

Determination of Trace Fluoride, Chloride, and Sulfate in Lithium-Containing Borated Waters

Lillian Chen, Brian De Borba, and Jeffrey Rohrer
Thermo Fisher Scientific, Sunnyvale, CA, USA

Key Words

Dionex IonPac AS14 Column, Nuclear Power, Pressurized Water Reactors

Introduction

Nuclear power plants are an important source of electrical energy. For nuclear power plants using pressurized water reactors (PWRs), boron is added in the form of boric acid as a neutron absorber in the reactor coolant to control the nuclear reactivity in the primary reactor.¹ The pH of the coolant is also crucial in maintaining the system because a pH lower than 6.9 (at an elevated temperature of ~300 °C) can cause accelerated corrosion of the fuel rod cladding.² Lithium hydroxide is added to the coolant to achieve the desired pH to prevent crud buildup.³ Trace anionic impurities in borated waters can catalyze corrosion. Therefore, monitoring the concentrations of trace anions is important to reduce the risks associated with operation and to maintain plant reliability.

Ion chromatography (IC) is the most popular technique for the determination of anions in power plant waters because it provides the required sensitivity to detect trace-level concentrations.⁴⁻⁸ Dionex™ (now part of Thermo Scientific™) Application Note (AN) 114 describes the determination of trace anions in high-purity waters using direct injection and two-step isocratic elution.⁵ In AN 114, the Thermo Scientific Dionex IonPac™ AS14 Analytical Column is used with manually prepared borate eluent to effectively resolve the weakly retained analytes in high-purity waters.

For the determination of trace anions in borated waters, Dionex AN 185⁶ describes the use of a Dionex IonPac AS15 Analytical Column with electrolytically produced potassium hydroxide gradient, and Dionex Application Update (AU) 175⁷ explains the use of a Dionex IonPac AS22 Analytical Column with tetraborate eluent generated by in-line mixing of boric acid with electrolytically generated potassium hydroxide. Although AN 185 and AU 175 demonstrate the labor-saving benefit of using the electrolytically generated eluent, the borate from the samples eluted between the anions of interest, which can pose a challenge for accurate quantification. To minimize interference from the borate peak, matrix elimination is applied in AN 185, thus increasing method complexity.

Dionex AN 166⁸ demonstrates early elution of the borate peak, which was well resolved from all the other anions in the samples using a Dionex IonPac AS14 column and tetraborate eluent produced by combining boric acid with electrolytically generated potassium hydroxide. This study uses an approach similar to AN 114. The Dionex IonPac AS14 column and manually prepared eluent are used with slightly modified elution conditions to determine fluoride, chloride, and sulfate in simulated borated waters with added lithium. Similar to the results seen in AN 166, the borate peak elutes early and is well resolved from the target anions, therefore obviating the need for matrix elimination.

Goal

To develop a simple and robust IC method for the determination of trace fluoride, chloride, and sulfate in lithium-containing borated waters

Equipment

- Thermo Scientific Dionex ICS-5000 system*, including:
 - SP Single Pump
 - DC Detector/Chromatography Compartment
 - AS-AP Autosampler** with Sample Syringe, 5.0 mL (P/N 074308) and 8.5 mL buffer line assembly (P/N 075520)
- Dionex IonPac ATC-3, Anion Trap Column, 4 × 35 mm (P/N 079932) for 2 mm systems
- Autosampler Vial Kit, Polystyrene, 10 mL, with caps and blue septa (P/N 074228)
- Thermo Scientific Dionex Chromeleon™ Chromatography Data System software version 7.1

A Dionex ICS-5000 system can also be used.

**A Dionex AS-DV Autosampler can also be used for sample delivery.

Consumables

- 125 mL I-Chem™ Sterile Nalgene High-Density Polyethylene (HDPE) Bottles (Fisher Scientific P/N N411-0125)
- Corning™ Class A 100 mL PMP Volumetric Flask (Fisher Scientific P/N 10-210-640)

Reagents and Standards

- Deionized (DI) water, Type I reagent grade, 18 MΩ-cm resistance or better
- Sodium Fluoride, Powder, ACS reagent (Fisher Scientific P/N S299)
- Sodium Chloride, Crystalline, ACS reagent (Fisher Scientific P/N S271)
- Sodium Sulfate, Anhydrous, ACS reagent (Fisher Scientific P/N S421)
- Sodium Hydroxide Solution, 50% w/w (Fisher Scientific P/N SS254)
- Boric Acid, ACS reagent, ≥99.5% (Fisher Scientific P/N A74)
- Lithium Hydroxide, monohydrate, ≥98%, ACS reagent (Fisher Scientific P/N AC41332)

Table 1. Masses of compounds used to prepare 100 mL of 1000 mg/L anion standards.

Anion	Compound	Mass (g)
Fluoride	Sodium fluoride (NaF)	0.2210
Chloride	Sodium chloride (NaCl)	0.1648
Sulfate	Sodium sulfate (Na ₂ SO ₄)	0.1479

Conditions

Columns:	Dionex IonPac AG14 Guard, 2 × 50 mm (P/N 046138)			
	Dionex IonPac AS14 Analytical, 2 × 250 mm (P/N 046129)			
Eluents:	A: 100 mM boric acid/75 mM sodium hydroxide B: DI water			
Eluent Conditions:	Time (min)	A (%)	B (%)	Comment
	0.00	10	90	Inject
	4.00	10	90	—
	4.01	65	35	Step to stronger eluent
	8.50	65	35	—
	8.51	10	90	Equilibrate eluent for next injection
	15.00	10	90	—
Flow Rate:	0.5 mL/min			
Inj. Volume:	1 mL			
Temperature:	25 °C (upper compartment) 30 °C (lower compartment)			
Detection:	Suppressed conductivity, Thermo Scientific Dionex ASRS™ 300 Anion Self-Regenerating Suppressor, 55 mA, external water mode			
System Backpressure:	~2170 psi			
Background Conductance:	1.3–3.4 µS			
Noise:	~1.2 nS/min peak-to-peak at the first step, ~8.4 nS/min peak-to-peak at the second step			
Run Time:	15 min			

Preparation of Solutions and Reagents

Use high-quality water (≥18 MΩ-cm resistivity) to prepare all the eluent solutions, standards, and sample solutions. Filter through a 0.2 µm porosity nylon filter under vacuum to remove particulates and reduce dissolved air. Keep the eluent solution blanketed under 34.5 kPa (5 psi) of helium or nitrogen at all times to reduce carbon dioxide intrusion.

Do not use glass containers. High-quality polystyrene and polypropylene vessels must be rinsed at least three times with filtered DI water, soaked overnight, and rinsed again before use. Wear Class 100 Nitrile gloves when preparing solutions.

Stock Anion Standard Solution

Prepare 1000 mg/L standards for each of the anions of interest by dissolving the corresponding mass of dried salt in DI water and bringing to a final volume of 100 mL, as shown in Table 1. Standards are stable for at least one month when stored at 4 °C. Store the 1000 mg/L stock standards in 125 mL I-Chem Sterile Nalgene Bottles.

Mixed Standard Solution

Prepare appropriate mixed standards from the 1000 mg/L stock standards. Select a range of analyte concentrations close to the expected analyte concentrations in the samples. Working standards of concentrations <100 µg/L must be prepared daily.

Eluent Solution

Eluent A: 100 mM boric acid/75 mM sodium hydroxide

Prepare Eluent A with a high-purity boric acid reagent. Dissolve 6.18 g of boric acid in 900 mL of DI water, add 6.00 g of 50% sodium hydroxide, and dilute to 1 L. Transfer this solution to an eluent container and vacuum degas for 5 min.

Note: Care must be taken to minimize air contact with the eluent; absorbed carbon dioxide will change the eluent characteristics. Keep the eluent blanketed with helium or nitrogen gas to prevent atmospheric carbon dioxide from entering.

200 mM Sodium Hydroxide (Dionex IonPac ATC-3 Regeneration Solution)

Weigh 990 g of DI water into an eluent reservoir bottle. Degas the water for approximately 5 min. Tare the bottle on the balance and add 16.0 g 50% sodium hydroxide directly to the bottle and swirl to mix. Quickly transfer the eluent reservoir bottle to the instrument and pressurize it with helium.

1000 mg/L Lithium Stock Solution

Dissolve 0.6044 g lithium hydroxide monohydrate in 100 mL DI water. Store this solution in a 125 mL high-density polyethylene (HDPE) container.

Simulated Boric Acid Treated Samples

Samples with a boron concentration in the range of 1000–2500 mg/L with added lithium in the concentration range of 1.8–5.0 mg/L were analyzed in this study.

Prepare the simulated boric acid/lithium hydroxide sample by adding an appropriate amount of boric acid solid and 1000 mg/L lithium stock solution to a 100 mL PMP Class A volumetric flask, then dilute to a final volume of 100 mL with DI water. Store the samples in 125 mL HDPE containers at 4 °C. Table 2 shows the masses of boric acid and 1000 mg/L lithium stock solution used to prepare the simulated samples.

Table 2. Amounts of boric acid and lithium stock solution used to prepare 100 mL of surrogate matrices.

Matrix Composition	Mass of Boric Acid (g)	Mass of Lithium Stock Solution* (g)
1000 mg/L B + 1.8 mg/L Li	0.5719	0.18
2000 mg/L B + 4.0 mg/L Li	1.1438	0.40
2500 mg/L B + 5.0 mg/L Li	1.4296	0.50

*1000 mg/L lithium stock solution

System Preparation and Configuration

Figure 1 shows the schematic diagram of the system configuration.

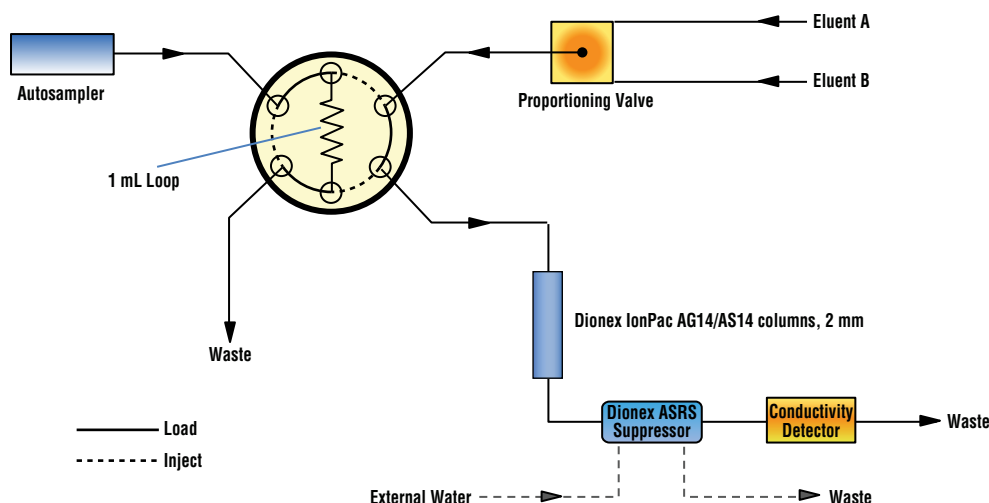


Figure 1. Schematic diagram of the system configuration.

Connect the Dionex IonPac ATC-3 Anion Trap Column for 2 mm systems to the outlet of the gradient pump. Condition it for 30 min with the strongest eluent that will be used during the two-step gradient program (65 mM boric acid/48.75 mM [65% Eluent A:35% Eluent B]) and direct the outlet to waste. Then connect the Dionex IonPac ATC-3 Anion Trap Column to the eluent line that leads to the injection valve. (For more information on Dionex IonPac ATC-3 Anion Trap Column operation, consult the product manual, Document No. 032697.)

Prepare the Dionex ASRS 300 suppressor for use by hydrating the internal membrane. Push 3 mL of DI water through the Eluent Out port and 5 mL of DI water through the Regen In port. Allow the suppressor to sit for 20 min to ensure complete hydration before installing it in the system. Configure the pressurized water reservoirs to supply external water for suppressor regeneration. Use at least two 4 L bottles plumbed in tandem to ensure uninterrupted external water delivery. Fill the reservoirs with DI water and apply 5–15 psi to the reservoir to deliver DI water through the regenerant channel. Ensure that the cap of the reservoir is sealed tightly. (For more information on installation and operation of a Dionex ASRS 300 suppressor, consult the product manual, Document No. 031956.)

Install and configure the Dionex AS-AP Autosampler in Push Mode. Follow the instruction in the Dionex AS-AP Autosampler Operator's Manual (Document No. 065361) to calibrate the sample transfer line to ensure accurate and precise sample injections. Due to the large sample injection volumes in this application, a sample syringe size of 5 mL must be installed and a buffer line assembly of 8.2 mL size is required.

Prepare a 1 mL sample loop by measuring approximately 86.5 in. (220 cm) of 0.030 in. i.d. tubing. Verify the volume of the loop by first weighing the empty tubing, then fill the tube with DI water, reweigh the filled tube, and calculate the volume. The total sample volume must be 1 mL $\pm$ 5%. Install the sample loop on the injection valve in the lower compartment of the DC. Flush the syringe, buffer line, and sample transfer line extensively to minimize contamination. Refill the wash reservoir with fresh DI water at least once every two days.

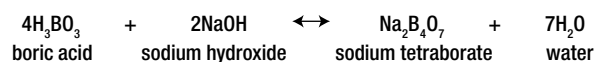
Install the Dionex IonPac AG14 Guard (2 $\times$ 50 mm) and the Dionex IonPac AS14 Analytical (2 $\times$ 250 mm) columns in the lower compartment of the DC. Connect the columns and suppressor using 0.005 in. i.d. tubing. Keep the lengths of the connecting tubing to a minimum. Condition the columns first with 65 mM boric acid/48.75 mM sodium hydroxide (65% Eluent A:35% Eluent B) for approximately 60 min. Equilibrate the columns with 10 mM boric acid/7.5 mM sodium hydroxide (Eluent A:Eluent B, 10:90) for at least five h to establish a stable background conductivity for the trace analysis.

Analyze a system blank by running the gradient program without injection. An equilibrated system has a background conductance of $\sim$ 1.3 μ S at the first-step elution and $\sim$ 3.4 μ S at the second-step elution. Analyze the reproducibility of at least three injections of a high-purity DI water blank to ensure that the Dionex AS-AP Autosampler is properly configured and there is no contamination.

Results and Discussion

Separation

The eluent used in this study was a mixture of sodium tetraborate and sodium hydroxide. The tetraborate eluent was made by a chemical reaction between boric acid and sodium hydroxide:



Producing tetraborate eluent by mixing boric acid and sodium hydroxide gives better eluent quality than the traditional method of dissolving sodium tetraborate in water, due to the purity of the chemicals available. The ratio of boric acid to sodium hydroxide was optimized to obtain the best resolution of carbonate from chloride.⁴ The eluent program started with a low eluent concentration to separate fluoride, chloride, and other weakly retained anions; then a step change to a higher concentration eluent was applied to accelerate the elution of the strongly retained anions, including sulfate. The total baseline shift between the eluent step change is typically 1.5 μ S.

The analytes of interest were eluted upon the steady baseline to ensure accurate quantification. The run time was also reduced by selecting a flow rate of 0.5 mL/min without significant loss in peak resolution.

A system blank and a DI water blank are shown in Figure 2, panels A and B, respectively. The typical background conductance ranges from 1.1 to 1.8 μS at the lower eluent concentration and from 2.6 to 3.2 μS at the higher eluent concentration. However, the background conductance gradually increases, due to exhaustion of the Dionex IonPac ATC-3 Anion Trap Column. Depending on the purity of the eluent and the degree of CO_2 intrusion, the Dionex IonPac ATC-3 Anion Trap Column used under the suggested experimental conditions can be effective from 10 to 14 days. A background conductance $>5 \mu\text{S}$ at the higher eluent concentration is an indication that the Dionex IonPac ATC-3 Anion Trap Column requires regeneration (refer to the Dionex IonPac ATC-3 Anion Trap Column Product Manual, Document No. 032697, for instruction).

In this study, an eluent containing 100 mM boric acid/75 mM sodium hydroxide was used to replace the two separate eluent solutions (9 mM boric acid/6.75 mM sodium hydroxide and 40 mM boric acid/30 mM sodium hydroxide) that are described in AN 114. With the more concentrated eluent, it is possible to modify the eluent condition by proportioning it with different amounts of water, thereby creating slight variations in concentration to adapt to different systems. The result is a reduction in manually prepared eluent and an increase in operational flexibility. The Dionex ICS-5000 system provides precise delivery of the eluent through the proportioning valve to deliver good retention time reproducibility of the target analytes.

To achieve sufficient sensitivity at low analyte concentrations while keeping the method simple and robust, a large direct-injection volume of 1 mL for the 2 mm column separation was used. Larger injection volumes may be used, but run times will be extended 1 min for each additional 0.75 mL injected. External water regeneration was chosen over the recycle mode to reduce baseline noise. Figure 3 shows a trace anion standard mixture prepared in DI water. Fluoride is well resolved from the system void and from the organic acids (glycolate, acetate, and formate). Sufficient resolution of chloride and sulfate from other anions is also obtained.

Column: Dionex IonPac AG14/AS14, 2 mm
 Eluent: 10 mM boric acid/7.5 mM NaOH, 0–4 min, step to 65 mM boric acid/48.75 mM NaOH at 4.01 min, 65 mM boric acid/48.75 mM NaOH, 4.01–8.50 min, step to 10 mM boric acid/7.5 mM at 8.51 min, 10 mM boric acid/7.5 mM, 8.51–15 min
 Flow Rate: 0.5 mL/min
 Inj. Volume: 1 mL
 Temperature: 30 °C
 Detection: Suppressed conductivity, Dionex ASRS 300 suppressor, 2 mm, 55 mA, external water mode

Peaks: 1. Acetate
 2. Formate
 3. Carbonate

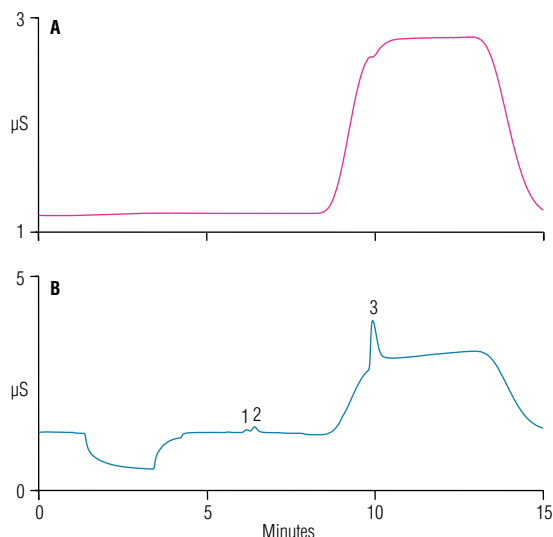


Figure 2. Representative chromatograms of (A) the system blank and (B) the DI water matrix blank.

Column: Dionex IonPac AG14/AS14, 2 mm
 Eluent: 10 mM boric acid/7.5 mM NaOH, 0–4 min, step to 65 mM boric acid/48.75 mM NaOH at 4.01 min, 65 mM boric acid/48.75 mM NaOH, 4.01–8.50 min, step to 10 mM boric acid/7.5 mM at 8.51 min, 10 mM boric acid/7.5 mM, 8.51–15 min
 Flow Rate: 0.5 mL/min
 Inj. Volume: 1 mL
 Temperature: 30 °C
 Detection: Suppressed conductivity, Dionex ASRS 300 suppressor, 2 mm, 55 mA, external water mode

Peaks: 1. Fluoride 5.0 $\mu\text{g/L}$ 7. Carbonate —
 2. Glycolate 10 8. Nitrate 10
 3. Acetate — 9. Phosphate 10
 4. Formate — 10. Sulfate 5.0
 5. Chloride 10 11. Oxalate 10
 6. Nitrite 10

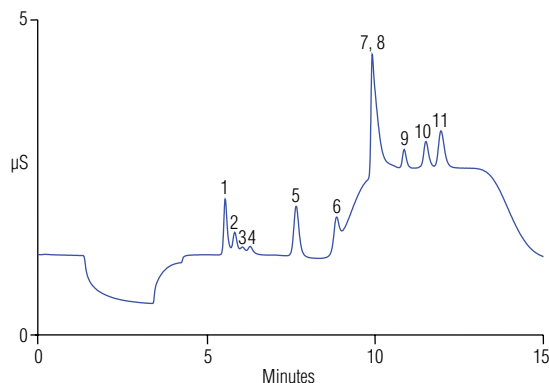


Figure 3. Separation of the trace anion standard.

Table 3. Calibration data and estimated method detection limits of fluoride, chloride, and sulfate.

Anion	Range (µg/L)	Coefficient of Determination (r^2)	MDL ^a in DI Water (µg/L)	MDL ^a in Borated Water ^b (µg/L)
Fluoride	0.1–3.0	0.9994	0.021	0.048
Chloride	0.3–10	0.9995	0.094	0.19
Sulfate	0.3–10	0.9999	0.11	0.31

^aMDL = (SD) × (t_f), where (t_f) is the Student's t value for a 99% confidence level ($t = 3.14$ for seven replicate injections)

^b2500 mg/L boron plus 5.0 mg/L lithium simulated sample

Calibration and Method Detection Limits (MDLs)

Calibration curves with five concentration levels were obtained with fluoride, chloride, and sulfate standards prepared in DI water. The results yielded linear relationships of peak area to concentration with coefficients of determination (r^2) greater than 0.999. The MDLs for fluoride, chloride, and sulfate were established from seven injections of a 0.2 µg/L fluoride, 1 µg/L chloride, and 1 µg/L sulfate standard mixture in DI water and seven injections of a 2500 mg/L boron plus 5.0 mg/L lithium simulated sample, respectively. The results of the calibration and MDLs are summarized in Table 3.

Analysis of Simulated Borated Waters

This method is applicable to high-purity power plant waters containing up to 2500 mg/L boron and 5 mg/L lithium. Figure 4A shows the chromatogram of a 2500 mg/L boron plus 5 mg/L lithium simulated sample. The large borate matrix peak that begins at 1.7 min returns to the baseline before the first analyte of interest elutes. No significant differences in peak efficiencies or retention times are observed for the peaks detected in the matrix blank compared to the standard prepared in DI water. Table 4 summarizes the results of fluoride, chloride, and sulfate in simulated samples containing 1000–2500 mg/L boron plus 1.8–5.0 mg/L lithium.

The simulated sample of 2500 mg/L boron plus 5 mg/L lithium spiked with low concentrations of common anions was also examined. As shown in Figure 4B, the anions of interest are all resolved from other anions; therefore, no interference occurs with the quantification.

Recoveries and Precision Study

To validate this method, recoveries of fluoride, chloride, and sulfate from three different simulated samples that contained 1000–2500 mg/L boron plus 1.8–5.0 mg/L lithium were studied. Each sample was spiked with anion concentrations comparable to the native concentrations present in the simulated samples. Three replicates of the spiked samples were analyzed and recoveries for the three anions in the three different simulated samples were in the range of 92–109% (Table 5). Figure 4C shows the chromatogram of the spiked 2500 mg/L boron plus 5 mg/L lithium simulated sample.

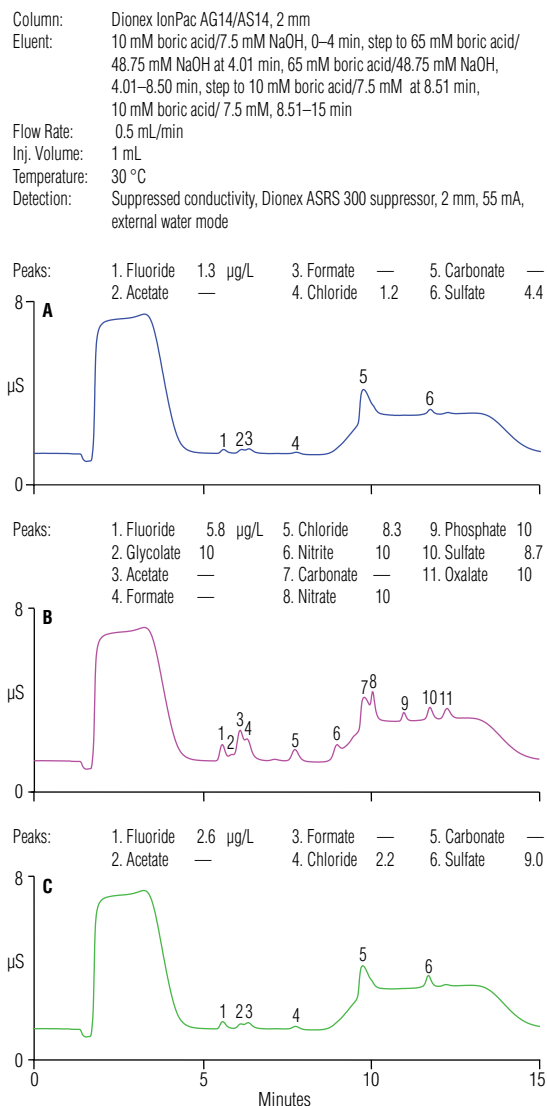


Figure 4. Chromatograms of (A) a 2500 mg/L B + 5.0 mg/L Li-simulated sample; (B) the same sample spiked with trace anions; and (C) the same sample spiked with 1.3 µg/L fluoride, 1.0 µg/L chloride, and 4.5 µg/L sulfate.

Table 4. Results of fluoride, chloride, and sulfate in different simulated borated waters.

Analyte ^a	1000 mg/L Boron + 1.8 mg/L Lithium			2000 mg/L Boron + 4 mg/L Lithium			2500 mg/L Boron + 5 mg/L Lithium		
	Amount Found (µg/L)	Retention Time RSD	Peak Area RSD	Amount Found (µg/L)	Retention Time RSD	Peak Area RSD	Amount Found (µg/L)	Retention Time RSD	Peak Area RSD
Fluoride	0.31	0.06	0.61	0.76	0.08	2.13	1.33	0.05	1.13
Chloride	0.53	0.26	1.96	0.73	0.18	2.43	1.23	0.01	1.61
Sulfate	0.83	0.43	3.50	2.81	0.14	1.73	4.51	0.14	0.63

^an = 3 injections

Table 5. Recoveries of fluoride, chloride, and sulfate in different simulated borated waters.

Analyte ^a	1000 mg/L Boron + 1.8 mg/L Lithium				2000 mg/L Boron + 4 mg/L Lithium				2500 mg/L Boron + 5 mg/L Lithium			
	Amount Added (µg/L)	Total Found (µg/L)	Peak Area RSD	Recovery (%)	Amount Added (µg/L)	Total Found (µg/L)	Peak Area RSD	Recovery (%)	Amount Added (µg/L)	Total Found (µg/L)	Peak Area RSD	Recovery (%)
Fluoride	0.41	0.69	0.79	92.6	0.81	1.52	0.30	94.2	1.32	2.58	0.44	94.6
Chloride	0.50	1.00	2.20	92.4	0.80	1.52	0.33	97.8	1.00	2.15	0.54	91.8
Sulfate	1.00	1.86	0.57	103	2.00	4.99	0.32	109	4.51	9.26	0.28	105

^an = 3 injections

Table 6. Retention time and peak area precisions.

Analyte ^a	0.2 µg/L Fluoride + 1 µg/L Chloride + 1 µg/L Sulfate		2500 mg/L Boron + 5 mg/L Lithium	
	Retention Time RSD	Peak Area RSD	Retention Time RSD	Peak Area RSD
Fluoride	0.07	1.10	0.27	3.09
Chloride	0.13	4.85	0.28	2.96
Sulfate	0.59	2.26	0.36	3.50

^an = 7 injections

Seven replicates of a standard mixture (0.2 µg/L fluoride, 1 µg/L chloride, and 1 µg/L sulfate) and 2500 mg/L boron plus 5.0 mg/L lithium simulated sample were injected (chromatograms shown in Figure 5). The retention time and peak area precisions were within 0.6% and 5%, respectively, as shown in Table 6.

Conclusion

This study demonstrates the determination of trace fluoride, chloride, and sulfate in simulated borated waters containing lithium using a borate eluent and a large-volume direct injection. This method is based on AN 114 and extends the analysis of anions in high-purity water to lithium-containing borated waters. Using a slightly modified eluent condition, the determination of fluoride, chloride, and sulfate is achieved with the early eluting borate peak resolved from fluoride and other target anions. Three different simulated samples are examined and the method is validated by recovery and precision studies on these simulated samples. This method also simplifies sample loading by replacing the manual loading performed using a syringe or pneumatic pressure in AN 114 with a Dionex AS-AP Autosampler. The MDLs of fluoride, chloride, and sulfate reported in this study are comparable to those in AN 114.

Column: Dionex IonPac AG14/AS14, 2 mm
 Eluent: 10 mM boric acid/7.5 mM NaOH, 0–4 min, step to 65 mM boric acid/48.75 mM NaOH at 4.01 min, 65 mM boric acid/48.75 mM NaOH, 4.01–8.50 min, step to 10 mM boric acid/7.5 mM at 8.51 min, 10 mM boric acid/7.5 mM, 8.51–15 min
 Flow Rate: 0.5 mL/min
 Inj. Volume: 1 mL
 Temperature: 30 °C
 Detection: Suppressed conductivity, Dionex ASRS 300 suppressor, 2 mm, 55 mA, external water mode

Peaks: 1. Fluoride 0.2 4. Chloride 1.0
 2. Acetate — 5. Carbonate —
 3. Formate — 6. Sulfate 1.0

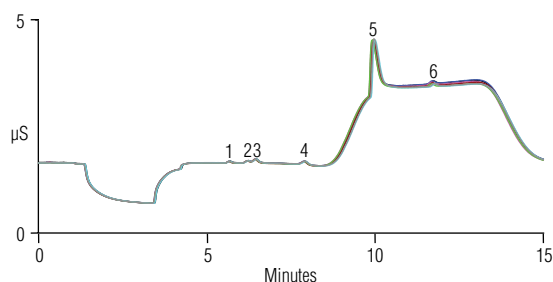


Figure 5. Overlay of seven chromatograms of a 0.2 µg/L fluoride, 1.0 µg/L chloride, and 1.0 µg/L sulfate standard mixture.

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