

Analysis of Haloacetic Acids Compliant with the Specified Test Method for Water Quality Standard in Japan

Haloacetic acids are formed when chlorine is used to disinfect drinking water. In the Japanese Water Supply Law, it is required to determine contaminant levels of monochloroacetic acid, dichloroacetic acid, and trichloroacetic acid.

In this note, haloacetic acids were analyzed with LC/MS

under conditions compliant with the specified test method for water quality standard in Japan. As a result, good linearity and repeatability was obtained.

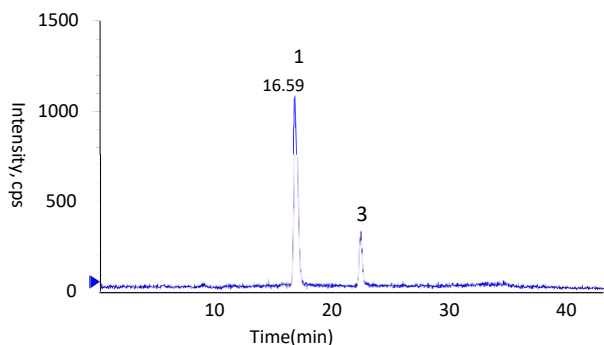
In addition, clean-up using a Meta SEP LC-Ba/Ag/H SPE cartridge was quite effective when high concentrations of anions, such as sulfate ion, are contained in sample water.

(Y.Tanaka)

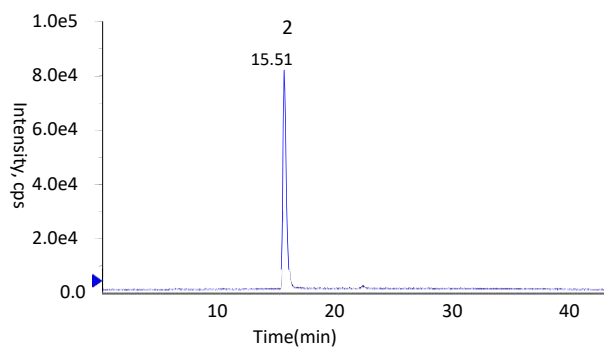
Chromatograms Obtained with Standard Solution

Concentration of each haloacetic acid was one-tenth of the target value.

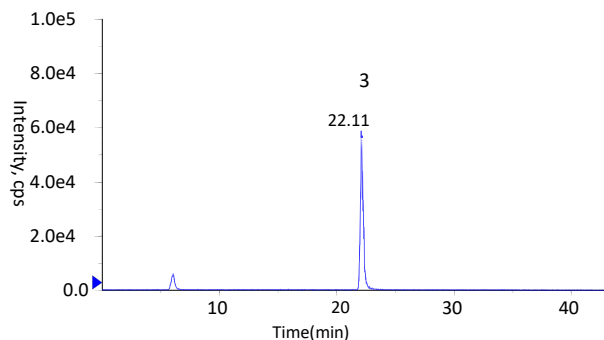
1. Monochloroacetic acid (MCAA) (Q1/Q3: 92.8/35.1, DP: -40 CE:-18) 2 µg/L



2. Dichloroacetic acid (DCAA) (Q1/Q3:126.7/82.8, DP: -40 CE:-14) 4 µg/L



3. Trichloroacetic acid (TCAA) (Q1/Q3: 160.7/116.8, DP: -35 CE:-10) 20 µg/L



Conditions

System : LC800 HPLC system

Column Eluent : InertSustain C18 (3 µm, 150 × 4.6 mm I.D.)

: A) CH₃OH

B) 0.2 % HCOOH in H₂O

A/B = 5/95 -38 min- 100/0 -12 min-

100/0, v/v (total 65 min)

Flow Rate : 0.2 mL/min

Col. Temp. : 30 °C

Detection : LC/MS/MS

(4000 Q TRAP : ESI, Negative, MRM)

CUR	IS	TEM	GS1	GS2
20	-4500	300	70	70

Inj. Vol. : 100 µL

HPLC Column

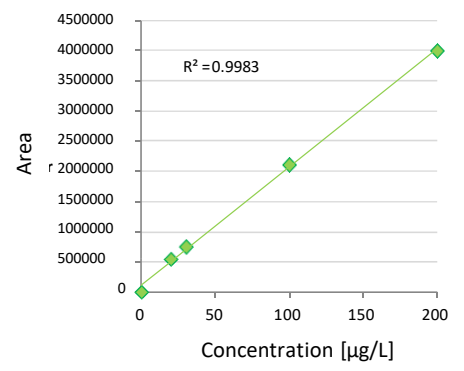
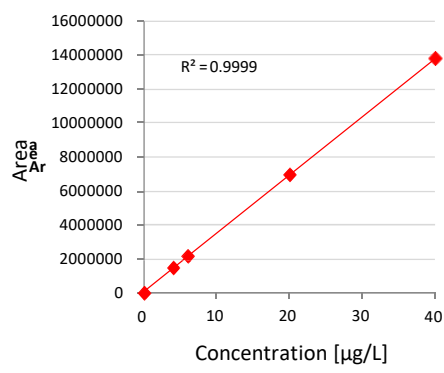
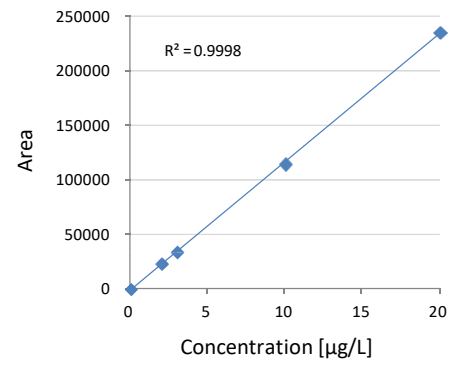
InertSustain C18
(3 µm, 150 × 4.6 mm I.D.)
Cat. No. 5020-07445

Repeatability at One-tenth of the Target Value

n=6	1. MCAA	2. DCAA	3. TCAA
Average	23850	1560000	605167
Standard deviation	459	20000	10008
CV [%]	1.9	1.3	1.7

Relationship between peak area and concentration

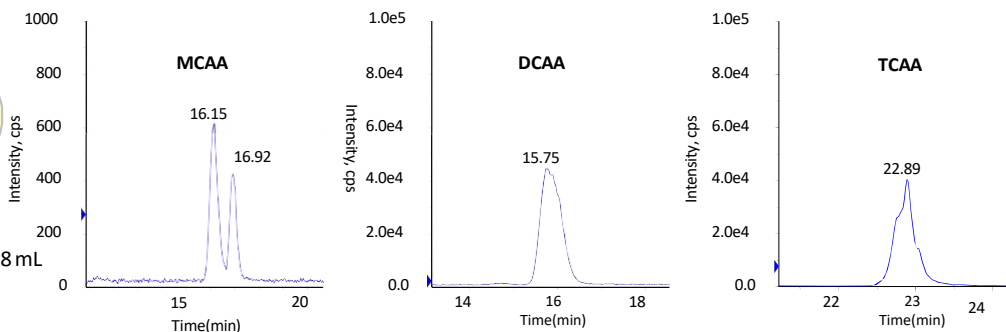
1. MCAA		2. DCAA		3. TCAA	
Conc. [µg/L]	Area	Conc. [µg/L]	Area	Conc. [µg/L]	Area
0	0	0	0	0	0
2	23400	4	1500000	20	548000
3	34100	6	2180000	30	743000
10	115000	20	6960000	100	2100000
20	236000	40	13800000	200	4000000



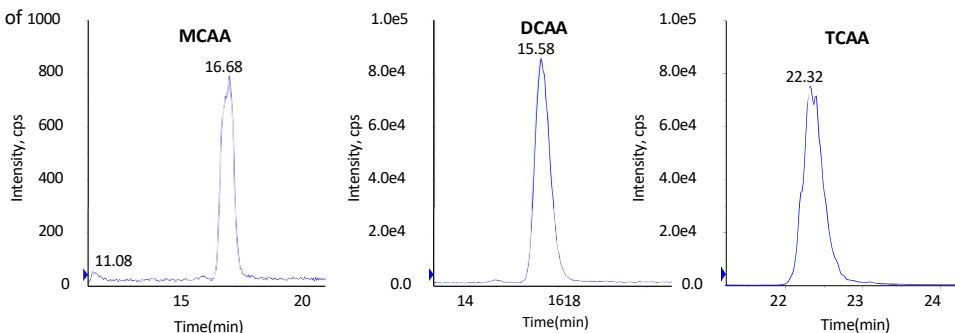
Especially when injection volume is increased to detect low level of haloacetic acids, chloride or sulfate ion in sample water often exerts bad influence on the analysis, such as peak splitting (shown below) and lowered sensitivity, which may deteriorate repeatability or quantitativity.

Chloride and sulfate ion can be removed by using a MetaSEP LC-Ba/Ag/H SPE cartridge. As a result of clean-up with the SPE cartridge, peak shape was improved as shown below. This SPE method also gave good recovery and reproducibility.

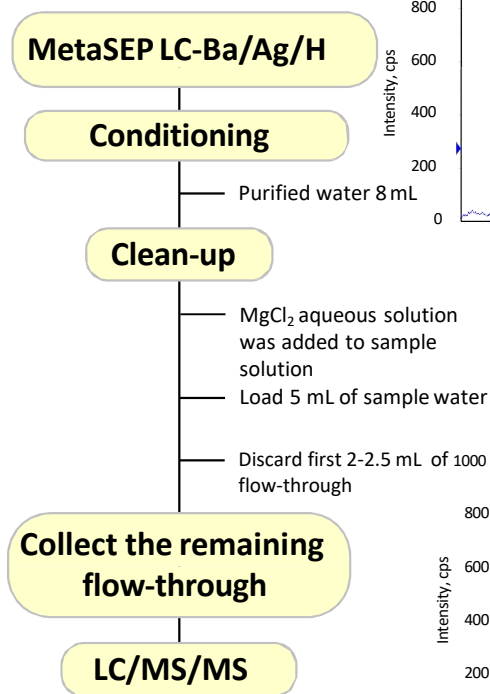
**Analysis of tap water without clean-up
(100 µL injection, one-tenth of the target value)**



**Analysis of tap water with clean-up
(100 µL injection, one-tenth of the target value)**



**Example of
Sample Pretreatment**



**Recovery of the sample pretreatment
(Standard addition, one-tenth of the target value)**

N=6	1. MCAA	2. DCAA	3. TCAA
Recovery [%]	93.1	99.8	97.3
CV[%]	3.8	6.2	6.4

**SPE cartridge;
MetaSEP SlimJ LC-Ba/Ag/H**



MetaSEP SlimJ LC-Ba/Ag/H
Cat. No. 8500-25100 50 pk

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