

ASTM D4327-03 Compliant Analysis of Anions in Drinking Water

T. Kaseda

User Benefits

- ◆ Enables acquisition of data with excellent linearity and repeatability in ASTM D4327-03 compliant analyses of anions.
- ◆ Applicable to anion analyses of not only drinking water, but also tap water.

Introduction

The ion chromatograph is widely used in detection and quantitative analysis of ion components in aqueous solutions. ASTM D4327-03⁽¹⁾ issued by ASTM International (the United States) specifies the test methods for analysis of 7 species of anions in drinking water or wastewater by suppressed ion chromatography.

This article introduces an ASTM D4327-03 compliant analysis of anions in drinking water using a Shimadzu HIC-ESP anion suppressor ion chromatograph. (Application News 01-00101-EN introduces an example of anion analysis of wastewater.)

Analysis of Standard Solution

Fig. 1 shows the obtained chromatogram of standard 7 anions specified in ASTM D4327-03 (fluoride ion, chloride ion, nitrite ion, bromide ion, nitrate ion, phosphate ion, sulfate ion), and Table 1 shows the analytical conditions.

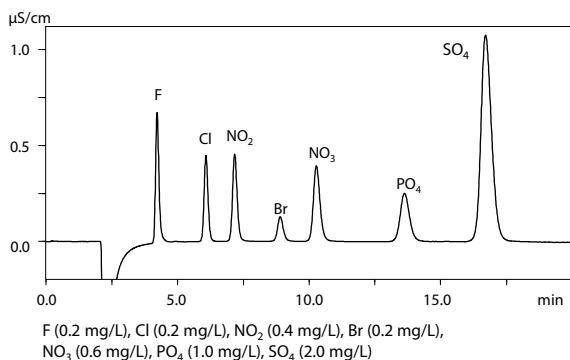


Fig. 1 Chromatogram of Anion Standard Solution

Table 1 Analysis Conditions

Column	: Shim-pack™ IC-SA2 ^{*1} (250 mm × 4.0 mm I.D., 9 µm)
Guard Column	: Shim-pack IC-SA2(G) ^{*2} (10 mm × 4.6 mm I.D., 9 µm)
Mobile phase	: 0.6 mmol/L Sodium Carbonate 12.0 mmol/L Sodium Hydrogen Carbonate
Flow rate	: 1.0 mL/min
Column temp.	: 30 °C
Injection volume	: 50 µL
Vial	: Shimadzu Vial, LC, 4 mL, Polypropylene ^{*3}
Detection	: Conductivity (CDD-10A VP)

*1 P/N: 228-38983-91

*2 P/N: 228-38983-92

*3 P/N: 228-31537-91

Linearity and Repeatability

A 4-level calibration curve was created for each of the 7 anions that are the targets of analysis in ASTM D4327-03 within a concentration range conforming to the standard. Fig. 2 shows the obtained calibration curves. All the coefficients of determination (r^2) of the calibration curves showed 0.990 or greater, as specified in ASTM D4327-03. Table 2 shows employed calibration levels.

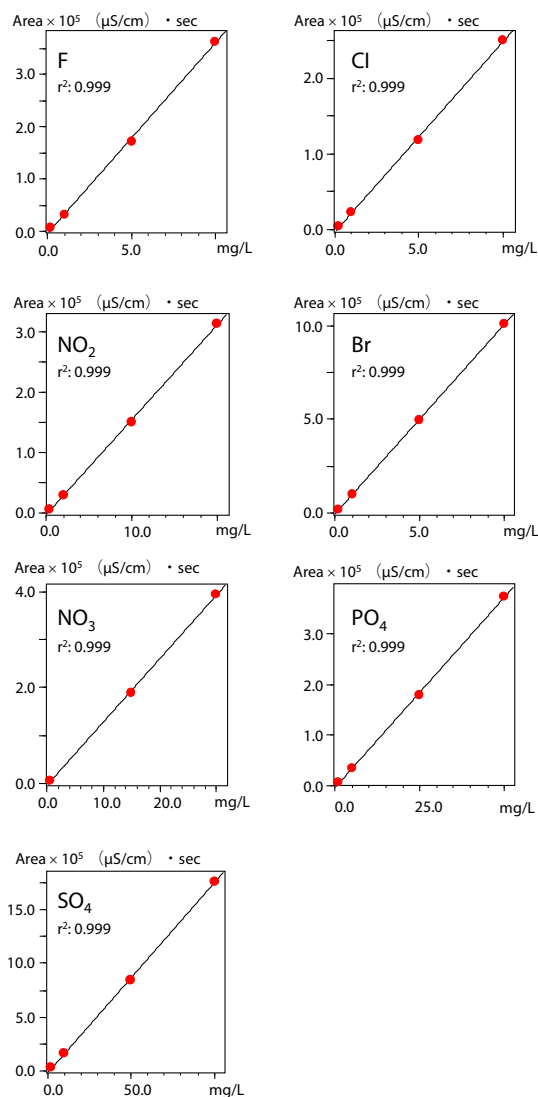


Fig. 2 Calibration Curves of 7 Anions

Table 2 Calibration levels of 7 Anions

	F	Cl	PO ₄	SO ₄	NO ₂	Br	NO ₃
STD1	0.20	0.20	1.00	2.00	0.40	0.20	0.60
STD2	1.00	1.00	5.00	10.00	2.00	1.00	3.00
STD3	5.00	5.00	25.00	50.00	10.00	5.00	15.00
STD4	10.00	10.00	50.00	100.00	20.00	10.00	30.00

Unit: mg/L

Repeatability was evaluated by the relative standard deviation (%RSD) of the retention time and peak area in 6 repeated analyses of the lower limit concentration of the calibration curves. Table 3 shows the concentrations of each anions, and the repeatability of their retention times, and area. In all cases, satisfactory repeatability was confirmed for both retention time and area.

Table 3 Repeatabilities of retention times and peak areas at lowest calibration levels

	F	Cl	PO ₄	SO ₄	NO ₂	Br	NO ₃
Concentration (mg/L)	0.20	0.20	1.00	2.00	0.40	0.20	0.60
Retention time %RSD	0.01	0.02	0.02	0.04	0.02	0.02	0.02
Peak area %RSD	0.57	0.79	0.91	0.81	0.43	0.52	0.63

■ Analysis of Drinking Water

Two types of commercially-available drinking water (A, B) were filtered with a 0.2 μm filter prior to the analysis. Fig. 3 shows the analysis results for the two drinking water samples, and Table 4 shows the quantitative values of each ion contained in the drinking water samples. The resulting concentrations results for components that exceeded the calibration range are shown in parentheses () as reference values.

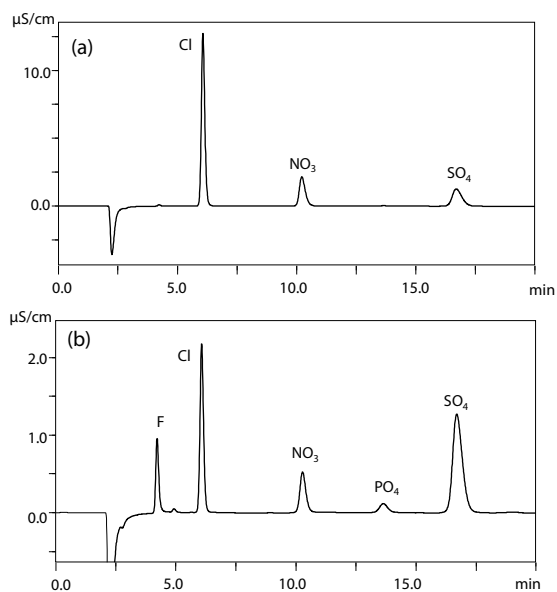


Fig. 3 Chromatograms of Commercial Drinking Water Samples (a) Drinking Water A, (b) Drinking Water B

Table 4 Concentrations of 7 anions in Drinking Water Samples

	F	Cl	PO ₄	SO ₄	NO ₂	Br	NO ₃
Drinking water A	N.D.	5.29	N.D.	2.96	N.D.	N.D.	3.17
Drinking water B	(0.35)	1.01	(0.86)	2.96	N.D.	N.D.	1.00

Unit: mg/L (N.D. = Not Detected)

Shim-pack is a trademark of Shimadzu Corporation in Japan and/or other countries.

■ Analysis of Tap Water

Using this method, a sample of tap water was analyzed after filtration with a 0.2 μm filter. Fig. 4 shows the analysis results. Table 5 shows the resulting concentration of each anion in the tap water. The concentrations of anions that exceeded the calibration range are shown in parentheses () as reference values.

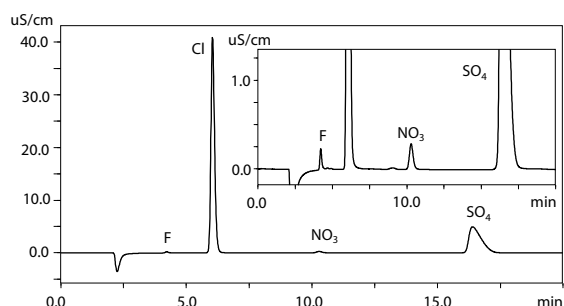


Fig. 4 Chromatogram of Tap Water and Enlarged Figure

Table 5 Concentrations of anions in Tap Water

	F	Cl	PO ₄	SO ₄	NO ₂	Br	NO ₃
Tap water	(0.18)	(16.51)	N.D.	10.61	N.D.	N.D.	0.69

Unit: mg/L (N.D. = Not Detected)

■ Spike-and-Recovery Test

Table 6 shows the results of a spike-and-recovery test of the tap water conforming to ASTM D4327-03. The test was conducted by spiking the sample with STD2 into Table 2. The recovery rate was calculated using the following equation, as specified in ASTM D4327-03.

Table 6 Percent recovery of the spike

F	Cl	PO ₄	SO ₄	NO ₂	Br	NO ₃
97.5	100.9	106.3	99.5	106.8	107.9	98.6

Unit: %

$$P=100 [A (V_s + V) - BV_s] / CV$$

- P :Percent recovery of the spike
- A :Analyte concentration (mg/L) in spiked sample
- B :Analyte concentration (mg/L) in unspiked sample
- C :Concentration (mg/L) of analyte in spiking solution
- V_s :Volume (mL) of sample used, and
- V :Volume (mL) added with spike

■ Conclusion

This article introduced analyses of anions contained in drinking water and tap water based on ASTM D4327-03 using a Shimadzu HIC-ESP suppressor ion chromatography.

In all cases, it was possible to obtain linearity within the allowable range specified in ASTM D4327-03 and good repeatability for the 7 species of anions specified in ASTM D4327-03.

In the analyses of the drinking water and tap water, satisfactory results were obtained for both separation from contaminants and the recovery rate.

<Reference>

- (1) ASTM D4327-03, Standard Test Method for Anions in Water by Chemically Suppressed Ion Chromatography, ASTM International, West Conshohocken, PA, 2003, www.astm.org