SHIMADZU

Application Data Sheet

No.34

GCMS

Gas Chromatograph Mass Spectrometer

Shortening Cycle Times for Analyzing Volatile Compounds

Introduction

Shortening analysis cycle times requires reviewing everything from the GC-MS measurement time to the preparation time required before the next measurement starts. The GC oven in the GCMS-QP2010 Ultra features a double-shot cooling system that significantly reduces the time for cooling the column. This datasheet reports the results of a study to shorten the cycle time of analyzing volatile organic compounds (VOCs) in water using headspace GC/MS.

Analysis Conditions and Results

Table 1 shows the analysis conditions and Fig. 1 shows the total ion current chromatogram. All 28 VOCs (3 of which were internal standards) were detected within a GC-MS measurement time of 5.8 seconds. Only 3.8 minutes were required for cooling the column after analysis, including an equilibration time of 1 minute.

Table 1: Analysis Conditions

HS	: TurboMatrix HS				
GC-MS	: GCMS-QP2010 Ultra				
[HS]		[GC]			
Head space mode	: Constant	Vaporization chamber temp			
Injection time	: 0.15 min*		: 200℃		
Zone temp. settings	: (O/N/T)	Column	: Rtx®-624 (length 20 m, 0.18 mm I.D., df=1.0 µm Restek)		
Oven temperature	: 70℃	Column oven temp.	: 50℃ (0.5min)→(35℃/min)→200℃ (1min)		
Needle temperature	: 100℃	Carrier gas	: Helium		
Transfer temperature	: 150℃				
Sample shaker	: OFF	[MS]			
GC cycle time	: 10min	Interface temp.	: 230℃	Ion source temp.	: 200℃
Pressurization time	: 1min	Solvent elution time	: 0.7 min	Data sampling time	: 1.2 – 5.8 min
Uptake time	: 0min	Measurement mode	: SIM	Event time	: 0.2 sec
Warming time	: 30min	Emission current	: 150 µA (high sensitivity)		
HS carrier gas pressure	: 180 kPa				

Note: The headspace sampler injection time can vary depending on the instrument status. Therefore, optimization is required.

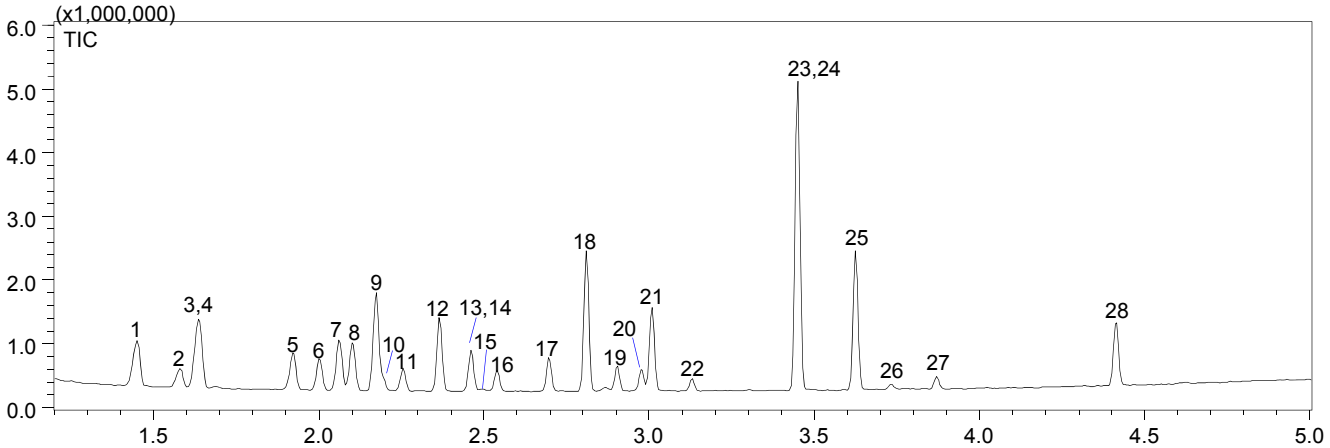

Fig. 1: Total Ion Current Chromatogram

1. 1,1-Dichloroethene, 2. Dichloromethane, 3. Methyl tert-butyl ether, 4. trans-1,2-Dichloroethylene, 5. cis-1,2-Dichloroethylene, 6. Chloroform, 7. 1,1,1-Trichloroethane, 8. Carbon tetrachloride, 9. Benzene, 10. 1,2-Dichloroethane, 11. Fluorobenzene, 12. Trichloroethylene 13. 1,2-Dichloropropane, 14. 1,4-Dioxane-d8, 15. 1,4-Dioxane, 16. Bromodichloromethane, 17. cis-1,3-Dichloropropene, 18. Toluene, 19. trans-1,3-Dichloropropene, 20. 1,1,2-Trichloroethane, 21. Tetrachloroethylene, 22. Dibromodichloromethane, 23,24. *m,p*-Xylene, 25. *o*-Xylene, 26. Bromoform, 27. 4-Bromofluorobenzene, 28. 1,4-Dichlorobenzene

Sensitivity and Reproducibility

Fig. 2 shows typical SIM chromatograms of 0.1 µg/L concentrations (5 µg/L for 1,4-dioxane). Table 2 indicates the accuracy for 7 repetitions of measuring 0.1 µg/L concentrations (5 µg/L for 1,4-dioxane). Good repeatability was obtained, even for high-speed analysis, with the %RSD for area ratios within 7 % for all compounds.

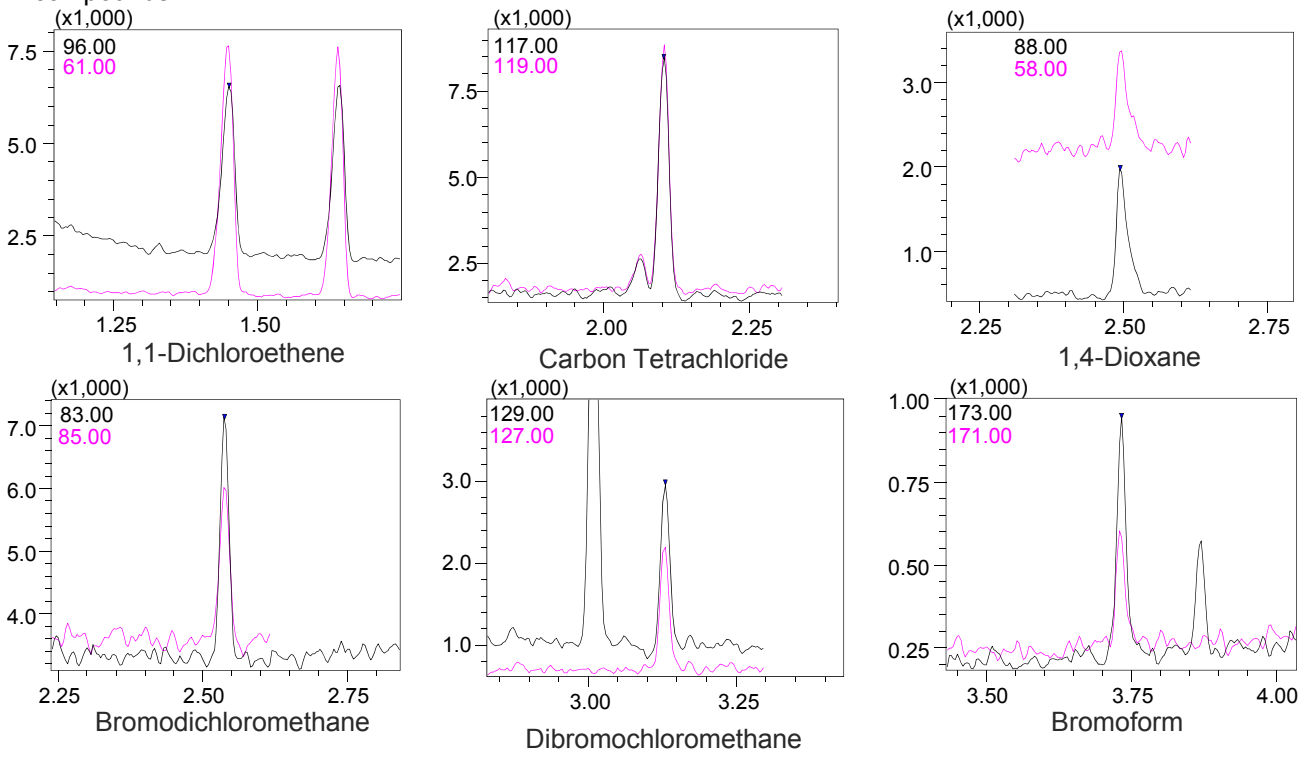

Fig. 2: SIM Chromatogram

Table 2: Area Ratio Repeatability (%RSD, n = 7)

ID	Compound Name	%RSD	ID	Compound Name	%RSD
1	1,1-Dichloroethene	1.24	15	1,4-Dioxane	6.48
2	Dichloromethane	6.43	16	Bromodichloromethane	2.18
3	Methyl tert-butyl ether	2.14	17	cis-1,3-Dichloropropene	4.05
4	trans-1,2-Dichloroethylene	4.14	18	Toluene	5.09
5	cis-1,2-Dichloroethylene	4.17	19	trans-1,3-Dichloropropene	4.60
6	Chloroform	3.17	20	1,1,2-Trichloroethane	4.59
7	1,1,1-Trichloroethane	1.01	21	Tetrachloroethylene	3.25
8	Carbon tetrachloride	2.68	22	Dibromodichloromethane	4.61
9	Benzene	1.45	23	*m,p*-Xylene	2.31
10	1,2-Dichloroethane	4.44	24	*o*-Xylene	1.39
11	Fluorobenzene	-	25	Bromoform	6.86
12	Trichloroethylene	1.93	26	4-Bromofluorobenzene	-
13	1,2-Dichloropropane	3.18	27	1,4-Dichlorobenzene	1.30
14	1,4-Dioxane-d8	-			

Summary

TurboMatrix HS enables overlapped heating of up to 12 vials. In combination with fast-GC/MS methods, it enables measuring six samples per hour.

SHIMADZU
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
URL http://www.shimadzu.com

First Edition: November 2011