

Determination of Trace Concentrations of Chlorite, Bromate, and Chlorate in Bottled Natural Mineral Waters

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Introduction

Bottled water has been one of the fastest growing beverage markets in the last five to ten years. Global consumption approached 41 billion gallons in 2004, an increase of 6.5% from 2003.¹ The bottled water industry markets to health conscious consumers as an alternative not only to tap water, but also to carbonated soft drinks and juice drinks.¹ Regardless of whether the water is delivered from a local municipality or is prepackaged in a bottle, the consumption of safe and reliable drinking water is essential to maintain a healthy lifestyle.

Bottled water must be disinfected to remove pathogenic microorganisms and ensure it is safe for human consumption. Water companies prefer ozone as a disinfectant because it is one of the most effective treatments available, it does not leave a taste, and there is no residual disinfectant in the bottled water.^{2,3} Some bottlers, however, use ultraviolet light or chlorine dioxide as alternative treatment methods.² Reactions between disinfectants and natural organic and inorganic matter in the source water can result in the production of undesirable disinfection byproducts (DBPs), such as chlorite, bromate, and trihalomethanes, that are potentially harmful to humans.⁴ Bromate, for example, can be formed by ozonation of water containing naturally occurring bromide, or may be present as an impurity in sodium hypochlorite used for treatment.⁵

Results from toxicological studies led the International Agency for Research on Cancer to conclude that bromate is a potential human carcinogen, even at low $\mu\text{g/L}$ (ppb) concentrations.⁶ The World Health Organization (WHO) estimated excess lifetime cancer risks of 10^{-4} , 10^{-5} , and 10^{-6} for drinking water containing bromate at 20, 2, and 0.2 $\mu\text{g/L}$, respectively.⁹ The U.S. EPA,⁷ European Commission,⁸ and the WHO⁹ set a maximum permissible limit of 10 $\mu\text{g/L}$ bromate in tap water. The U.S. FDA¹⁰ adopted the same regulatory limit for bottled water. In Europe, natural mineral waters and spring waters treated by ozonation have a maximum permissible limit of 3 $\mu\text{g/L}$ bromate.¹¹

Traditionally, ion chromatography (IC) with suppressed conductivity detection has been used for determination of bromate and other DBPs in drinking water, as described in EPA Method 300.1.¹² This method describes the use of a high-capacity Thermo Scientific™ Dionex™ IonPac™ AS9-HC column with a carbonate eluent and large loop injection to achieve a method detection limit (MDL) of 1.4 $\mu\text{g/L}$ bromate. In early 2006, the U.S. EPA enacted Stage 2 of the disinfectants/disinfection byproducts (D/DBP) rule, maintaining the maximum permissible limit for bromate but adding three additional analytical methods to further improve the selectivity and sensitivity for bromate.¹³ U.S. EPA Methods 317.0 and 326.0 combine suppressed conductivity detection and absorbance detection after postcolumn addition to achieve bromate MDLs less than 0.2 $\mu\text{g/L}$.^{14,15} IC coupled to inductively coupled plasma mass spectrometry has also been demonstrated for the determination of low concentrations of bromate in environmental waters, permitting a bromate MDL of 0.3 $\mu\text{g/L}$.¹⁶

A high-capacity Dionex IonPac AS19 column with an electrolytically generated hydroxide eluent, large loop injection, and suppressed conductivity detection can achieve a calculated bromate MDL of 0.34 $\mu\text{g/L}$.¹⁷ Absorbance detection after postcolumn addition can reduce this MDL to less than 0.2 $\mu\text{g/L}$, using EPA Methods 317.0 and 326.0.^{18,19} In this application note, we compare the Dionex IonPac AS19 column using an electrolytically generated hydroxide eluent to the Dionex IonPac AS23 column using an electrolytically generated carbonate/bicarbonate eluent for the determination of chlorite, bromate, and chlorate in natural mineral waters. We compare the linearity, method detection limits, precisions, and recovery for three mineral waters obtained from three European countries to determine whether these columns have the sensitivity required to meet current EPA and EU requirements.

Equipment

Thermo Scientific™ Dionex™ ICS-2000 Reagent-Free™ Ion Chromatography (RFIC™) system* was used in this work. The Dionex ICS-2000 is an integrated ion chromatograph and consists of:

- Eluent generator
- Pump with in-line vacuum degas
- Column heater
- Hydroxide system:
 - Thermo Scientific Dionex EluGen™ EGC II KOH cartridge
- Thermo Scientific Dionex CR-ATC Continuously Regenerated Anion Trap Column
- Carbonate system:
 - Dionex EluGen EGC II K₂CO₃ cartridge
- EPM Electrolytic pH Modifier to generate the carbonate/bicarbonate eluent
- EGC Carbonate Mixer
- Two 4 L plastic bottle assemblies (for external water mode of suppression)
- Thermo Scientific Dionex AS Autosampler
- Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System (CDS) Software

*Equivalent or improved results can be achieved using the Thermo Scientific Dionex ICS-2100 system.

Reagents and Standards

- Deionized water, Type I reagent grade, 18 MΩ-cm resistivity or better
- Sodium chlorite (NaClO₂, 80% pure)
- Sodium bromate (NaBrO₃)
- Sodium chlorate (NaClO₃)

Conditions

Columns:	(A) Dionex IonPac AS19 Analytical, 4 × 250 mm Dionex IonPac AG19 Guard, 4 × 50 mm (B) Dionex IonPac AS23 Analytical, 4 × 250 mm Dionex IonPac AG23 Guard, 4 × 50 mm
Eluent:	(A) 10 mM KOH from 0–10 min, 10–45 mM from 10–25 min, 45 mM from 25–30 min* (B) 4.5 mM K ₂ CO ₃ /0.8 mM KHCO ₃
Eluent Source:	(A) Dionex EGC II KOH with Dionex CR-ATC (B) Dionex EGC II K ₂ CO ₃ with EPM
Flow Rate:	1.0 mL/min
Temperature:	30 °C
Injection:	250 µL
Detection:	(A) Suppressed conductivity, Thermo Scientific Thermo Scientific™ Dionex™ ASRS™ ULTRA II Suppressor, 4 mm, auto-suppression, recycle mode, 130 mA current (B) Suppressed conductivity, Dionex ASRS ULTRA II suppressor, 4 mm, auto-suppression, external water mode, 25 mA current
CRD:	(A) 4 mm format
Background Conductance:	(A) <1 µS (B) 18–20 µS
System Backpressure:	~2200 psi
Run Time:	30 min

*Method returns to 10 mM KOH for 3 min prior to injection.

Preparation of Solutions and Reagents

Eluent Solution for the Dionex IonPac AS23 Column 4.5 mM Carbonate/0.8 mM Bicarbonate

Generate the carbonate/bicarbonate eluent on-line by pumping high quality deionized water (18 MΩ-cm resistivity or better) through the Dionex EluGen EGC II K₂CO₃ cartridge and EPM. The Chromeleon software will track the amount of eluent used and calculate the remaining lifetime.

Alternatively, prepare the eluent solution by adding 10 mL of the Dionex IonPac AS23 Eluent Concentrate to a 1 L volumetric flask containing approximately 700 mL of degassed deionized water. Bring to volume and mix thoroughly. The 0.45 M sodium carbonate/0.08 M sodium bicarbonate concentrate can also be prepared from the salts by combining 47.7 g sodium carbonate (MW=106 g/mole) and 6.72 g sodium bicarbonate (MW=84 g/mole) in a 1 L volumetric flask containing approximately 700 mL of degassed deionized water. Bring to volume and mix thoroughly.

Stock Standard Solutions

Prepare 1000 mg/L stock standard solutions of chlorite, bromate, and chlorate by dissolving 0.1676 g, 0.1180 g, and 0.1275 g, respectively, of the corresponding sodium salts in separate 100 mL volumetric flasks of DI water.

Calibration Standard Solutions

Prepare a secondary stock solution containing 1 mg/L each of chlorite and chlorate and a separate secondary stock solution containing 1 mg/L bromate by performing the appropriate dilutions of the 1000 mg/L stock standards. Calibration standards can then be prepared from the secondary solutions using the appropriate dilutions. Dilute working standards should be prepared monthly, except those that contain chlorite, which must be prepared every two weeks, or sooner if evidence of degradation is indicated by repeated QC failures. Concentration ranges used in this application note are shown in Table 1.

Table 1. Calibration data, retention time precisions, peak area precisions, and method detection limits for DBP anions.

Dionex IonPac AS19 Column						
Analyte	Range (µg/L)	Linearity (r ²)	Retention Time ^a RSD (%)	Peak Area RSD (%)	MDL Standard (µg/L)	Calculated MDL (µg/L)
Chlorite	2–50	0.9999	0.04	1.20	1.0	0.18
Bromate	1–25	0.9995	0.03	1.40	2.0	0.31
Chlorate	2–50	0.9999	0.01	0.54	1.0	0.28
Dionex IonPac AS23 Column						
Chlorite	10–50	0.9999	0.07	2.20	5.0	1.02
Bromate	5–25	0.9998	0.07	2.63	5.0	1.63
Chlorate	10–50	0.9998	0.11	2.48	9.0	2.05

^aRSD= relative standard deviation, n = 10 for a standard consisting of 10 ppb bromate and 20 ppb each of chlorite and chlorate.

Sample Preparation

For the present analysis, mineral waters B and C were degassed for 10–15 min under vacuum due to an excess amount of bicarbonate in the samples. Increased amounts of bicarbonate in the sample can produce shifts in retention times as shown in Figures 1A and 1B. In addition, due to the presence of significantly high concentrations of sulfate in mineral water C, the sample was diluted 1:5 with DI water prior to analysis.

Results and Discussion

The Dionex IonPac AS23 column is a high-capacity anion-exchange column specifically designed to be used with carbonate/bicarbonate eluent for the determination of the trace DBPs, chlorite, bromate, and chlorate, together with common inorganic anions, including bromide (precursor to bromate), in drinking waters. To simplify the method and avoid manual eluent preparation, this column can be used with electrolytically generated potassium carbonate that is modified by an Electrolytic pH Modifier (EPM) to automatically generate the carbonate/bicarbonate eluent that is required for analyte separation. The Dionex IonPac AS23 column was developed using a unique polymer technology to achieve a capacity of 320 $\mu\text{eq}/\text{column}$, higher than the Dionex IonPac AS9-HC column (190 $\mu\text{eq}/\text{column}$) described in EPA Method 300.1. The combination of an optimized selectivity for DBP anions, high anion exchange capacity, and improved selectivity of carbonate from inorganic anions and oxyhalides, makes this column an ideal replacement for the Dionex IonPac AS9-HC column.

In this application, we compare the Dionex IonPac AS23 column to the hydroxide-selective AS19 column for the determination of trace DBP anions in natural mineral waters. Figure 2 compares the separation for chlorite, bromate, and chlorate on the Dionex IonPac AS19 and AS23 columns. As shown, both columns provide good selectivity for the target DBP anions.

The linear calibration ranges, MDLs, and quality control standard (QCS) performances were evaluated for the hydroxide and carbonate eluent systems. The hydroxide eluent system was calibrated using four increasing concentrations of chlorite and chlorate (2–50 $\mu\text{g}/\text{L}$) and five increasing concentrations of bromate (1–25 $\mu\text{g}/\text{L}$). For the carbonate-based system, chlorite and chlorate were calibrated from 10–50 $\mu\text{g}/\text{L}$ whereas bromate was calibrated from 5–25 $\mu\text{g}/\text{L}$ using three different concentrations. Each system produced a linear response in its respective range with a correlation coefficient greater than 0.999. The improved sensitivity of the hydroxide eluent system, however, allowed a lower minimum reporting limit (MRL) than the carbonate-based system. The MDLs for the target DBPs were determined for each system by performing seven replicate injections of reagent water fortified with the calibration standards at concentrations of three to five times the estimated instrument detection limits.

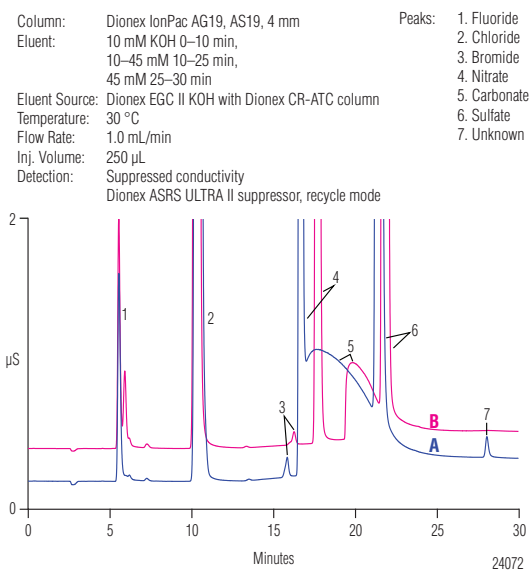


Figure 1. Comparison of mineral water B A) before vacuum degas and B) after vacuum degas.

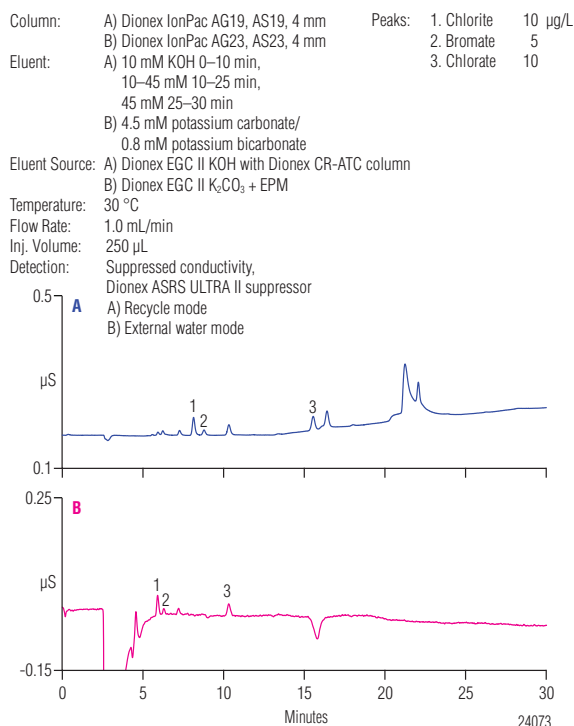


Figure 2. Separation of disinfection byproducts using the A) Dionex IonPac AS19 column and B) Dionex IonPac AS23 column.

Table 1 compares the calibration data, retention time and peak area precisions for a QCS, and MDLs for the Dionex IonPac AS19 column with an electrolytically generated hydroxide eluent to the AS23 with an electrolytically generated carbonate/bicarbonate eluent. The calculated MDL of bromate with the Dionex IonPac AS19 column was 0.31 µg/L compared to 1.63 µg/L using the Dionex IonPac AS23 column. This demonstrates that hydroxide eluents improve the sensitivity for bromate compared to carbonate-based eluents and are therefore more suitable to meet the current European regulatory requirement of 3 µg/L bromate in natural mineral waters. Either the Dionex IonPac AS19 or AS23 based IC systems are capable of measuring the 10 µg/L requirement of bromate for tap water or U.S. bottled water according to the regulations established by the U.S. EPA, U.S. FDA, WHO, and European Commission.

In the U.S., mineral water is defined as water that contains no less than 250 ppm total dissolved solids (TDS) and that originates from a geologically and physically protected underground water source. Mineral content must be maintained at a constant level and no minerals may be added to the water.¹⁸ In Europe, mineral water is defined as microbiologically wholesome water, originating from an underground water table or deposit and emerging from a spring tapped at one or more natural or bored exits. It can contain less than 50 ppm TDS.¹⁹ The total mineral content of the waters can vary significantly, with higher mineral concentrations generally appearing in Russia, the Baltic States, and Germany. The differences between regions are most likely a result of differences in the overall compositions of the waters and the geological locations.²⁰

In this application, three natural mineral waters from different European countries with TDSs that varied significantly from 136 to 2359 ppm were evaluated. The properties of the investigated water samples are summarized in Table 2. As shown, the ionic strength of mineral water C is significantly higher than observed in typical drinking waters. The absence of bromate in the bottled mineral waters analyzed indicated that ozonation was not used for disinfection.

Table 2. Concentrations in mg/L of cations and anions in the investigated mineral water samples.

Mineral water	Na ⁺	K ⁺	Mg ²⁺	Ca ²⁺	F ⁻	Cl ⁻	NO ₃ ⁻	HCO ₃ ⁻	SO ₄ ²⁻
A	11.8	6.2	8	11.5	— ^a	13.5	6.3	71	8.1
B	4.5	0.5	8	32.0	— ^a	5.0	< 2	133	7.0
C	4.2	— ^a	117	510	1.8	3.0	< 0.1	278	1445

^aNot specified.

Tables 3 and 4 summarize typical recoveries for single-operator data obtained using the Dionex IonPac AS19 and AS23 columns, respectively, for trace concentrations of DBP anions in three European natural mineral water samples. As shown, chlorite and bromate were not detected in any of the samples analyzed, whereas only a trace concentration of chlorate was detected in mineral water A. To determine the accuracy of the method, the samples were spiked with 5 µg/L bromate and 10 µg/L each of chlorite and chlorate. Calculated recoveries for the spiked mineral water samples were in the range of 86–97% and 84–111% using the Dionex IonPac AS19 and AS23 columns, respectively. The analyte recoveries using either a hydroxide or carbonate/bicarbonate eluent were within the acceptable range of 75–125% according to the criteria described in

Table 3. Recoveries of disinfection byproduct anions in natural mineral waters using the Dionex IonPac AS19 column.

Mineral Water	Analyte	Amount Found (µg/L)	Amount Added (µg/L)	Recovery (%)
A	Chlorite	<MDL	10	87.7
	Bromate	<MDL	5.0	96.0
	Chlorate	4.4	10	91.1
B	Chlorite	<MDL	10	86.4
	Bromate	<MDL	5.0	97.4
	Chlorate	<MDL	10	90.7
C	Chlorite	<MDL	10	87.6
	Bromate	<MDL	5.0	94.7
	Chlorate	<MDL	10	92.8

Table 4. Recoveries of disinfection byproduct anions in natural mineral waters using the Dionex IonPac AS23 column.

Mineral Water	Analyte	Amount Found (µg/L)	Amount Added (µg/L)	Recovery (%)
A	Chlorite	<MDL	10	107.6
	Bromate	<MDL	5.0	91.2
	Chlorate	4.6	10	99.3
B	Chlorite	<MDL	10	110.6
	Bromate	<MDL	5.0	93.5
	Chlorate	<MDL	10	92.9
C	Chlorite	<MDL	10	104.3
	Bromate	<MDL	5.0	83.9
	Chlorate	<MDL	10	102.6

EPA Method 300.1. Figure 3 compares chromatograms of mineral water A using the Dionex IonPac AS19 and AS23 columns. Figure 4 shows the same chromatograms spiked with 5 µg/L bromate and 10 µg/L each of chlorite and chlorate, which resulted in good recoveries for both eluents. Although bromide was not quantified in this study, the estimated concentrations were approximately 16 µg/L in mineral waters A and B and 2 µg/L in mineral water C. Therefore, ozonation of mineral waters A and B could potentially produce bromate. To demonstrate the applicability of detecting bromate at concentrations significantly less than the 3 µg/L European regulatory limit for ozonated mineral waters, mineral water A was spiked with 0.5 µg/L bromate (Figure 5). As shown, bromate can be observed easily at this concentration, with good peak-to-peak baseline noise of 0.3–0.5 nS.

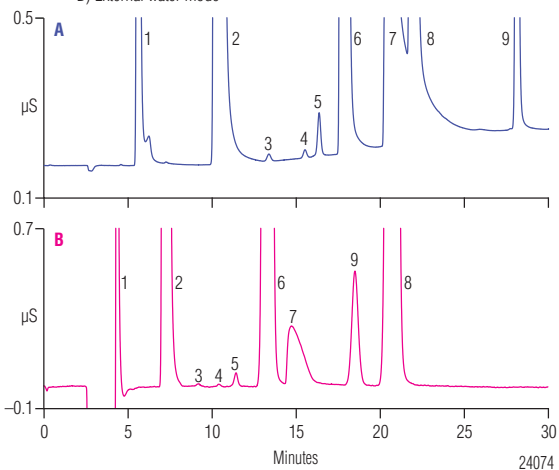
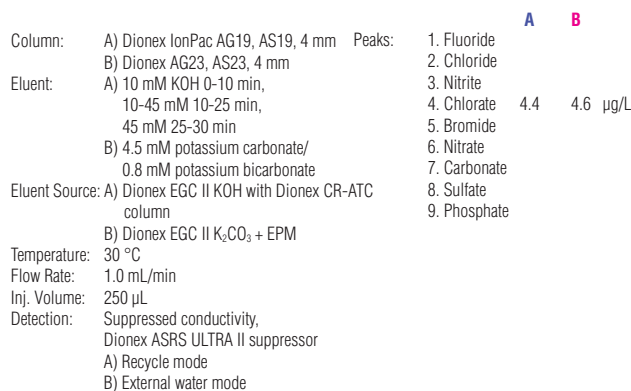


Figure 3. Comparison of the A) Dionex IonPac AS19 and B) Dionex IonPac AS23 columns for the separation of DPB anions in mineral water A.

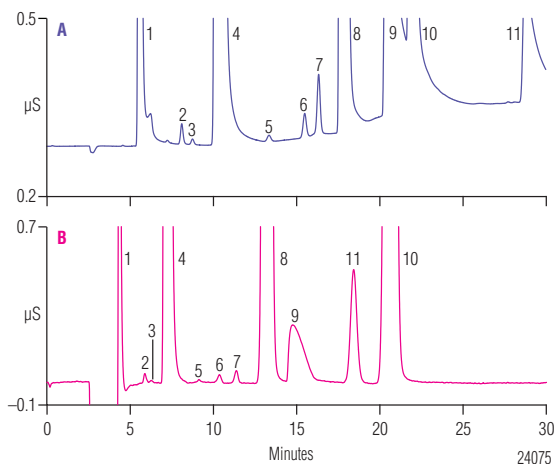
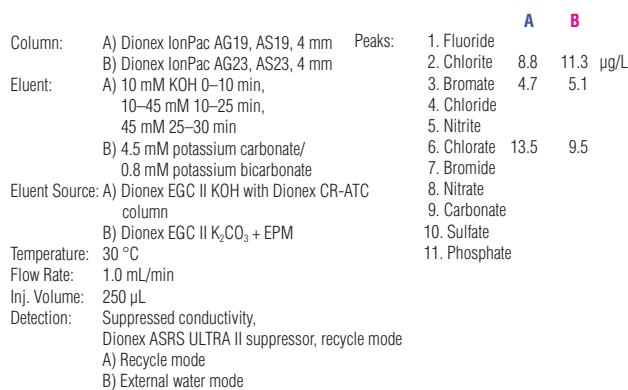


Figure 4. Comparison of the A) Dionex IonPac AS19 and B) Dionex IonPac AS23 columns for the separation of trace concentrations of common anions and DPB anions spiked in mineral water A.

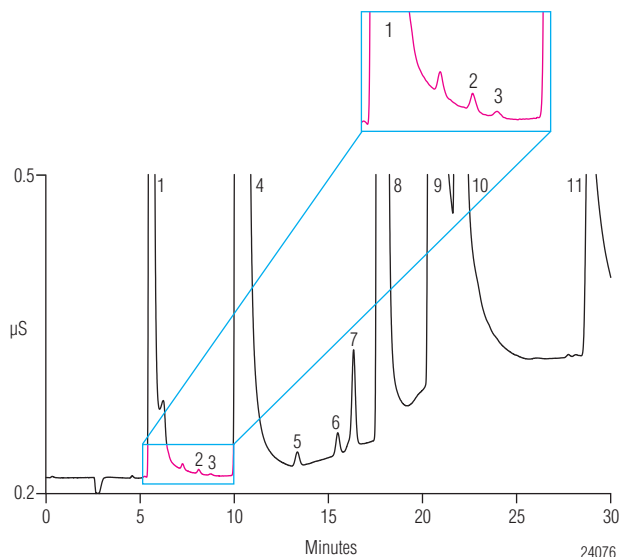
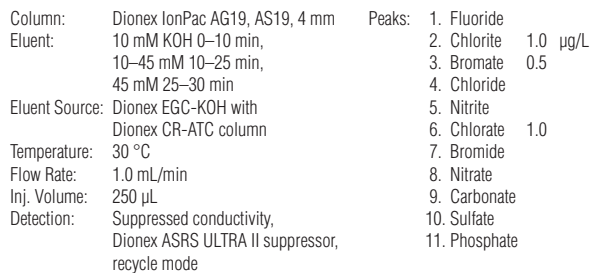


Figure 5. Chromatogram of mineral water A spiked with 1 µg/L each chlorite and chlorate and 0.5 µg/L bromate.

Conclusion

The Dionex IonPac AS19 column using an electrolytically generated hydroxide eluent was compared to the Dionex IonPac AS23 column using an electrolytically generated carbonate/bicarbonate eluent for the determination of trace concentrations of DBP anions in natural mineral waters. The improved sensitivity using a hydroxide eluent allowed the detection of lower concentrations of bromate, a potential human carcinogen, in drinking waters. Therefore, the Dionex IonPac AS19 with an electrolytically generated hydroxide eluent is recommended for laboratories that must comply with EU Directive 2003/40/EC, which permits a maximum of 3 µg/L bromate in mineral waters treated with ozone. The use of either the Dionex IonPac AS19 column with a hydroxide eluent or Dionex IonPac AS23 column with a carbonate/bicarbonate eluent provides the required sensitivity to meet the maximum permissible limit of 10 µg/L bromate currently required by most regulatory agencies. Both columns demonstrated good resolution between bromate and chloride and comparable recovery for mineral water samples spiked with known concentrations of chlorite, bromate, and chlorate. In addition, hydroxide or carbonate/bicarbonate eluents can be generated on-line from deionized water, freeing the operator from manually preparing eluents. This increases the automation, ease-of-use, and reproducibility between analysts and laboratories.

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