to eliminate loss of analyte.

8.4 No preservation is required. Unpreserved samples may be held for a maximum of 28 days from the time of collection (Reference 15.6).

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 bromide 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 2.

\[
\text{MDL} = (t) \times (S)
\]

*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 bromide 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 bromide 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 Bromide Cartridge (Part #A002763) 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 15.10) 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 15.10).

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 1, 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 Begin pump flow with the start-up solution (Section 7.2.2). Verify a stable baseline (Section 10.3).

11.1.2 After the baseline has been verified according to Section 10.3, place all reagents on-line and allow to pump at least 10–15 minutes. Verify there are no bubbles in the flowcell. Obtain a stable baseline at 520 nm and autozero the baseline before beginning analysis.

11.1.3 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.

11.1.4 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.

11.1.5 When analysis is complete, pump start-up solution through the system for at least 10–15 minutes. Stop the pump, release the tension on all pump tubes, and power off the system.

11.2 Operating Notes

11.2.1 Flush the system with Kleenflow Basic after every 1–2 days of operating this method.

11.2.2 For best results, all glassware should be cleaned with 0.1 N nitric acid (Section 7.2.9).

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

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<th>Range:</th>
<th>0.20–50 mg/L*</th>
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<tr>
<td>Throughput:</td>
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</tr>
<tr>
<td>Precision:</td>
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</tr>
<tr>
<td>0.20 mg/L</td>
<td>&lt;2% RSD</td>
</tr>
<tr>
<td>0.50 mg/L</td>
<td>&lt;2% RSD</td>
</tr>
<tr>
<td>10 mg/L</td>
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<tr>
<td>50 mg/L</td>
<td>&lt;1% RSD</td>
</tr>
<tr>
<td>Method Detection Limit (MDL):</td>
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</tbody>
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*Use a 200-µL sample loop.

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<tr>
<th>Range:</th>
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<td>Precision:</td>
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<td>0.10 mg/L</td>
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<tr>
<td>Method Detection Limit (MDL):</td>
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**Use a 200-µL sample loop.

<table>
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<th>Range:</th>
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<td>Throughput:</td>
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<td>0.50 mg/L</td>
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</table>

†The legs of the injection valve should be connected together with no additional loop for a 30-µL sample volume.

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 15.5).
15.0 References


15.5 *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.


15.10 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

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<th>Symbol</th>
<th>Definition</th>
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<td>°C</td>
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<td>±</td>
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16.1.2 Alphabetical characters

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17.0 Figures

Figure 1. Calibration (0.20–50 mg/L), 200-µL Sample Loop

Figure 2. Calibration Curve (0.20–50 mg/L), Second Order
Figure 3. Calibration (0.10–10 mg/L), 200-μL Sample Loop

Figure 4. Calibration Curve (0.10–10 mg/L), Second Order
Figure 5. Calibration (0.50–100 mg/L), 30-µL Sample Loop

Figure 6. Calibration Curve (0.50–100 mg/L), Third Order
Figure 7. Detailed Flow Diagram for Bromide by FIA on a Flow Solution 3000, Cartridge Part #A002763
Figure 8. Precision at 0.10 mg/L, 200-µL Sample Loop (<4% RSD)

Figure 9. Precision at 0.20 mg/L, 200-µL Sample Loop (<2% RSD)
Figure 10. Precision at 0.50 mg/L, 30-µL Sample Loop (<2% RSD)

Figure 11. Precision at 10 mg/L, 200-µL Sample Loop (<1% RSD)
Figure 12. Precision at 100 mg/L, 30-µL Sample Loop (<1% RSD)

Figure 13. Calibration Results (0.20–50 mg/L), 200-µL Sample Loop
### Figure 14. Calibration Results (0.10–10 mg/L), 200-µL Sample Loop

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</tr>
</tbody>
</table>

Calibration:

- Linear fit: $y = 0.9999x + 0.001$
- Coefficient of determination: $R^2 = 0.9999$
- No drift peaks

### Figure 15. Calibration Results (0.50–100 mg/L), 30-µL Sample Loop

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<td>CAL 10</td>
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</tr>
</tbody>
</table>

Calibration:

- Linear fit: $y = 0.9999x + 0.001$
- Coefficient of determination: $R^2 = 0.9999$
- No drift peaks
Figure 16. WinFLOW Method Editor—Detector/Channel Settings

Figure 17. WinFLOW Method Editor—Flow Control Settings
Figure 18. WinFLOW Method Editor—Signal Filter and Marking Settings

Figure 19. WinFLOW Method Editor—Calibration and Quantitation Settings
Figure 20. WinFLOW Method Editor—Timed Events Editor

Figure 21. 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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