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Application News

Nexera™ IC

Analysis of Inorganic Anions in Drinking Water According to EPA Method 300.1 Using Nexera IC -Part A-

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User Benefits

- Nexera IC system is suitable for analyzing drinking water in compliance with EPA Method 300.1 (Part A).
- Utilizing optimized conditions for the Shim-pack™ IC SA3 allows for the separation of seven inorganic anions in under 20 minutes.
- Employing an electrodialytic 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[1)] (EPA Method 300.1).

The anion suppressor installed in Nexera IC (Fig.1) is designed to improve sensitivity by replacing sodium ions in the eluent with hydrogen ions before detection. In this report, we introduce examples of analysis of seven inorganic anions in accordance with EPA Method 300.1 Part A 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.

Seven common anions listed in Part A include Fluoride (F), Chloride (Cl), Nitrite as N (NO_2-N), Bromide (Br), Nitrate as N (NO_3-N), Phosphate as P (PO_4-P) and Sulfate (SO_4).

For the surrogate solution, 0.50 mg/mL dichloroacetic acid (DCA) was used. All samples were added DCA to achieve a final concentration of 1 mg/L.

■ Samples

a) Standard Solutions: Mixed commercially available standard solutions for ion chromatography and diluted them with ultra-pure water to prepare five-point calibration samples (STD1~5). DCA was added to all standard solutions at a final concentration of 1 mg/L.

b) Analytical Samples: Two types of commercially available mineral water (Mineral Water S and Mineral Water C), as well as tap water. DCA was added to all analytical samples at a final concentration of 1 mg/L.

c) Continuing Calibration Check Standard: Mixed commercially available standard solutions for ion chromatography and diluted them with ultra-pure water to prepare a standard sample with the same concentration as STD3.

■ Flow Chart

Fig. 2 shows the flow chart of Nexera IC.

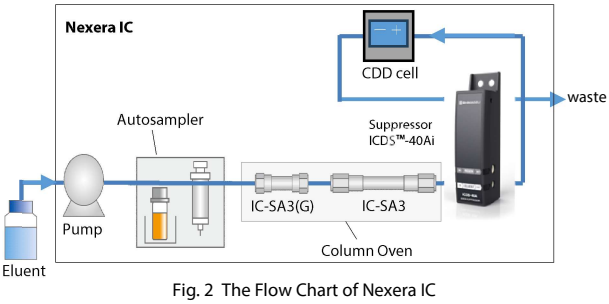

Fig. 2 The Flow Chart of Nexera IC

■ Analytical Conditions

By optimizing the concentration and flow rate of the eluent for the Shim-pack IC-SA3, we achieved analysis completion within 20 minutes. Table 1 shows the analytical conditions.

Table 1 Analytical Conditions

Column	:	Shim-pack IC-SA3 *1
Guard column	:	Shim-pack IC-SA3 (G) *2
Mobile phase	:	4.5 mmol/L Sodium Carbonate
Flow rate	:	0.85 mL/min
Column temp.	:	40 °C
Injection volume	:	50 µL
Vial	:	Shimadzu Vial, LC, 4 mL, Polypropylene*3
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 relevant coefficients (r^2). All anions had all relevant coefficients 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 A.

Table 2 The Linear and Calibrations' Concentration of Each Anion

Anions	F	Cl	NO_2-N	Br	NO_3-N	PO_4-P	SO_4
	Conc. of Calibration Standards (mg/L)	Conc. of Calibration Standards (mg/L)	Conc. of Calibration Standards (mg/L)	Conc. of Calibration Standards (mg/L)	Conc. of Calibration Standards (mg/L)	Conc. of Calibration Standards (mg/L)	Conc. of Calibration Standards (mg/L)
STD1	0.01	0.2	0.01	0.04	0.01	0.05	0.1
STD2	0.10	2.0	0.10	0.40	0.10	0.50	1.0
STD3	0.50	10	0.50	2.00	0.50	2.50	5.0
STD4	0.75	15	0.75	3.00	0.75	3.74	7.5
STD5	1.00	20	1.00	4.00	1.00	5.00	10
Relevant Coefficients (r^2)	0.9999	0.9995	0.9998	0.9999	0.9999	0.9995	0.9995

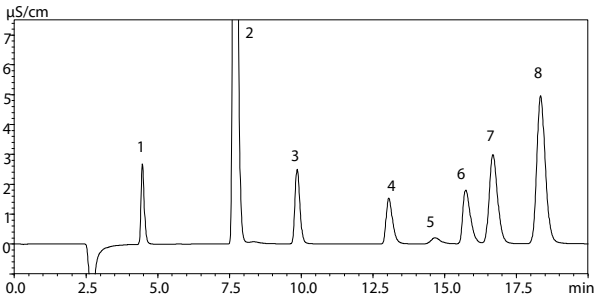

Fig. 3 Chromatogram of Standard Solution (STD 3)
(Peak 1 F, 2 Cl, 3 NO_2-N, 4 Br, 5 DCA, 6 NO_3-N, 7 PO_4-P, 8 SO_4)

■ Method Detection Limit

The MDL was calculated as (t) x (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 ranged from 0.0004 to 0.003 mg/L, values well below the corresponding Maximum Contaminant Level (MCL)[2] for fluoride, nitrite and nitrate.

Table 3 Formula of MDL

MDL=(t)×(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

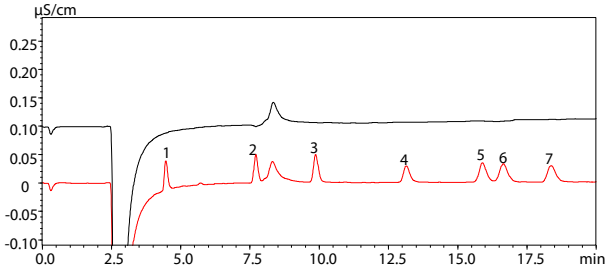

Fig. 4 Analysis Result of MDL Standard Solution (Red Line) and Blank (Black Line)
(Peak 1 F, 2 Cl, 3 NO_2-N, 4 Br, 5 NO_3-N, 6 PO_4-P, 7 SO_4)

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

Anions	MDL Standard Concentration (mg/L)	Standard Deviation SD	MDL (mg/L)
F	0.01	0.0004	0.001
Cl	0.02	0.0002	0.0005
NO_2-N	0.01	0.0002	0.0006
Br	0.04	0.0004	0.001
NO_3-N	0.01	0.0001	0.0004
PO_4-P	0.03	0.0006	0.002
SO_4	0.03	0.0009	0.003

■ 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, 0.2 mL of DCA was added to 100 mL of each sample. 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
F	0.074	1.20	0.071	0.56	0.253	0.16
Cl	15.3	0.03	3.42	0.07	0.9246	0.05
NO_2-N	N.D.		N.D.		N.D.	
Br	0.012	2.32	0.009	3.36	N.D.	
NO_3-N	0.038	0.79	0.689	0.06	0.126	0.15
PO_4-P	N.D.		0.044	0.56	0.153	0.26
SO_4	7.87	0.05	4.16	0.09	2.06	0.26
DCA	1.05	0.39	1.06	0.18	1.09	0.11

* 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 100±10%, 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
F	0.50	99.0	0.16	100.0	0.38	102.0	0.87
Cl	10	100.1	0.04	101.0	0.03	99.6	0.04
NO_2-N	0.50	98.9	0.08	98.8	0.13	98.9	0.16
Br	2.0	98.4	0.04	99.7	0.10	99.5	0.09
NO_3-N	0.50	98.2	0.04	101.2	0.09	98.3	0.23
PO_4-P	2.50	96.9	0.03	96.8	0.10	97.3	0.09
SO_4	5.0	104.3	0.06	102.3	0.03	99.6	0.05
DCA	1.0	92.1	0.45	93.0	0.25	94.3	0.43

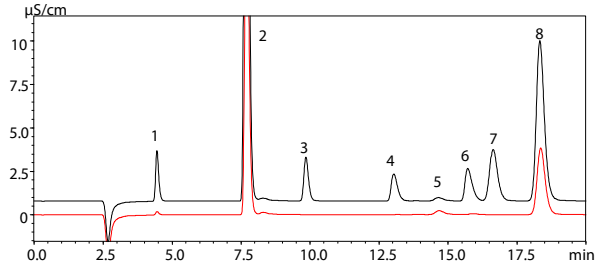

Fig. 5 The Chromatograms of Tap Water (Red Line) and Spiked Sample (Black Line)
(Peak 1 F, 2 Cl, 3 NO_2-N, 4 Br, 5 DCA, 6 NO_3-N, 7 PO_4-P, 8 SO_4)

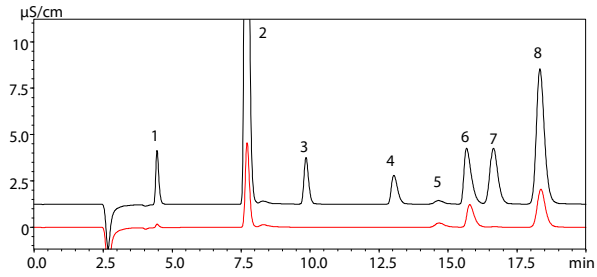

Fig. 6 The Chromatograms of Mineral Water S (Red Line) and Spiked Sample (Black Line)
(Peak 1 F, 2 Cl, 3 NO_2-N, 4 Br, 5 DCA, 6 NO_3-N, 7 PO_4-P, 8 SO_4)

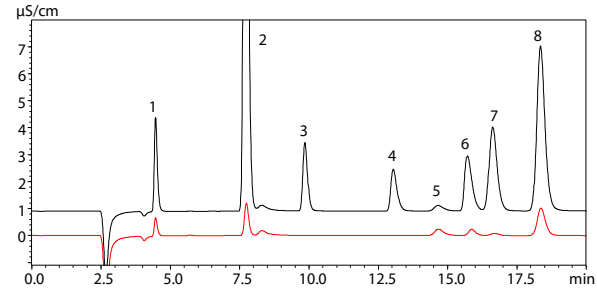

Fig. 7 The Chromatograms of Mineral Water C (Red Line) and Spiked Sample (Black Line)
(Peak 1 F, 2 Cl, 3 NO_2-N, 4 Br, 5 DCA, 6 NO_3-N, 7 PO_4-P, 8 SO_4)

■ Continuous Analysis

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 16 hours. The fluctuation of the concentrations obtained from the continuous analysis (analysis concentration / set concentration x 100) is shown in Fig. 8 and Fig. 9. The fluctuation of all measured anions during the analysis were within 100±10%, confirming the validity of the calibration curve throughout study period.

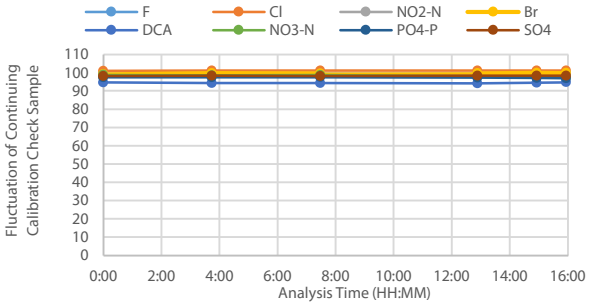

Fig. 8 The Fluctuation of the Continuing Calibration Check Standard over Approximately 16 hours

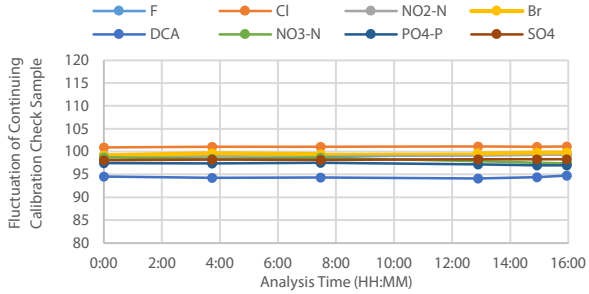

Fig. 9 The Fluctuation of the Continuing Calibration Check Standard over Approximately 16 hours (Enlarged View)

■ Conclusion

Using the Nexera IC, we introduced the analysis of anions in Part A in accordance with EPA Method 300.1. Seven anions were separated using a Shim-pack IC-SA3 and analyzed with a conductivity detector, and the total analysis time was within 20 minutes. The results showed that DCA recoveries fell within the QC acceptance criteria (90–115%). Spike recovery tests demonstrated excellent recoveries (97–104%) with low %RSD (<0.87). The continuing calibration check standards remained within 100 ± 10% throughout the study period. All verification tests yielded satisfactory results, confirming compliance with EPA Method 300.1.

<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 No. 01-00487

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