

Anion Determinations in Municipal Drinking Water Samples Using EPA Method 300.1 (A) on an Integrated IC System

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Key Words

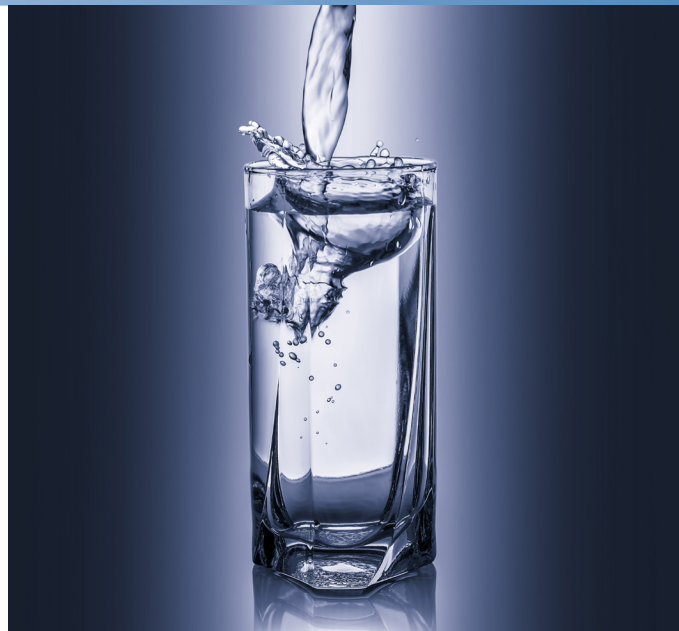
Drinking Water, Environmental, Dionex IonPac AS22, Anions, Dionex ICS-1100, Dionex ICS-1600

Goal

Demonstrate EPA Method 300.1 (A) using a Dionex IonPac AS22 column and an integrated IC system

Introduction

The determination of common inorganic anions in drinking water is one of the most important ion chromatography (IC) applications worldwide. In the U.S., the Environmental Protection Agency (EPA) defines the Maximum Contamination Levels (MCL) of toxic contaminants, such as fluoride, nitrite, and nitrate in the Primary Drinking Water regulations.¹ The MCLs were established to minimize exposure to toxic contaminants. For example, high levels of fluoride are associated with skeletal and dental fluorosis, whereas methemoglobinemia results in increased infant mortalities caused by high nitrite and nitrate concentrations.¹ Chloride and sulfate concentrations are regulated under the the U.S. National Secondary Drinking Water Guidelines for aesthetic characteristics. Both the Primary and Secondary Drinking Water Regulations are adopted by most U.S. states, as well as many industrialized countries. Since the mid-1980s, IC has been approved for inorganic anion compliance monitoring under the U.S. EPA Method 300.0.²



This method updates the previous application in 1998, Application Note 133 (Determination of Inorganic Anions in Drinking Water by Ion Chromatography, with the latest column and instrumentation) which was approved for EPA Method 300.1 (A).³

Here we discuss using a Thermo Scientific™ Dionex™ IonPac™ AS22 anion-exchange column to separate and quantify inorganic anions with manually-prepared carbonate/bicarbonate eluents on a Thermo Scientific Dionex ICS-1100 or a Thermo Scientific Dionex ICS-1600 IC system. This method provides an economical way to meet EPA Method 300.1 (A) regulatory testing requirements.

Equipment

- Dionex ICS-1100 Integrated IC system*
- Thermo Scientific Dionex AS-DV Autosampler
- Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System (CDS), version 7.2

*or any other Dionex IC system

Reagents and Standards

- 18 MΩ-cm resistivity degassed deionized water
- Thermo Scientific™ Dionex™ Combined Seven Anion Standard II, NIST traceable (P/N 057590)
- Thermo Scientific™ Dionex™ AS22 Carbonate/Bicarbonate Concentrate (P/N 063965)

Samples

Municipal drinking and surface waters.

Conditions

Columns:	Dionex IonPac AG22, 4 × 50 mm Dionex IonPac AS22, 4 × 250 mm
Eluent:	4.5 mM Sodium carbonate, 1.4 mM sodium bicarbonate
Flow Rate:	1.2 mL/min
Column Temperature:	30 °C
Injection Volume:	5 µL
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ AERS™ 500 Anion Electrolytically Regenerated Suppressor, recycle mode; 31 mA
Background Conductance:	~22 µS
Noise:	<10 nS
System Backpressure:	~1800 psi

Standard and Sample Preparation

It is important to use 18 MΩ-cm resistivity, deionized water for standard and eluent. Using deionized water with less than 18 MΩ-cm resistivity can reduce sensitivity, introduce contamination, and affect calibration, thereby resulting in inaccurate quantification.

Prepare standards by diluting the NIST-traceable Dionex Seven Combined II Standard with deionized water.

Sample Preparation

The surface water sample was filtered (0.2 µm) prior to injection. All other samples were injected without pretreatment.

Table 1. Consumables list.

Product Name	Type	Part Number
Dionex IonPac AG22	Carbonate eluent anion-exchange guard column, 4 × 50 mm	064139
Dionex IonPac AS22	Carbonate eluent anion-exchange analytical column, 4 × 250 mm	064141
Dionex AERS 500 Suppressor	Anion suppressor for 4 mm i.d. columns	082540
Dionex AS-DV Autosampler Vials, 5 mL	PolyVials and 20 µm filter caps, 250 each	038141
	PolyVials and plain caps, 250 each	038008
4 L Eluent Bottle	4 L Eluent bottle recommended for convenience to replace 2 L eluent bottle	066019
Dionex AS22 Carbonate/Bicarbonate Eluent Concentrate	Eluent concentrate (100x) optimized for Dionex IonPac AS22 column conditions	063965

Eluent Preparation

Using manually prepared eluent is a traditional approach for IC and is what is used in this application update. However, manual eluent preparation can create variability. An alternative to manually prepared eluents is eluent generation which electrolytically generates precise and accurate eluent inline for isocratic or gradient separations. Eluent generation is an approved method for EPA Method 300.1. The use of eluent generation is applied in Application Note 154: Determination of Inorganic Anions in Environmental Waters Using a Hydroxide-Selective Column and Application Note 175: Determination of Sulfate and Chloride in Ethanol by Ion Chromatography.^{4,5} Both applications of eluent generation are approved for EPA Method 300.1 (A).

4.5 mM Sodium Carbonate, 1.4 mM Sodium Bicarbonate Eluent Preparation

To prepare 4 L of eluent, pipet 20 mL of the Dionex AS22 Carbonate/Bicarbonate Eluent Concentrate into a 2 L volumetric flask containing ~1000 mL of previously degassed 18 MΩ resistivity deionized water. Gently swirl to mix. Dilute to the 2 L mark with degassed deionized water, stopper, and invert the flask at least three times to mix. Transfer the eluent to a 4 L eluent bottle and repeat the process for the second 2 L of eluent. Connect the bottle to the instrument eluent line, and prime the pump to draw the new eluent through the eluent line and pump. For the best chromatography, equilibrate for 30 min prior to running samples and check standards.

Instrument Setup

To set up this application, hydrate the 4 mm Dionex AERS 500 suppressor according to the product manual and Quick Start instructions.^{6,7} Allow 20 min for the suppressor membranes to fully hydrate. Install and condition the columns with eluent for 30 min while temporarily directing the eluent leaving the column to waste. Install the fully hydrated suppressor and complete the installation of the columns in the Dionex ICS-1100 system or Dionex ICS-1600 system, according to Figure 1. For more detailed instructions, refer to the cited Quick Start Instructions, product manuals, and the instrument installation and operator's manuals.⁶⁻¹³

Results and Discussion

Table 2 shows the comparison of the Dionex IonPac AS22 column to the Dionex IonPac AS14 column (AN 133). The Dionex IonPac AS22 column has nearly 3x higher capacity than the Dionex IonPac AS14 with shorter run times than the previous columns while still meeting the performance limits specified in EPA Methods 300.0 and 300.1. This column replaces the Dionex IonPac AS4A-SC, AS12A, AS14 and AS14A columns. The listed columns improved upon the separation of acetate and fluoride which was problematic on the Dionex IonPac AS4A column.

Table 2. Column comparison.

	Dionex IonPac AS14	Dionex IonPac AS22
Bead Type	Macroporous	Supermacroporous
Bead size	9 μm	6 μm
Porosity	1000 \AA	2000 \AA
Functional Group	Alkyl quaternary ammonium ion	Alkanol quaternary ammonium ion
Cross-linking	55% DVB	55% DVB
Capacity	65 μeq	210 μeq

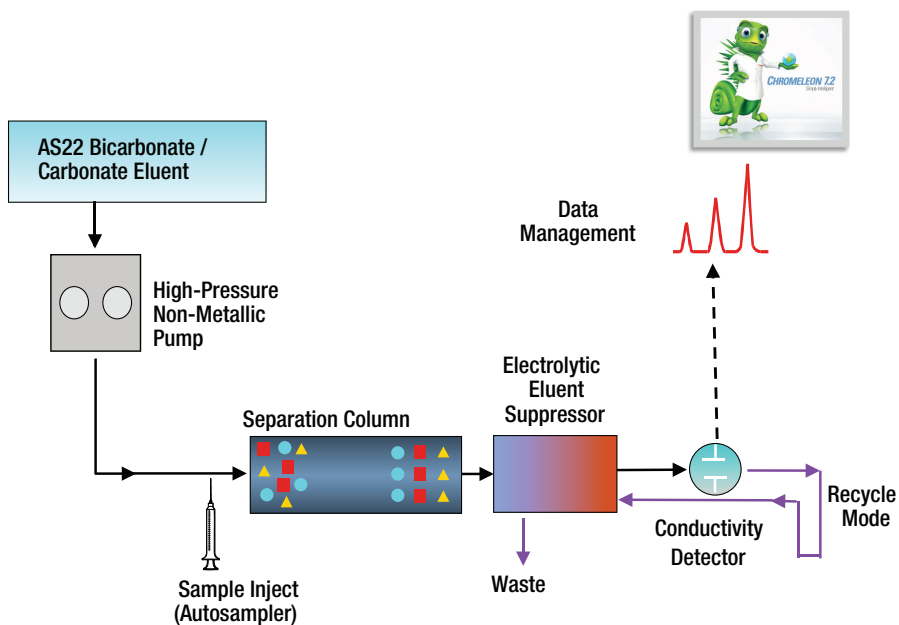


Figure 1. Flow diagram for the Dionex ICS-1100 or Dionex ICS-1600 Integrated IC systems.

Figures 2 and 3 show the same concentration standard separated with the optimum conditions for each column. In Figure 2 from AN 133, a 50 μL sample was separated at 1.2 mL/min (Figure 2). All eight anions were eluted within 14 min. In comparison, all eight anions eluted from the Dionex IonPac AS22 column within 12 min, saving two min of run time (Figure 3). Additionally, the sample injection volume in this method was reduced to 5 μL at the expense of sensitivity to allow injection of undiluted samples. The elution order is similar for both columns indicating similar selectivity which allows easy implementation of this method. The exception is the carbonate dip which elutes just prior to chloride on the Dionex IonPac AS14 column but between nitrate and phosphate on the Dionex IonPac AS22 column which provides more accurate quantification of chloride.

In both methods, the anions were detected by suppressed conductivity with a Dionex electrolytic suppressor as they elute from the column. In this method, the Dionex AERS 500 suppressor provides improved peak efficiencies, thermal stability, and faster recoveries after stopping and restarting operations. This suppressor eliminates the need for preparation and use of chemical regenerants.

Linearity and Method Detection Limits

To evaluate the method as run on the AS22 column, the Linear Calibration Range (LCR), method detection limits (MDL), 4 day reproducibility, and recoveries of standard added to the samples and laboratory blank (LB) were determined. MDL standards were prepared by fortifying the LB with a concentrated standard to achieve a signal to noise of 3 to 5 as calculated according to EPA method 300.1 (A) by multiplying the standard deviation ($n = 7$) and 3.14 t-test factor. Table 3 shows the results for the MDL, LCR, and Coefficient of Determination for seven anions. The LCR was optimized for direct injections of undiluted drinking water samples at the expense of sensitivity; as a result the MDLs are 10x higher than reported in AN 133 due to the 10x smaller sample injection volume. Nitrite, nitrate, and phosphate results were also expressed as mg/L of nitrite as nitrogen ($\text{NO}_2\text{-N}$), mg/L of nitrate as nitrogen ($\text{NO}_3\text{-N}$), mg/L of phosphate as phosphorous ($\text{PO}_4\text{-P}$), respectively, for reporting requirements. The results show a linear response between peak area and concentration, $r^2 > 0.999$.

Column: Dionex IonPac AG14, Dionex IonPac AS14
 Eluent: 3.5 mM Sodium carbonate/1 mM Sodium bicarbonate
 Flow Rate: 1.2 mL/min
 Inj. Volume: 50 μL
 Detection: Suppressed conductivity, Thermo Scientific™ Dionex™ ASRS™ Anion Self Regenerating Suppressor, AutoSuppression, recycle mode

Peaks: 1. Fluoride 5 5. Bromide 25 mg/L
 2. Acetate 20 6. Nitrate 25
 3. Chloride 10 7. Phosphate 40
 4. Nitrite 15 8. Sulfate 30

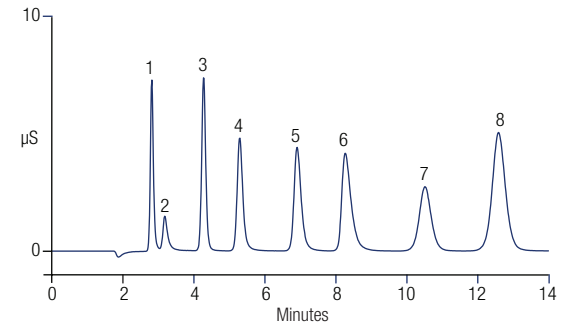


Figure 2. Separation of eight anions using the Dionex IonPac AS14 column.

Column: Dionex IonPac AG22, 4 x 50 mm, Dionex IonPac AS22, 4 x 250 mm
 Eluent: 4.5 mM Sodium carbonate/1.4 mM sodium bicarbonate
 Flow Rate: 1.2 mL/min
 Injection: 5 μL
 Detection: Suppressed conductivity, AutoSuppression, Dionex AERS 500 suppressor, 31 mA, recycle mode

Peaks: 1. Fluoride 5 5. Bromide 25 mg/L
 2. Acetate 20 6. Nitrate 25
 3. Chloride 10 7. Phosphate 40
 4. Nitrite 15 8. Sulfate 30

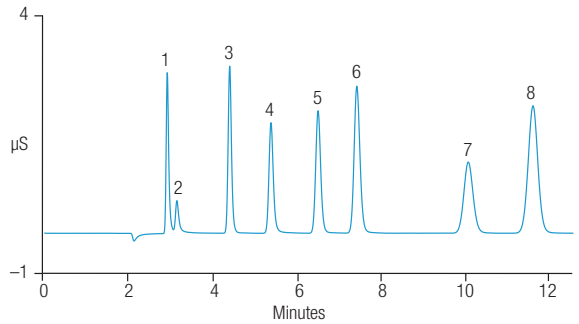


Figure 3. Separation of eight anions using the Dionex IonPac AS22 column.

Table 3. MDL, Linear calibration range, and coefficient of determination.

	MDL [^] (mg/L)	LCR ^{^^} (mg/L)	Coefficient of Determination (r ²)
Fluoride	0.042	0.2 to 20	0.9996
Chloride	0.082	1 to 100	0.9993
Nitrite (NO ₂ -N) ⁺	0.222 (0.068)	1 to 100 (0.306 to 30.6)	0.9990
Bromide	0.292	1 to 100	0.9997
Nitrate (NO ₃ -N) ⁺⁺	0.266 (0.060)	1 to 100 (0.226 to 22.6)	0.9996
<i>o</i> -Phosphate (PO ₄ -P) ⁺⁺⁺	0.763 (0.249)	2 to 200 (0.653 to 65.3)	0.9994
Sulfate	0.282	1 to 100	0.9996

⁺ NO₂-N: nitrite as nitrogen

⁺⁺ NO₃-N: nitrate as nitrogen

⁺⁺⁺ PO₄-P: phosphate as phosphorous

[^] n = 7

^{^^} LCR: Linear Calibration Range

MDL = using t-test, 3.14 x Standard Deviation for n = 7

Reproducibility

To evaluate reproducibility of this method, a 5-fold dilution of Dionex Combined Seven Anion II Standard (4 mg/L fluoride; 20 mg/L chloride, nitrite, nitrate, and sulfate; 40 mg/L phosphate) was injected continuously for four days while monitoring the retention times and peak areas for all seven analytes. The seven anions had excellent retention time reproducibility, with RSDs from 0.1 for fluoride to 0.54 for sulfate. The peak area reproducibilities were good with RSDs < 0.7 for all peaks except fluoride and nitrite, which had RSDs of 0.83 and 1.2 respectively.

Sample Analysis

Four California municipal drinking water samples (Berkeley, Sunnyvale, southern portion of San Jose, and Palm Desert), and two surface water samples (Whitewater River in Palm Desert, CA, and Lake Tahoe near Homewood, CA) were analyzed (Figures 4–5).

The Berkeley, California municipal drinking water sample had the lowest chloride and sulfate concentrations, whereas the south San Jose, California sample had the highest anion concentrations of the drinking water samples.

The two surface water samples from the Whitewater River in Palm Desert, CA and the Lake Tahoe sample from Homewood, CA were very different. The Lake Tahoe sample is from a very large resort lake that is fed by snow run-off. In contrast the Whitewater River from the Palm Desert area periodically forms from agricultural run-off and sparse rain, feeds the Salton Sea, and dries to a river bed during the dry season. As expected, the lake sample has relatively low ionic concentrations, as compared to the municipal drinking samples, while the river sample has considerably higher concentrations.

Column: Dionex IonPac AG22, 4 × 50 mm,
Dionex IonPac AS22, 4 × 250 mm
Eluent: 4.5 mM Sodium carbonate/1.4 mM Sodium bicarbonate
Flow Rate: 1.2 mL/min
Injection: 5 µL
Detection: Suppressed conductivity, Dionex AERS 500 suppressor, 31 mA, recycle mode
Samples: A: City of Berkeley, CA
B: City of Sunnyvale, CA
C: City of Palm Desert, CA
D: City of San Jose, CA

Peaks:		A	B	C	D
1. Fluoride		0.84	0.79	0.61	0.20 mg/L
2. Chloride		5.9	11.6	10.6	37.7
3. Nitrite		0.60	—	—	—
4. Nitrate		—	0.73	9.1	5.05
5. Carbonate		—	—	—	—
6. Sulfate		6.7	11.5	25.1	37.3

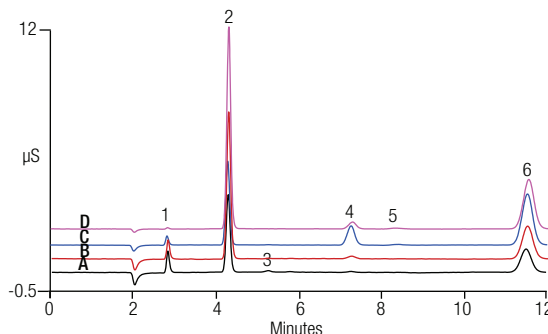


Figure 4. Municipal drinking water samples.

Column: Dionex IonPac AG22, 4 × 50 mm,
Dionex IonPac AS22, 4 × 250 mm
Eluent: 4.5 mM Sodium carbonate/1.4 mM Sodium bicarbonate
Flow Rate: 1.2 mL/min
Injection: 5 µL
Detection: Suppressed conductivity, Dionex AERS 500 suppressor, 31 mA, recycle mode
Sample Prep.: A: None, B: Filtered, 0.2 µm
Samples: A: Lake Tahoe, CA
B: Whitewater River, Palm Desert

Peaks:		A	B
1. Fluoride		0.09	0.23
2. Chloride		2.2	49.3
3. Nitrate		—	19.3
4. Phosphate		—	1.9
5. Sulfate		1.8	82.0

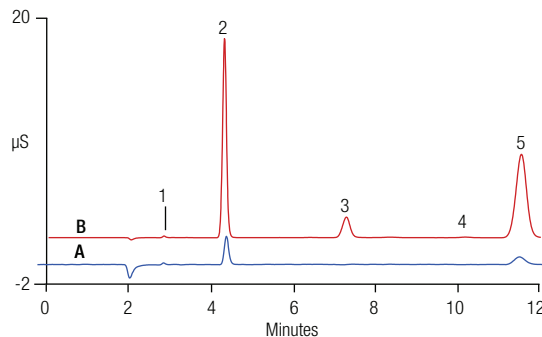


Figure 5. Surface water samples.

Sample Accuracy

To determine the method accuracy, the Laboratory Blank (LB) and five samples were fortified with known concentrations of each anion and analyzed for the total concentrations. The results of these recovery experiments are shown in Tables 4 and 5. The Laboratory Fortified Blank (LFB) shown in Table 4 has good recoveries of all seven anions, 96 to 106%.

The samples also had good recoveries, 90 to 101% indicating good method accuracy for samples and the LFB.

Conclusion

- This method using the high capacity Dionex IonPac AS22 column provides similar selectivity and faster separations and at standard flow rates than previously shown in Application AN 133.
- This application meets the EPA Method 300.1 (A) requirements for determinations of inorganic anions in municipal drinking water.

Table 4. Recoveries in the Laboratory Fortified Blank.

Analyte	Laboratory Fortified Blank (LFB)*	
	Added (mg/L)	Recovery (%)
Fluoride	2.0	102
Chloride	10	106
Nitrite (NO ₂ -N) ⁺	2.0 (0.62)	102
Bromide	2.0	98.2
Nitrate (NO ₃ -N) ⁺⁺	4.0 (0.88)	95.8
<i>o</i> -Phosphate (PO ₄ -P) ⁺⁺⁺	4.0 (1.3)	104
Sulfate	10	101

n = 7

* LFB: No analytes were measured in Laboratory Blank (LB)

⁺ NO₂-N: nitrite expressed as nitrogen;

⁺⁺ NO₃-N, NO₂-N: nitrite or nitrate as nitrogen

⁺⁺⁺ PO₄-P: phosphate as phosphorous

Table 5. Recovery results from fortified municipal drinking and surface water samples.

	Municipal Drinking Water Samples						Surface Water Samples			
	Berkeley		Palm Desert		South San Jose		Homewood Resort, Lake Tahoe		Whitewater River, Palm Springs	
	Added (mg/L)	Recovery (%)	Added (mg/L)	Recovery (%)	Added (mg/L)	Recovery (%)	Added (mg/L)	Recovery (%)	Added (mg/L)	Recovery (%)
Fluoride	1.0	93.6	1.0	91.6	1.0	95.0	1.0	94.6	1.0	94.2
Chloride	5.0	95.0	10	99.8	10	95.6	2.0	93.8	25	97.2
Nitrite (NO ₂ -N) ⁺	1.0 (0.3)	90.8	1.0 (3.1)	91.1	1.0 (3.1)	95.2	1.0	95.4	1.0	93.4
Bromide	1.0	93.0	1.0	96.3	1.0	92.8	1.0	95.2	1.0	93.2
Nitrate (NO ₃ -N) ⁺⁺	1.0 (0.2)	91.4	10 (2.3)	95.2	10 (2.3)	94.3	1.0	94.4	10 (2.3)	92.4
Phosphate (PO ₄ -P) ⁺⁺⁺	2.0 (0.6)	90.6	2.0 (0.6)	92.2	2.0 (0.6)	90.8	2.0 (0.6)	91.5	5.0 (3.3)	91.9
Sulfate	5.0	98.2	10	98.4	15	98.2	2.0	101.2	40	96.3

n = 7

⁺ NO₂-N: nitrite as nitrogen

⁺⁺ NO₃-N: nitrate as nitrogen

⁺⁺⁺ PO₄-P: phosphate as phosphorous

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