

Monitoring Inorganic Anions and Cations During Desalination

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Introduction

As of 2009, there were 14,450 desalination plants worldwide producing more than 60 million cubic meters of water a day.¹ Because of the growing demand for water and the limited supply of fresh water, desalination increasingly is being used to produce potable and irrigation water from salty or brackish water. The global market for desalination to generate supplies of potable water is projected to grow at an annual rate of 10% over the next 10 years. Seawater desalination is a \$10 billion industry today and is forecasted to reach \$16 billion in 2020.¹

A wide variety of desalination techniques are currently available and more are being developed. Most use distillation or membrane techniques. The performance of desalination processes is evaluated by monitoring the common anions and cations in the feed, intermediate, and final water product. For the final drinking water product, ion chromatography (IC) is approved for monitoring primary and secondary anions according to U.S. Environmental Protection Agency (EPA) Method 300.0,² and Federal and State regulatory agencies ensure that U.S. National Primary and Secondary Drinking Water Standards are met.

Common cations, though not considered contaminants, are monitored and reported by many public water suppliers in the United States. Cations, particularly calcium and magnesium, are measured to determine water hardness. In addition to calcium and magnesium, ammonium is also measured and regulated in public water supplies in EU countries and Japan. During desalination, the levels of divalent cations affect performance of membrane processes like reverse osmosis (RO).³ High levels of calcium or magnesium result in frequent fouling of the membranes, which is highly undesirable. Therefore, it is critical to monitor anions and cations at all stages of desalination.



Another challenge for the desalination of seawater is the removal of boron, which is typically found at concentrations of 4.5 mg/L. World Health Organization 2008 guidelines suggest a concentration of 0.5 mg/L,⁴ whereas the U.S. EPA recommends a maximum lifetime exposure of 0.6 mg/L.⁵ Depending on pH levels, boron can exist in ionic and non-ionic forms. Above pH 8, the removal efficiency using RO is enhanced due to the formation of borate. RO membranes remove ions better than non-ionic forms of the same compounds. This suggests that raising the pH may improve the removal efficiency of boron. However, raising the pH too high results in the formation of scales formed by the precipitation of carbonate salts of calcium and magnesium, which can disrupt membrane performance. In addition to monitoring the pH, it is important to know the concentration of scale-forming divalent cations calcium and magnesium in order to maintain optimal RO membrane performance.

Compared to traditional sources of water, desalination is an energy-intensive process that requires expensive infrastructure. The potential benefits of desalination are constantly being evaluated because of the high economic and environmental costs. Hence, efficient water monitoring techniques are needed to understand the robustness of desalination processes.

This work describes an IC method using a Thermo Scientific™ Dionex™ ICS-3000 Reagent-Free™ Ion Chromatography (RFIC™) system with Thermo Scientific™ Dionex™ IonPac™ AS18 anion-exchange and Dionex IonPac CS12A cation-exchange columns, electrolytically generated hydroxide and methanesulfonic acid eluents, and suppressed conductivity detection to simultaneously measure the common anions and cations in water samples obtained from desalination processes. This method uses a 2 mm column format for anion separations, a 3 mm column format for cation separations, and electrolytically generated eluents that require only the addition of deionized water for continuous operation. The linearity, method detection limits (MDLs), precision, and recovery of anions and cations in saline and drinking water matrices for this method are discussed here. This IC method supports all the monitoring needs of a desalination facility because it can measure anions and cations in diverse matrices ranging from seawater to drinking water.

Equipment

Dionex ICS-3000 RFIC system* with eluent generation (RFIC-EG) including:

- Thermo Scientific Dionex DP Dual Pump module
- Thermo Scientific Dionex EG Eluent Generator module
- Thermo Scientific Dionex DC Detector/Chromatography module (single- or dual-temperature zone configuration)
- Thermo Scientific Dionex AS Autosampler (with Simultaneous Injection Upgrade Kit,)
- Thermo Scientific Dionex EGC II KOH cartridge
- Thermo Scientific Dionex CR-ATC Continuously Regenerated Anion Trap Column
- Thermo Scientific Dionex EGC II MSA cartridge
- Thermo Scientific Dionex CR-CTC Continuously Regenerated Cation Trap Column
- Thermo Scientific™ Dionex™ Chromeleon™ 6.8 Chromatography Data System (CDS) Workstation
- Polystyrene Autoselect vials with caps and septa, 10 mL
- Thermo Scientific™ Nalgene™ 125 mL Narrow-Mouth HDPE Bottles
- Nalgene 250 mL Narrow-Mouth HDPE Bottles
- Thermo Scientific™ Nalgene™ 250 mL 0.2 µm nylon filter units
- Nalgene 1000 mL 0.2 µm nylon filter units

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

Reagents and Standards

- Deionized water, Type I reagent grade, 18 MΩ-cm resistivity or better, filtered through a 0.2 µm filter immediately before use
- Fluoride Standard 1000 mg/L
- Chloride Standard 1000 mg/L
- Nitrite Standard 1000 mg/L
- Bromide Standard 1000 mg/L
- Sulfate Standard 1000 mg/L
- Nitrate Standard 1000 mg/L
- Phosphate Standard 1000 mg/L
- Lithium Standard 1000 mg/L
- Sodium Standard 1000 mg/L
- Ammonium Standard 1000 mg/L
- Potassium Standard 1000 mg/L
- Magnesium Standard 1000 mg/L
- Calcium Standard 1000 mg/L
- Sodium Chloride
- Sodium Sulfate
- Sodium Nitrite
- Sodium Bromide
- Sodium Nitrate
- Potassium Phosphate Monobasic
- Lithium Chloride
- Ammonium Chloride
- Potassium Chloride
- Magnesium Chloride Hexahydrate
- Calcium Chloride Dihydrate
- Combined Six Cation Standard-II
- Combined Seven Anion Standard

Conditions

Anion Determinations

Columns:	Dionex IonPac AG18, 2 × 50 mm Dionex IonPac AS18, 2 × 250 mm
Eluent:	22 mM KOH from 0–7 min, 22–40 mM KOH from 7–8 min, 40 mM KOH from 8–18 min*
Eluent Source:	Dionex EGC II KOH cartridge with Dionex CR-ATC column
Injection Volume:	4 µL
Flow Rate:	0.25 mL/min
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ ASRS™ 300 Anion Self-Regenerating suppressor, 2 mm, recycle mode, suppressor current 15 mA
Background Conductance:	<1 µS

Cation Determinations

Columns:	Dionex IonPac CG12A-5 µm, 3 × 30 mm Dionex IonPac CS12A-5 µm, 3 × 150 mm
Eluent:	20 mM MSA
Eluent Source:	Dionex EGC II MSA cartridge with Dionex CR-ATC column
Injection Volume:	10 µL
Flow Rate:	0.50 mL/min
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ CSRS™ 300 Cation Self-Regenerating suppressor, 2 mm, recycle mode, suppressor current 30 mA
Background Conductance:	<0.5 µS

Both Anion and Cation Determinations

Temperature:	30 °C (column and detector compartment)
Noise:	~0.5–1.0 nS (conductivity)
System Backpressure:	~2500 psi
Run Time:	20 min (including column equilibration time)

*The column equilibrates for 2 min at 22 mM KOH prior to injection.

Preparation of Solutions and Reagents

Eluent Solutions

Generate potassium hydroxide (KOH) and methanesulfonic acid (MSA) eluents online by pumping high-quality degassed, deionized (DI) water through the Dionex EGC II KOH and Dionex EGC II MSA cartridges, respectively. Chromeleon CDS software tracks the amount of KOH and MSA used and calculates the remaining lifetime. Although electrolytic eluent generation delivers the best performance, manually prepared eluents may be used, if needed.

Stock Standard Solution

Certified standard solutions can be purchased from Thermo Scientific or other commercial sources. When commercial standards are not available, 1000 mg/L stock standard solutions can be prepared by dissolving appropriate amounts of the required analyte in DI water in a plastic volumetric flask (Table 1). Store in plastic containers at 4 °C. Stock standards are stable for at least 3 months.

Working Standard Solutions

Prepare composite working standards at lower analyte concentrations by diluting appropriate volumes of the 1000 mg/L stock with DI water. Prepare working standards containing <100 mg/L anions or cations daily. Store standard solutions at <6 °C when not in use.

Table 1. Mass of compound required to prepare 1 L of 1000 mg/L stock standard solutions.

Analyte	Compound	Amount(g)
Fluoride	Sodium fluoride (NaF)	2.210
Chloride	Sodium chloride (NaCl)	1.648
Nitrite	Sodium nitrite (NaNO ₂ -N)	4.926
Bromide	Sodium bromide (NaBr)	1.288
Nitrate	Sodium nitrate (NaNO ₃ -N)	6.068
Sulfate	Sodium sulfate (Na ₂ SO ₄)	1.479
Phosphate	Potassium phosphate monobasic (KH ₂ PO ₄ -P)	4.394
Lithium	Lithium chloride (LiCl)	6.108
Sodium	Sodium chloride (NaCl)	2.542
Ammonium	Ammonium chloride (NH ₄ Cl)	2.965
Potassium	Potassium chloride (KCl)	1.907
Magnesium	Magnesium chloride hexahydrate (MgCl ₂ • 6H ₂ O)	8.365
Calcium	Calcium chloride dihydrate (CaCl ₂ • 2H ₂ O)	3.668

Sample Preparation

Artificial Seawater

Prepare simulated seawater by diluting the salts listed in Table 2 into 1 L of DI water following the method of Kester et al.⁶ with the exclusion of strontium chloride. This yields a solution with approximately 3.5% salinity.

Table 2. Salts added to form simulated seawater (1 L).

Compound	Amount (g)
Sodium chloride	2.393
Sodium sulfate	4.008
Potassium chloride	0.677
Sodium bicarbonate	0.196
Potassium bromide	0.098
Boric acid	0.026
Sodium fluoride	0.003

Commercial Aquarium Sea Salt

Follow package directions (1/2 cup of salt per gallon of DI water) to prepare commercially available synthetic sea salt, creating a solution of approximately 3.5% salinity. Prepare a 1 L portion with 30 g of aquarium salt. (A sea salt density of approximately 2.2 g/cm³ was used to convert the preparation directions to metric units.⁷)

Seawater (From California's San Francisco Bay)

Collect surface seawater in a Nalgene 250 mL HDPE bottle that has been precleaned before sample collection. Store the sample on ice until it can be filter sterilized through a Nalgene 250 mL, 0.2 µm nylon filter unit. After filtration, store the sample at <6 °C. Filter all samples through a 0.2 µm nylon filter unit before injection.

System Preparation and Configuration

Configure the Dionex AS autosampler for simultaneous injection into the anion and cation detection systems. In the simultaneous mode, the Dionex AS autosampler delivers sample to two independent IC systems. The sample is injected simultaneously and equally to both systems (two injection valves are required). Dual analyses can be performed with only one sample. A 5 or 10 mL syringe and an 8.5 mL sampling needle assembly are required for simultaneous injections. Full-loop injections are required for this mode.

Using Chromeleon CDS software, configure the two chromatography systems and the Dionex AS autosampler into a single timebase and assign each system a unique device name and channel. Use one control panel to monitor and control both systems and all samples in one sequence. The system also may be configured for sequential injection into the two IC systems. In the sequential option, the sample is delivered to the first system, flow is rerouted (diverted), and then sample is delivered to the second system.⁸

Results and Discussion

Using the Dionex IonPac CS12A and Dionex IonPac AS18 columns, the common anions and cations were easily resolved in 20 min (Figure 1 A and B). Note that this method provided good resolution between sodium and ammonium, the two analytes that can be challenging to resolve, especially when one is in a large excess relative to the other. This method also achieved good retention time for fluoride, which was well resolved from the void volume.

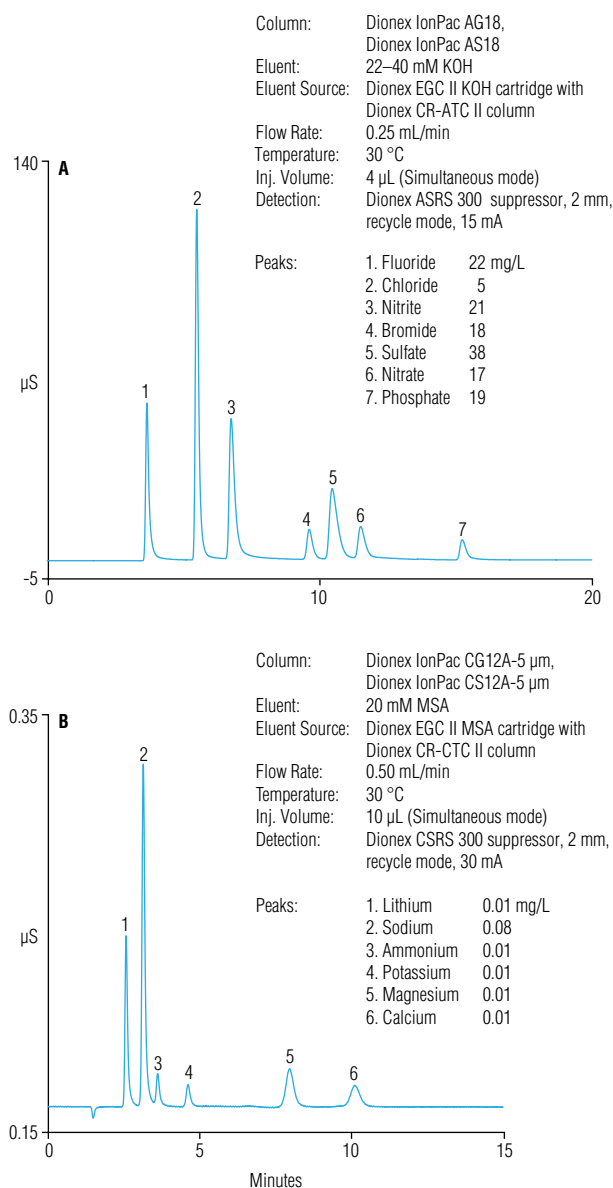


Figure 1. Separation of common A) anions using the Dionex IonPac AS18 column and B) cations using the Dionex IonPac CS12A column.

Table 3. Linear range, MDLs, and precisions for anions and cations.

Analyte	Range (mg/L)	Corr. Coeff. (r ²)	MDL Standard (µg/L)	MDL (µg/L) ^a	QCS (mg/L)	Retention Time Precision (RSD) ^b	Peak Area Precision (RSD) ^b
Lithium	0.02–16	0.9999	1	0.08	1	0.07	0.20
Sodium	0.10–100	0.9999	4	0.13	4	<0.01	0.29
Ammonium ^c	0.01–8	0.9997	5	0.10	5	<0.01	0.97
Potassium	0.02–16	0.9997	10	0.10	10	0.03	0.61
Magnesium	0.02–80	0.9998	5	0.53	5	0.03	0.09
Calcium	0.02–80	0.9999	5	0.36	5	0.04	0.15
Fluoride	0.08–100	0.9996	10	0.62	2	0.05	0.05
Chloride	0.24–300	0.9999	10	0.66	20	0.02	0.04
Nitrite (-N)	0.08–100	0.9994	20 (67 as NO ₃ ⁻)	0.51 (-N)	2 (6.7 as NO ₃ ⁻)	0.03	0.07
Bromide	0.08–100	0.9994	25	0.46	2	0.02	0.05
Sulfate	0.16–201	0.9994	20	0.67	2	0.01	0.07
Nitrate (-N)	0.02–22	0.9999	27 (120 as NO ₃ ⁻)	0.24 (-N)	2.3 (10 as NO ₃ ⁻)	0.02	0.08
Phosphate (-P)	0.03–33	0.9999	27 (70 as PO ₄ ³⁻)	0.15 (-P)	0.7 (2 as PO ₄ ³⁻)	0.04	0.14

^a MDL = (t) × (S) where t = Student's t value for a 99% confidence level and a standard deviation estimate with n - 1 degrees of freedom [t = 3.14 for seven replicates of the MDL Standard], and S = standard deviation of the replicate analysis

^b Relative standard deviation, n = 7

^c Quadratic fit

Table 3 summarizes the calibration data, the method detection limits (MDLs), retention time, and peak area precisions for the common anions and cations. MDLs and precision data were obtained from seven replicate injections of the MDL and QCS standards, respectively, prepared in DI water. Anion and cation MDL standards were prepared at concentrations of 3–5× the estimated method detection limits.

Correlation coefficient values obtained from the calibration plots were between 0.9994 and 0.9999. The calibration curves were linear for all anions and cations except

ammonium. Analytes that form weak acids or bases in the suppressor are known to exhibit nonlinear behavior. A quadratic curve-fitting function was used for ammonium. The retention time precision ranged from <0.01–0.07%, and the peak area precision ranged from 0.04–0.97%. The high retention time precisions are attributed to consistent generation of high-purity KOH and MSA using the eluent generator module and the respective continuously regenerated trap columns (Dionex CR-ATC and Dionex CR-CTC columns).

Table 4. Between-day (n = 3) retention time (RT) and peak area precisions (triplicate injections of spiked sample).

Cations					Anions				
San Francisco Bay Water									
Analyte	RT (min)	RT Precision (RSD)	Peak Area (μS* min)	Peak Area Precision (RSD)	Analyte	RT (min)	RT Precision (RSD)	Peak Area (μS* min)	Peak Area Precision (RSD)
Lithium	2.57	<0.01	0.72	0.13	Fluoride	3.63	<0.01	0.39	0.57
Sodium	3.19	0.06	18.4	0.07	Chloride	5.45	0.00	40.3	0.31
Ammonium	3.61	0.02	0.32	0.20	Nitrite	6.77	0.02	0.61	0.64
Potassium	4.59	0.03	0.51	0.40	Bromide	9.63	0.03	0.14	0.82
Magnesium	7.87	0.02	4.31	0.12	Sulfate	10.53	0.01	5.14	0.20
Calcium	9.96	0.02	0.91	0.38	Nitrate	11.57	0.01	0.26	0.63
					Phosphate	15.44	0.03	0.09	0.65
Commercial Aquarium Sea Salt									
Lithium	2.57	0.05	0.72	0.15	Fluoride	3.63	0.05	0.37	0.52
Sodium	3.19	0.02	18.4	0.05	Chloride	5.45	0.02	40.2	0.18
Ammonium	3.61	<0.01	0.29	0.54	Nitrite	6.77	0.02	0.63	0.79
Potassium	4.59	0.03	0.52	0.30	Bromide	9.63	0.01	0.14	0.63
Magnesium	7.91	0.02	4.32	0.09	Sulfate	10.54	0.03	5.11	0.26
Calcium	10.08	0.01	0.88	0.41	Nitrate	11.57	0.01	0.26	0.68
					Phosphate	15.45	0.02	0.09	1.01
Artificial Seawater									
Lithium	2.57	0.05	0.71	0.22	Fluoride	3.63	<0.01	0.37	0.39
Sodium	3.20	0.04	21.2	0.05	Chloride	5.45	0.01	45.83	0.22
Ammonium	3.61	<0.01	0.31	0.52	Nitrite	6.77	0.02	0.62	0.61
Potassium	4.59	0.03	0.58	0.37	Bromide	9.63	0.01	0.15	0.62
Magnesium	7.91	0.01	4.56	0.36	Sulfate	10.52	0.02	3.92	0.27
Calcium	10.07	0.03	0.94	0.50	Nitrate	11.57	0.01	0.27	0.24
					Phosphate	15.44	0.02	0.09	0.55

Method performance was evaluated by measuring recoveries in samples of spiked saline (Table 4) and drinking water (Table 5). Samples were spiked with analytes at a level that was 50–100% of the amount determined in the original sample. The between-day precision for anions and cations in the spiked samples ranged from < 0.01–1.6% over three days.

Figure 2 shows the separation of A) anions and B) cations in water from California's San Francisco Bay. The bay water sample is representative of the typical feed water into a desalination plant. The bay water sample was diluted 200-fold so that measured levels were within the calibrated range. The major inorganic anions in bay water are chloride and sulfate, and the major inorganic cations are sodium, potassium, magnesium, and calcium.

Table 5. Between-day (n = 3) retention time (RT) and peak area precisions (triplicate injections of spiked sample).

Cations					Anions				
Tap Water									
Analyte	RT (min)	RT Precision (RSD)	Peak Area (µS* min)	Peak Area Precision (RSD)	Analyte	RT (min)	RT Precision (RSD)	Peak Area (µS* min)	Peak Area Precision (RSD)
Lithium	2.57	0.05	0.75	0.13	Fluoride	3.63	0.04	0.82	0.34
Sodium	3.16	0.04	8.34	0.10	Chloride	5.47	0.01	7.47	0.32
Ammonium	3.61	0.02	0.48	0.14	Nitrite	6.78	0.01	0.29	0.46
Potassium	4.59	<0.01	0.27	0.15	Bromide	9.64	0.02	0.12	0.76
Magnesium	7.91	0.02	4.11	0.19	Sulfate	10.53	0.03	5.13	0.34
Calcium	9.97	0.01	6.57	0.18	Nitrate	11.56	0.01	0.31	0.66
					Phosphate	15.44	0.01	0.09	0.89
Bottled Mineral Water									
Lithium	2.57	0.03	0.75	0.13	Fluoride	3.63	0.04	0.90	0.49
Sodium	3.16	<0.01	8.57	0.07	Chloride	5.47	0.01	6.12	0.33
Ammonium	3.61	0.04	0.29	0.54	Nitrite	6.76	0.02	0.22	0.65
Potassium	4.59	0.01	0.61	0.09	Bromide	9.61	0.01	0.12	0.76
Magnesium	7.80	0.02	11.4	0.12	Sulfate	10.44	0.03	8.52	0.50
Calcium	9.73	0.01	18.5	0.21	Nitrate	11.52	0.02	0.74	0.34
					Phosphate	15.51	0.03	0.09	0.53

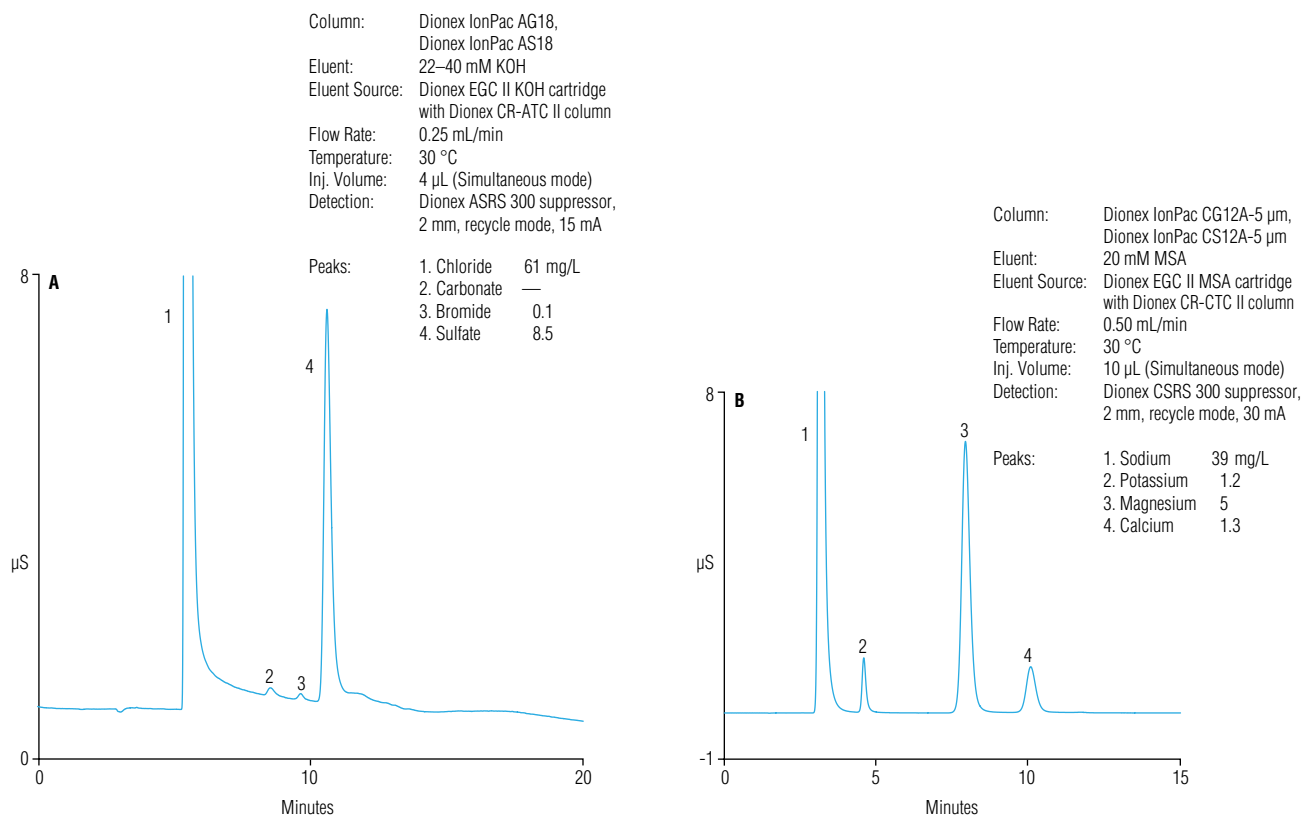


Figure 2. San Francisco, CA bay water: determination of common A) inorganic anions using the Dionex IonPac AS18 column and B) cations using the Dionex IonPac CS12A column.

Table 6. Between-day (n = 3) retention time (RT) and peak area precisions (triplicate injections of spiked sample).

Analyte	San Francisco Bay Water		Artificial Seawater		Commercial Aquarium Sea Salt		Bottled Mineral Water		Tap Water	
	Amount Added (mg/L)	Recovery* (%)	Amount Added (mg/L)	Recovery* (%)	Amount Added (mg/L)	Recovery* (%)	Amount Added (mg/L)	Recovery* (%)	Amount Added (mg/L)	Recovery* (%)
Lithium	1	93.5	1	92.4	1	98.1	1.0	96.9	1	97.2
Sodium	40	89.9	40	95.3	40	90.9	20.1	93.1	20	96.6
Ammonium	1	108.3	1	105.7	1	105.9	1.0	97.9	1	99.9
Potassium	2	94.5	2	96.7	2	99.6	1.0	94.9	1	95.7
Magnesium	5	97.2	5	97.4	5	97.6	5.0	94.6	5	97.1
Calcium	2	88.4	2	83.7	2	82.8	15.0	95.7	15	85.6
Fluoride	1	109.6	1	107.0	1	106.9	1	98.1	1	98.7
Chloride	74	87.2	74	92.7	74	89.7	16	84.2	16	85.9
Nitrite (-N)	1	100.7	1	101.7	1	105.3	1	37.2	1	41.5
Bromide	1	84.4	1	83.6	1	84.1	1	87.9	1	88.4
Sulfate	16	81.2	16	83.9	16	84.9	16	93.9	16	84.4
Nitrate (-N)	0.2	98.4	0.2	103.7	0.2	100.0	0.2	100.9	0.2	101.3
Phosphate (-P)	0.3	88.0	0.3	84.3	0.3	86.6	0.3	88.4	0.3	85.0

* Average over 3 days

As seen in Figure 2 and Table 6, all anions and cations were well resolved and had acceptable recoveries (80–120%) using the criteria outlined in U.S. EPA Method 300.0.

Figure 3 (A and B) shows the chromatogram for all anions and cations in Sunnyvale, CA drinking water. Tap water samples have fluoride, chloride, and sulfate as the predominant inorganic anions. Table 6 lists the recoveries of anions and cations in the drinking water matrices. All anions and cations were well resolved and, with the exception of nitrite, had acceptable recoveries (80–120%). The low recovery of nitrite can be attributed to biological activity in these samples (which is minimal in the high saline matrices) and the instability of nitrite in oxidizing environments, such as chlorinated water or other oxidizing disinfectants in drinking water.

In summary, the current methods using the Dionex IonPac AS18 and Dionex IonPac CS12A columns provide acceptable recoveries for anions and cations in both saline and drinking water matrices. This work shows methods that can be used for diverse matrices that are typically encountered in a desalination plant.

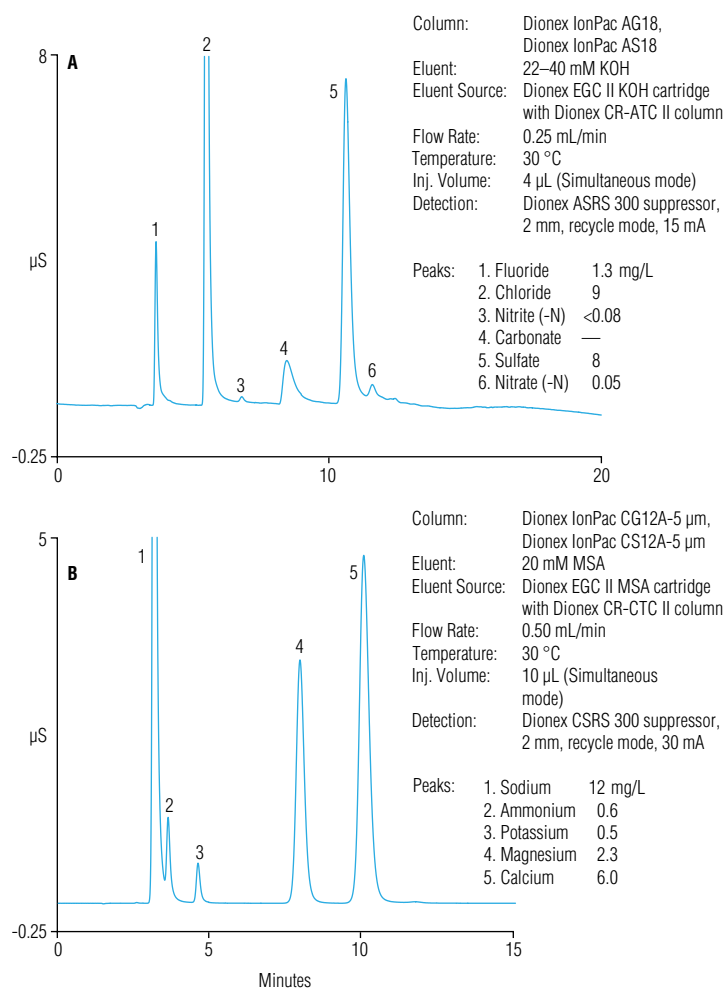


Figure 3. Sunnyvale, CA tap water: determination of common A) inorganic anions using the Dionex IonPac AS18 column and B) cations using the Dionex IonPac CS12A column.

Conclusion

Dionex IonPac AS18 and Dionex IonPac CS12A columns with electrolytically generated hydroxide and MSA eluents can simultaneously determine anions and cations in saline and drinking water matrices. The capacities of the Dionex IonPac AS18 and Dionex IonPac CS12A columns allow sample analysis with minimal sample pretreatment. The RFIC-EG system allows continuous operation of the instrument with minimal maintenance. Only water for eluent generation and suppressor regeneration must be added to keep the instrument running for sample analysis. Additionally, the smaller column format generates less waste and uses less eluent, saving both time and money. The methods were shown to be accurate by the good recovery of anions and cations in a wide variety of samples including natural and artificial seawater and drinking water. These methods are robust for all ion-monitoring needs of a typical desalination facility and support a varying range of matrices from seawater to drinking water.

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