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APPLICATION NOTE 72440

Determination of inorganic anions in wastewater using a capillary ion chromatography system

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Keywords

Dionex IonPac AS18-4µm capillary column, ICS-4000, drinking water, suppressed conductivity detection, RFIC system, Safe Drinking Water Act, Clean Water Act

Goal

To demonstrate the determination of inorganic anions in wastewater using a capillary ion chromatography (IC) system with a Thermo Scientific™ Dionex™ IonPac™ AS18-4µm capillary column and a 100 nL injection valve

Introduction

Ion chromatography (IC) is an established technique worldwide for monitoring inorganic anions in water, including surface, ground, drinking, and wastewaters. In the U.S., water quality is regulated through the Safe Drinking Water Act (SDWA) and the Clean Water Act (CWA).[1, 2] The SDWA ensures the integrity and safety of U.S. drinking water, whereas the goal of the CWA is to minimize the discharge of pollutants into the environment. IC methods have been approved for compliance monitoring of inorganic anions in drinking water since the 1980s through U.S. EPA Method 300.0,[3] which was updated in 1997 to U.S. EPA Method 300.1.[4] As an approved CWA Test Method, IC was adopted for wastewater analysis under the U.S. EPA Office of Water and the National Pollution Discharge Elimination System (NPDES) permits program.[2-5] In addition, many standard-setting organizations (including the International Organization for Standardization (ISO), American Society for

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Testing and Materials (ASTM), and American Water Works Association (AWWA) have validated IC methods for the determination of inorganic anions.[6,7]

IC methods for environmental water analysis have been demonstrated in Thermo Scientific Application Notes using standard or microbore flow rate columns with both carbonate/bicarbonate and hydroxide eluents.[8-11] Scaling down from standard bore (4 mm i.d.) to capillary format (0.4 mm i.d.) brings many benefits to IC users. The system can be always on and ready for analysis because of its low eluent consumption (15 mL of water a day at 0.01 mL/min flow rate) and therefore no time is wasted starting the system and preparing it for analysis. Additionally, the amount of waste generated is significantly reduced and the eluent generation cartridge (EGC) can last 18 months under standard continuous operation, which translates into reduced overall cost of ownership. Unfortunately, using Capillary IC with the standard 400 nL injection valve requires most water samples to be diluted, including wastewater.[12] This limits the benefits of capillary IC.

This application note describes the determination of inorganic anions in water samples using the Thermo Scientific™ Dionex™ ICS-4000 Integrated Capillary HPIC™ System in combination with the Dionex IonPac AS18-4µm Capillary column and a small, internal loop injection valve (100 nL). Using this valve, inorganic anions in undiluted municipal drinking water and wastewater samples can be determined in 13 min. The linear range, method detection limits, and recovery from fortified sample matrices are described.

Experimental

Equipment and consumables

- Thermo Scientific™ Dionex™ ICS-4000 Integrated Capillary HPIC™ System with RFIC-EG and Conductivity Detection (P/N 075130)*
- Thermo Scientific™ Dionex™ AS-AP Autosampler with 250 µL syringe
- Thermo Scientific™ Dionex™ 100 nL injection valve
- Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) software, version 7.2 SR4

*Equivalent results can be achieved using Thermo Scientific Dionex ICS-5000+ HPIC system equipped for capillary IC.

Table 1. Consumables list.

Product name	Description	P/N
Thermo Scientific™ Dionex™ EGC™ KOH Eluent Generator Cartridge	Potassium Hydroxide Eluent Generator Cartridge, Capillary	072076
Thermo Scientific™ Dionex™ CR-ATC™ Continuously Regenerated Anion Trap Column	Dionex CR-ATC (Capillary) Continuously Regenerated Anion Trap Column	072078
HP EG Degasser	Degasser used with eluent generation: HP degasser and tubing (capillary)	088231
Thermo Scientific™ Dionex™ ACES™ 300 Suppressor	Anion capillary electrolytic suppressor	072052
Thermo Scientific™ Dionex™ IonPac™ AG18-4µm Guard Column	Anion guard column, 0.4 × 35 mm	076033
Thermo Scientific™ Dionex™ Ion-Pac™ AS18-4µm Analytical Column	Anion analytical column, 0.4 × 150 mm	082314
Thermo Scientific™ Nalgene™ Syringe Filter	Syringe filters, PES membrane, 0.2 µm. This type is compatible with IC analysis.	725-2520*
Dionex AS-AP Autosampler Vials	Package of 100, 10 mL, polystyrene vials, caps, blue septa	074228
Thermo Scientific™ Dionex™ OnGuard™ II RP Cartridges	Polymeric reversed-phase cartridge 1 cc, 12 pack 1 cc, 48 pack 2.5 cc, 48 pack	 082760 057083 057084

* Fisher Scientific P/N 09-740-113

Reagent and standards

- Degassed deionized (DI) water, 18 MΩ·cm resistance or better
- Sodium and potassium salts, A.C.S. reagent grade or better, for preparing anions standards

Conditions	
Columns:	Dionex IonPac AS18-4µm Analytical Column, 0.4 × 150 mm Dionex IonPac AG18-4µm Guard Column, 0.4 × 35 mm
Eluent:	26 mM KOH
Eluent Source:	Dionex EGC KOH cartridge with CR-ATC
Flow Rate:	0.0120 mL/min
Injection Volume:	100 nL (full loop)
Column Temperature:	35 °C
Detection:	Suppressed conductivity, Dionex ACES 300 (Capillary) Suppressor, recycle mode, 8 mA current
Detection/Suppressor Compartment:	30 °C
Cell Temperature:	35 °C
Background Conductance:	< 1 µs
System Backpressure:	~1,800 psi
Noise:	< 2 nS/min
Run Time:	13 min

Preparation of solutions and reagents

Stock anion standard solutions (1,000 mg/L)

Stock standard solutions can be prepared by dissolving the appropriate amounts of the required analytes in 100 mL of DI water according to Table 2. Several of the 1,000 mg/L standard solutions are available from Thermo Scientific and their part numbers are also listed in Table 2. Stock standards for most anions are stable for at least 6 months at 4 ˚C. The nitrite and phosphate standards are stable for one month when stored at 4 ˚C.

Working standard solutions

Diluted working standard solutions are prepared using the 1,000 mg/L stock standards. Eight levels of calibration standards were used in this study for fluoride, chloride, nitrite, bromide, sulfate, nitrate, and phosphate (Table 3). Two concentrated mixed standards – level 8 and mixed stock – were prepared and stored at 4 °C for up to a month. Levels 1 through 7 working standards, which contain less than 100 mg/L anions, were prepared fresh daily by diluting the mixed standard stock with DI water.

Table 4 shows the anion standard concentrations used to calculate the method detection limits (MDLs) and the concentration of the quality control standard (QCS) used to determine retention time stability and peak area precision.

Table 2. Masses of compounds used to prepare 100 mL of 1000 mg/L anion standards.

Analyte	P/N	Compound	Amount* (mg)
Fluoride	037158	Sodium fluoride (NaF)	221.0
Chloride	037159	Sodium chloride (NaCl)	164.9
Nitrite		Sodium nitrite ($NaNO_2$)	150.0
Bromide		Sodium bromide (NaBr)	128.8
Nitrate		Sodium nitrate ($NaNO_3$)	137.1
Sulfate	037160	Sodium sulfate (Na_2SO_4)	147.9
Phosphate		Potassium phosphate, monobasic (KH_2PO_4)	143.3

*Compound must be dry.

Table 3. Calibration standards (mg/L).

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Mixed Stock
Fluoride	0.10	0.20	1.0	2.5	10	25	50	100	100
Chloride	0.20	0.40	2.0	5.0	20	50	100	300	200
Nitrite	0.10	0.20	1.0	2.5	10	25	50	100	100
Bromide	0.10	0.20	1.0	2.5	10	25	50	100	100
Sulfate	0.20	0.40	2.0	5.0	20	50	100	200	200
Nitrate	0.10	0.20	1.0	2.5	10	25	50	100	100
Phosphate	0.10	0.20	1.0	2.5	10	25	50	100	100

Table 4. Concentration of MDLs and QCS standards (mg/L).

Analyte	MDL Calculation Standard (mg/L)	QCS Used to Test Peak Retention Time and Peak Area Precisions (mg/L)
Fluoride	0.010	1.0
Chloride	0.004	2.0
Nitrite	0.010	1.0
Bromide	0.010	1.0
Sulfate	0.200	2.0
Nitrate	0.010	1.0
Phosphate	0.100	1.0

Sample preparation

A drinking water sample was collected locally. The other environmental water samples, including raw water, treated water, and wastewater (effluent), were obtained from a local water district lab.

All samples were filtered through a 0.2 µm PES syringe filter, discarding the first 300 µL of the effluent. To prolong column lifetimes, we also recommend pretreatment of wastewater sample with a reversed-phase cartridge to remove hydrophobic organic material.

Diluted water samples were prepared by mixing one part water sample and four parts DI water.

Spiked samples were prepared by adding mix standards in five-fold diluted samples (Table 5).

Table 5. Concentration of mixed spiking standards (mg/L).

Analyte	Spike 1 (mg/L)	Spike 2 (mg/L)
Fluoride	2	4
Chloride	60	120
Nitrite	2	4
Bromide	2	4
Sulfate	40	80
Nitrate	10	20
Phosphate	2	4

Results and discussion

Separation

The Dionex IonPac AS18-4µm column is a hydroxide-selective anion-exchange column developed to determine inorganic anions in environmental waters. Figure 1 shows a separation of common anions within 13 min using the Dionex IonPac AS18-4µm capillary column with a 100 nL injection valve. As this figure shows, all seven common inorganic anions were well resolved.

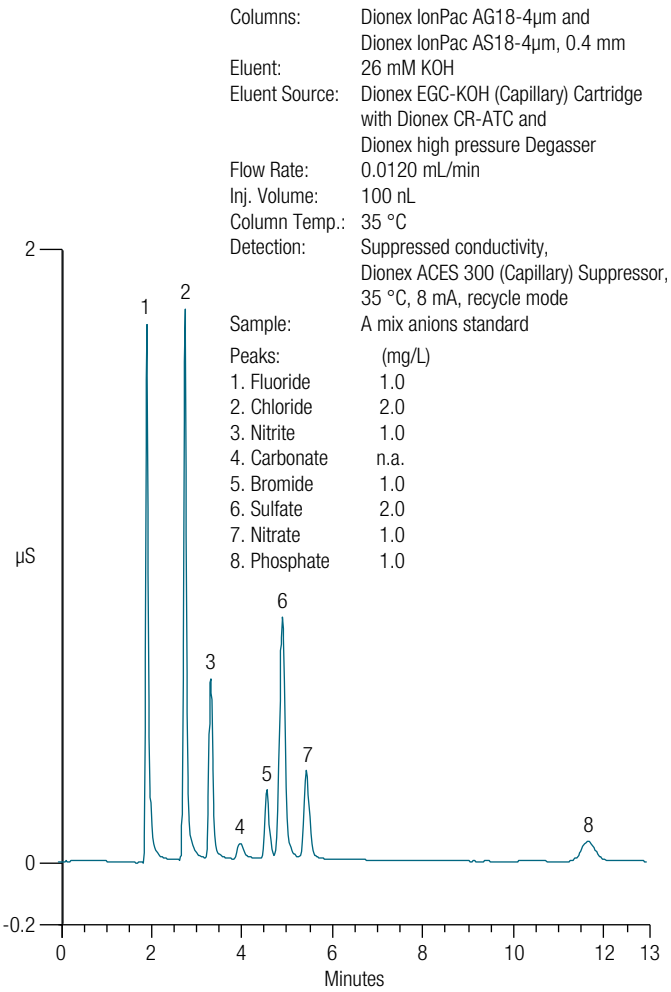

Figure 1. Separation of common anions using the Dionex IonPac AS18-4µm column and 100 nL injection valve.

Linearity, method detection limits (MDL), and precision

The quality control section of U.S. EPA Method 300.0 (Section 9.0) requires a demonstration of linearity, method detection limits (MDLs), and acceptable instrument performance by the analysis of a QCS prior to performing analyses using the method. Here, an eight-point calibration was used for fluoride, chloride, nitrite, bromide, sulfate, nitrate, and phosphate with three injections made for each concentration (Table 3). MDLs were determined by performing seven replicate injections of standards at a concentration of three to five times the estimated instrument detection limits (Table 4). Retention time and peak area precisions were determined from 30 replicate injections of a QCS prepared in DI water (Table 4). Table 6 shows the linear concentration ranges, the coefficients of determination (r^2), calculated MDLs, and retention time and peak area precisions. The MDLs, which ranged from 1.4 to 34.2 µg/L, are comparable to the method using a a 4 mm Dionex IonPac AS18 column with a 25 µL injection. The retention time stability is excellent (<0.05%), which is consistent with results typically produced using an electrolytically generated, high-purity potassium hydroxide eluent. However, the peak area precisions are 2–3% vs. <1% using a 25 µL injection. This may be a result of using a small injection volume (100 nL).

Table 6. Linearity, method detection limits (MDLs), and retention time and peak area precisions obtained using a Dionex IonPac AS18-4µm column with a 100 nL injection.

Analyte	Range (mg/L)	Coefficient of Determination (r^2)	Calculated * MDL (µg/L)	Retention Time Precision (n=30) (RSD)	Peak Area Precision (n=30) (RSD)
Fluoride	0.1–100 0.1–10	0.9970 0.9999	1.7	0.04	2.1
Chloride	0.2–300	1.000	1.4	<0.01	2.3
Nitrite	0.1–100 0.1–25	0.9986 1.000	3.7	0.05	2.3
Bromide	0.1–100	1.000	2.8	0.02	2.4
Sulfate	0.2–200	1.000	20.7	0.03	2.8
Nitrate	0.1–100	0.9997	4.9	0.02	2.3
Phosphate	0.1–100	0.9999	34.2	0.05	3.2

*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)

S=standard deviation of the replicate analyses

Figure 2 shows the calibration plots illustrating linearity. Chloride, bromide, sulfate, nitrate, and phosphate exhibited a large linear calibration range, but fluoride and nitrite did not exhibit a linear response at high concentrations. Because fluoride and nitrite concentrations in water samples are low, smaller concentration ranges of 0.1–10 mg/L for fluoride and 0.1–25 mg/L for nitrite were used for their respective determinations.

Using a Dionex IonPac AS18-4µm capillary column with a 100 nL injection valve, we determined inorganic anions in drinking water, treated water, raw waters, and wastewaters. Figures 3–8 show the chromatograms of inorganic anions in water samples, diluted water samples, and fortified diluted water samples. Table 7 lists the inorganic anions found and their concentrations in six water samples. The amounts were determined in both five-fold diluted and undiluted waters. As shown in Table 7, the amounts found were similar with and without sample dilution for all samples, even for the wastewater samples containing high concentrations of chloride (>250 mg/L) and sulfate (>150 mg/L). Therefore, using a 100 nL injection valve, water samples can be analyzed without a time-consuming dilution step that may also introduce error.

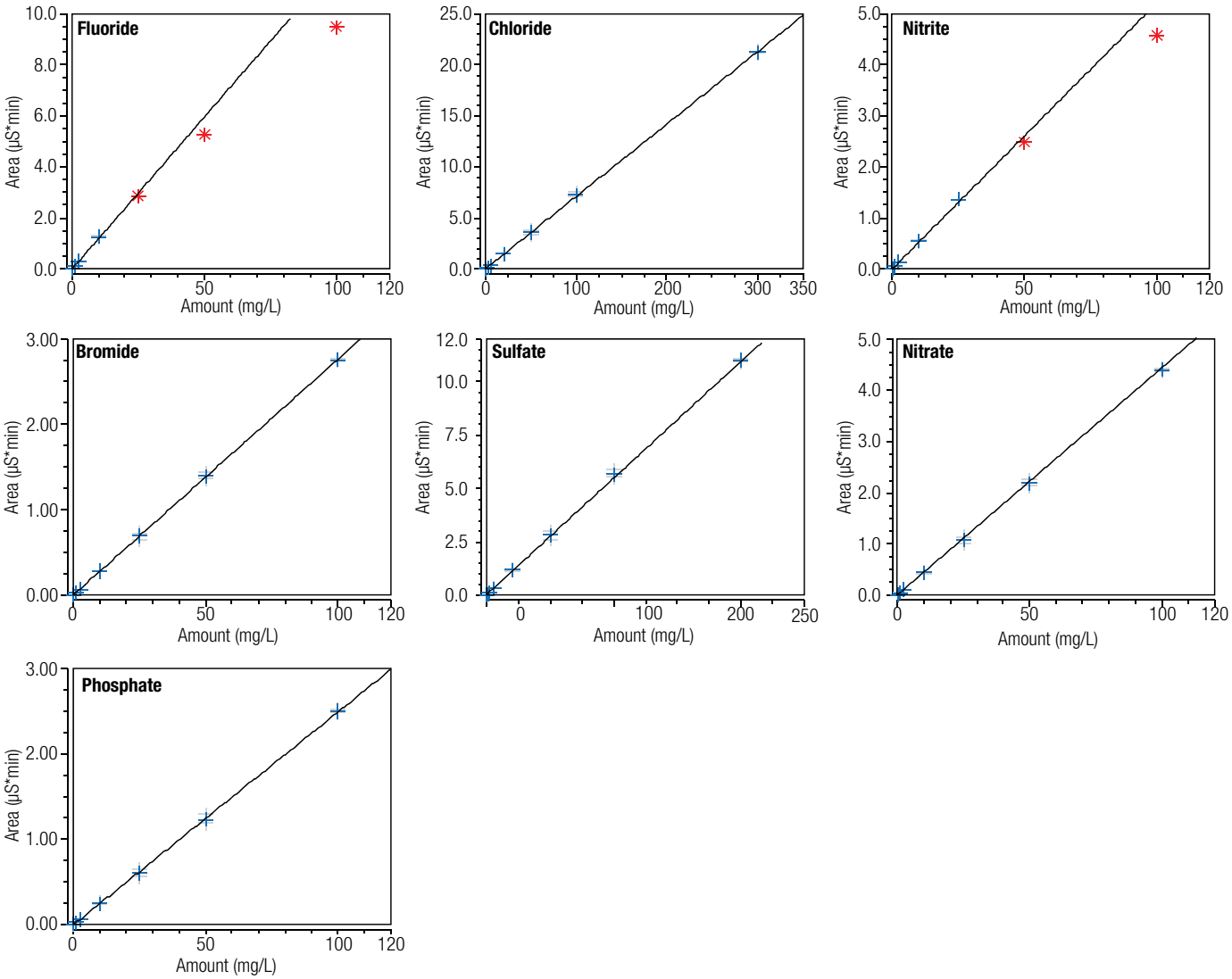

Figure 2. Calibration plots.

Columns:	Dionex IonPac AG18-4µm and Dionex IonPac AS18-4µm, 0.4 mm
Eluent:	26 mM KOH
Eluent Source:	Dionex EGC-KOH (Capillary) Cartridge with Dionex CR-ATC and Dionex high pressure Degasser
Flow Rate:	0.0120 mL/min
Inj. Volume:	100 nL
Column Temp.:	35 °C
Detection:	Suppressed conductivity, Dionex ACES 300 (Capillary) Suppressor, 35 °C, 8 mA, recycle mode
Sample Prep.:	Filtered, 0.2 µm
Samples:	A. Sample: Drinking water, undiluted B. Spiked 5-fold diluted sample C. 5-fold diluted sample

Peaks:	A	B	C	(mg/L)
1. Fluoride	0.8	2.1	0.1	
2. Chloride	10.0	63.5	1.9	
3. Nitrite	0.0	1.8	0.0	
4. Carbonate	-	-	-	
5. Bromide	0.0	1.9	0.0	
6. Sulfate	19.4	45.1	4.2	
7. Nitrate	1.2	9.5	0.7	
8. Phosphate	0.2	2.0	0.0	

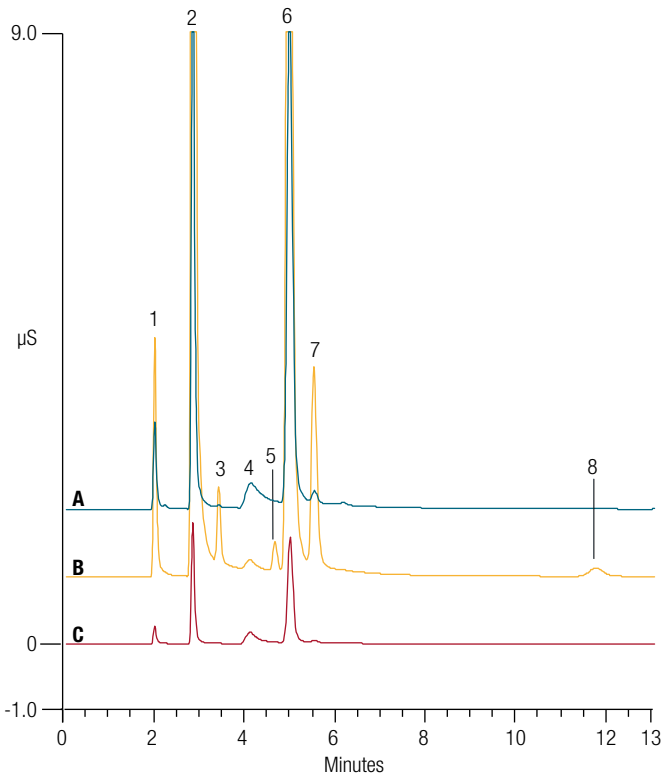

Figure 3. Determination of inorganic anions in drinking water.

Columns:	Dionex IonPac AG18-4µm and Dionex IonPac AS18-4µm, 0.4 mm
Eluent:	26 mM KOH
Eluent Source:	Dionex EGC-KOH (Capillary) Cartridge with Dionex CR-ATC and Dionex high pressure Degasser
Flow Rate:	0.0120 mL/min
Inj. Volume:	100 nL
Column Temp.:	35 °C
Detection:	Suppressed conductivity, Dionex ACES 300 (Capillary) Suppressor, 35 °C, 8 mA, recycle mode
Sample Prep.:	Filtered, 0.2 µm
Samples:	A. Sample: Treated water, undiluted B. Spiked 5-fold diluted sample C. 5-fold diluted sample

Peaks:	A	B	C	(mg/L)
1. Fluoride	0.6	2.1	0.1	
2. Chloride	88.5	79.7	18.4	
3. Nitrite	0.0	1.8	0.0	
4. Carbonate	-	-	-	
5. Bromide	0.8	1.8	0.4	
6. Sulfate	43.5	50.0	9.0	
7. Nitrate	4.1	9.9	1.1	
8. Phosphate	0.8	2.2	0.3	

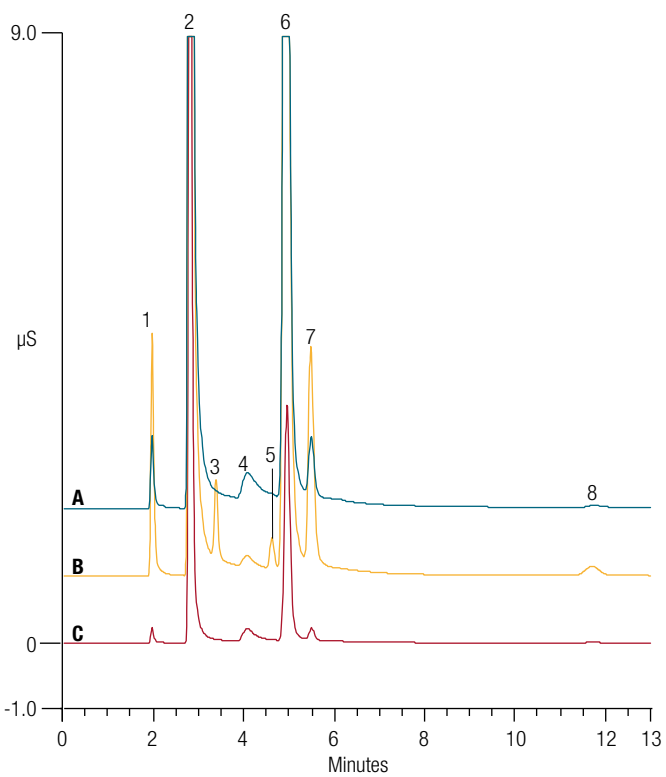

Figure 4. Determination of inorganic anions in treated water.

Columns:	Dionex IonPac AG18-4µm and Dionex IonPac AS18-4µm, 0.4 mm
Eluent:	26 mM KOH
Eluent Source:	Dionex EGC-KOH (Capillary) Cartridge with Dionex CR-ATC and Dionex high pressure Degasser
Flow Rate:	0.0120 mL/min
Inj. Volume:	100 nL
Column Temp.:	35 °C
Detection:	Suppressed conductivity, Dionex ACES 300 (Capillary) Suppressor, 35 °C, 8 mA, recycle mode
Sample Prep.:	Filtered, 0.2 µm
Samples:	A. Sample: Raw water 1, undiluted B. Spiked 5-fold diluted sample C. 5-fold diluted sample

Peaks:	A	B	C	(mg/L)
1. Fluoride	0.0	2.1	0.0	
2. Chloride	89.2	78.8	17.7	
3. Nitrite	0.0	1.8	0.0	
4. Carbonate	-	-	-	
5. Bromide	1.1	1.9	0.4	
6. Sulfate	33.1	47.5	6.5	
7. Nitrate	3.9	9.8	1.1	
8. Phosphate	0.4	2.0	0.2	

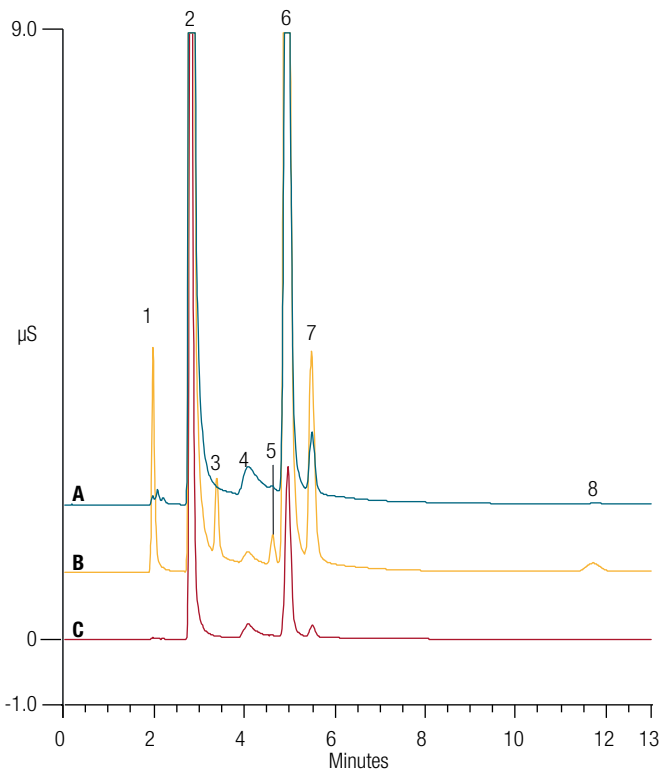

Figure 5. Determination of inorganic anions in raw water 1.

Columns:	Dionex IonPac AG18-4µm and Dionex IonPac AS18-4µm, 0.4 mm
Eluent:	26 mM KOH
Eluent Source:	Dionex EGC-KOH (Capillary) Cartridge with Dionex CR-ATC and Dionex high pressure Degasser
Flow Rate:	0.0120 mL/min
Inj. Volume:	100 nL
Column Temp.:	35 °C
Detection:	Suppressed conductivity, Dionex ACES 300 (Capillary) Suppressor, 35 °C, 8 mA, recycle mode
Sample Prep.:	Filtered, 0.2 µm
Samples:	A. Sample: Raw water 2, undiluted B. Spiked 5-fold diluted sample C. 5-fold diluted sample

Peaks:	A	B	C	(mg/L)
1. Fluoride	0.0	2.0	0.0	
2. Chloride	86.2	78.5	17.7	
3. Nitrite	0.0	1.8	0.0	
4. Carbonate	-	-	-	
5. Bromide	1.0	1.9	0.4	
6. Sulfate	31.5	47.2	6.4	
7. Nitrate	3.7	9.8	1.0	
8. Phosphate	0.4	2.0	0.2	

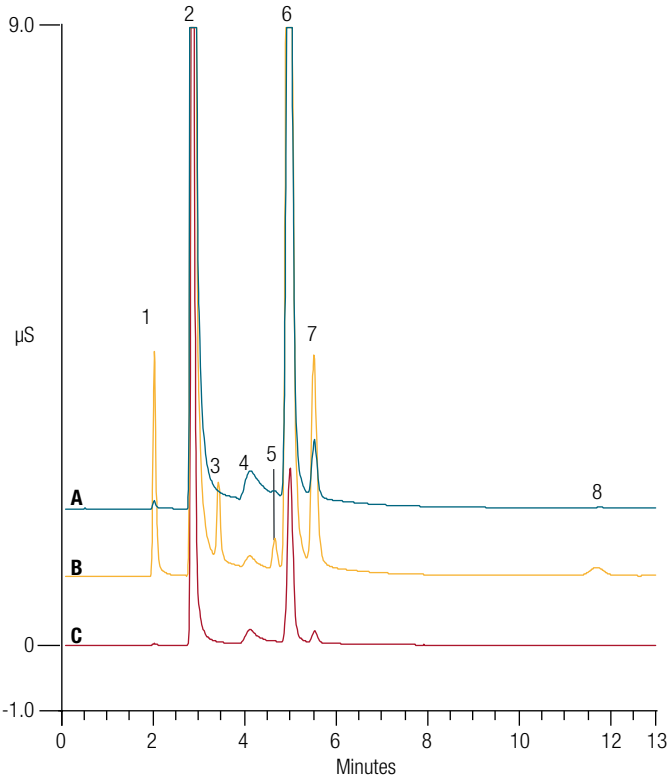

Figure 6. Determination of inorganic anions in raw water 2.

Columns:	Dionex IonPac AG18-4µm and Dionex IonPac AS18-4µm, 0.4 mm
Eluent:	26 mM KOH
Eluent Source:	Dionex EGC-KOH (Capillary) Cartridge with Dionex CR-ATC and Dionex high pressure Degasser
Flow Rate:	0.0120 mL/min
Inj. Volume:	100 nL
Column Temp.:	35 °C
Detection:	Suppressed conductivity, Dionex ACES 300 (Capillary) Suppressor, 35 °C, 8 mA, recycle mode
Sample Prep.:	Filtered, 0.2 µm and passed through a Dionex OnGuard II RP cartridge
Samples:	A. Sample: Wastewater 1, undiluted B. Spiked 5-fold diluted sample C. 5-fold diluted sample

Peaks:	A	B	C	(mg/L)
1. Fluoride	0.5	2.2	0.1	
2. Chloride	222.8	105.6	44.5	
3. Nitrite	5.8	3.0	1.1	
4. Carbonate	-	-	-	
5. Bromide	0.7	1.8	0.4	
6. Sulfate	154.1	70.9	30.8	
7. Nitrate	61.3	22.1	12.1	
8. Phosphate	2.1	2.6	0.7	

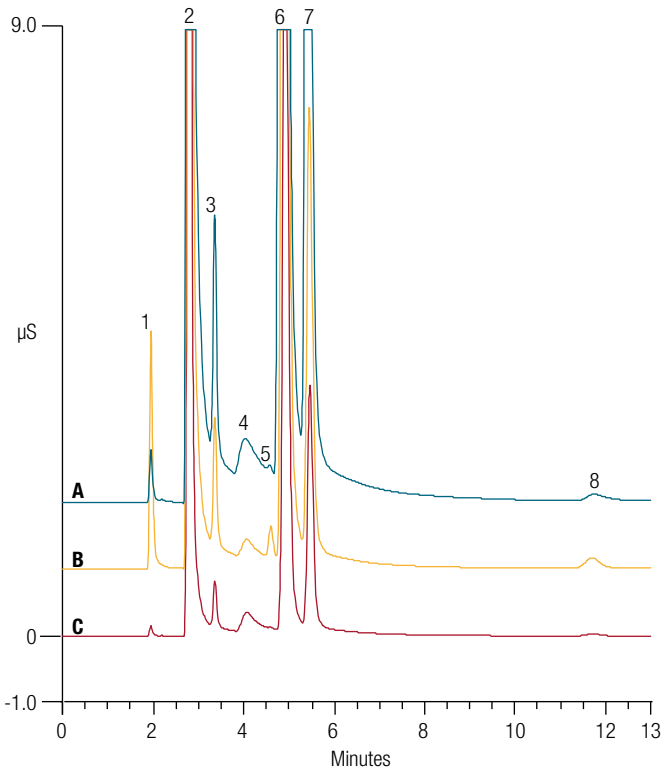

Figure 7. Determination of inorganic anions in wastewater 1.

Columns:	Dionex IonPac AG18-4µm and Dionex IonPac AS18-4µm, 0.4 mm
Eluent:	26 mM KOH
Eluent Source:	Dionex EGC-KOH (Capillary) Cartridge with Dionex CR-ATC and Dionex high pressure Degasser
Flow Rate:	0.0120 mL/min
Inj. Volume:	100 nL
Column Temp.:	35 °C
Detection:	Suppressed conductivity, Dionex ACES 300 (Capillary) Suppressor, 35 °C, 8 mA, recycle mode
Sample Prep.:	Filtered, 0.2 µm and passed through a Dionex OnGuard II RP cartridge
Samples:	A. Sample: Wastewater 2, undiluted B. Spiked 5-fold diluted sample C. 5-fold diluted sample

Peaks:	A	B	C	(mg/L)
1. Fluoride	0.3	2.1	0.1	
2. Chloride	259.6	112.5	51.0	
3. Nitrite	0	1.8	0.0	
4. Carbonate	-	-	-	
5. Bromide	0.7	1.9	0.5	
6. Sulfate	91.0	58.5	18.0	
7. Nitrate	82.5	26.9	16.3	
8. Phosphate	5.1	3.1	1.2	

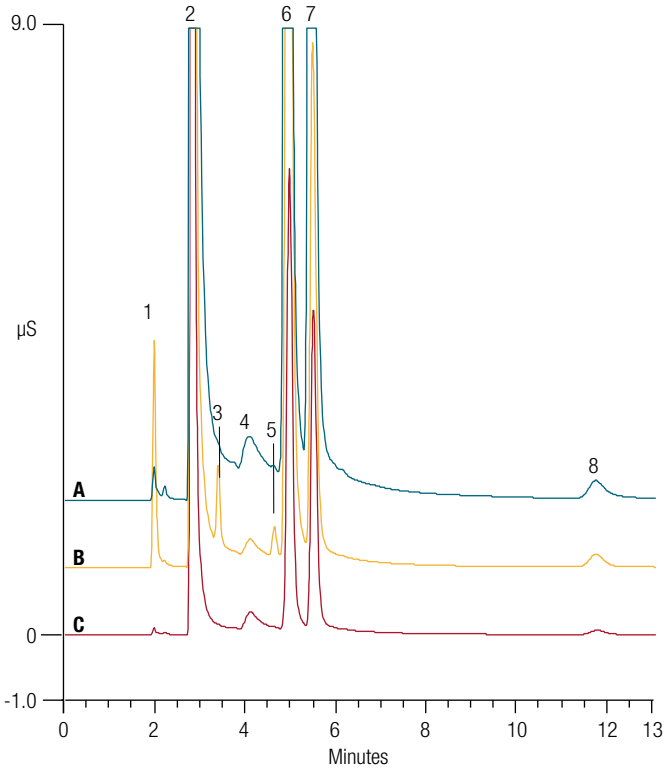

Figure 8. Determination of inorganic anions in wastewater 2.

Table 7. Comparison of the determinations of inorganic anions in waters between diluted and undiluted waters samples (mg/L).

	Drinking Water		Treated Water		Raw Water 1	
	Method A*	Method B*	Method A	Method B	Method A	Method B
Fluoride	0.8	0.6	0.6	0.5	0.0	0.0
Chloride	10.0	9.6	88.5	91.9	89.2	88.4
Nitrite	0.0	0.0	0.0	0.0	0.0	0.0
Bromide	0.0	0.0	0.8	1.9	1.1	2.3
Sulfate	19.4	20.8	43.5	45.1	33.0	32.4
Nitrate	1.2	3.6	4.1	5.6	3.9	5.4
Phosphate	0.2	0.0	0.8	1.4	0.4	1.1
	Raw Water 2		Wastewater 1		Wastewater 2	
Fluoride	0.0	0.0	0.5	0.3	0.3	0.1
Chloride	86.2	88.5	223	222	260	255
Nitrite	0.0	0.0	5.8	5.4	0.0	0.0
Bromide	1.0	1.9	0.7	2.2	0.7	2.6
Sulfate	31.5	31.9	154.1	153.9	91.0	90.2
Nitrate	3.7	5.0	61.3	60.7	82.5	81.6
Phosphate	0.4	0.8	2.1	3.5	5.1	6.2

*Method A-Amount determined (mg/L) using undiluted sample. Method B- Amount (mg/L) determined using five-fold diluted sample.

The performance of the IC method using the Dionex IonPac AS18-4µm capillary column with 100 nL injection valve was also evaluated through a single-operator precision and bias study using spiked water samples. Table 8 shows typical recovery results for common inorganic anions spiked into drinking water, raw (unfinished) drinking water, treated raw water, and wastewaters. As shown in the Table 8, good recovery (86–106%) was obtained for most of the common inorganic anions including fluoride, chloride, nitrite, sulfate, nitrate, and phosphate. Bromide recovery was low (66.8–83%) for raw water and wastewater. This may be a result of the low spiking concentration (2 mg/L) and the high concentration of carbonate.

Table 8. Recoveries of inorganic anions in drinking water, raw water, treated water, and wastewaters (%).

	Drinking Water		Treated Water		Raw Water 1	
	Spiked 1	Spiked 2	Spiked 1	Spiked 2	Spiked 1	Spiked 2
Fluoride	104	105	104	102	103	106
Chloride	103	103	102	102	102	104
Nitrite	91.0	94.2	88.9	93.3	90.5	94.8
Bromide	91.8	90.8	71.5	80.6	70.0	80.9
Sulfate	102	103	102	104	103	104
Nitrate	87.3	99.9	87.2	89.5	87.3	97.5
Phosphate	102	98.5	93.1	93.1	89.9	93.3
	Raw Water 2		Wastewater 1		Wastewater 2	
Fluoride	98.2	103	105	102	104	108
Chloride	101	105	102	102	102	106
Nitrite	90.4	95.6	96.7	96.4	89.6	95.1
Bromide	73.6	83.4	66.8	76.0	71.0	75.6
Sulfate	102	104	100	101	101	104
Nitrate	87.9	102	100	102	106	102
Phosphate	90.8	95.4	92.2	92.5	90.0	96.5

Conclusion

This application note describes the determination of inorganic anions in water samples using a capillary IC system in combination with a Dionex IonPac AS18-4µm capillary column and a small injection valve (100 nL) in 13 min. The study demonstrates that the method has a large linear range (e.g., 0.2–300 mg/L for chloride), is sensitive, precise, and accurate. The capillary IC system has low eluent consumption, so the system can always be kept on and ready for analysis. By using the smaller injection valve, inorganic anions in municipal drinking water, raw water, and wastewater samples can be determined easily without dilution.

References

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