

# *Data Pack*

## *Ion Chromatography Methods*

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*June 2009*

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# Introduction

This Datapack contains application information on several Lachat methods utilizing Ion Chromatography. The Lachat QuikChem 8500 Series analyzer is the only analytical instrument to offer an Ion Chromatography (IC) option to complement its Flow Injection Analysis (FIA) capability.

These technologies are complementary analytical techniques. FIA is used for high-productivity applications, while IC is used to profile samples for their component ions. Both techniques play important roles in the environmental testing laboratory.

By incorporating both technologies into a single instrument, the following benefits are achieved:

- Using a unique, patented software algorithm called the Shared Peripheral System (SPS), simultaneous and independent operation of FIA and IC is possible on the same instrument platform.
- Shared use of several peripherals including sampler, dilutor, pump, electronics unit and data station.
- Uniform operating protocols
- Reduced footprint
- Unified training, service and support

The QuikChem IC+ Option is available with the original system purchase, integrated at the factory, or as a simple field upgrade to an existing QuikChem 8500 series instrument.

With the availability of FIA and IC on a single instrument, Lachat Instruments offers the most comprehensive ion analysis solution available today.

Please check with your local Lachat representative for updates on new IC methods.

For technical assistance, price information, and ordering, please contact Lachat Instruments:

**In the US:** (800) 247-7613

**International:** (970) 669-3050

**On the Web:** [www.lachatinstruments.com](http://www.lachatinstruments.com)

**E-mail for Support:** [support@lachatinstruments.com](mailto:support@lachatinstruments.com)

**E-mail for Sales:** [sales@lachatinstruments.com](mailto:sales@lachatinstruments.com)

Method No	Range	MDL	Matrix	Comments	Rev Date
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## Lachat Ion Chromatography Methods

*As of June 2009*

### Anions

<b>10-510-00-1-A1 #</b>			Waters and water extracts of soil	USEPA method 300.0 (A), 100 µL loop, Run time: 11 min.	29-Nov-01
Bromide	0.05 – 5.0	0.018	mg Br <sup>-</sup> /L		
Chloride	0.5 – 50.0	0.004	mg Cl <sup>-</sup> /L		
Fluoride	0.05 – 5.0	0.004	mg F <sup>-</sup> /L		
Nitrate	0.05 – 5.0	0.004	mg NO <sub>3</sub> <sup>-</sup> - N/L		
Nitrite	0.05 – 5.0	0.008	mg NO <sub>2</sub> <sup>-</sup> - N/L		
Phosphate, ortho	0.05 – 5.0	0.012	mg HPO <sub>4</sub> <sup>2-</sup> - P/L		
Sulfate	1.0 – 100	0.012	mg SO <sub>4</sub> <sup>2-</sup> /L		
<b>10-510-00-1-A2 #</b>			Waters and water extracts of soil	USEPA method 300.0 (A), High-range method, 100 µL loop, Run time: 11 min.	29-Nov-01
Bromide	0.1 – 5	NA	mg Br <sup>-</sup> /L		
Chloride	2 – 100	NA	mg Cl <sup>-</sup> /L		
Fluoride	0.2 – 10	NA	mg F <sup>-</sup> /L		
Nitrate	0.2 – 10	NA	mg NO <sub>3</sub> <sup>-</sup> - N/L		
Nitrite	0.1 – 5	NA	mg NO <sub>2</sub> <sup>-</sup> - N/L		
Phosphate, ortho	0.2 – 10	NA	mg HPO <sub>4</sub> <sup>2-</sup> - P/L		
Sulfate	4 – 200	NA	mg SO <sub>4</sub> <sup>2-</sup> /L		
<b>10-510-00-1-A3 #</b>			Waters and water extracts of soil	USEPA method 300.0 (A), Low-range method, 200 µL loop, Run time: 11 min.	29-Nov-01
Bromide	0.025 – 2.5	0.005	mg Br <sup>-</sup> /L		
Chloride	0.25 – 25	0.012	mg Cl <sup>-</sup> /L		
Fluoride	0.025 – 2.5	0.004	mg F <sup>-</sup> /L		
Nitrate	0.025 – 2.5	0.002	mg NO <sub>3</sub> <sup>-</sup> - N/L		
Nitrite	0.025 – 2.5	0.005	mg NO <sub>2</sub> <sup>-</sup> - N/L		
Phosphate, ortho	0.025 – 2.5	0.003	mg HPO <sub>4</sub> <sup>2-</sup> - P/L		
Sulfate	0.5 – 50	0.003	mg SO <sub>4</sub> <sup>2-</sup> /L		
<b>10-510-00-1-A4 #</b>			Waters and water extracts of soil	USEPA method 300.0 (A), Chloride to Nitrite-N ratio 800:1, Run time: 16 min.	29-Nov-01
Bromide	0.16 – 3.0	0.02	mg Br <sup>-</sup> /L		
Chloride	32 – 600	NA	mg Cl <sup>-</sup> /L		
Fluoride	0.04 – 0.75	0.008	mg F <sup>-</sup> /L		
Nitrate	0.04 – 0.75	0.005	mg NO <sub>3</sub> <sup>-</sup> - N/L		
Nitrite	0.04 – 0.75	0.02	mg NO <sub>2</sub> <sup>-</sup> - N/L		
Phosphate, ortho	0.10 – 1.875	0.02	mg HPO <sub>4</sub> <sup>2-</sup> - P/L		
Sulfate	32 – 600	NA	mg SO <sub>4</sub> <sup>2-</sup> /L		
<b>10-510-00-1-C</b>			Waters, wastewaters	Common inorganic anions, 50 µL sample loop, Run time: 14 min.	08-Sep-03
Bromide	0.06 – 6.0	0.02	mg Br <sup>-</sup> /L		
Chloride	0.6 – 60	0.005	mg Cl <sup>-</sup> /L		

# USEPA Accepted Methods

^ USEPA Equivalent Methods

\* Methods with External Quality Standards

Method No	Range	MDL	Matrix	Comments	Rev Date
Fluoride	0.04 – 4.0	0.006	mg F <sup>-</sup> /L		
Nitrate	0.06 – 6.0	0.007	mg NO <sub>3</sub> <sup>-</sup> - N/L		
Nitrite	0.016 – 1.6	0.002	mg NO <sub>2</sub> <sup>-</sup> - N/L		
Phosphate, ortho	0.06 – 6.0	0.015	mg HPO <sub>4</sub> <sup>2-</sup> - P/L		
Sulfate	2.0 – 200	0.03	mg SO <sub>4</sub> <sup>2-</sup> /L		
<b>10-510-00-1-E1 ^#</b>			Waters, wastewaters	Fast IC, Separation of all seven common anions in 6.5 minutes, 3 ranges included, Omnion 3.0 only.	31-Oct-08
Bromide	0.05 – 5.0	0.016	mg Br <sup>-</sup> /L		
Chloride	0.5 – 50	0.029	mg Cl <sup>-</sup> /L		
Fluoride	0.05 – 5.0	0.004	mg F <sup>-</sup> /L		
Nitrate	0.05 – 5.0	0.008	mg NO <sub>3</sub> <sup>-</sup> - N/L		
Nitrite	0.05 – 5.0	0.033	mg NO <sub>2</sub> <sup>-</sup> - N/L		
Phosphate, ortho	0.05 – 5.0	0.015	mg HPO <sub>4</sub> <sup>2-</sup> - P/L		
Sulfate	1.0 – 100	0.02	mg SO <sub>4</sub> <sup>2-</sup> /L		
<b>10-510-00-1-E2 ^#</b>			Waters, wastewaters	Low range.	31-Oct-08
Bromide	0.025 – 2.5	0.015	mg Br <sup>-</sup> /L		
Chloride	0.015 – 2.5	0.006	mg Cl <sup>-</sup> /L		
Fluoride	0.025 – 2.5	0.003	mg F <sup>-</sup> /L		
Nitrate	0.025 – 2.5	0.0048	mg NO <sub>3</sub> <sup>-</sup> - N/L		
Nitrite	0.025 – 2.5	0.0048	mg NO <sub>2</sub> <sup>-</sup> - N/L		
Phosphate, ortho	0.025 – 2.5	0.0098	mg HPO <sub>4</sub> <sup>2-</sup> - P/L		
Sulfate	0.5 – 50	0.020	mg SO <sub>4</sub> <sup>2-</sup> /L		
<b>10-510-00-1-E3 ^#</b>			Waters, wastewaters	High range.	31-Oct-08
Bromide	0.1 – 5	0.038	mg Br <sup>-</sup> /L		
Chloride	2.0 – 100	0.016	mg Cl <sup>-</sup> /L		
Fluoride	0.2 – 10.0	0.016	mg F <sup>-</sup> /L		
Nitrate	0.2 – 10.0	0.029	mg NO <sub>3</sub> <sup>-</sup> - N/L		
Nitrite	0.1 – 5.0	0.010	mg NO <sub>2</sub> <sup>-</sup> - N/L		
Phosphate, ortho	0.2 – 10.0	0.034	mg HPO <sub>4</sub> <sup>2-</sup> - P/L		
Sulfate	4.0 – 200	0.144	mg SO <sub>4</sub> <sup>2-</sup> /L		
<b>10-510-13-1-A *</b>			Waters	USEPA method 218.6, Chromium (VI) w/ post-column derivatization, Run time: 5.5 minutes.	09-Sep-03
Chromate	0.2 – 10	0.06	µg Cr(VI)/L		
<b>10-511-00-1-A1 #</b>			Waters, wastewaters	Rapid IC for anions, 50 µL loop, Run time: 4 min.	23-Jan-02
Chloride	1.0 – 100	0.004	mg Cl <sup>-</sup> /L		
Nitrate	0.2 – 20.0	0.003	mg NO <sub>3</sub> <sup>-</sup> - N/L		
Phosphate, ortho	0.05 – 5.0	0.006	mg HPO <sub>4</sub> <sup>2-</sup> - P/L		
Sulfate	1.0 – 100	0.014	mg SO <sub>4</sub> <sup>2-</sup> /L		
<b>10-511-00-1-A2 #</b>			Waters, wastewaters	High-range method, 25 µL loop, Run time: 4 min.	10-Nov-99

# USEPA Accepted Methods

^ USEPA Equivalent Methods

\* Methods with External Quality Standards

Method No	Range	MDL	Matrix	Comments	Rev Date
Chloride	1.5 – 150	0.01	mg Cl <sup>-</sup> /L		
Nitrate	0.25 – 25	0.005	mg NO <sub>3</sub> <sup>-</sup> - N/L		
Phosphate, ortho	0.1 – 10	0.016	mg HPO <sub>4</sub> <sup>2-</sup> - P/L		
Sulfate	2.5 – 250	0.04	mg SO <sub>4</sub> <sup>2-</sup> /L		
<b>10-540-00-1-C #</b>			Waters, wastewaters	USEPA method 300.1, Disinfection byproducts, Run time: 38 min., use Omnion 3.0.	25-Nov-08
Bromate	5 – 50	1.15	µg BrO <sub>3</sub> <sup>-</sup> /L		
Bromide	10 – 100	2.01	µg Br <sup>-</sup> /L		
Chlorate	20 – 200	5.00	µg ClO <sub>3</sub> <sup>-</sup> /L		
Chlorite	5 – 50	2.61	µg ClO <sub>2</sub> <sup>-</sup> /L		
<b>21-510-00-1-A</b>			Beverages		03-Feb-09
Chloride	1 – 50	0.045	mg Cl <sup>-</sup> /L		
Fluoride	0.2 – 10	0.037	mg F <sup>-</sup> /L		
Nitrate	0.2 – 10	0.021	mg NO <sub>3</sub> <sup>-</sup> - N/L		
Phosphate, ortho	4 – 200	0.062	mg HPO <sub>4</sub> <sup>2-</sup> - P/L		
Sulfate	4 – 200	0.102	mg SO <sub>4</sub> <sup>2-</sup> /L		
<b>70-510-00-1-C</b>			High purity waters	Trace-level method	16-Jan-09
Bromide	2 – 40	0.67	µg Br <sup>-</sup> /L		
Chloride	1 – 20	0.22	µg Cl <sup>-</sup> /L		
Fluoride	1 – 20	0.39	µg F <sup>-</sup> /L		
Nitrate	1 – 20	0.20	µg NO <sub>3</sub> <sup>-</sup> - N/L		
Nitrite	1 – 20	0.40	µg NO <sub>2</sub> <sup>-</sup> - N/L		
Phosphate, ortho	3 – 60	0.60	µg HPO <sub>4</sub> <sup>2-</sup> - P/L		
Sulfate	1.5 – 30	0.45	µg SO <sub>4</sub> <sup>2-</sup> /L		
<b>Cations</b>					
<b>10-520-00-1-D1</b>				For use with Omnion 3.0	16-Feb-05
Ammonium	0.8 – 32	0.16	mg NH <sub>4</sub> <sup>+</sup> /L		
Calcium	1.6 – 64	0.60	mg Ca <sup>2+</sup> /L		
Lithium	0.25 – 10	0.05	mg Li <sup>+</sup> /L		
Magnesium	0.8 – 32	0.16	mg Mg <sup>2+</sup> /L		
Potassium	1.6 – 64	0.32	mg K <sup>+</sup> /L		
Sodium	1.8 – 72	0.36	mg Na <sup>+</sup> /L		
<b>10-520-00-1-D2</b>				For use with Omnion 3.0	16-Feb-05
Ammonium	0.2 – 4.0	0.04	mg NH <sub>4</sub> <sup>+</sup> /L		
Calcium	0.25 – 5.0	0.053	mg Ca <sup>2+</sup> /L		
Lithium	0.05 – 1.0	0.01	mg Li <sup>+</sup> /L		
Magnesium	0.25 – 5.0	0.05	mg Mg <sup>2+</sup> /L		
Potassium	0.2 – 4.0	0.04	mg K <sup>+</sup> /L		
Sodium	0.2 – 4.0	0.04	mg Na <sup>+</sup> /L		
<b>10-520-00-1-D3</b>				For use with Omnion 3.0	17-Feb-09
Ammonium	5 – 250	3.49	µg NH <sub>4</sub> <sup>+</sup> /L		
Calcium	25 – 1,250	7.44	µg Ca <sup>2+</sup> /L		
Lithium	8 – 400	0.58	µg Li <sup>+</sup> /L		
Magnesium	12 – 600	2.60	µg Mg <sup>2+</sup> /L		

# USEPA Accepted Methods

^ USEPA Equivalent Methods

\* Methods with External Quality Standards

Method No	Range	MDL	Matrix	Comments	Rev Date
Potassium	20 – 1,000	5.74	µg K <sup>+</sup> /L		
Sodium	10 – 500	1.44	µg Na <sup>+</sup> /L		

### Organic Acids

Method No	Range	MDL	Matrix	Comments	Rev Date
<b>21-550-00-1-B</b>			Beverages	Use with Omnion 3.0.	03-Feb-09
Acetic Acid	3 – 300	1.16	mg/L		
Adipic Acid	4.5 – 450	3.34	mg/L		
Citric Acid	3 – 300	0.45	mg/L		
Formic Acid	3 – 300	0.50	mg/L		
Fumaric Acid	3 – 300	0.45	mg/L		
Lactate / Lactic Acid	3 – 300	0.90	mg/L		
Malic Acid	3 – 300	0.60	mg/L		
Malonic Acid	3 – 300	1.07	mg/L		
Oxalic Acid	3 – 300	0.71	mg/L		
Succinic Acid	3 – 300	0.77	mg/L		
Tartaric Acid	3 – 300	0.92	mg/L		

# USEPA Accepted Methods

^ USEPA Equivalent Methods

\* Methods with External Quality Standards

QuikChem Method 10-510-00-1-E

# Inorganic Anions in Waters

(USEPA 300.0 Part A, Fast Method)

## Mid Range

0.05 to 5 mg F<sup>-</sup>/L

0.50 to 50 mg Cl<sup>-</sup>/L

0.05 to 5 mg NO<sub>2</sub><sup>-</sup>-N/L

0.05 to 5 mg Br<sup>-</sup>/L

0.05 to 5 mg NO<sub>3</sub><sup>-</sup>-N/L

0.05 to 5 mg HPO<sub>4</sub><sup>2-</sup>-P/L

1.0 to 100 mg SO<sub>4</sub><sup>2-</sup>/L

(See Section 1.4 for low and high range concentrations)

## – Principle –

This is a chemically-suppressed, ion chromatography (IC) method. The anions are separated based on their affinities toward the stationary phase in the column. A suppressor cartridge, packed with high-capacity cation exchange resin, chemically suppresses the background conductance of the eluent and, at the same time, converts the anionic species into species of higher conductance. The method provides results that are equivalent to those of USEPA method 300.0 Part A and those of ISO methods 10304-1 and 10304-2.

## – Interferences –

See Section 4 of the method for information on interferences.

## – Special Apparatus –

This method is for use with **Omnion 3.0** only.



## 1. SCOPE AND APPLICATION

1.1 The method covers the determination of the following inorganic anions:

Bromide	Nitrite-N
Chloride	Ortho-Phosphate-P
Fluoride	Sulfate
Nitrate-N	

1.2 The matrices applicable to this method are as follows:

Drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, and reagent waters.

1.3 The single laboratory Method Detection Limits (MDL defined in Section 3.10) for the above analytes are listed in Tables 1-3, Section 17.3.1 of this method. The MDL for a specified matrix may differ from those listed, depending upon the nature of the sample.

1.4 The ranges tested for each anion is as follows:

<u>Analyte</u>	<u>Mid Range</u>	<u>Range, mg/L</u>
bromide, fluoride, nitrite-N, nitrate-N, $\sigma$ -phosphate-P		0.05 to 5
chloride		0.5 to 50
sulfate		1 to 100

**Note:** The calibrated range for this method is two orders of magnitude. These ranges were based on a **20  $\mu$ L** sample loop.

<u>Analyte</u>	<u>Low Range</u>	<u>Range, mg/L</u>
bromide, fluoride, nitrite-N, nitrate-N, $\sigma$ -phosphate-P		0.025 to 2.5
chloride		0.015 to 25
sulfate		0.5 to 50

**Note:** The calibrated range for this method is two orders of magnitude. These ranges were based on a **50  $\mu$ L** sample loop.

<u>Analyte</u>	<u>High Range</u>	<u>Range, mg/L</u>
fluoride, nitrate-N, $\sigma$ -phosphate-P		0.2 to 10.0
bromide, nitrite-N		0.1 to 5.0
chloride		2.0 to 100
sulfate		4.0 to 200

**Note:** The calibrated range for this method is two orders of magnitude. These ranges were based on a **10  $\mu$ L** sample loop.

1.5 This method is recommended for use only by analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatogram.

- 1.6 When this method is used to analyze unfamiliar samples for any of the above anions, anion identification should be supported by the use of a fortified sample matrix covering the anions of interest. The fortification procedure is described in Section 9.
- 1.7 Bromide and nitrite react with most oxidants employed as disinfectants for drinking waters. The utility of measuring these anions in treated water should be considered prior to conducting the analysis.
- 1.8. Users of the method data should state the data-quality objectives prior to analysis. Users of the method must demonstrate the ability to generate acceptable results with this method, using the procedures described in Section 9, Quality Control.

## METHOD SUPPORT DATA

**Table 1. Method Detection Limits for Inorganic Anions in Reagent Water – Mid Range**

Analyte	Known Conc. (mg/L)	Determined Conc. (mg/L)	Std. Dev. (mg/L)	MDL (mg/L)	PQL (mg/L)
Fluoride	0.02	0.019	0.0012	0.004	0.012
Chloride	0.025	0.056	0.009	0.029	0.092
Nitrite-N	0.02	0.025	0.010	0.033	0.105
Bromide	0.05	0.040	0.005	0.016	0.051
Nitrate-N	0.02	0.026	0.0026	0.008	0.026
Phosphate-P	0.04	0.041	0.0047	0.015	0.047
Sulfate	0.05	0.078	0.006	0.020	0.064

**Note:** The instrument was calibrated by including the MDL standard in the calibration model (Section 7.11). Seven replicates of a standard were analyzed. The MDL equals the standard deviation of the determined values for seven replicate runs multiplied by 3.142, corresponding to a 99% confidence value. The PQL (practical quantitation limit) equals the standard deviation multiplied by 10.

**Table 2. Method Detection Limits for Inorganic Anions in Reagent Water – Low Range**

Analyte	Known Conc. (mg/L)	Determined Conc. (mg/L)	Std. Dev. (mg/L)	MDL (mg/L)	PQL (mg/L)
Fluoride	0.01	0.013	0.001	0.003	0.011
Chloride	0.03	0.034	0.0018	0.006	0.019
Nitrite-N	0.01	0.013	0.0009	0.003	0.009
Bromide	0.025	0.028	0.005	0.015	0.049
Nitrate-N	0.01	0.012	0.0015	0.0048	0.015
Phosphate-P	0.02	0.024	0.003	0.0098	0.031
Sulfate	0.05	0.054	0.0064	0.020	0.064

**Note:** The instrument was calibrated by including the MDL standard in the calibration model (Section 7.11). Seven replicates of a standard were analyzed. The MDL equals the standard deviation of the determined values for seven replicate runs multiplied by 3.142, corresponding to a 99% confidence value. The PQL (practical quantitation limit) equals the standard deviation multiplied by 10.

**Table 3. Method Detection Limits for Inorganic Anions in Reagent Water – High Range**

Analyte	Known Conc. (mg/L)	Determined Conc. (mg/L)	Std. Dev. (mg/L)	MDL (mg/L)	PQL (mg/L)
Fluoride	0.20	0.20	0.005	0.016	0.052
Chloride	2.0	1.93	0.005	0.016	0.05

Nitrite-N	0.10	0.10	0.003	0.010	0.033
Bromide	0.10	0.10	0.012	0.038	0.12
Nitrate-N	0.20	0.20	0.009	0.029	0.09
Phosphate-P	0.20	0.20	0.011	0.034	0.11
Sulfate	4.0	3.80	0.046	0.144	0.46

**Note:** The instrument was calibrated by including the MDL standard in the calibration model (Section 7.11). Seven replicates of a standard were analyzed. The MDL equals the standard deviation of the determined values for seven replicate runs multiplied by 3.142, corresponding to a 99% confidence value. The PQL (practical quantitation limit) equals the standard deviation multiplied by 10.

**Table 4. Single-Operator Precision of Standard Anions – Mid Range (Standard B)**

(10 replicates of each sample were analyzed)

Analyte	Known Conc. (mg/L)	Determined Conc. (mg/L)	Std. Dev. (mg/L)	%RSD
Fluoride	3.0	2.94	0.021	0.71
Chloride	30.0	29.9	0.19	0.63
Nitrite-N	3.0	2.98	0.036	1.22
Bromide	3.0	2.93	0.06	2.05
Nitrate-N	3.0	3.05	0.013	0.42
Phosphate-P	3.0	2.93	0.024	0.81
Sulfate	60.0	60.4	0.28	0.46

**Table 5. Single-Operator Precision of Standard Anions – Mid Range (Standard C)**

(10 replicates of each sample were analyzed)

Analyte	Known Conc. (mg/L)	Determined Conc. (mg/L)	Std. Dev. (mg/L)	%RSD
Fluoride	1.0	0.94	0.004	0.45
Chloride	10.0	9.50	0.03	0.31
Nitrite-N	1.0	0.96	0.006	0.63
Bromide	1.0	0.93	0.009	1.02
Nitrate-N	1.0	0.99	0.006	0.60
Phosphate-P	1.0	0.93	0.008	0.85
Sulfate	20.0	19.27	0.116	0.60

**Table 6. Single-Operator Precision of Standard Anions – Mid Range (Standard D)**

(10 replicates of each sample were analyzed)

Analyte	Known Conc. (mg/L)	Determined Conc. (mg/L)	Std. Dev. (mg/L)	%RSD
Fluoride	0.4	0.38	0.002	0.56
Chloride	4.0	3.78	0.008	0.22
Nitrite-N	0.4	0.39	0.004	1.07
Bromide	0.4	0.38	0.013	3.43
Nitrate-N	0.4	0.39	0.006	1.48
Phosphate-P	0.4	0.39	0.008	2.18
Sulfate	8.0	7.83	0.017	0.22

**Table 7. Single-Operator Precision of Standard Anions – Low Range (Standard B)**  
(10 replicates of each sample were analyzed)

<b>Analyte</b>	<b>Known Conc. (mg/L)</b>	<b>Determined Conc. (mg/L)</b>	<b>Std. Dev. (mg/L)</b>	<b>%RSD</b>
Fluoride	1.5	1.48	0.006	0.38
Chloride	15.0	15.4	0.05	0.31
Nitrite-N	1.5	1.48	0.01	0.71
Bromide	1.5	1.50	0.008	0.57
Nitrate-N	1.5	1.53	3.44e-08	2.25e-06
Phosphate-P	1.5	1.48	0.005	0.33
Sulfate	30.0	30.6	0.05	0.17

**Table 8. Single-Operator Precision of Standard Anions – Low Range (Standard C)**  
(10 replicates of each sample were analyzed)

<b>Analyte</b>	<b>Known Conc. (mg/L)</b>	<b>Determined Conc. (mg/L)</b>	<b>Std. Dev. (mg/L)</b>	<b>%RSD</b>
Fluoride	0.5	0.48	0.003	0.56
Chloride	5.0	4.90	0.006	0.12
Nitrite-N	0.5	0.48	0.002	0.45
Bromide	0.5	0.49	0.003	0.61
Nitrate-N	0.5	0.50	0.002	0.40
Phosphate-P	0.5	0.47	0.003	0.60
Sulfate	10.0	9.84	0.011	0.11

**Table 9. Single-Operator Precision of Standard Anions – High Range (Standard B)**  
(10 replicates of each sample were analyzed)

<b>Analyte</b>	<b>Known Conc. (mg/L)</b>	<b>Determined Conc. (mg/L)</b>	<b>Std. Dev. (mg/L)</b>	<b>%RSD</b>
Fluoride	6.0	6.12	0.03	0.47
Chloride	60.0	60.1	0.28	0.47
Nitrite-N	3.0	3.03	0.03	1.06
Bromide	3.0	3.05	0.07	2.21
Nitrate-N	6.0	6.06	0.02	0.36
Phosphate-P	6.0	6.01	0.05	0.76
Sulfate	120	119	0.67	0.56

**Table 10. Single-Operator Precision of Standard Anions – High Range (Standard C)**  
(10 replicates of each sample were analyzed)

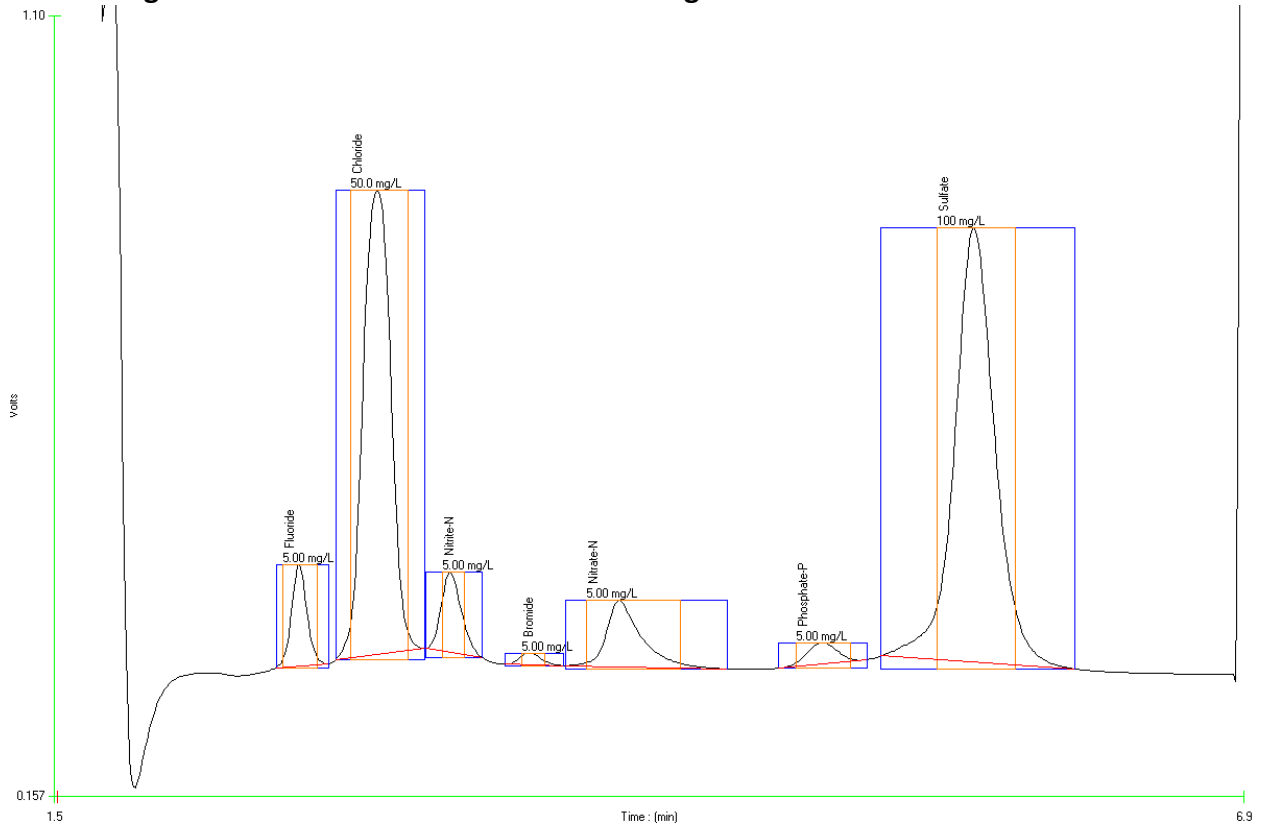
<b>Analyte</b>	<b>Known Conc. (mg/L)</b>	<b>Determined Conc. (mg/L)</b>	<b>Std. Dev. (mg/L)</b>	<b>%RSD</b>
Fluoride	2.0	2.0	0.01	0.48
Chloride	20.0	19.4	0.00	0.00
Nitrite-N	1.0	0.99	0.01	0.91
Bromide	1.0	1.03	0.03	3.06
Nitrate-N	2.0	1.94	0.01	0.50
Phosphate-P	2.0	1.95	0.02	1.21
Sulfate	40.0	39.3	0.08	0.21

**Table 11. Single-Operator Accuracy and Precision of Standard Anions Spikes**  
(Seven replicates of each sample were analyzed)

Analyte	Sample Type	Spike mg/L	Mean Recovery -- % --	Standard Deviation - mg/L -
Fluoride	RW	0.5	93.7	0.002
	DW	0.5	87.7	0.005
	SW	0.5	86.1	0.005
	WW	2.0	84.4	0.008
Chloride	RW	5.0	93.6	0.01
	DW	5.0	93.2	0.02
	SW	5.0	92.1	0.018
	WW	15.0	95.6	0.097
Nitrite-N	RW	0.5	92.8	0.003
	DW	0.5	93.9	0.003
	SW	0.5	94.9	0.005
	WW	1.0	105.0	0.025
Bromide	RW	0.5	93.4	0.006
	DW	0.5	90.9	0.008
	SW	0.5	93.6	0.012
	WW	1.0	85.8	0.048
Nitrate-N	RW	0.5	95.4	0.01
	DW	0.5	100.8	0.006
	SW	0.5	98.7	0.003
	WW	2.0	78.4	0.017
O-Phosphate-P	RW	0.5	92.8	0.008
	DW	0.5	94.5	0.005
	SW	0.5	92.5	0.008
	WW	2.0	103.5	0.027
Sulfate	RW	10	95.5	0.016
	DW	10	94.9	0.05
	SW	10	92.4	0.053
	WW	40	83.8	0.125

RW; Reagent Water, DW; Drinking Water, SW; Surface Water, WW; Wastewater

# Chromatogram of calibration standard A - Mid Range



File Name: 10-21 mr cal support.omn

Acq. Date: 21 October 2008



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
CINCINNATI, OHIO 45268

Lynn Egan  
legan@hach.com  
Applications Chemist  
Lachat Instruments  
P.O. Box 389, Mail Stop #1  
Loveland, CO 80539-0389

February 20, 2009

RE ATP Case No. D08-0007:

Per the terms of the Alternate Test Procedure (ATP) program, the Office of Ground Water and Drinking Water's Technical Support Center (OGWDW/TSC) has determined that QuikChem Method 10-510-00-1-E [10/29/2008] is an acceptable version of EPA Method 300.1, Part A. Subsequently, the method may be used for drinking water compliance monitoring under National Primary Drinking Water Regulations (NPDWR).

We appreciate your interest in the development of environmental monitoring methods. If you have any questions regarding the review of this alternate test procedure, please contact Steve Wendelken by e-mail at: [wendelken.steve@epa.gov](mailto:wendelken.steve@epa.gov) or by telephone at: 513-569-7491.

Sincerely,

A handwritten signature in cursive script, appearing to read "Steven C. Wendelken".

Steven C. Wendelken, PhD.  
U.S. EPA, OGWDW-TSC  
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Fax: (513) 569-7837  
[wendelken.steve@epa.gov](mailto:wendelken.steve@epa.gov)

# Inorganic Anions in High Purity Waters

1.0 to 20.0  $\mu\text{g F}^-/\text{L}$

1.0 to 20.0  $\mu\text{g Cl}^-/\text{L}$

1.0 to 20.0  $\mu\text{g NO}_2^-/\text{N/L}$

2.0 to 40.0  $\mu\text{g Br}^-/\text{L}$

1.0 to 20.0  $\mu\text{g NO}_3^-/\text{N/L}$

3.0 to 60.0  $\mu\text{g HPO}_4^{2-}/\text{P/L}$

1.5 to 30.0  $\mu\text{g SO}_4^{2-}/\text{L}$

## – Principle –

This method describes determination of trace anions for trace-level determination of common anions in high purity waters, e.g., as used in the power generation and semiconductor manufacturing industries. The anions are separated based on their affinities toward the stationary phase in the column. A suppressor cartridge, packed with high-capacity cation exchange resin, chemically suppresses the background conductance of the eluent and, at the same time, converts the anionic species into species of higher conductance.

## – Interferences –

See Section 4 of the method for information on interferences.

## – Special Apparatus –

This method is for use with **Omnion 3.0** only.



# 1. METHOD SUPPORT DATA

**Table 1. Method Detection Limits for Inorganic Anions in Reagent Water**

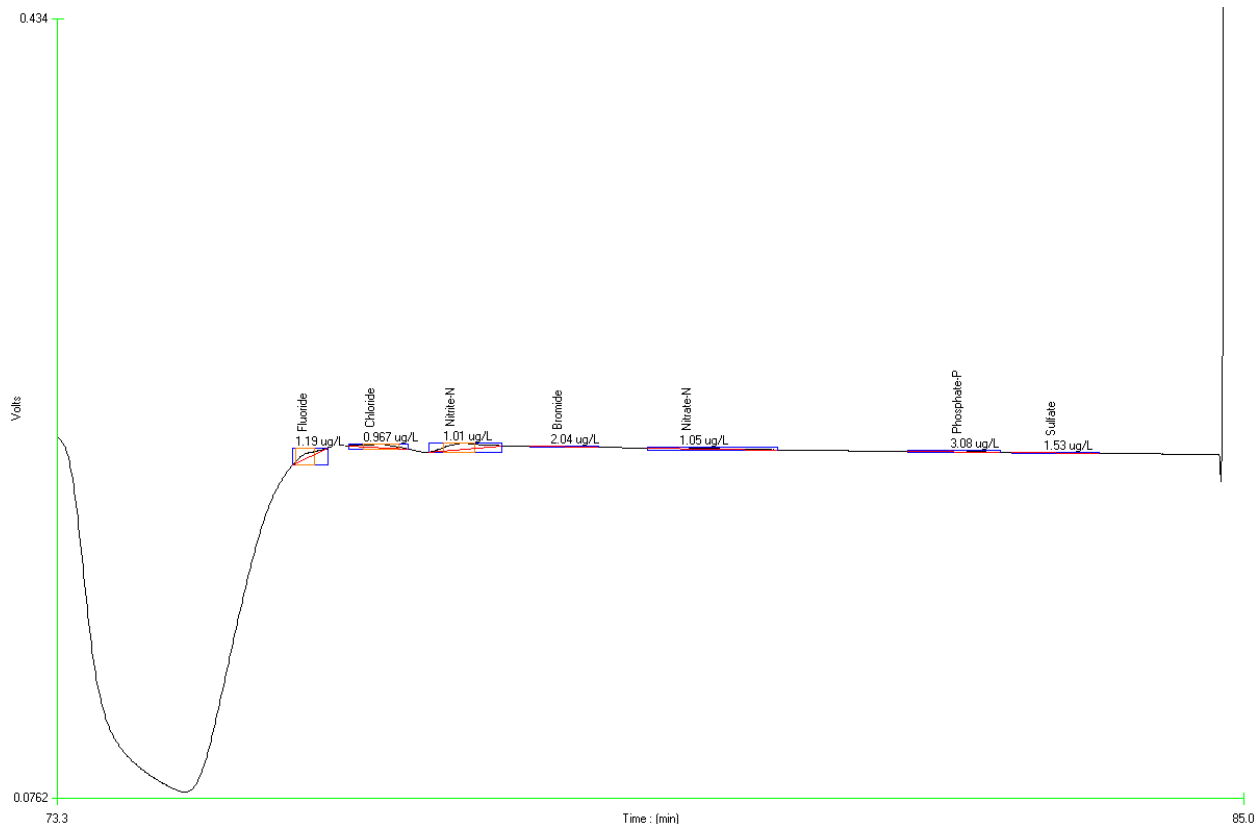
Analyte	Known Conc. (µg/L)	Determined Conc. (µg/L)	Std. Dev. (µg/L)	MDL (µg/L)	PQL (µg/L)
Fluoride	1.0	0.97	0.08	0.39	1.38
Chloride	1.0	1.02	0.14	0.22	0.78
Nitrite-N	1.0	1.22	0.24	0.40	1.41
Bromide	2.0	1.88	0.14	0.67	2.38
Nitrate-N	1.0	1.00	0.04	0.20*	0.39
Phosphate-P	3.0	3.04	0.18	0.60**	1.81
Sulfate	1.5	1.70	0.16	0.45	1.58

**Note:** The instrument was calibrated by including the MDL standard in the calibration model (Section 7.11). Ten replicates of a standard were analyzed. The MDL equals the standard deviation of the determined values for ten replicate runs multiplied by 2.821, corresponding to a 99% confidence value. The PQL (practical quantitation limit) equals the standard deviation multiplied by 10.

\*Claiming 0.20, actual calculated mdl was 0.11 µg/L, however since this value was less than 20% of the known value.

\*\* Claiming 0.60, actual calculated mdl was 0.51 µg/L, however since this value was less than 20% of the known value.

**Figure 1. Chromatogram of MDL Replicate**



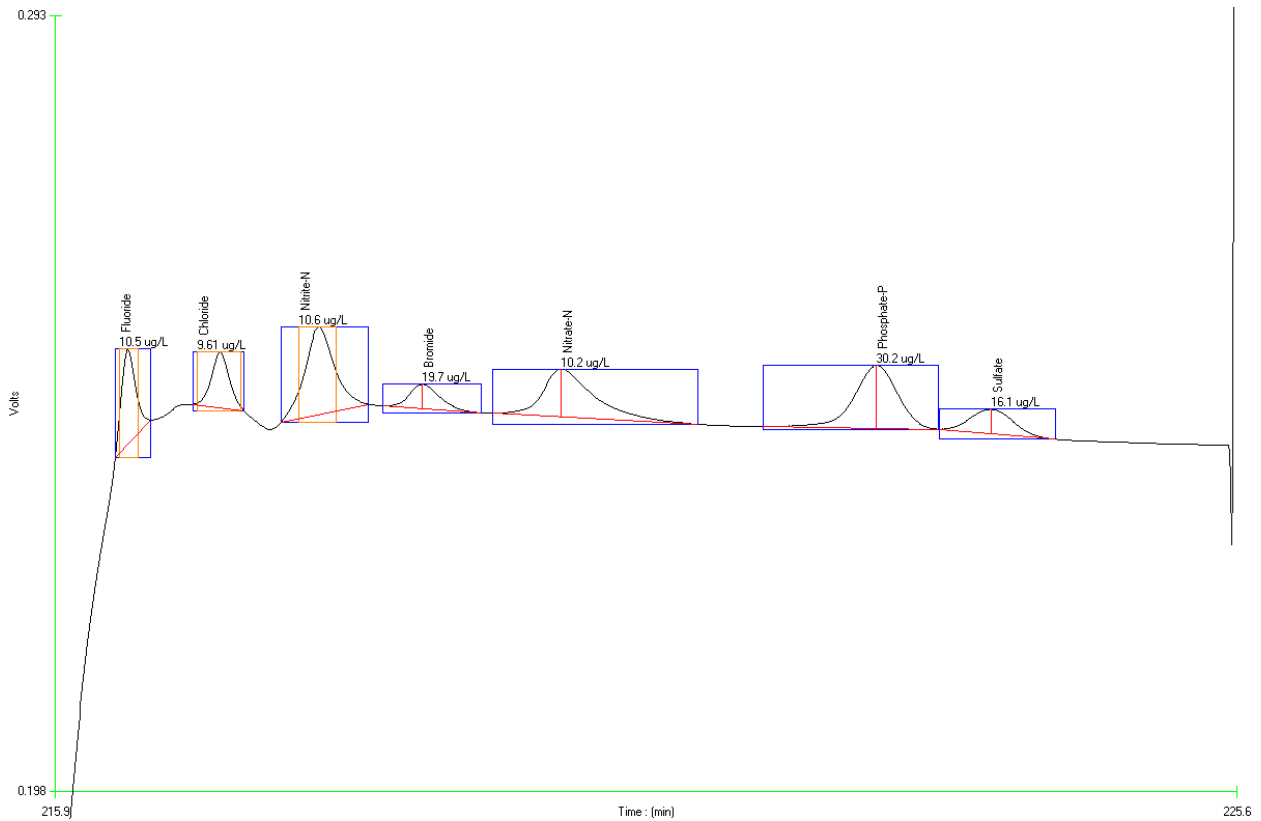
File Name: 12-17 mdl spike.omn

Acq. Date: 17 December 2008

**Table 2. Single-Operator Precision of Standard Anions (Standard B)**  
 (10 replicates of each sample were analyzed)

Analyte	Known Conc. (µg/L)	Determined Conc. (µg/L)	Std. Dev. (µg/L)	%RSD
Fluoride	10	10.20	0.46	4.47
Chloride	10	9.56	0.22	2.35
Nitrite-N	10	10.88	0.19	1.72
Bromide	20	19.97	0.18	0.92
Nitrate-N	10	10.23	0.07	0.66
Phosphate-P	30	30.59	0.77	2.51
Sulfate	15	16.16	0.16	0.98

**Figure 2. Chromatogram of Standard B Precision Replicate**



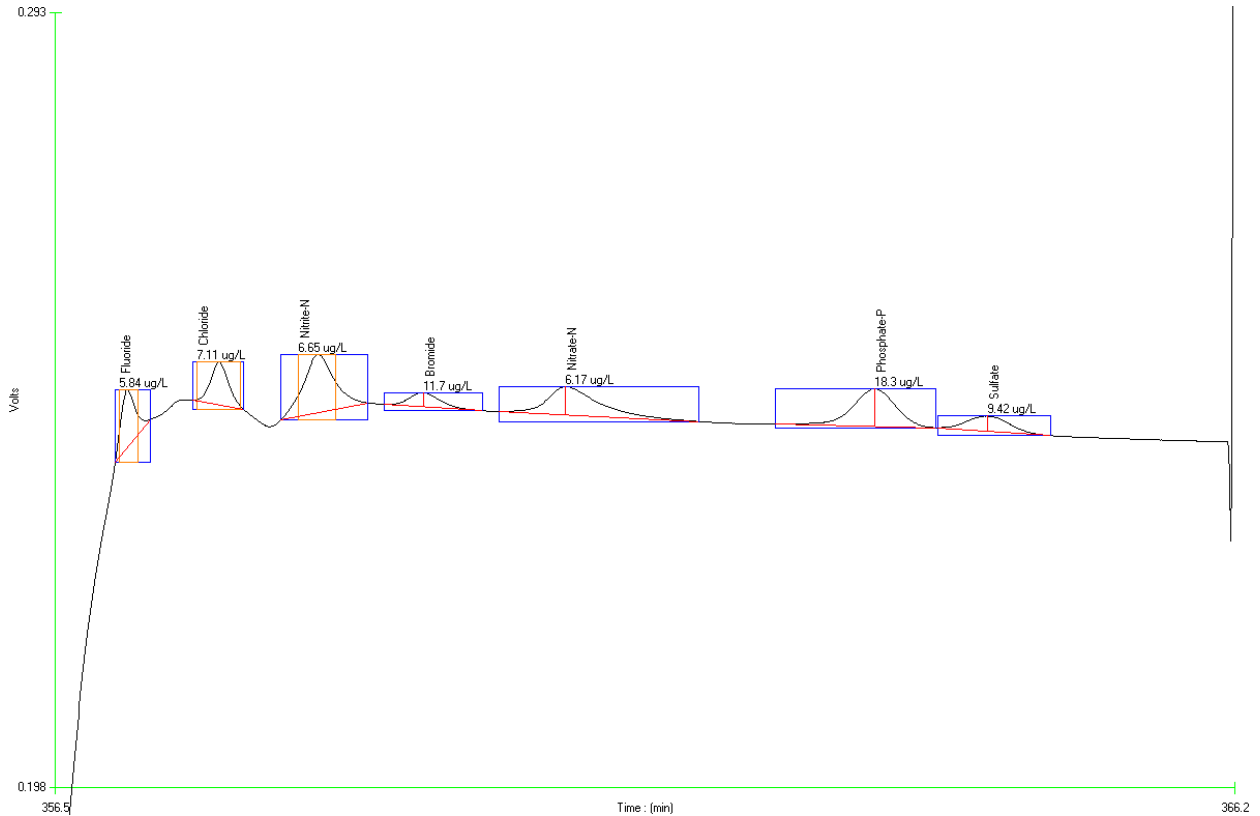
File Name: 12-15 cal support.omn

Acq. Date: 15 December 2008

**Table 3. Single-Operator Precision of Standard Anions (Standard C)**  
 (10 replicates of each sample were analyzed)

Analyte	Known Conc. (µg/L)	Determined Conc. (µg/L)	Std. Dev. (µg/L)	%RSD
Fluoride	6	5.84	0.36	6.16
Chloride	6	7.13	0.10	1.41
Nitrite-N	6	6.79	0.12	1.73
Bromide	12	11.88	0.15	1.24
Nitrate-N	6	6.22	0.08	1.36
Phosphate-P	18	18.21	0.15	0.84
Sulfate	9	9.66	0.13	1.36

**Figure 3. Chromatogram of Standard C Precision Replicate**



File Name: 12-15 cal support.omn

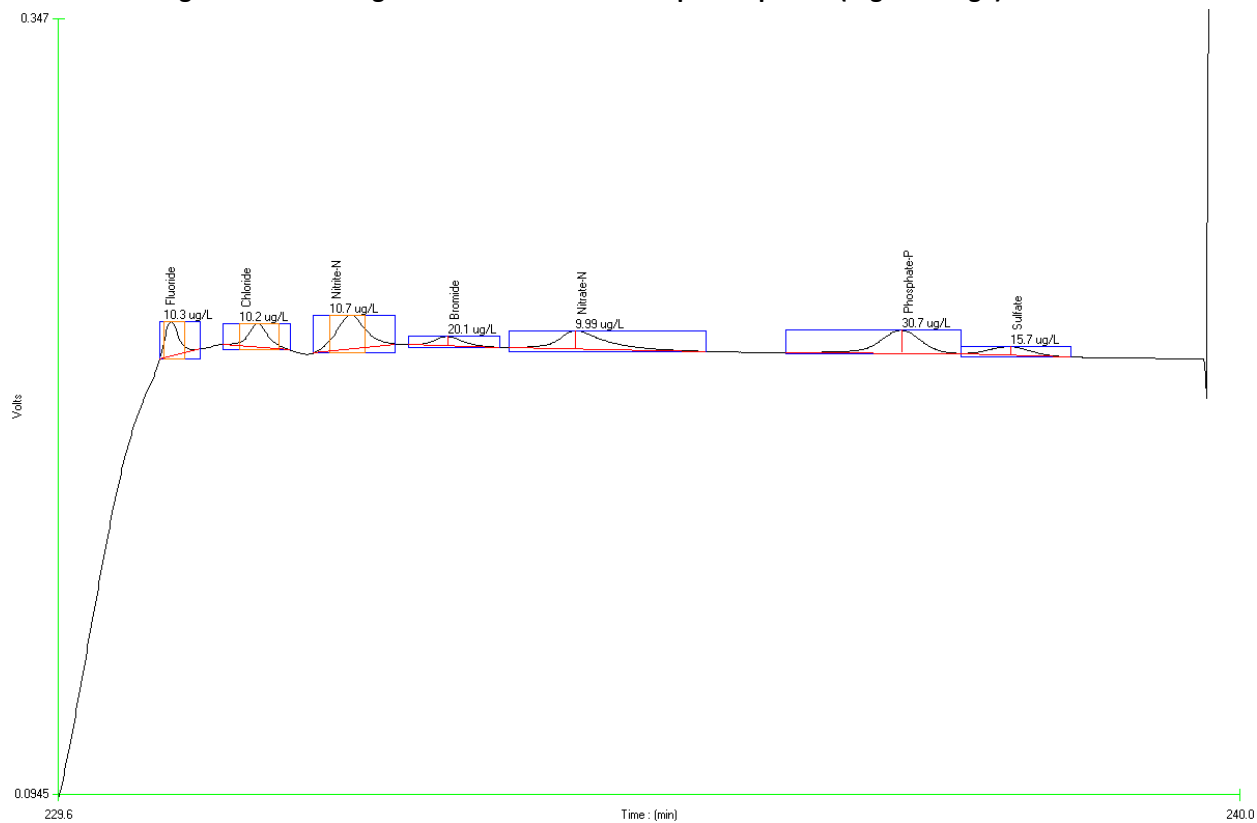
Acq. Date: 15 December 2008

**Table 4. Recovery of Spikes in De-ionized Water (Higher Range)**

(2 replicates of each sample were analyzed)

Analyte	Unspiked (µg/L)	Spiked Conc (µg/L)	Spike Level (µg/L)	% Recovery
Fluoride	-0.11	10.90	10	110.14
Chloride	1.96	10.10	10	81.40
Nitrite-N	0.43	10.70	10	102.75
Bromide	0.12	20.10	20	99.90
Nitrate-N	0.35	9.97	10	96.19
Phosphate-P	0.66	30.90	30	100.81
Sulfate	0.63	15.55	15	99.50

**Figure 4. Chromatogram of De-ionized Water Spike Replicate (Higher Range)**



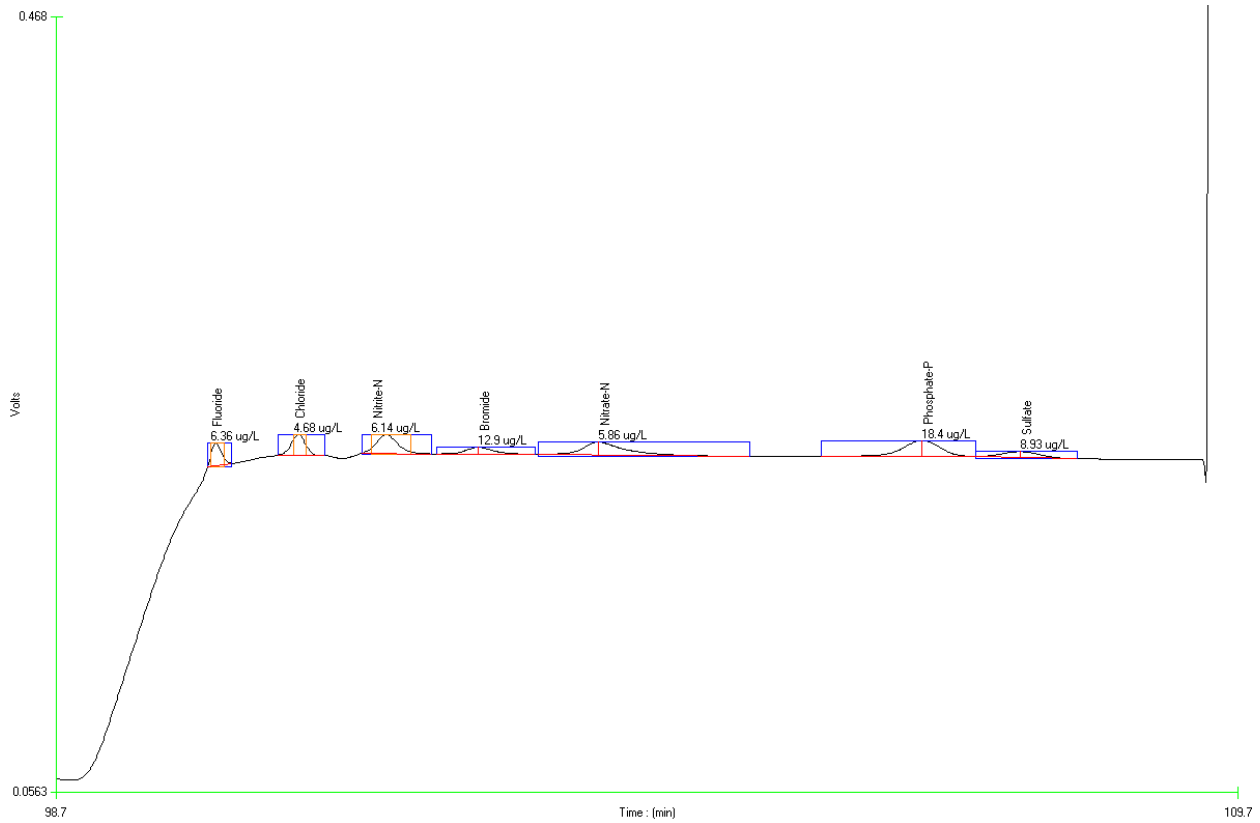
File Name: 12-17 mdl spike.omn

Acq. Date: 17 December 2008

**Table 5. Recovery of Spikes in De-ionized Water (Lower Range)**  
 (2 replicates of each sample were analyzed)

Analyte	Unspiked (µg/L)	Spiked Conc (µg/L)	Spike Level (µg/L)	% Recovery
Fluoride	-0.07	6.60	6	111.01
Chloride	-1.15	4.74	6	98.17
Nitrite-N	-0.24	6.26	6	108.32
Bromide	-0.35	12.45	12	106.67
Nitrate-N	0.19	5.98	6	96.43
Phosphate-P	0.30	18.45	18	100.82
Sulfate	-1.08	8.99	9	111.83

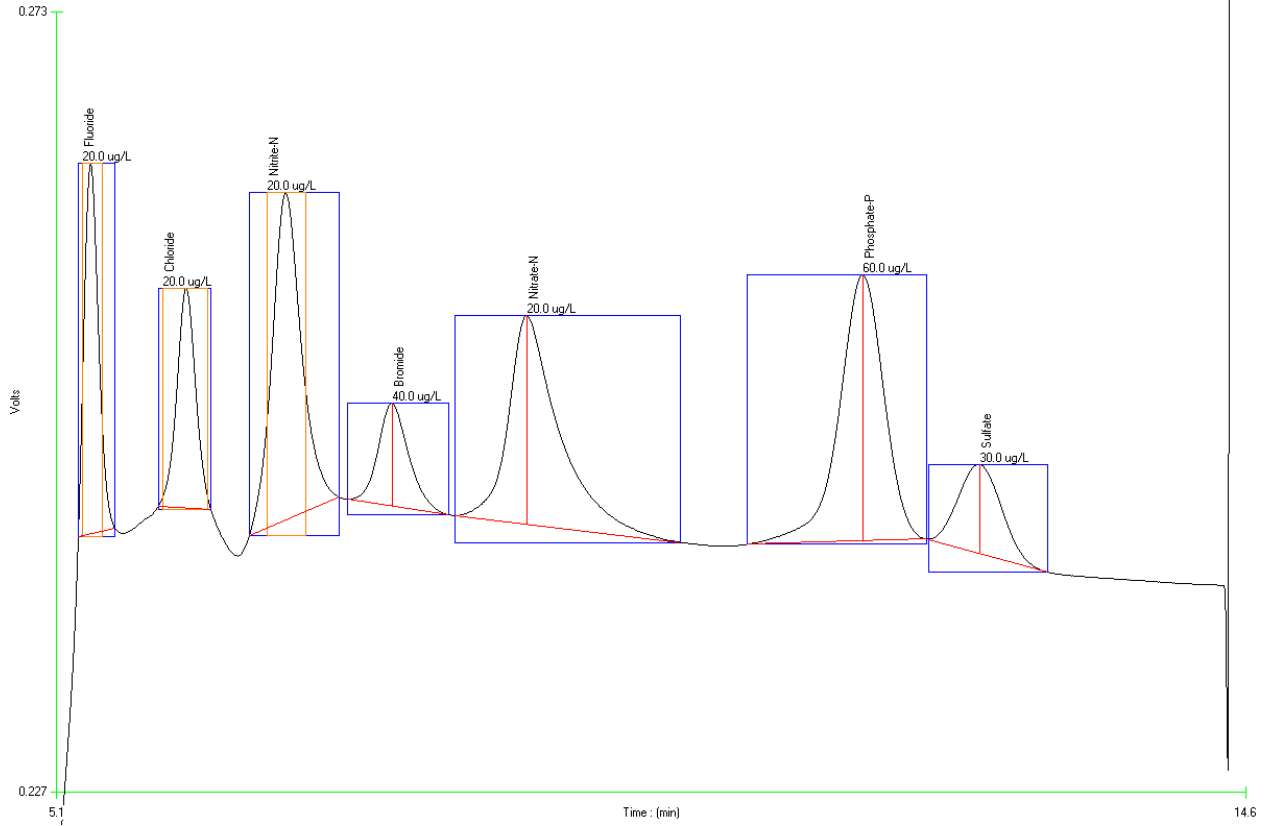
**Figure 5. Chromatogram of De-ionized Water Spike Replicate (Lower Range)**



File Name: 12-12 cal spike.omn

Acq. Date: 12 December 2008

# Chromatogram of calibration standard A



File Name: 12-15 cal support.omn  
Acq. Date: 15 December 2008

# Inorganic Disinfection Byproducts in Drinking Waters

5 to 50  $\mu\text{g ClO}_2^-/\text{L}$   
5 to 50  $\mu\text{g BrO}_3^-/\text{L}$   
20 to 200  $\mu\text{g ClO}_3^-/\text{L}$   
10 to 100  $\mu\text{g Br}^-/\text{L}$

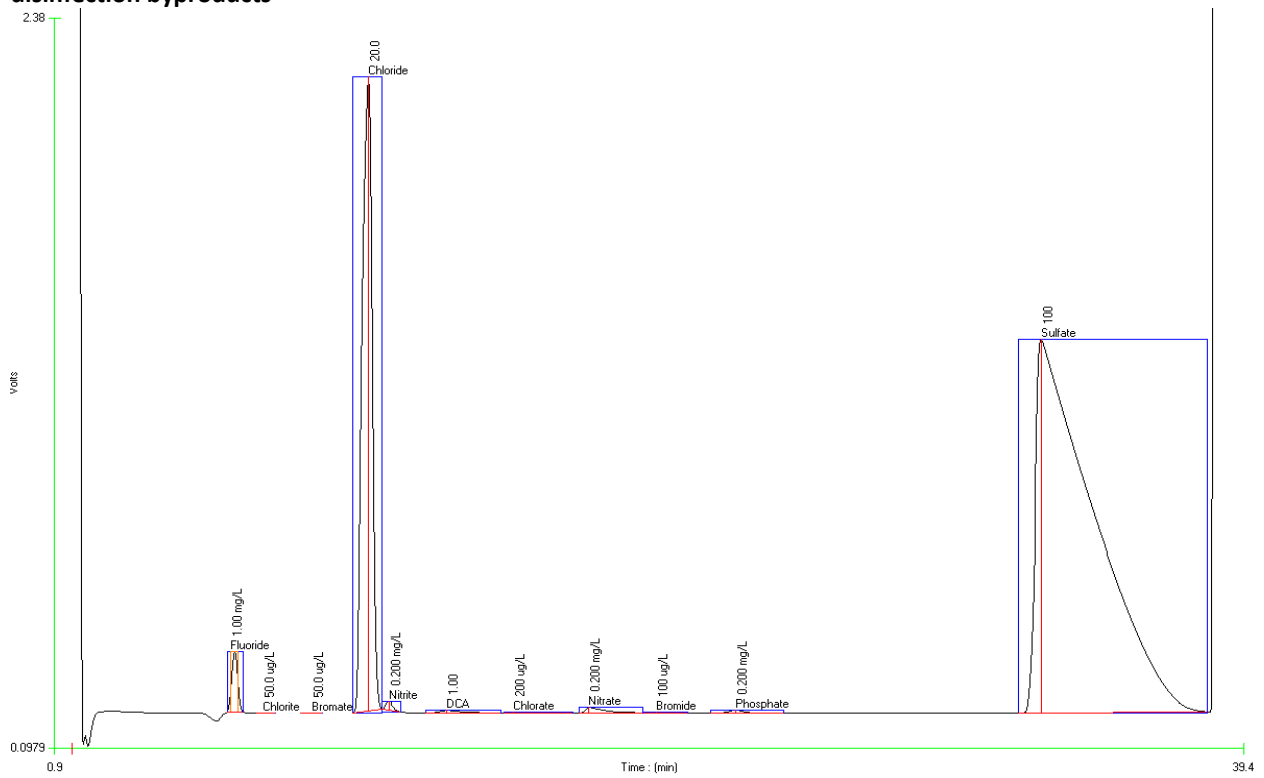
## – Principle –

This is a chemically-suppressed ion chromatography (IC) method. The anions are separated based on their affinities toward the stationary phase in the column. A suppressor cartridge, packed with high-capacity cation exchange resin, chemically suppresses the background conductance of the eluent and, at the same time, converts the anionic species into species of higher conductance. The method provides results that are similar to those of USEPA Method 300.1.

## – Special Apparatus –

This method is for use with **Omnion 3.0** only and requires Lachat Instruments QuikChem 8500 IC+ Ion Chromatography System.

**Figure 1. Chromatogram of Calibration Standard A Showing Separation of common inorganic anions and the disinfection byproducts**

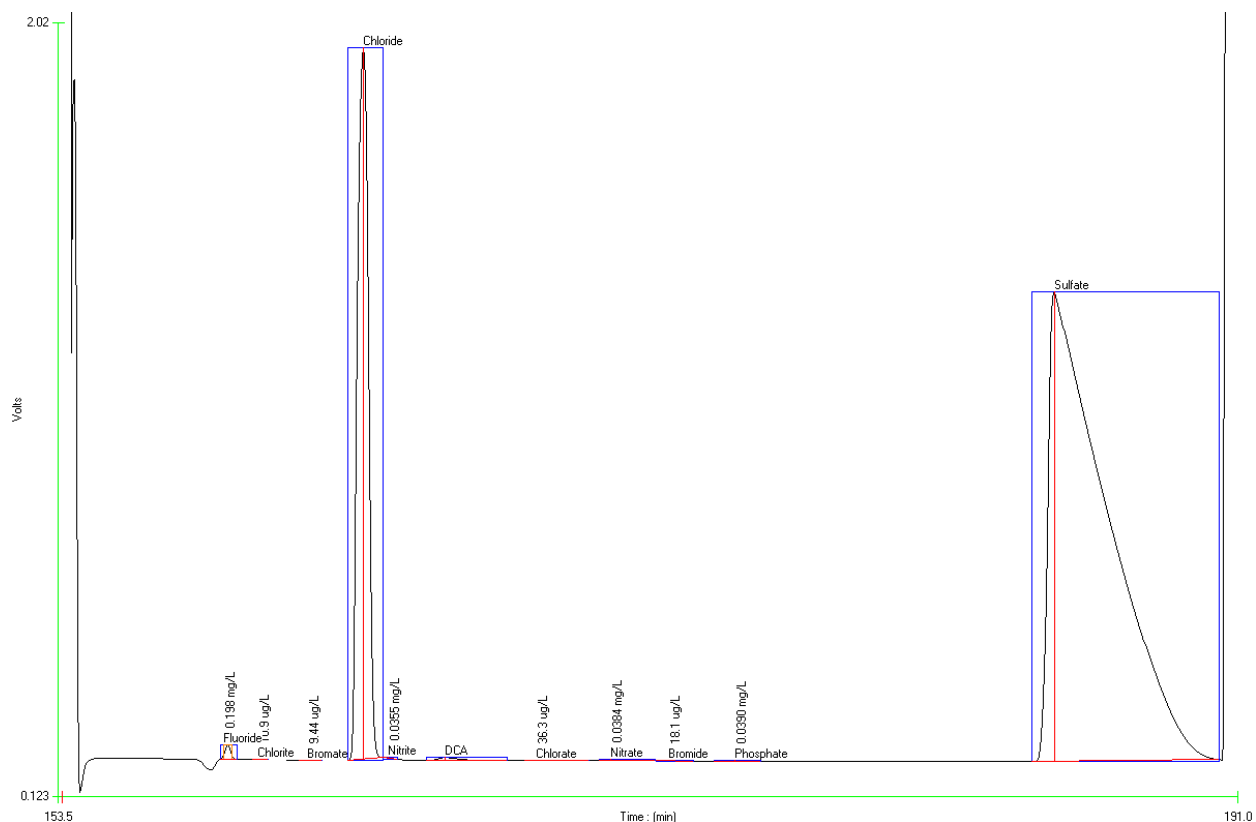


File Name: 11-20 cal mdl.omn

Acq. Date: 20 November 2008



**Figure 2. Chromatogram of MDL Replicate**



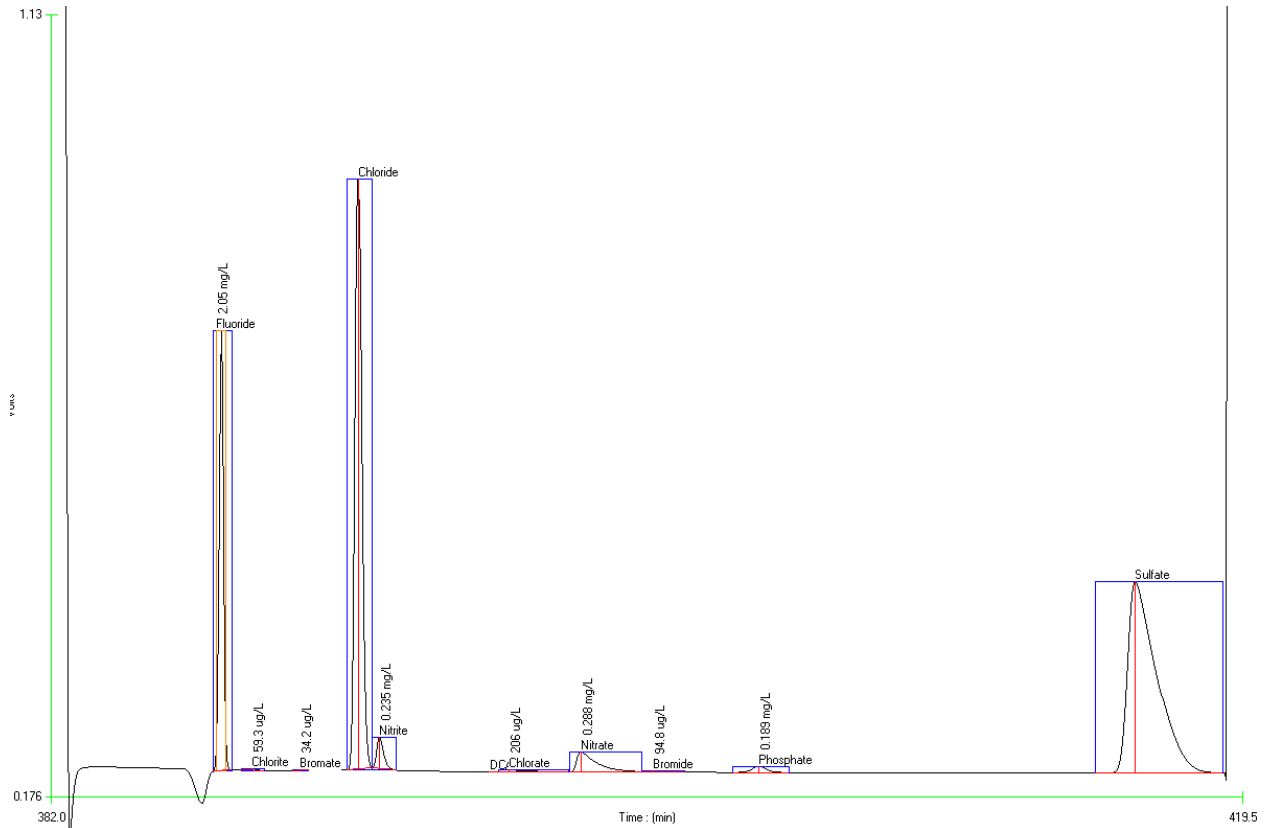
File Name: 11-20 cal mdl.omn  
Acq. Date: 20 November 2008

**Table 1. Precision and MDL Determination**

Anion	Known amount	Mean	Std. Dev.	MDL	PQL	RSD	Recovery
	$\mu\text{g/L}$	$\mu\text{g/L}$	$\mu\text{g/L}$	$\mu\text{g/L}$	$\mu\text{g/L}$	%	%
Fluoride	200	207.4	5.91	18.56	59.1	2.85	103.7
Chlorite	10.0	9.75	0.83	2.61	8.31	8.52	97.5
Bromate	10.0	9.96	0.37	1.15	3.66	3.68	99.6
Nitrite-N	40.0	34.54	0.66	2.09	6.65	1.93	86.4
Chlorate	40.0	37.2	1.59	5.00	15.93	4.28	93.0
Bromide	20.0	18.73	0.64	2.01	6.40	3.41	93.6
Nitrate-N	40.0	38.64	0.27	0.85	2.70	0.70	96.6
Phosphate-P	40.0	38.76	0.66	2.09	6.65	1.72	96.9

**Note:** Seven replicates of a standard were analyzed. The MDL equals the standard deviation of the determined values for seven replicate runs multiplied by 3.14, corresponding to a 99% confidence value. The PQL (practical quantitation limit) equals the standard deviation multiplied by 10.

**Figure 3. Chromatogram of Drinking Water Spike Replicate**



File Name: 11-19 spike\_20.omn

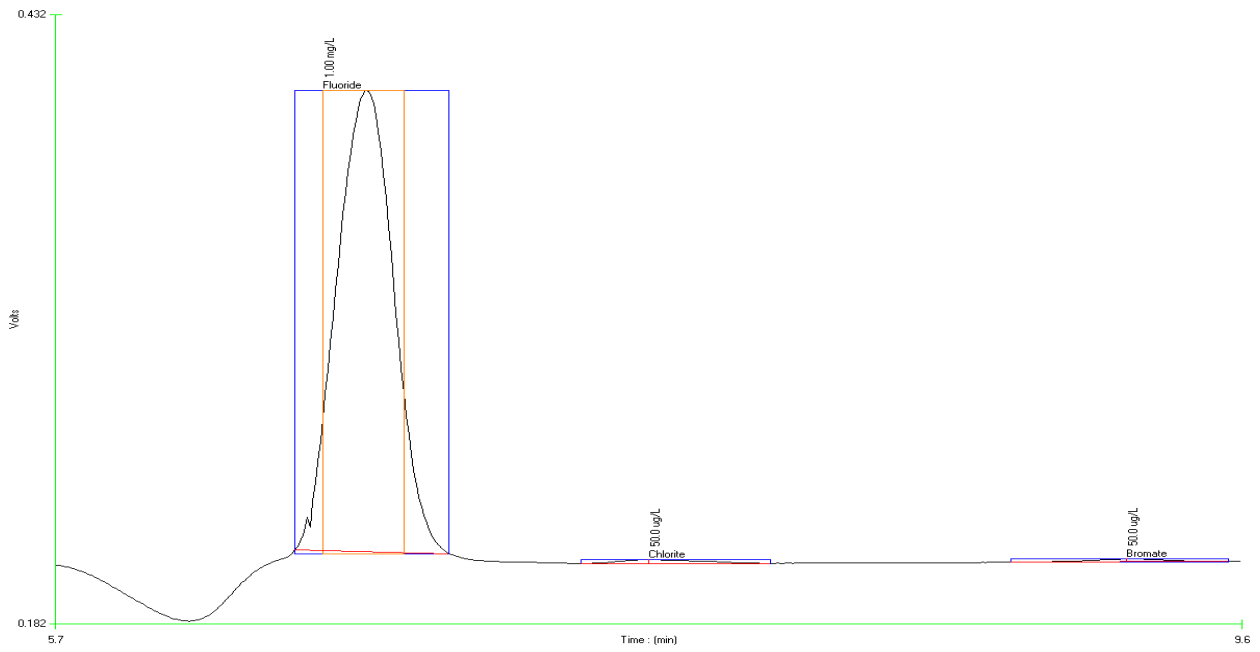
Acq. Date: 20 November 2008

**Table 2. Recovery of Spikes in Drinking Water**

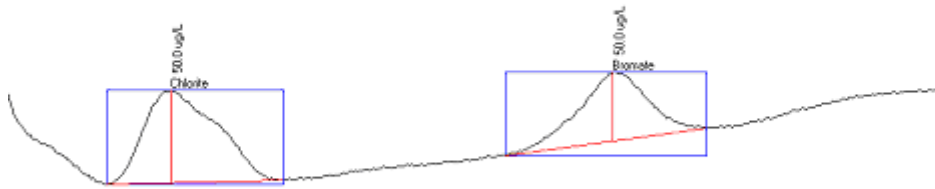
Analyte	Unspiked Tapwater <sup>a</sup>	Spiked amount <sup>a</sup>	Spiked total <sup>a</sup>	% Recovery
Fluoride	1.07	1.0	2.06	99.0
Chlorite	3.69	50.0	59.4	111.3
Bromate	-16.8	50.0	34.4	102.5
Nitrite-N	12.6	200	236	111.5
Chlorate	9.16	200	210	100.7
Bromide	4.64	100	95.9	91.3
Nitrate-N	107	200	288	90.8
Phosphate	-4.31	200	190	97.4

<sup>a</sup> Results for fluoride are in mg/L, all others are in ug/L.

**Figure 4. Chromatogram Showing Separation of Fluoride, Chlorite and Bromate.**

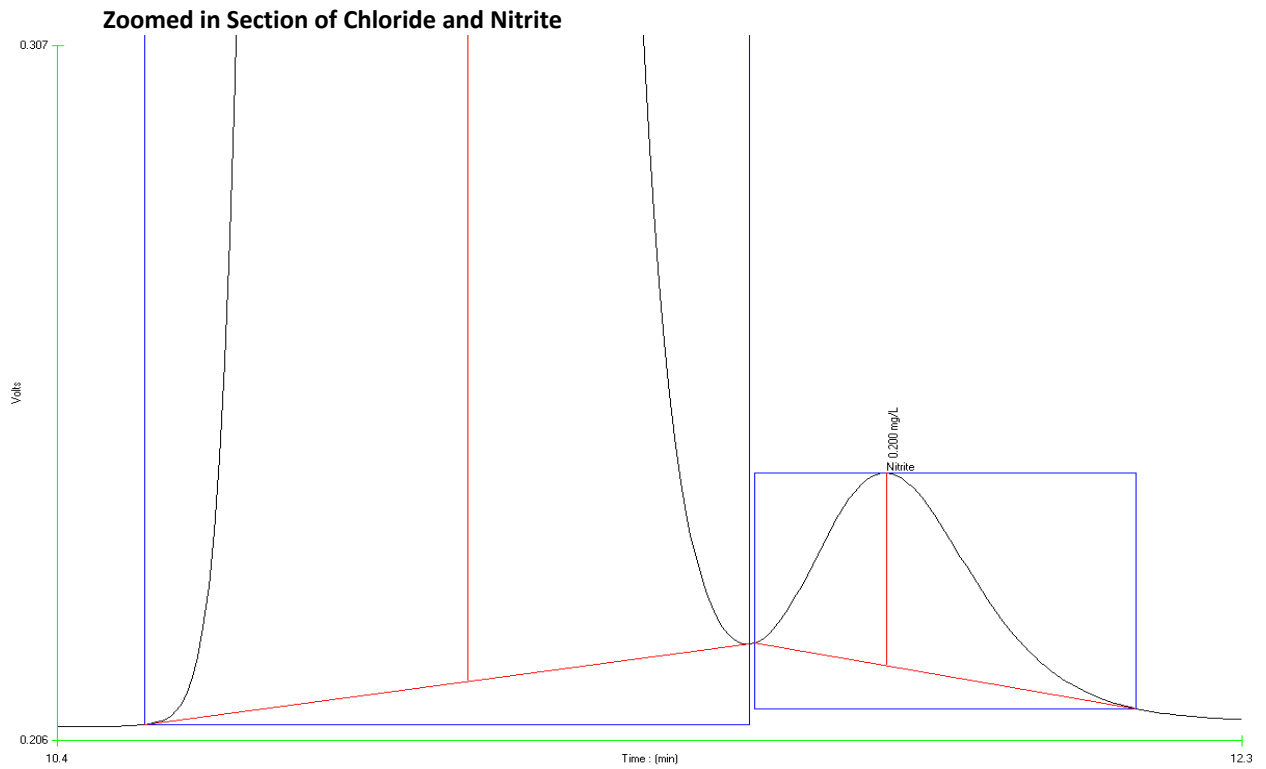
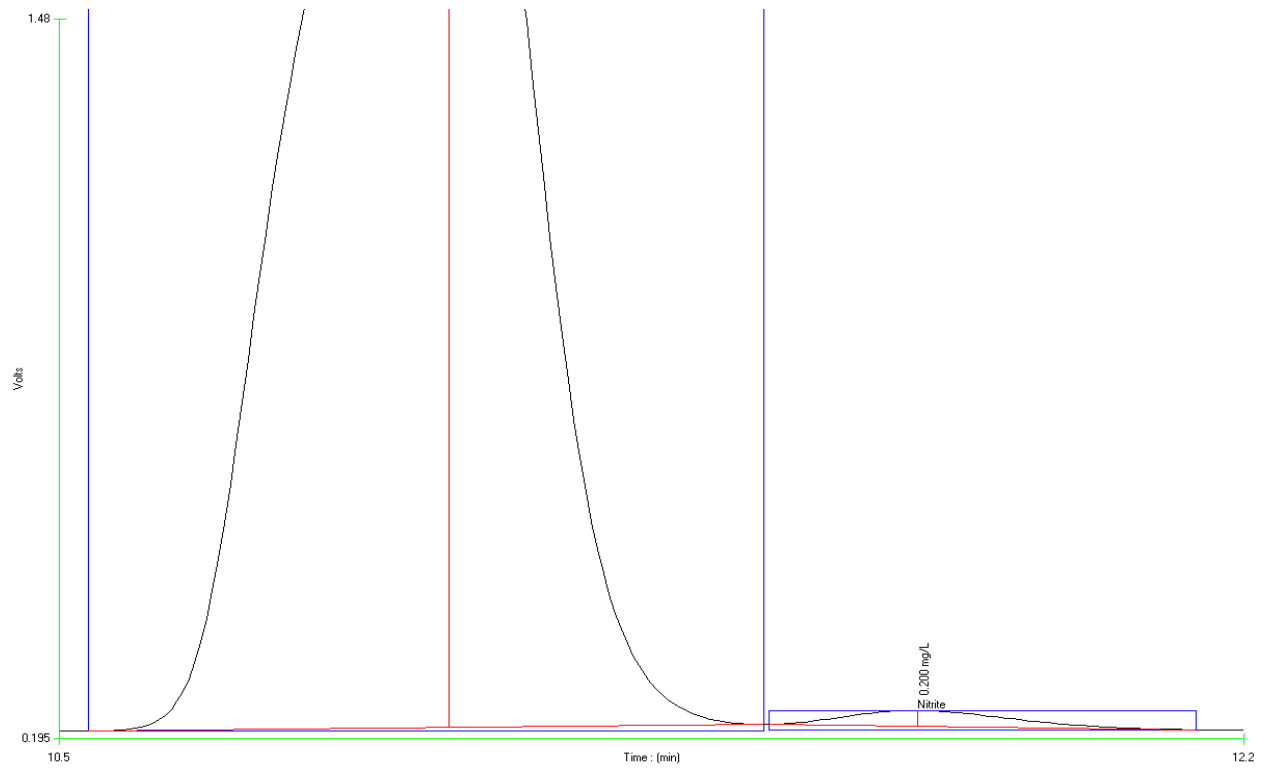


**Zoomed in Section of Chlorite and Bromate**



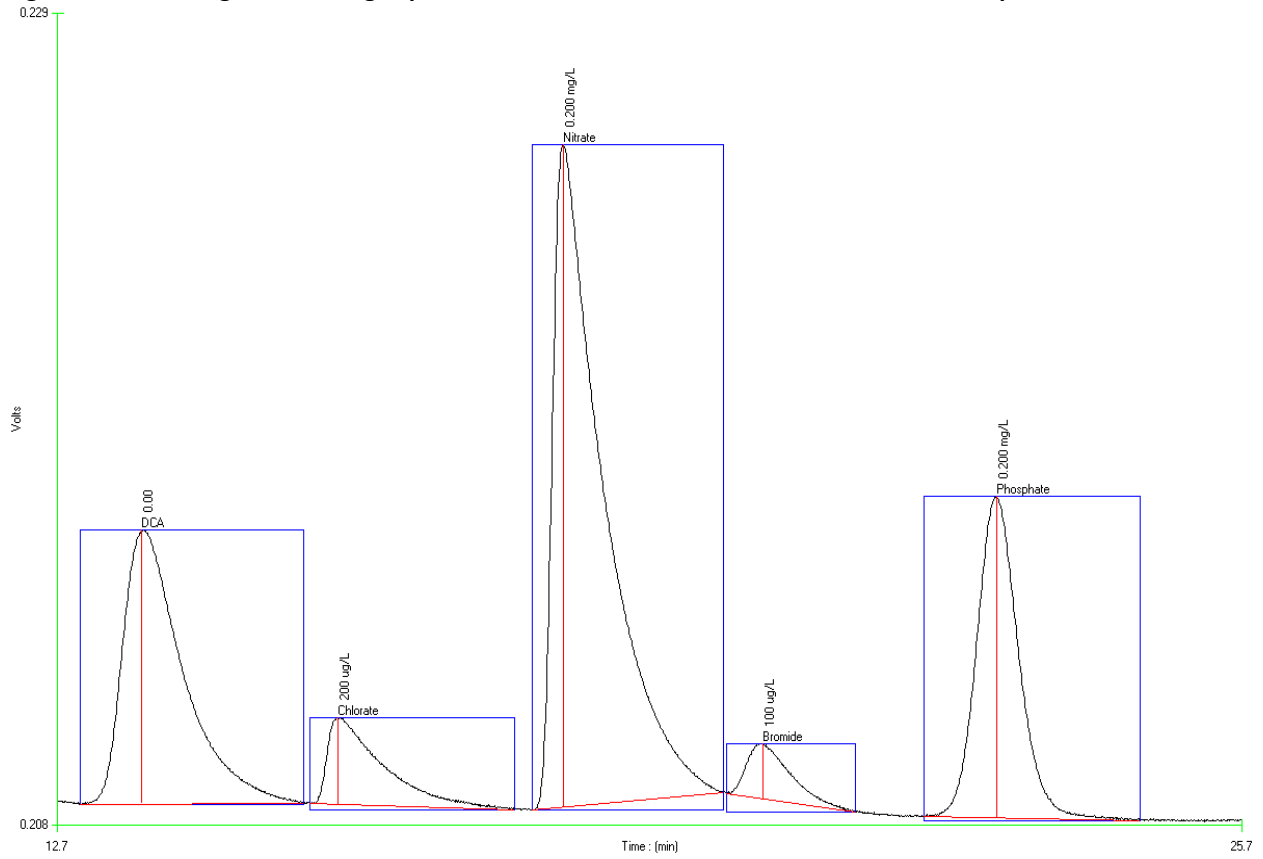
File Name: 11-20 cal mdl.omn  
Acq. Date: 20 November 2008

Figure 5. Chromatogram Showing Separation of Chloride and Nitrite.

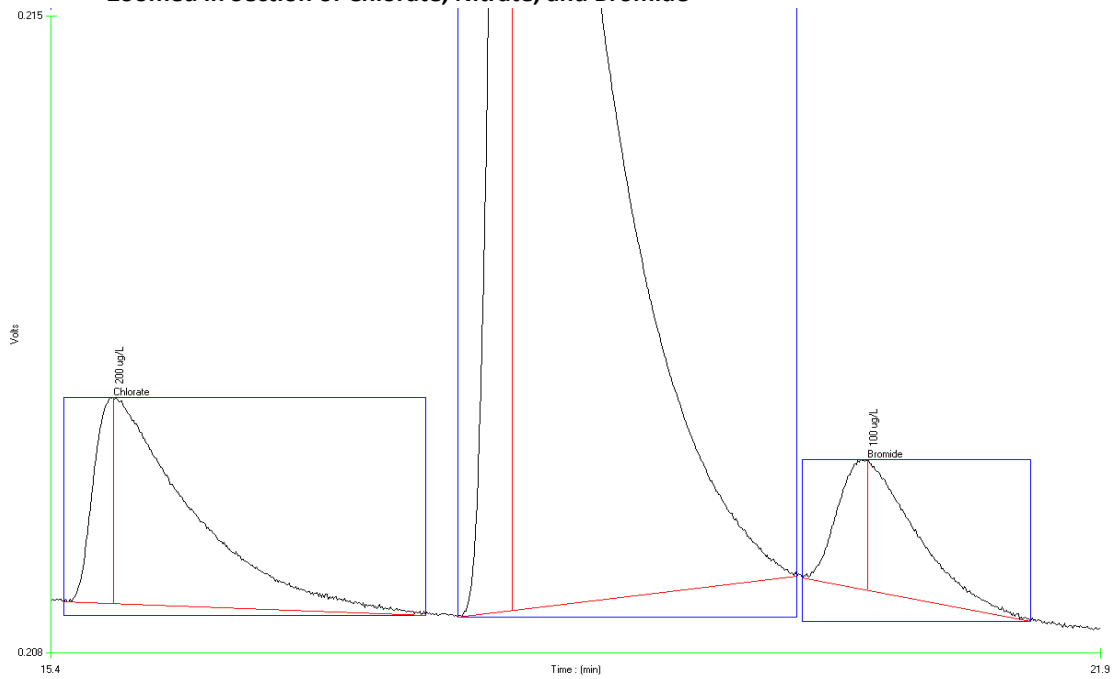


File Name: 11-20 cal mdl.omn  
Acq. Date: 20 November 2008

Figure 6. Chromatogram Showing Separation of DCA, Chlorate, Nitrate, Bromide and Phosphate.



Zoomed in Section of Chlorate, Nitrate, and Bromide



File Name: 11-20 cal mdl.omn  
Acq. Date: 20 November 2008



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
CINCINNATI, OHIO 45268

Lynn Egan  
legan@hach.com  
Applications Chemist  
Lachat Instruments  
P.O. Box 389, Mail Stop #1  
Loveland, CO 80539-0389

January 27, 2009

RE ATP Case No. D09-0002:

Per the terms of the Alternate Test Procedure (ATP) program, the Office of Ground Water and Drinking Water's Technical Support Center (OGWDW/TSC) has determined that QuikChem Method 10-540-00-1-C [11/24/2008] is an acceptable version of EPA Method 300.1, Part B. Subsequently, the method may be used for drinking water compliance monitoring under National Primary Drinking Water Regulations (NPDWR).

We appreciate your interest in the development of environmental monitoring methods. If you have any questions regarding the review of this alternate test procedure, please contact Steve Wendelken by e-mail at: [wendelken.steve@epa.gov](mailto:wendelken.steve@epa.gov) or by telephone at: 513-569-7491.

Sincerely,

A handwritten signature in cursive script that reads "Steve Wendelken".

Steven C. Wendelken, PhD.  
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[wendelken.steve@epa.gov](mailto:wendelken.steve@epa.gov)

# Perchlorate in Drinking Waters

2 to 100  $\mu\text{g ClO}_4^-/\text{L}$

## – Principle –

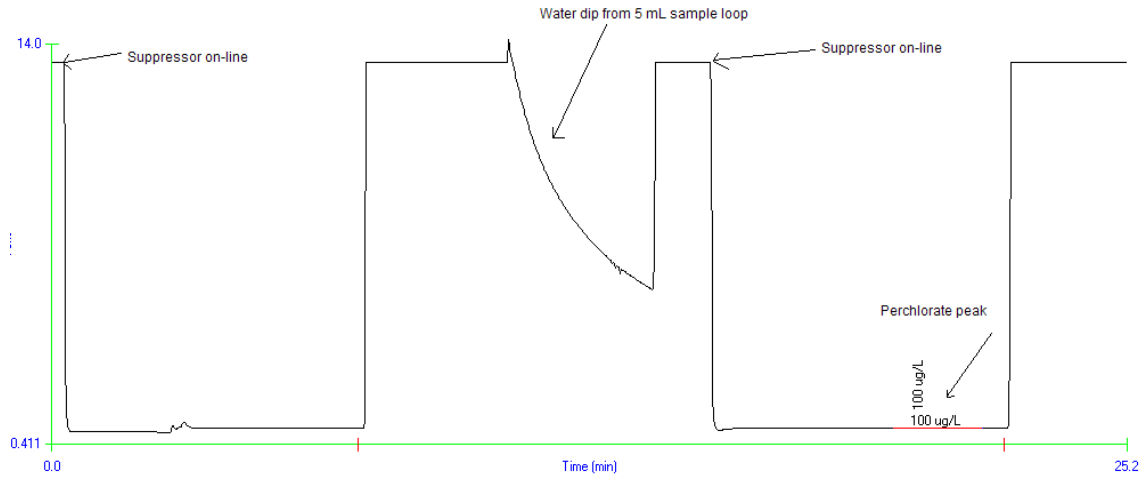
This is a chemically-suppressed, ion chromatography (IC) method. The anions are separated based on their affinities toward the stationary phase in the column. The eluent contains lithium hydroxide and 4-cyanophenol to provide excellent separation from excess levels of common matrix anions with minimal broadening of the perchlorate peak. A suppressor cartridge, packed with high-capacity cation exchange resin, chemically suppresses the background conductance of the eluent and, at the same time, converts the anionic species into species of higher conductance. The method provides results that are equivalent to those of USEPA Method 314.0.

## – Interferences –

1. Interferences can be caused by substances with retention times that are similar to and overlapping those of the analyte of interest. In particular, matrix levels of o-phosphate exceeding 15 mg-P/L may diminish the determined values for perchlorate due to peak overlap. Sample dilution and/or fortification can be used to solve many interference problems associated with overlapping retention times. However, sample dilution will typically affect the MDL and MRL values proportionally. Dilution of the eluent may be a useful alternative procedure.
2. Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.
3. High background levels of common matrix anions such as chloride, carbonate, and sulfate have the potential to cause false negative results by diminishing the conductivity response for perchlorate. This method contains support data to demonstrate full recovery of perchlorate at a fortified level of 5  $\mu\text{g/L}$  in a matrix containing chloride and sulfate up to 200 mg/L. This threshold must be determined by the individual analytical laboratory during the Initial Demonstration of Capability (IDC) and confirmed in each analysis batch using the Instrument Performance Check (IPC) Solution.
4. Pretreatment cartridges can be effective as a means to eliminate certain matrix interferences such as chloride and sulfate. With any proposed pretreatment, the analyst must verify that the target analyte is not affected by monitoring recovery after pretreatment and that no background contaminants are introduced by the pretreatment.
5. Close attention should be given to the potential for carry-over peaks from one injection, which have the potential to affect the proper detection of perchlorate in a subsequent sample injection. It is the responsibility of the user to confirm that no late eluting peaks have carried over into a subsequent analysis, thereby compromising the integrity of the analytical results.

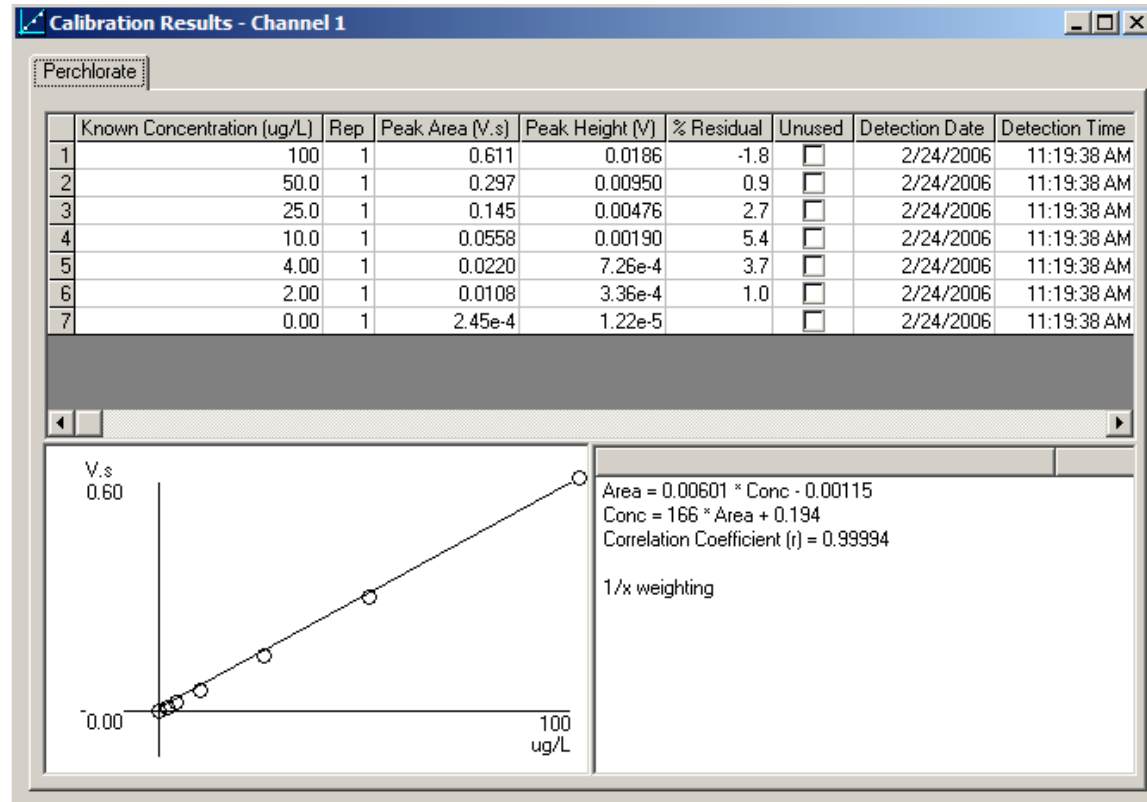
## – Special Apparatus –

\* This method is for use with **Omnion 3.0** only.



Screen capture of chromatogram for perchlorate using the QC8500.

### Calibration for Perchlorate





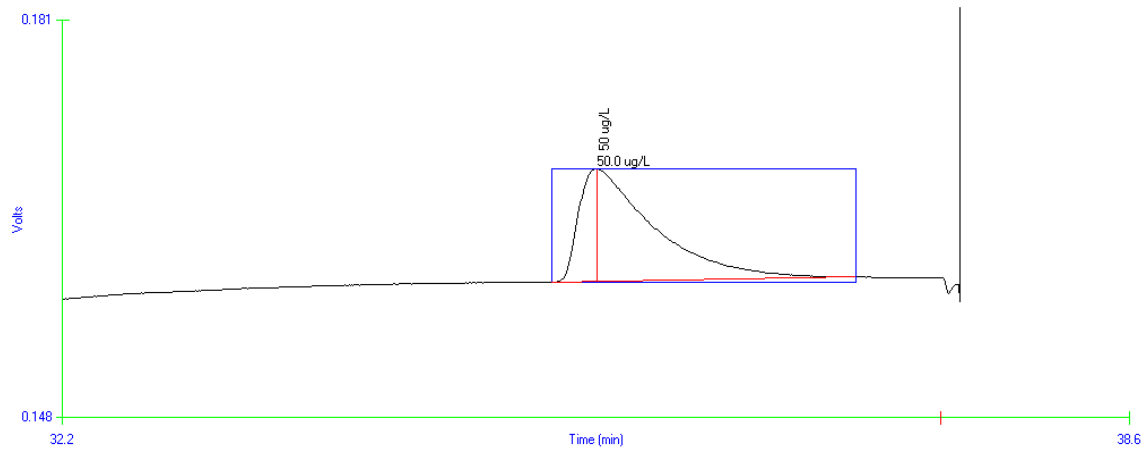
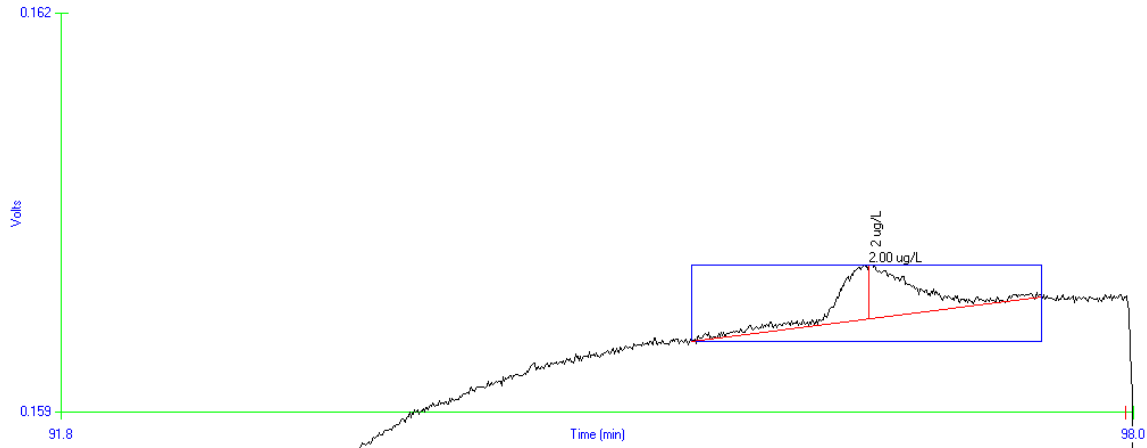


Figure 1: Chromatogram showing peak for 50 ug Perchlorate

Precision Study for Perchlorate using 50  $\mu\text{g ClO}_4^-/\text{L}$  standard

Replicate	Area	Calculated Concentration, $\mu\text{g ClO}_4^-/\text{L}$
1	0.004932	49.383
2	0.004895	49.013
3	0.004932	49.383
4	0.004922	49.284
5	0.004894	49.004
6	0.004955	49.615
7	0.004948	49.541
8	0.004944	49.499
9	0.004950	49.561
10	0.004934	49.402
	<b>Average</b>	<b>49.37</b>
	<b>Standard Deviation</b>	<b>0.214</b>
	<b>% RSD</b>	<b>0.43</b>



**Figure 2: Chromatogram showing peak for 2 ug perchlorate.**

Date acquired: 25Feb 06

**Method Detection Limit and Minimum Level using 2 µg ClO<sub>4</sub><sup>-</sup>/L standard**

Replicate	Absorbance	Calculated Concentration, mg N/L
1	0.0001810	1.9992
2	0.0001822	2.0112
3	0.0001684	1.8726
4	0.0002029	2.2167
5	0.0001887	2.0753
6	0.0001668	1.8573
7	0.0002016	2.2046
	<b>Average</b>	<b>2.034</b>
	<b>Standard Deviation</b>	<b>0.1433</b>
	<b>MDL (3.14*s)</b>	<b>0.45</b>
	<b>ML (3.18*MDL)</b>	<b>1.43</b>

**Spike Recovery in Drinking waters**

Sample	Unspiked Value, µg ClO <sub>4</sub> <sup>-</sup> /L	Spiked Value, µg ClO <sub>4</sub> <sup>-</sup> /L	Spike Level, µg ClO <sub>4</sub> <sup>-</sup> /L	% Recovery
DI	0.194	48.95	50	97.51
Tap 1	0.196	48.50	50	96.61
Tap 2	0.196	49.56	50	98.73
Tap 3	0.210	47.06	50	93.70

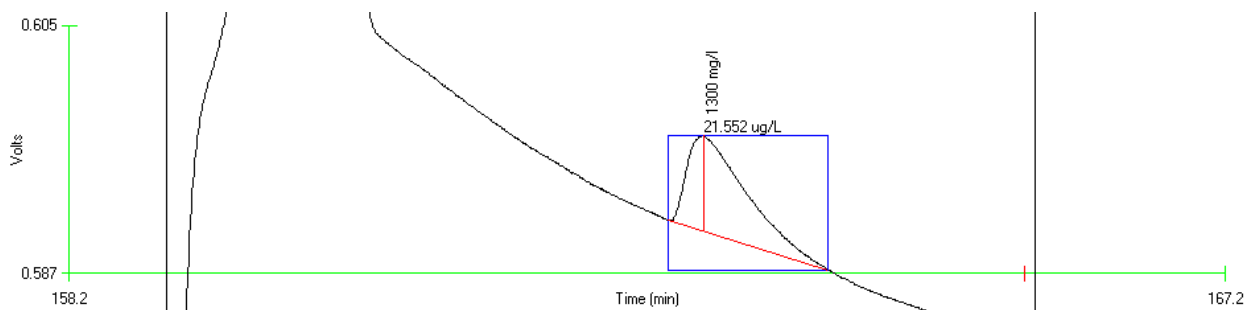
Spike recoveries of 93.70 – 98.73% were obtained in a variety of drinking water and wastewater matrixes.

### Matrix Conductivity Threshold (MCT)

The MCT was determined as outlined in Section 9.2.8. Initially experiments indicated the MCT was at a conductance of 9.8 ms/cm (See Table 1 and Figure 3). This was determined based on the peak area to height (A/H) ratio for 25 ppb  $\text{ClO}_4^-$  standards in samples containing increasing concentrations of the mixed anion standard (see section 9.2.8.6). However the recovery of a 2 ppb  $\text{ClO}_4^-$  standard (the minimum reporting level or MRL) at this established MCT was less than 70%. Therefore, the MCT was determined by measuring 2 ppb  $\text{ClO}_4^-$  standards in samples containing increasing concentrations of the mixed anion standard (see section 9.2.8.11). **The MCT determined in this manner for a 2 ppb  $\text{ClO}_4^-$  MRL was 4.1 ms/cm** (see Table 2 and Figure 4). The MCT was also determined for MRL standards of 4 and 5 ppb  $\text{ClO}_4^-$  (see Table 2).

**Table 1: MCT based on peak to height ratios**

Mixed anion mg/L	Conductance ms/cm	Peak Area	Peak Height	PDA/H ratio	$\text{ClO}_4^-$ ppb	PD A/H	Rec %
0	0.1073	0.291	0.011	26.45455	28.188	0%	113%
200	1.7325	0.269	0.009436	28.50784	26.121	8%	104%
300	2.544	0.254	0.008947	28.3894	24.616	7%	98%
400	3.295	0.263	0.008813	29.84228	25.483	13%	102%
500	4.12	0.254	0.008521	29.80871	24.619	13%	98%
600	4.835	0.252	0.008253	30.53435	24.488	15%	98%
800	6.305	0.241	0.007861	30.65768	23.46	16%	94%
1000	7.705	0.227	0.007393	30.70472	22.091	16%	88%
<b>1300</b>	<b>9.79</b>	<b>0.221</b>	<b>0.006939</b>	<b>31.84897</b>	<b>21.552</b>	<b>20%</b>	<b>86%</b>
1500	11.01	0.204	0.006287	32.44791	19.871	23%	79%
2000	14.155	0.185	0.005488	33.70991	18.032	27%	72%
2500	17.31	0.156	0.004731	32.974	15.356	25%	61%



**Figure 3: MCT test.** 25  $\mu\text{g ClO}_4^-/\text{L}$  standard containing 1300 mg/l of mixed anion standard with a conductance of 9.8 ms/cm.

File Name: MCT\_001a.omn

Acq. Date: 18 September 2006

**Table 2: The MCT based on the recovery of the MRL standard at a given conductance. The MCT is the conductance at which the recovery of the MRL falls between 70-130%**

Sample ID	Conductance ms/cm	ClO <sub>4</sub> <sup>-</sup> ppb	Rec %	Peak Area	Peak Height
2 ppb ClO <sub>4</sub> <sup>-</sup>	-----	1.923	96.2%	0.016	0.000773
200-2	1.7	1.959	98.0%	0.016	0.000626
300-2	2.5	1.587	79.4%	0.012	0.000591
400-2	3.3	1.47	73.5%	0.011	0.000538
<b>500-2</b>	<b>4.1</b>	<b>1.447</b>	<b>72.4%</b>	<b>0.011</b>	<b>0.000523</b>
4 ppb ClO <sub>4</sub> <sup>-</sup>	-----	3.754	93.9%	0.035	0.001486
300-4	2.5	3.139	78.5%	0.028	0.001227
400-4	3.3	3.228	80.7%	0.029	0.00123
500-4	4.1	2.94	73.5%	0.026	0.00115
<b>700-4</b>	<b>5.5</b>	<b>2.945</b>	<b>73.6%</b>	<b>0.026</b>	<b>0.0011</b>
800-4	6.3	2.563	64.1%	0.022	0.000988
1000-4	7.7	2.176	54.4%	0.018	0.000883
5 ppb ClO <sub>4</sub> <sup>-</sup>	-----	4.619	92.4%	0.044	0.001882
400-5	2.5	6.316	126.3%	0.062	0.001977
500-5	3.3	3.701	74.0%	0.034	0.001516
600-5	4.1	3.504	70.1%	0.032	0.001356
<b>700-5</b>	<b>5.5</b>	<b>3.726</b>	<b>74.5%</b>	<b>0.035</b>	<b>0.001379</b>
800-5	6.3	3.402	68.0%	0.031	0.001325
1000-5	7.7	2.987	59.7%	0.027	0.001138

**Note:** the sample ID format of ZZZ\_Y refers to the mixed anion concentration (ZZZ) and the ClO<sub>4</sub><sup>-</sup> concentration (Y). For example, 700-4 is sample containing 700 mg/l of the mixed anion standard and 4 ppb ClO<sub>4</sub><sup>-</sup>.

QuikChem Method 21-510-00-1-A

# Inorganic Anions in Beverages

0.2 to 10 mg F<sup>-</sup>/L

1.0 to 50 mg Cl<sup>-</sup>/L

0.2 to 10 mg NO<sub>3</sub><sup>-</sup>-N/L

4 to 200 mg HPO<sub>4</sub><sup>2-</sup>-P/L

4 to 200 mg SO<sub>4</sub><sup>2-</sup>/L

## – Principle –

This is a chemically-suppressed, ion chromatography (IC) method. The anions are separated based on their affinities toward the stationary phase in the column. A suppressor cartridge, packed with high-capacity cation exchange resin, chemically suppresses the background conductance of the eluent and, at the same time, converts the anionic species into species of higher conductance. The method provides results that are equivalent to those of USEPA method 300.0 Part A and those of ISO methods 10304-1 and 10304-2.

## – Interferences –

See Section 4 of the method for information on interferences.

## – Special Apparatus –

This method is for use with **Omnion 3.0** only.

**Table 1. Method Detection Limits for Inorganic Anions in Reagent Water**

Analyte	Known Conc. (mg/L)	Determined Conc. (mg/L)	Std. Dev. (mg/L)	MDL (mg/L)	PQL (mg/L)
Fluoride	0.20	0.19	0.012	0.037	0.117
Chloride	1.0	1.00	0.014	0.045	0.143
Nitrate-N	0.20	0.19	0.007	0.021	0.067
Phosphate-P	4.0	3.95	0.020	0.062	0.198
Sulfate	4.0	3.93	0.032	0.102	0.326

Note: The instrument was calibrated by including the MDL standard in the calibration model (Section 7.11). Seven replicates of a standard were analyzed. The MDL equals the standard deviation of the determined values for seven replicate runs multiplied by 3.142, corresponding to a 99% confidence value. The PQL (practical quantitation limit) equals the standard deviation multiplied by 10.

**Table 3. Single-Operator Precision of Standard Anions – (Standard B)**

(10 replicates of each sample were analyzed)

Analyte	Known Conc. (mg/L)	Determined Conc. (mg/L)	Std. Dev. (mg/L)	%RSD
Fluoride	6.0	5.97	0.026	0.43
Chloride	30.0	30.04	0.158	0.52
Nitrate-N	6.0	6.05	0.161	2.66
Phosphate-P	120	124.6	0.516	0.41
Sulfate	120	119.0	1.25	1.05

**Table 4. Single-Operator Precision of Standard Anions – (Standard C)**

(10 replicates of each sample were analyzed)

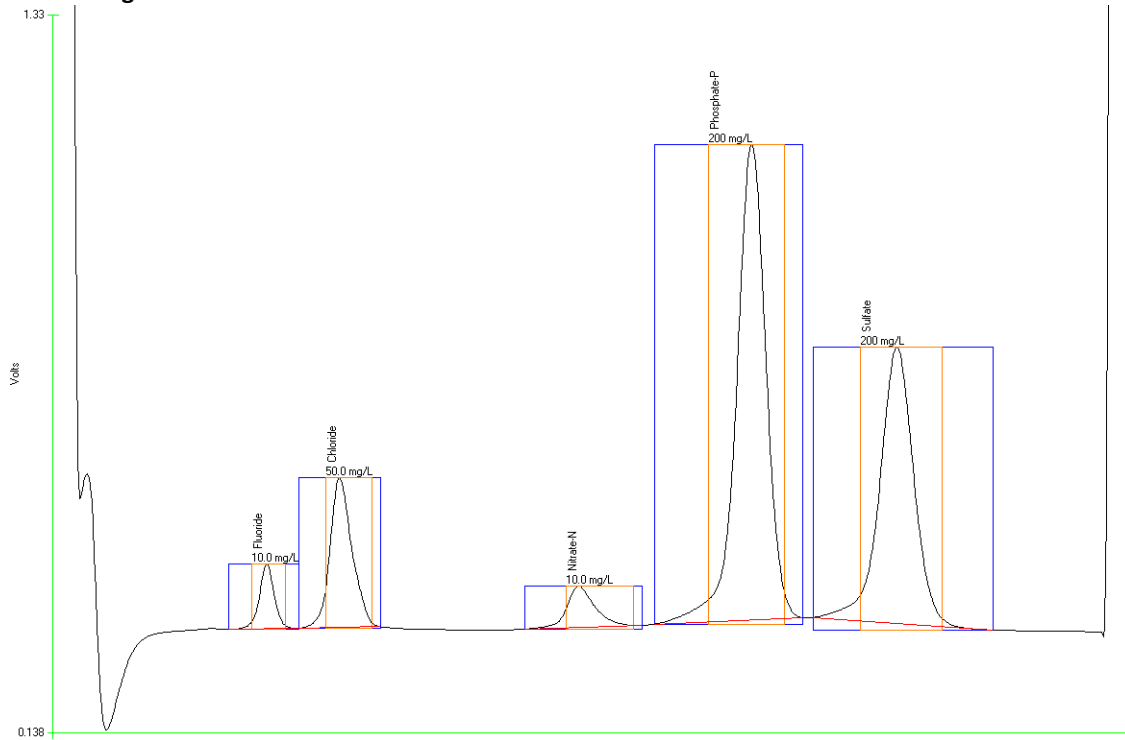
Analyte	Known Conc. (mg/L)	Determined Conc. (mg/L)	Std. Dev. (mg/L)	%RSD
Fluoride	2.0	1.94	0.007	0.38
Chloride	10.0	9.97	0.023	0.23
Nitrate-N	2.0	2.03	0.03	1.47
Phosphate-P	40.0	39.3	0.048	0.12
Sulfate	40.0	39.2	0.092	0.23

**Table 5. Single-Operator Precision of Beverage Sample (Coca-Cola)**

(10 replicates of each sample were analyzed)

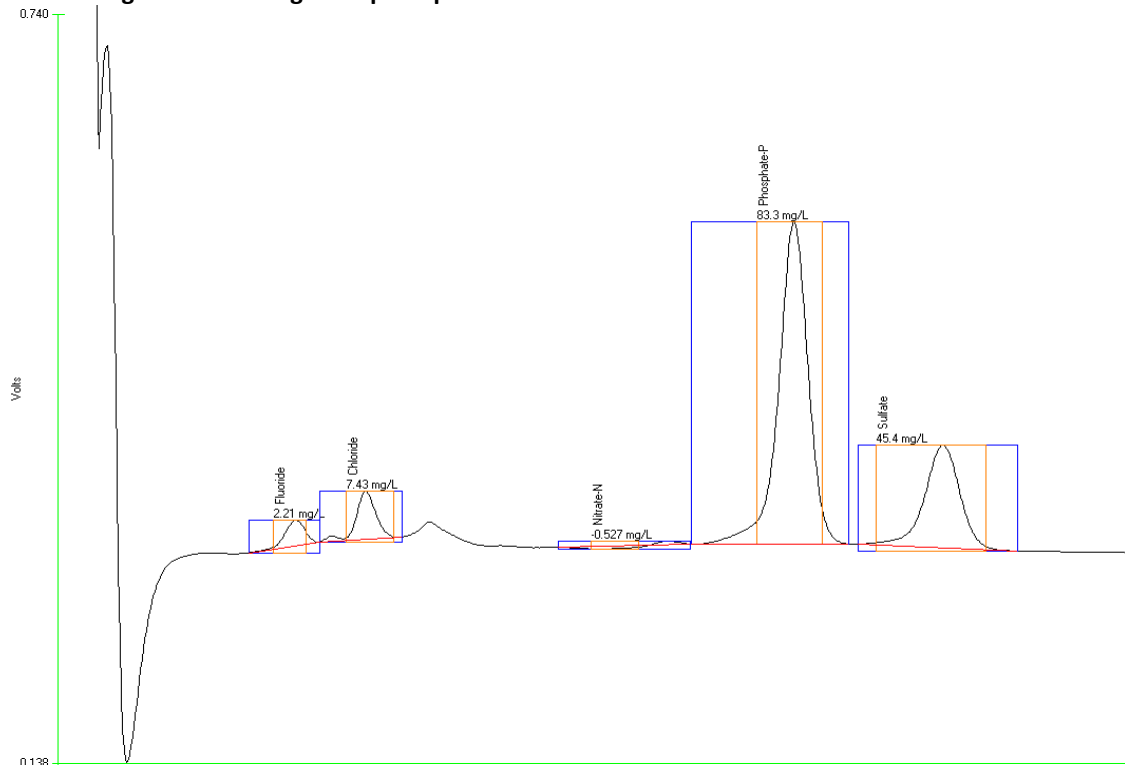
Analyte	Determined Conc. (mg/L)	Std. Dev. (mg/L)	%RSD
Fluoride	2.24	0.021	0.95
Chloride	7.41	0.093	1.25
Nitrate-N	ND	ND	ND
Phosphate-P	83.1	0.378	0.45
Sulfate	44.5	0.673	1.51

### Chromatogram of calibration standard A



File Name: 11-3 cal support.omn  
Acq. Date: 3 November 2008

### Chromatogram of Beverage Sample replicate



File Name: 11-3 bev.omn  
Acq. Date: 3 November 2008

# Determination of Mono- and Di-valent Cations in Waters by Ion Chromatography

## Non - Suppressed Conductivity Method

<b>Range 1:</b>	0.25 to 10 mg Li <sup>+</sup> /L 1.8 to 72 mg Na <sup>+</sup> /L 0.8 to 32 mg NH <sub>4</sub> <sup>+</sup> /L 1.6 to 64 mg K <sup>+</sup> /L 0.8 to 32 mg Mg <sup>2+</sup> /L 1.6 to 64 mg Ca <sup>2+</sup> /L
<b>Range 2:</b>	0.05 to 1.0 mg Li <sup>+</sup> /L 0.20 to 4.0 mg Na <sup>+</sup> /L 0.20 to 4.0 mg NH <sub>4</sub> <sup>+</sup> /L 0.20 to 4.0 mg K <sup>+</sup> /L 0.25 to 5.0 mg Mg <sup>2+</sup> /L 0.25 to 5.0 mg Ca <sup>2+</sup> /L
<b>Range 3:</b>	8.0 to 400 µg Li <sup>+</sup> /L 10 to 500 µg Na <sup>+</sup> /L 5.0 to 250 µg NH <sub>4</sub> <sup>+</sup> /L 20 to 1000 µg K <sup>+</sup> /L 12 to 600 µg Mg <sup>2+</sup> /L 25 to 1250 µg Ca <sup>2+</sup> /L

### -- Principle --

This is a non-suppressed ion chromatography (IC) method, for use with **Omnion 3.0** software. The stationary phase of the cation profiling columns is made of a silica-based material coated with polybutadiene/maleic acid copolymer to provide a weak cation-exchange phase. The resulting low exchange-capacity stationary phase is suitable for simultaneous determination of mono- and di-valent cations in waters. The eluent used is methanesulfonic acid. The separated cations are detected due to a drop in conductivity relative to the background conductivity of the eluent. The Inverse Polarity (makes the peaks appear positive) and Zero features of the conductivity detector are used. Different ranges are accommodated by the use of different sample loops. **This method is for use with Omnion 3.0.**



**Table 1. Detection Limits in Reagent Water Range 1**

Analyte	Known Conc.(mg/L)	Determined Conc.(mg/L)	Std. Dev. (mg/L)	MDL (mg/L)
Lithium	0.25	0.255	0.0033	0.05
Sodium	1.8	1.89	0.017	0.36
Ammonium	0.80	0.822	0.018	0.16
Potassium	1.6	1.60	0.071	0.32
Magnesium	0.8	0.775	0.022	0.16
Calcium	1.8	1.88	0.19	0.6

**Table 2. Single-Operator Precision of Determined Cations for Range 1**  
(ten replicates were analyzed)

Cation	Known Amount, mg/L	Determined Amount, mg/L	Std. Dev., mg/L	%RSD	% of known amount
Lithium	2.5	2.482	0.023	0.93	99.28
Sodium	18	17.46	0.35	2.0	97.0
Ammonium	8	7.86	0.28	3.56	98.25
Potassium	16	16.02	0.063	0.39	100.13
Magnesium	8	7.95	0.065	0.82	99.38
Calcium	16	15.86	0.26	1.64	99.13

**Table 3. Detection Limits in Reagent Water Range 2**

Analyte	Known Conc.(mg/L)	Determined Conc.(mg/L)	Std. Dev. (mg/L)	MDL (mg/L)*
Lithium	0.05	0.051	0.0004	0.01
Sodium	0.20	0.198	0.0015	0.04
Ammonium	0.2	0.209	0.0012	0.04
Potassium	0.2	0.26	0.005	0.04
Magnesium	0.25	0.25	0.0063	0.05
Calcium	0.25	0.23	0.017	0.053

\* Reported as 1/5<sup>th</sup> the analyte level used in the determination if calculation was less than this.

**Table 4. Single-Operator Precision of Determined Cations for Range 2**  
(ten replicates were analyzed)

<b>Cation</b>	<b>Known Amount, mg/L</b>	<b>Determined Amount, mg/L</b>	<b>Std. Dev., mg/L</b>	<b>%RSD</b>	<b>% of known amount</b>
Lithium	0.2	0.21	0.0008	0.40	105
Sodium	0.8	0.75	0.011	1.54	93.75
Ammonium	0.8	0.79	0.007	0.83	98.75
Potassium	0.8	0.84	0.007	0.86	1.05
Magnesium	1.0	0.99	0.012	1.2	99
Calcium	1.0	0.99	0.041	4.16	99

**Table 5: Spike Recoveries in Water Samples**

		<b>Lithium</b>	<b>Sodium</b>	<b>Ammonium</b>	<b>Potassium</b>	<b>Magnesium</b>	<b>Calcium</b>
<b>Tap Water</b>							
	<b>Unspiked (mg/L)</b>	0	3.64	0	0.74	1.68	overrange
	<b>Spiked (mg/L)</b>	1.75	8.0	1.86	4.64	3.7	overrange
	<b>% Recovery</b>	97.22	95.5	93.0	97.89	100.54	
<b>Spike Level</b>		1.8	4.0	2.0	4.0	2.0	4.0
<b>Distilled Water</b>							
	<b>Unspiked (mg/L)</b>	0	0.274	0	0	0	0
	<b>Spiked (mg/L)</b>	1.7	4.68	1.91	3.96	1.87	3.99
	<b>% Recovery</b>	94.44	109.5	95.5	99.0	93.5	99.75

**Table 6. Detection Limits in Reagent Water Range 3**  
(Seven replicates were analyzed)

<b>Analyte</b>	<b>Known Conc.(µg/L)</b>	<b>Determined Conc.(µg/L)</b>	<b>Std. Dev. (µg/L)</b>	<b>MDL (µg/L)</b>
<b>Lithium</b>	8.00	7.70	0.186	0.58
<b>Sodium</b>	10.0	11.3	0.458	1.44
<b>Ammonium</b>	5.00	5.47	1.11	3.49
<b>Potassium</b>	20.0	21.5	1.83	5.74
<b>Magnesium</b>	12.0	14.0	0.826	2.60
<b>Calcium</b>	25.0	23.0	2.37	7.44

**Table 7. Single-Operator Precision (Standard B) of Determined Cations for Range 3**  
(ten replicates were analyzed)

Cation	Known Amount, $\mu\text{g/L}$	Determined Amount, $\mu\text{g/L}$	Std. Dev., mg/L	%RSD	% of known amount
Lithium	200	202.4	0.70	0.35	101.2
Sodium	250	249.2	1.32	0.53	99.68
Ammonium	125	129.9	1.37	1.05	103.9
Potassium	500	507.1	2.64	0.52	101.4
Magnesium	300	297.8	2.74	0.92	99.27
Calcium	625	662.3	9.01	1.36	106.0



**Chromatogram showing separation of Standard C using the PN 2809 cation profiling column. Range 2.**  
Date acquired: 25 Oct 2004

## Organic Acids in Beverages

3 to 300 mg oxalic acid/L	3 to 300 mg succinic acid/L or lactic acid/L
3 to 300 mg citric acid/L	3 to 300 mg formic acid/L
3 to 300 mg tartaric acid/L	3 to 300 mg acetic acid/L
3 to 300 mg malic acid/L	3 to 300 mg fumaric acid/L
3 to 300 mg malonic acid/L	4.5 to 450 mg adipic acid/L

### – Principle –

This is a chemically-suppressed ion chromatography (IC) method. The polymer-based stationary phase is able to separate weakly acidic organic and inorganic compounds by an ion-exclusion mechanism. Chromatographic retention occurs as the neutral protonated form of the weak acid analyte permeates the intra-particle volume of the stationary phase, whereas dissociated ionic species are excluded, thereby passing through the column unretained. The eluent is dilute sulfuric acid. Within this eluent, the organic acids are dissociated to varying extents, which dictates their retention times. A suppressor cartridge, packed with high-capacity cation exchange resin in the tetramethylammonium form, improves the signal-to-noise ratio for conductivity detection in two ways. First, hydrogen ion from the eluent is replaced by tetramethylammonium cation, which chemically suppresses the background conductivity of the eluent by 60%, thus diminishing noise. Second, the eluent pH is raised to about 7, which increases the dissociation for weakly acidic analytes, thus increasing sensitivity.

### – Interferences –

See Section 4 of the method for information on interferences.

### – Special Apparatus –

This method is for use with **Omnion 3.0** only.

1.1 The method covers the determination of the following organic acid anions:

Oxalic acid	Citric acid
Tartaric acid	Malic acid
Malonic acid	Succinic or Lactic acid*
Formic acid	Acetic acid
Fumaric acid	Adipic acid

\*Either succinic or lactic acid can be analyzed since these two acids have the same retention time and will interfere with each other, see interferences.

1.2 The matrices applicable to this method are as follows: Wine, fruit juice and other beverages.

1.3 The single laboratory Method Detection Limits (MDL) for the above analytes are listed in Table 1. The MDL for a specified matrix may differ from those listed, depending upon the nature of the sample.

1.4 The range tested for each organic acid is as follows:

<u>Analyte</u>	<u>Range, mg/L</u>
Oxalic acid, Citric acid, Tartaric acid, Malic acid, Malonic acid, Succinic acid, Lactic acid, Formic acid, Acetic acid, Fumaric acid	3.0 to 300
Adipic acid	4.5 to 450

**Note:** The calibrated range for the method is 100. These ranges were based on a 50 µL sample loop.

1.5 This method is recommended for use only by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatogram.

1.6 When this method is used to analyze unfamiliar samples for any of the above analytes, analyte identification should be supported by spiking the suspected analyte into the sample matrix and then comparing the retention times obtained from successive chromatograms of the unknown sample and the spiked matrix.

**Table 1. Detection Limits in 7 mM Sulfuric Acid Matrix**  
(Seven replicates were analyzed)

Analyte	Known Conc. (mg/L)	Determined Conc. (mg/L)	Std Dev.	MDL (mg/L)	PQL (mg/L)
Oxalic acid	3	2.81	0.23	0.71	2.27
Citric acid	3	3.23	0.14	0.45	1.44
Tartaric acid	3	3.25	0.29	0.92	2.92
Malic acid	3	3.10	0.19	0.60	1.92
Malonic acid	3	3.08	0.34	1.07	3.40
Succinic acid	3	3.27	0.24	0.77	2.45
Lactic acid	3	2.29	0.32	0.90	3.20
Formic acid	3	3.11	0.16	0.50	1.60
Acetic acid	3	3.00	0.37	1.16	3.69
Fumaric acid	3	2.87	0.14	0.45	1.43
Adipic acid	4.5	3.57	1.06	3.34	10.64

**Note:** Seven replicates of a standard were analyzed. The MDL was calculated as the standard deviation of the determined values for seven replicate runs multiplied by 3.142, corresponding to a 99% confidence value. The PQL (practical quantitation limit) equals the standard deviation multiplied by 10.

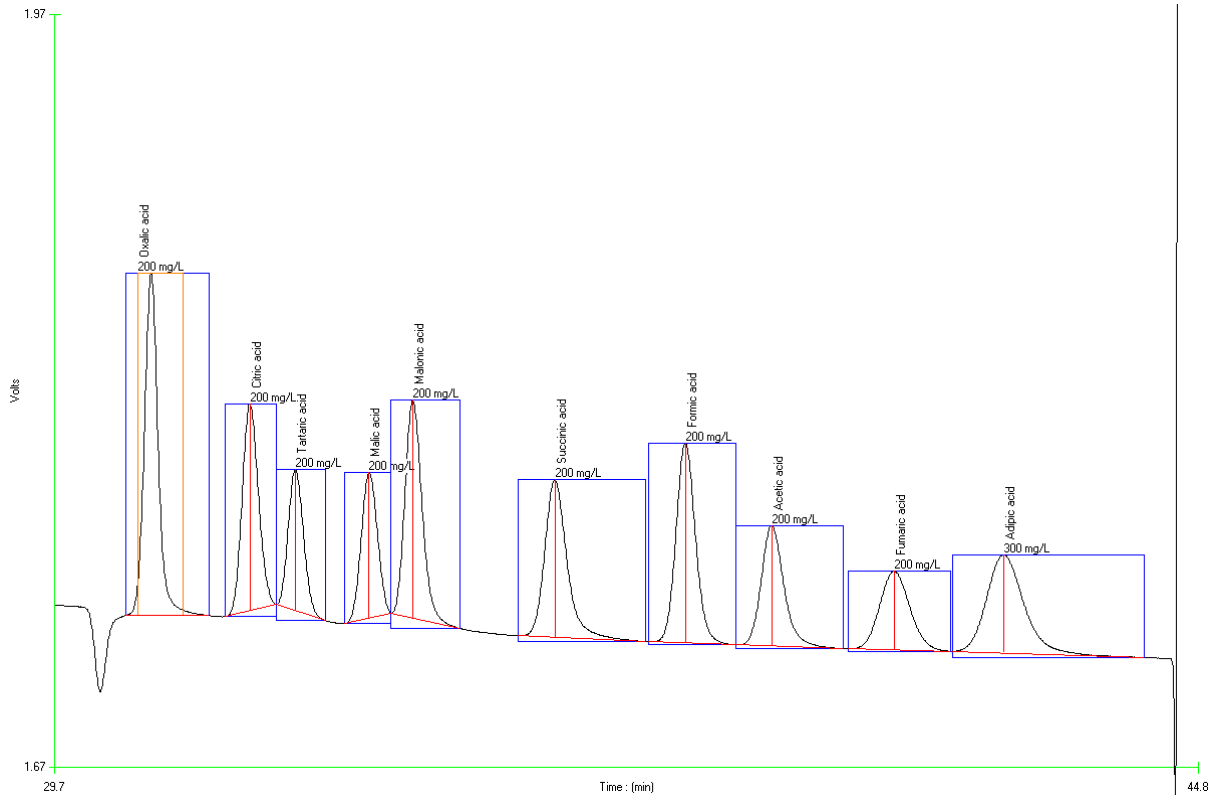
**Table 2. Single-Operator Precision for Organic Acids in 7 mM Sulfuric Acid Matrix**  
(Ten replicates were analyzed)

Organic acid	Known Amount mg/L	Determined amount mg/L	Std Dev. mg/L	Recovery %	RSD %
Oxalic acid	100	101.8	0.63	101.8	0.62
Citric acid	100	98.14	0.29	98.14	0.30
Tartaric acid	100	103.3	0.82	103.3	0.80
Malic acid	100	101.4	0.52	101.4	0.51
Malonic acid	100	98.61	0.22	98.61	0.23
Succinic acid	100	94.79	0.49	94.79	0.51
Formic acid	100	100.2	0.44	100.2	0.44
Acetic acid	100	96.3	0.41	96.3	0.43
Fumaric acid	100	101.1	0.57	101.1	0.56
Adipic acid	150	139.9	0.57	93.27	0.41

**Table 3. Single-Operator Precision for Organic Acids in 7 mM Sulfuric Acid Matrix**  
(Ten replicates were analyzed)

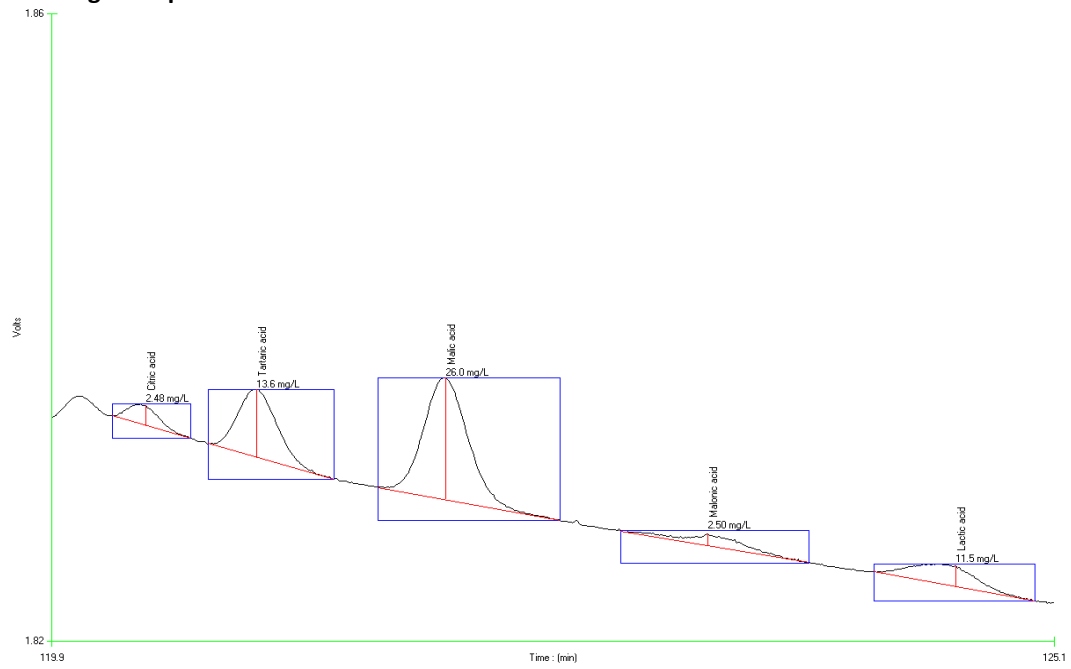
Organic acid	Known Amount mg/L	Determined amount mg/L	Std Dev. mg/L	Recovery %	RSD %
Oxalic acid	200	189.5	1.56	94.75	0.87
Citric acid	200	189.0	0.0	94.50	0.0
Tartaric acid	200	214.2	0.63	107.1	0.30
Malic acid	200	202.6	0.84	101.3	0.42
Malonic acid	200	187.6	0.52	93.80	0.28
Lactic acid	200	204.8	0.63	102.4	0.31
Formic acid	200	200.1	0.57	100.05	0.28
Acetic acid	200	192.6	0.84	96.3	0.44
Fumaric acid	200	203.7	0.67	101.8	0.33
Adipic acid	300	306.0	0.94	102.0	0.31

Chromatogram showing calibration standard separation using the QS-OA1 organic acids profiling column.

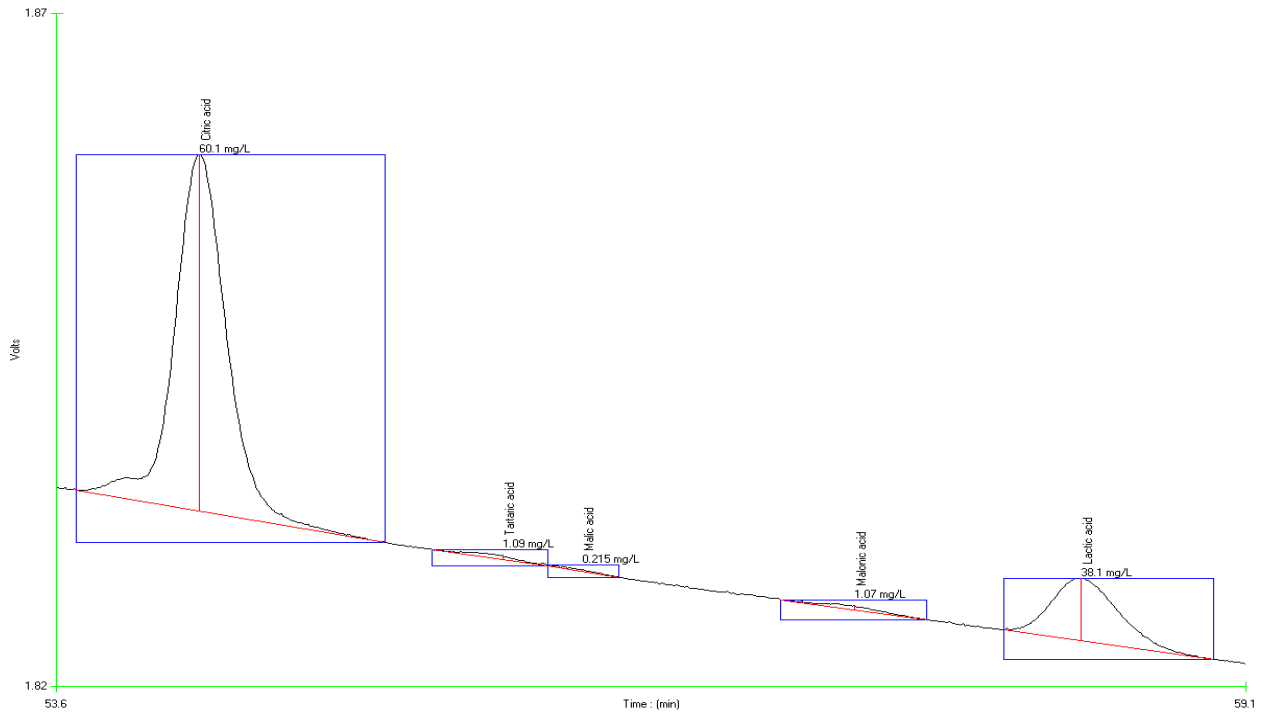


File name: 1-6 cal.omn  
Acq Date: 6 January 2009

### Beverage Samples



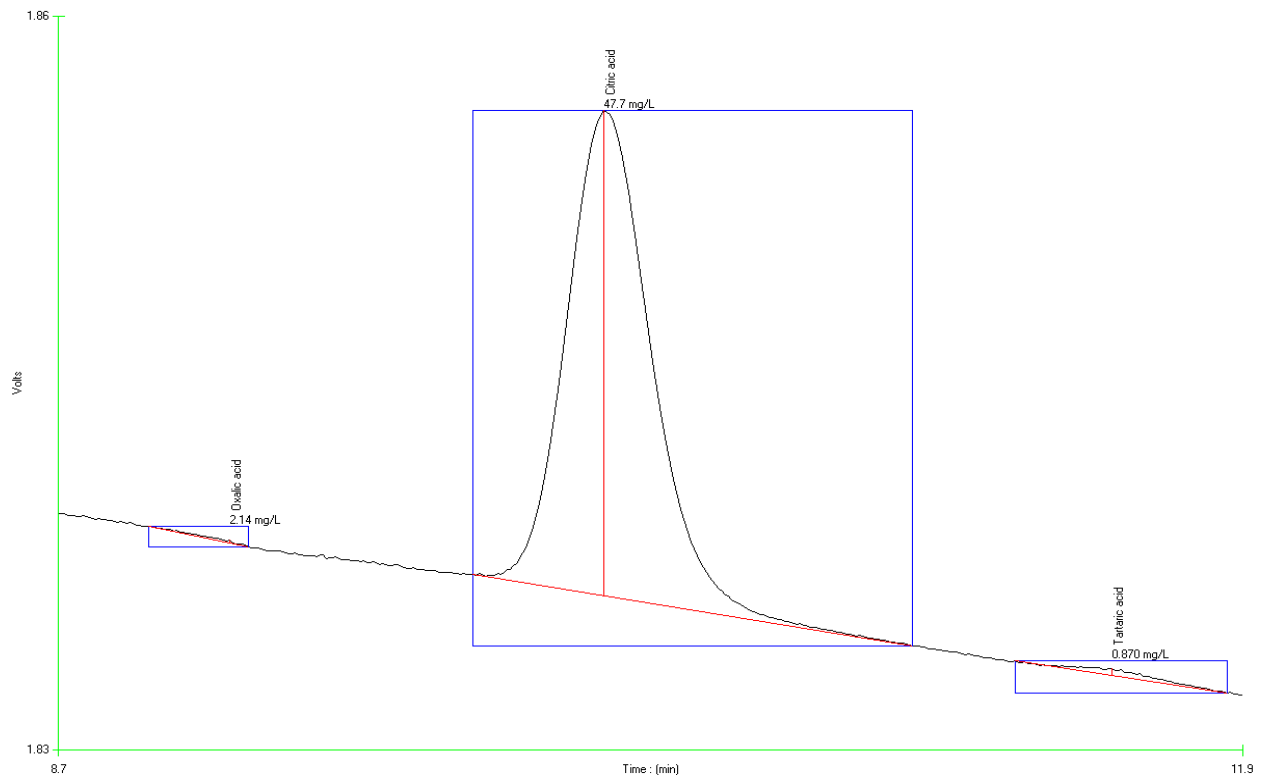
White Wine: Diluted 100-fold and filtered through 0.45 mM membrane filter  
File name: 1-16 samples.omn  
Acq Date: 16 January 2009



Orange Juice: Diluted 100-fold and filtered through a 0.45  $\mu$ M membrane filter

File name: 1-16 samples.omn

Acq Date: 16 January 2009



Lemonade: Diluted 100-fold and filtered through a 0.45  $\mu$ M membrane filter.

File name: 1-16 samples.omn

Acq Date: 16 January 2009



Lachat does custom method development! If you have a need you do not see addressed, please let us know. We would be happy to help you!