

Analysis of Water sample using GC/MS - Acrylamide-

In March 2000, the Ministry of Welfare issued guidelines for the evaluation of chemicals in tap water, in which the specifications for acrylamide were set and a test method for it using the gas chromatography was prescribed.

This data sheet presents an analysis according to these guidelines. The guidelines prescribes a sequence of operations, comprising bromination of acrylamide, solvent extraction, dehydration, concentration, addition of alkali, and GC analysis.

However, in this example, an analysis was performed using the NCI-GC/MS, according to the flowchart in Fig. 1 and the analytical conditions shown in Table 1. The derivatization reactions initially convert acrylamide to dibromopropyl amide and then to 2-bromopropyl amide. Fig. 2 summarizes the derivatization reactions. A 10mg/L standard sample was analyzed using the EI method. Fig. 3 shows the total ion chromatogram and Fig. 4 shows the mass spectrum.

Table 1 Analytical conditions

Model : GCMS-QP5050A (EI/CI/NCI Model)	
- GC -	- MS -
Column : ZB-WAX (30m×0.25mm I.D. df = 0.5μm)	Interface Temp. : 200°C
Column Temp. : 60°C (2min) - 10°C /min - 200°C (2min)	Ionization method : EI/NCI (Isobutane 50kPa)
Injector Temp. : 200°C	Scan Range : m/z 35-400
Injection method : Splitless (Sampling Time = 2min)	Scan Interval : 0.5sec

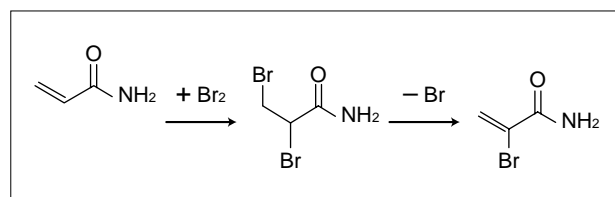


Fig. 2 Derivatization Reactions

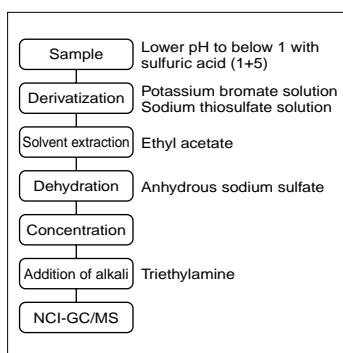


Fig.1 Flow Chart of Analysis

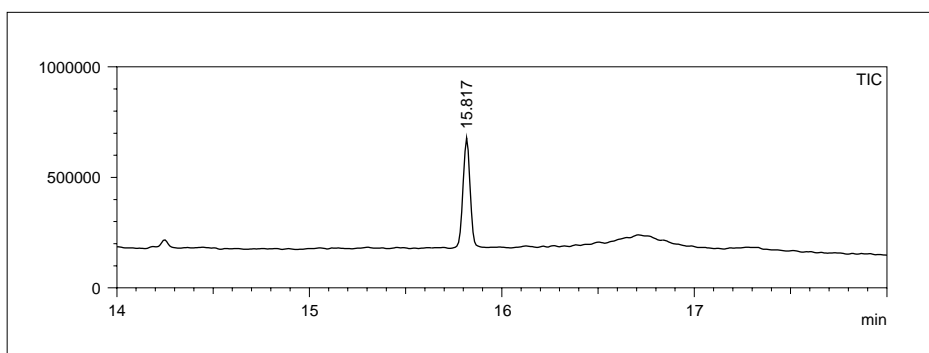


Fig. 3 Total Ion Chromatogram of 10 mg/L Standard Sample using the EI method

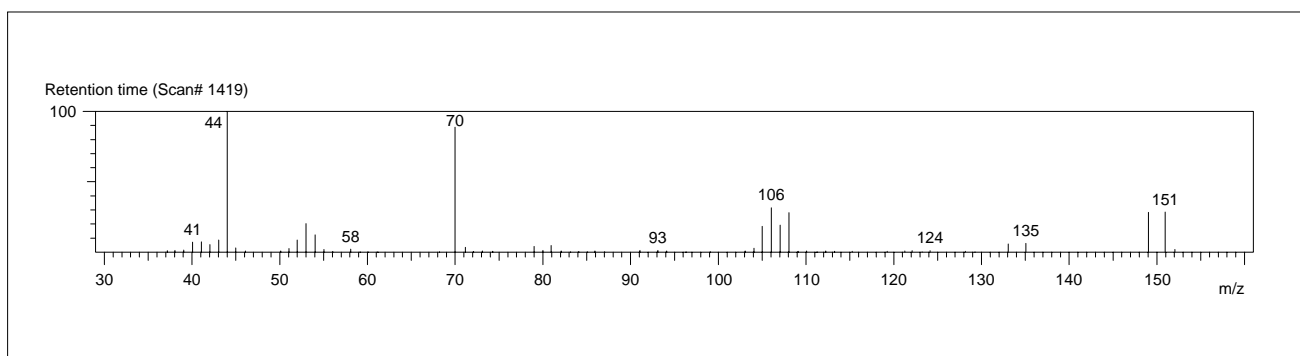


Fig. 4 EI Mass Spectrum of Standard Sample

Analysis of a 0.5 μ g/L standard sample using the NCI method produced the chromatogram in Fig. 5 and mass spectrum in Fig. 6. The calibration curve created by measuring standard samples from 0.5 μ g/L to 20 μ g/L shows good linearity, as shown in Fig. 7. Fig. 8 shows a chromatogram obtained from a real water sample and Fig. 9 shows the corresponding quantitation calculation results. In this example, impurities were detected near the peak of the target compound, but the excellent selectivity of the MS handled this analysis without any problems. The evaluation criterion for acrylamide in tap water is 0.05 μ g/L and the minimum limit of determination is 0.02 μ g/L. The sample pretreatment process results in the sample concentration being increased by 50 times, so that the minimum limit of determination is 1 μ g/L, providing a sufficient sensitivity margin.

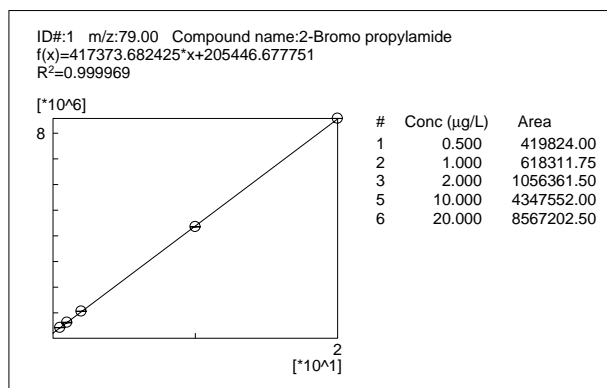


Fig. 7 Calibration Curve

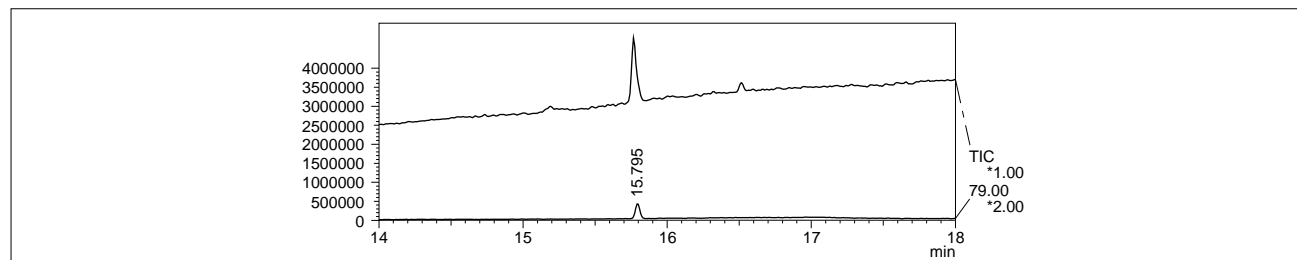
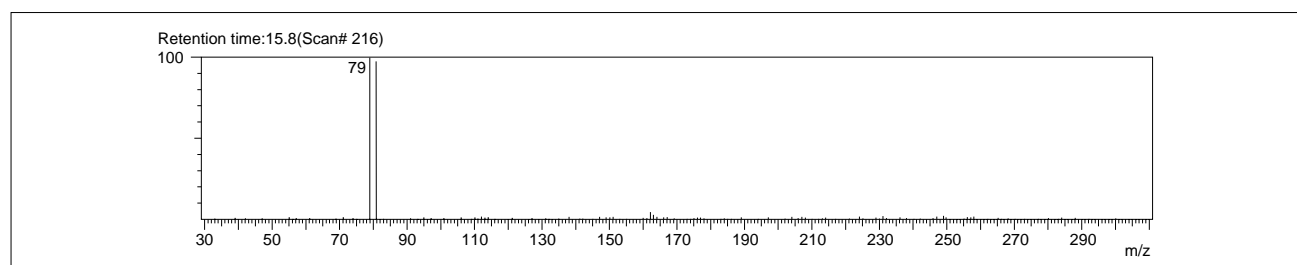
Fig. 5 Chromatogram of 0.5 μ g/L Standard Sample using the NCI method

Fig. 6 Mass Spectrum of Standard Sample using the NCI method

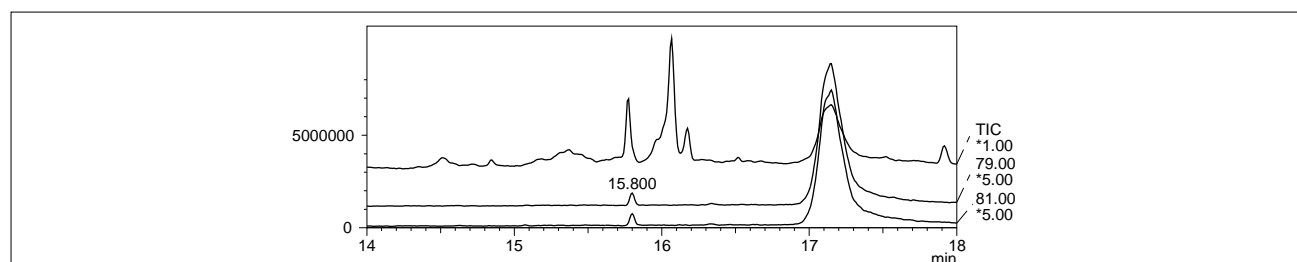


Fig. 8 Chromatogram of Real Sample using the NCI method

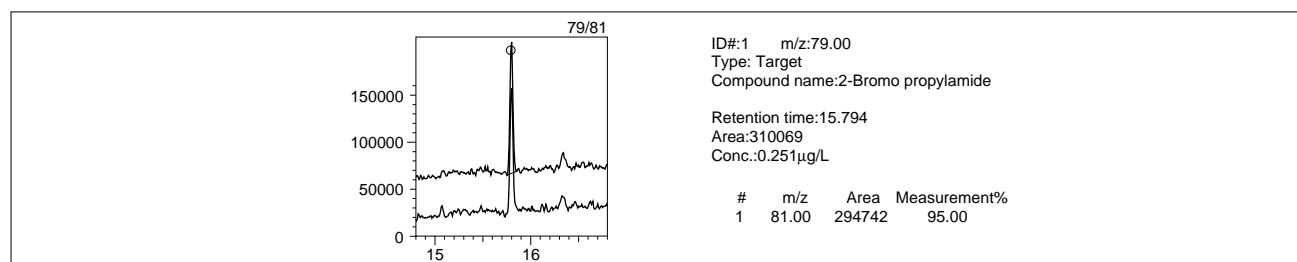


Fig. 9 Quantitation of Real Sample



SHIMADZU CORPORATION. International Marketing Division

3. Kanda-Nishikicho 1-chome, Chiyoda-ku, Tokyo 101-8448, Japan Phone: 81(3)3219-5641 Fax: 81(3)3219-5710
Cable Add.:SHIMADZU TOKYO

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