SHIMADZU
Excellence in Science

Application Data Sheet

No.131

GC-MS
Gas Chromatograph Mass Spectrometer

Analysis of Leachate from Water Supply Equipment Using Purge and Trap GC/MS

Water supply equipment refers to materials or equipment used in water supply facilities. The quality of these materials is required to conform to the standards designated by ordinances from the Ministry of Health, Labour and Welfare, as per "tests related to the quality of materials in equipment" (notification no. 45 by the Ministry of Health, Labour and Welfare in 2000). This, in turn, was prescribed on the basis of "ministerial ordinance regarding the technical standards for water supply facilities" (ordinance no. 15 by the Ministry of Health and Welfare in 2000). The six components shown in Table 1 are configured in the purge and trap (PT) GC/MS method. They differ significantly in terms of boiling point and solubility in water, so the method for the PT system is divided into two parts. However, since the sample volumes and trap tubes are completely the same in the two PT methods, system switching is not required, which means that consecutive analyses can be performed efficiently.
This Data Sheet presents the results of an investigation regarding the ease of quantifying the components in the leachate from water supply equipment via a purge and trap GC/MS.

Table 1: Purge and Trap (PT) Measurement Methods and the Components Targeted for Measurement

PT Method 1	1,3-butadiene, 1,2-butadiene	**PT Method 2**	Vinyl acetate, epichlorohydrin, styrene, N,N-dimethylaniline

Experiment

A standard product containing 1,3-butadiene, 1,2-butadiene, vinyl acetate, epichlorohydrin, styrene, and N,N-dimethylaniline was diluted with methanol to prepare a series of six mixed standard solutions at 0.25 μg/mL, 1.25 μg/mL, 2.5 μg/mL, 12.5 μg/mL, and 25 μg/mL.
Standard samples with 0.1 μg/L, 0.5 μg/L, 1 μg/L, 5 μg/L, and 10 μg/L of each component were prepared by adding a 2 μL fraction of the mixed standard solution at each concentration to 5 mL of Volvic (mineral water).
The prepared standard samples were measured using the analytical conditions listed in Table 2.
Note that the fluorobenzene and 4-bromofluorobenzene (the internal standard substances) were diluted with methanol to prepare a 12.5 μg/mL internal standard solution. This was added utilizing the system's internal standard automatic addition function in order to reach a concentration in water of 5 μg/L.

Table 2: Analytical Conditions

Purge and Trap Gas Analyzer: AquaPT 6000
Gas Chromatograph Mass Spectrometer GCMS-QP2020

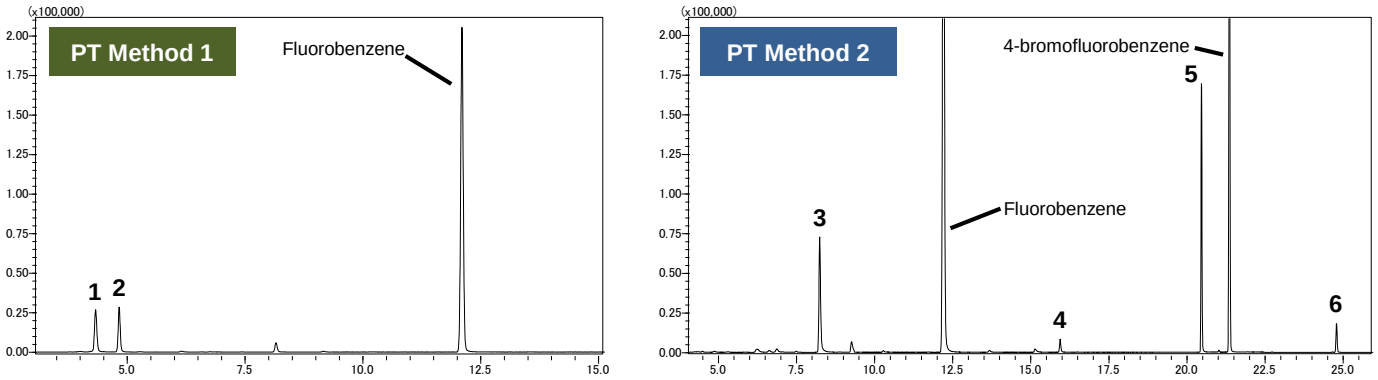
PT1	Trap Tube:	AQUA TRAP 1	PT2	Trap Tube:	AQUA TRAP 1
	Sample Volume:	5 mL		Sample Volume:	5 mL
	MCS*1:	Not used.		MCS*1:	Not used.
	Purge Time:	1.5 min		Purge Time:	18 min
	Purge Flowrate:	40 mL/min		Purge Flowrate:	60 mL/min
	Sample Heater:	ON (30 °C)		Sample Heater:	ON (30 °C)
	Dry Purge Time:	0.5 min		Dry Purge Time:	1 min
	Number of Rinse Cycles	3 cycles		Number of Rinse Cycles	9 cycles*2
	Desorption Temperature:	220 °C		Desorption Temperature:	220 °C
	Desorption Time:	2 min		Desorption Time:	2 min

GC			MS		
	Column:	InertCap AQUATIC (60 m × 0.25 mm I.D., 1.00 μm)		Ion Source Temperature:	200 °C
	Injection Port Temperature:	150 °C		Interface Temperature:	200 °C
	Injection Mode:	Split		Measurement Mode:	SIM mode
	Split Ratio:	3		Event Time:	0.3 sec
	Purge Flowrate:	3.5 mL/min			
	Control Mode:	Constant pressure (180 kPa)			
	Oven Temperature:	40 °C (1 min) → (3 °C/min) → 80 °C → (20 °C/min) → 200 °C (10 min)			

*1: Moisture Control System
*2: Up to nine cycles can be configured, and residue prone N,N-dimethylaniline can be analyzed with good accuracy.

Results

Fig. 1 shows the total ion current chromatogram obtained by measuring the 1 μg/mL standard sample in scan mode. Fig. 2 shows the SIM chromatograms for each component of the 0.1 μg/L standard sample. Sufficient sensitivity was obtained, even at concentrations of 1/10 or less of the reference level. In addition, Table 3 shows the results for the linearity (correlation coefficient: R) of the calibration curve (0.1 μg/L, 0.5 μg/L, 1 μg/L, 5 μg/L, and 10 μg/L) and the repeated analysis accuracy. A favorable result of 5 % or less was obtained for the repeated analysis accuracy.



1. 1,3-butadiene; 2. 1,2-butadiene; 3. Vinyl acetate; 4. Epichlorohydrin; 5. Styrene; 6. N,N-dimethylaniline

Fig. 1: Total Ion Current Chromatogram for the Standard Sample (1 μg/L) (Left: PT Method 1; Right: PT Method 2)

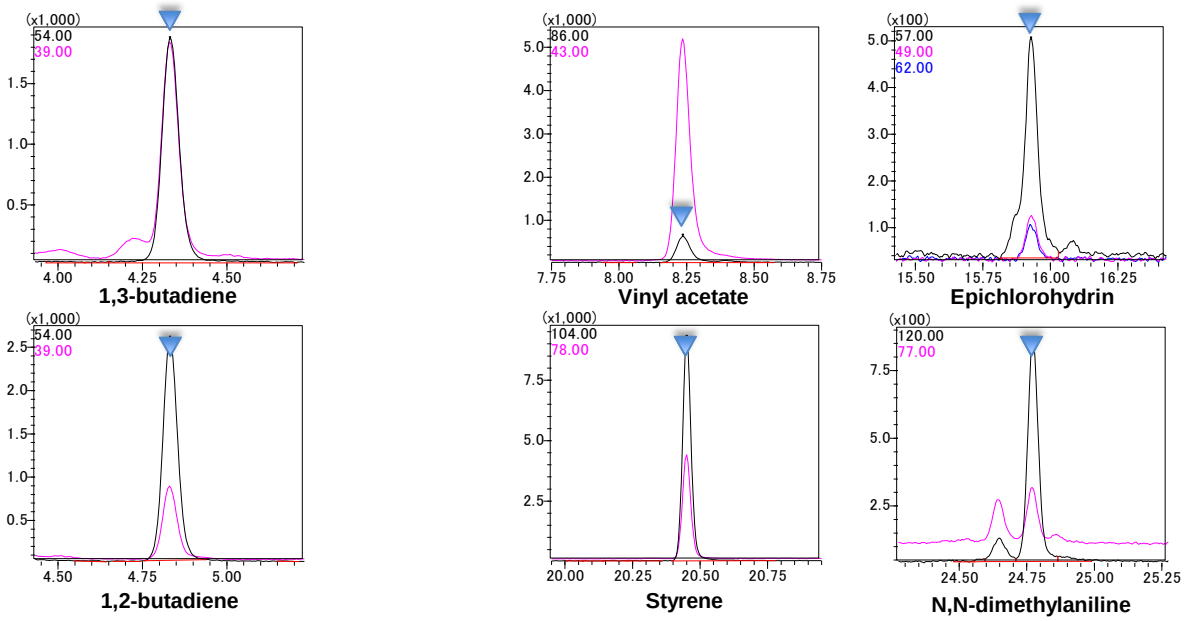


Fig. 2: SIM Chromatograms for the Standard Sample (0.1 μg/L) (Left: PT Method 1; Right: PT Method 2)

Table 2: Calibration Curve Linearity and Repeated Analysis Accuracy

ID	Compound Name	Correlation Coefficient: R	Coefficient of Variation (%)
1	1,3-butadiene	0.9988	2.3
2	1,2-butadiene	0.9991	1.5

* 0.5 μg/L, n=5

ID	Compound Name	Correlation Coefficient: R	Coefficient of Variation (%)
3	Vinyl acetate	0.9997	4.6
4	Epichlorohydrin	0.9999	3.4
5	Styrene	0.9998	2.8
6	N,N-dimethylaniline	0.9984	3.8

* 0.1 μg/L, n=5

Conclusions

From the results of an investigation of analytical conditions using the AquaPT 6000 as the purge and trap, and the GCMS-QP2020 as the GC/MS, it is evident that using this system enabled the measurement of components in the leachate from water supply equipment with high sensitivity and high accuracy.

First Edition: March, 2017

Shimadzu Corporation

www.shimadzu.com/an/