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# Determination of Inorganic Oxyhalide Disinfection Byproduct Anions and Bromide in Drinking Water Using Ion Chromatography with the Addition of a Postcolumn Reagent for Trace Bromate Analysis

## INTRODUCTION

The chlorination of drinking water can produce trihalomethanes and other suspected carcinogenic disinfection byproducts (DBPs) that endanger human health.<sup>1</sup> Unfortunately, common alternatives to chlorination also can produce harmful DBPs. The use of chlorine dioxide for the disinfection of drinking water generates the inorganic oxyhalide DBPs chlorite and chlorate, and the presence of chlorate has been reported in waters treated with hypochlorite.<sup>2</sup> Ozonation, an increasingly prevalent and effective disinfection technique, produces bromate as a DBP anion if the source water contains naturally occurring bromide.<sup>3</sup> Bromate has been judged by both the World Health Organization (WHO) and the U.S. Environmental Protection Agency (EPA) as a potential carcinogen, even at very low  $\mu\text{g/L}$  levels. The U.S. EPA has estimated a potential cancer risk equivalent to 1 in  $10^4$  for a lifetime exposure to drinking water containing bromate at  $5 \mu\text{g/L}$ .<sup>4</sup>

The U.S. EPA has recently issued new rules that require public water supplies to control previously unregulated microbes (e.g., cryptosporidium and giardia) and cancer-causing DBPs in finished drinking water. The Stage 1 D/DBP Rule specifies a Maximum Contaminant Level (MCL) for bromate of  $10 \mu\text{g/L}$  and an MCL for chlorite of  $1000 \mu\text{g/L}$ .<sup>5</sup> The EPA intends to convene Stage 2 of the D/DBP Rule in the near future, while both Germany and Japan are considering regulatory limits for inorganic DBPs.<sup>6</sup>

The recent efforts by global regulatory agencies to monitor levels and establish regulatory limits has generated considerable interest in the development of improved analytical methods for the determination of trace level inorganic oxyhalide DBPs. The determination of bromate and other inorganic DBPs traditionally has been accomplished by ion chromatography (IC) using an IonPac® AS9-SC anion-exchange column with a carbonate/bicarbonate eluent and suppressed conductivity detection, as described in U.S. EPA Method 300.0 (B).<sup>7</sup> EPA Method 300.1 was published as an update to Method 300.0 in 1997. Method 300.1 specifies the use of an IonPac AS9-HC column and suppressed conductivity detection for the determination of bromate, bromide, chlorite, and chlorate at low  $\mu\text{g/L}$  levels by direct injection.<sup>8</sup> The detection limit for bromate determined by IC with suppressed conductivity detection can be further reduced to  $1 \mu\text{g/L}$  by using preconcentration after appropriate sample cleanup.<sup>1</sup>

Postcolumn derivatization can also be used to improve detection limits when using IC for inorganic DBP analysis. The use of IC with dual postcolumn addition of hydrochloric acid and then chlorpromazine can achieve a method detection limit (MDL) for bromate of  $0.49 \mu\text{g/L}$ .<sup>9</sup> Iodate, chlorite, and bromate have been detected by using a postcolumn reaction with excess bromide under acidic conditions. The tribromide ion formed can be detected spectrophotometrically at

267 nm, allowing an MDL of less than 0.5 µg/L for bromate with a large-volume injection.<sup>10</sup> Sub-µg/L MDLs for bromate have also been reported by workers using other postcolumn reagents, such as fuchsin or excess iodide under acidic conditions.<sup>11,12</sup> In addition to postcolumn reaction (PCR) methods, electrospray tandem mass spectrometry (MS-MS) and inductively coupled plasma mass spectrometry (ICP-MS) have been used as specific detection techniques for the ion chromatographic analysis of bromate. The use of electrospray MS-MS detection can achieve an MDL for bromate of approximately 0.1 µg/L; the use of ICP-MS detection has been reported to permit an MDL for bromate of 0.8 µg/L.<sup>13,14</sup>

This Application Note describes an improved IC method to quantify low levels of oxyhalide DBP anions and bromide in reagent water, bottled water, and finished drinking water. The method uses an IonPac AS9-HC column and suppressed conductivity detection, followed by postcolumn addition of *o*-dianisidine (ODA) to enhance visible absorbance detection of the bromate ion. This method allows quantification of all the key oxyhalide anions and bromide at low µg/L levels by using conductivity detection, and the postcolumn addition of ODA followed by visible detection allows quantification of bromate down to 0.5 µg/L. This method requires only a single postcolumn reagent delivered pneumatically with conventional postcolumn instrumentation.<sup>2</sup> The approach described in this Application Note is technically equivalent to that described in U.S. EPA Method 317.0 titled “Determination of Inorganic Oxyhalide Disinfection By-Products in Drinking Water Using Ion Chromatography with the Addition of a Postcolumn Reagent for Trace Bromate Analysis”.<sup>15</sup>

## EQUIPMENT

Dionex DX-500 ion chromatographic system consisting of:  
GP50 Gradient Pump with vacuum degas option  
ED40 Conductivity Detector with DS3 Detector Cell  
AD20 UV/Vis Absorbance Detector with 10-mm cell  
AS50 Autosampler  
PC10 Pneumatic Postcolumn Delivery Module (P/N 50601)  
PCH-2 Postcolumn Reaction Heater (P/N 39348)  
Knitted Reaction Coil, 500 µL, potted (for PCH-2) (P/N 39349)  
Two 4-L plastic bottle assemblies (for external water mode suppression)  
PeakNet® 5.1 Chromatography Workstation

## REAGENTS AND STANDARDS

Deionized water, Type I reagent grade, 18 MΩ-cm resistivity or better  
0.5 M Carbonate Anion Eluent Concentrate (Dionex P/N 37162)  
*o*-Dianisidine, dihydrochloride salt (ODA; Sigma D-3252)  
Iron (II) sulfate heptahydrate (Fe<sub>2</sub>SO<sub>4</sub>•7H<sub>2</sub>O; Aldrich 21,542-2)  
Ethylenediamine (EDA; Sigma E-1521)  
Nitric acid, (70%; J.T. Baker INSTRANA-ANALYZED 9598-00)  
Methanol (spectrophotometric grade; Sigma M-3641)  
Potassium bromide (KBr; J.T. Baker 2998)  
Sodium bromide (NaBr; Aldrich 31,050-6)  
Sodium bromate (NaBrO<sub>3</sub>; EM SX 03785-1)  
Sodium chlorate (NaClO<sub>3</sub>; Fluka 71370)  
Sodium chlorite (NaClO<sub>2</sub>; Fluka 71388, ~80% pure)  
Bromate standard, 1000 mg/L, NaBrO<sub>3</sub> in H<sub>2</sub>O (SPEX CertiPrep AS-BRO39-2Y)  
Bromide standard, 1000 mg/L, NaBr in H<sub>2</sub>O (SPEX CertiPrep AS-BR9-2Y)  
Chlorate standard, 1000 mg/L, NaClO<sub>3</sub> in H<sub>2</sub>O (SPEX CertiPrep AS-CLO39-2Y)  
Chlorite standard, 1000 mg/L, NaClO<sub>2</sub> in H<sub>2</sub>O (SPEX CertiPrep AS-CLO29-2Y)

## CONDITIONS

Columns: Dionex AG9-HC, 50 x 4 mm ID guard column (P/N 51791)  
Dionex AS9-HC, 250 x 4 mm ID analytical column (P/N 51786)  
Eluent: 9.0 mM Sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>)  
Flow Rate: 1.3 mL/min  
Temperature: Ambient  
Sample Volume: 225 µL  
Detection: Suppressed conductivity:  
ASRS®-ULTRA (P/N 53946), AutoSuppression® external water mode, 100 mA current, DS3 Cell (P/N 44130), 35 °C, 1.7%/°C.  
Background Conductance: ~24 µS  
System Backpressure: ~2300 psi  
Run Time: 25 min

## PCR

Detection: Absorbance at 450 nm (tungsten lamp)

Postcolumn

Reagent Flow: 0.7 mL/min

Postcolumn

Heater Temp.: 60 °C

## PREPARATION OF SOLUTIONS AND REAGENTS

### Reagent Water

Distilled or deionized water, 18 M $\Omega$ -cm or better, free of the anions of interest and filtered through a 0.2- $\mu$ m filter.

### Eluent Solution (9 mM Sodium Carbonate)

Dilute 18 mL of 0.5 M sodium carbonate concentrate to 1 L with deionized water. Unless the in-line degas option is being used, sparge eluent prior to use with helium or sonicate under vacuum for 10 min.

### Postcolumn Reagent

Add 40 mL of 70% nitric acid to about 300 mL reagent water in a 500-mL volumetric flask. Add 2.5 g KBr and stir to dissolve. Dissolve 250 mg of *o*-dianisidine • 2 HCl in 100 mL methanol and add to the nitric acid/KBr solution. Bring to volume with reagent water. Prepare in advance, set aside overnight until the slight champagne color fades, and filter through a 0.45- $\mu$ m filter. Discard any PCR reagent that is not colorless or nearly colorless after sitting overnight. The reagent is stable for one month when stored at room temperature.

### Stock Standard Solutions

Purchase certified solutions or prepare stock standard solutions by dissolving the corresponding mass of the salt for each of the anions of interest (see Table 1) in reagent water and dilute to 100 mL.

**Table 1 Masses of Compounds Used to Prepare 100 mL of 1000 mg/L Anion Standards**

Anion	Compound	Mass (g)
BrO <sub>3</sub> <sup>-</sup>	Sodium bromate (NaBrO <sub>3</sub> )	0.1180
Br <sup>-</sup>	Sodium bromide (NaBr)	0.1288
ClO <sub>3</sub> <sup>-</sup>	Sodium chlorate (NaClO <sub>3</sub> )	0.1275
ClO <sub>2</sub> <sup>-</sup>	Sodium chlorite (NaClO <sub>2</sub> )	0.1680*

\*Because sodium chlorite is usually available only as an 80% technical grade salt, the 80% purity is accounted for in the 0.1680 g mass cited above. If an alternate purity is used, make an appropriate adjustment in the mass of salt used after determining the exact percentage of NaClO<sub>2</sub>, which can be done using an iodometric titration procedure.<sup>16</sup>

Prepare a mixed anion calibration stock standard at 20 mg/L by combining 2 mL of each of the bromide, chlorite, and chlorate stock standards in a 100-mL volumetric flask. Mix and bring to volume with reagent water. These standards are stable for at least 1 month when stored at < 6 °C.

Because bromate decomposes in the presence of chlorite, prepare a bromate-only calibration stock standard at 5 mg/L by adding 0.5 mL of the bromate stock standard to a 100-mL volumetric flask and bringing to volume with reagent water. This standard is stable for 2 weeks when stored at < 6 °C.

### Working Standard Solutions

Use reagent water to prepare appropriate dilutions of the calibration stock standards as needed.

### Ethylenediamine (EDA) Preservative Solution

Dilute 2.8 mL of ethylenediamine (99%) to 25 mL with reagent water. Prepare fresh monthly.

### Ferrous Iron Solution [1000 mg/L Fe (II)]

Add 6  $\mu$ L concentrated nitric acid to about 15 mL reagent water in a 25 mL volumetric flask. Add 0.124 g ferrous sulfate heptahydrate (FeSO<sub>4</sub>•7H<sub>2</sub>O), dissolve, and bring to volume with reagent water (final pH ~ 2). Prepare fresh every 2 days.

### Sulfuric Acid Solution (0.5 N)

Dilute 1.4 mL of concentrated sulfuric acid to 100 mL with reagent water.

## SAMPLE PREPARATION

When taking a sample from a treatment plant that uses chlorine dioxide or ozone, the sample must be sparged immediately with an inert gas (e.g., nitrogen, argon, or helium) for 5 min. Add 1.00 mL of EDA Preservative Solution per 1.0 L of sample to prevent conversion of residual hypochlorite or hypobromite to chlorate or bromate. This also prevents metal-catalyzed conversion of chlorite to chlorate. The samples preserved in this manner are stable for at least 14 days when stored in amber glass bottles at 4 °C.<sup>17</sup>

After appropriate preservation, most samples can be filtered through a 0.45- $\mu$ m filter and directly injected onto the ion chromatograph. However, each sample that contains excess chlorite must be treated to remove chlorite and then reanalyzed for bromate, because

elevated levels of chlorite can interfere with the quantification of bromate by PCR.

The treatment procedure to remove chlorite requires two portions of sample. Place two 10-mL aliquots of the sample into separate 20-mL beakers. Fortify one aliquot with bromate at a level approximating the native concentration of bromate in the untreated sample. This laboratory fortified matrix (LFM) will indicate correct performance of the chlorite removal step. Acidify both aliquots with 33  $\mu\text{L}$  of sulfuric acid reagent and confirm the final pH (5–6) with pH test strips. Add 40  $\mu\text{L}$  of ferrous iron solution, mix, and allow to react for 10 min. Filter the treated samples through a 0.45- $\mu\text{m}$  nylon filter to remove precipitated ferric hydroxide, and then pass the solution through a hydronium form cation-exchange cartridge (Dionex OnGuard<sup>®</sup>-H, P/N 39596) to remove excess soluble iron. The treated samples must be analyzed within 30 h.

### SYSTEM PREPARATION AND SET-UP

Configure the IC with the PCR system as depicted in Figure 1. Determine the PCR flow rate by collecting the combined effluent from the IC pump and the PCR module in a 10-mL graduated cylinder for 1 min. The PCR flow rate is the difference between the total flow rate and that of the IC pump. Adjust the air pressure of the postcolumn delivery module (PC10) and remeasure the flow rate until the correct flow rate of 0.7 mL/min is established. Confirm this flow rate on a weekly basis or whenever detector response for a calibration check standard deviates beyond quality control acceptance criteria.

To determine target anions at trace concentrations, it is essential to have low baseline noise. Minimize baseline

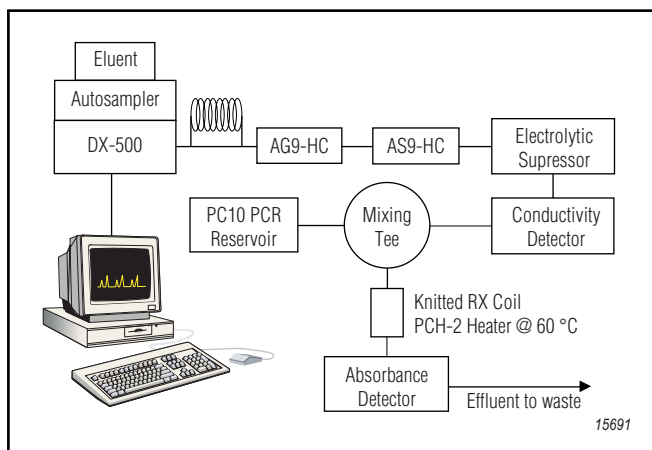


Figure 1. IC system configuration for EPA Method 317.0.

noise by taking the following steps during system set-up. Install the ASRS-ULTRA in the external water mode rather than the recycle mode. Prior to sample analysis, determine a system blank by analyzing 225  $\mu\text{L}$  of deionized water using the method described above. An equilibrated system has a background conductance of  $\sim 24 \mu\text{S}$ , peak-to-peak noise of  $\sim 5 \text{ nS}$  per minute, and no peaks eluting at the same retention time as the anions of interest.

### RESULTS AND DISCUSSION

Figure 2 shows the chromatograms of a mixed anion standard containing 10  $\mu\text{g/L}$  bromate and 15  $\mu\text{g/L}$  each of chlorite, bromide, and chlorate obtained by using dual A) suppressed conductivity and B) UV/Vis absorbance after postcolumn reaction with ODA. The bromate peak is baseline-resolved from chlorite on both detector channels; however, it shows a significantly enhanced

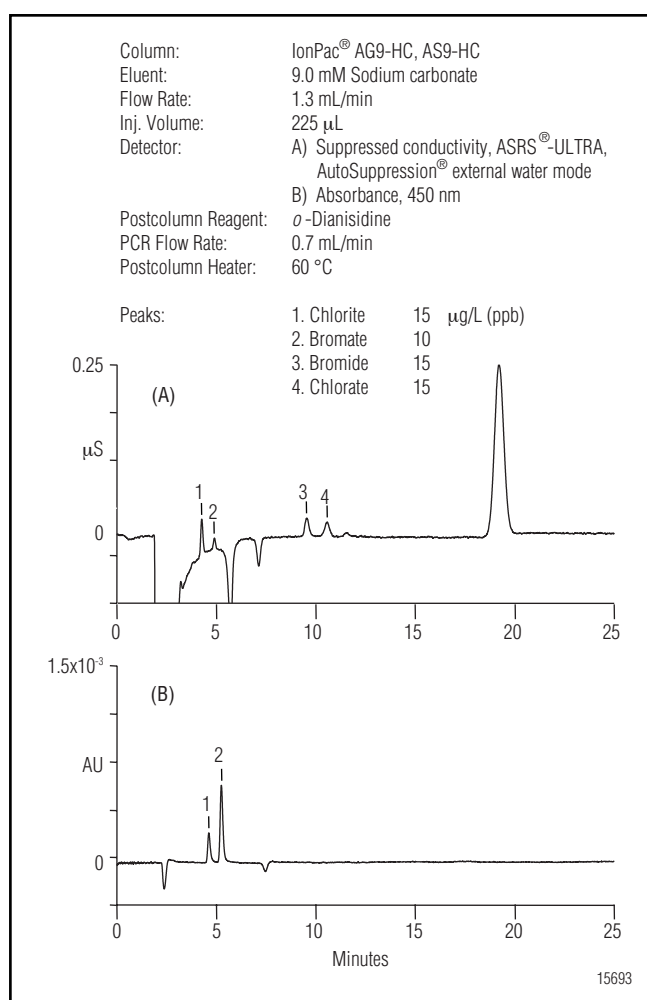


Figure 2. Separation of a low-ppb DBP anion standard using an IonPac AS9-HC column: A) suppressed conductivity detection and B) visible absorbance detection after PCR with *o*-dianisidine.

response on the absorbance detector after PCR with ODA compared to the response obtained on the conductivity detector.

Table 2 summarizes the calibration data and method detection limits (MDLs) obtained for the oxyhalide DBP anions and bromide using dual conductivity and PCR detection. The MDL for each analyte was established by making seven replicate injections of a reagent water blank fortified at a concentration of 3 to 5 times the estimated instrument detection limit.<sup>2</sup> The use of PCR addition and UV/Vis detection allows quantification of bromate down to 0.5 µg/L without compromising detection limits obtained with suppressed conductivity detection for the other anions of interest.<sup>6</sup> Note that the use of electronic smoothing (Olympic, 25 points, 5 sec, 1 iteration) of the UV/Vis signal improves the calculated MDL for bromate.<sup>2</sup> Figure 3 demonstrates the effect of smoothing on the performance of the PCR detection for a 1.0 µg/L bromate standard. No significant loss of peak response is observed after smoothing, although baseline noise is reduced by a factor of approximately 2x, which results in a similar improvement in the detection limit (Table 2).

**Table 2 Linear Ranges and MDLs for Oxyhalides and Bromide**

Solute	Range (µg/L)	r <sup>2</sup>	MDL Standard (µg/L)	Calculated MDL* (µg/L)
Chlorite	5.0–500	0.9999	5.0	1.80
Bromate–conductivity	5.0–50	0.9986	5.0	1.22
Bromide	5.0–500	0.9999	5.0	1.90
Chlorate	5.0–500	0.9999	5.0	1.85
Bromate–UV/Vis (smoothed)	0.5–15	0.9986	1.0	0.09
Bromate–UV/Vis (no smoothing)	0.5–15	0.9986	1.0	0.19

\*MDL = (t) x (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 of the MDL standard], and S = standard deviation of the replicate analysis.

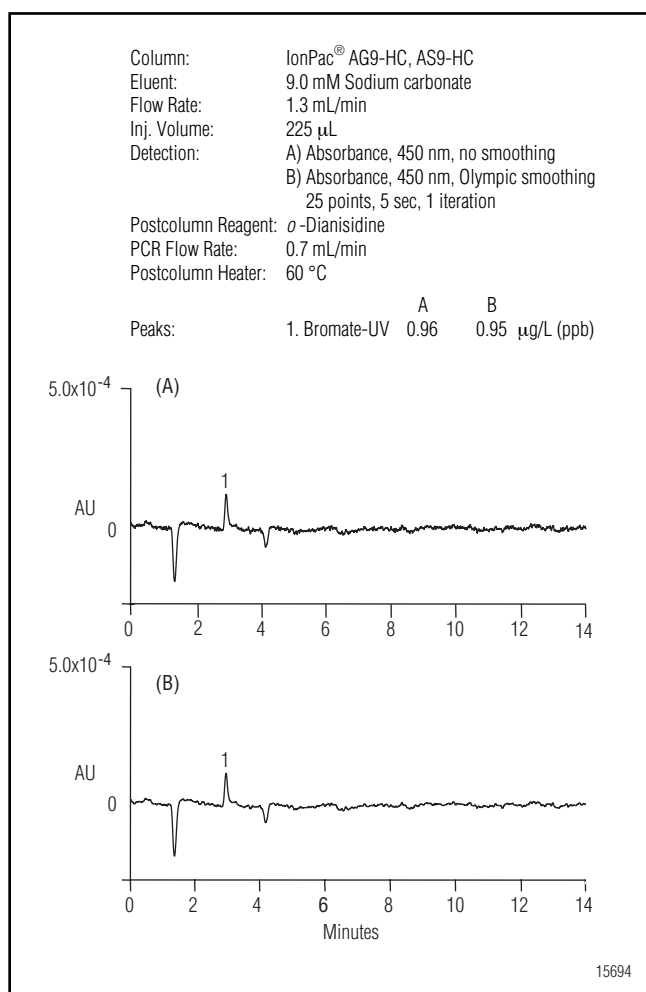


Figure 3. Effect of smoothing on bromate determination: A) unsmoothed data and B) smoothed data (Olympic, 25 points, 5 sec, 1 iteration).

Figures 4–7 illustrate the performance of the method for the determination of inorganic oxyhalide DBP anions and bromide in drinking and bottled water samples. Figure 4 shows the chromatograms from a direct injection of drinking water (from Sunnyvale, California) obtained by using dual A) suppressed conductivity and B) UV/Vis absorbance after postcolumn reaction with ODA. Neither chlorite nor bromate are observed in the drinking water sample; however, bromide and chlorate (frequently observed as a disinfection byproduct from the use of hypochlorite) are well resolved from the sample matrix.

Figure 5 shows the chromatograms of the same drinking water sample spiked with chlorite, bromate, bromide, and chlorate at levels of 108, 11.3, 36, and

72  $\mu\text{g/L}$ , respectively. The chromatograms were obtained using, in series, dual A) suppressed conductivity and B) UV/Vis absorbance after postcolumn reaction with ODA. Quantitative recoveries were obtained for all anions, as shown in Table 3. The benefits of PCR with UV/Vis detection for bromate determination can clearly be seen in Figure 5B: bromate peak response is significantly enhanced compared to the response on the conductivity detector and no response is observed for the large peak from about 20  $\mu\text{g/L}$  chloride that elutes immediately after bromate. The use of PCR with UV/Vis detection allows the quantification of bromate down to 0.5  $\mu\text{g/L}$  in the presence of 200 mg/L chloride (a 400,000-fold excess) with no sample pretreatment.

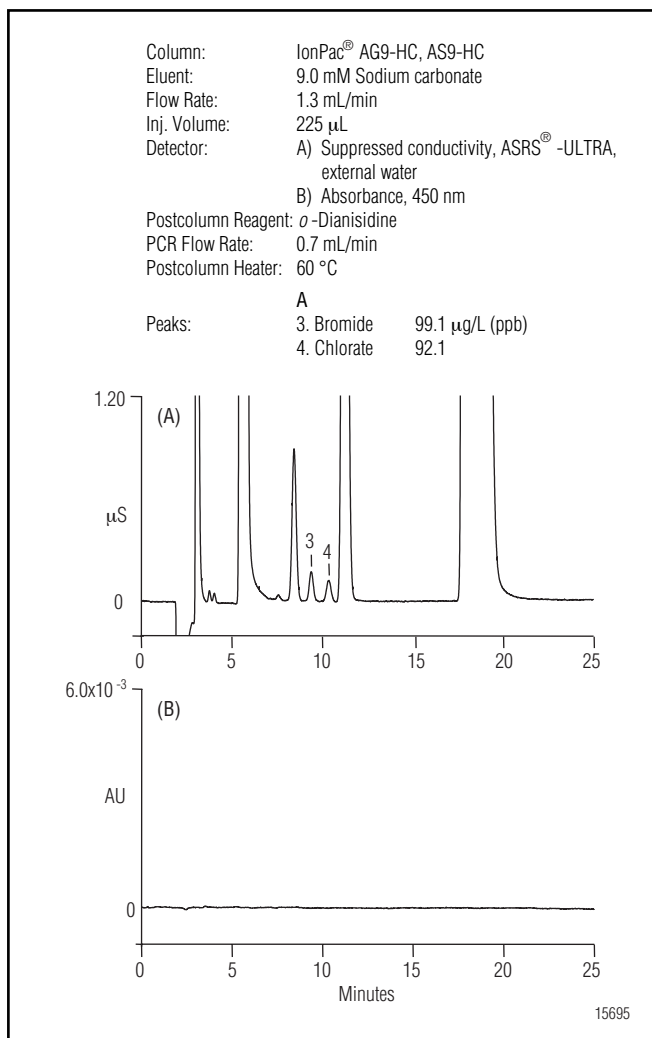


Figure 4. Determination of DBP anions in tap water: A) suppressed conductivity detection and B) visible absorbance detection after PCR with *o*-dianisidine.

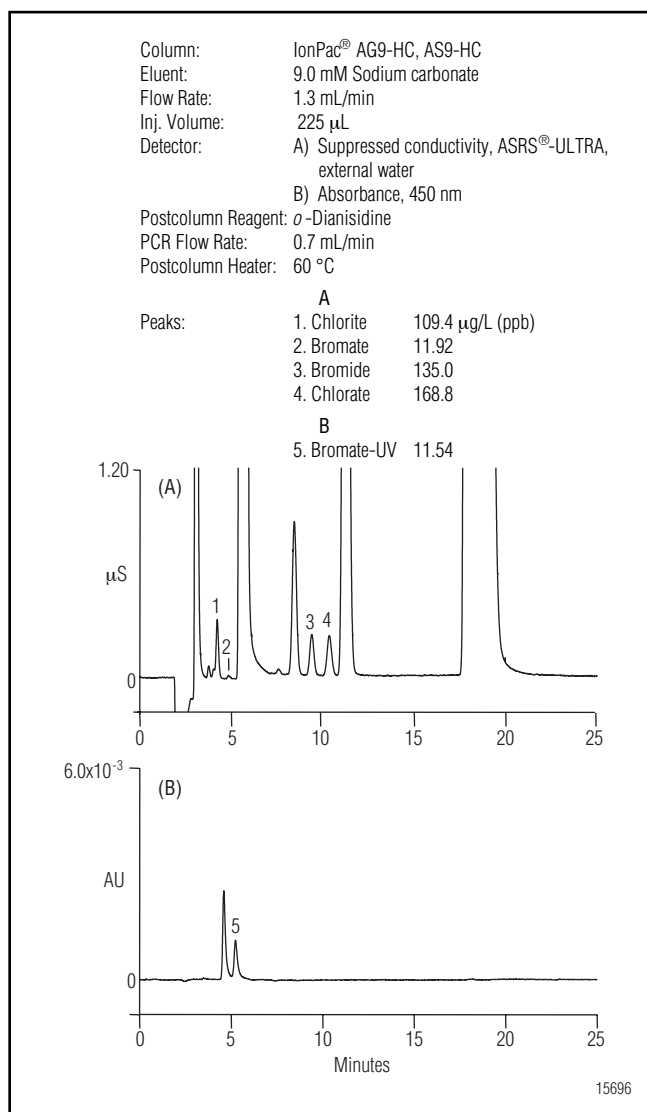


Figure 5. Determination of DBP anions in spiked tap water: A) suppressed conductivity detection and B) visible absorbance detection after PCR with *o*-dianisidine.

Figure 6 shows the chromatograms from a direct injection of bottled spring water obtained using, in series, dual A) suppressed conductivity and B) UV/Vis absorbance after postcolumn reaction with ODA. In this instance, both bromate and bromide are observed in the bottled water sample. Bromate, which is formed during ozonation of source water containing bromide, is present at about 2 µg/L and can clearly be seen in the UV/Vis chromatogram, although no peak is evident on the conductivity detector. Figure 7 shows the chromatograms of the same bottled water sample spiked with chlorite, bromate, bromide, and chlorate at levels of 126, 13.2, 42, and 84 µg/L, respectively. These chromato-

grams were obtained by using, in series, dual A) suppressed conductivity and B) UV/Vis absorbance after postcolumn reaction with ODA. Table 3 shows that quantitative recoveries were again obtained for all anions. Table 3 also shows the recoveries obtained for bromate spiked into the same drinking and bottled water samples at a lower concentration of 2.2 µg/L when using UV/Vis absorbance after postcolumn reaction with ODA. This method permits quantitative recoveries (80–120%) for bromate at levels down to 1 µg/L when using PCR and UV/Vis detection.

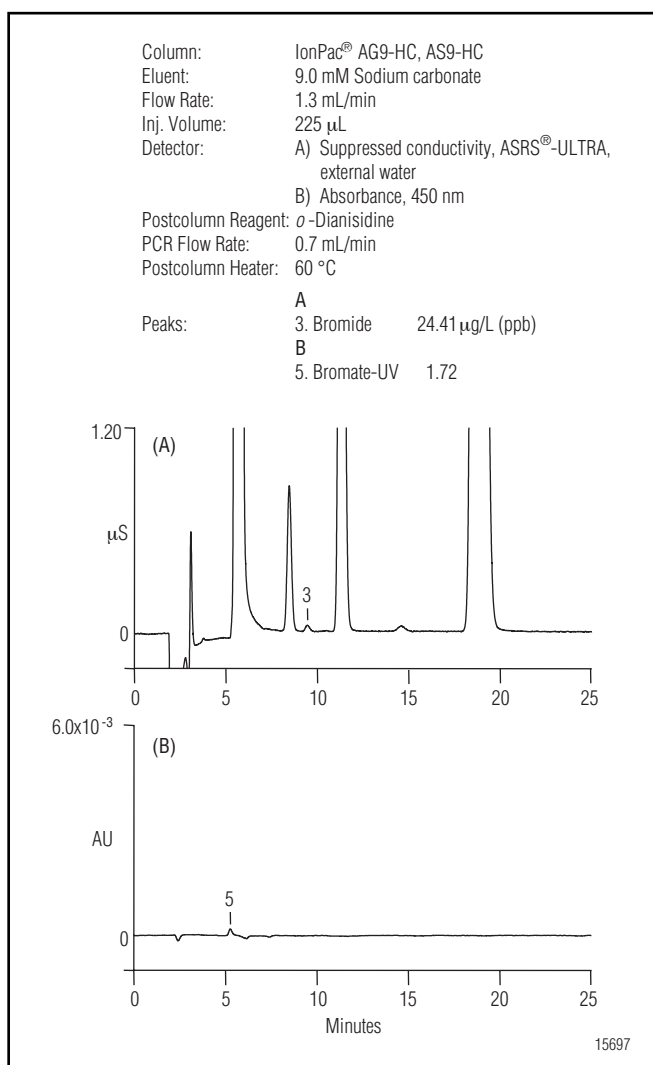


Figure 6. Determination of DBP anions in bottled water: A) suppressed conductivity detection and B) visible absorbance detection after PCR with *o*-dianisidine.

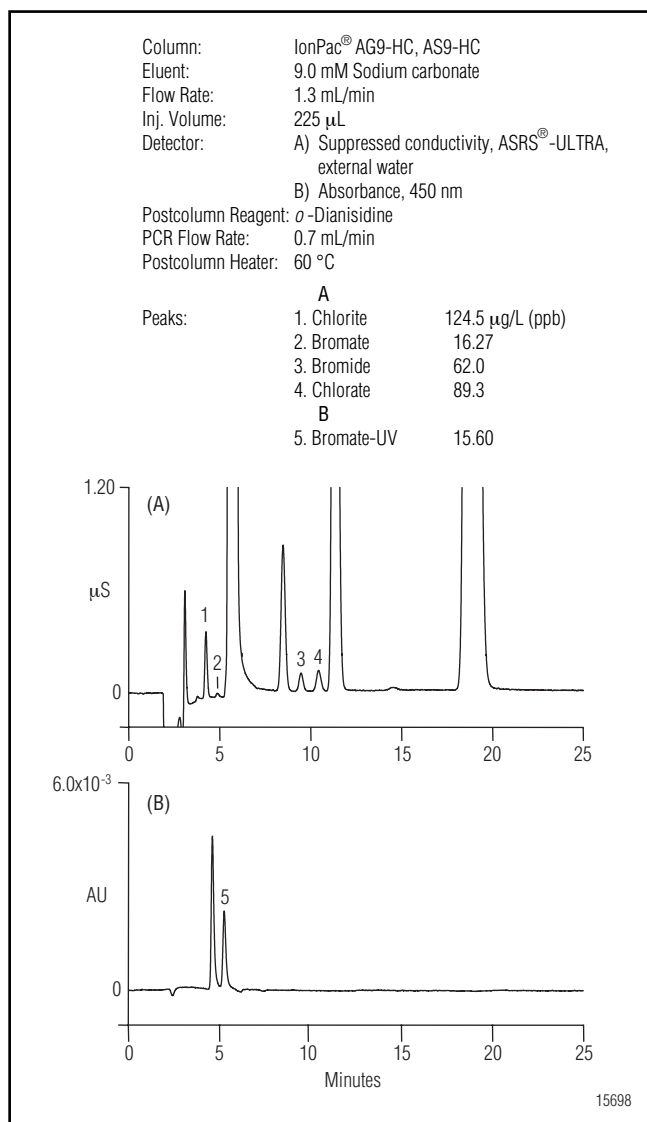


Figure 7. Determination of DBP anions in spiked bottled water: A) suppressed conductivity detection and B) visible absorbance detection after PCR with *o*-dianisidine.

**Table 3 Anion Recoveries for Spiked Water Samples**

Anion*	Tap Water		Bottled Water	
	Amount Added (µg/L)	Recovery	Amount Added (µg/L)	Recovery
Chlorite	108	104%	126	104%
Bromate–conductivity	11.3	105%	13.2	105%
Bromide	36.0	100%	42	100%
Chlorate	72	107%	84	107%
Bromate–UV/Vis	11.3	102%	13.2	102%
Bromate–UV/Vis**	2.2	91%	2.2	96%

\*Data were obtained from multi-analyte spikes into tap and bottled water samples.

\*\*Bromate only (2.2 µg/L) was added to tap and bottled water samples to determine low level recovery for this anion using UV/Vis detection.

### REMOVAL OF CHLORITE INTERFERENCE

When chlorine dioxide is used to disinfect drinking water, the DBP anion chlorite is found in the finished drinking water. Chlorite, like bromate, reacts with *o*-dianisidine to form a complex that absorbs at 450 nm. High chlorite levels can interfere with quantification of low-level bromate.<sup>2</sup> One approach to minimize the interference from chlorite is to remove the chlorite by reduction with ferrous sulfate, as described in the “Sample Preparation” section. This treatment was evaluated by applying it to a series of simulated chlorine dioxide-treated tap waters, which had been spiked with varying levels of bromate, and the corresponding LFMs. The results, summarized in Table 4, show that acceptable recoveries of bromate are obtained after such treatment. This treatment approach is recommended when analysis of low-level bromate is required in chlorine dioxide-treated drinking waters.

**Table 4 Bromate Recovery from Simulated Chlorine Dioxide Treated Waters (STW)\***

	Spiked STW Fe (II)-Treated		Laboratory Fortified Matrix Fe (II)-Treated	
	Amount Added (µg/L)	Recovery	Amount Added (µg/L)	Recovery
STW	0	ND	2.0	90%
STW-1	1.75	74%	2.0	78%
STW-2	2.15	80%	4.0	75%
STW-3	4.61	76%	6.0	82%
STW-4	5.14	80%	8.0	75%

\* Chlorite present at 100 µg/L

### SUMMARY

The IC method described in this Application Note, which uses an IonPac AS9-HC column and suppressed conductivity detection, followed by postcolumn addition of *o*-dianisidine with UV/Vis detection specifically to enhance bromate response, allows the determination of all the key oxyhalide anions and bromide at low µg/L levels in drinking and bottled waters. The use of postcolumn addition and UV/Vis detection allows the quantification of bromate in the range of 0.5-15 µg/L without compromising the suppressed conductivity detection of chlorite, bromide, and chlorate. Conductivity detection is recommended for the quantification of bromate in the range of 15–50 µg/L.



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## SUPPLIERS

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