SHIMADZU APPLICATION NEWS

LA146-E025

● GASCHROMATOGRAPHY MASS SPECTROMETRY

No.M220

Analysis of 1,4-Dioxane in Water using GC/MS

1,4-dioxane is used in various types of industrial solvents, such as lacquer, resin, paint remover, etc. When high concentration vapors are inhaled, acutely toxic symptoms, such as dizziness and nausea, occur. Further, it has been learned from recent animal test that long-term oral ingestion of low concentrations of 1,4-dioxane generates toxic and carcinogenic effects.
1,4-dioxane is highly soluble in water, and there is concern that its discharge in waste water and waste fumes causes a risk to the environment and to health. Accordingly, the Ministry of Health, Labor and Welfare of Japan fixed the criteria level for 1,4-dioxane in tap water at 0.05mg/L in the revision for the Drinking Water Test Method announced in 2003.
This Application News introduces one of the analysis example of the determination of 1,4-dioxane using solid phase extraction GC/MS.

■ Outline of Analysis

The internal standard 1,4-dioxane-d_8 was added to 200mL of test water, which was the passed through a styrene divinylbenzene polymer solid phase column connected to an activated carbon solid phase column. The styrene divinylbenzene polymer solid phase column acts as a filter, and the sample is retained in the activated carbon solid phase column. The subsequent processes were performed only with the activated carbon solid phase column. After washing the activated carbon solid phase column with pure water, pure nitrogen gas was passed through it to remove the water, and elution was carried out using acetone. Then pure nitrogen gas was blown for concentration, and GC/MS-SIM measurement was performed. Fig. 1 shows the analysis flow chart.

Table 1 Analytical Conditions

Model	: GCMS-QP2010
-GC-	
Column	: Rtx-1701(30m×0.25mm I.D. df=1µm) (14% cyanopropylphenyl 86% demethylpolysiloxane)
Column Temp.	: 40˚C(2min)-5˚C/min-90˚C -10˚C/min-250˚C(5min)
Carrier Gas	: He (Constant linear velocity mode)
Linear Velocity	: 45cm/s
Injector Temp.	: 200˚C
Injection Method	: Split (Split Ratio=10:1)
Injection Volume	: 1µL
-GCMS-	
Interface Temp.	: 250˚C
Ion box Temp.	: 200˚C
Ionization Method	: EI
-Scan Mode-	
Scan Range	: m/z 45-350
Scan Interval	: 0.5s
-SIM Mode-	
SIM Monitoring Ion	: m/z 88, 58, 96, 64
SIM Sampling Interval	: 0.2s

Table 2 Monitor Ion Table

	Quantitation Ion	Reference Ion
1,4-Dioxane	88	58
1,4-Dioxane-d_8	96	64

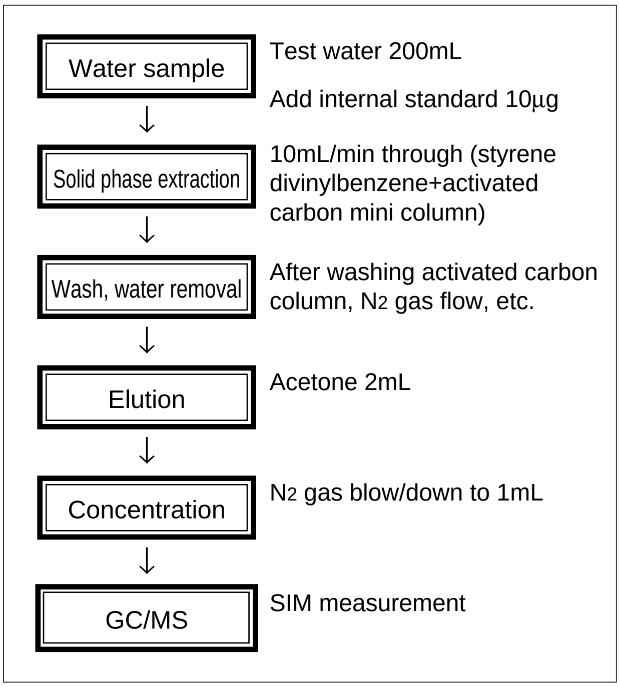

Fig.1 Flow Chart

■ Analysis with EI Scan Mode

Fig. 2 shows the TIC (Total Ion Chromatogram) of the measurement target 1,4-dioxane and internal standard 1,4-dioxane-d8 at 10mg/L standard solution, and Fig. 3 and Fig. 4 show the mass spectra. The analytical conditions are shown in Table 1.

■ Analysis with EI SIM Mode

It is possible to perform measurements with high sensitivity using the SIM (Selected Ion Monitoring) mode. The analytical conditions are shown in Table 1. Table 2 shows monitored ions. Fig. 5 shows the mass chromatogram of the 0.01mg/L standard sample, and Fig. 6 shows the calibration curve for concentrations over a range of 0.01 to 100mg/L. Standard solution was added to the tap water, and then recovery testing was performed. Fig. 7 shows the mass chromatogram and Fig. 8 shows the quantitative results. When tap water is measured, 0.005mg/L is 1/10 the minimum criteria level of 0.05mg/L, is required as the detection limit.
Pretreatment of the sample is performed to concentrated 200 times, so the tap water concentration 0.005mg/L corresponds to 1mg/L in the final solution, demonstrating that sufficient sensitivity can be obtained.

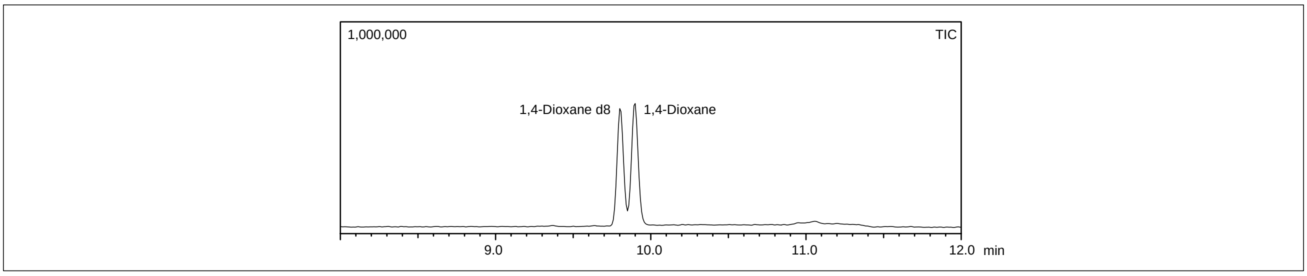

Fig.2 TIC of 10mg/L Standard Sample

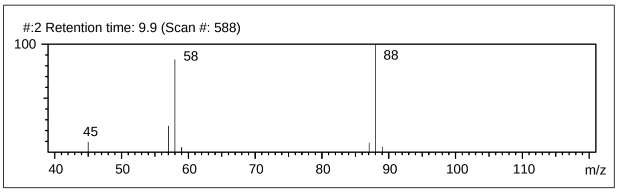

Fig.3 Mass spectrum of 1,4-Dioxane

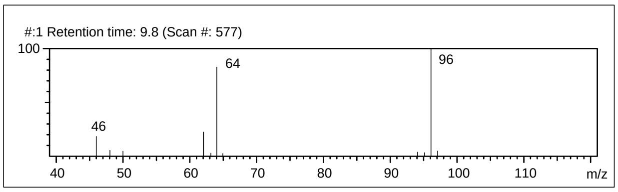

Fig.4 Mass spectrum of 1,4-Dioxane-d8

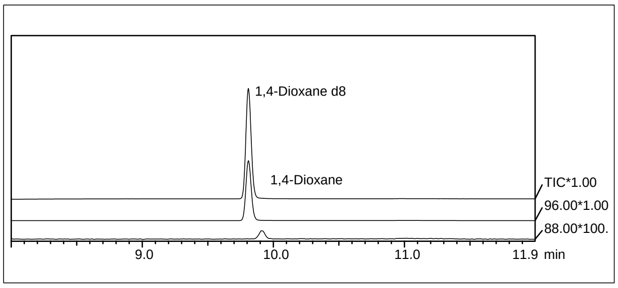

Fig.5 Mass chromatgram of 0.01 mg/L Standard Sample

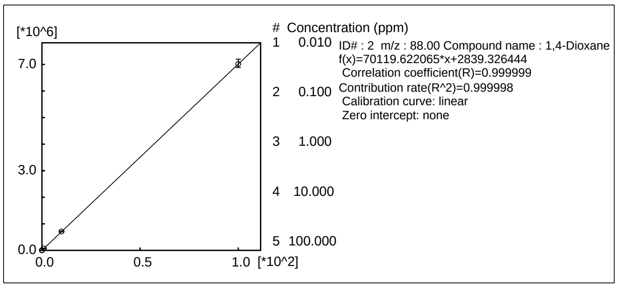

Fig.6 Calibration curve

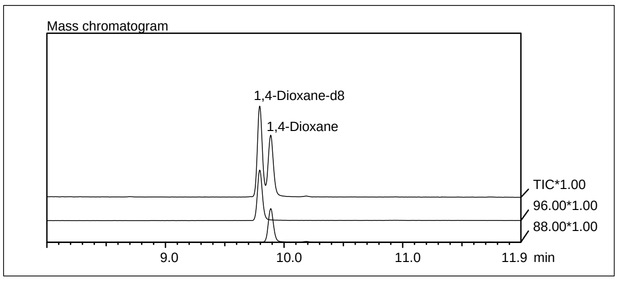

Fig.7 Mass Chromatogram of Tap Water spiked with STD

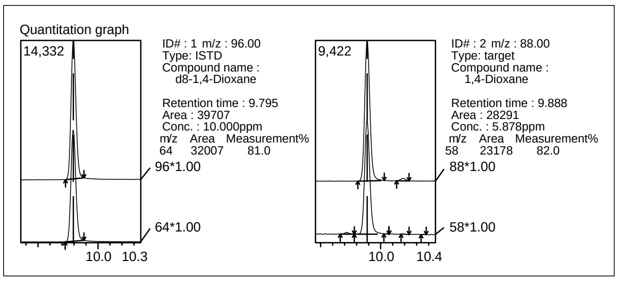

Fig.8 Quantitation Results for Tap Water spiked with STD

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