



## Environmental ion chromatography

Thermo Scientific application note compendium

## Introduction

## Inorganic anions and cations

## Disinfection byproducts

## Toxic contaminants

**Introduction to environmental water analysis**

Everyone is impacted by the quality of water and by the air we breath. Wastewater treatment plants and industrial facilities must comply with strict discharge limits for environmental safety, while the quality of surface and ground water will dictate a treatment strategy that will ultimately result in safe drinking water. Thermo Fisher Scientific is committed to enhancing the quality of our global water resources. As innovation leaders in ion and liquid chromatography, our analytical instruments are used by governments and industry to provide solutions for environmental water testing for a wide range of regulated and emerging inorganic elements and organic compounds. As pioneers of suppression technology, we started a revolution in ion chromatography (IC) that increased the sensitivity and accuracy of ion determination. As constant innovators, we developed Thermo Scientific™ Reagent-Free™ (RFIC™) IC systems that set a new benchmark for ion analysis. Today, RFIC systems with eluent generation provide the ultimate in sensitivity and ease of use.

**Ground and surface water**

Surface water is the largest source of fresh water used for human consumption. The U.S. Geological Survey implemented the National Water-Quality Assessment (NAWQA) Program in 1991 to develop long-term data on streams, rivers, groundwater, and aquatic systems. The data support national, regional, state, and local policies and decisions related to water-quality management. The NAWQA program is designed to answer the following questions:

- What is the condition of our nation's streams, rivers, and groundwater?
- How are these conditions changing over time?
- How do natural features and human activities affect these conditions, and where are those effects most pronounced?

Thermo Fisher Scientific has codeveloped several methods with the U.S. Environmental Protection Agency (EPA) Office of Ground Water and Drinking Water. This collaboration has strengthened with the development of unique technology, including electrolytic suppression and RFIC systems with eluent generation.





### Drinking and bottled water

Currently, less than 1% of the planet's water is available for human consumption. With surface water contamination and groundwater resources overexploited, the need for effective water analysis and monitoring has never been higher. The Safe Drinking Water Act (SDWA) is a U.S. federal law which sets legal limits on the levels of certain contaminants in drinking water. Under the SDWA, the U.S. EPA enforces the National Primary Drinking Water Regulations (NPDWRS or primary standards) that apply to all public water systems. The NPDWR mandates maximum concentration levels of certain drinking water contaminants, also called "maximum contaminant levels" or "MCLs". The EPA also provides a list of acceptable techniques for treating drinking water to reduce regulated contaminants to acceptably low levels. In Europe, the Drinking Water Directive provides the essential quality standards. These quality standards were developed using guidelines from the World Health Organization (WHO) and the European Commission's Scientific Advisory Committee. Member States of the European Union can add additional requirements for substance regulation; however, they cannot set lower standards for these substances. Drinking water must be reported to the European Commission every three years. Contaminant levels in drinking water are continuously subject to reassessment by the above regulatory bodies, both in regard to revised levels, as well as the addition of new contaminants to the list of existing regulated substances. Thermo Fisher Scientific provides a variety of solutions for inorganic and organic drinking water contaminants.

### Wastewater

Aqueous waste from residences, industry, and agriculture, may contain a wide range of potential contaminants and concentrations. Industries discharge a variety of pollutants in their wastewater, including heavy metals, organic toxins, oils, nutrients, and solids, all of which endanger ecosystems and pose a threat to human health. In some areas, treated wastewater is recycled for irrigation purposes and even as drinking water. This reuse of water is gaining closer scrutiny as demand increases for water resources.

### Air

Monitoring the composition of ambient particulate matter of 2.5  $\mu\text{m}$  or less ( $\text{PM}_{2.5}$ ) plays an important role in air quality management. The chemical composition of  $\text{PM}_{2.5}$  includes a large number of mineral oxides, sulfate, nitrate, polycyclic aromatic hydrocarbons, organic acids, and organic chlorine that directly impact atmospheric quality and influence human health. They can also act as markers for the origins of such pollution, which can contribute to strategies to control the impact of these sources on the environment.

# Determination of inorganic anions in drinking water using a compact ion chromatography system – EPA 300 and 300.1

Introduction

## Inorganic anions and cations

### Inorganic anions in drinking water using IC EPA 300 and 300.1

Fast and low-cost determination of anions in municipal drinking water

Inorganic anions in drinking water, wastewater, and high ionic strength water

Oxyhalides and bromide in municipal and bottled waters using a compact IC system

Trace concentrations of oxyhalides and bromide in municipal and bottled waters

Fast determination of oxyhalides and bromide in drinking water using IC with a single quadrupole mass spectrometer

Improved IC separation of ethylenediamine carbamate and fluoride, and carbonate and sulfate in drinking water

Nitrite and nitrate in wastewater using capillary IC with UV detection

Low-cost determination of inorganic cations and ammonium in environmental waters

Anions and carboxylic acids in urban fine particles (PM<sub>2.5</sub>)

### Disinfection byproducts

### Toxic contaminants

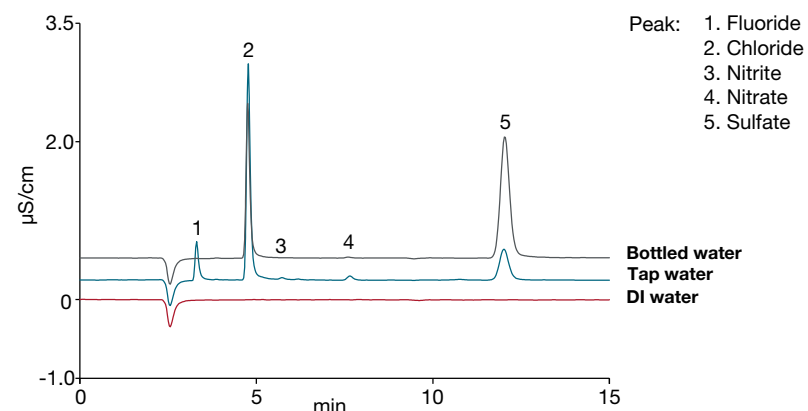


The determination of inorganic anions in municipal drinking, waste, and bottled waters according to the U.S. EPA Methods 300.0 and 300.1 are two of the most popular and widely used IC methods. We used a Thermo Scientific™ Dionex™ Aquion™ ion chromatography system with a Thermo Scientific™ Dionex™ AS-DV autosampler to determine seven anions (fluoride, chloride, bromide, nitrite, nitrate, phosphate, and sulfate) in drinking water samples.

### Concentrations of seven anions in two water samples

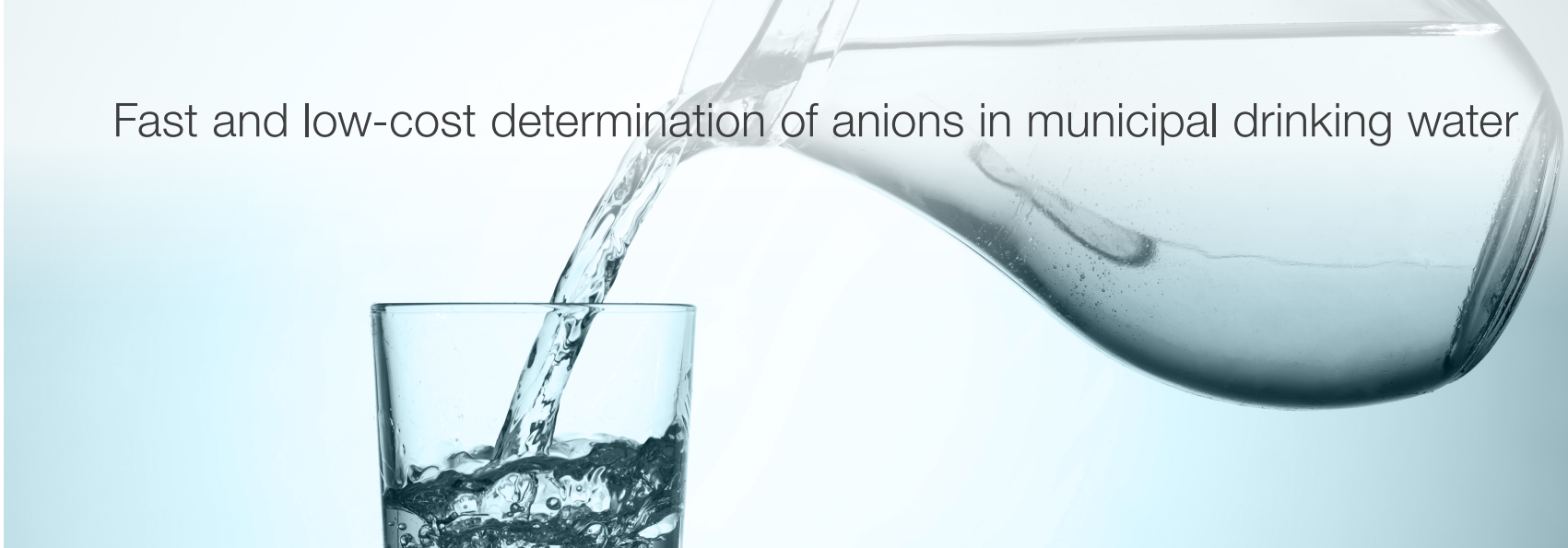
Anion	Tap water		Bottled water	
	mg/L	RSD (n = 3)	mg/L	RSD (n = 3)
Fluoride	0.73	0.09	n.d.	n.d.
Chloride	6.08	0.21	4.39	0.19
Nitrite	0.20	0.20	n.d.	n.d.
Bromide	ND	ND	ND	ND
Nitrate	0.32	0.27	0.08	0.97
Phosphate	0.27	0.55	n.d.	n.d.
Sulfate	3.48	0.3	13.4	0.29

ND = not detected



Chromatograms of deionized water (DI), tap water, and bottled water samples





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Helpful resources and links

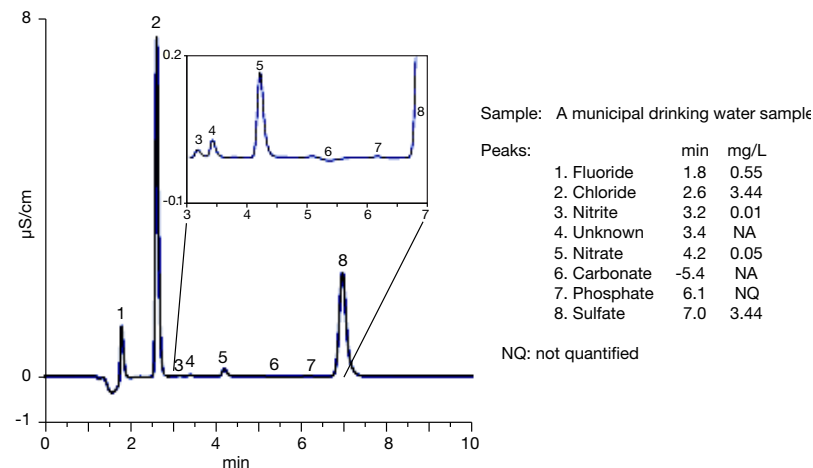
IC is a well-accepted technique for monitoring inorganic anions in water, including surface, ground, drinking, and waste waters. IC methods have been approved by the U.S. EPA for compliance monitoring of inorganic anions in drinking water since the 1980s through U.S. EPA Method 300.0 that was updated in 1997 to U.S. EPA Method 300.1.

**Linearity obtained using the Dionex IonPac AS22-Fast-4µm column with a 25.0 µL injection**

Analyte	Range (mg/L)	Coefficient of determination (r <sup>2</sup> )*
Fluoride	0.1–20	1.000
Chloride	0.2–100	0.999
Nitrite-N	0.1–20	1.000
Bromide	0.1–20	0.999
Nitrate-N	0.1–20	0.996
Phosphate-P	0.2–50	0.995
Sulfate	0.2–100	0.997

\*Calibration type is linear and forced through the origin

This work demonstrates the determination of inorganic anions in drinking water by IC using a Thermo Scientific™ Dionex™ IonPac™ AS22-Fast-4µm column set on a Thermo Scientific™ Dionex™ Easion™ ion chromatography system in displacement chemical regeneration (DCR) mode. The Dionex Easion IC system is an integrated, single-channel low-cost system designed for isocratic applications with suppressed conductivity detection. Coupled to the Dionex AS autosampler, it provides a fast and low-cost choice for routine water analysis.



Analysis of municipal drinking water using the Dionex IonPac AS22-Fast-4µm columns

For more information click [here](#)



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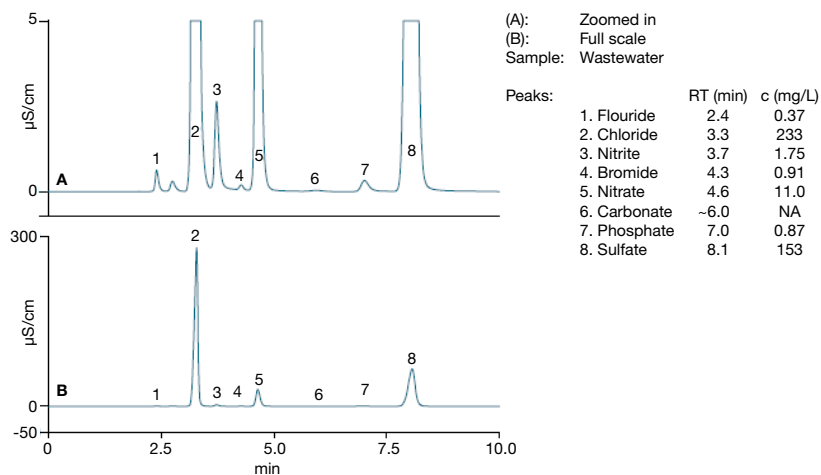
**Disinfection byproducts**

**Toxic contaminants**

Helpful resources and links

IC is a well-accepted technique for monitoring inorganic anions in water, including surface, ground, drinking, and wastewaters. As an approved Clean Water Act (CWA) Test Method, IC was adopted for wastewater analysis under the U.S. EPA Office of Water and the National Pollution Discharge Elimination System (NPDES) permits program. In addition, many standard-setting organizations (including the International Organization for Standardization (ISO), American Society for Testing and Materials (ASTM), and American Water Works Association (AWWA) have validated IC methods for the determination of inorganic anions.

This application note demonstrates the determination of inorganic anions in water samples (both drinking water and wastewater) using a Thermo Scientific™ Dionex™ ICS-5000+ IC system with a Thermo Scientific™ Dionex™ IonPac™ AS29-Fast-4µm anion-exchange column. The application note also shows the benefits of this column compared to a Thermo Scientific™ Dionex™ IonPac™ AS4A-SC column, the column originally specified in U.S. EPA Method 300.0 Part A.



Determination of inorganic anions in wastewater using a Dionex IonPac AS29-Fast-4µm column

**Linearity, method detection limits (MDLs), retention time, and peak area precisions obtained using a Dionex IonPac AS29-Fast-4µm column with a 10 µL injection**

Analyte	Range (mg/L)	Coefficient of determination (r <sup>2</sup> )	Calculated MDL* (mg/L)	Retention time precision (RSD)	Peak area precision (RSD)
Fluoride	0.1–100	1	0.02	0.1	0.7
Chloride	0.02–1000	0.999	0.004	< 0.01	0.5
Nitrite-N	0.01–100	1	0.003	< 0.01	0.7
Bromide	0.01–100	0.989 (0.994)**	0.02	< 0.01	0.7
Nitrate-N	0.01–100	0.996	0.003	< 0.01	0.5
Phosphate-P	0.01–100	0.995 (1.00)**	0.035	< 0.01	3.7
Sulfate	0.02–500	0.998	0.01	< 0.01	0.6

\*MDL = (t) × (S), 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), S = standard deviation of the replicate analyses.

\*\*Calibration type is linear and forced through the origin. The values in parentheses (bromide and phosphate) are quadratic fits that were forced through the origin.

For more information click [here](#)



# Trace concentrations of oxyhalides and bromide in municipal and bottled water using a compact IC system

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**Disinfection byproducts**

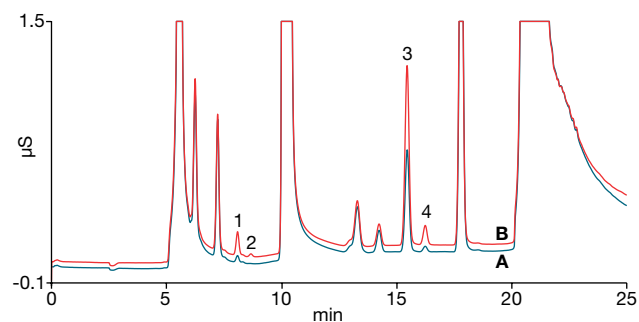
**Toxic contaminants**

Helpful resources and links

Most municipal water authorities must treat their water to produce safe drinking water. Disinfection protects public water systems from potentially dangerous microbes. However, the disinfectants can react with naturally occurring materials in the water to form disinfection byproducts (DBPs), such as oxyhalides, which may pose health risks. Currently, the only solution is to limit bromate formation during the water treatment process. Careful monitoring of bromate concentration ensures that it does not exceed safe drinking water standards.

This study demonstrates that oxyhalides and bromide can be determined accurately in municipal drinking water and bottled water using a Dionex™ IonPac™ AS19-4µm column and a Thermo Scientific™ Dionex™ Integriion™ HPIC™ system. The excellent resolution of the 4 µm small-particle column allows for faster analysis without compromising data quality. The Dionex Integriion HPIC system provides excellent reproducibility, thereby yielding greater quantification accuracy and consistently reliable results.

Samples	(A): Drinking water	
	(B): Spiked drinking water	
Peaks	(A): 1. Chlorite	11.1 µg/L
	3. Chlorate	137
	4. Bromide	11.0
Peaks	(B): 1. Chlorite	29.8 µg/L
	2. Bromate	4.64
	3. Chlorate	238
	4. Bromide	31.7



Oxyhalides and bromide spiked in drinking water #2

**Recoveries of trace oxyhalides and bromide in drinking water**

Analyte	Drinking water 1			Drinking water 2			Drinking water 3		
	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	10.8	20	90.3	11.6	20	91.8	< MDL	20	105.2
Bromate	< MDL	5	90.6	< MDL	5	90.5	< MDL	5	99.0
Chlorate	146.3	100	106.3	138.2	100	104.5	108.3	100	103.0
Bromide	13.5	20	103.7	11.6	20	102.0	90.3	100	104.8

For more information click [here](#)



# Determination of trace concentrations of oxyhalides and bromide in municipal and bottled waters

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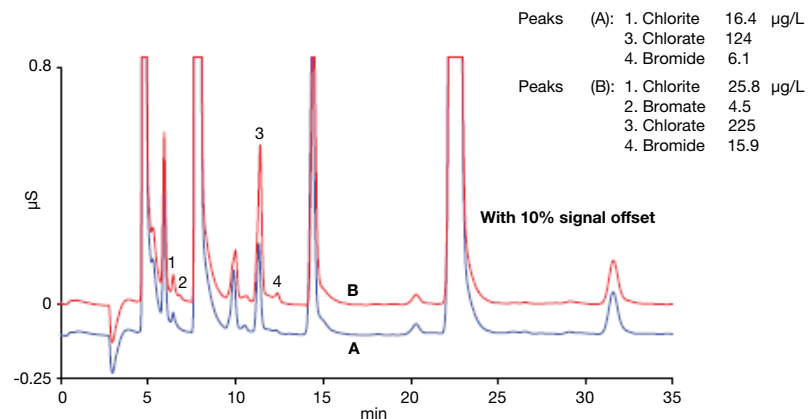
**Toxic contaminants**

Helpful resources and links

Public water systems are required to treat source water to bring safe drinking water to thousands of households. Disinfection, an essential step in the water treatment process, inactivates dangerous microbes and pathogens. The most commonly used chemical disinfectants are chlorine, chlorine dioxide, chloramine, and ozone. However, the disinfectants themselves can react with naturally occurring materials in the water to form disinfection byproducts (DBPs) that may pose health risks. For example, chlorination of drinking water can produce trihalomethanes, haloacetic acids, and chlorate. Chlorine dioxide treatment generates chlorite and chlorate, and chloramine is known to generate chlorate. Ozone reacts with natural sources of bromide found in water supplies to produce bromate. Inorganic DBPs, also referred to as DBP anions or oxyhalides, such as bromate, chlorate, and chlorite, are regulated or monitored by regulatory agencies.

**Method detection limits of oxyhalides and bromide in DI water and simulated drinking water with a Dionex CRD 300 device**

Analyte	MDL standard concentration (µg/L)	Calculated MDL in DI H <sub>2</sub> O (µg/L)	Calculated MDL in simulated drinking water (µg/L)
Chlorite	1	0.37	0.34
Bromate	2	0.57	0.63
Chlorate	3	0.66	0.58
Bromide	3	0.52	0.61



Determination of DBP anions and bromide in (A) tap water 2 and (B) spiked tap water 2 with a Dionex CRD 300 device

This application note demonstrates that oxyhalides and bromide can be successfully determined at concentrations required for regulatory standards and guidelines using a carbonate eluent on a Thermo Scientific™ Dionex™ ion chromatography system equipped with a Thermo Scientific™ Dionex™ IonPac™ AS23-4µm column, Thermo Scientific™ Dionex™ CRD 300 carbonate removal device, and a Thermo Scientific™ Dionex™ AERS™ 500 carbonate anion electrolytically regenerated suppressor.

For more information click [here](#)





# Fast determination of oxyhalides and bromide in drinking water using a compact ion chromatography system coupled with a single quadrupole mass spectrometer

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**Toxic contaminants**

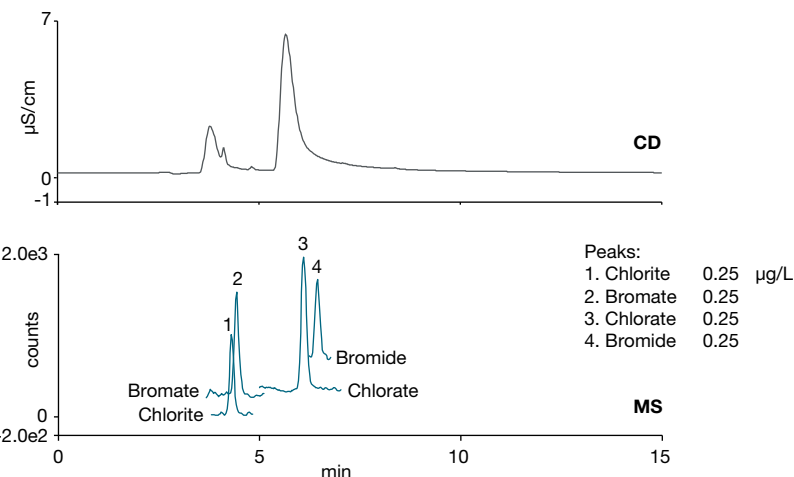
Helpful resources and links

Most municipal water authorities disinfect their water in order to provide their communities with safe drinking water. These disinfectants can react with naturally occurring material in the water to form unintended disinfection byproducts (DBPs), which may pose health risks. For example, chlorination of drinking water can produce trihalomethanes, haloacetic acids, and chlorate. Major regulatory bodies worldwide have set the maximum allowable concentration for bromate in drinking water at 10 µg/L. Although, discussions to lower this limit are ongoing, more sensitive analytical methods would be required.

**MDL and LOD using MS detection**

Analyte	MDL (µg/L)	MDL in HIW* (µg/L)	LOD (µg/L)	LOD in HIW (µg/L)
<b>Chlorite</b>	0.030	0.036	0.025	0.029
<b>Bromate</b>	0.038	0.033	0.035	0.031
<b>Chlorate</b>	0.036	0.045	0.028	N/A
<b>Bromide</b>	0.049	N/A	0.050	N/A

\*HIW = High ionic strength water



CD (top) and MS profile (bottom) of a 0.25 µg/L calibration standard (No ISTD)

In this application note, oxyhalides and bromide are accurately determined in municipal drinking water and bottled water using a 15-min isocratic IC-MS method. The method provides an advantage over the traditional ion chromatography-conductivity detector (IC-CD) method, in that it offers positive analyte-identification, particularly advantageous for complex samples.

For more information click [here](#)



# Improved ion chromatography column for separation of ethylenediamine carbamate and fluoride, and carbonate and sulfate in drinking water

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**Disinfection byproducts**

**Toxic contaminants**

Helpful resources and links

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. In addition, bromate can also be formed when chlorinated water is exposed to the UV rays in sunlight. The determination of trace concentrations of bromate and other oxyhalides in drinking water preserved with ethylenediamine (EDA) to maintain the integrity of chlorite and bromate can be very challenging.

This study demonstrates the Thermo Scientific™ Dionex™ IonPac™ AS30 high capacity, hydroxide-selective column that offers improved separation of inorganic anions and oxyhalides in samples preserved with EDA. The Dionex IonPac AS30 column was specifically developed to resolve the EDA carbamate artifact and fluoride as well as carbonate and sulfate better than the Thermo Scientific™ Dionex™ IonPac™ AS27 and the Dionex IonPac AS19 columns, and provide good resolution of dichloracetate (DCA, a surrogate anion) from potentially interfering matrix anions.

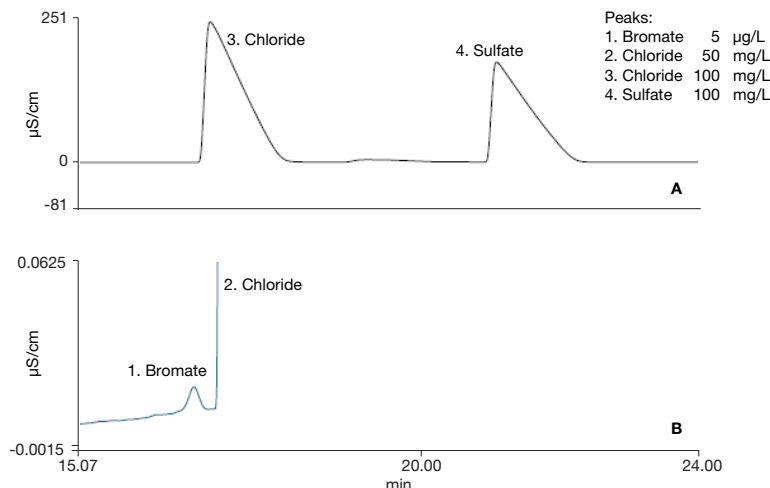
**Linearity and detection limits obtained using a Dionex IonPac AS30 column**

Analyte	Range (µg/L)	Coefficient of determination (r <sup>2</sup> )	MDL standard (µg/L)	Calculated MDL <sup>a,b</sup> (µg/L)
Chlorite	1–100	0.9995	0.5	0.15
Bromate	1.25–30	0.9999	1.0	0.29
Bromide	10–500	0.9999	2.5	0.58
Chloride	10–500	0.9998	2.5	0.60

a50 µL injection volume

bMDL =  $\sigma t_{S,99}$  where  $t_{S,99} = 3.14$  for  $n = 7$

For more information click [here](#)



Separation of bromate and chloride on a Dionex IonPac AS30 column in (A) High ion strength water (HIW) sample, and (B) ½ HIW sample



# Determination of nitrite and nitrate in wastewater using capillary IC with UV detection

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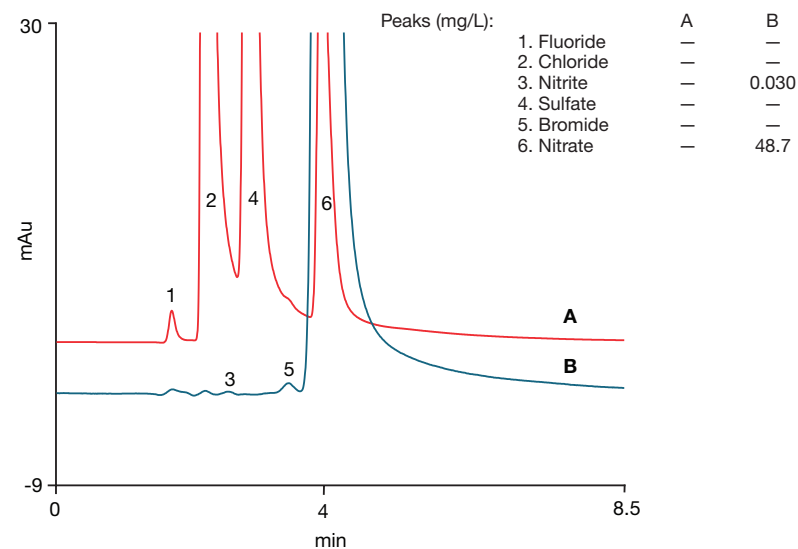
## Disinfection byproducts

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Helpful resources and links



Ion chromatography with suppressed conductivity detection is an effective technique to simultaneously determine common inorganic anions in environmental water and drinking water. However in some samples such as mineral water, wastewater, and brine, accurate quantification of some anions present at low concentrations can be challenging due to the high ionic strength of the sample. Ion chromatography with UV detection provides an alternate approach for determining nitrite and nitrate without compromising sensitivity. By combining suppressed conductivity with UV detection, the suppressor reduces the background noise. The Dionex Capillary RFIC system delivers fast turnaround by reducing eluent preparation, system startup, and equilibration times. This method is a solution for nitrite analysis when high concentrations of chloride can mask the presence of nitrite.



Separation of inorganic anions in a municipal wastewater sample spiked with 0.030 mg/L nitrite

For more information click [here](#)



# Low-cost determination of inorganic cations and ammonium in environmental waters

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Helpful resources and links

In the U.S., water quality is regulated through the Safe Drinking Water Act (SDWA) and the Clean Water Act (CWA) and enforced through the U.S. EPA. As a well-accepted technique, IC has been used for compliance monitoring of inorganic anions in water, including surface, ground, drinking, and waste waters following U.S. EPA Methods 300 and 300.1. Although the common alkali and alkaline earth cations, and ammonium are not considered primary drinking water contaminants in the U.S., they are routinely monitored and reported here and are regulated in Europe and Japan. Ammonium is measured in the U.S. for wastewater discharge compliance.

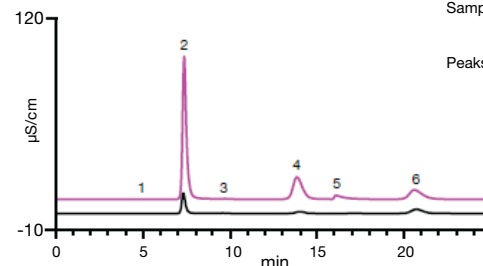
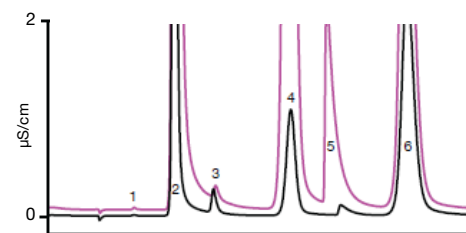
### Linearity obtained using a Dionex IonPac CS16 column with a 25.0 µL injection

Analyte	Range (mg/L)	Coefficient of determination (r <sup>2</sup> )*
Lithium	0.01–20	0.999
Sodium	0.04–500	0.999
Ammonium	0.05–125	0.997**
Magnesium	0.05–125	1.000
Potassium	0.1–250	1.000
Calcium	0.1–250	1.000

\*Calibration type is linear and forced through the origin

\*\*Calibration type is quadratic and forced through the origin

This work demonstrates the determination of cations and ammonium in environmental waters by IC using a Thermo Scientific™ Dionex™ IonPac™ CS16 column set on a Dionex Easion IC system in displacement chemical regeneration (DCR) mode.



Samples: (A) Black, a municipal drinking water sample  
(B) Red, a waste water sample

Peaks:	min	conc. (mg/L)	
		A	B
1. Lithium	5.0	0.00	0.01
2. Sodium	7.4	20.3	146.0
3. Ammonium	9.6	0.8	0.4
4. Magnesium	14.0	2.4	28.3
5. Potassium	16.7	0.7	13.9
6. Calcium	21.0	10.9	24.8

Analysis of a municipal drinking water and a waste water using the Dionex IonPac CS16 columns

For more information click [here](#)



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Fast and low-cost determination of anions in municipal drinking water

Inorganic anions in drinking water, wastewater, and high ionic strength water

Oxyhalides and bromide in municipal and bottled waters using a compact IC system

Trace concentrations of oxyhalides and bromide in municipal and bottled waters

Fast determination of oxyhalides and bromide in drinking water using IC with a single quadrupole mass spectrometer

Improved IC separation of ethylenediamine carbamate and fluoride, and carbonate and sulfate in drinking water

Nitrite and nitrate in wastewater using capillary IC with UV detection

Low-cost determination of inorganic cations and ammonium in environmental waters

**Anions and carboxylic acids in urban fine particles (PM<sub>2.5</sub>)**

**Disinfection byproducts**

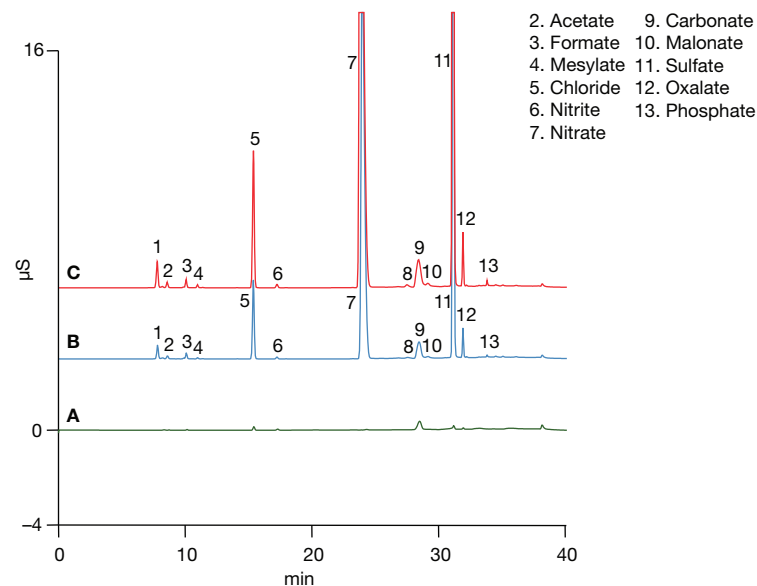
**Toxic contaminants**

Helpful resources and links

Identifying the composition of ambient fine particle pollution, particles that are 2.5 µm and smaller in diameter (PM<sub>2.5</sub>), plays an important role in air quality management. The chemical composition of PM<sub>2.5</sub> includes many mineral oxides, sulfate, nitrate, polycyclic aromatic hydrocarbons, organic acids, and organic chlorine that directly impact atmospheric quality and influence human health. Although the simultaneous determination of water-soluble anions and carboxylic acids in PM<sub>2.5</sub> is difficult to accomplish by other analytical methods, it has already been demonstrated using IC.

**Calibration data and MDLs**

Anion	Regression equation	(r <sup>2</sup> )	Range (mg/L)	MDL (µg/L)
Fluoride	A = 0.3498c - 0.0054	1	0.02–5	06
Acetate	A = 0.0794c + 0.0034	0.9985	0.02–5	12.5
Formate	A = 0.1678c + 0.0024	0.9999	0.02–5	07
Mesylate	A = 0.072c - 0.0038	0.9994	0.02–5	18
Chloride	A = 0.206c - 0.0015	0.9999	0.024–6	4.5
Nitrite	A = 0.1398c - 0.0049	0.9999	0.02–5	14
Nitrate	A = 0.1197c - 0.0045	0.9995	0.024–6	13
Succinate	A = 0.076c + 0.0028	0.9987	0.02–5	17
Malonate	A = 0.089c + 0.0039	0.9983	0.02–5	18.5
Sulfate	A = 0.1554c + 0.0313	0.9999	0.02–5	10.5
Oxalate	A = 0.1072c - 0.0013	1	0.02–5	11
Phosphate	A = 0.0685c - 0.0012	0.9999	0.02–5	12



(A) blank, (B) air sample, and (C) sample spiked with a mixed anion standard solution

Standard methods (e.g., U.S. EPA Method 26A and ASTM International's standard test method (ASTM) D5085-02) can be used to determine chloride, nitrate, and sulfate in air; however, the number of analytes and method sensitivity are inadequate when compared to newer technologies and current knowledge of air contaminants. This application note shows an efficient IC method for the sensitive determination of inorganic anions and carboxylic acids using the Thermo Scientific™ Dionex™ IonPac™ AS11-HC column (fluoride, acetate, formate, mesylate, chloride, nitrate, succinate, malonate, sulfate, and oxalate) in airborne PM.

For more information click [here](#)



# Speciation of bromine compounds in ozonated drinking water using ion chromatography and inductively coupled plasma mass spectrometry

Introduction

Inorganic anions and cations

Disinfection byproducts

**Speciation of bromine compounds in ozonated drinking water using IC and ICP-MS**

Disinfection byproduct anions and bromide in drinking water using a reagent-free IC system and postcolumn trace bromate analysis

Sub- $\mu\text{g/L}$  bromate in municipal and natural mineral waters using preconcentration with two dimensional IC and suppressed conductivity detection

Low-level haloacetic acids, bromate, and dalapon in drinking water using IC-MS

Fast determination of nine haloacetic acids, bromate, and dalapon at trace levels in drinking water samples

Haloacetic acids, bromate, and dalapon in drinking water using ion chromatography coupled to high-resolution accurate-mass (IC-HRAM) mass spectrometry

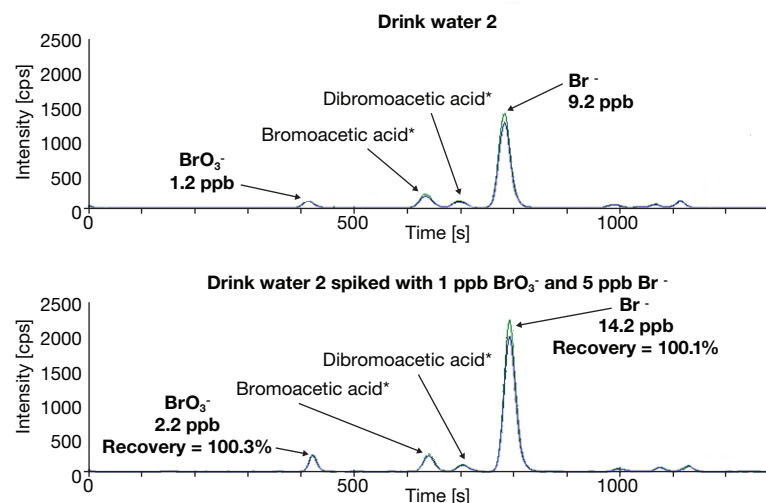
Toxic contaminants

Bromine speciation in drinking water is required worldwide by major regulatory bodies. A maximum contaminant level (MCL) of  $10 \mu\text{g/L}$  in the U.S. for bottled drinking water and in the EU of  $3 \mu\text{g/L}$  for any water treated with ozonation are stipulated for the bromate anion. Differentiating bromate from bromide is therefore important due to the toxicity differences between the two species. Bromine analysis with inductively coupled plasma mass spectrometry (ICP-MS) is challenging because the ionization potential of bromine is relatively high, and it lowers sensitivity in ICP based techniques. Additionally, there are several isobaric interferences on  $m/z$  81 and 79 (the two Br stable isotopes). The U.S. EPA Method 321.8 provides an analytical procedure for bromate determination in drinking water using ion chromatography inductively coupled plasma mass spectrometry (IC-ICP-MS). This method significantly reduces the risk for contamination and has the advantage of a stable and quantitative bromate recovery through the IC column, and it tolerates high salt conditions. The method also provides guidance for reducing polyatomic interferences that overlap with the two most abundant Br isotopes.

## Possible sources of isobaric interference

Interferent source	MASS 79	MASS 81
Plasma	$^{40}\text{Ar}^{38}\text{Ar}^1\text{H}^+$	$^{40}\text{Ar}^{40}\text{Ar}^1\text{H}^+$
Sulfate	–	$\text{SO}_3\text{H}^+$
Phosphate	$^{31}\text{P}^{16}\text{O}_3^+$	$^{31}\text{P}^{16}\text{O}_3^1\text{H}_2^+$
Potassium	$^{40}\text{Ar}^{39}\text{K}^+$	–

For more information click [here](#)



**Separation for Br species in water sample 2 (upper trace) and the same sample with a 1  $\mu\text{g/L}$  bromate and 5  $\mu\text{g/L}$  bromide spike (lower trace).** Note the consistency in elution times for all the species detected, the excellent separation and quantitative recovery.

Combining the resolving capability of IC with the detection power of ICP-MS allows fast, easy and reliable determination of bromine species in drinking waters. This method enables robust and reliable speciation of bromine containing species in drinking water, with no salinity-related recovery issues. Thanks to the high sensitivity and completely metal free sample and mobile phase flow paths, the application complies to U.S. EPA 321.8 requirements and provides MDLs well below the values necessary for accurate monitoring of water from various sources.



# Determination of disinfection byproduct anions and bromide in drinking water using a reagent-free ion chromatography system followed by postcolumn addition of an acidified on-line generated reagent for trace bromate analysis

Introduction

Inorganic anions and cations

Disinfection byproducts

Speciation of bromine compounds in ozonated drinking water using IC and ICP-MS

**Disinfection byproduct anions, bromide in drinking water using a reagent-free IC system and post-column trace bromate analysis**

Sub- $\mu\text{g/L}$  bromate in municipal and natural mineral waters using preconcentration with two dimensional IC and suppressed conductivity detection

Low-level haloacetic acids, bromate, and dalapon in drinking water using IC-MS

Fast determination of nine haloacetic acids, bromate, and dalapon at trace levels in drinking water samples

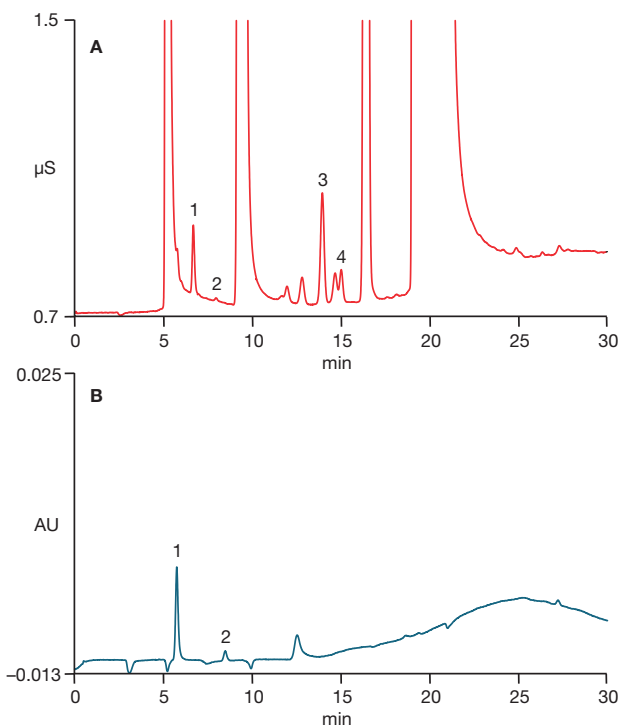
Haloacetic acids, bromate, and dalapon in drinking water using ion chromatography coupled to high-resolution accurate-mass (IC-HRAM) mass spectrometry

Toxic contaminants

Helpful resources and links

Considerable efforts have focused on developing improved analytical methods for determining trace concentrations of inorganic DBPs in drinking water to meet current regulatory requirements. Traditionally, IC with suppressed conductivity detection has been used to determine chlorite, bromate, and chlorate in environmental waters, as described

in U.S. EPA Method 300.0 (B). In this application note, we demonstrate the performance of the Dionex IonPac AS19 column for U.S. EPA Method 326.0. This method allows quantification of bromate to  $1 \mu\text{g/L}$  by suppressed conductivity detection with a hydroxide eluent and  $0.5 \mu\text{g/L}$  using postcolumn reaction with UV detection.



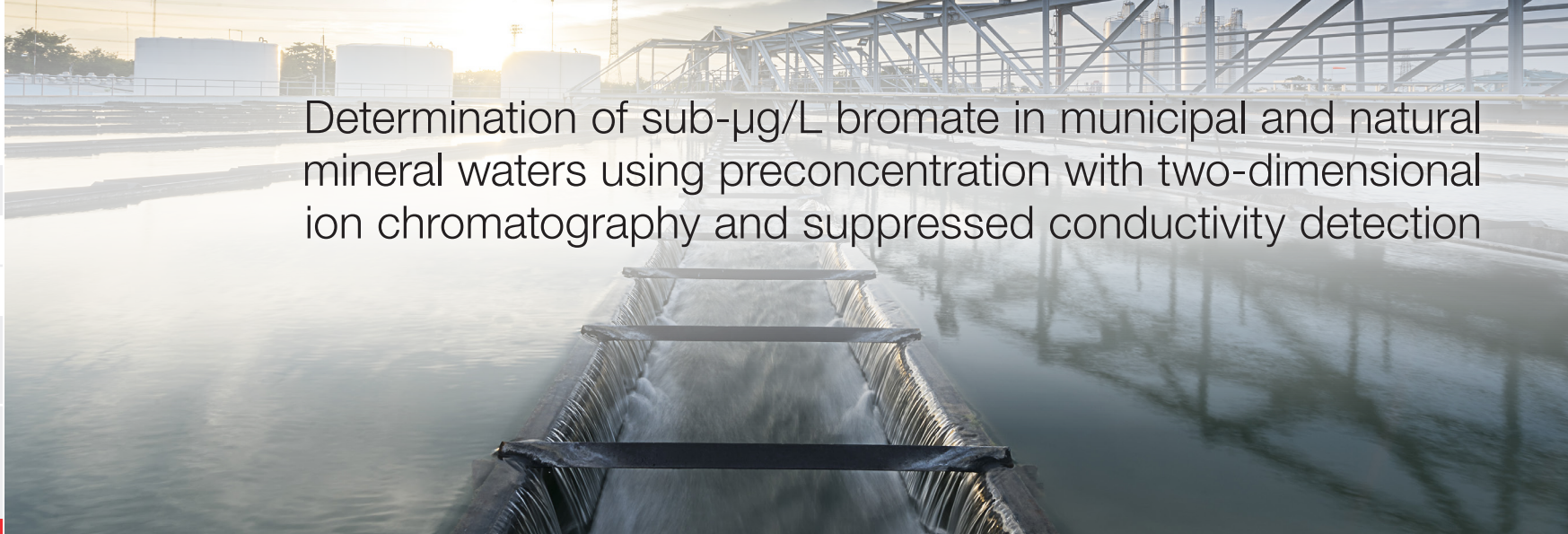
Column: Dionex IonPac AG19/AS19 columns, 4 mm  
 Detection: A: Dionex ASRS 300 suppressor, 4 mm, external water mode  
 B: Absorbance, 352 nm  
 Postcolumn Reagent: Acidified potassium iodide (KI)

Peaks $\mu\text{g/L}$ (ppb):	A	B
1. Unknown	—	1. Unknown —
2. Bromate	2.4	2. Bromate 2.8
3. Chlorate	62.4	
4. Bromide	17.5	

**Determination of trace DBP anions and bromide in tap water B using suppressed conductivity detection and UV absorbance after PCR with acidified iodide**

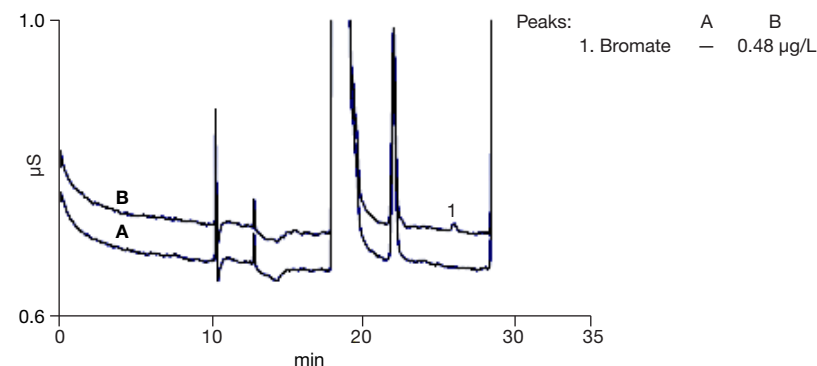
For more information click [here](#)





# Determination of sub- $\mu\text{g/L}$ bromate in municipal and natural mineral waters using preconcentration with two-dimensional ion chromatography and suppressed conductivity detection

Determining low concentrations of bromate in high-ionic-strength matrices using suppressed conductivity detection is subject to potential interferences and loss of sensitivity. Although postcolumn reaction methods do not generally suffer from interferences by common anions, column overloading with high-ionic-strength samples can still cause peak broadening and an associated loss of response. In this application note, we demonstrate the use of a two-dimensional (2-D) IC system for the determination of trace concentrations of bromate in municipal and natural mineral waters with high-ionic-strength matrices. This 2-D IC method achieves bromate detection limits equivalent to or better than postcolumn addition methods. The 2-D IC method avoids the cost and disposal of the chemicals required for postcolumn configurations and simplifies the experimental setup.



Chromatogram of (A) mineral water C and (B) mineral water C fortified with 0.5  $\mu\text{g/L}$  bromate

Linearity obtained using a Dionex IonPac CS16 column with a 25.0  $\mu\text{L}$  injection

Analyte	Range (mg/L)	Linearity ( $r^2$ )*	MDL standard ( $\mu\text{g/L}$ )	SD ( $\mu\text{g/L}$ )	Calculated MDL ( $\mu\text{g/L}$ )
Bromate	0.15-15	0.9995	0.20	0.012	0.036

\*Quadratic fit

For more information click [here](#)





# Determination of low-level haloacetic acids, bromate, and dalapon in drinking water using IC-MS

Introduction

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Speciation of bromine compounds in ozonated drinking water using IC and ICP-MS

Disinfection byproduct anions, bromide in drinking water using a reagent-free IC system and postcolumn trace bromate analysis

Sub- $\mu\text{g/L}$  bromate in municipal and natural mineral waters using preconcentration with two dimensional IC and suppressed conductivity detection

**Low-level haloacetic acids, bromate, and dalapon in drinking water using IC-MS**

Fast determination of nine haloacetic acids, bromate, and dalapon at trace levels in drinking water samples

Haloacetic acids, bromate, and dalapon in drinking water using ion chromatography coupled to high-resolution accurate-mass (IC-HRAM) mass spectrometry

Toxic contaminants

Helpful resources and links

Municipal water authorities most commonly disinfect water using chemical disinfectants such as chlorine, chlorine dioxide, chloramine, and ozone. However, chlorination of drinking water can produce trihalomethanes, haloacetic acids (HAAs), and chlorate, while bromate is formed when disinfecting ozone reacts with natural sources of bromide. These disinfection byproducts (DBPs) may pose health risks. Dalapon, a

herbicide used to control grasses in a wide variety of crops and can be introduced to waterways from runoff is another potential drinking water contaminant of concern. U.S. EPA Method 557 has been validated for the determination of HAAs, bromate, and dalapon using ion chromatography (IC) coupled with electrospray ionization tandem mass spectrometry (IC-ESI-MS/MS).

Summary of IC-MS method performance values and comparison of its MDL to U.S. EPA Method 557

Analyte	Retention time (min)	Internal standard	U.S. EPA Method 557 DL ( $\mu\text{g/L}$ )	MDL ( $\mu\text{g/L}$ )	Calibration range ( $\mu\text{g/L}$ )	( $r^2$ ) value
MCAA*	6.5	MCAA-2-13C	0.2	0.1	0.1–100	0.999
MBAA*	7.2	MBAA-1-13C	0.06	0.03	0.1–100	0.998
Bromate	7.7	MBAA-1-13C	0.02	0.12	0.5–100	0.998
Dalapon	12.3	DCAA-2-13C	0.04	0.12	0.5–100	0.999
DCAA*	13.4	DCAA-2-13C	0.06	0.03	0.1–100	0.998
BCAA	15.0	DCAA-2-13C	0.11	0.16	0.5–100	0.998
DBAA*	17.1	DCAA-2-13C	0.02	0.16	0.5–100	0.999
TCAA*	24.8	TCAA-2-13C	0.09	0.67	1–100	0.999
BDCAA	27.0	TCAA-2-13C	0.05	2.79	4–100	0.998
CDBAA	30.4	TCAA-2-13C	0.04	1.04	4–100	1
TBAA	35.1	TCAA-2-13C	0.07	4.55	10–100	1

\*HAA5

For more information click [here](#)



Speciation of bromine compounds in ozonated drinking water using IC and ICP-MS

Disinfection byproduct anions, bromide in drinking water using a reagent-free IC system and postcolumn trace bromate analysis

Sub- $\mu\text{g/L}$  bromate in municipal and natural mineral waters using preconcentration with two dimensional IC and suppressed conductivity detection

Low-level haloacetic acids, bromate, and dalapon in drinking water using IC-MS

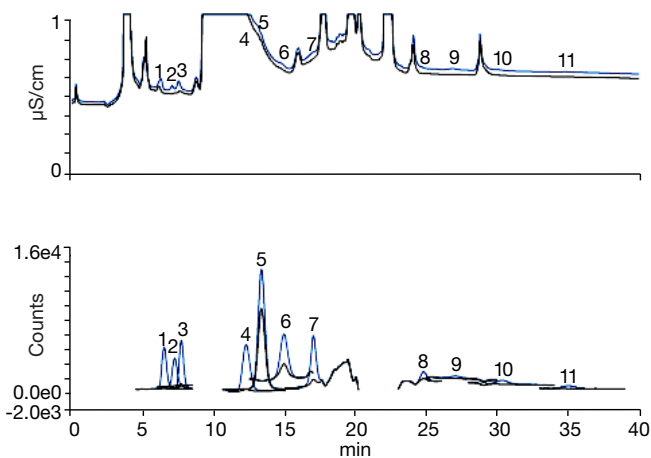
Fast determination of nine haloacetic acids, bromate, and dalapon at trace levels in drinking water samples

Haloacetic acids, bromate, and dalapon in drinking water using ion chromatography coupled to high-resolution accurate-mass (IC-HRAM) mass spectrometry



This application note evaluated the determination of HAAs, bromate, and dalapon in drinking water using a Thermo Scientific™ Dionex™ ICS-6000 HPIC™ system with DP pump and Thermo Scientific™ Dionex™

IonPac™ AS31 column coupled with a Thermo Scientific™ ISQ™ EC single quadrupole mass spectrometer. This method is sensitive ( $< 1 \mu\text{g/L}$ ) for HAA5, which is currently regulated by the U.S. EPA.



(A) Gray, Water sample 3  
(B) Blue, Water sample 3 spiked with 10  $\mu\text{g/L}$  mixed standards

No.	Peak	RT (min)	Conc. ( $\mu\text{g/L}$ )	
			A	B
1.	MCAA	6.5	ND	10.7
2.	MBAA	7.2	ND	11.1
3.	Bromate	7.7	0.33	12.3
4.	Dalapon	12.3	ND	10.4
5.	DCAA	13.4	21.8	33.7
6.	BCAA	15.0	4.9	14.7
7.	DBAA	17.1	0.4	9.5
8.	TCAA	24.8	4.7	16.5
9.	BDCAA	27.0	ND	9.9
10.	CDBAA	30.4	ND	10.3
11.	TBAA	35.1	ND	8.8

ND: Not detectable

Chromatogram of (A) mineral water C and (B) mineral water C fortified with 0.5  $\mu\text{g/L}$  bromate

For more information click [here](#)



# Fast determination of nine haloacetic acids, bromate, and dalapon at trace levels in drinking water samples by tandem IC-MS/MS

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Speciation of bromine compounds in ozonated drinking water using IC and ICP-MS

Disinfection byproduct anions, bromide in drinking water using a reagent-free IC system and postcolumn trace bromate analysis

Sub- $\mu\text{g/L}$  bromate in municipal and natural mineral waters using preconcentration with two dimensional IC and suppressed conductivity detection

Low-level haloacetic acids, bromate, and dalapon in drinking water using IC-MS

**Fast determination of nine haloacetic acids, bromate, and dalapon at trace levels in drinking water samples**

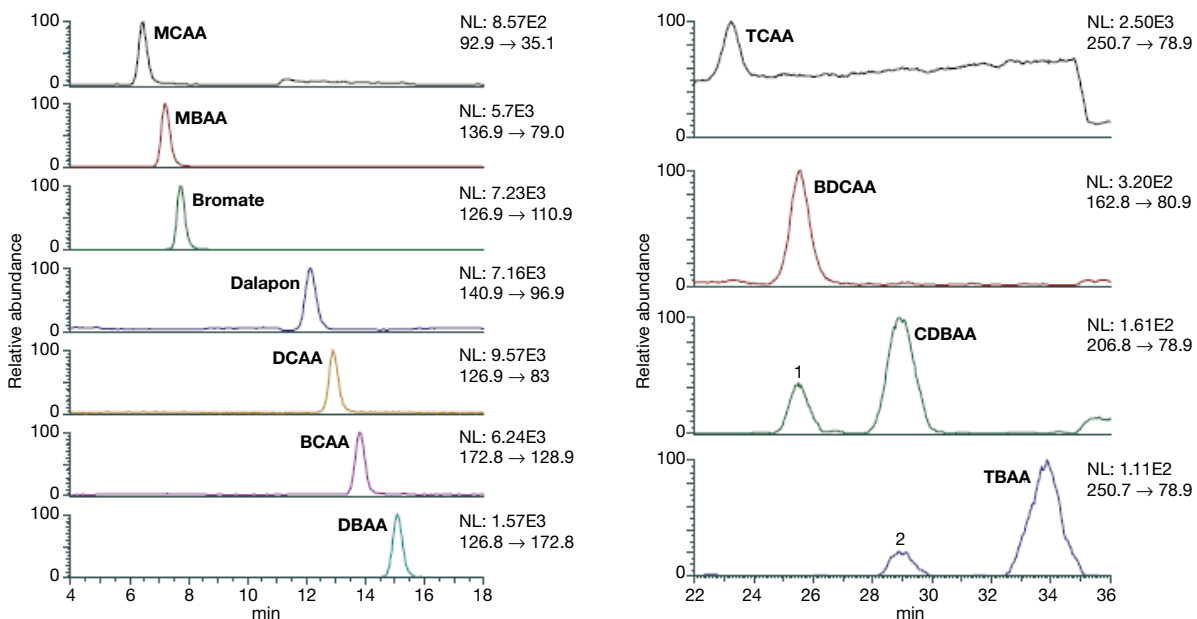
Haloacetic acids, bromate, and dalapon in drinking water using ion chromatography coupled to high-resolution accurate-mass (IC-HRAM) mass spectrometry

Toxic contaminants

Helpful resources and links

Haloacetic acids (HAAs) are a class of undesirable disinfection by-products (DBPs) formed during the disinfection of drinking water in which routine water disinfectants such as chlorine or chloramine are used to kill pathogenic microorganisms. Because of their suspected carcinogenicity, mutagenicity, as well as developmental, reproductive, and hepatic toxicity,

the World Health Organization (WHO) has established guidelines for these DBPs in drinking water. In the U.S., these guidelines are regulated by the EPA. In U.S. EPA Method 557, IC-ESI-MS/MS is used to directly determine HAAs, bromate, and dalapon in drinking water samples.



IC-MS/MS SRM chromatograms of nine HAAs, bromate, and dalapon with a concentration of 5  $\mu\text{g/L}$  in LSSM

For more information click [here](#)



Speciation of bromine compounds in ozonated drinking water using IC and ICP-MS

Disinfection byproduct anions, bromide in drinking water using a reagent-free IC system and postcolumn trace bromate analysis

Sub-µg/L bromate in municipal and natural mineral waters using preconcentration with two dimensional IC and suppressed conductivity detection

Low-level haloacetic acids, bromate, and dalapon in drinking water using IC-MS

**Fast determination of nine haloacetic acids, bromate, and dalapon at trace levels in drinking water samples**

Haloacetic acids, bromate, and dalapon in drinking water using ion chromatography coupled to high-resolution accurate-mass (IC-HRAM) mass spectrometry



This application note reports the application of a new ion exchange column Dionex IonPac AS31 column for the IC-MS/MS determination of nine haloacetic acids, bromate, and dalapon present at low concentrations in drinking water. The new IC-MS/MS method can

determine all analytes in water samples in 35 min, a 39% faster analysis time than the original U.S. EPA Method 557, which uses the Thermo Scientific™ Dionex™ IonPac™ AS24 column.

**IC-MS/MS method detection limits (MDL) and lowest concentration minimum reporting level (LCMRL) obtained for HAAs, bromate, and dalapon**

Analyte (µg/L, n = 7)	EPA reported DL	Calculated DL	EPA reported LCMRL	Calculated LCMRL
MCAA	0.20	0.099	0.58	0.15
MBAA	0.064	0.028	0.19	0.035
Bromate	0.02	0.012	0.042	0.039
Dalapon	0.038	0.031	0.41	0.20
DCAA	0.055	0.036	0.13	0.096
BCAA	0.11	0.087	0.16	0.15
DBAA	0.015	0.009	0.062	0.058
TCAA	0.09	0.061	0.25	0.14
BDCAA	0.05	0.027	0.19	0.055
CDBAA	0.041	0.042	0.080	0.10
TBAA	0.067	0.067	0.27	0.25

For more information click [here](#)



# Determination of haloacetic acids, bromate, and dalapon in drinking water using ion chromatography coupled to high-resolution accurate-mass (IC-HRAM) mass spectrometry

Introduction

Inorganic anions and cations

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Speciation of bromine compounds in ozonated drinking water using IC and ICP-MS

Disinfection byproduct anions, bromide in drinking water using a reagent-free IC system and postcolumn trace bromate analysis

Sub- $\mu\text{g/L}$  bromate in municipal and natural mineral waters using preconcentration with two dimensional IC and suppressed conductivity detection

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**Haloacetic acids, bromate, and dalapon in drinking water using ion chromatography coupled to high-resolution accurate-mass (IC-HRAM) mass spectrometry**

Toxic contaminants

Helpful resources and links

Haloacetic acids (HAAs) are one of the most commonly detected classes of disinfection by-products (DBPs) and have captured considerable attention due to their adverse biological effects on human and aquatic

organisms. Additional water contaminants of concern are bromate, which can arise as a byproduct of the ozonation of bromide containing water and the herbicide dalapon.

IC-HRAM MS lowest concentration minimum reporting level (LCMRL,  $\mu\text{g/L}$ )

Compound	EPA reported QQQ MS/MS	t-SIM/ddMS <sup>2</sup>	PRM	Full MS/dd-MS <sup>2</sup>
MCAA	0.58	0.1	0.1	0.1
MBAA	0.19	0.1	0.1	0.1
Bromate	0.042	0.0011	0.001	0.01
Dalapon	0.41	0.0025	0.025	0.01
DCAA	0.13	0.05	0.1	0.1
BCAA	0.16	0.00375	0.0054	0.012
DBAA	0.062	0.0025	0.00375	0.01
TCAA	0.25	0.075	0.05	0.075
BDCAA	0.19	0.0075	0.0077	0.11
CDBAA	0.08	0.00375	0.00375	0.18
TBAA	0.27	0.00375	0.028	> 0.375

For more information click [here](#)



Speciation of bromine compounds in ozonated drinking water using IC and ICP-MS

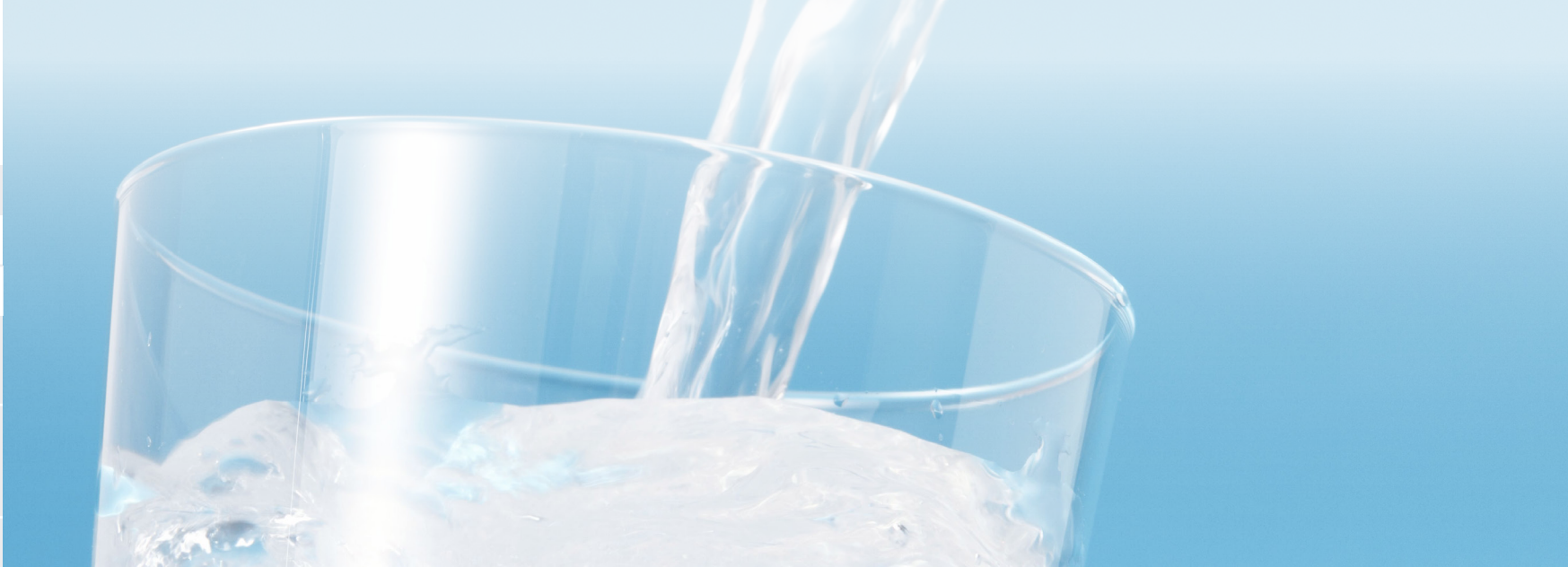
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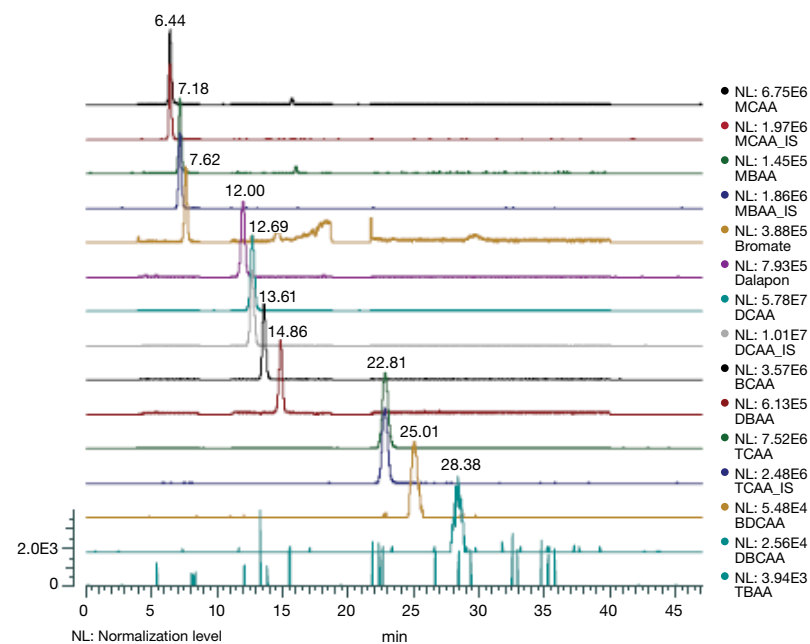
Low-level haloacetic acids, bromate, and dalapon in drinking water using IC-MS

Fast determination of nine haloacetic acids, bromate, and dalapon at trace levels in drinking water samples

**Haloacetic acids, bromate, and dalapon in drinking water using ion chromatography coupled to high-resolution accurate-mass (IC-HRAM) mass spectrometry**



In this application note, a fast, simple, and sensitive quantitation assay with high confidence was developed for determination of HAAs, bromate, and dalapon in drinking water using a recently introduced Dionex IonPac AS31 column, Dionex ICS-6000 HPIC system, and a Thermo Scientific™ Q Exactive™ HF mass spectrometer. Thermo Scientific™ Q Exactive™ hybrid quadrupole-Orbitrap™ series mass spectrometers offer analysts a range of scan modes that provide functionality comparable to the modes provided by conventional tandem quadrupole mass spectrometers. Compared to QQQ MS, the availability of high-resolution MS multiple techniques offers flexibility and convenience, bringing greater possibilities and confidence to the analysts in achieving more conclusive results to their experiments.



Chromatograms of a drinking water sample containing the preservative and internal standards (4  $\mu\text{g/L}$  each) using Full MS/dd-MS2 mode. Here, the y-axis scale is set to absolute (where the title is intensity), and the normalization is set to local.

For more information click [here](#)



Introduction

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**Hexavalent chromium in drinking water**

Chromium species using IC coupled to ICP-MS

Metal cyanide complexes by IC and UV absorbance detection

Total cyanide in wastewater and drinking water using ion exclusion chromatography with pulsed amperometric detection (ICE-PAD)

Cyanide in alkaline solutions using ion chromatography

Perchlorate in environmental waters using IC-MS

Perchlorate by U.S. EPA Method 332.0 using IC-MS

Perchlorate in drinking water using ion chromatography

Polar pesticides in drinking water using IC-MS/MS

Validation of IC-MS/MS method for determining polar pesticides

Adsorbable organic halogen in wastewater using CIC

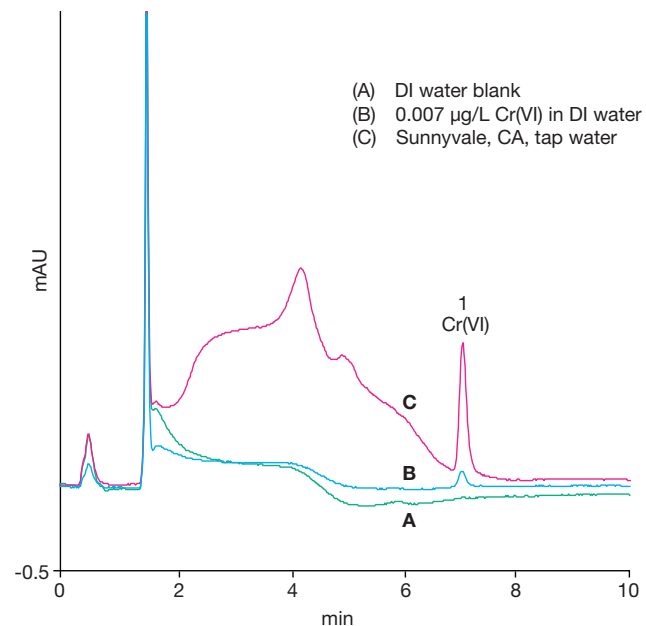
AOF by combustion IC—PFAS in aqueous samples

HPAE-PAD for determination of saccharides in atmospheric aerosol samples

Helpful resources and links

All hexavalent chromium Cr(VI) compounds are strong oxidizing agents and considered toxic and potentially carcinogenic. Hence, chromates are regulated in the environment and are a primary drinking water contaminant. Currently, dissolved hexavalent chromium is measured as chromate according to a modified version of U.S. EPA Method 218.6, that is based on anion exchange chromatography on a Thermo Scientific™ Dionex™ IonPac™ AS7 IC column and detection after post-column reaction with diphenylcarbazide. This permits a method detection limit (MDL) for chromate at 0.02 µg/L and can support a reporting limit of 0.06 µg/L.

This application note presents modifications to the existing U.S. EPA method 218.6 to allow sufficient sensitivity for determining hexavalent chromium (i.e., Cr(VI) as  $\text{CrO}_4^{2-}$ ) at the proposed California level of 0.02 µg/L. This includes the use of a 2 mm Dionex IonPac AS7 IC column set while appropriately reducing the flow rates and reaction coil volume. The resulting MDL for Cr(VI) as  $\text{CrO}_4^{2-}$  at 0.001 µg/L will allow a minimum quantitation limit of 0.003 µg/L, which is more than enough for the proposed California of 0.02 µg/L.



Determination of Cr(VI) in (A) DI water blank, (B) 0.007 g/L standard in DI water, and (C) Sunnyvale, CA, tap water sample with a measured concentration of 0.05 g/L Cr(VI) on a Thermo Scientific™ Dionex™ ICS-3000 system. Postcolumn reagent delivered by the second pump of DP. Flow cell: standard (PEEK).

Method detection limits for Cr(VI) in HIW based on a 1000 µL injection

Chromate concentration (µg/L)	Standard deviation (µg/L)	RSD (%)	MDL* (µg/L)
0.001	0.0003	10.03	0.0009
0.005	0.0004	6.62	0.0013

\*MDL for Cr(VI) for 1000 µL (n = 7) injections

For more information click [here](#)



# Determination of chromium species using ion chromatography coupled to inductively coupled plasma mass spectrometry

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Hexavalent chromium in drinking water

Chromium species using IC coupled to ICP-MS

Metal cyanide complexes by IC and UV absorbance detection

Total cyanide in wastewater and drinking water using ion exclusion chromatography with pulsed amperometric detection (ICE-PAD)

Cyanide in alkaline solutions using ion chromatography

Perchlorate in environmental waters using IC-MS

Perchlorate by U.S. EPA Method 332.0 using IC-MS

Perchlorate in drinking water using ion chromatography

Polar pesticides in drinking water using IC-MS/MS

Validation of IC-MS/MS method for determining polar pesticides

Adsorbable organic halogen in wastewater using CIC

AOF by combustion IC—PFAS in aqueous samples

HPAE-PAD for determination of saccharides in atmospheric aerosol samples

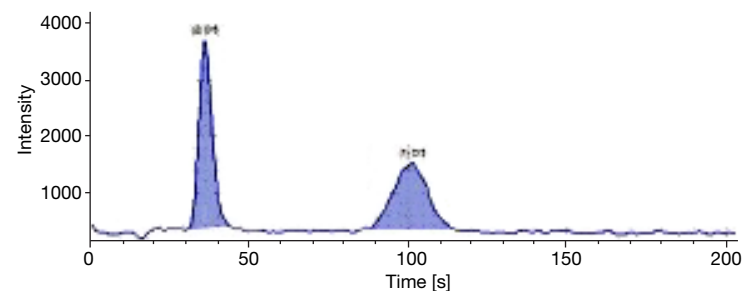
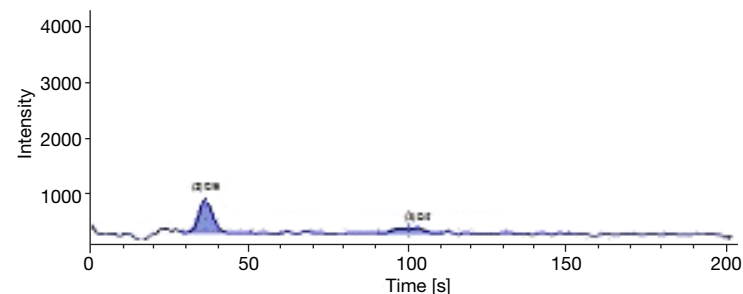
Helpful resources and links

In trace amounts, trivalent chromium (Cr (III)) is considered an essential nutrient that promotes insulin, sugar, and lipid metabolism. In contrast, hexavalent chromium (Cr (VI)) is toxic and can lead to respiratory tract, stomach, and intestinal irritation, anemia, and is known to be a human carcinogen. Cr (VI) can leach into drinking water sources naturally but drinking water can also be contaminated by industrial processes. Because of the varying toxicity attributable to the different oxidation states of chromium, simply knowing the total chromium concentration in a solution is not sufficient to determine its true toxicity following exposure, and therefore speciation analysis is required. While ICP-MS can readily determine the total amount of an element present, chromatographic separation prior to the ICP-MS system is required to separate the different elemental species. Because Cr (III) and Cr (VI) have different charges, ion chromatography (IC) using anion exchange is the ideal separation method for analysis of these species.

In this application note, the Dionex Aquion IC system using a Thermo Scientific™ Dionex™ IonPac™ AG7 column was coupled with the Thermo Scientific™ iCAP™ RQ ICP-MS to determine the concentration of Cr (III) and Cr (VI) in drinking water.

## Analytical figures of merit

	Cr (VI)	Cr (III)
Retention time (s)	36 ± 0.2	101 ± 1.2
Sensitivity (kcps/μg-L <sup>-1</sup> ) <sup>-1</sup>	114	123
Detection limit (ng-L <sup>-1</sup> )	4.0	9.0



Chromatograms showing the injection of tap water (top) and spiked tap water (bottom, 0.1 μg·L<sup>-1</sup>). For better comparability, all are scaled identically. First peak is Cr (VI), second Cr (III).

For more information click [here](#)





# Determination of metal cyanide complexes by ion chromatography with on-line sample preconcentration and UV absorbance detection

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Perchlorate by U.S. EPA Method 332.0 using IC-MS

Perchlorate in drinking water using ion chromatography

Polar pesticides in drinking water using IC-MS/MS

Validation of IC-MS/MS method for determining polar pesticides

Adsorbable organic halogen in wastewater using CIC

AOF by combustion IC—PFAS in aqueous samples

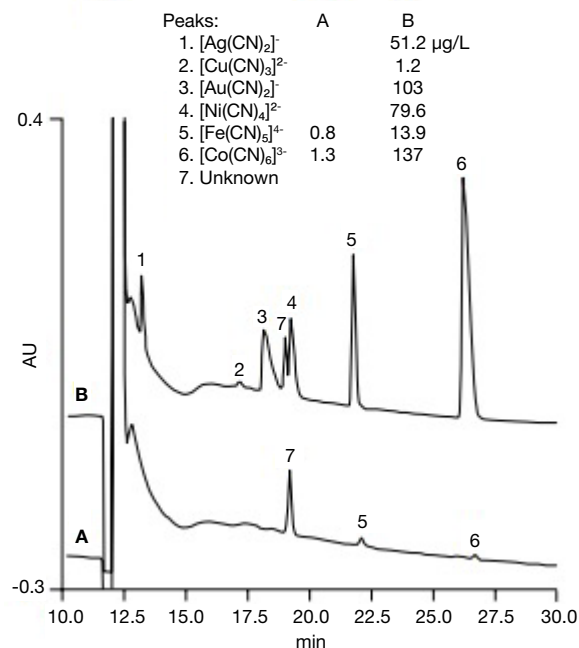
HPAE-PAD for determination of saccharides in atmospheric aerosol samples

Several methods measure free cyanide, but rely on some operational definition to distinguish between weak and strong cyanide complexes. Ion chromatography resolves each individual metal cyanide complex during an automated, 30 min separation. IC thus allows a precise differentiation of complexes of limited toxicity from those of greater toxicity. This method of on-line preconcentration allows determination of metal cyanide complexes at  $\mu\text{g/L}$  concentrations in a variety of environmental water matrices.

This method provides good recoveries for the gold, iron, and cobalt cyanide complexes in all matrices studied, and for the nickel cyanide complex in all matrices except wastewater. This method shows increased bias for the silver and copper cyanide complexes, especially in higher-ionic strength matrices.

## Linear ranges and MDLs for metal cyanide complexes

	Cr (VI)	Cr (III)
Retention time (s)	$36 \pm 0.2$	$101 \pm 1.2$
Sensitivity ( $\text{kcps}/\mu\text{g}\cdot\text{L}^{-1}$ ) <sup>-1</sup>	114	123
Detection limit ( $\text{ng}\cdot\text{L}^{-1}$ )	4.0	9.0



Anion-exchange separation with preconcentration and absorbance detection at 215 nm of metal cyanide complexes in groundwater from a manufactured gas plant (MGP) site. Groundwater matrix blank (A) and matrix spiked with metal cyanide complexes (B) as shown.

Helpful resources and links

For more information click [here](#)





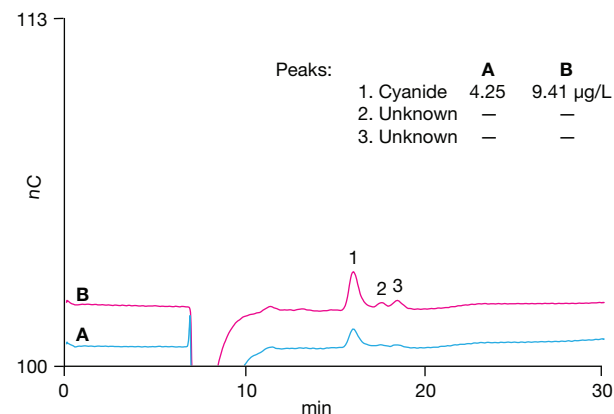
# Determination of total cyanide in municipal wastewater and drinking water using ion-exclusion chromatography with pulsed amperometric detection (ICE-PAD)

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- Perchlorate in drinking water using ion chromatography
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- Adsorbable organic halogen in wastewater using CIC
- AOF by combustion IC—PFAS in aqueous samples
- HPAE-PAD for determination of saccharides in atmospheric aerosol samples

Cyanide is a well-known acute toxin that prevents cellular respiration and interferes with iodine uptake by the thyroid gland, causing goiters and other long-term iodine deficiency diseases. Cyanide is regulated by the U.S. EPA for drinking water, surface water, and wastewater due to these health concerns. For drinking and surface waters, the EPA has established an Maximum Contaminate Level (MCL) of 200 µg/L free cyanide. For saltwater bodies, the continuous and maximum discharges are 1 µg/L total cyanide.

In U.S. EPA Methods 335.2, 335.3, and 335.4, samples are individually digested then determined spectrophotometrically or by titration. These methods are complicated and subject to interference from solutions and sulfur-containing compounds. Traditional chromatography methods, such as ion-exchange and ion-exclusion (ICE) can eliminate some of these interferences; however, cyanide may not be fully resolved from chloride and sulfide at mg/L levels. ICE is preferred because strong acid anions such as chloride and sulfate are excluded from the column, and cyanide is resolved from sulfide. IC using pulsed amperometric detection (PAD), is sensitive, selective, and suitable for direct determinations of cyanide. Using PAD with a platinum working electrode combines the advantages of IC with the sensitivity, selectivity, and stability to directly detect cyanide without interferences from chloride and sulfide.



Comparison of (A) 10-fold dilution of cyanide certified wastewater sample and (B) Sample A with 5 µg/L cyanide added

Sample	Amount found (µg/L) <sup>a</sup>	Amount added (µg/L)	Average recovery <sup>3</sup> (%)
100 mM sodium hydroxide	< LOD	1.06	110 ± 6.4
Filtered 100 mM sodium hydroxide	< LOD	5.02	102 ± 1.0
10-fold dilution of certified cyanide wastewater sample (4.0 µg/L total cyanide)	4.25 ± 0.07	4.99	102 ± 0.9
Municipal drinking water	0.67 ± 0.02	0.99	97.4 ± 2.0
Filtered municipal wastewater effluent without base	< LOD	Not tested	—
Filtered municipal wastewater effluent with base	5.99 ± 0.09	4.97	99.5 ± 1.0

<sup>a</sup> n = 6

Average cyanide determinations over three days

For more information click [here](#)



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Polar pesticides in drinking water using IC-MS/MS

Validation of IC-MS/MS method for determining polar pesticides

Adsorbable organic halogen in wastewater using CIC

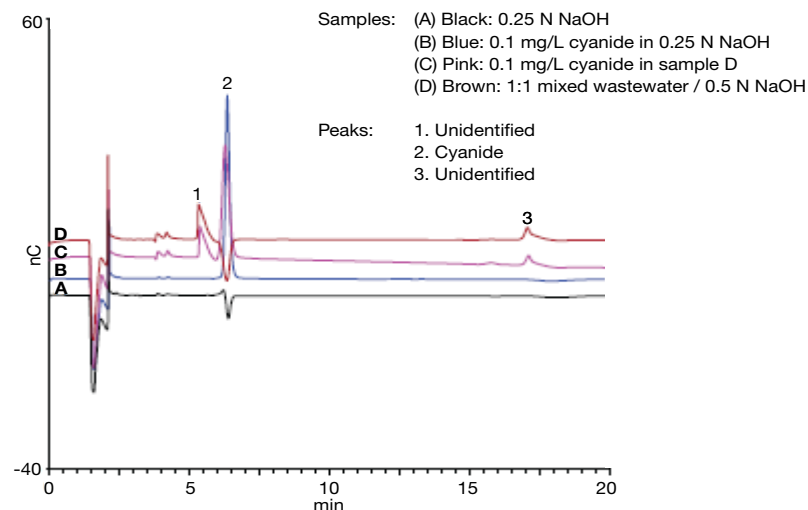
AOF by combustion IC—PFAS in aqueous samples

HPAE-PAD for determination of saccharides in atmospheric aerosol samples

Helpful resources and links

Free cyanide includes hydrogen cyanide (HCN) and cyanide (CN<sup>-</sup>) and is extremely toxic and regulated. IC coupled with electrochemical detection (ED) is used for the determination of cyanide, including direct determination of cyanide in strongly alkaline solutions using IC with DC amperometric detection. Unfortunately, the DC amperometry method exhibits electrode fouling problems over time.

This application note presents an updated IC method with pulsed amperometric detection (PAD) for the determination of cyanide in different absorbing solutions, including the 1.25 M NaOH solution specified in U.S. EPA Method 335.2 and the 0.25 M NaOH solution specified in U.S. EPA Method 335.4. Determination of cyanide in drinking water and wastewater using this method is also demonstrated. The improved method uses a 2 mm version of the Dionex IonPac AS7 column and a 50 µL injection.



Chromatograms of wastewater and cyanide-spiked wastewater

### Linearity and method detection limit for cyanide in different matrices

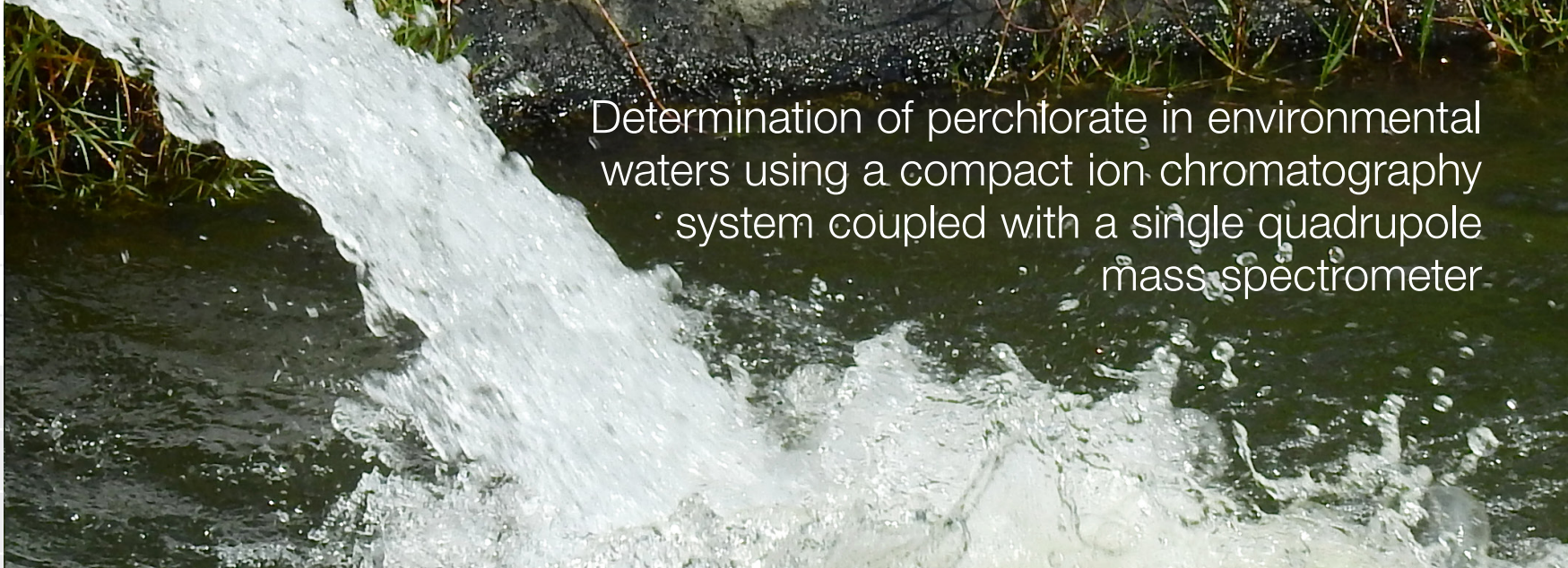
Analyte	Matrix	Injection volume (µL)	Range (mg/L)	Coefficient of determination (r <sup>2</sup> )*	Calculated MDL (mg/L)
Cyanide	Water	50	0.005–2	0.999	0.0001**
Cyanide	0.25 N NaOH	50	0.005–2	0.9999	0.01
Cyanide	1.25 N	50	0.005–2	0.9999	0.03

\*Quadratic fit

\*\*MDL = (t) × (S). Where t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees freedom (t = 3.14 for seven replicates). S = standard deviation of the replicate analyses.

For more information click [here](#)





# Determination of perchlorate in environmental waters using a compact ion chromatography system coupled with a single quadrupole mass spectrometer

Perchlorate has been used as an oxidizer in rockets, munitions, and fireworks since the 1950s. It has been found to cause thyroid dysfunction and has been linked to tumors in humans. Perchlorate is regulated under the Safe Drinking Water Act (2011), although the Maximum Contaminant Level Goal (MCLG) and Maximum Contaminant Level (MCL) have not yet been established. Determination of perchlorate in environmental samples has also gained the attention of the International Standards Organization (ISO).

IC with suppressed conductivity detection and electrospray ionization/mass spectrometry is one of the most sensitive and robust characterization methods available for perchlorate. MS provides lower detection limits in high-ionic-strength matrices than conductivity detection alone. MS also provides peak identification information for perchlorate by monitoring the appropriate mass/charge ratio (*m/z*).

## Method calibration and LOD for IC-MS of perchlorate

Name	Quantitation ion	Calibration method	Coefficient of determination ( <i>r</i> <sup>2</sup> )	LOD* (ng/L)
Perchlorate in deionized water over the range of 125–5000 ng/L	<i>m/z</i> 99	Internal standard	0.9994	20–60
		External standard	1.000	
Perchlorate in LSSM over the range of 400–5000 ng/L	<i>m/z</i> 101	Internal standard	0.9997	30–60
		External standard	0.9999	
Perchlorate in LSSM over the range of 125–5000 ng/L	<i>m/z</i> 99	Internal standard	0.9995	NA
		External standard	0.9996	
Perchlorate in LSSM over the range of 125–5000 ng/L	<i>m/z</i> 101	Internal standard	0.9993	30–60
		External standard	0.9999	

\*LOD = 3 × S/N

For more information click [here](#)



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**Perchlorate in environmental waters using IC-MS**

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Perchlorate in drinking water using ion chromatography

Polar pesticides in drinking water using IC-MS/MS

Validation of IC-MS/MS method for determining polar pesticides

Adsorbable organic halogen in wastewater using CIC

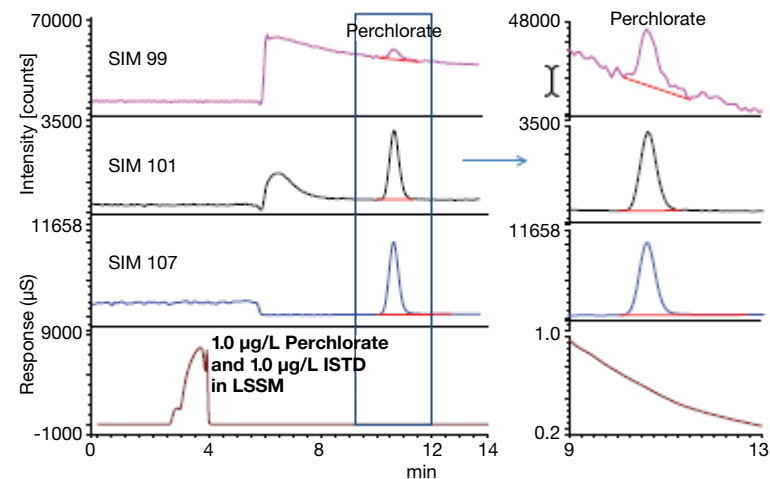
AOF by combustion IC—PFAS in aqueous samples

HPAE-PAD for determination of saccharides in atmospheric aerosol samples

Helpful resources and links



This application update revises AN151 using a Thermo Scientific™ Dionex™ IonPac™ AS20 column set, on a Dionex Integriion HPIC system, coupled with the ISQ EC single-quadrupole mass spectrometer. If compliance to U.S. EPA Method 332.0 is required, refer to [AN72587](#).



The separation of perchlorate in deionized water and Laboratory Synthetic Sample Matrix (LSSM, 1000 mg/L each chloride, carbonate, and sulfate) using a Dionex IonPac AS20 column set. 1 µg/L perchlorate in LSSM with enlarged box section.

For more information click [here](#)



# Determination of perchlorate by U.S. EPA Method 332.0 using a compaction chromatography system coupled with mass spectrometry

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- Cyanide in alkaline solutions using ion chromatography
- Perchlorate in environmental waters using IC-MS

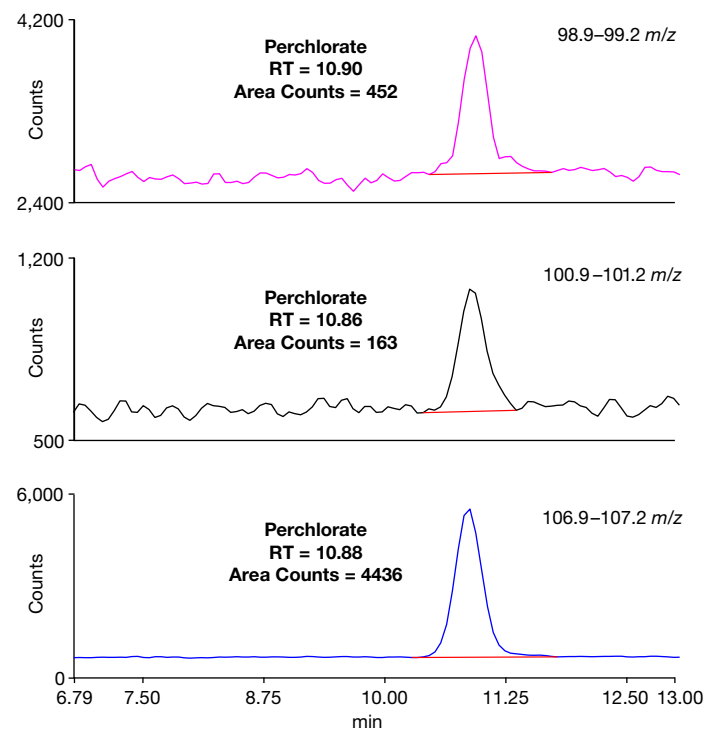
**Perchlorate by U.S. EPA Method 332.0 using IC-MS**

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- HPAE-PAD for determination of saccharides in atmospheric aerosol samples

Perchlorate has been used as an oxidizer in rockets, munitions, and fireworks since the 1950s. It has been found to cause thyroid dysfunction and has been linked to tumors. Perchlorate is regulated under the Safe Drinking Water Act (2011) although the Maximum Contaminant Level Goal (MCLG) and Maximum Contaminant Level (MCL) have not yet been established. Some U.S. states have promulgated enforceable standards for the levels of perchlorate in drinking water. Massachusetts and California have established standards for perchlorate levels in drinking water of 2 µg/L and 6 µg/L, respectively. More than 10 other states have also set advisory levels or health-based goals for perchlorate in drinking water ranging from 1 to 18 µg/L. This application note describes how to meet these levels using ion chromatography and single quad mass spectrometry.

**Single laboratory accuracy in drinking waters and external quality control samples**

Sample	Certified concentration (ng/L)	Measured concentration (ng/L)	Recovery (%)
Fortified drinking water 1	125	139	111
Fortified drinking water 2	1000	980	98.0
External quality control sample 1	500	478	95.6
External quality control sample 2	1000	1019	101.9



Mass chromatogram of a standard containing 125 ng/L perchlorate and 1.0 µg/L internal standard

For more information click [here](#)





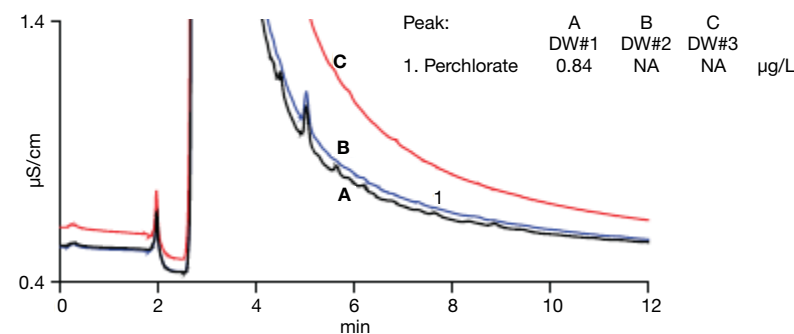
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- AOF by combustion IC—PFAS in aqueous samples
- HPAE-PAD for determination of saccharides in atmospheric aerosol samples

Perchlorate is widely used as the oxidizing component in solid propellants for rockets, munitions, and fireworks. However, perchlorate can contaminate soil, groundwater, and drinking water, leading to adverse health effects. The U.S. EPA placed perchlorate on the first Contaminant Candidate List (CCL1) in 1998 and determined that perchlorate meets the Safe Drinking Water Act's criteria for regulation as a contaminant in February 2011. However, perchlorate is still not federally regulated. Multiple analytical methods are available for perchlorate analysis. U.S. EPA Method 314.0 uses ion chromatography with suppressed conductivity detection.

This application note updates AU148 with use of a high-resolution Thermo Scientific™ Dionex™ IonPac™ AS16-4µm 2 mm column. In comparison to the conventional Thermo Scientific™ Dionex™ IonPac™ AS16 column, the Dionex IonPac AS16-4µm column exhibits higher peak efficiency while maintaining chromatographic selectivity. Using the Dionex IonPac AS16-4µm column increases the sample throughput by 20% and saves three minutes per injection, thereby improving productivity for perchlorate determinations in drinking water.

### Recoveries of perchlorate spiked in drinking waters

Drinking water	Amount found (µg/L)	Amount added (µg/L)	Recovery (%)
#1	0.840	4	99.6
#2	NA	4	104
#3	NA	4	98.8



Overlay of chromatograms of (A) DW #1 and (B) spiked DW #1

For more information click [here](#)





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Validation of IC-MS/MS method for determining polar pesticides

Adsorbable organic halogen in wastewater using CIC

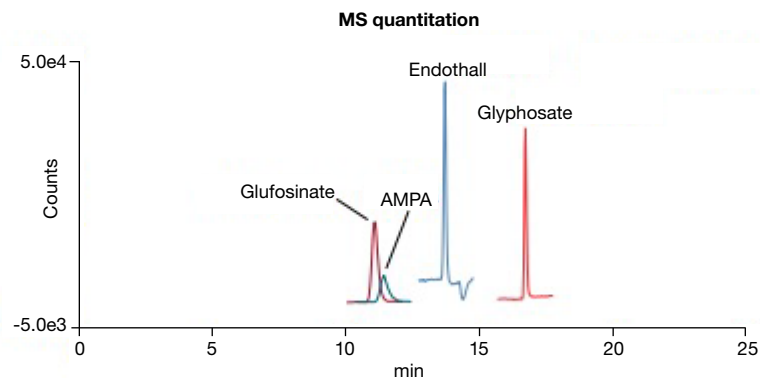
AOF by combustion IC—PFAS in aqueous samples

HPAE-PAD for determination of saccharides in atmospheric aerosol samples

Helpful resources and links

Glyphosate, the active ingredient in the herbicide Roundup™ has potential adverse effects on human health and although the bacteria in soil break it down into aminomethylphosphonic acid (AMPA), wastewater discharge and drinking water samples in the United States and Europe have tested positive for glyphosate. The determination of glyphosate and other polar compounds (including the herbicides endothall and glufosinate) presents a difficult analytical challenge due to their ionic structures, poor volatility, and absence of chromophores.

The objective of the present work was to develop a method that uses ion chromatography coupled to a single quadrupole mass spectrometer (IC-MS) to simultaneously determine the herbicides endothall, glyphosate, AMPA, and glufosinate. Using the method developed, water samples were directly injected for analysis, and chromatographic separation was achieved in 25 min. Performance data for the method such as recovery, precision, sensitivity, and calibration range were also reported. Together these data show that the IC-MS can successfully determine the four targeted analytes in drinking water samples.



Drinking water spiked with 10 ppb each of four pesticides

**Method detection limits (MDL) and limits of detection (LOD), DL, and LCMRL**

	LOD (µg/L)	MDL (µg/L)	DL (µg/L)	LCMRL (µg/L)	Standards used to determine LCMRL (µg/L)
<b>Endothall</b>	0.33	0.33	0.36	0.40	0.4, 0.5, 1, 2
<b>Glyphosate</b>	0.08	0.10	0.11	0.34	0.2, 0.3, 0.4, 0.5
<b>AMPA</b>	0.31	0.32	0.43	0.75	0.4, 0.5, 1, 2
<b>Glufosinate</b>	0.16	0.13	0.13	0.31	0.3, 0.4, 0.5, 1

For more information click [here](#)





# Validation of an IC-MS/MS method for the determination of polar compounds at ng/L level in water samples

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Perchlorate in drinking water using ion chromatography

Polar pesticides in drinking water using IC-MS/MS

**Validation of IC-MS/MS method for determining polar pesticides**

Adsorbable organic halogen in wastewater using CIC

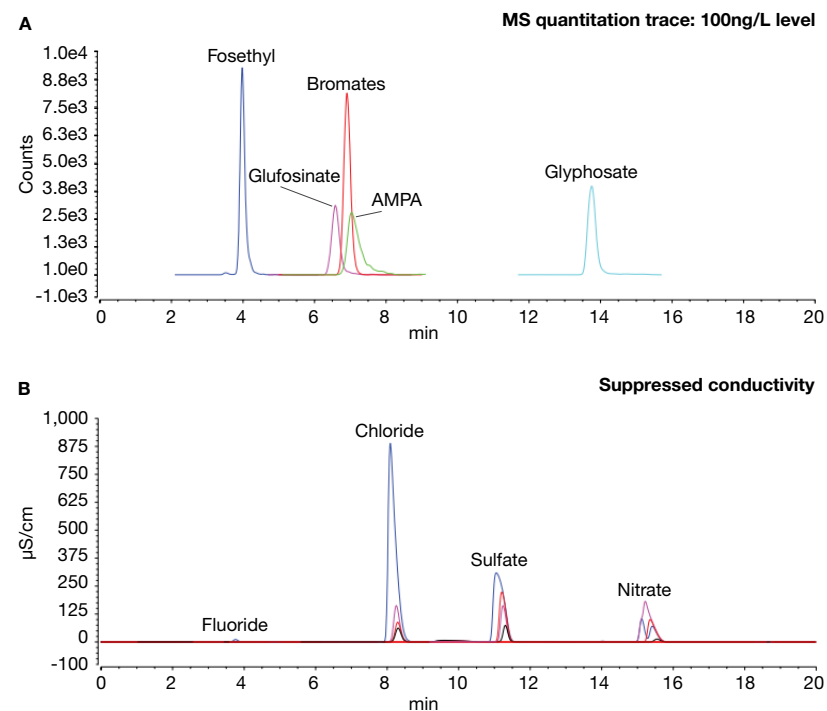
AOF by combustion IC—PFAS in aqueous samples

HPAE-PAD for determination of saccharides in atmospheric aerosol samples

Helpful resources and links

The analysis of glyphosate and other polar anionic pesticides and their metabolites presents an analytical challenge. Recent developments in the hyphenation of IC and MS has facilitated novel options to determine polar pesticides. IC is the preferred separation technique for polar ionic analytes, such as anions, cations, ionic metabolites, and sugars. In triple quadrupole MS/MS systems, MS offers low detection limits and high detection selectivity when operated in selected reaction monitoring (SRM) mode.

This work aimed to develop and validate an IC-MS/MS method for direct analysis of polar ionic pesticides in different water samples and assess the applicability under routine conditions. This method allows a direct injection of 30  $\mu$ L of different types of water samples to determine aminomethylphosphonic acid (AMPA), 2-chloro-4-methylsulfonylbenzoic acid (CMBA), ethephon, fosetylaluminum, glyphosate, glufosinate, maleic hydrazide, and bromate in a single chromatographic run, without derivatization or a concentration step.



Chromatographic separation of fosetyl-aluminum, glufosinate, AMPA, bromate, and glyphosate—100 ng/L standard in Evian water (A), and overlay of the conductivity traces for the four water matrices selected for validation (B)

For more information click [here](#)



# Determination of adsorbable organic halogen in wastewater using a combustion ion chromatography system

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Perchlorate by U.S. EPA Method 332.0 using IC-MS

Perchlorate in drinking water using ion chromatography

Polar pesticides in drinking water using IC-MS/MS

Validation of IC-MS/MS method for determining polar pesticides

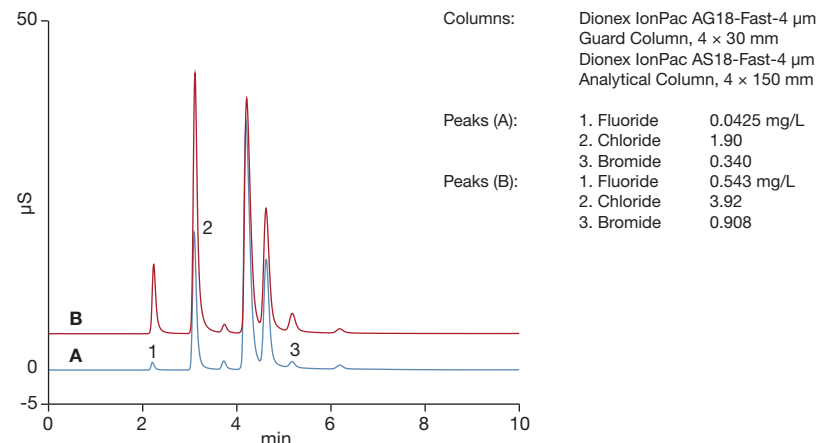
**Adsorbable organic halogen in wastewater using CIC**

AOF by combustion IC—PFAS in aqueous samples

HPAE-PAD for determination of saccharides in atmospheric aerosol samples

Helpful resources and links

Organohalogens are toxic and persistent compounds that are often given high priority in the monitoring of environmental pollution. Two of the most frequently used sum parameters for organohalogens—adsorbable organic halogens (AOX) and extractable organic halogens (EOX)—are general indicators of industrial pollution. AOX represents the equivalent amount of fluorine, chlorine, and bromine contained in organic compounds, expressed as chloride substances that can be adsorbed from water onto activated carbon. Organic halogen compounds cannot be directly analyzed by IC, therefore, in this note automated combustion ion chromatography (CIC) is used to determine these organic halogen contaminants. In CIC, the samples are first combusted, and the resultant gases are released into an absorption solution, which is directly injected into an IC.



### Recoveries of AOX spiked in wastewaters (n = 3)

Analyte	Wastewater 1			Wastewater 2			Wastewater 3		
	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 (%)
Fluoride	1.71	101	103	2.69	101	104	8.86	101	103
Chloride	315	400	102	80.5	100	103	399	400	98.3
Bromide	293	230	103	27.3	115	102	68.7	115	104

For more information click [here](#)



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HPAE-PAD for determination of saccharides in atmospheric aerosol samples

Helpful resources and links

Perfluoroalkyl and polyfluoroalkyl substances (PFAS) are used in a wide variety of consumer products and found in firefighting foams, which are primary groundwater contaminants at airports and military bases. Living organisms can accumulate PFAS compounds in their tissue,

which can build up over time and impact their health. Several national and international standardization and regulatory bodies have developed methods specifically for the determination of PFAS in aqueous samples.

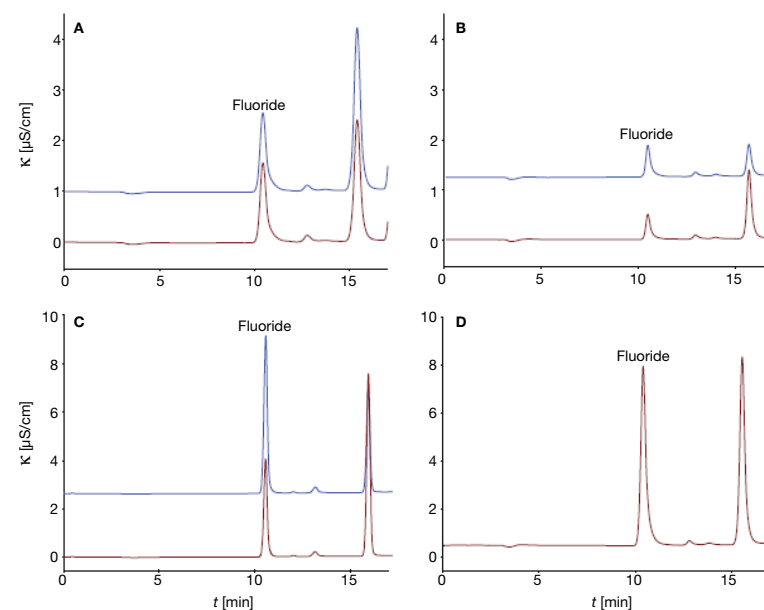
These methods are mostly based on solid-phase extraction (SPE) followed by LC-MS/MS detection for only a select number of PFAS. Conventional IC cannot directly analyze non-ionic PFAS. Automated combustion ion chromatography (CIC), however, is used to determine organic halogen contaminants, such as adsorbable organically bound halogens (AOX).

### Recovery data for perfluorobutanesulfonate (PFBS) and 4-fluorobenzoic Acid (4-FBA)

Sample	Surface water		Wastewater		
	PFBS recovery	Sample	PFBS recovery	4-FBA recovery	Sample
1	94	1	102	90	
2	105	2	91	83	
3	99	3	86	82	
4	92	4	89	ND	
5	109	5	93	ND	
6	98	6	85	83	
7	98	7	94	127	
8	99				

For more information click [here](#)

This customer application note describes an adsorbable organic fluorine (AOF)-CIC method, as a complement to existing approaches, that provides an easy-to-use and economically attractive way to generate a cumulative parameter and can help to optimize the utilization of the more expensive LC-MS/MS and GC-MS/MS instrumentation by selecting and only analyzing “suspicious” samples.



Selection of representative chromatograms obtained after adsorption on activated carbon and combustion; (A) municipal wastewaters, (B) groundwaters, (C) surface waters, (D) industrial wastewater (diluted 1 to 10)



# An HPAE-PAD method for determination of saccharides in atmospheric aerosol samples

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- Perchlorate by U.S. EPA Method 332.0 using IC-MS
- Perchlorate in drinking water using ion chromatography
- Polar pesticides in drinking water using IC-MS/MS

- Validation of IC-MS/MS method for determining polar pesticides
- Adsorbable organic halogen in wastewater using CIC
- AOF by combustion IC—PFAS in aqueous samples

**HPAE-PAD for determination of saccharides in atmospheric aerosol samples**

Helpful resources and links

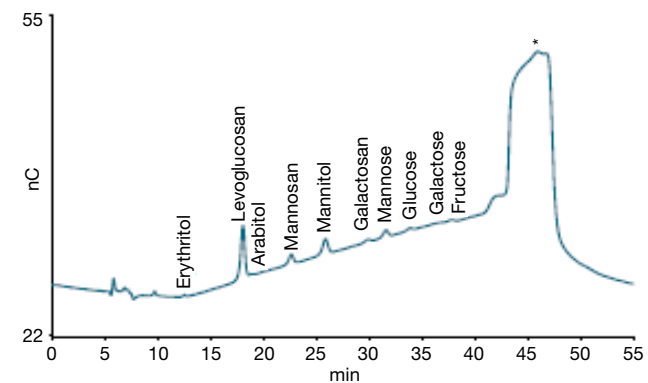
Anhydro sugars, sugar alcohols, monosaccharides, and disaccharides are among the major classes of water-soluble organic compounds (WSOC) in atmospheric aerosols. Atmospheric saccharides originate from two main sources: biomass burning (natural or anthropogenic) and natural biogenic detritus. Saccharides have been proposed as markers to evaluate the contribution of these sources to atmospheric aerosols. Anhydro sugars levoglucosan, mannosan, and galactosan originate from the combustion

of cellulose and hemicellulose due to wildfires or residential wood burning. High performance anion-exchange chromatography with pulsed amperometric detection (HPAE-PAD) has been used to determine saccharides from atmospheric aerosol samples. HPAE is capable of separating complex mixtures of carbohydrates, and PAD is a powerful detection technique with a broad linear range and very low detection limits.

**Spike recovery studies (n = 3)**

Recovery (%)										
Erythritol	Xylitol	Levoglucosan	Mannosan	Mannitol	Galactosan	Mannose	Glucose	Galactose	Fructose	Sucrose
<b>Spike 0.5 mg/L</b>										
89.8	93.7	89.9	86.1	89.5	82.3	86.9	99.4	92.1	116	112
<b>Spike 5 mg/L</b>										
98.5	99.7	100.5	98.3	99.7	98.0	99.4	100	99.8	103	108

This application note describes a single column method using HPAE-PAD to determine anhydro sugars, simple sugars, and sugar alcohols present in atmospheric aerosol samples. Using a Thermo Scientific™ Dionex™ CarboPac™ MA1 column, 11 saccharides can be resolved in a single 55 min method. Additionally, three sugar alcohols can be resolved by modifying the initial eluent concentration, taking the total number of analytes that can be resolved to 14.



Simulated atmospheric aerosol sample analyzed using Method A described in the text (\*sucrose present in the sample matrix)

For more information click [here](#)



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