Reduction of Analysis Time using the GCMS-QP2010

The headspace-GC/MS (HS-GC/MS) is often used for the measurement of volatile organic compounds (VOCS) in environmental water samples. The 2001 revision of the Drinking Water Testing Method in Japan prescribes the HS-GC/MS for measurement of VOCs in tap water. Reducing the analysis time is essential for the improvement of productivity. Analysis time can be shorted by reducing column length, increasing carrier gas flow rate, and increasing the column temperature, but these methods result in poorer resolution. Conversely, the usual method to improve the resolution is using a column with smaller inner diameter. However, reduced carrier gas flow rate using the HS-GC/MS makes detection sensitivity less due to the reduction of the headspace gas introduced

into the column, and also the broadening of the chromatogram peaks. Water which is also introduced into the mass spegtrometer, may cause fluctuations in sensitivity due to issues related to the vacuum condition

The Shimadzu GCMS-QP2010 offers high sensitivity and can ensure sufficiently high vacuum due to the large-capacity vacuum evacuation system (incorporating 260 L/sec and 65 L/sec turbo molecular pumps). These features allow analysis time to be easily reduced by use of a small diameter column.

This data sheet introduces examples of reducing the analysis time from 30-minute to 15-minute cycles when analyzing the 23VOCs according to the Drnking Water Testing Method using GCMS-QP2010.

■ Analytical Conditions

Table 1 Analytical Conditions

	Analytical Conditions
Model	: TurboMatrix HS-40
	: GCMS-QP2010
-HS-	
Sample Amount	: 15mL(NaCl 4.5g)
Injection Time	: 0.12min
Transfer Temp.	: 180°C
Carrier Gas	: 130kPa
Sample Temp.	: 70°C
Needle Temp.	: 150°C
Cycle Time	: 15min
-GC-	
Column	: DB-624(30m×0.25mm I.D. df=1.4 μ m)
Column Temp.	: 50°C(3.5min)-25°C/min-115°C-40°C/ min-220°C(2min)
Injection Temp.	: 200°C
-MS-	
Interface Temp.	: 220°C
Ionsource Temp.	: 170°C
Ionization Method	: EI

Table 2 List of VOCs

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ID	Compound	SIM		
1	1, 1-Dichloroethylene	96	61	98
2	Dichloromethane	84	86	
3	trans-1, 2-Dichloroethylene	96	61	98
4	cis-1, 2-Dichloroethylene	96	61	98
5	Chloroform	83	85	
6	1, 1, 1-Trichloroethane	97	99	61
7	Carbontetrachloride	117	119	121
8	Benzene	78	77	52
9	1, 2-Dichloroethane	62	49	64
10	Trichloroethylene	130	95	132
11	1, 2-Dichloropropane	63	62	
12	Bromodichloromethane	83	85	
13	cis-1, 3-Dichloropropene	75	49	
14	Toluene	91	92	
15	trans-1, 3-Dichloropropene	75	49	
16	1, 1, 2-Trichloroethane	83	97	85
17	Tetrachloroethylene	166	164	129
18	Dibromochloromethane	129	127	131
19	m, p-Xylene	106	105	
20	o-Xylene	106	105	
21	Bromoform	173	171	175
22	p-Dichlorobenzene	146	148	111
23	Fluorobenzene	96	70	
24	p-Bromofluorobenzene	174	176	

■ Separation

Fig. 1 shows a SIM chromatogram of a $2\mu g/L$ solution of 23VOCs measured over a 15-minute cycle. If 1, 1, 1-trichloroethane and carbon tetrachloride, and

ethyl benzene and m, p-xylene in the 23 VOC components are separated clearly.

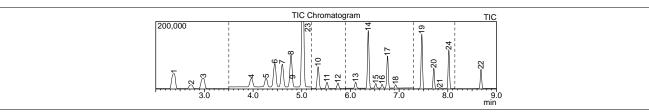


Fig.1 SIM Chromatogram of VOCs (2.0μg/L)

■ Sensitivity and Repeatability

Fig. 2 shows SIM chromatograms of 0.1μg/L of bromoform standard sample and a blank sample. Bromoform shows the lowest sensitivity in the 23VOCs, but in this measurement, it is easily detected at 0.1μg/L. Table 3 shows the repeatability results for three consecutive measurements of the four trihalomethanes components at 0.1μg/L:(chloroform, bromodichloromethane, dibromochloromethane, and bromoform). The Drinking Water Testing Method defines the minimum limit of determination where the CV value does not exceed 20%. This repeatability result more than meets the detection limit.

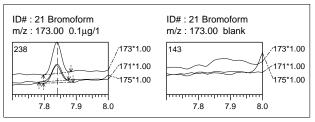


Fig. 2 SIM Chromatograms of Bromoform (0.1µg/L and blank)

Table 3 Repeatability

	1	2	3	Mean	CV (%)
Chloroform	0.1024	0.0975	0.1055	0.1018	3.97
Bromodichloromethane	0.1031	0.1040	0.1038	0.1036	0.48
Dibromochloromethane	0.1049	0.1060	0.1050	0.1053	0.60
Bromoform	0.1050	0.1036	0.1067	0.1051	1.48
			$(\mu g/L)$	$(\mu g/L)$	

■ Calibration Curves

Fig.3 shows the calibration curves of the four trihalomethanes in concentrations from $0.1\mu g/L$ to $20\mu g/L$. Satisfactory linearity was achieved, with correlation coefficients exceeding 0.9995 for all components.

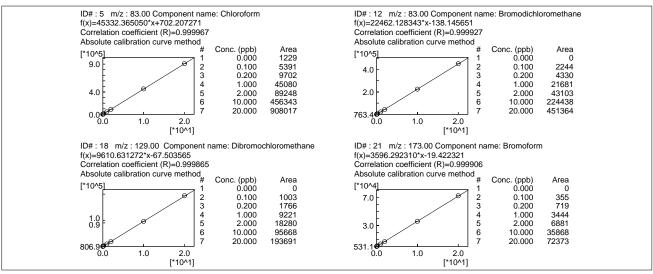


Fig. 3 Calibration Curves for Trihalomethanes

■ Stability

Twenty samples of tap water were analyzed after a calibration curve was created using a standard sample. A $2\mu g/L$ standard sample was measured after 10 and 20 measurements of tap water and the stability

determined by comparing the results of the $2\mu g/L$ standard sample already measured, to create the calibration curve. Almost no change was observed. Fig. 4 shows the compared chromatograms.

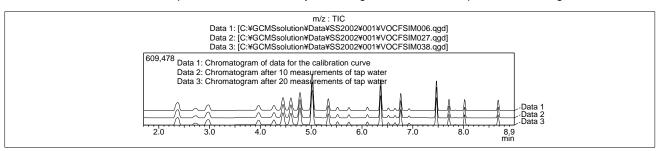


Fig. 4 Repeatability of Chromatograms

