

Application News

No. C95

Liquid Chromatography Mass Spectrometry

Analysis of Haloacetic Acids in Drinking Water Using Triple Quadrupole LC/MS/MS (LCMS-8050)

Haloacetic acids (HAAs), by-products of water disinfection, are formed from naturally occurring organic and inorganic materials in water which react with the disinfectants chlorine and chloramine. The Japanese Ministry of Health, Labour and Welfare has established criterion values for three of these substances, monochloroacetic acid (MCAA: 0.02 mg/L), dichloroacetic acid (DCAA: 0.04 mg/L), and trichloroacetic acid (TCAA: 0.2 mg/L). The official analytical method for measuring these haloacetic acids utilizes solvent extraction and diazomethane derivatization prior to GC/MS quantitation.

In April, 2012, this method was amended to include LC/MS/MS as an additional method for measuring haloacetic acids. These LC/MS methods, which permit

analysis of HAAs directly from water samples, offer greater sample throughput by eliminating the solvent extraction and derivatization steps required when using GC/MS. This Application News introduces the use of the LCMS-8050 triple quadrupole mass spectrometer for analysis of these HAAs in accordance with the official LC/MS methodology requirements.

In this high speed method, MCAA, DCAA, and TCAA are eluted at 3.1, 3.4, and 5.2 minutes, respectively. Fig. 1 shows an MRM chromatogram of these HAAs each at a concentration of 0.001 mg/L. The calibration curve in Fig. 2 demonstrates linearity from 0.001 to 0.1 mg/L for each substance, and peak area repeatability at 0.001 mg/L (less than 1/10 the criterion value) (n=5), which was less than 3 % (%RSD).

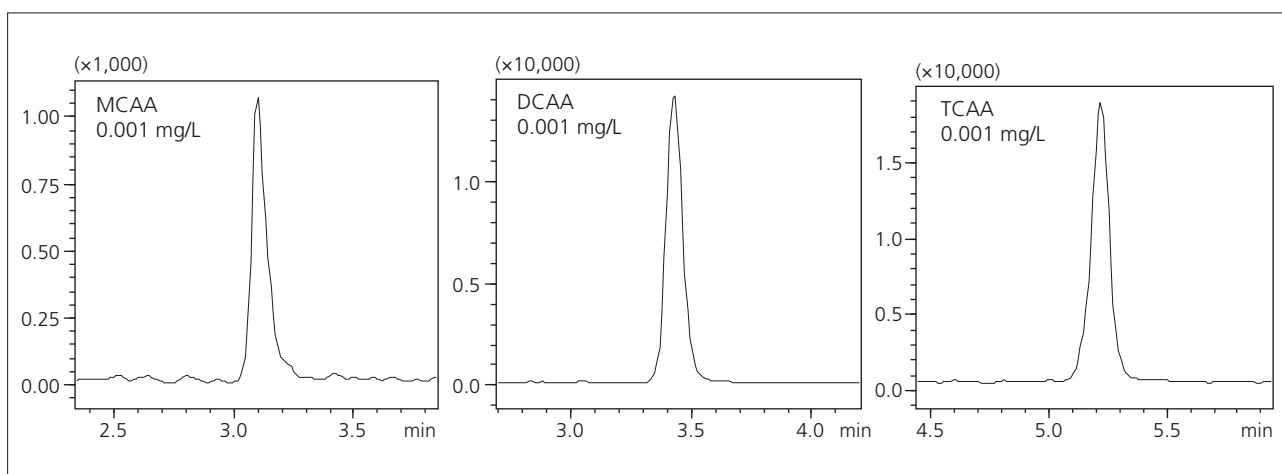


Fig. 1 MRM Chromatograms of MCAA, DCAA and TCAA (0.001 mg/L)

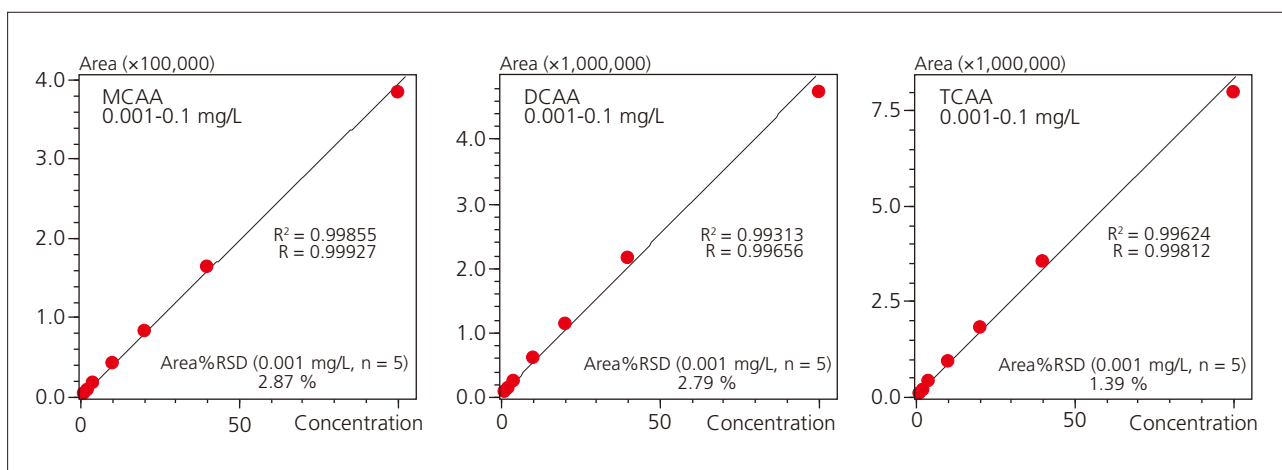


Fig. 2 Calibration Curve Linearity and Peak Area Repeatability

Quantitation and spike and recovery testing of haloacetic acids in tap water were conducted. To reduce residual chlorine in the tap water, sodium ascorbate was added at a ratio of 2 mg/100 mL. Fig. 3 shows MRM chromatograms obtained from tap water spiked with the three HAAs, each at 0.001 mg/L. The

recovery rate was determined using the average area value obtained in five repeat measurements. The official method specifies that anions present at high concentrations in the test water must be reduced "as needed." However, during these analyses, no anion contamination-related interference was observed.

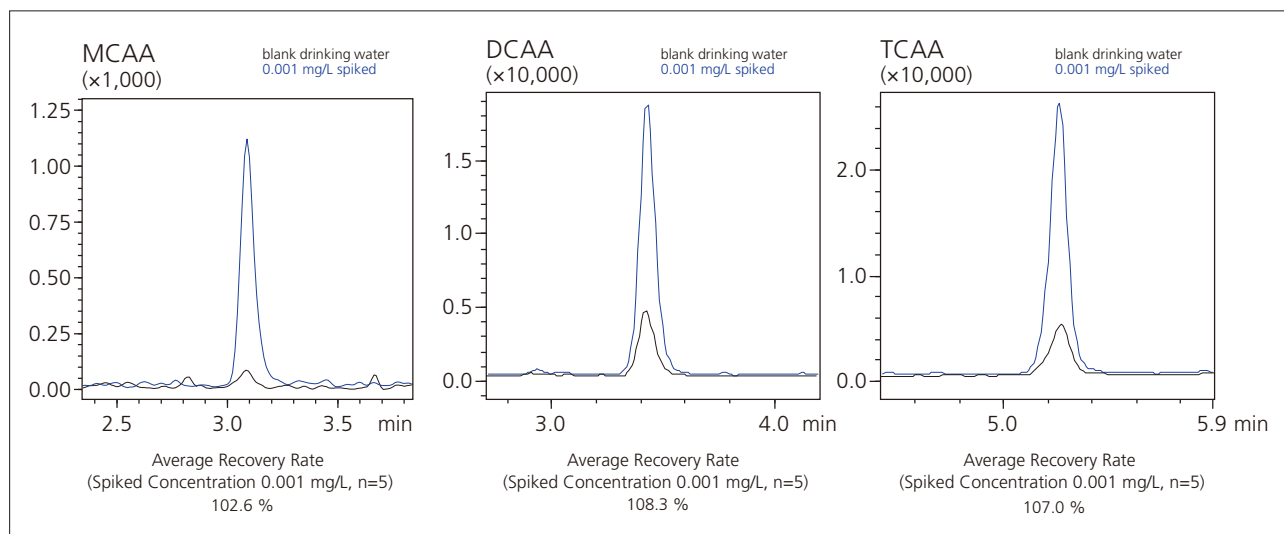


Fig. 3 MRM Chromatograms of Drinking Water Blank and Spiked with MCAA, DCAA and TCAA Respectively (0.001 mg/L)

The spike and recovery testing was conducted using tap water samples collected from four different regions (Samples 1 to 4). The results indicated no dependence on the water sampling region, with recoveries ranging

from 90 to 110 % from each collection location (see Table 1). Further, the concentrations of haloacetic acids detected were all below the criterion value.

Table 1 Quantitation and Recovery Results for Tap Water Samples

	Sample 1		Sample 2		Sample 3		Sample 4	
	Sample Conc. (mg/L)	Recovery (%)	Sample Conc. (mg/L)	Recovery (%)	Sample Conc. (mg/L)	Recovery (%)	Sample Conc. (mg/L)	Recovery (%)
MCAA	Tr.	102.6	0.00076	103.6	0.00069	94.9	0.00034	100.4
DCAA	Tr.	108.3	0.01151	101.7	0.00742	102.9	0.00635	92.3
TCAA	Tr.	107.1	0.00861	107.2	0.00622	104.5	0.00452	102.9

Table 2 Analytical Conditions

Column	: CAPCELL PAK MGIII (150 mm L. × 3 mm I.D., 3 μm)
Mobile Phases	: A 0.2 % Formic acid-water : B 0.2 % Formic acid-methanol
Flowrate	: 0.5 mL/min
Column Temperature	: 50 °C
Injection Volume	: 25 μL
Probe Voltage	: -3.5 kV (ESI-negative mode)
DL Temperature	: 150 °C
Block Heater Temperature	: 100 °C
Interface Temperature	: 100 °C
Nebulizing Gas Flow	: 3 L/min
Drying Gas Flow	: 5 L/min
Heating Gas Flow	: 15 L/min
MRM Transition	: MCAA; m/z 93.00 > 35.00 : DCAA; m/z 126.90 > 82.90 : TCAA; m/z 161.10 > 116.90

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> LCMS-8050

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