

## Analysis of Inorganic Anions in Drinking Water According to EPA Method 300.1 Using Nexera IC Ion Chromatograph -Part B-

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### User Benefits

- ◆ Nexera IC system is suitable for analyzing drinking water in compliance with EPA Method 300.1 (Part B).
- ◆ Utilizing optimized conditions for the Shim-pack™ IC-SA3 allows for the separation of four inorganic disinfection by-products in under 16 minutes.
- ◆ Employing an electroalytic suppressor with superior baseline suppression enables highly sensitive analysis.

### Introduction

Reliable determination of inorganic anions in drinking water is essential for regulatory compliance and protection of public health. In the United States, the Environmental Protection Agency (EPA) provides methods for the analysis of inorganic anions in water by ion chromatography in Method 300.1<sup>1)</sup> (EPA Method 300.1).

The anion suppressor installed in the Nexera IC (Fig. 1) removes conductive ions from the eluent before detection, reducing background and noise and enhancing analyte signals.

In this report, we introduce examples of analysis of four inorganic disinfection by-products (DBPs) in accordance with EPA Method 300.1 Part B using the Nexera IC.



Fig. 1 Nexera™ IC

### EPA Method 300.1

In EPA Method 300.1, a total of 10 types of anions are subject to measurement, which are divided into common anions (Part A) and inorganic disinfection by-products (Part B) according to the difference in designated injection amounts.

Disinfection by-products listed in Part B include Chlorite (ClO<sub>2</sub>), Bromate (BrO<sub>3</sub>), Bromide (Br) and Chlorate (ClO<sub>3</sub>). Among them, Br is included in both Part A because of its importance as a common anion and Part B because of its important role as a disinfection by-product precursor.

For the surrogate solution, dichloroacetic acid (DCA) was used. And ethylenediamine (EDA) was spiked as the preservation solution.

### Samples

**a) Standard Solutions:** Mixed commercially available standard solutions for ion chromatography and diluted them with ultra-pure water to prepare six-point calibration samples (STD1–6). DCA and EDA was added to all standard solutions at a final concentration of 1 mg/L and 50 mg/L respectively.

**b) Analytical Samples:** Two types of commercially available mineral water (Mineral Water S and Mineral Water C), as well as tap water.

**c) Continuing Calibration Check Standard:** Same solution as standard solution STD3.

### Flow Chart

Fig. 2 shows the flow chart of Nexera IC.

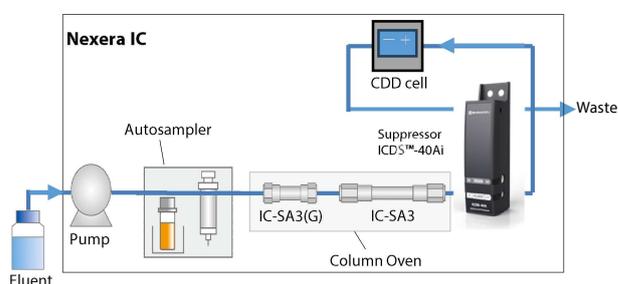


Fig. 2 The Flow Chart of Nexera IC

### Analytical Conditions

By optimizing the concentration and flow rate of the eluent for the Shim-pack IC-SA3, we achieved analysis completion within 16 minutes. Table 1 shows the analytical conditions.

Table 1 Analytical Conditions

Column	: Shim-pack IC-SA3 <sup>*1</sup>
Guard column	: Shim-pack IC-SA3 (G) <sup>*2</sup>
Mobile phase	: 4.5 mmol/L Sodium Carbonate
Flow rate	: 0.85 mL/min
Column temp.	: 40 °C
Injection volume	: 200 µL
Vial	: Shimadzu Vial, LC, 4 mL, Polypropylene <sup>*3</sup>
Detection	: Conductivity

\*1 P/N : 228-41600-91

\*2 P/N : 228-41600-92

\*3 P/N : 228-31537-91

### Analysis of Standard Solutions

The Quality Control (QC) of EPA Method 300.1 requires prior confirmation of method detection limit (MDL), linearity and recovery to evaluate column, analytical methods and system performance.

Table 2 indicates the concentrations of each calibration standard and coefficient of determination (r<sup>2</sup>). All anions' coefficients of determination exceeded 0.999, meeting the QC standards outlined in the EPA Method 300.1. Fig. 3 presents the results of a mixed standard solution (STD3) of Part B.

Table 2 The Linear and Calibrations' Concentration of Each Anion

Anions (µg/L)	ClO <sub>2</sub>	BrO <sub>3</sub>	Br	ClO <sub>3</sub>
STD1	2	2	2	10
STD2	5	5	5	25
STD3	10	10	10	50
STD4	50	50	50	250
STD5	100	100	100	500
STD6	200	200	200	1000
r <sup>2</sup>	0.9999	0.9999	0.9999	0.9996

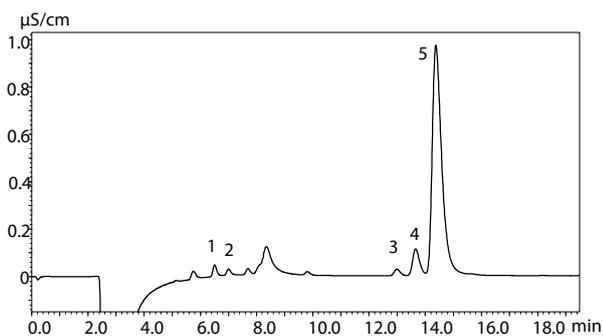


Fig. 3 The Chromatogram of Standard Solution (STD 3)  
(Peak: 1 ClO<sub>2</sub>, 2 BrO<sub>3</sub>, 3 Br, 4 ClO<sub>3</sub>, 5 DCA)

### Method Detection Limit

The MDL was calculated as  $(t) \times (S)$  after preparing the MDL standard solution according to the procedure described in EPA Method 300.1 and performing seven consecutive analyses. The formula is shown in Table 3, where  $t$  is student's  $t$  value for 99% confidence level ( $t=3.14$  for seven replicates) and  $S$  is standard deviation from seven injections. The results of MDL standard solution are shown in Fig. 4 and Table 4.

MDLs for the target anions ranged from 0.3 to 0.6 μg/L, values well below the corresponding Maximum Contaminant Level (MCL)<sup>2</sup> for chlorite (1.0 mg/L) and bromate (0.010 mg/L).

Table 3 Formula of MDL

$$MDL=(t) \times (s)$$

$t$ =Student's  $t$  value for  $n-1$  degrees of freedom at the 99% confidence level;  
 $t = 3.143$  for six degrees of freedom  
 $s$  = standard deviation of the replicate analyses

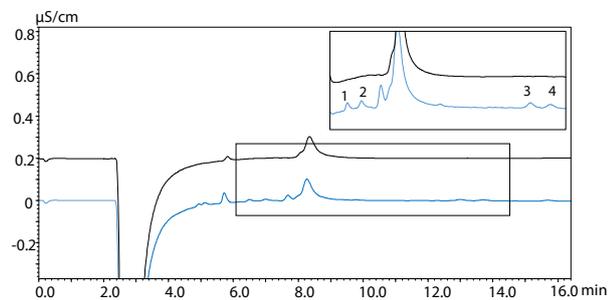


Fig. 4 Analysis Result of MDL Standard Solution (Blue Line) and Blank (Black Line) (Peak: 1 ClO<sub>2</sub>, 2 BrO<sub>3</sub>, 3 Br, 4 ClO<sub>3</sub>)

Table 4 MDL Standard Concentrations and the Calculated MDLs (n=7)

Anions	MDL Standard Concentration (mg/L)	Standard Deviation SD	MDL (mg/L)
ClO <sub>2</sub>	2	0.08	0.3
BrO <sub>3</sub>	2	0.2	0.6
Br	2	0.1	0.3
ClO <sub>3</sub>	2	0.2	0.5

### Sample Analysis and Recoveries

We analyzed three types of samples (tap water, mineral water S, and mineral water C) and performed seven consecutive analyses following the procedures according to EPA Method 300.1. As a pre-treatment, reagents were added to the sample to adjust the DCA concentration to 1 mg/L and the EDA concentration to 50 mg/L.

The results of the sample determinations are summarized in Table 5.

Table 5 Analysis Results of Analytical Samples (n=7)

Anions	Tap Water		Mineral Water S		Mineral Water C	
	Mean Conc. (mg/L)	%RSD	Mean Conc. (mg/L)	%RSD	Mean Conc. (mg/L)	%RSD
ClO <sub>2</sub>	N.D.		N.D.		N.D.	
BrO <sub>3</sub>	N.D.		N.D.		4.2	3.41
Br	14.7	0.64	35.2	0.34	N.D.	
ClO <sub>3</sub>	31.5	0.45	N.D.		N.D.	

\* N.D.: <MDL

The performance and accuracy of EPA Method 300.1 were confirmed by spike recovery tests. Samples were evaluated by spiking a standard solution.

Recoveries were calculated according to EPA Method 300.1. The formula is shown in Table 6. The recoveries obtained from the analysis of the fortified samples are summarized in Table 7.

The spike recoveries of all samples were within the range of 92–111%, which meet the QC requirement range of 75–125%. Furthermore, the recoveries of DCA as surrogate were also fell within the acceptable range of 90–115% as per QC requirements.

Fig. 5 shows chromatograms of tap water and its spiked sample, and Fig. 6 shows chromatograms of mineral water S and its spiked sample and Fig. 7 shows chromatograms of mineral water C and its spiked sample.

Table 6 Formula of Samples' Recovery

$$R = \frac{C_F - C}{F} \times 100$$

$R$  = percent recovery

$C_F$  = fortified sample concentration

$C$  = sample background concentration

$F$  = concentration equivalent added to sample

Table 7 The Results of Spike Recovery Test (n=7)

Anions	Spike Conc. (mg/L)	Tap water		Mineral Water S		Mineral Water C	
		Recovery %	%RSD	Recovery %	%RSD	Recovery %	%RSD
ClO <sub>2</sub>	10	100.4	0.78	101.4	1.22	95.3	1.33
BrO <sub>3</sub>	10	101.1	1.12	96.1	1.57	92.3	0.48
Br	10	109.0	0.34	93.4	0.30	111.4	0.88
ClO <sub>3</sub>	50	95.9	0.10	98.0	0.20	97.6	0.14
DCA	1000	101.9	0.08	104.3	0.06	97.6	0.08

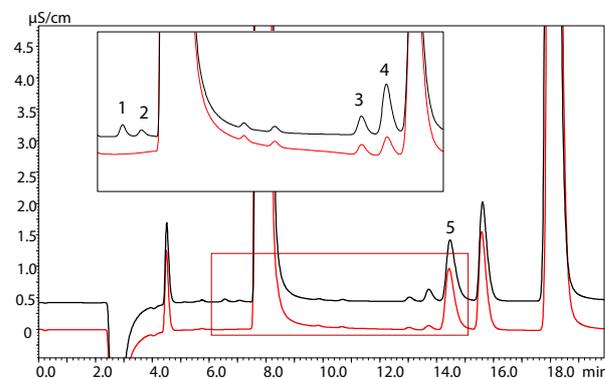


Fig. 5 The Chromatograms of Tap Water (Red Line) and Spiked Sample (Black Line)  
(Peak: 1 ClO<sub>2</sub>, 2 BrO<sub>3</sub>, 3 Br, 4 ClO<sub>3</sub>, 5 DCA)

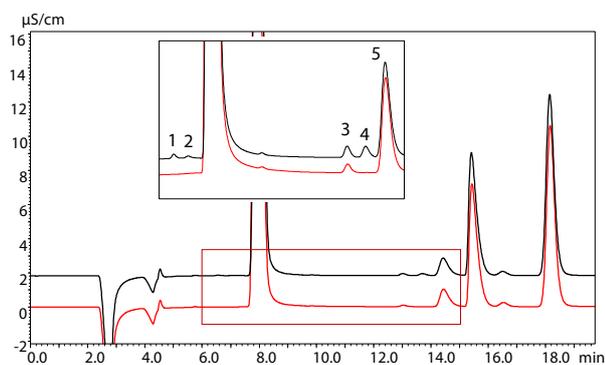


Fig. 6 The Chromatograms of Mineral Water S (Red Line) and Spiked Sample (Black Line)  
(Peak: 1 ClO<sub>2</sub>, 2 BrO<sub>3</sub>, 3 Br, 4 ClO<sub>3</sub>, 5 DCA)

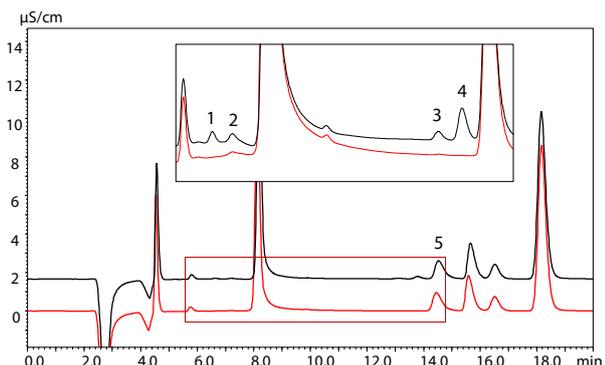


Fig. 7 The Chromatograms of Mineral Water C (Red Line) and Spiked Sample (Black Line)  
(Peak: 1 ClO<sub>2</sub>, 2 BrO<sub>3</sub>, 3 Br, 4 ClO<sub>3</sub>, 5 DCA)

### ■ Calibration Curve Stability

To evaluate continuous analysis stability, EPA Method 300.1 requires confirmation of the efficacy of calibration curves during analysis by analyzing the continuing calibration check standard (STD3) for every ten samples.

In this time, the samples, including the continuing calibration check standard, were analyzed for approximately 60 hours. The fluctuation of the concentrations obtained from the continuous analysis (analysis concentration / set concentration × 100) is shown in Fig. 8.

The fluctuation of all measured anions during the analysis were within 100±10%, the fluctuations were fell within the acceptable range of 85–115% (red dot line) as per QC requirements, confirming the validity of the calibration curve throughout the analysis period.

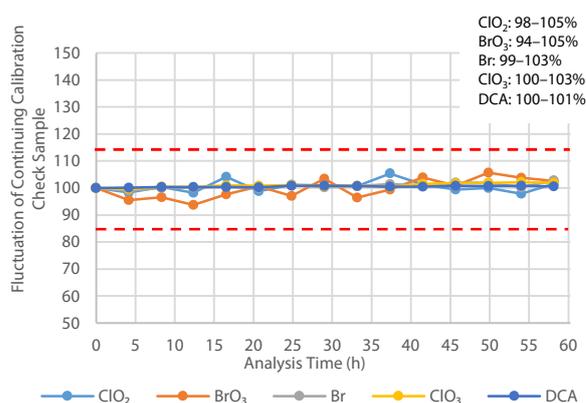


Fig. 8 The Fluctuation of the Continuing Calibration Check Standard over Approximately 60 hours (Enlarged View)

### ■ Conclusion

Using the Nexera IC system, anion analysis was implemented in Part B in accordance with EPA Method 300.1. Four target anions were successfully separated on a Shim-pack IC SA3 column and detected by conductivity detection, with a total analysis time of less than 16 minutes.

The Nexera IC demonstrated outstanding analytical performance, as DCA recoveries consistently met the quality control acceptance criteria (90–115%). Spike recovery experiments further confirmed the robustness and accuracy of the method, yielding excellent recoveries in the range of 92–111% with very low precision values (%RSD < 1.57). In addition, continuing calibration check standards remained stable and within 100 ± 10% throughout the entire study period.

All method verification tests produced satisfactory results, confirming full compliance with EPA Method 300.1 and highlighting the high reliability and reproducibility of the Nexera IC platform.

#### <References>

- 1) EPA Method 300.1 Determination of inorganic anions in drinking water by ion chromatography Revision 1.0
- 2) [National Primary Drinking Water Regulations](#) (2026. 02)

#### <Related Applications>

1. US EPA 300 Method-Compliant Environmental and Water Analysis, [Application News No. L553](#)
2. Analysis of inorganic anions in tap water according to EPA Method 300.1 using Ion Chromatography, [Application News 1-00487](#)
3. Analysis of Inorganic Anions in Drinking Water According to EPA Method 300.1 Using Nexera IC -Part A-, [Application News 1-01104](#)

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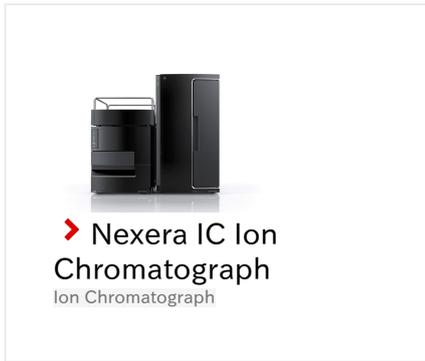
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