Efficient and Fast Separations of Inorganic Anions in Water Samples Using a 4 µm Particle Size Microbore Column with a High-Pressure Ion Chromatography System

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Technical Note 127

Key Words
Drinking water, Wastewater, Municipalities, IonPac AS18-4µm, HPIC, ICS-5000+

Goal
Demonstrate high resolution and short run times using a 4 µm resin anion-exchange separation column on a high-pressure ion chromatography system.

Introduction

In 1993, the U.S. EPA Method 300.0 (A) defined the use of the Thermo Scientific™ Dionex™ IonPac™ AS4A anion-exchange column to determine inorganic anions in environmental waters using a manually prepared carbonate/bicarbonate eluent and suppressed conductivity detection.[1] With the introduction of Reagent-Free™ ion chromatography (RFIC™) systems, hydroxide-based eluents were electrolytically generated inline on anion-exchange columns optimized for hydroxide chemistry. Reagent-free IC systems deliver reproducible and precise eluent concentrations that are produced inline by the addition of just water. These systems eliminate labor that would normally be spent on manual eluent preparation, removing a potential source of error and operator-to-operator variability.

The Dionex IonPac AS18-4µm column is a newly developed high capacity column with AS18 chemistry using 4 µm resin particles.[2] The Dionex IonPac AS18 column was developed as an alternative to the AS4A column; the AS18 is designed for use with EPA Method 300.0 (A) using Electrolytically Generated hydroxide eluents. These smaller resin particles allow an optimal combination of fast speed and high resolution. For the determination of inorganic anions in water samples, a comparison of the new 4 µm versus the previous 7.5 µm resin Dionex IonPac AS18 column is performed.

With the introduction of high-pressure IC, the Thermo Scientific™ Dionex™ ICS-5000+ Reagent-Free HPIC™ system has been designed to operate at pressures up to 5000 psi using all column formats: standard (4 mm i.d.), microbore (2 mm i.d.), and capillary (0.4 mm i.d.). Higher operating pressures allow the use of faster flow rates, which result in shorter run times.

This document presents the advantages of using the small-particle size microbore (2 mm i.d.) Dionex IonPac AS18-4µm column, in combination with the Dionex ICS-5000+ HPIC system to obtain fast and efficient separation of inorganic anions in municipal drinking and wastewater samples.

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Equipment

Dionex ICS-5000+ HPIC system

- Thermo Scientific Dionex SP/DP Pump module
- Thermo Scientific Dionex EG Eluent Generator module with high-pressure degas module
- Thermo Scientific Dionex DC Detector/ Chromatography module
- Thermo Scientific Dionex AS-AP Autosampler
- Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data system (CDS) software ver. 6.8 or 7.1

Reagents and Standards

18 MΩ-cm degassed deionized water

Fisher Scientific reagents, ACS Grade

Samples

Municipal drinking and wastewater samples collected from two cities in California.

Conditions	
Columns:	Dionex IonPac AG18-4µm, 2 × 50 mm Dionex IonPac AS18-4µm, 2 × 150 mm
Eluent Source:	Thermo Scientific Dionex EGC 500 KOH Eluent Generator Cartridge Thermo Scientific Dionex CR-ATC 500 Continuously Regenerated Anion Trap Column
Eluent:	23 mM KOH
Flow Rate:	0.25, 0.30, 0.38 mL/min for flow rate experiments 0.38 mL/min for samples
Column Temp.:	30 °C
Inj. Volume:	5 µL
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ ASRS™ 300 Self-Regenerating Suppressor, recycle; 15, 18, 22 mA
Background Conductance:	< 1.0 µS
Noise:	< 3 nS
System backpressure: 2500–3600 psi	

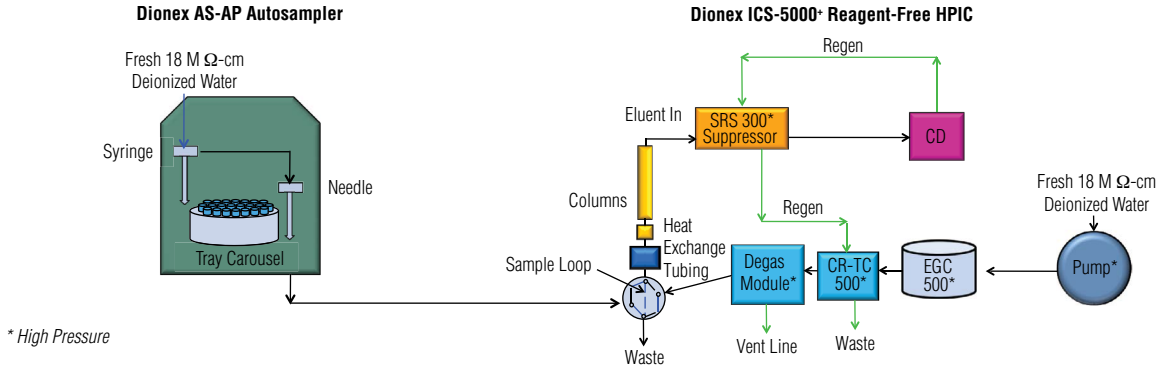

Figure 1. Flow diagram for the high-pressure Dionex ICS-5000+ Reagent-Free HPIC system.

Table 1. Consumables Table.

Product name	Type, Capillary	Dionex Part Number
Dionex EGC 500 KOH*	Anion Eluent Generator cartridge	075778
Dionex CR-ATC 500*	Anion electrolytic trap column	075550
Dionex IonPac AG18-4µm	Anion guard column	076037
Dionex IonPac AS18-4µm	Anion separation column	076036
Dionex ASRS 300*	Anion suppressor	064555
Dionex HP Degas Module*	Degas module	075522
Dionex HP fittings (blue)	Bolts / Ferrules*	074449 / 074373
EG Degas HP cartridge	High pressure degas cartridge, up to 5000 psi	074459
Dionex AS-AP autosampler vials	Package of 100, polystyrene vials, caps, blue septa	074228

Standard and Sample Preparation

The calibration standard solutions were prepared by diluting individual standard stock solutions to obtain the final concentrations listed in Figures 2–4.

Sample solutions were diluted with 18 MΩ-cm resistivity deionized water and filtered with a syringe filter (0.45 µm) without any pretreatment prior to injection. The amount of sample dilution is indicated in Figure 4.

Instrument Setup and Installation

Tip: To achieve the best chromatography with high-pressure IC, it is important to use high pressure connectors and ferrules (see Table 1) for all connections prior to the suppressor. The high-pressure Dionex ICS-5000⁺ HPIC Reagent-Free system is designed to operate from 2000 to 5000 psi. To set up this application, plumb the consumables and modules of this system according to Figure 1.

Install and hydrate the Dionex EGC 500 cartridge, Dionex CR-TC 500 trap column, and 2 mm Dionex SRS 300 suppressor according to the product manual instructions. Complete the installation according to the flow diagram, using high-pressure connectors and ferrules (blue) for all of the fittings from the pump prior to the suppressor. Standard pressure fittings can be used for the suppressor and detector connections. Detailed instructions are described in Technical Note TN 129, the product manuals, and the instrument installation and operator's manuals.[3–6]

Results and Discussion

Figure 2 shows a comparison of the smaller-particle-size (4 µm) column Dionex IonPac AS18-4µm, 2 × 150 mm with the larger-particle-size (7.5 µm) column Dionex IonPac AS18-Fast, 7.5 µm, 2 × 150 mm for the separation of nine anions in a standards mixture. Note that only half the amount of standard mixture was injected onto the 4 µm column. Good separation was obtained with both columns, although resolution and peak efficiency were enhanced with the smaller-particle-size column (4 µm). This better resolution can be exploited to achieve faster run times by using elevated flow rates without the loss of peak discrimination.

Figure 3 illustrates the analysis of nine inorganic anions using a Dionex IonPac AS18-4µm, 2 × 150 mm column with several flow rates. At a flow rate of 0.38 mL/min, all of the analytes were separated within 6 minutes with no loss of resolution compared to the slower runs. At this flow rate, a backpressure of 3600 psi is produced, which is well within the maximum pressure limit (5000 psi) of the Dionex ICS-5000⁺ HPIC system.

Column:	A: Dionex IonPac AS18-Fast, 2 × 150 mm B: Dionex IonPac AS18-4µm, 2 × 150 mm
Eluent Source:	A: Dionex EGC III KOH B: Dionex EGC 500 KOH
Eluent:	23 mM KOH
Flow Rate:	0.25 mL/min
Inj. Volume:	A: 5 µL; B: 2.5 µL
Column Temp.:	30 °C
Detection:	Suppressed conductivity, Dionex ASRS 300, 2 mm, recycle, 15 mA

Peaks:

1. Fluoride	0.5 mg/L	5. Carbonate	20.0 mg/L
2. Chlorite	5.0	6. Bromide	10.0
3. Chloride	3.0	7. Sulfate	10.0
4. Nitrite	5.0	8. Nitrate	10.0
		9. Chlorate	10.0

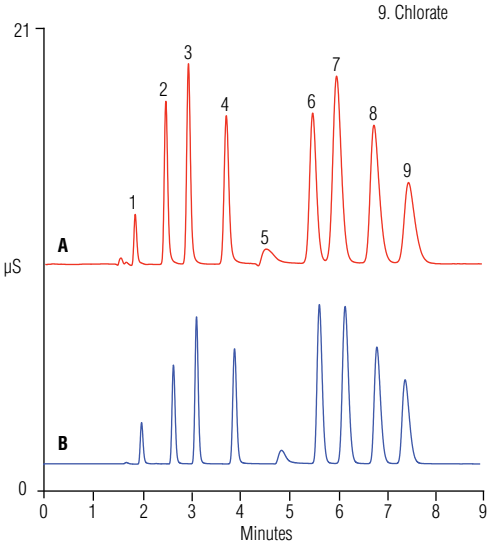

Figure 2. Comparison of the separation of nine inorganic anions using small-particle (4 µm) and large-particle (7.5 µm) analytical columns.

Column:	Dionex IonPac AG18-4µm, Dionex IonPac AS18-4µm, 2 × 150 mm
Eluent Source:	Dionex EGC 500 KOH
Eluent:	23 mM KOH
Flow Rate:	A: 0.25, B: 0.30, C: 0.38 mL/min
Inj. Volume:	5 µL
Column Temp.:	30 °C
Detection:	Suppressed conductivity, Dionex ASRS 300, 2 mm, recycle A: 15, B: 18, C: 22 mA
Back Pressure:	A: 2500; B: 3000; C: 3600 psi

Peaks:

1. Fluoride	0.5 mg/L	6. Bromide	10.0 mg/L
2. Chlorite	1.0	7. Sulfate	10.0
3. Chloride	3.0	8. Nitrate	20.0
4. Nitrite	5.0	9. Chlorate	10.0
5. Carbonate	--		

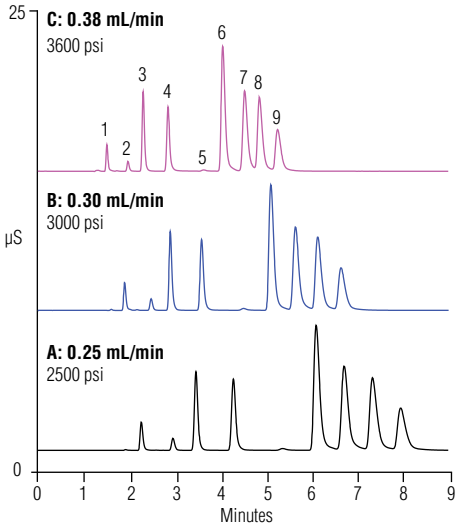

Figure 3. Comparison of the separation of inorganic anions using different flow rates.

Figure 4 shows the determination of inorganic anions in municipal drinking and waste water samples using a Dionex IonPac AS18-4µm, 2 × 150 mm column. The five anions that are commonly found in municipal water samples were resolved within 5 min, demonstrating the speed with which such runs can be performed using this column.

Column:	Dionex IonPac AG18-4µm, Dionex IonPac AS18-4µm, 2 × 150 mm
Eluent Source:	Dionex EGC 500 KOH cartridge
Eluent:	23 mM KOH
Flow Rate:	0.38 mL/min
Inj. Volume:	5 µL
Column Temp.:	30 °C
Detection:	Suppressed conductivity Dionex ASRS 300, 2 mm, recycle, 22 mA
Samples:	A. City 1 Drinking Water B. City 2 Drinking Water C. City 1 Wastewater D. City 2 Wastewater
Dilutions:	A: Undiluted B: 5-fold dilution C-D: 10-fold dilution

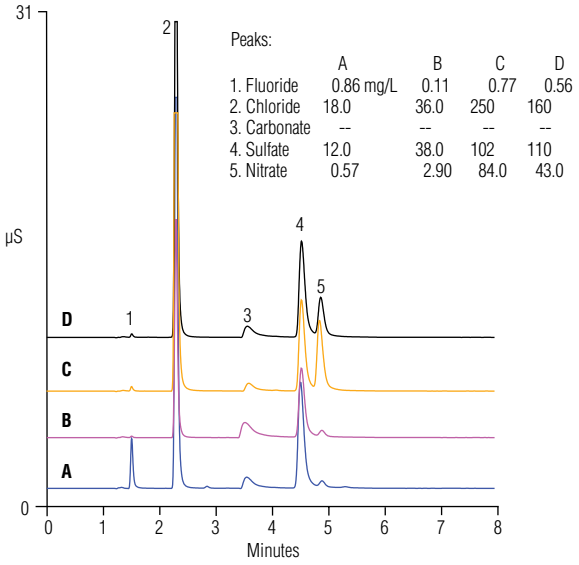

Figure 4. Determination of inorganic anions in municipal drinking and waste water samples.

Conclusion

This technical note demonstrates how the Dionex IonPac AS18-4 µm anion-exchange column can be used in combination with the Dionex ICS-5000⁺ HPIC system to obtain efficient separations with high resolution. It was demonstrated that all five anions present in samples eluted within 6 minutes. The efficiency of the Dionex IonPac AS18-4 µm analytical column permits the use of higher flow rates, reduces run times and, therefore, increases throughput for determination of inorganic anions in environmental water samples.

Additional information on inorganic anion determination in environmental and drinking water samples using standard bore and microbore IC are reviewed in application note AN 154 and application brief AB 132.[7–8]

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