Determination of Bromate in Bottled Mineral Water Using the CRD 300 Carbonate Removal Device

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Application Note 208

Introduction

Drinking and bottled waters are commonly disinfected with ozone. Ozone is highly effective and, unlike many other disinfectants, does not remain in the water or change its taste. Unfortunately, when bromide is present in water, it is converted to bromate by the ozone treatment. Bromate is recognized as a potential human carcinogen, which has led to the regulation of its concentration in drinking and bottled water. Major regulatory bodies worldwide (e.g., U.S. EPA and the European Commission) have set a maximum allowable bromate concentration in drinking water of 10 µg/L.[1] In Europe, the limit was lowered to 3 µg/L for bottled natural mineral and spring waters disinfected by ozonation.[2]

Over the past two decades, we have led the effort in developing sensitive and robust ion chromatography (IC) methods for determining bromate and other oxyhalides (e.g., chlorite and chlorate). U.S. EPA Method 300.0 (B) and 300.1 (B) used the Thermo Scientific™ Dionex™ IonPac™ AS9-SC and IonPac AS9-HC columns, respectively, along with suppressed conductivity detection for bromate, chlorite, and chlorate determinations in drinking water. In 1997, we introduced the AS9-HC column to allow the direct injection of 250 µL of drinking water to easily meet the 10 µg/L regulatory requirement. This method was documented in Dionex (now part of Thermo Scientific) Application Note (AN) 81.[3] Since then, we have developed a number of products and techniques, and worked with regulatory agencies and international standards organizations to improve the sensitivity and ruggedness of bromate determinations as well as the types of samples that can be directly injected.

Our products were instrumental in the development of the postcolumn derivatization techniques in U.S. EPA Methods 317.0 and 326.0. These methods used the Dionex IonPac AS9-HC column and our suppression technology for conductivity detection of oxyhalides combined with postcolumn addition and absorbance detection for enhanced determination of bromate. EPA Methods 317.0 and 326.0 are documented in Dionex (now part of Thermo Scientific) AN 136 and AN 149.[4,5] To improve the sensitivity for bromate using direct injection, we developed the Dionex IonPac AS19 column. This column was designed for use with hydroxide eluents rather than the carbonate eluents used with the Dionex AS9-HC column.

Hydroxide eluents offer improved sensitivity for suppressed conductivity detection as compared to carbonate eluents. This improved sensitivity was documented in Dionex (now part of Thermo Scientific) AN 167.[6] Hydroxide eluents are also advantageous because they can be generated easily using an eluent generator as part of a Reagent-Free™ IC (RFIC™) system. RFIC systems improve reproducibility and simplify analysis. The Dionex IonPac AS19 separation can also replace the AS9-HC separation in EPA Methods 317.0 and 326.0, which is documented in Dionex (now part of Thermo Scientific) AN 168 and AN 171.[7,8] The Dionex IonPac AS19 was also used with an isocratic hydroxide eluent rather than the typical gradient for analysis of drinking water for bromate.[9] This method, presented in Application Update 154 (AU 154), cannot determine all the common inorganic anions in a single injection like the gradient method in AN 167. For determination of sub µg/L concentrations of bromate in drinking water and higher ionic strength matrices without postcolumn derivatization, We developed a two-dimensional IC technique (AN 187) that uses a Dionex IonPac AS19 column in the first dimension, and an AS24 column, developed specifically for determining haloacetic acids and bromate by IC-MS and IC-MS/MS, in the second dimension.[10]

Dionex (now part of Thermo Scientific) AN 184 shows that the Dionex IonPac AS19 method in AN 167 could be used to meet the 3 µg/L European limit for bromate in natural mineral and spring waters disinfected by ozonation.[11] The same application note compares the Dionex IonPac AS19 chromatography to chromatography with the AS23, a column that uses carbonate eluents and was designed to replace the AS9-HC. The Dionex IonPac AS23 has a higher capacity than the AS9-HC, and a different selectivity for the carbonate ion so that it is less likely to interfere with bromate determinations. AN 184 shows that poorer sensitivity associated with using carbonate eluents when compared to hydroxide eluents made the Dionex IonPac AS23 performance inferior to that of the AS19.

This application note describes the use of the Thermo Scientific Dionex CRD 300 Carbonate Remove Device to remove the majority of carbonate from the eluent and allow hydroxide-like performance and detection sensitivity. This device was used with the Dionex IonPac AS23 to determine bromate in bottled mineral water samples. Detection sensitivity when using the Dionex CRD 300 Carbonate Removal Device was improved compared to chromatography without the Dionex CRD 300 device. Scientists responsible for water analysis can choose the column and eluent chemistry that best meets their needs to reliably determine bromate at concentrations below the common 10 µg/L regulatory limit.

Equipment

- Thermo Scientific Dionex ICS-2000 Reagent-Free Ion Chromatography System* equipped with the following for carbonate/bicarbonate eluent generation:
  - Thermo Scientific Dionex EluGen™ EGC II K_2CO_3 cartridge (P/N 058904)
  - Thermo Scientific EPM Electrolytic pH Modifier (P/N 063175)
  - Thermo Scientific Dionex EGC Carbonate Mixer (P/N 079943)
- Dionex CRD 300 Carbonate Removal Device (4 mm) with VC Vacuum Pump (P/N 068474)
- Thermo Scientific Dionex Chromeleon™ 6.8 Chromatography Management Software

*This application can be run on any of our systems equipped for carbonate/bicarbonate eluent generation. Alternately, this application can be run with a manually prepared carbonate/bicarbonate eluent.

Reagents and Standards

- Deionized water, type I reagent grade, 18 MΩ-cm resistivity or better
- Sodium chlorite, 80% ($NaClO_2$, Fluka)
- Potassium bromate ($KBrO_3$, Fluka)
- Sodium chlorate ($NaClO_3$, Fluka)
- Individual stock standards of fluoride, chloride, and sulfate, 1000 mg/L each (Merck)

Preparation of Solutions and Reagents

Carbonate Eluent Generation

The eluent generator (EG) produces the eluent using the Dionex EluGen EGC II K_2CO_3 cartridge, EPM Electrolytic pH Modifier, EGC Carbonate Mixer, and deionized water supplied by the pump. The eluent concentration is controlled by the Chromeleon software. Backpressure tubing must be added to achieve 2300–2500 psi backpressure that will allow the EG degasser to function properly. See the Dionex ICS-2000 Operator's Manual Section 2.4.4, "Eluent Generator" for instructions on adding backpressure.

To set up the Dionex EGC II K_2CO_3, see the EGC II K_2CO_3 Cartridge, Electrolytic pH Modifier, and EGC Carbonate Mixer Product Manual (Doc. No. 065075) for more information.

Manual Eluent Preparation

From Eluent Concentrate

Prepare 1 L of eluent by adding 10 mL of the Dionex IonPac AS23 Eluent Concentrate (P/N 064161) to a 1 L volumetric flask. Bring to volume with DI water and mix thoroughly.

From Manually Prepared Stock Solutions

Stock Carbonate/Bicarbonate Eluent Preparation

1.0 M Na_2CO_3 and 1.0 M $NaHCO_3$

Weigh 10.596 g sodium carbonate and 8.400 g sodium bicarbonate into separate 100 mL volumetric flasks. Bring each to volume with DI water.

Dionex IonPac AS23 Eluent (4.5 mM Na_2CO_3 /0.8 mM $NaHCO_3$)

For 1L, prepare by adding 4.5 mL of 1.0 M Na_2CO_3 and 0.8 mL of 1.0 M $NaHCO_3$ to a 1 L volumetric flask, bring to volume with DI water, and mix thoroughly.

Stock Standard Solutions

Prepare 1000 mg/L stock standard solutions of fluoride, chloride, sulfate, chlorite, bromate, and chlorate by weighing 0.221 g, 0.165 g, 0.148 g, 0.168 g, 0.131 g, and 0.128 g, respectively, into separate 100 mL volumetric flasks. Bring each to volume with DI water.

Secondary Standards

The stock standards are used to prepare the 1000 µg/L secondary standards of chlorite, bromate, and chlorate. Take a defined volume of the stock standard and dilute it 1 to 1000 with DI water (e.g., dilute 100 µL to 100 mL in a 100 mL volumetric flask). Use these standards to prepare the working standards and to spike the bottled mineral water sample.

Working Standards

Prepare the standards for calibration and MDL studies by mixing defined volumes of the 1000 mg/L stock standard solutions of fluoride, chloride, and sulfate and the 1000 µg/L secondary standards of chlorite, bromate, and chlorate. For example, to prepare the working standard containing 0.5 mg/L fluoride, 50 mg/L chloride, 100 mg/L sulfate, and 40 µg/L of each of the oxyhalides, add 0.05 mL of the fluoride stock standard, 5 mL of the chloride stock standard, 10 mL of the sulfate stock standard, and 4 mL of each oxyhalide secondary standard to a 100 mL volumetric flask and bring to volume.

Sample

The bottled mineral water sample was purchased from a local market in Bangkok, Thailand, and was bottled at its source in the mountains of Thailand. The label reported the presence of fluoride, chloride, sulfate, and bicarbonate, but not their concentrations.

CRD 300 in Vacuum Mode Setup

The Dionex CRD 300 Carbonate Removal Device in vacuum mode uses a vacuum pump to evacuate the regenerant chamber of the device so that CO_2 gas is literally sucked out of the eluent. A bleed tube feeds a trickle of fresh air into the regenerant chamber to constantly sweep out the CO_2 gas. To operate the Dionex CRD 300 device in vacuum mode, mount the Dionex CRD 300 device directly on top of the suppressor and plumb the eluent from the Eluent Out of the suppressor to the Eluent In of the Dionex CRD 300 device. The Eluent Out of the Dionex CRD 300 device is connected to the conductivity cell In and conductivity cell Out goes to waste if the system is running in external water mode. If the system is operated in recycle mode, connect conductivity cell Out to the suppressor Regen In. Connect the vacuum tubing to the vacuum port of the vacuum pump and to the ballast bottle. Connect a length of 1/8 in. Teflon® tubing from the ballast bottle to the Regen Out of the CRD 300. Make sure the third port on the ballast bottle is closed and air tight. Connect 15 cm of red (0.005 in. i.d.) PEEK™ tubing to the Regen In of the CRD 300; this is the air bleed assembly. Begin eluent flow before beginning vacuum operation. When eluent flow is established, turn on the vacuum pump. The background conductivity should drop almost immediately. When the eluent pump is turned off, immediately turn off the vacuum pump. Avoid operating the vacuum pump while eluent flow is stopped. A TTL can be wired to automate stopping the vacuum pump.

Conditions

Condition A (Eluent Generation and Dionex CRD 300)

Column:	Dionex IonPac AS23, 4 × 250 mm (P/N 064149) Dionex IonPac AG23, 4 × 50 mm (P/N 064147)
Eluent:	Dionex EGC II K_2CO_3 (P/N 058904) Dionex EPM (P/N 063175) 4.5 mM K_2CO_3/0.8 mM $KHCO_3$
Flow Rate:	1.0 mL/min
Inj. Volume:	250 µL
Temperature:	30 °C
Suppressor:	Suppressed conductivity, Thermo Scientific Dionex ASRS™ 300, 4 mm (P/N 064554), external water mode, 25 mA CRD 300, 4 mm, (P/N 064637) vacuum mode
Background:	< 1.5 µS
Noise:	~ 0.3 nS
Back Pressure:	~2200 psi

Condition B (Manual Eluent Preparation and no Dionex CRD 300)

Column:	Dionex IonPac AS23, 4 × 250 mm (P/N 064149) Dionex IonPac AG23, 4 × 50 mm (P/N 064147)
Eluent:	4.5 mM Na_2CO_3/0.8 mM $NaHCO_3$
Flow Rate:	1.0 mL/min
Inj. Volume:	250 µL
Column Temp:	30 °C
Suppressor:	Suppressed conductivity, Dionex ASRS 300, 4 mm (P/N 064554), external water mode, 25 mA
Background:	17-19 µS
Noise:	~ 3.0 nS
Back Pressure:	~1800 psi

Results and Discussion

Chromatography

Bromate, chlorite, and chlorate were resolved from seven common inorganic anions using a Dionex IonPac AS23 column under its recommended eluent conditions (4.5 mM Na_2CO_3/0.8 mM $NaHCO_3$). Chromatogram B in Figure 1 shows this separation. The background conductivity after suppression using the carbonate eluent is between 18 and 19 µS. The higher the background, the higher the noise, and this results in a lower signal-to-noise ratio (i.e., lower sensitivity). The background of the suppressed hydroxide eluent used for the Dionex IonPac AS19 column is < 1 µS. In order for the carbonate eluent system of the Dionex IonPac AS23 to approach the detection limits delivered by the hydroxide eluent system of the AS19, the background must be reduced. The Dionex CRD 300 device was designed to remove carbonate from the eluent (after suppression) and thereby reduce the background to improve detection limits. Chromatogram A shows the same Dionex IonPac AS23 separation as B using a Dionex CRD 300 device. Note that the background has been reduced to about 1 µS, the injection dip at about 2 min is greatly reduced in size, and there is a noticeable improvement in analyte sensitivity. Throughout this application note, we compare the determination of bromate, chlorite, and chlorate with the Dionex IonPac AS23 and suppressed conductivity, both with and without the Dionex CRD 300 device.

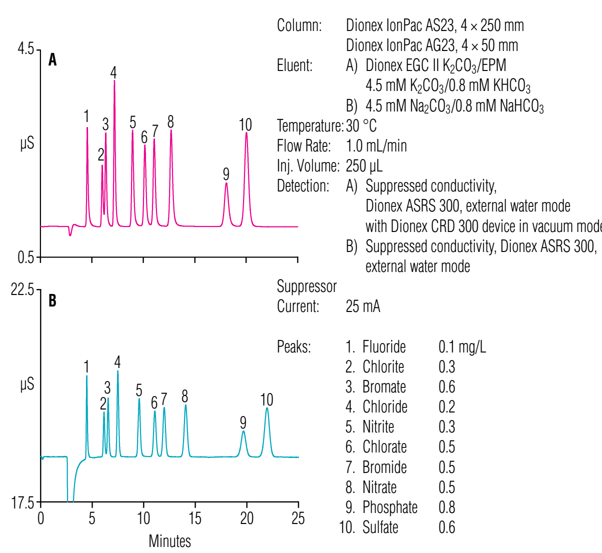


Figure 1. Chromatography of a mixed anion standard A) with a Dionex CRD 300 Carbonate Removal Device and electrolytically prepared eluent, and B) without a Dionex CRD 300 device and with manually prepared eluent.

Figure 2 shows single injections from the MDL determinations of bromate, chlorite, and chlorate with and without the Dionex CRD 300 device. Fluoride, (0.5 mg/L), chloride (50 mg/L), and sulfate (100 mg/L) were added to the MDL standards to simulate the ionic strength of bottled water samples. Due to the higher background and noise of the system without the Dionex CRD 300 device (Chromatogram B, Figure 2), higher analyte concentrations were used for the MDL test compared to the system with the Dionex CRD 300 device. Table 1 shows the results of the MDL determination. For all three oxyhalide analytes, the MDL is lower for the system with the Dionex CRD 300 device. The MDL values without the Dionex CRD 300 device are similar to those determined with the Dionex IonPac AS23 in AN 184. The values when using the Dionex CRD 300 device, though lower than without, are not as low as those determined with the Dionex IonPac AS19 and hydroxide eluent in AN 184.

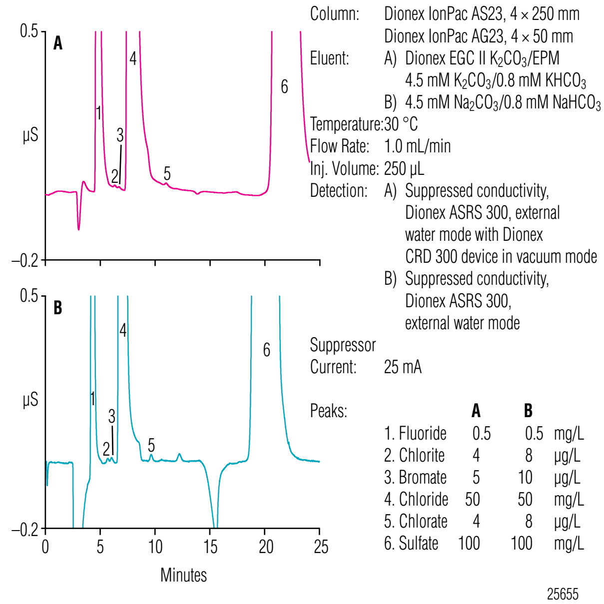


Figure 2. Example chromatograms from the MDL determination A) with a Dionex CRD 300 Carbonate Removal Device, and B) without a Dionex CRD 300 device.

Table 1. MDL determinations of chlorite, bromate, and chlorate with and without a Dionex CRD 300 Carbonate Removal Device.

Injection No.	Height (µS)					
	With Dionex CRD 300 Device			Without Dionex CRD 300 Device		
	Chlorite	Bromate	Chlorate	Chlorite	Bromate	Chlorate
	4 µg/L	5 µg/L	4 µg/L	8 µg/L	10 µg/L	8 µg/L
1	0.0057	0.0041	0.0076	0.0099	0.0121	0.0189
2	0.0051	0.0042	0.0071	0.0114	0.0128	0.0199
3	0.0053	0.0042	0.0065	0.0093	0.0115	0.0204
4	0.056	0.0043	0.0074	0.0105	0.0132	0.0215
5	0.059	0.0047	0.0074	0.0111	0.0133	0.0205
6	0.0061	0.0045	0.0077	0.0103	0.0125	0.0201
7	0.0057	0.0042	0.0076	0.0114	0.0111	0.0199
Average	**0.0056**	**0.0043**	**0.0073**	**0.0105**	**0.0124**	**0.0202**
RSD	**5.97**	**5.04**	**5.42**	**7.41**	**6.67**	**3.94**
MDL (µg/L)	**0.75**	**0.79**	**0.68**	**1.86**	**2.10**	**0.99**

Another calibration was performed for both systems using consistent concentrations of fluoride, chloride, and sulfate (0.5 mg/L, 2 mg/L, and 10 mg/L, respectively) in standards with three levels of chlorite, bromate, and chlorate concentrations; 10, 20, and 40 µg/L. Overlays of three calibration standards are shown in Figure 3 and the results are in Table 2. The calibration data are equivalent.

Both systems were used to analyze a bottled mineral water sample from the mountains of Thailand. Figure 4 shows the analysis of this sample and Table 3 reports the results of the analysis. The sample had just over 10 µg/L bromate and 1–2 µg/L chlorate, suggesting a second disinfection process besides ozonation was used. Due to the noise of the system without the Dionex CRD 300 device, the chlorate peak could not be identified with confidence. To evaluate accuracy, known amounts of bromate, chlorite, and chlorate were spiked into the bottled mineral water sample. Figure 5 shows the chromatography from this study and Table 4 shows that all analytes were recovered at >85%. In this experiment, the recovery was better for the system with the Dionex CRD 300 device.

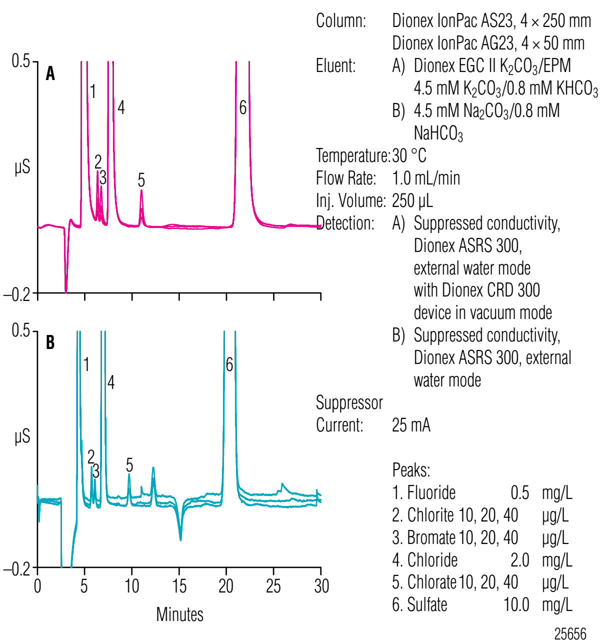


Figure 3. Overlay of chromatograms of three concentration levels of chlorite, bromate, and chlorate in a mixed anion standard A) with a Dionex CRD 300 Carbonate Removal Device, and B) without a Dionex CRD 300 device.

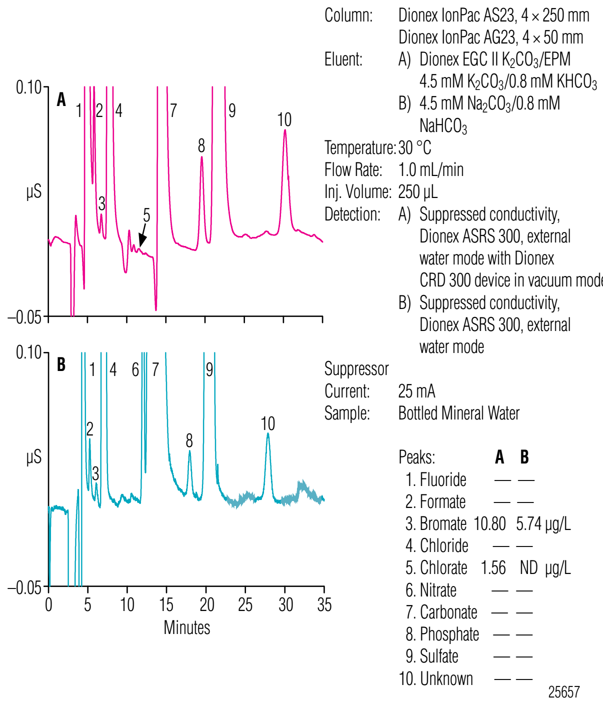


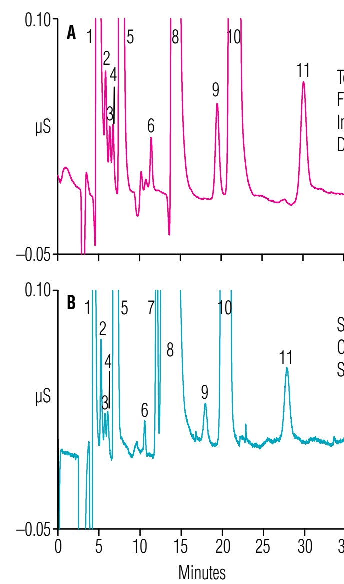
Figure 4. Chromatography of a bottled mineral water sample A) with a Dionex CRD 300 Carbonate Removal Device, and B) without a Dionex CRD 300 device.

Table 2. Chromeleon calibration report for chlorite, bromate, and chlorate with and without a Dionex CRD 300 Carbonate Removal Device.

Peak Name	Points	R-Square (%)	
		With Dionex CRD 300 Device	Without Dionex CRD 300 Device
Chlorite	3	99.9961	99.9748
Bromate	3	100.0000	99.9986
Chlorate	3	99.9995	99.9637

Table 3. Determination of bromate and chlorate in a bottled mineral water sample with and without a Dionex CRD 300 Carbonate Removal Device.

Injection No.	With Dionex CRD 300 Device (µg/L)		Without Dionex CRD 300 Device (µg/L)	
	Bromate	Chlorate	Bromate	Chlorate
1	11.0	1.52	5.33	ND
2	10.9	1.55	6.23	ND
3	10.9	1.35	5.02	ND
4	10.1	1.91	6.25	ND
5	11.3	1.48	5.89	ND
Average	10.8	1.56	5.74	—
RSD	4.34	13.42	9.61	—



Column: Dionex IonPac AS23, 4 × 250 mm
Dionex IonPac AG23, 4 × 50 mm
Eluent: A) Dionex EGC II K_2CO_3/EPM 4.5 mM K_2CO_3/0.8 mM $KHCO_3$
B) 4.5 mM Na_2CO_3/0.8 mM $NaHCO_3$
Temperature: 30 °C
Flow Rate: 1.0 mL/min
Inj. Volume: 250 µL
Detection: A) Suppressed conductivity, Dionex ASRS 300, external water mode with Dionex CRD 300 device in vacuum mode
B) Suppressed conductivity, Dionex ASRS 300, external water mode
Suppressor Current: 25 mA
Sample: Bottled mineral water spiked with chlorite, bromate, and chlorate

Peaks:	A	B
1. Fluoride	—	—
2. Formate	—	—
3. Chlorite	9.88	8.58 µg/L
4. Bromate	20.51	15.30 µg/L
5. Chloride	—	—
6. Chlorate	12.02	8.50 µg/L
7. Unknown-1	—	—
8. Carbonate	—	—
9. Phosphate	—	—
10. Sulfate	—	—
11. Unknown -2	—	—

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Figure 5. Chromatography of a bottled mineral water sample spiked with chlorite, bromate, and chlorate (10 µg/L each) A) with a Dionex CRD 300 Carbonate Removal Device, and B) without a Dionex CRD 300 device.

Table 4. Spike recovery of chlorite, bromate, and chlorate with and without a Dionex CRD 300 Carbonate Removal Device.

	With Dionex CRD 300 Device			Without Dionex CRD 300 Device		
	Chlorite	Bromate	Chlorate	Chlorite	Bromate	Chlorate
Sample	ND[a]	10.83	1.56	ND[a]	5.74	ND[a]
Spike	10	10	10	10	10	10
Measured[b] Amount	9.88	20.51	12.02	8.58	15.30	8.50
RSD	2.39	1.60	2.45	2.39	1.60	2.45
Recovery (%)	98.8	98.5	104	85.8	97.2	85.0

Summary

This application note shows that using the Dionex CRD 300 Carbonate Removal Device with the Dionex IonPac AS23, bromate can be determined in bottled mineral water at concentrations < 5 µg/L. The method sensitivity for bromate and other oxyhalides approaches that of the hydroxide eluent system featured in Dionex (now part of Thermo Scientific) AN 184.

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