

Cooperation with the China Academy of Urban Planning and Design for Field VOC Measurement of Drinking Water Sources with the Agilent 5975T LTM GC/MSD

Application Note

Environmental

Author

Suli Zhao
Agilent Technologies (Shanghai) Co., Ltd

Abstract

This study describes the use of the compact Agilent 5975T Low Thermal Mass GC/MS for analyzing drinking water quality following a natural disaster in China. The results demonstrate that given its portability, short cycle times, and accurate detection capabilities, the 5975T is an excellent choice for emergency response analyses.

Introduction

Agilent was requested to provide onsite water quality monitoring after a major earthquake in China. Rapid, reliable, lab quality results in the field were necessary in order to make decisions about how to avoid a national emergency from potentially contaminated drinking water.

The Agilent 5975T Low Thermal Mass (LTM) GC/MSD is a mobile combination gas chromatograph/mass selective detector(GC/MSD) instrument configured with a purge and trap concentrator, portable enough to bring to the field, but accurate and sensitive enough to provide reproducible, lab-quality results. These are necessary features to comply with regulatory standards for drinking water safety.

A method was developed on the Agilent 5975T LTM GC/MSD to rapidly analyze drinking water for potential pollutants. This method was tested in the field with samples from both Beijing and Tianjin drinking water sources. The method results were compared and verified with those from the environmental monitor center of Huai River and the Beijing hydrographic environmental monitor station, which both use the Agilent 5975T LTM GC/MSD for drinking water analyses.



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Experimental

Target compounds/compounds of interest

There were 22 target compounds analyzed in this application. These target compounds were chosen as the typical compounds regulated in drinking water for that area of China. The following 22 compounds were targets in this study:

1. 1,1-Dichloroethane
2. *Trans*-1,2-dichloroethane
3. *Cis*-1,2-dichloroethylene
4. Vinyl chloride
5. Methylene chloride
6. Chloroform
7. 1,1,1-Trichloroethane
8. Carbon tetrachloride
9. 1,2-dichloroethane
10. Trichloroethylene
11. Bromodichloromethane
12. 1,1,2-Trichloroethane
13. Dibromochloro methane
14. Tetrachloroethylene
15. Chlorobenzene
16. Bromoform
17. 1,4-Dichlorobenzene
18. 1,2-Dichlorobenzene
19. 1,2,4-Trichlorobenzene
20. 1,3,5-Trichlorobenzene
21. Hexachlorobutadiene
22. 1,2,3-Trichlorobenzene

Instruments

The drinking water contaminants were analyzed onsite, with an Agilent 5975T LTM GC/MSD, using the low thermal mass rapid method. Instrument conditions are listed in Table 1.

Reagents and chemicals

- Calibration standards: 22 VOCs, 1,000 mg/L (from J&K)
- Methanol, HPLC grade
- Pure water

Table1. Instrument Conditions

Purge & Trap system	Tekmar Stratum
Purge temperature	Ambient
MS Instrument	Transportable 5975T LTM GC/MSD
GC column	DB-624 LTM, 20 m × 0.18 mm, 1 µm
Oven temperature	45 °C (1.0 minute), 50 °C/min, 150 °C, 100 °C/min, 220 °C (2.2 minutes)
Constant flow	1.4 mL/min
Inject	220 °C, 20:1
Mass range	<i>m/z</i> 45~300
EI	230 °C
Ion source	150 °C
Calibration levels	0.5, 1, 10, 25, and 40 µg/L in water
Valve oven temperature	140 °C
Transfer line temperature	140 °C
Sample mount temperature	90 °C
Purge ready temperature	35 °C
Standby by flow	10 mL/min
PrePurge time	0.5 minutes
PrePurge flow	40 mL/min
Purge time	5 minutes
Purge flow	40 mL/min
Condenser ready temperature	40 °C
Condenser purge temperature	20 °C
Dry purge time	0.5 minutes
Dry purge temperature	20 °C
Dry purge flow	100 mL/min
GC start	At desorb
Desorb preheat temperature	245 °C
Desorb time	2 minutes
Desorb temperature	250 °C
Desorb flow	300 mL/min
Bake time	5.00 minutes
Bake temperature	280 °C
Bake flow	400 mL/min
Condenser bake temperature	200 °C

Sample preparation

Calibration levels

1. Add 10 μL from 1,000 mg/L VOC standards to 1 mL methanol, to make a 10 mg/L solution.
2. Dilute 100 μL of the 10 mg/L solution to 1 mL of 1 mg/L solution.
3. Dilute the 1 mg/L solution with water to make calibration solutions of 0.5 and 1.0 $\mu\text{g/L}$.
4. Dilute the 10 mg/L solution with water to the following levels: 4, 10, 25, and 40 $\mu\text{g/L}$, to produce the following calibration solutions: 0.5, 1.0, 4.0, 10, 25, and 40 $\mu\text{g/L}$.
5. Inject the calibration solutions with 5 mL purge-trap syringe.

Results and Discussion

Drinking water samples were analyzed in a mobile lab, located near the source, for 22 target compounds at a concentration of 25 $\mu\text{g/L}$. A sample chromatogram is illustrated in Figure 1. As the figure indicates, there was good separation of all compounds, with good peak shape. The average run time for this sample analysis was complete in less than 5 minutes for all runs.

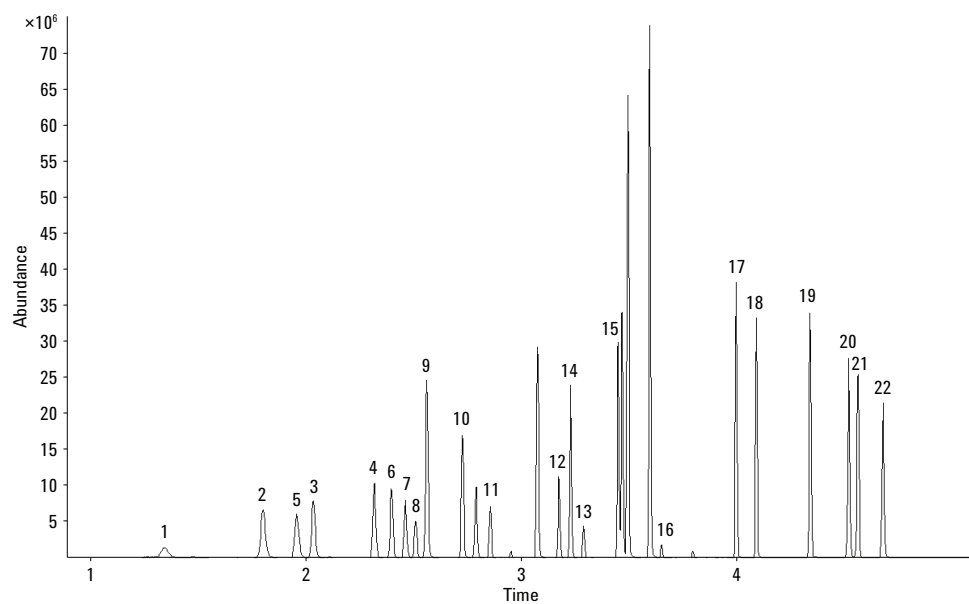


Figure 1. Total ion chromatogram of water analysis for 22 target VOCs at a concentration 25 $\mu\text{g/L}$.

Table 2 shows the retention times and correlation coefficients (R^2) for target compounds, as corresponding to the analysis shown in Figure 1. As the data show, all retention times are less than 5 minutes, which substantiates a 5-minute cycle time with the 5975T LTM GC/MSD. All R^2 values are ≥ 0.9993 , which indicates acceptable accuracy and data reproducibility for drinking water verification.

Calibration curves were created during the study for all target VOC. Figures 2–5 show example calibration curves for four of the VOCs: 1,1 dichloroethylene, *trans*-1,2-dichloroethylene, vinyl chloride, and chloroform. As the figures illustrate, all calibrations were linear, demonstrating good instrument response for the 5975T LTM GC/MSD.

Table 2. Retention Times and Correlation Coefficients for 22 Target VOC's in Wastewater

No.	Peak name	Retention time (min)	R^2	No.	Peak name	Retention time (min)	R^2
1.	1,1-Dichloroethane	1.81	0.9997	12.	1,1,2-Trichloroethane	3.18	0.9998
2.	<i>Trans</i> -1,2-dichloroethane	2.04	0.9999	13.	Dibromochloro methane	3.292	0.9996
3.	<i>Cis</i> -1,2-dichloroethylene	2.322	0.9999	14.	Tetrachloroethylene	3.233	0.9999
4.	Vinyl chloride	1.359	0.9999	15.	Chlorobenzene	3.454	0.9999
5.	Methylene chloride	1.965	0.9996	16.	Bromoform	3.653	0.9998
6.	Chloroform	2.403	0.9997	17.	1,4-Dichlorobenzene	4.001	0.9999
7.	1,1,1-Trichloroethane	2.469	0.9988	18.	1,2-Dichlorobenzene	4.094	0.9999
8.	Carbon tetrachloride	2.515	0.9996	19.	1,2,4-Trichlorobenzene	4.343	0.9998
9.	1,2-dichloroethane	2.565	0.9999	20.	1,3,5-Trichlorobenzene	4.523	0.9993
10.	Trichloroethylene	2.733	0.9999	21.	Hexachlorobutadiene	4.564	1.0000
11.	Bromodichloromethane	2.86	0.9994	22.	1,2,3-Trichlorobenzene	4.682	0.9998

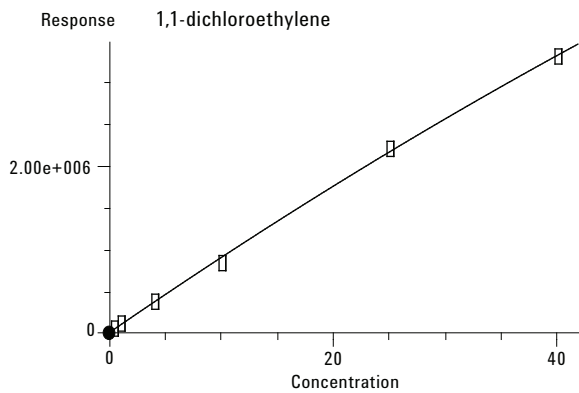


Figure 2. Calibration curve for 1,1-dichloroethylene.

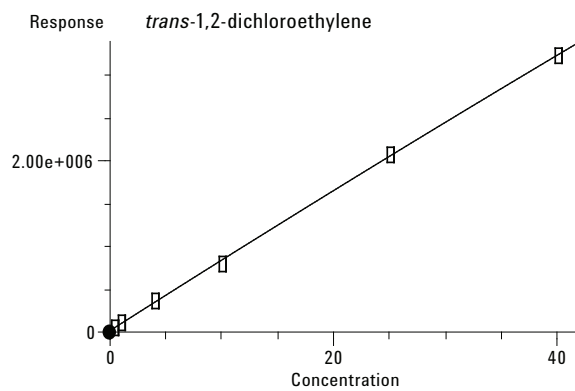


Figure 3. Calibration curve for *trans*-1,2-dichloroethylene.

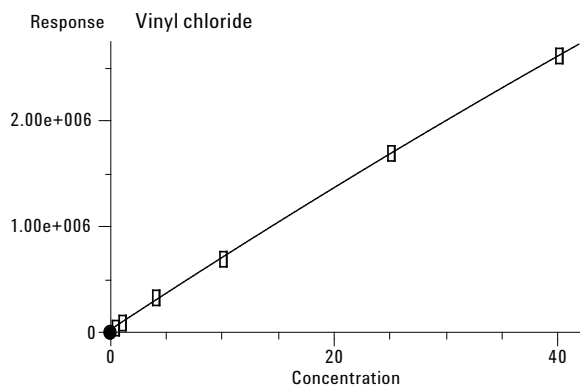


Figure 4. Calibration curve for vinyl chloride.

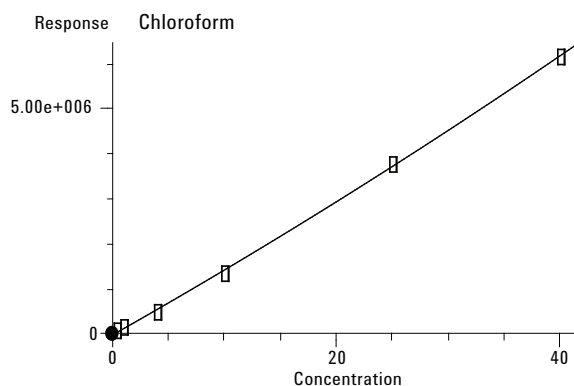


Figure 5. Calibration curve for chloroform.

Average detection levels for the 22 target VOCs are as listed in Table 3. All compounds were detected at levels between 0.06–0.18 µg/L, which are below the acceptable detection limit for safe levels of these compounds in drinking water. As these data indicate, the 5975T LTM GC/MSD was capable of accurately analyzing for the targets at low detection levels.

Conclusions

During an earthquake relief effort in China, the Agilent 5975T LTM GC/MSD was chosen by a state regulated mobile monitoring lab as the primary instrument for monitoring the quality and safety of drinking water. This is because the 5975T is the first commercial remote combination GC/MSD in the industry that can provide results of similar quality to those from a stationary lab. The results of this study prove that this is true. The low thermal mass rapid method on the 5975T GC/MSD reduces routine analysis cycle time from over 30 minutes to within 5–10 minutes, and provides results with high accuracy, sensitivity and reproducibility. Moreover, the small size and low power consumption of 5975T LTM GC/MSD provides the versatility for analyses in remote disaster locations.

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Table 3. Detection Levels of 22 VOCs in Drinking Water with the Agilent 5975T LTM GC/MSD

No.	Peak name	MDL (µg/L)
1.	1,1-Dichloroethane	0.13
2.	<i>Trans</i> -1,2-dichloroethane	0.09
3.	<i>Cis</i> -1,2-dichloroethylene	0.09
4.	Vinyl chloride	0.12
5.	Methylene chloride	0.10
6.	Chloroform	0.10
7.	1,1,1-Trichloroethane	0.11
8.	Carbon tetrachloride	0.07
9.	1,2-dichloroethane	0.10
10.	Trichloroethylene	0.06
11.	Bromodichloromethane	0.18
12.	1,1,2-Trichloroethane	0.06
13.	Dibromochloro methane	0.14
14.	Tetrachloroethylene	0.13
15.	Chlorobenzene	0.13
16.	Bromoform	0.10
17.	1,4-Dichlorobenzene	0.09
18.	1,2-Dichlorobenzene	0.14
19.	1,2,4-Trichlorobenzene	0.11
20.	1,3,5-Trichlorobenzene	0.13
21.	Hexachlorobutadiene	0.08
22.	1,2,3-Trichlorobenzene	0.13

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