

Improved Determination of Trace Concentrations of Oxyhalides and Bromide in Drinking Water Using a Hydroxide-Selective Column

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Key Words

Dionex IonPac AS27 Column, Dionex AERS 500 Anion Electrolytically Regenerated Suppressor, Bromate, Chlorite, Chlorate, Disinfection Byproducts, Dichloroacetate, EPA Method 300.1

Goal

To demonstrate use of the Thermo Scientific™ Dionex™ IonPac™ AS27 column to separate bromate and other oxyhalides in environmental waters containing the ethylenediamine (EDA) preservative

Introduction

Disinfection treatment is used to protect municipal water from potentially dangerous microbes. The most common chemical disinfectants are chlorine, chlorine dioxide, chloramine, and ozone.^{1,2} These chemical disinfectants can react with natural organic and inorganic matter in the source water to produce disinfection byproducts (DBPs) that are potentially harmful to humans. For example, chlorine dioxide treatment generates the inorganic oxyhalide DBPs chlorite and chlorate, whereas ozone reacts with naturally occurring bromide to produce bromate.² In addition, bromate can also be formed when chlorinated water is exposed to the UV rays in sunlight.³

Of these DBPs, bromate has received the most attention due to its potential negative health effects. The International Agency for Research on Cancer has identified bromate as an animal carcinogen and a potential human carcinogen.⁴ Therefore, when conditions exist that can cause bromate formation (i.e., ozonation and photochemical reaction), the finished product must be tested to ensure that bromate levels are safe for human consumption.



The World Health Organization (WHO) has estimated an excess lifetime cancer risk of 10^{-4} , 10^{-5} , and 10^{-6} for drinking water containing bromate at levels of 20, 2, and 0.2 µg/L, respectively, and has suggested a guideline level of 10 µg/L bromate in drinking water.⁵ The U.S. Environmental Protection Agency (EPA); European Commission; and Japanese Ministry of Health, Labor, and Welfare have established a regulatory maximum contaminant level (MCL) of 10 µg/L bromate in municipal drinking water.⁶⁻⁸ The limits of detection required by the European Commission for bromate determination are 25% of the MCL, or 2.5 µg/L, for drinking water. In addition, the European Commission established a lower MCL of 3 µg/L bromate in natural mineral water and spring water treated by ozonation.⁹

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The determination of trace concentrations of bromate and other oxyhalides in high-ionic-strength matrices can be very challenging. U.S. EPA Methods 300.1 Part B, 317.0, and 326.0 have been the standard approaches for determining DBPs in drinking water using the Dionex IonPac AS9-HC column with a carbonate eluent.¹⁰⁻¹² The Dionex IonPac AS19 column was developed for use with hydroxide eluents to improve the sensitivity of bromate using a direct injection; this method is described in Thermo Scientific Application Note 167.¹³ Besides providing superior sensitivity by suppressed conductivity detection, hydroxide eluents can be easily generated with Reagent-Free™ ion chromatography (IC) (RFIC™) systems. These systems simplify analysis and improve reproducibility when transferring methods between laboratories.

The Dionex IonPac AS19 column can also replace the Dionex IonPac AS9-HC column in EPA Methods 317.0 and 326.0, as described in Thermo Scientific Application Notes 168 and 171.^{14,15} To determine sub-µg/L bromate in high-ionic-strength matrices without postcolumn derivatization, a two-dimensional IC method was developed (EPA Method 302.0). This method uses the Dionex IonPac AS19 column in the first dimension and the Dionex IonPac AS24 column in the second dimension, followed by suppressed conductivity detection.¹⁶

Column Selection

This study demonstrates the Dionex IonPac AS27 high-capacity, hydroxide-selective, anion-exchange column for determination of trace concentrations of chlorite, bromate, chlorate, and bromide in drinking water samples. The Dionex IonPac AS27 column was specifically developed for this application as an alternative to the Dionex AS19 column. The Dionex IonPac AS27 offers several key advantages over the Dionex AS19 column, including the ability to determine trace bromate in the presence of the EDA preservative, good resolution of dichloroacetate (DCA, a surrogate anion) from potentially interfering matrix anions, and improved resolution between bromate and chloride. In addition, with the Dionex IonPac AS27 column, the resolution between bromate and chloride is more than double that achieved using the Dionex IonPac AS19 column. Therefore, the quantification of bromate in samples containing high concentrations of chloride is significantly improved when using the Dionex IonPac AS27 column.

EDA Preservative

The EDA preservative is required in all standards and samples to preserve the integrity of chlorite and bromate. Chlorite is susceptible to degradation through catalytic reactions with dissolved iron, but EDA prevents this reaction by chelating iron and other destructive metal cations. EDA also preserves bromate by binding with hypobromous acid/hypobromite, which is an intermediate formed as a byproduct of the reaction of either ozone or hypochlorous acid/hypochlorite with bromide.¹⁰

Equipment, Software, and Consumables

- A Thermo Scientific™ Dionex™ ICS-5000+ HPIC™ system,* capable of supporting high-pressure IC, including:
 - SP Single Pump
 - EG Eluent Generator
 - DC Detector/Chromatography Compartment
- Thermo Scientific Dionex AS-AP Autosampler, with 1000 µL sample syringe (P/N 074307) and 1200 µL Buffer Line Assembly (P/N 074989) with 250 µL injection loop
- Thermo Scientific Dionex EGC 500 Potassium Hydroxide (KOH) Eluent Generator Cartridge (P/N 075778)
- Thermo Scientific Dionex CR-ATC 500 Continuously Regenerated Anion Trap Column (P/N 075550)
- Thermo Scientific™ Dionex™ AERS™ 500 Anion Electrolytically Regenerated Suppressor (P/N 082540)
- Vial Kit, 10 mL, Polystyrene with Caps and Blue Septa (P/N 074228)
- Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System software, version 7.2

* This application also can be performed on any Dionex ICS system capable of eluent generation.

Reagents and Standards

- Deionized (DI) water, Type I reagent grade, 18 MΩ-cm resistivity or better
- Sodium Chlorite, 80% (Fisher Scientific P/N AC22323-5000)
- Sodium Bromate, 99.7–100.3% (Fisher Scientific P/N S253-250)
- Sodium Chlorate, 99% (Fisher Scientific P/N AC44641-1000)
- Sodium Bromide, ≥99% (Fisher Scientific P/N S255-500)
- Ethylenediamine, 99+% (Fisher Scientific P/N AC22042-2500)
- Sodium Dichloroacetate, 98% (Fisher Scientific P/N AC338280100)
- Sodium Nitrate, ≥99% (Fisher Scientific P/N S343-500)
- Potassium Phosphate Monobasic, ≥99% (Fisher Scientific P/N P285-500)
- Sodium Carbonate Anhydrous, ≥99.5% (Fisher Scientific P/N S263-500)
- Thermo Scientific Dionex Fluoride Standard, 1000 mg/L, (Fisher Scientific P/N DX037158)
- Thermo Scientific Dionex Chloride Standard, 1000 mg/L (Fisher Scientific P/N DX037159)
- Nitrite Standard, 1000 mg/L (Fisher Scientific P/N USICC007)
- Nitrate Standard, 1000 mg/L NO₃ (Fisher Scientific P/N 53-074)
- Thermo Scientific Dionex Sulfate Standard, 1000 mg/L (Fisher Scientific P/N DX037160)

Conditions (Applicable to Figures 2–5)

Columns:	Dionex IonPac AG27 Guard, 4 × 50 mm (P/N 088438) Dionex IonPac AS27 Analytical, 4 × 250 mm (P/N 088437)
Eluent:	0–10 min, 12 mM KOH; 10–12 min, 12–20 mM; 12–20 min, 20 mM; step to 60 mM at 20 min; 20–29 min, 60 mM; step to 12 mM at 29 min; 29–30 min, 12 mM*
Eluent Source:	Dionex EGC 500 Eluent Generator Cartridge with Dionex CR-ATC 500 Continuously Regenerated Anion Trap Column
Flow Rate:	1.0 mL/min
Inj. Volume:	250 µL
Temperature:	30 °C
Detection:	Suppressed conductivity, Dionex AERS 500 suppressor, 4 mm, recycle mode, 149 mA current
Background Conductance:	<0.5 µS
Noise:	~0.5 nS/min, peak-to-peak
System Backpressure:	~2500 psi
Run Time:	30 min

* This method equilibrates at 12 mM KOH for 7 min prior to injection.

Preparation of Solutions and Reagents

Stock Standard Solutions

For several of the anions of interest, 1000 mg/L standard solutions can be purchased from Fisher Scientific or other commercial sources. When commercial standards are not available, prepare 1000 mg/L standards by dissolving the appropriate amounts of the required analytes in 100 mL of DI water as specified in Table 1. Stock standards for most anions are stable for at least 6 months when stored at 4 °C. The chlorite standard is only stable for 2 weeks when stored protected from light at 4 °C. The nitrite and phosphate standards are only stable for 1 month when stored at 4 °C.

DCA Stock Standard Solution

To prepare 500 mg/L of DCA, dissolve 0.059 g of sodium DCA in 80 mL of DI water in a 100 mL volumetric flask. Fill to the mark with DI water, cap, and mix thoroughly by manual inversion. Transfer the solution to a polypropylene bottle where it will be stable for 3 months at 4 °C.

Table 1. Masses of compounds used to prepare 100 mL of 1000 mg/L anion standards.

Analyte	Compound	Amount (g)
Fluoride	Sodium Fluoride (NaF)	0.2210
Chlorite	Sodium Chlorite (NaClO ₂), 80%	0.1676
Bromate	Sodium Bromate (NaBrO ₃)	0.1180
Chloride	Sodium Chloride (NaCl)	0.1649
Nitrite	Sodium Nitrite (NaNO ₂)	0.1500
Chlorate	Sodium Chlorate (NaClO ₃)	0.1275
Bromide	Sodium Bromide (NaBr)	0.1288
Nitrate	Sodium Nitrate (NaNO ₃) ¹	0.1371
Carbonate	Sodium Carbonate (Na ₂ CO ₃)	0.1766
Sulfate	Sodium Sulfate (Na ₂ SO ₄)	0.1479
Phosphate	Potassium Phosphate, Monobasic (KH ₂ PO ₄) ²	0.1433

¹To prepare nitrate as nitrogen, use 0.607 g/100 mL of sodium nitrate for a 1000 mg/L standard.

²To prepare phosphate as phosphorus, use 0.439 g/100 mL of potassium phosphate monobasic for a 1000 mg/L standard.

Working Standard Solutions

Prepare dilute working standard solutions using the 1000 mg/L stock standards. Prepare working standards containing <100 µg/L anions fresh daily. Use five levels of calibration standards for chlorite and bromate, but use six levels for chlorate and bromide to cover the expected concentration ranges found in drinking water samples. Use additional anions listed in Table 1 to prepare the high-ionic-strength water (HIW) listed in Footnote 2 of Table 1B in EPA Method 300.1. The HIW sample must contain 100 mg/L chloride, 10 mg/L nitrate as nitrogen, 100 mg/L carbonate, 100 mg/L sulfate, and 10 mg/L phosphate as phosphorus. Fortify all standards and simulated HIW with 1 mg/L DCA.

Preservation Solution

As specified in section 7.4 of EPA Method 300.1, dilute 2.8 mL of 99% EDA to 25 mL with DI water to prepare a 100 mg/mL solution of EDA. Store this solution at 4 °C when not in use and prepare fresh monthly. Use 50 µL of this solution per 100 mL of standard or sample so the final concentration will be 50 mg/L.

Sample Preparation

Filter samples, as necessary, through a 0.45 µm syringe filter and discard the first 300 µL of the effluent. To prevent degradation of chlorite or formation of bromate from hypobromous acid/hypobromite, preserve the samples by adding 50 µL of EDA preservation solution per 100 mL of sample. All samples require a final fortified concentration of 1 mg/L DCA, so add 0.2 mL of 500 mg/L DCA stock solution per 100 mL of sample. The recovery of DCA must fall between 90 and 115%.

Results and Discussion

The Dionex IonPac AS27 column is a high-capacity, hydroxide-selective, anion-exchange column specifically developed for the separation of trace concentrations of bromate and other oxyhalides from common inorganic anions in environmental samples. The selectivity of the Dionex IonPac AS27 column has been optimized to allow quantification of low concentrations of bromate in the presence of the 50 mg/L EDA preservative, which is required for EPA Method 300.1(B). The EDA preservative can react with carbonate and produce artifacts that interfere with early eluting analytes, such as chlorite and bromate. In comparison to the Dionex IonPac AS19 column described in Thermo Scientific Application Note 167, the Dionex IonPac AS27 column has similar selectivity and capacity, but offers better resolution of bromate from artifacts produced by the EDA preservative, better resolution between bromate and chloride, and improved selectivity for the DCA surrogate.

When installing a new column, Thermo Scientific strongly recommends reproducing the quality assurance report (QAR) that is included in the column packaging. Figure 1 shows a QAR separation of six common inorganic anions, plus chlorite, bromate, and chlorate, using an isocratic 20 mM KOH eluent. As shown, all target analytes are well resolved using isocratic eluent conditions.

The separation was then optimized using a multistep hydroxide gradient to resolve chlorite, bromate, chlorate, bromide, and DCA from common inorganic anions found in drinking water samples. The optimized separation developed for this application (Figure 2) shows that the Dionex IonPac AS27 column provides good resolution of the DBP anions, bromide, and DCA from common anions within 30 min using a hydroxide gradient.

Linearity and Method Detection Limits (MDLs)

Before analyzing samples, the linear calibration range, MDLs, and acceptable performance of a quality control sample (QCS) were demonstrated. A five-point calibration range was used for chlorite and bromate, whereas a six-point calibration range was used for chlorate and bromide. The MDLs for chlorite, bromate, chlorate, and bromide were determined by performing seven replicate injections of reagent water fortified at a concentration of 3× to 5× the estimated instrument detection limits.

Table 2 shows typical calculated MDLs in reagent water using the Dionex IonPac AS27 column combined with an electrolytic eluent generator and a 250 µL injection. Using this method, the calculated MDL for bromate was 0.12 µg/L—more than 60% lower than previously reported in AN 167 using the Dionex IonPac AS19 column. In addition, the MDLs for other DBP anions and bromide were also significantly lower than previously reported using the Dionex IonPac AS19 column. The lower detection limits are attributed to the lower baseline noise using the Dionex AERS 500 suppressor relative to the Thermo Scientific™ Dionex™ ASRS™ ULTRA II Anion Self-Regenerating Suppressor used in Application Note 167.

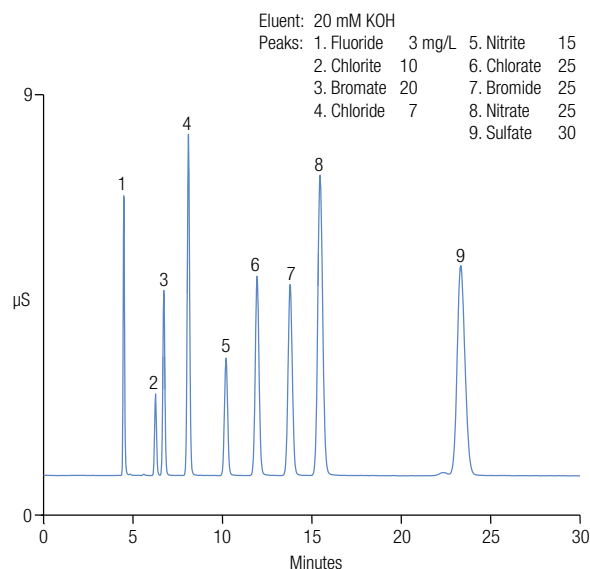


Figure 1. Dionex IonPac AS27 column QAR separation using an isocratic KOH eluent.

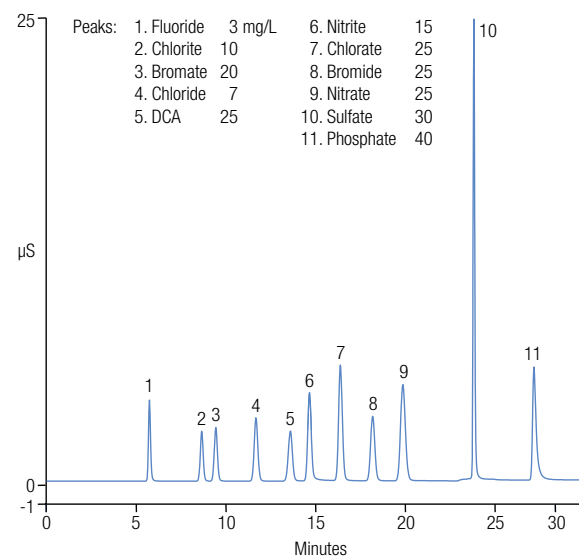


Figure 2. Separation of common anions, DBPs, and DCA using the Dionex IonPac AS27 column with a hydroxide gradient.

Table 2. Linearity and detection limits obtained using the Dionex IonPac AS27 column.

Analyte	Range (µg/L)	Linearity (r ²)	MDL Standard (µg/L)	Calculated MDL ^b (µg/L)
Chlorite	1–100	0.9993	0.5	0.10
Bromate	1.0–25	0.9990	0.5	0.12
Chlorate	10–500	0.9998	0.5	0.14
Bromide	10–500	0.9992	1.0	0.21

^a 250 µL injection volume.

^b MDL = $\sigma t_{s,99}$ where $t_{s,99} = 3.14$ for $n = 7$

Table 3. Recoveries of trace oxyhalides and bromide spiked into environmental waters.

Analyte	Drinking Water A			Drinking Water B			Drinking Water C			HIW		
	Amount Found (µg/L)	Amount Added (µg/L)	Recovery (%)	Amount Found (µg/L)	Amount Added (µg/L)	Recovery (%)	Amount Found (µg/L)	Amount Added (µg/L)	Recovery (%)	Amount Found (µg/L)	Amount Added (µg/L)	Recovery (%)
Chlorite	4.1	10.0	91.6	1.8	5.0	87.9	<MDL	10.0	82.9	<MDL	10.3	87.9
Bromate	0.98	1.0	92.6	0.5	1.0	104.1	<MDL	1.1	103.9	<MDL	1.1	108.2
DCA	—	985	99.9	—	983	99.3	—	984	97.4	—	984	96.4
Chlorate	92.5	100	97.1	169	165	101.5	73.7	80	92.9	<MDL	54.4	93.6
Bromide	85.0	97.6	98.1	160	165	100	127.0	151	97.1	10.2	53.1	91.2

As part of the initial demonstration of performance in accordance with Section 9.2.2 in EPA Method 300.1, the laboratory must analyze a QCS to verify the calibration and assess acceptable instrument performance. A QCS consisting of 50 µg/L each of chlorite, chlorate, and bromide; 5 µg/L bromate; and 1000 µg/L DCA was prepared and seven replicate injections were performed. The average recoveries from these injections ranged from 92.6 to 99.4%, which is well within the $\pm 15\%$ specification in EPA Method 300.1. The retention time and peak area precisions were $\leq 0.02\%$ and $\leq 1.23\%$, respectively. The recoveries and precisions demonstrate good initial performance of the method for the determination of oxyhalides and bromide.

Accuracy and Precision

The performance of the Dionex IonPac AS27 column was also evaluated through a single-operator precision and bias study using spiked municipal water samples. Tap water samples were obtained from three different cities located within the San Francisco Bay Area. In addition, a synthetic HIW sample was also included to evaluate the method performance in a high-ionic-strength matrix.

Table 3 summarizes the recoveries of the DBP anions, bromide, and DCA spiked into four different matrices, then separated on the Dionex IonPac AS27 column. Overall, the recoveries ranged from 82.9 to 108.2%, which is within the acceptable range according to the criteria outlined in EPA Method 300.1. All samples were fortified with 1 mg/L DCA surrogate, for which the EPA has specified a recovery range between 90 and 115%. As shown in Table 3, the recoveries of DCA were well within this specification, with recoveries ranging from 96.4 to 99.9%. For bromate, the recoveries ranged from 92.6 to 108.2% after spiking each sample with ~ 1 µg/L; this is well below the MCL of 10 µg/L. These excellent recoveries demonstrate the lack of interference from artifacts caused by adding the EDA preservative to the samples.

Drinking Water C was the highest-ionic-strength sample among the drinking waters investigated in this study. Although this sample produced the lowest recovery of chlorite at 82.9%, the recovery of bromate was 103.9%, even when spiked at a low concentration of 1.1 µg/L. Figures 3A and 3B compare chromatograms of unfortified and fortified Drinking Water C. As shown, all fortified anions are resolved from potential matrix interferences in the sample.

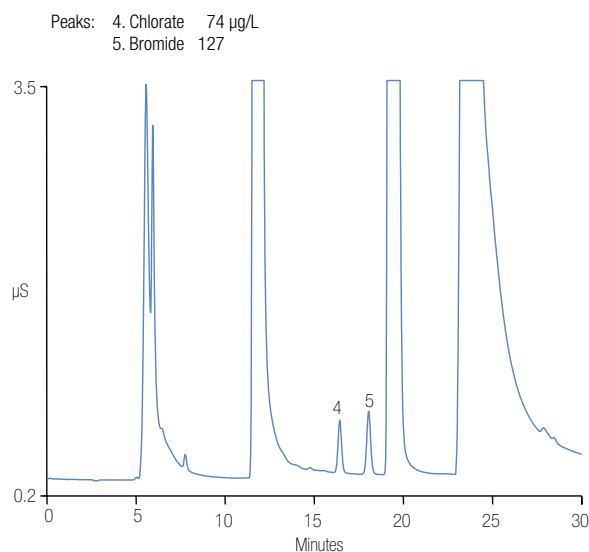


Figure 3A. Separation of DBP anions and bromide in unfortified Drinking Water C.

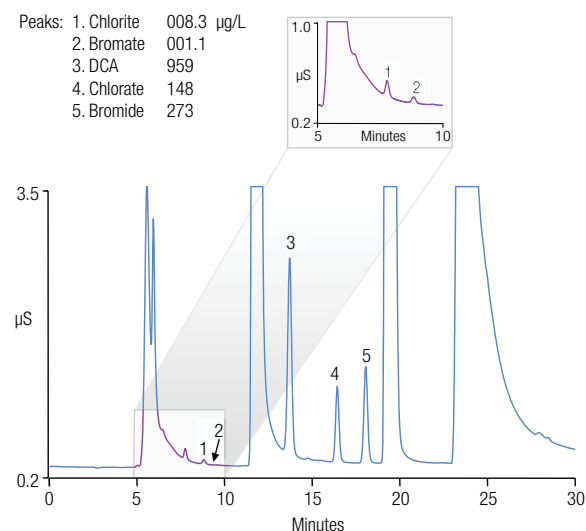


Figure 3B. Drinking Water C fortified with DBP anions, bromide, and DCA. (Fortification concentrations are shown in Table 3).

To demonstrate the applicability of the Dionex IonPac AS27 column to accurately quantify trace DBP anions and bromide in a high-ionic-strength matrix, an HIW sample containing 100 mg/L each of chloride, carbonate, and sulfate; 10 mg/L nitrate-N; and 10 mg/L phosphate-P was analyzed. Figure 4 shows a spiked HIW sample in which recoveries of the fortified anions ranged from 87.9 to 108.2%. The precision of the method using the Dionex IonPac AS27 column in combination with an electrolytic eluent generation was determined from seven replicate injections of the samples spiked with trace concentrations of DBPs and bromide. Overall, the calculated peak area precisions varied from 0.18 to 3.20% for the target analytes. However, the peak area precisions for most of the spiked anions were <1%. The high precision of this method is consistent with results typically encountered when using a RFIC system.

Conclusion

To determine trace concentrations of DBP anions and bromide in municipal drinking water samples by IC, use of the hydroxide-selective Dionex IonPac AS27 column combined with an electrolytic eluent generator is an improvement over the Dionex IonPac AS19 column. Although either column is suitable for this application, the Dionex IonPac AS27 column is preferred when samples contain the EDA preservative and when the DCA surrogate is used in the analysis. This study demonstrates a sensitive and accurate approach for determining trace concentrations of bromate and other DBP anions using a direct sample injection. The data shows good linearity, MDLs, and accuracy, as well as improved resolution between bromate and the artifacts generated when using EDA.

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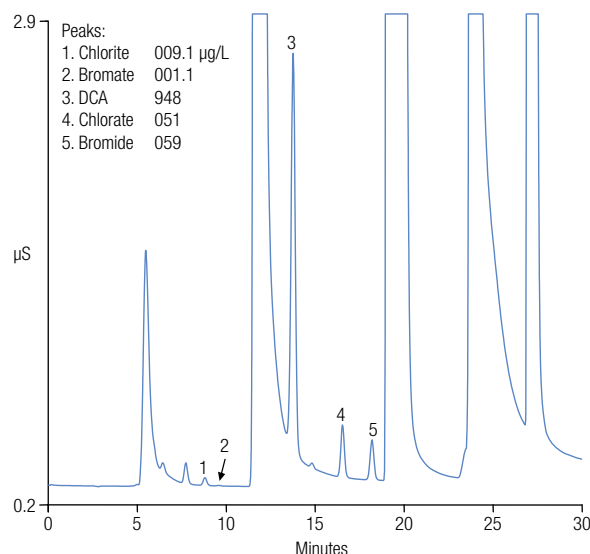


Figure 4. Separation of DBP anions, bromide, and DCA in a fortified HIW sample.

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