

Analysis of Environmental Waters for Cations and Ammonium Using a Compact Ion Chromatography System

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Key Words

RFIC system, Integrion, Dionex IonPac CS16-4 μ m, wastewater, drinking water

Goal

Determine inorganic cations and ammonium in environmental waters using a compact high-pressure ion chromatography system with a 4 μ m particle cation-exchange column to shorten analysis time.

Introduction

Common alkali and alkaline earth cations are not considered primary drinking water contaminants in the U.S., but they are monitored and reported by many public water suppliers and are regulated in the EU and Japan.

While calcium and magnesium are essential minerals that have beneficial human health effects, they are routinely measured to determine water hardness, an important parameter for control of corrosion and scaling.¹

Ammonia is a colorless, pungent gas that is highly soluble in water, where it exists in equilibrium between a molecular form associated with water and the ionized form (the ammonium cation, NH_4^+). Its toxicity to aquatic life depends upon the extent of dissociation, which in turn depends upon temperature and pH. Ammonia can enter environmental waters as a product of anaerobic decomposition of nitrogen-containing compounds or from waste streams containing ammonia. Ammonium, which is formed upon dissolution of ammonia in water, is routinely measured in the U.S. for wastewater discharge compliance monitoring and in the EU and Japan in both wastewater and drinking water.²

In AN 141, the Thermo Scientific™ Dionex™ IonPac™ CS16 cation-exchange column was combined with the Thermo Scientific Dionex DX-600 chromatography system, which used an electrolytically generated methanesulfonic acid (MSA) eluent, and suppressed conductivity detection to determine dissolved alkali and alkaline earth cations and ammonium in drinking water, wastewater, and aqueous soil extracts.⁴ The linear range,



method detection limits (MDLs), and analyte recoveries were discussed and the effect of potential interferences on method performance during the analysis of typical environmental samples was evaluated.

This Application Update revises AN141 using the compact, integrated Thermo Scientific™ Dionex™ Integrion™ HPIC™ system with a Thermo Scientific™ Dionex™ IonPac™ CS16-4 μ m cation-exchange column.

The simplified, intuitive flow-path-based plumbing, thermally isolated temperature-controlled module compartments, and reagent-free capability of the Dionex Integrion HPIC system shortens time to equilibration and ensures consistent results.

The Dionex IonPac CS16-4 μ m column is a high-capacity cation exchange column with 100% solvent compatibility and medium hydrophobicity. The high capacity of 5370 $\mu\text{eq}/\text{column}$ is achieved by using a high density of grafted carboxylic acid cation exchange groups. The high capacity improves performance for trace-level determinations of cations in high ionic strength matrices by extending the linear range and resolving higher concentration ratios of sodium and ammonium. The 4 μ m particle size produces more efficient separations, facilitating peak integration and permitting faster flow rates without compromising resolution. These features result in shorter run times and make higher sample throughput possible.

While faster flow rates produce higher back pressure, the high pressure capability (up to 5000 psi with reagent-free IC (RFIC) and 6000 psi with manual eluent preparation) of the Dionex Integrion HPIC system extends the range of eluent flow rates that can be used, allowing the system to take maximum advantage of smaller particle columns.

Software

- Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System (CDS) software, version 7.2 SR4

Reagents and Standards

- Deionized (DI) water, Type I reagent grade, 18 MΩ-cm resistivity or better
- ULTRA Scientific
 - Lithium standard, 1000 mg/L (P/N ICC-104)
 - Sodium standard, 1000 mg/L (P/N ICC-107)
 - Ammonium standard, 1000 mg/L (P/N ICC-101)
 - Potassium standard, 1000 mg/L (P/N ICC-106)
 - Magnesium standard, 1000 mg/L (P/N ICC-105)
 - Calcium standard, 1000 mg/L (P/N ICC-103)
- Fisher Scientific, ACS Grade
 - Lithium chloride (P/N L121)
 - Sodium chloride (P/N S271)
 - Ammonium chloride (P/N A661)
 - Potassium chloride (P/N P217)
 - Magnesium chloride hexahydrate (P/N M33)
 - Calcium chloride dihydrate (P/N C79)
- Thermo Scientific™ Dionex™ Combined Six Cation Standard-II (P/N 046070)

Conditions

Columns:	Dionex IonPac CG16-4µm Guard and Dionex IonPac CS16-4µm Separation 4 mm (i.d.) column set
Eluent Source:	Dionex EGC 500 MSA cartridge with Dionex CR-CTC 600 Continuously Regenerated Anion Trap Column
Eluent Conc.:	30 mM MSA
Flow Rate:	0.9 mL/min
Column Temp.:	40 °C
Inj. Volume:	10 µL (Full loop)
Detection:	Suppressed conductivity, Dionex CERS 500 Electrolytic Suppressor, 4 mm, recycle mode, 79 mA
Detector Compart. Temp.:	15 °C
Background:	<1 µS
Noise:	~ 0.2 nS
Backpressure:	~4100 psi
Run Time:	22 min

Preparation of Standards and Reagents

Stock Standard Solutions

Purchase certified solutions or prepare 1000 mg/L stock standard solutions of each of the cations of interest by accurately weighing the amounts of reagent-grade salts given in Table 2. Dissolve in reagent water in a 100 mL plastic volumetric flask. Dilute to volume with reagent water. Store in plastic containers at 4 °C. Stock standards are stable for at least three months.

Table 1. Consumables list.

Product Name	Description	Part Number
Thermo Scientific™ Dionex™ EGC 500 MSA cartridge	Methanesulfonic acid eluent generator cartridge	075779
Thermo Scientific™ Dionex™ CR-CTC 600	Continuously regenerated cation trap column	088663
Thermo Scientific™ Dionex™ CERS™ 500 Electrolytically Regenerated Suppressor, 4 mm	Cation electrolytically regenerated suppressor, 4 mm	082542
Dionex IonPac CG16-4µm Column, 4 mm	Guard column, 4 × 50 mm	088585
Dionex IonPac CS16-4µm Column, 4 mm	Separation column, 4 × 250 mm	088584
Thermo Scientific™ Nalgene™ Syringe Filter	Syringe filters, 25 mm, PES membrane, 0.2 µm	Thermo Scientific 7252520/ Fisher Scientific 09-740-113
Dionex AS-AP Autosampler vials	Polystyrene vials with caps and blue septa, 10 mL, Package of 100	074228

Table 2. Mass of compound required to prepare 100 mL of a 1 g/L solution of cation.

Analyte	Compound	Mass (g)
Li ⁺	LiCl	0.6108
Na ⁺	NaCl	0.2542
NH ₄ ⁺	NH ₄ Cl	0.2965
K ⁺	KCl	0.1907
Mg ²⁺	MgCl ₂ • 6H ₂ O	0.8365
Ca ²⁺	CaCl ₂ • 2H ₂ O	0.3668

Working Standard Solutions

Prepare working standards by diluting appropriate volumes of the 1000 mg/L stock standards with reagent water. The calibration standard concentrations used for lithium, potassium, magnesium, and calcium were 0.03, 0.08, 0.16, 0.32, 0.8, 1.6, 3.2, 8, 16, 80 mg/L; for sodium 0.1, 0.2, 0.4, 1, 2, 4, 10, 20, 40, 100, 200, and 1000 mg/L; for ammonium 0.02, 0.04, 0.08, 0.16, 0.4, 0.8, 1.6, 4, 8, and 40 mg/L. Prepare working standards daily if they contain less than 100 mg/L of the cations.

Sample Preparation

Filter all water samples through a 0.2 µm Nalgene PES syringe filter. A 0.45 µm pore size filter of the same material should also be effective. Discard the first 300 µL of filtrate and filter the remainder directly into a clean plastic autosampler vial. Prepare aqueous soil extracts by adding 30 mL of 30 mM MSA to 3.0 g of soil. Extract in an ultrasonic bath for 30 min. Centrifuge 5 min, ~3400 × g to clear the solution and then filter through a 0.2 µm IC syringe filter as above.

System Preparation and Configuration

Configure the system as described in Technical Note 175⁵, using the Dionex Integriion Operator's Manual for additional reference.⁶ Install and condition the Dionex EGC 500 MSA eluent generator cartridge as described in the product manual.⁷ Install and hydrate the Dionex CR-CTC 600 trap column⁸ and the Dionex CERS 500 suppressor.⁹ Install and condition the guard and separation columns for 30 min prior to installing the columns inline with the suppressor.¹⁰

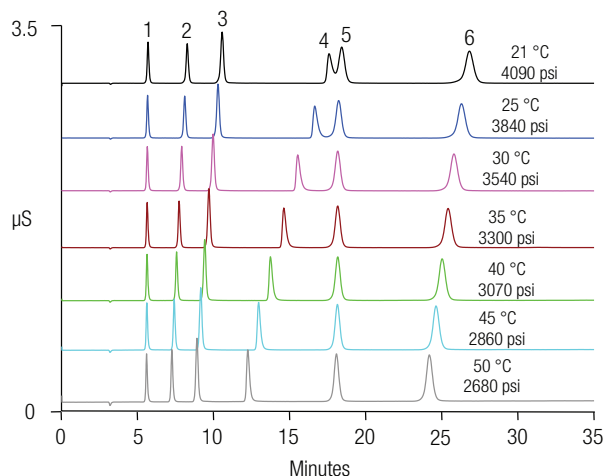
Method Detection Limit Calculation

The method detection limit (MDL) is defined as the minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero. To establish the single operator, within-day MDL for each analyte, make seven replicate injections of reagent water fortified with each analyte at a concentration of three to five times the estimated instrument detection limit.¹¹ Perform all the calculations defined in the method and report the concentration values in the appropriate units.¹²

Results and Discussion

Impact of Temperature on Cation Separation

On the Dionex IonPac CS16-4µm column, the cation retention time varies with temperature (Figure 1). Of the six cations analyzed, the retention time of potassium was the most sensitive to temperature. This retention time variability can be exploited to optimize selectivity among analytes, but a constant temperature should be maintained to ensure the best possible retention time and minimize the misidentification of peaks. A column oven should be used for the best retention time reproducibility. Additionally, a thermally controlled detector compartment adds to the performance of the system by cooling the electrolytic suppressor, thus improving the signal to noise ratio of the detector and potentially extending suppressor longevity.

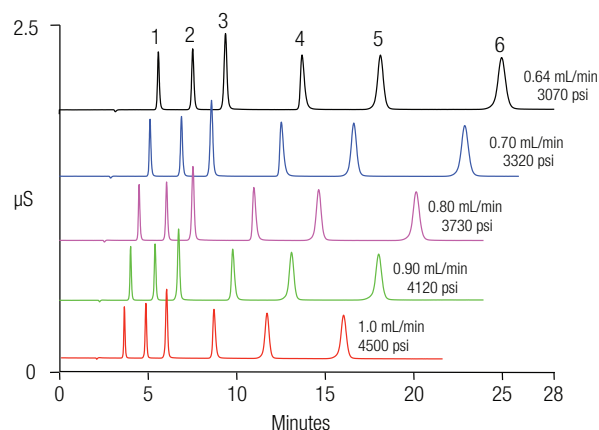


Column:	Dionex IonPac CG16-4µm/CS16-4µm columns, 4 mm i.d.
Eluent Source:	Dionex EGC 500 MSA cartridge, Dionex CR-CTC 600 trap column, high pressure degasser
MSA Conc.:	30 mM
Flow Rate:	0.64 mL/min
Inj. Volume:	10 µL
Col. Temp.:	21–50 °C
Detector Compartment Temp.:	15 °C
Detection:	Suppressed conductivity, Dionex CERS 500 Cation Electrolytic Suppressor, recycle mode, 57 mA
Peaks:	
	1. Lithium 0.1 mg/L
	2. Sodium 0.4
	3. Ammonium 0.5
	4. Potassium 1.0
	5. Magnesium 0.5
	6. Calcium 1.0

Figure 1. Comparison of cation separation at various temperatures.

Impact of Eluent Flow Rate on Run Time and Peak Resolution

The cation standard solution was initially run at the Quality Assurance Report (QAR) conditions of 0.64 mL/min, but to reduce the run time, flow was increased up to 1 mL/min (Figure 2). Even at this maximum recommended flow rate, the cation standards were baseline resolved. Good peak resolution (> 1.5) at all the flow rates was achieved using a 4- μm -particle column, which has greater peak efficiencies than standard, larger-particle-size columns. A flow rate of 0.9 mL/min was used for the remainder of the data presented here because it produced a relatively short run time while yielding sufficient resolution to quantify ammonium in the presence of a 10,000-fold excess of sodium. Using this flow rate and a 40 °C column temperature, calcium, the longest retained cation, eluted within 18 min, seven minutes faster than when using the conditions in AN141. One of the consequences of using smaller-particle columns is an increase in system pressure, which rose from 3070 to 4500 psi when the flow rate was increased from 0.64 to 1.0 mL/min. Pressures above 3000 psi necessitate the use of a high pressure IC system, such as the Dionex Integration HPIC system.



Column: Dionex IonPac CG16-4 μm /CS16-4 μm columns, 4 mm i.d.
 Eluent Source: Dionex EGC 500 MSA cartridge, Dionex CR-CTC 600 trap column, high pressure degasser
 MSA Conc.: 30 mM
 Flow Rate: 0.64–1.0 mL/min
 Inj. Volume: 10 μL
 Col. Temp.: 40 °C
 Detector Compartment Temp: 15 °C
 Detection: Suppressed conductivity, Dionex CERS 500 Cation Electrolytic Suppressor, recycle mode, 57–88 mV

Peaks:

1. Lithium	0.1 mg/L
2. Sodium	0.4
3. Ammonium	0.5
4. Potassium	1.0
5. Magnesium	0.5
6. Calcium	1.0

Figure 2. Comparison of cation retention times at different flow rates.

Method Linear Calibration Ranges and Method Detection Limits

Table 3 summarizes the calibration data and method detection limits (MDLs) obtained for the six cations determined. To establish the linear calibration range, the peak responses to concentration were determined using injections of calibration standards over a 2000-fold or greater concentration range. The high capacity of the Dionex IonPac CS16-4 μm column results in a calibration curve that is linear over three orders of magnitude for most of the cations, except ammonium. The nonlinear dependence of area on amount is common for analytes that form weak acids or weak bases in the suppressor.¹³ A quadratic curve-fitting function extends the calibration curve for ammonium to a concentration of 40 mg/L. Coefficients of determination (r^2) ranged from 0.9996 to 1.000.

Table 3. Linear range and MDLs for cations and ammonium.

Cation	Range (mg/L)	Coefficient of Determination (r^2)	MDL Standard ($\mu\text{g/L}$)	Calculated MDL ^a ($\mu\text{g/L}$)
Li ⁺	0.03–80	1.000	1	0.12
Na ⁺	0.1–1000	0.9999	4	0.70
NH ₄ ⁺ ^b	0.02–40	0.9996	5	0.89
K ⁺	0.03–80	0.9999	10	1.12
Mg ²⁺	0.03–80	1.000	5	0.75
Ca ²⁺	0.03–80	0.9999	10	2.24

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

^b Quadratic fit

Suppressed conductivity allowed detection down to the low $\mu\text{g/L}$ level with a 10 μL sample injection (Table 3). The MDLs were comparable to those obtained in AN141. Depending on the ionic strength and nature of the sample matrix, lower detection limits may be achieved by injecting more sample.

Method Precision

The Dionex Integration system, configured with automated eluent generation, conveniently generates a high purity MSA eluent with a very stable composition, leading to highly reproducible results. This stability is reflected in the retention time (t_R) and peak area reproducibility summarized in Table 4 for seven injections of a quality control standard (QCS). The t_R precision ranged from 0.01% to 0.03% and the area precision from 0.08% to 0.19% RSD, which was 3- to 20-fold and 5- to 10-fold better than the values obtained in AN141, respectively.

Table 4. Retention time and peak area precision results for cations and ammonium.

Cation	QCS ^a (mg/L)	t _r Precision (RSD) ^b	Area Precision (RSD) ^b
Li ⁺	1	0.01	0.16
Na ⁺	4	0.02	0.16
NH ₄ ⁺	5	0.03	0.08
K ⁺	10	0.02	0.19
Mg ²⁺	5	0.01	0.16
Ca ²⁺	10	0.01	0.19

^a Quality Control Standard

^b Relative standard deviation, n = 7

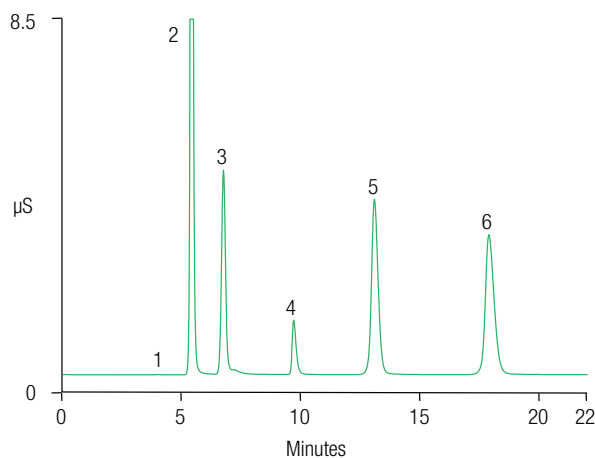
A typical way to validate the performance of methods used for environmental analysis is through precision and bias studies on spiked samples.¹⁴ For the method presented here, the levels of inorganic cations and ammonium in various environmental water samples was first determined. Samples were then spiked with the analytes at a level that was 50–100% of this amount. Table 5 summarizes the spike recovery of inorganic cations from various environmental water matrices. Recoveries were ~ 100% in all matrices, ranging from 89–117%, which is within an acceptable range of 80–120%.¹⁴

Table 5. Recovery of inorganic cations in water samples.

Reagent Water			
Cation	Measured (mg/L)	Amount Added (mg/L)	Recovery (%)
Li ⁺	<0.03	0.5	117%
Na ⁺	<0.1	0.5	90%
NH ₄ ⁺	<0.02	0.5	113%
K ⁺	<0.03	0.5	106%
Mg ²⁺	<0.03	0.5	102%
Ca ²⁺	<0.03	0.5	100%
Drinking Water			
Li ⁺	<0.03	0.5	107%
Na ⁺	5.4	10	102%
NH ₄ ⁺	0.62	1	112%
K ⁺	0.33	0.5	103%
Mg ²⁺	0.44	0.5	97%
Ca ²⁺	4.4	5	98%
Municipal Wastewater			
Li ⁺	<0.03	0.5	117%
Na ⁺	34.0	50	100%
NH ₄ ⁺	9.6	15	89%
K ⁺	3.6	5	103%
Mg ²⁺	6.5	10	99%
Ca ²⁺	11.0	15	100%

Cations in Environmental Water

The Dionex IonPac CS16-4μm column is a high capacity column, therefore, environmental samples, such as municipal wastewater, which can have a wide range of ionic strength, can be analyzed with minimal dilution while still be able to determine low concentration analytes (Figure 3).



Column: Dionex IonPac CG16-4μm/CS16-4μm columns, 4 mm i.d.

Eluent Source: Dionex EGC 500 MSA cartridge, Dionex CR-CTC 600 trap column, high pressure degasser

MSA Conc.: 30 mM

Flow Rate: 0.9 mL/min

Inj. Volume: 10 μL

Col. Temp.: 40 °C

Detector Compartment

Temp.: 15 °C

Detection: Suppressed conductivity, Dionex CERS 500 Cation Electrolytic Suppressor, recycle mode, 79 mA

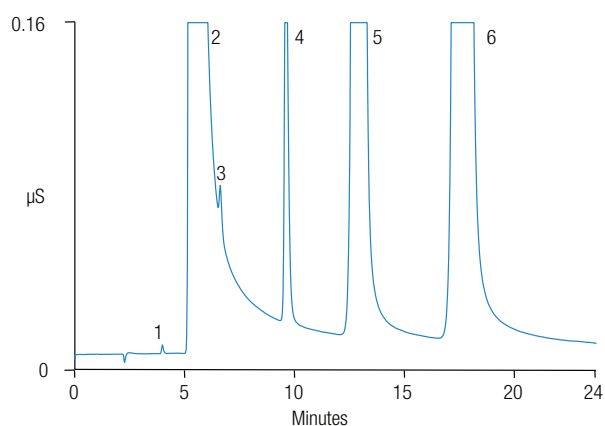
Sample: Municipal wastewater, filtered, 0.2 μm, diluted 5-fold

Peaks:

1. Lithium	< 0.03 mg/L
2. Sodium	34.0
3. Ammonium	9.6
4. Potassium	3.6
5. Magnesium	6.5
6. Calcium	11.0

Figure 3. Determination of inorganic cations and ammonium in municipal wastewater.

The Dionex IonPac CS16-4 μ m column is ideal for the determination of low concentrations of ammonium in environmental waters. It provides improved resolution of sodium from ammonium and alkanolamines, even for high ionic strength samples. Figure 4 illustrates the determination of trace-level ammonium in the presence of high sodium (1:10,000). For quantification, the ammonium peak was integrated using the rider detection function in the Dionex Chromeleon CDS software setting the skimming to exponential. A recovery of 119% was obtained when 0.02 mg/L ammonium was spiked into a sample containing 200 mg/L sodium, as per the figure. Even more disparate ratios can be measured if the elution conditions are modified using gradient conditions such that a lower eluent concentration is used initially to improve the sodium and ammonium separation and then increased to expedite the elution of the later-eluting cations. A lower flow rate can also be used.



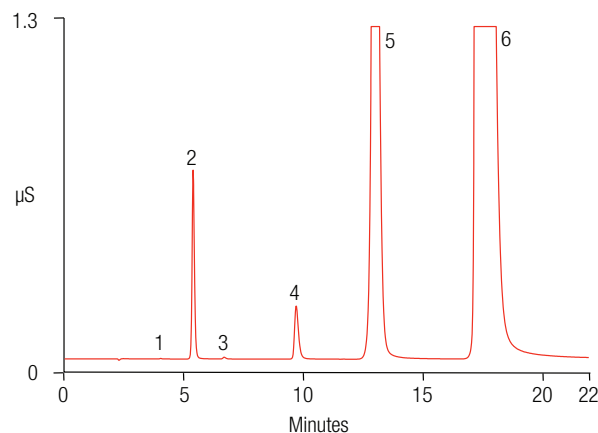
Column: Dionex IonPac CG16-4 μ m/CS16-4 μ m columns,
4 mm i.d.
Eluent Source: Dionex EGC 500 MSA cartridge,
Dionex CR-CTC 600 trap column,
high pressure degasser
MSA Conc.: 30 mM
Flow Rate: 0.9 mL/min
Inj. Volume: 10 μ L
Col. Temp.: 40 $^{\circ}$ C
Detector
Compartment
Temp.: 15 $^{\circ}$ C
Detection: Suppressed conductivity, Dionex CERS 500 Cation
Electrolytic Suppressor, recycle mode, 79 mA

Peaks:

1. Lithium	< 0.03 mg/L
2. Sodium	200.0
3. Ammonium	0.02
4. Potassium	0.5
5. Magnesium	8.0
6. Calcium	20

Figure 4. Determination of trace ammonium in the presence of high sodium (1:10,000).

The Dionex IonPac CS16-4 μ m column packing is compatible with acidic eluents and samples such as acid digests, acid-preserved samples, or acidic soil extracts (Figure 5). The method permits the injection of 10 μ L sample volume containing as much as 100 mmol/L hydronium directly on column. In case of higher acid concentrations, a simple dilution can be used as sample preparation to lower the acid concentration.



Column: Dionex IonPac CG16-4 μ m/CS16-4 μ m columns,
4 mm i.d.
Eluent Source: Dionex EGC 500 MSA cartridge,
Dionex CR-CTC 600 trap column,
high pressure degasser
MSA Conc.: 30 mM
Flow Rate: 0.9 mL/min
Inj. Volume: 10 μ L
Col. Temp.: 40 $^{\circ}$ C
Detector
Compartment
Temp.: 15 $^{\circ}$ C
Detection: Suppressed conductivity, Dionex CERS 500 Cation
Electrolytic Suppressor, recycle mode, 79 mA
Sample: Acidified soil, filtered, 0.2 μ m, diluted 10-fold

Peaks:

1. Lithium	< 0.03 mg/L
2. Sodium	0.79
3. Ammonium	< 0.03
4. Potassium	0.66
5. Magnesium	5.3
6. Calcium	51.0

Figure 5. Determination of inorganic cations and ammonium in acidic soil extract.

Strongly retained compounds from injected environmental water samples can accumulate on the column and degrade its performance. Signs of a fouled column include loss of capacity, loss of resolution, shortened retention times, higher noise and background, spurious peaks, and peak tailing. The Dionex IonPac CS16-4 μ m column can be flushed with acetonitrile to remove hydrophobic cations and organic contaminants, with HCl to remove polyvalent cations and acid-soluble contaminants, and with oxalic acid to remove transition metals.¹⁰

Conclusion

This application used the Dionex IonPac CS16-4 μ m column with 30 mM MSA eluent at 40 °C and suppressed conductivity detection to determine inorganic cations and ammonium at concentrations ranging from 0.1–80 mg/L. The high pressure capability of the Dionex Integrion HPIC system allowed a flow rate of 0.9 mL/min to be used, which reduced the run time from 25 to just over 18 min. The high capacity and resolution of the Dionex IonPac CS16-4 μ m column enabled the analysis of a wide range of environmental waters, and resolved trace ammonium in the presence of a 10,000-fold higher concentration of sodium. The retention time precision of 0.03% or less and the peak area precision of 0.2% or less for standards in the low mg/L range demonstrated the exceptional reproducibility of the analytical solution presented. The use of HPIC instrumentation provides the possibility to use smaller particle size columns at higher flow rates to reduce the run time, while maintaining the chromatographic resolution.

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