

Application of Eluent Generation for Trace Anion Analysis of Borated Waters

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Introduction

Boric acid is used in pressurized water reactor (PWR) power plants to control the nuclear reaction, because boron is a good neutron absorber. In PWR plants, measuring anionic contaminants in borated waters is an important part of corrosion monitoring. The presence of low- $\mu\text{g/L}$ (ppb) concentrations of chloride and sulfate can make the stainless steel components of a power plant, such as steam generators, boiler tubes, condenser tubes, and turbine blades, susceptible to stress-induced corrosion cracking.¹ Therefore, monitoring the presence of these anions is important. A reliable method is needed to determine anionic contamination at trace levels in borated waters.

Anions in borated waters are currently determined using ion chromatography (IC) with a sodium tetraborate eluent. This approach has several disadvantages. Preparing bottled eluents is labor intensive and operator dependent, leading to inconsistent results. Making a tetraborate eluent manually from tetraborate salts is problematic, particularly when using nonhydrated salts. When running tetraborate gradients, the concentration of the suppressed product—boric acid—increases with the gradient, leading to baseline shifts. Using boric acid and sodium hydroxide to prepare a tetraborate eluent is another approach, however contamination from carbonate causes a large baseline shift during the gradient that makes peak integration difficult.

Recently, a new automated method disclosed how to prepare tetraborate eluents using an Thermo Scientific Dionex EG50 Eluent Generator in conjunction with a boric acid eluent.² In this approach, the boric acid concentration is held constant while varying the potassium hydroxide concentration to create a gradient. The eluent generator can generate potassium hydroxide eluents that are free of carbonate contamination. Carbonate-free eluents result in consistent background conductivity and provide consistent peak response. This method minimizes baseline shifts during gradients and results in more reliable peak area integration. This application note describes a rapid, high-volume, direct-injection technique that uses an Thermo Scientific™ Dionex™ IonPac™ AS14 2 mm analytical column. Common anions such as fluoride, chloride, nitrate, phosphate, and sulfate can be determined at levels below 1 $\mu\text{g/L}$ (ppb) in borated waters with up to 7500 mg/L boron in less than 30 min.

This method is also applicable for use with standard-bore (4 mm) chromatography by using a standard-bore Thermo Scientific Dionex GP50 pump, 4 mm Dionex IonPac AS14 column, and 4 mm Thermo Scientific™ Dionex™ ASRS™ Anion Self-Regenerating Suppressor at a flow rate of 1.5 mL/min. A 1 mL loop with a standard bore configuration will have one quarter the peak response compared to using a microbore system.

Equipment

Thermo Scientific Dionex ICS-2500 IC system* consisting of:

- GS50 Gradient Pump with vacuum degas
- CD25A Conductivity Detector with DS-3
- EG50 Eluent Generator with Thermo Scientific Dionex EGC II KOH Cartridge
- LC30 Chromatography Enclosure equipped with Rheodyne Model 9126 injector PEEK, rear loading
- Thermo Scientific Dionex AS40 Autosampler
- Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System (CDS) Software
- Columns:
 - Dionex IonPac AS14 Analytical, 2 mm i.d.
 - Dionex IonPac AG14 Guard, 2 mm i.d.
- Suppressor:
 - Thermo Scientific™ Dionex™ ASRS™ ULTRA II Anion Self-Regenerating Suppressor, 2 mm 300 cm of green 0.75 mm (0.030 in.) PEEK tubing to make a 1000 µL sample loop
- Suppression External Water Installation Kit includes 25 psi regulator and a 4 L water reservoir
- Upchurch Plastic Tubing Cutter
- Filter unit, 0.2 µm, nylon
- Ion chromatography filters, 13 mm, 0.2 µm pore size (optional)

*Equivalent or improved results can be achieved using the Thermo Scientific Dionex ICS-5000+ or the Thermo Scientific Dionex ICS-2100 system.

Reagents, Standards, and Samples

- Deionized water, Type I reagent-grade, 18 MΩ-cm resistance
- Boric acid
- Sodium borate, tetrahydrate
- Fluoride standard 1000 mg/L, 100 mL
- Chloride standard 1000 mg/L, 100 mL
- Sulfate standard 1000 mg/L, 100 mL
- Nitrate standard 1000 mg/L, 100 mL
- Phosphate standard 1000 mg/L, 100 mL
- ACS reagent-grade materials for preparing anion standards optional

50 mM Boric Acid Eluent

Weigh 3.09 g of boric acid. Carefully add this amount to a 1 L volumetric flask containing about 500 mL of deionized water (with a specific resistance of 18 MΩ-cm or greater). Dilute to the 1 L mark and mix thoroughly. Use a stir bar or ultrasonic bath to ensure that the boric acid is completely dissolved. Filter the solution through a 0.2 µm nylon filter unit. Transfer this solution to an eluent container and vacuum degas for 5 min.

Anion Standard Solutions (1000 mg/L)

Several of the analytes of interest are available as 1000 mg/L anion standard solutions. When commercial standards are unavailable, 1000 mg/L standards can be prepared by dissolving the appropriate amounts of the corresponding mass for the target analytes in 1000 mL of deionized water according to Table 1. We recommend making a 100 mL final volume of 1000 mg/L stock standards in 125 mL high-density polyethylene (HDPE) containers. Concentrated standards are stable for at least one month when stored at 4 °C.

Table 1. Amounts used to prepare 1 L of 1000 mg/L anion standards.

Anion	Compound	Mass (g)
Fluoride	Sodium fluoride (NaF)	2.210
Chloride	Sodium chloride (NaCl)	1.648
Nitrate	Sodium nitrate (NaNO ₃)	1.371
Sulfate	Sodium sulfate (Na ₂ SO ₄)	1.479
Phosphate	Potassium phosphate, monobasic (KH ₂ PO ₄)	1.433

Calibration

Working Standard Solutions

The anion concentrations in borated water samples were determined using external standards prepared in deionized water. Composite working standards were prepared by diluting appropriate volumes of the 1000 mg/L stock standards with deionized water. Working standards containing <100 mg/L anions should be prepared daily.

ATC-HC Regeneration Solution

100 mM Sodium Borate

Thoroughly dissolve 38.14 sodium borate, tetrahydrate (MW 381.42 g/mole) in 700 mL degassed deionized water (with a specific resistance of 18 MΩ-cm or greater) in a 1 L volumetric flask. Dilute to a final volume of 1000 mL.

Conditions

Columns:	Dionex IonPac AS14 Analytical, 2 mm i.d. Dionex IonPac AG14 Guard, 2 mm i.d.
Eluent:	50 mM boric acid titrated with potassium hydroxide from an Dionex EG50 Eluent Generator
Temperature:	30 °C
Gradient:	4.0 mM KOH from 0–6.0 min with 50 mM boric acid 4.0 to 30.0 mM KOH from 6–18 min with 50 mM boric acid
Flow Rate:	0.38 mL/min
Detection:	Suppressed conductivity
Suppressor:	Dionex ASRS ULTRA II suppressor, 2 mm, AutoSuppression, external water mode
Suppressor current:	38–46 mA
Expected Background Conductivity:	3 µS (30 mM potassium hydroxide/50 mM boric acid)
Recommended System Backpressure:	15.2–16.6 MPa (2200–2400 psi)

Pump Program Method

Time (min)	Flow (mL/min)	50 mM Boric Acid (%)	Valve	Dionex EG40 Conc. (mM)	Comments
–7.00	0.38	100	Load	4.0	4.0 mM KOH
–2.4	0.38	100	Load	4.0	Load Loop, Dionex AS40 Autosampler on
–0.1	0.38	100	Load	4.0	Dionex AS40 Autosampler off
0.00	0.38	100	Inject	4.0	Inject, 4.0 mM
6.00	0.38	100	Inject	4.0	Begin gradient to 30 mM KOH
18.0	0.38	100	Inject	30.0	30 mM KOH
30.0	0.38	100	Load	30.0	30 mM KOH

System Preparation and Setup

This section describes the procedure for the initial installation and start-up of the Dionex ASRS ULTRA suppressor, Thermo Scientific Dionex ATC-HC Anion Trap Column, and Dionex EluGen cartridge. Prepare the Dionex ASRS suppressor for use by hydrating the eluent chamber and regen chamber with deionized water. Wait at least 20 min before pumping eluent through the eluent chamber. (For more information on Dionex ASRS suppressor operation, consult the Quickstart Instructions for the Dionex ASRS ULTRA Suppressor.)

Prepare the Dionex ATC-HC column by rinsing with 100 mL of 100 mM tetraborate at 2.0 mL/min. This can be done off-line without the GS50 by pressurizing an eluent bottle with helium at 34.5 kPa (5 psi). Then rinse the Dionex ATC column with boric acid at 2.0 mL/min for 20 min. Install the Thermo Scientific Dionex EGC II KOH EluGen cartridge according to the instructions in the *Operator's Manual for the Dionex EG50 Eluent Generator System*. Place the Dionex ATC-HC column between the GS50 outlet and the Dionex EGC II KOH cartridge inlet as shown in Figure 1.

Sample Loading

Load the sample into the sample loop with an Dionex AS40 Autosampler using the 5 mL vials. For analysis of samples containing target anions at µg/L concentration levels, the probability of contamination during sample collection and storage is high. Sample and standard containers, as well as autosampler vials and caps, should be rinsed with deionized water prior to use. *Use only Dionex AS40 autosampler sample vial caps without filters when performing low-level analyses.*

Samples that contain high amounts of particulate matter should be filtered with filters that will not contribute significant amounts of the analytes of interest.

Loading Samples in the Autosampler Vials for Routine Analysis

1. Rinse the vials and caps 3–5 times with DI water.
2. Fill the 5 mL vial with the standard, sample, or blank.
3. Insert the cap into the vial with the insertion tool and load into the sample cassette.

Loading Samples in the Autosampler Vials for Trace Analysis

1. Rinse the vials and caps 3–5 times with DI water.
2. Place the vials and caps in a large, precleaned container and soak for 4 h in DI water.
3. Drain or empty the container and refill it with DI water. Soak the caps and vials for an additional 24 h before use.
4. Fill the 5 mL vial with the standard, sample, or blank.
5. Insert the cap into the vial with the insertion tool and load into the sample cassette.

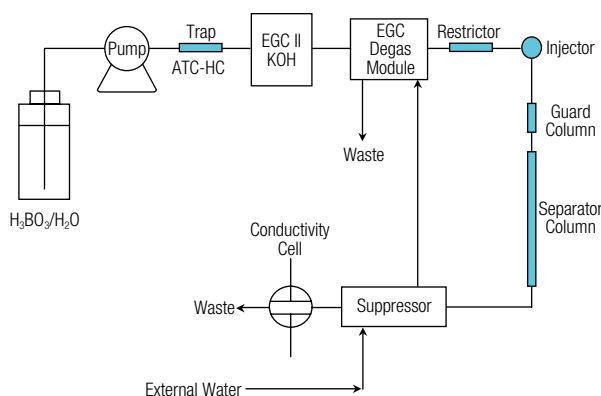


Figure 1. System configuration with the Dionex EG50 generator using boric acid eluent.

Configure the pressurized water reservoir as shown in Section 3.4, "Plumbing for the AutoSuppression External Water Mode Operation" of the *Product Manual for the Dionex Anion Self-Regenerating Suppressor ULTRA II*. Fill the reservoir with deionized water with a specific resistance of 18 MΩ-cm or greater. Adjust the reservoir pressure from 0 to 172 kPa (0 to 25 psi) to deliver external water regenerant of 5–7 mL/min before applying current. Ensure that the reservoir cap is sealed tightly. After applying the current, the flow rate will drop due to gas formation in the regenerant.

Make a 1000 µL sample loop by cutting a 220 cm portion of 0.030 in (0.75 mm) i.d. PEEK tubing. In cases where a loop or tubing with a different internal diameter is desired, refer to Table 2 to calculate the length needed. The volume of a loop can be verified by measuring the weight difference between the sample loop filled with deionized water and the dry, empty loop. The inside diameter of tubing varies by as much as 20% (e.g., 0.010 ± 0.002 in). For example a 250 µL loop prepared with 0.030 in i.d. PEEK tubing had a dry weight of 12.8327 g and weighed 13.0953 g filled with water. This weight difference and a repeat measurement suggest the loop is a 262 µL loop. Connect the columns and suppressor in the IC system by using 0.005 in (0.125 mm) tubing. Keep the lengths of connecting tubing as short as possible to minimize system void volume, which ensures efficient 2 mm column operation. Carefully use a plastic tubing cutter and make sure that the surfaces of the tubing cuts have straight and smooth surfaces. Irregularity on the surface of a tubing end can result in unwanted dead volume.

Turn flow on the gradient pump to begin the flow of eluent through the system. If the system backpressure is below 14 MPa (2000 psi), then a portion of the yellow PEEK 0.003 in (0.075 mm) tubing should be added between the outlet of the degas assembly in the Dionex EG50 generator and the inlet of the injection valve. Confirm that there are no leaks in the chromatographic pathway. For more information consult the *Operator's Manual for the Dionex EG50 Eluent Generator System*. Turn on the Dionex EG50 generator to deliver 30 mM KOH using Chromeleon CDS software and allow the background conductivity to reach a steady value.

Table 2. Volume per unit length for various tubing internal diameters.

Material	Color	Internal Diameter (Inches)	Internal Diameter (mm)	Estimated Volume (µL/cm)
PEEK	Red	0.005	0.125	0.126
PEEK	Black	0.010	0.250	0.506
PEEK	Orange	0.020	0.500	2.022
PEEK	Green	0.030	0.750	4.550

Results and Discussion

The Dionex IonPac AS14 column provides the best selectivity for fluoride, chloride, nitrate, sulfate, and phosphate using a borate eluent. Fluoride is well resolved from the water dip and free of interference from weakly retained organic acids such as glycolate, acetate, and formate. The best separation of the target analytes was achieved with a gradient from 4 to 30 mM potassium hydroxide using 50 mM boric acid. The Dionex IonPac AS14 2 mm column (with a capacity of 65 µeq/column) was selected because it has more than three times the capacity of the Dionex IonPac AS4A-SC column (20 µeq/column capacity). Due to the high capacity, the Dionex IonPac AS14 column can tolerate larger sample volumes by direct injection.

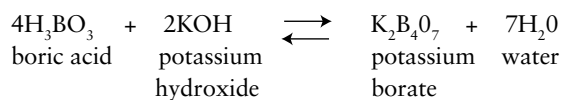
The microbore format, chosen for the analytical columns and suppressor, has several advantages for trace-level analysis, including a fourfold increase in mass sensitivity for the microbore (2 mm) over the standard bore (4 mm) format with no change in concentration sensitivity. The increased mass sensitivity allows smaller sample volumes to be injected. The microbore format also uses less eluent and produces less eluent waste.

The Dionex ASRS ULTRA II suppressor provides low background and noise and facilitates low detection limits for the target anions. The DS-3 conductivity cell minimizes the effects of temperature-induced baseline drift. Separations were performed at 30 °C. A high-capacity anion trap column Dionex ATC-HC column) in the borate form was used to trap anionic contaminants in the boric acid eluent. The Dionex ATC-HC column is placed between the outlet of the gradient pump and the inlet of the Dionex EluGen cartridge. The Dionex ATC-HC column requires periodic regeneration with 100 mM sodium borate. Do not use a Thermo Scientific Dionex CR-ATC Continuously Regenerated Anion Trap Column for this application, because it would remove anionic compounds—including borate ions—from the boric acid eluent.

The Dionex EG50 Eluent Generator enhances IC performance for the determination of anions at trace levels.³ In this application, the Dionex EG50 generator was used to electrolytically produce high-purity, carbonate-free KOH eluents with boric acid as the carrier stream. Gradient separations with carbonate-free hydroxide eluents are highly precise and have negligible baseline shifts and lower background conductivity. Because the present method uses a constant stream of boric acid, all the benefits of electrolytically generated hydroxide eluents are realized, including better retention time reproducibility and improved signal-to-noise ratios.

The optimized gradient method for this application was quickly developed using the Dionex EG50 Eluent Generator. The Dionex EG50 generator was programmed with a number of isocratic methods and linear gradient eluent methods to evaluate a variety of eluent conditions. Without the Dionex EG50 generator, it would have been necessary to proportion from a manually prepared eluent to achieve different gradient conditions.

The tetraborate eluent is made by a chemical reaction of boric acid with potassium hydroxide according to the following reaction:



The 50 mM boric acid eluent is "titrated" with the potassium hydroxide generated in-line through the eluent generator. Four moles of boric acid react with two moles of potassium hydroxide to form one mole of tetraborate. For example, 8 mM boric acid is titrated with 4 mM potassium hydroxide to form 2 mM tetraborate.

By making the tetraborate eluent in-line with the Dionex EG50 generator, carbonate is not mixed into the solution and therefore the chromatography is improved. Figure 1 illustrates how this eluent is made on-line. This technique has several advantages. The eluent strength can be adjusted by the on-line addition of potassium hydroxide. The background is maintained at the same level during gradient runs, because the boric acid concentration is held constant during the run.

Method Performance

Figure 2 shows separation of a trace anion standard prepared in deionized water using the Dionex EG50 generator with the boric acid method. The large system void corresponds to the time required for the 1 mL sample to pass through the chromatographic system. The method began with an initial eluent concentration of 4 mM potassium hydroxide/50 mM boric acid to elute weakly retained ions. Fluoride is well resolved from the system void and resolved from the organic acids (glycolate, acetate, and formate) under these conditions. Depending on the amount of carbonate present in the sample, chloride may coelute with carbonate. Degassing the sample may minimize the presence of carbonate. The eluent concentration in this method increases from 4 to 30 mM potassium hydroxide in 12 min to elute stronger retained anions such as nitrate, phosphate, and sulfate. The pump program returns to a 4 mM potassium hydroxide/50 mM boric acid eluent to equilibrate for 7 min prior to the next injection.

This method is applicable to high-purity power plant waters containing up to 7500 mg/L boron as boric acid. Figure 3 shows an analysis of 2000 mg/L boron containing trace levels of anionic contamination. The large borate matrix peak beginning at 2 min returns to baseline before the first analytes of interest elute. No significant difference in peak efficiencies or retention times is observed compared to the standard prepared in deionized water. Chloride can elute on the downward slope of the carbonate peak, complicating integration. However, by using the "Maximum Rider Ratio" command in the Chromeleon chromatography software this peak can be reproducibly integrated as a rider on the slope of the carbonate peak.

Column:	Dionex IonPac AG14/AS14, 2 mm	Peaks:	1. Fluoride	30 µg/L
Eluent:	50 mM boric acid		2. Unidentified	—
Flow Rate:	0.38 mL/min		3. Carbonate	—
Inj. Volume:	1000 µL		4. Chloride	30
Suppressor:	Dionex ASRS ULTRA suppressor, 2 mm, 100 mA, external water mode		5. Nitrate	30
			6. Phosphate	28
			7. Sulfate	35

Gradient:	Time (min)	Concentration (KOH, mM)
	0	4.0
	6	4.0
	18	30.0
	25	30.0

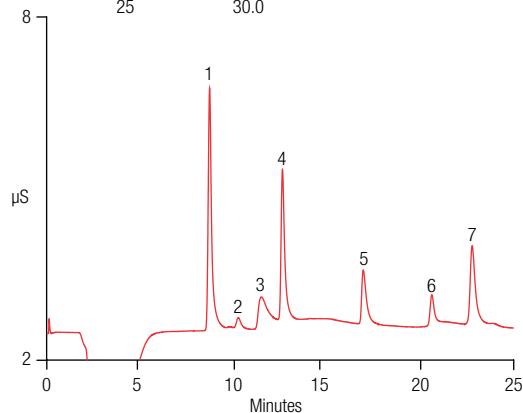


Figure 2. Trace anion standard in deionized water.

Column:	Dionex IonPac AG14/AS14, 2 mm	Peaks:	1. Borate matrix	µg/L
Eluent:	50 mM boric acid		2. Fluoride	0.80
Flow Rate:	0.38 mL/min		3. Unidentified	—
Inj. Volume:	1000 µL		4. Unidentified	—
Suppressor:	Dionex IonPac AG14/AS14, 2 mm, 100 mA, external water mode		5. Carbonate	—
			6. Chloride	5.68
			7. Unidentified	—
			8. Nitrate	18.6
			9. Sulfate	37.8

Gradient:	Time (min)	Concentration (KOH, mM)
	0	4.0
	6	4.0
	18	30.0
	26	30.0

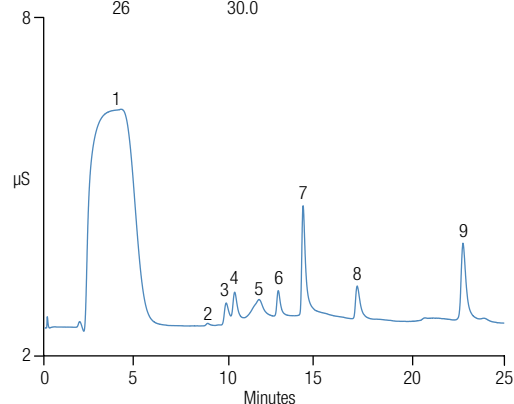


Figure 3. Trace anions in 2000 mg/L boron.

To start trace-level analysis, first establish a blank. You can easily perform this task by injecting high-purity deionized water as a sample, using the same set of containers, pipetting devices, and so forth. A baseline anion concentration is established above which reliable quantification can be made. Make several replicate injections to establish a precise reading. Figure 4 shows a representative blank (from a typical laboratory point-of-use deionization system). It is not uncommon to have trace levels of chloride, nitrate, and sulfate from a typical laboratory point-of-use deionized water unit. Figure 5 shows a representative system blank. The data for the system blank were collected without a sample injection. Accomplish this action by using Chromeleon to select "Blank" as the "Sample Type" (explained in the help section under "Blank run sample"). No peaks were detected in the system blank, confirming that no significant anionic contaminants were present in the boric acid and the trap column is functioning properly.

The total baseline shift between the weak and strong eluent concentration in the separation presented in this application note would typically be 2 μS with a manually prepared tetraborate eluent. The Dionex EG50 generator-based method reduces the baseline shift to less than 0.1 μS . The Dionex ATC-HC anion trap column traps anionic contaminants in the boric acid eluent. This trap column should be periodically regenerated by conditioning it with at least 100 mL of 100 mM sodium tetraborate solution. The frequency of trap column regeneration depends on the level of contamination in the 50 mM boric acid eluent.

For the best performance at trace levels, baseline noise must be kept to a minimum. A system for trace analysis typically requires about 1 h from start-up to establish a stable background conductivity. For this reason, it is best to keep the system running continuously. Ensure that you have an adequate supply of the eluents, external water, and waste containers to allow the system to run unattended.

The Dionex ICS-2500 system and Chromeleon CDS software provide the analyst with the ability to monitor baseline noise. In a representative 1.0 min section, an equilibrated system in good working order should be able to deliver a noise reading of less than 5 nS peak to peak. In the diagnostic menu under "Pressure statistics", the Dionex GS50 Gradient Pump displays the measured pressure from the pressure transducer. In a 1 min segment, the pump pressure reading difference should be less than 138 kPa (20 psi).

Calibration curves were obtained using anion standards prepared in deionized water. Three replicate injections were used at spikes of 1, 3, 10, and 30 ppb for fluoride, chloride, nitrate, sulfate, and phosphate in the matrix. Results for the anions of interest yielded a linear response with coefficients of determination (r^2) greater than 0.997. Table 3 summarizes the method detection limits (MDLs) for the target analytes calculated based on three times the signal-to-noise ratio. Remember that the lowest quantifiable analyte concentration is generally three to five times greater than the lowest detectable concentration.

Column:	Dionex IonPac AG14/AS14, 2 mm	Peaks:	1. Unidentified	—	$\mu\text{g/L}$
Eluent:	50 mM boric acid		2. Unidentified	—	
Flow Rate:	0.38 mL/min		3. Carbonate	—	
Inj. Volume:	1000 μL		4. Chloride	0.32	
Suppressor:	Dionex ASRS ULTRA suppressor, 2 mm, 100 mA, external water mode		5. Unidentified	—	
			6. Nitrate	0.67	
			7. Sulfate	0.71	
Gradient:	Time (min)	Concentration (KOH, mM)			
	0	4.0			
	6	4.0			
	18	30.0			
	25	30.0			

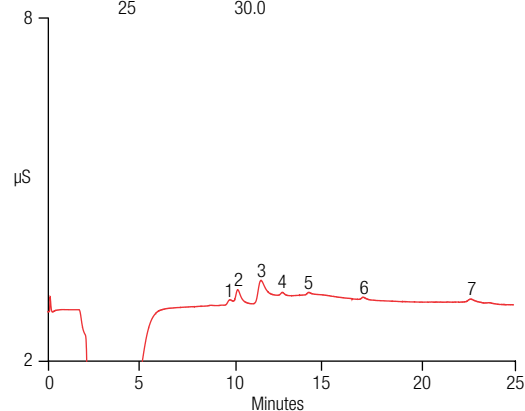


Figure 4. Representative deionized water blank.

Column:	Dionex IonPac AG14/AS14, 2 mm
Eluent:	50 mM boric acid
Flow Rate:	0.38 mL/min
Inj. Volume:	1000 μL
Suppressor:	Dionex ASRS ULTRA suppressor, 2 mm, 100 mA external water mode

Gradient:	Time (min)	Concentration (KOH, mM)
	0	4.0
	6	4.0
	18	30.0
	26	30.0

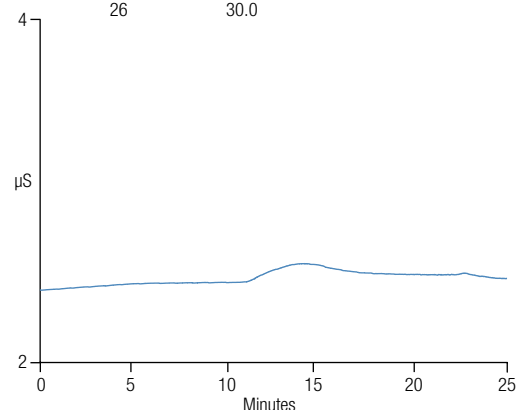


Figure 5. Representative system blank.

Table 3. MDLs for determination of trace anions in borated waters using eluent generation and boric acid eluent.

Anion	MDL, $\mu\text{g/L}$
Fluoride	0.030
Chloride	0.053
Nitrate	0.10
Phosphate	0.32
Sulfate	0.090

*Calculated based on three times signal-to-noise

Precautions

The deionized water used for preparing rinse solution, eluent, and standards should be free of measurable levels of ionic impurities, organics, microorganisms, and particulate matter larger than 0.2 µm. Soak containers for at least 24 h with deionized water and rinse several times prior to use. When conducting analyses at trace levels, the sources of contamination are numerous. Wear vinyl disposable gloves when handling equipment that makes contact with eluent, standard, or samples. After putting on the gloves, rinse them with deionized water, then air dry. Do not dry with paper towels. All containers should be dedicated to this analysis and copiously rinsed with 18 MΩ-cm or better deionized water before use. Exercise caution when handling anything that could have contact with the blank, unknown, or standards. The flow path of the chromatographic instrumentation (eluent containers, injector, pump, valves, tubing, columns, suppressor, and conductivity cell) is a potential source of contamination. Rinse with high-purity water to reduce residual contamination.

The Dionex ATC-HC column should be periodically regenerated with the procedure described in the "System Preparation and Setup" section. Monitoring the blank for any significant increase in anionic contamination will indicate when regeneration is necessary. Monitoring the background conductivity will indicate when regeneration is necessary. A significant increase beyond 3 µS indicates that the Dionex ATC-HC column has exceeded its capacity to trap ionic contaminants and should be regenerated. The frequency of regeneration depends on the quality of the deionized water and usage rate of the instrument. Expect to regenerate the Dionex ATC-HC column at least every 30 days of continuous (24/7) operation.

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2. Ling, R.; Srinivasan, K.; Saini, S.; Pohl, C.; Avdalovic, N.; Presented at the International Ion Chromatography Symposium, San Diego, CA, September 2003; Poster A-15.
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