

## ASTM D4327-03 Compliant Analysis of Anions in Wastewater

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### User Benefits

- ◆ Enables acquisition of data with excellent linearity and repeatability in ASTM D4327-03 compliant analyses.
- ◆ Separation of contaminant components and target anions in wastewater is possible by using the Shim-pack™ IC-SA3.
- ◆ This test method is also applicable to anion analyses of drinking water.

### Introduction

The ion chromatograph is widely used in detection and quantitative analysis of ion components in aqueous solutions. ASTM D4327-03<sup>(1)</sup> issued by ASTM International in the United States specifies the test methods for analysis of the seven anions (fluoride ion, chloride ion, nitrite ion, bromide ion, nitrate ion, phosphate ion, sulfate ion) in drinking water or wastewater by suppressed ion chromatograph.

This article introduces an ASTM D4327-03 compliant analysis of anions in industrial wastewater using a Shimadzu HIC-ESP anion suppressor ion chromatograph. (Application News 01-00102-EN introduces an anion analysis of drinking water.) A high resolution-type column, Shim-pack IC-SA3 was used because industrial wastewater has a high content of contaminants.

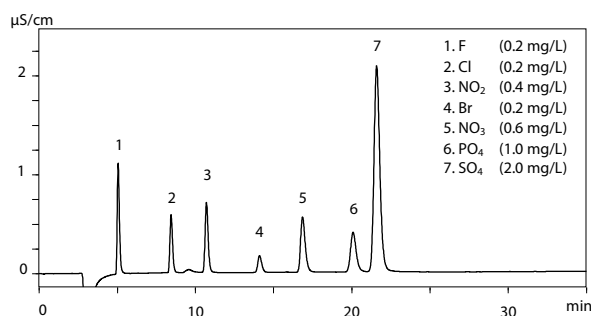


Fig. 1 Chromatogram of Anion Standard Solution

### Analysis of Standard Solution

Table 1 shows the analysis conditions of the standard solution of the seven anions, and Fig. 1 shows the chromatogram of the seven anions. The peak immediately after the chloride ion is a system peak of the carbonate ion, which is included in the mobile phase.

Table 1 Analytical Conditions

Column	: Shim-pack IC-SA3 <sup>*1</sup> (250 mm × 4.0 mm I.D., 5 μm)
Guard Column	: Shim-pack IC-SA3(G) <sup>*2</sup> (10 mm × 4.6 mm I.D., 5 μm)
Mobile phase	: 3.6 mmol/L Sodium Carbonate
Flow rate	: 0.8 mL/min
Column temp.	: 40 °C <sup>*3</sup>
Suppressor	: Electrodeialysis-type suppressor ICDS-40A
Injection volume	: 50 μL
Vial	: Shimadzu Vial, LC, 4 mL, Polypropylene <sup>*4</sup>
Detection	: Conductivity

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

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

\*3 The temperature can be changed according to the separation pattern of the components in the analysis sample. In the analysis of the industrial wastewater used in this experiment, satisfactory separation of contaminants and phosphate ions was possible at 40 °C.

\*4 P/N: 228-31537-91

### Linearity and Repeatability

A 4-level calibration curve was created for the seven anions which are the targets of analysis in ASTM D4327-03 in a concentration range conforming to the ASTM 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 the employed calibration levels.

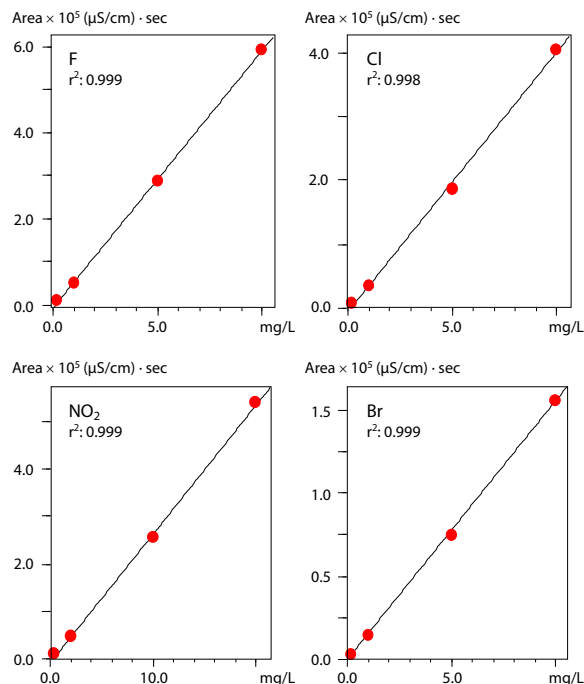


Fig. 2-1 Calibration Curves of Seven Anions

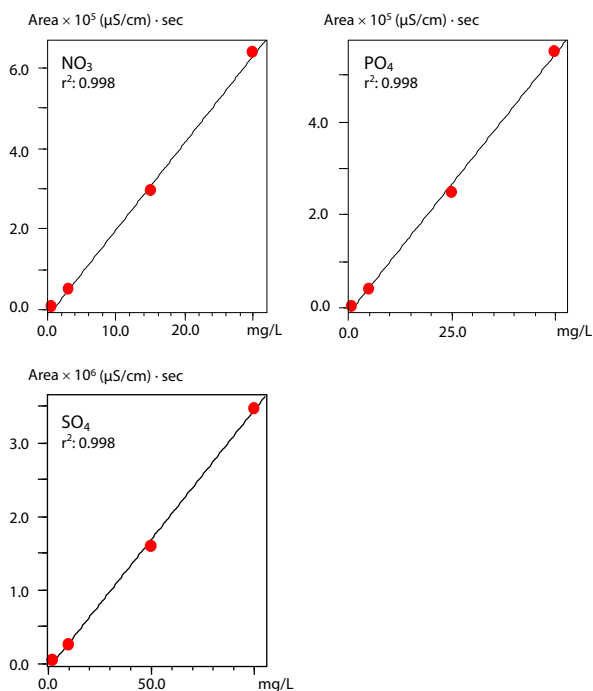


Fig. 2-2 Calibration Curves of Seven Anions

Table 2 Calibration Levels of Seven Anions

	F	Cl	PO <sub>4</sub>	SO <sub>4</sub>	NO <sub>2</sub>	Br	NO <sub>3</sub>
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 concentration of each anion and the repeatability of the retention times and area. Good repeatability was confirmed for the retention times and area of all anions.

Table 3 Repeatabilities of Retention Times and Peak Areas at Lowest Calibration Levels

	F	Cl	PO <sub>4</sub>	SO <sub>4</sub>	NO <sub>2</sub>	Br	NO <sub>3</sub>
Concentration (mg/L)	0.20	0.20	1.00	2.00	0.40	0.20	0.60
Retention time %RSD	0.01	0.01	0.03	0.03	0.02	0.03	0.04
Area %RSD	0.33	0.35	0.39	0.19	0.18	0.59	0.21

## ■ Analysis of Industrial Wastewater

Using this technique, industrial wastewater was filtered with a 0.2 µm filter and then diluted 10 times with ultrapure water prior to the analysis. Fig. 3 shows the analysis results. Although contaminant peaks appeared in close proximity to some components such as the fluoride ion and phosphate ion, satisfactory separation of those components was also possible by using a Shim-pack IC-SA3 as the analysis column. Table 4 shows the resulting concentration of each anion in the industrial wastewater.

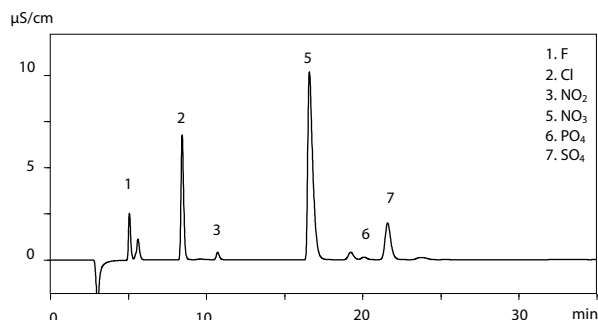


Fig. 3 Chromatogram of Industrial Wastewater

Table 4 Concentration of Industrial Wastewater (After Conversion to Stock Solution)

	F	Cl	PO <sub>4</sub>	SO <sub>4</sub>	NO <sub>2</sub>	Br	NO <sub>3</sub>
Concentration	6.2	27.3	14.6	40.1	5.9	N.D.	142.9

Unit: mg/L

## ■ Conclusion

This article introduced analyses of anions based on ASTM D4327-03 using a Shimadzu HIC-ESP anion suppressor ion chromatograph. Satisfactory separation of contaminants contained in industrial wastewater was possible by using this instrument in combination with a high resolution-type Shim-pack IC-SA3 column.

This analysis technique is applicable not only to industrial wastewater, but also to anion analysis of drinking water.

<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](http://www.astm.org)

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