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Application News

LCMS™-8060NX High Performance Liquid Chromatograph Mass Spectrometer

Analysis of Acrylamide in Tap Water Using Triple Quadrupole LC/MS/MS

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User Benefits

- Quantitative analysis of acrylamide, which is listed as an Item for Further Study in Japan's Drinking Water Quality Standards, is possible at a concentration of 1/20 the target value of 0.5 µg/L.
- LC/MS/MS enables analysis of acrylamide without a sample concentration pretreatment process.
- It is possible to quantify acrylamide in drinking water with a satisfactory recovery rate.

■ Introduction

Polyacrylamide is a highly effective flocculating agent which is used as a chemical for water treatment, but may also contain its monomer acrylamide as a contaminant.

As of August 2021, acrylamide was classified as a Class 1 Designated Chemical Substance under Japan's Law concerning Pollutant Release and Transfer Register (PRTR Law) due to concerns regarding its harmful effect on health, and a target value of 0.0005 mg/L (0.5 µg/L) was set as an Item for Further Study in the Drinking Water Quality Standards (DWQSs).

Although an acrylamide analysis method involving sample preparation by solid-phase extraction has been reported [1], this article introduces the results of an analysis using the LCMS-8060NX liquid chromatograph mass spectrometer (Fig. 1) in which the concentration process was omitted and the tap water was injected directly.

A good recovery rate was obtained in the spike-and-recovery test, and analysis with high accuracy was possible at a concentration of 0.05 µg/L, which is 1/10 of the target value for acrylamide.

Fig. 1 Nexera™ X3 + LCMS™-8060NX

■ Analysis Conditions

Table 1 shows the HPLC and MS analysis conditions used in the measurement of acrylamide.

Table 1 Analysis Conditions

[HPLC conditions] (Nexera™ X3)		
Column	: CAPCELL PAK C18 AQ (150 mm × 2.0 mm I.D., 3 µm, Osaka Soda)	
Mobile phases	: A) 0.01 % Formic Acid in Water B) 0.01 % Formic Acid in Acetonitrile	
Gradient Program	: B .conc. 2 % (0-4.9 min) - 100 % (5-9 min) - 2 % (9.1-15 min)	
Flow rate	: 0.2 mL/min	
Column Temp.	: 40 °C	
Injection volume	: 10 µL	
[MS conditions] (LCMS-8060NX)		
Ionization	: ESI (Positive mode)	
Probe Voltage	: + 0.5 kV	
Nebulizing gas flow	: 3 L/min	
Drying gas flow	: 10 L/min	
Heating gas flow	: 10 L/min	
DL Temp.	: 200 °C	
Heat Block Temp.	: 500 °C	
Interface Temp.	: 350 °C	
MRM transition	: Acrylamide	*m/z* 72.10>55.15
	$^{13}C_3$-Acrylamide	*m/z* 75.10>58.15

■ MRM Chromatogram of Acrylamide

Fig. 2 shows the MRM chromatogram obtained when acrylamide was measured under the HPLC and MS analysis conditions shown in Table 1. The results confirmed that detection is amply possible at a concentration of 0.025 µg/L.

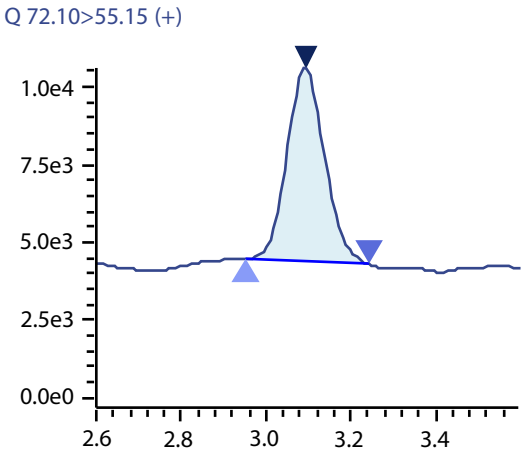

Fig. 2 MRM Chromatogram of Acrylamide (0.025 µg/L)

■ Calibration Curve of Acrylamide

Standard samples of acrylamide were prepared by dilution with water to concentrations from 0.025 to 0.5 µg/L. In this process, an internal standard ($^{13}C_3$-acrylamide) was added as 0.1 µg/L total concentration for each sample.

Using these samples, a calibration curve for acrylamide was prepared by the internal standard method. Fig. 3 shows the obtained calibration curve.

The coefficient of determination (r^2) was r^2>0.999 and the accuracy of all calibration points was 80 to 120 %, confirming that the calibration curve was satisfactory.

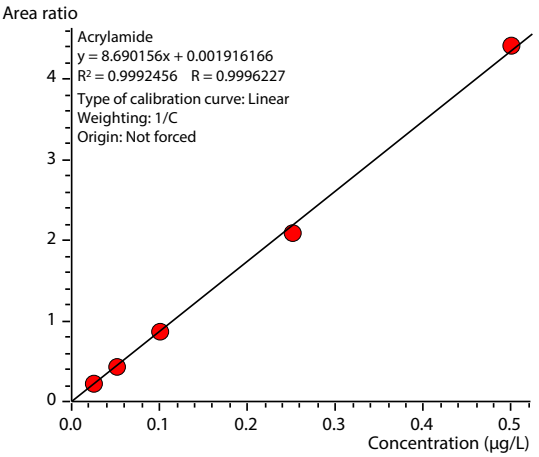

Fig. 3 Calibration Curve of Acrylamide

■ Repeatability of Acrylamide Analysis

A repeated analysis (n=5) was conducted with a standard sample of acrylamide with a concentration of 0.05 µg/L, which is 1/10 of the target value, and repeatability was confirmed. Fig. 4 shows the MRM chromatograms of each analysis. Repeatability accuracy (concentration RSD) was 1.9 %, showing good repeatability.

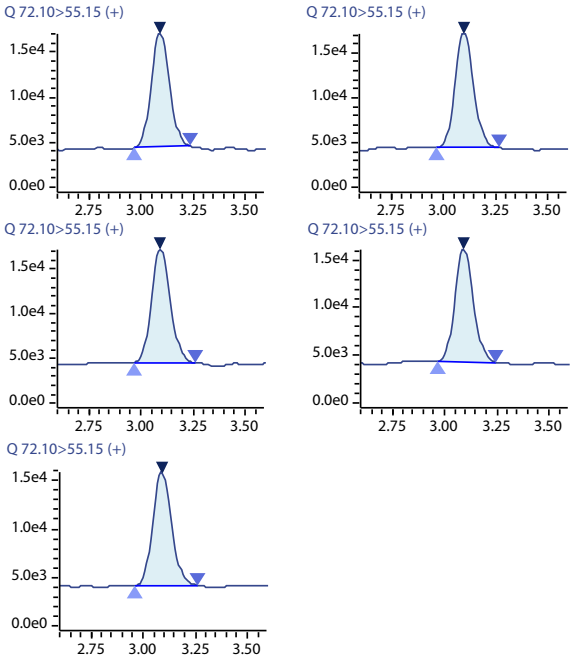

Fig. 4 MRM Chromatograms of Acrylamide (0.05 µg/L)

■ Spike-and-Recovery Test of Tap Water

A spike-and-recovery test was carried out using tap water sampled in Kanagawa Prefecture, Japan. Because decomposition of acrylamide by chlorine has been reported [1], the sampled tap water was dechlorinated by adding 0.02 g/L of sodium ascorbate.

A spiked tap water sample was prepared by spiking the dechlorinated tap water with 0.05 µg/L of acrylamide. Fig. 5 shows the MRM chromatograms obtained by analysis of the tap water and the spiked tap water sample.

The recovery rate of the tap water was 100.3 %, and the repeatability accuracy (concentration RSD) of the spiked sample was 2.5 % (Table 2).

Satisfactory results were obtained for both the recovery rate and repeatability accuracy, demonstrating that tap water samples can also be analyzed with good accuracy.

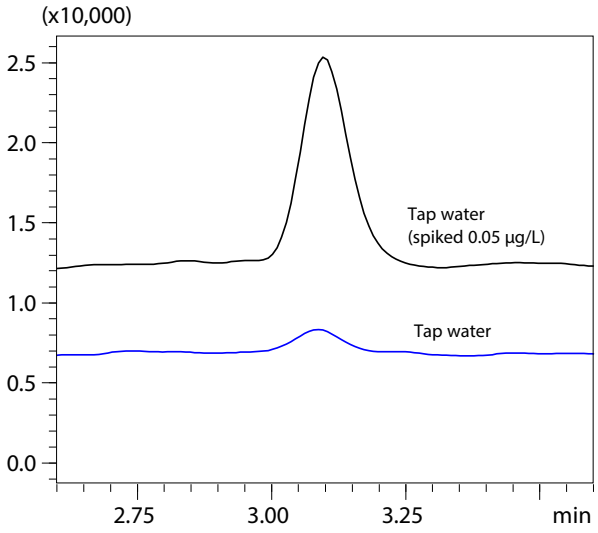

Fig. 5 MRM Chromatograms of Tap Water and Spiked Tap Water

Table 2 Recovery Rate and Repeatability of Spiked Tap Water

Recovery rate	Repeatability accuracy (concentration RSD)
100.3 %	2.5 %

■ Conclusion

- An analysis of acrylamide, which is specified as an Item for Further Study in Japan's Drinking Water Quality Standards, was carried out using an LCMS-8060NX, and sufficient sensitivity was obtained at 0.025 µg/L (target value: 0.5 µg/L).
- A good recovery rate and repeatability were obtained in a spike-and-recovery test of a tap water sample, confirming that it is possible to analyze acrylamide in tap water with high accuracy.

<Reference>

(1) Standard Methods for the Examination of Water 2020 Ed., III. Organic Compounds, 16. Acrylamide (Japan Water Works Association)

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