

Analysis of Chloral Hydrate and Haloacetonitriles

■ Introduction

The tap water is disinfected with chlorinating agents, and the behavior of chlorination byproducts is attracting attention.

The Drinking Water Test Method of Japan revised in July 2001 stipulates the internal standard method using 1,2,3-trichloropropane for measuring chloral hydrate and haloacetonitriles generated as byproducts of disinfection. Haloacetonitriles, chloroacetonitrile and bromochloroacetonitrile were recently added in this revision. Chloral hydrate and haloacetonitriles can be analyzed simultaneously.

This Application News introduces an example of analyzing chloral hydrate and haloacetonitriles, including chloroacetonitrile and bromochloroacetonitrile, by the internal standard method using GC/MS.

■ Analytical Conditions

Table 1 shows the analytical conditions and Table 2 shows the monitor ions for the SIM mode of target substances.

■ Calibration Curve

Calibration curves were created for each substance over a range of 10 μ g/L to 1000 μ g/L with the internal standard method. As the internal standard, 50 μ L of 1,2,3-trichloropropane solution at a concentration of 10mg/L was added to 2mL of samples. Excellent linearity was obtained for all substances.

■ Data

Fig. 1 through 7 show the mass spectrum, SIM mass chromatogram for a 10 μ g/L sample, and the calibration curve for each substance.

Table 1 Analytical Conditions

Instrument: GCMS-QP2010 with AOC-20i Autosampler	
GC	
Column	: J&W DB-1 (30m \times 0.25mm I.D. df=1 μ m)
Column Temp.	: 40°C(10min) \rightarrow 20°C/min \rightarrow 200°C(3min)
Carrier Gas	: Liner Velocity Constant Mode(43cm/sec)
Injection method	: Splitless
Sampling Time	: 2min
Injection Temp.	: 250°C
Injection Volume	: 1 μ L
MS	
Interface Temp.	: 250°C
Ion Source Temp.	: 200°C

Table 2 Monitor Ion

Compound Name	Molecular Formula	Monitor Ion
Chloral Hydrate	CCl ₃ CH(OH) ₂	82,111,146
Chloroacetonitrile	CH ₂ ClCN	75,77
Trichloroacetonitrile	CCl ₃ CN	108,110
Dichloroacetonitrile	CHCl ₂ CN	74,82
Bromochloroacetonitrile	CHBrClCN	74,155
Dibromochloroacetonitrile	CHBr ₂ CN	118,120
1,2,3-Trichloropropane	C ₃ H ₅ Cl ₃	75,110

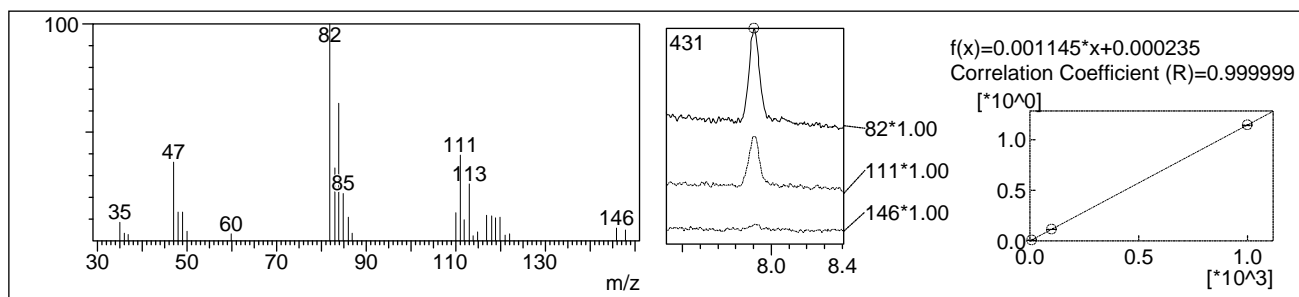


Fig.1 Chloral hydrate

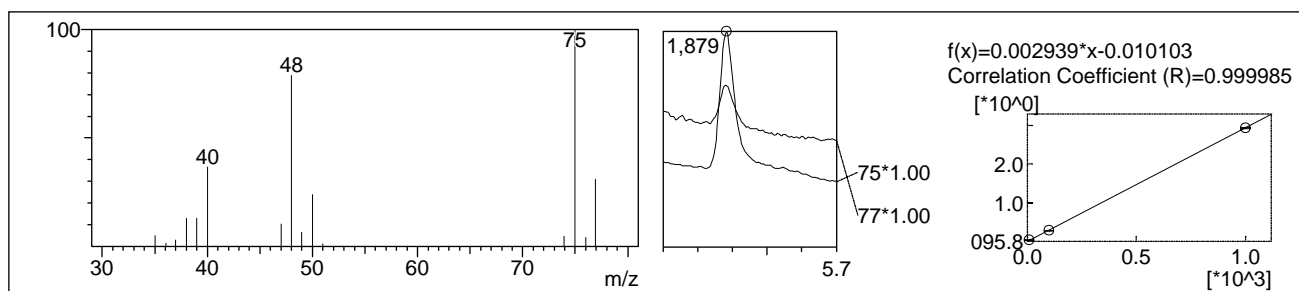


Fig.2 Chloroacetonitrile

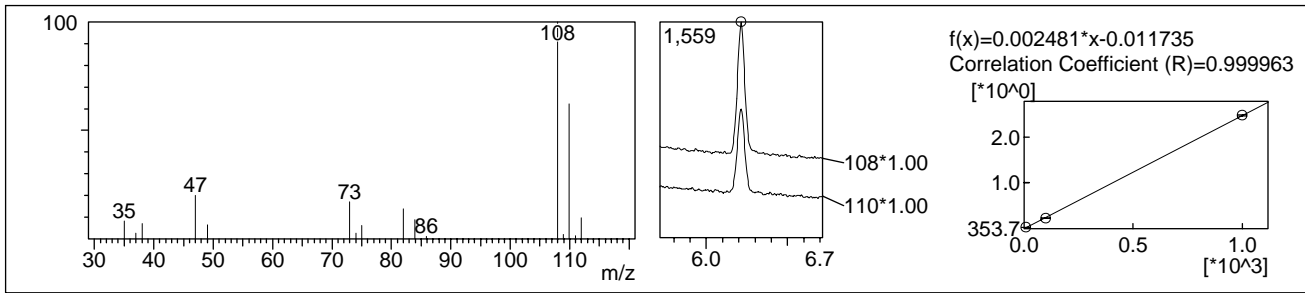


Fig.3 Trichloroacetonitrile

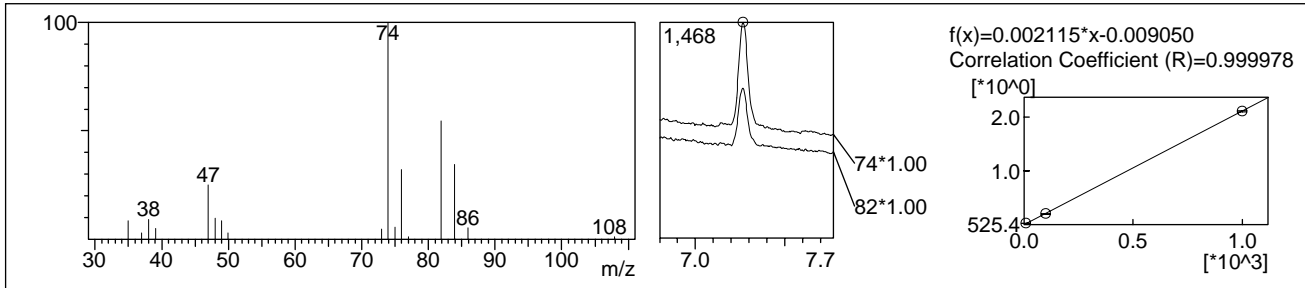


Fig.4 Dichloroacetonitrile

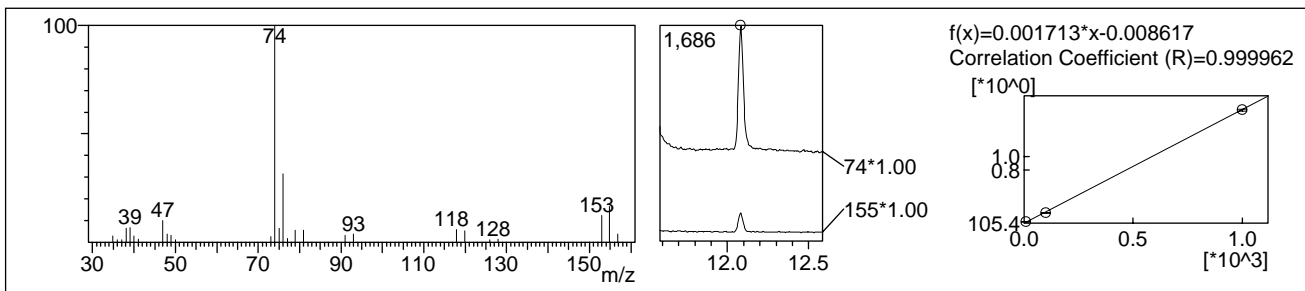


Fig.5 Bromochloroacetonitrile

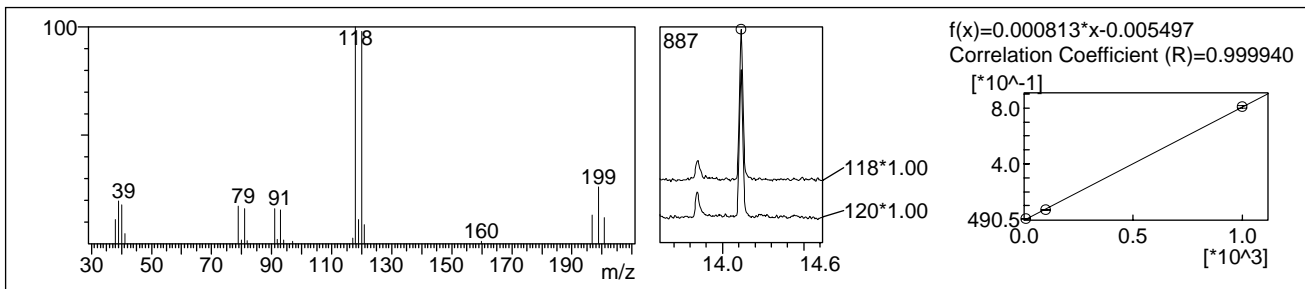


Fig.6 Dibromoacetonitrile

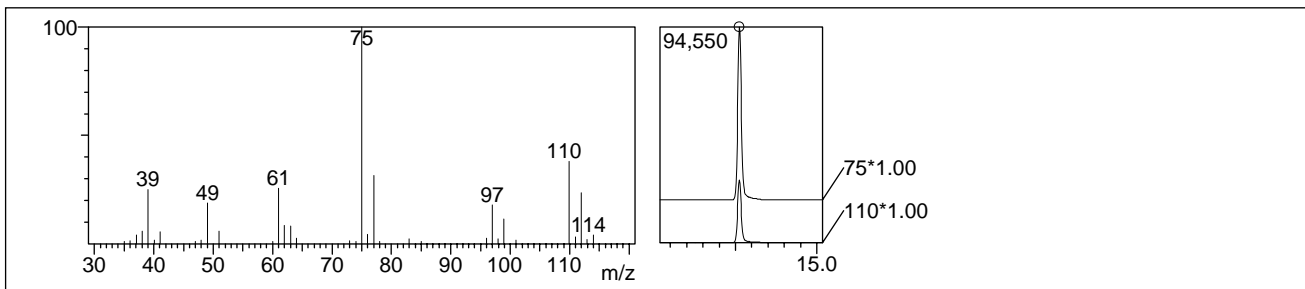


Fig.7 1,2,3-Trichloropropane