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GC-MS HS-20 NX, GCMS-QP2050

Application News

Measurement of Volatile Organic Compounds in Water by Headspace GC-MS with Nitrogen Carrier Gas

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User Benefits

- Compared with helium, nitrogen carrier gas is inexpensive and readily available.
- Using the headspace sampler in trap mode enables highly sensitive measurement of volatile organic compounds in water.
- An electrically cooled trap enables the measurement of low boiling point and high boiling point components in a single analysis.

■ Introduction

Due to the health issues associated with volatile organic compounds (VOCs), standards and regulations are established for VOC levels in water. VOCs are preferably measured by methods that involve simple sample pretreatment, and headspace GC-MS is one of these methods commonly used to measure VOC levels.

GC-MS is normally performed with helium carrier gas, but helium gas could require more cost and lead time to obtain for analytical use every year. There is a need for a GC-MS analysis method that can be performed using relatively inexpensive and readily available nitrogen carrier gas. While nitrogen carrier gas results in inferior analytical sensitivity compared with helium carrier gas, trap mode sampling can salvage the reduction of the sensitivity.

This Application News presents an example measurement of VOCs in water using nitrogen carrier gas performed on the GCMS-QP2050 gas chromatograph mass spectrometer and the HS-20 NX headspace sampler in trap mode.

Fig. 1 GCMS-QP2050 and HS-20 NX

■ Samples and Analysis Conditions

Standard solutions were prepared by diluting 25 standard VOC mixture in methanol to 1, 2.5, 5, 10, 25, 50, 100, and 250 mg/L. The standard solutions were combined with internal standards 1,4-dioxane-d8 at 100 mg/L, fluorobenzene at 12.5 mg/L, and p-bromofluorobenzene at 12.5 mg/L.

Calibration standards were then prepared by adding 3 g of sodium chloride, 10 mL of mineral water, and 2 µL of standard solution to a 20 mL headspace vial and capping the vial. Internal standard levels in the calibration standards were 1,4-dioxane-d8 at 20 µg/L, fluorobenzene at 2.5 µg/L, and p-bromofluorobenzene at 2.5 µg/L. The calibration standards were then agitated until the sodium chloride was completely dissolved. Calibration curves were prepared with solutions containing VOCs at 0.2, 0.5, 1, 2, 5, and 10 µg/L in water (5, 10, 20, and 50 µg/L for 1,4-dioxane). Repeatability was verified with a solution containing VOCs at 0.2 µg/L in water (5 µg/L for 1,4-dioxane). The analysis conditions used are shown in Table 1.

Table 1 Equipment Configuration and Analysis Conditions

<GC-MS>	GCMS-QP2050
<Head Space Sampler>	HS-20 NX
<HS>	
Mode	: Trap (Tenax TA)
Oven Temp.	: 70 °C
Sample Line Temp.	: 150 °C
Transfer Line Temp.	: 150 °C
Trap Cooling Temp.	: 25 °C
Trap Heating Temp.	: 230 °C
Trap Waiting Temp.	: 25 °C
Multi Injection	: 5
Vial Pressure	: 70 kPa
Dry Purge Pressure	: 20 kPa
Vial Heating Time	: 30 min
Vial Pressurization Time	: 1 min
Pressure Equilibrating Time	: 0.1 min
Loading Time	: 0.5 min
Load Equilibrating Time	: 0.1 min
Dry Purge Time	: 3 min
Injection Time	: 5 min
Needle Flush Time	: 5 min
<GC>	
Injection Mode	: Split
Split Ratio	: 15
Carrier Gas	: N_2
Carrier Gas Control	: Const. Linear Velocity (23.7 cm/sec)
Column	: InertCap AQUATIC (60 m, 0.25 mm i.d., df = 1 µm)
Oven Program	: 40 °C (1 min)→10 °C/min→120 °C (0 min) →5 °C/min→140 °C (0min)→10 °C/min →200 °C (0 min)
<MS>	
Ion Source Temp.	: 200 °C
Interface Temp.	: 200 °C
Data Acquisition Mode	: SIM
SIM Monitoring *m/z*	: See Table 2
Event Time	: 0.3 sec
TMP Evacuation Rate	: 255 L/sec

■ Confirmation of Calibration Curves and Repeatability

Fig. 2 shows a total ion current chromatogram (TICC) from the 5 µg/L calibration standards. The chromatogram shows the separation between the peaks of every compound. Fig. 3 shows mass chromatograms from the 0.2 µg/L calibration standards (1,4-dioxane at 5 µg/L). Every compound was detected with good sensitivity, as shown by an S/N of ≥10. Fig. 4 shows calibration curves for selected compounds, and Table 2 shows analysis parameters, quantitative results, and calibration curve data. For some components, the high concentration region of the calibration curve was not linear and a quadratic fit was used. The analytical quality was excellent for all compounds. The repeatability and accuracy were less than 5 %, and within ±20 %, respectively.

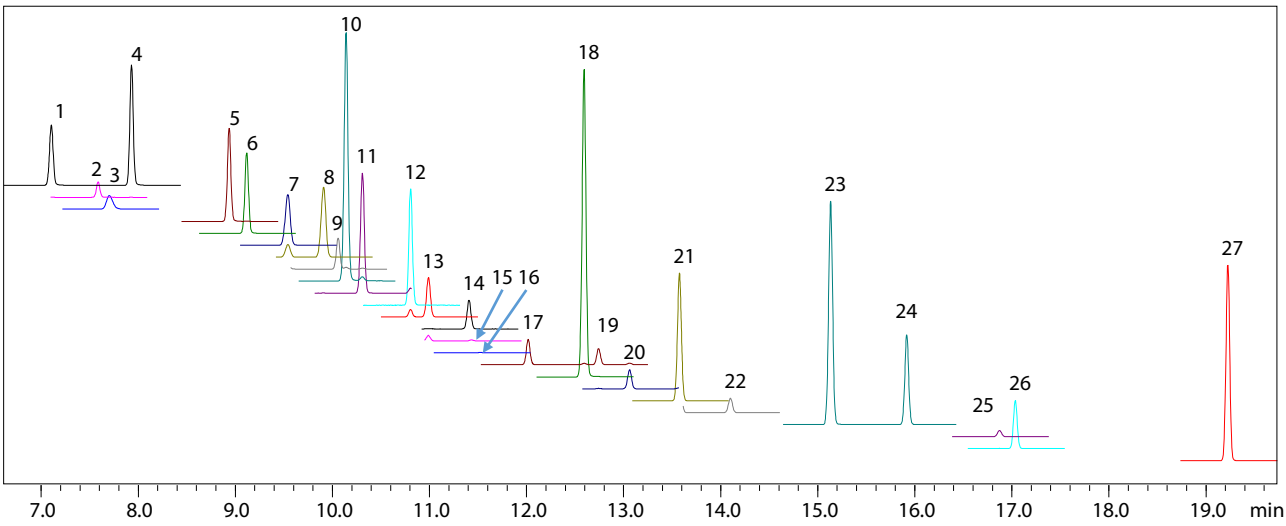

Fig. 2 Total Ion Current Chromatogram (TICC) of 5 µg/L Calibration Standards

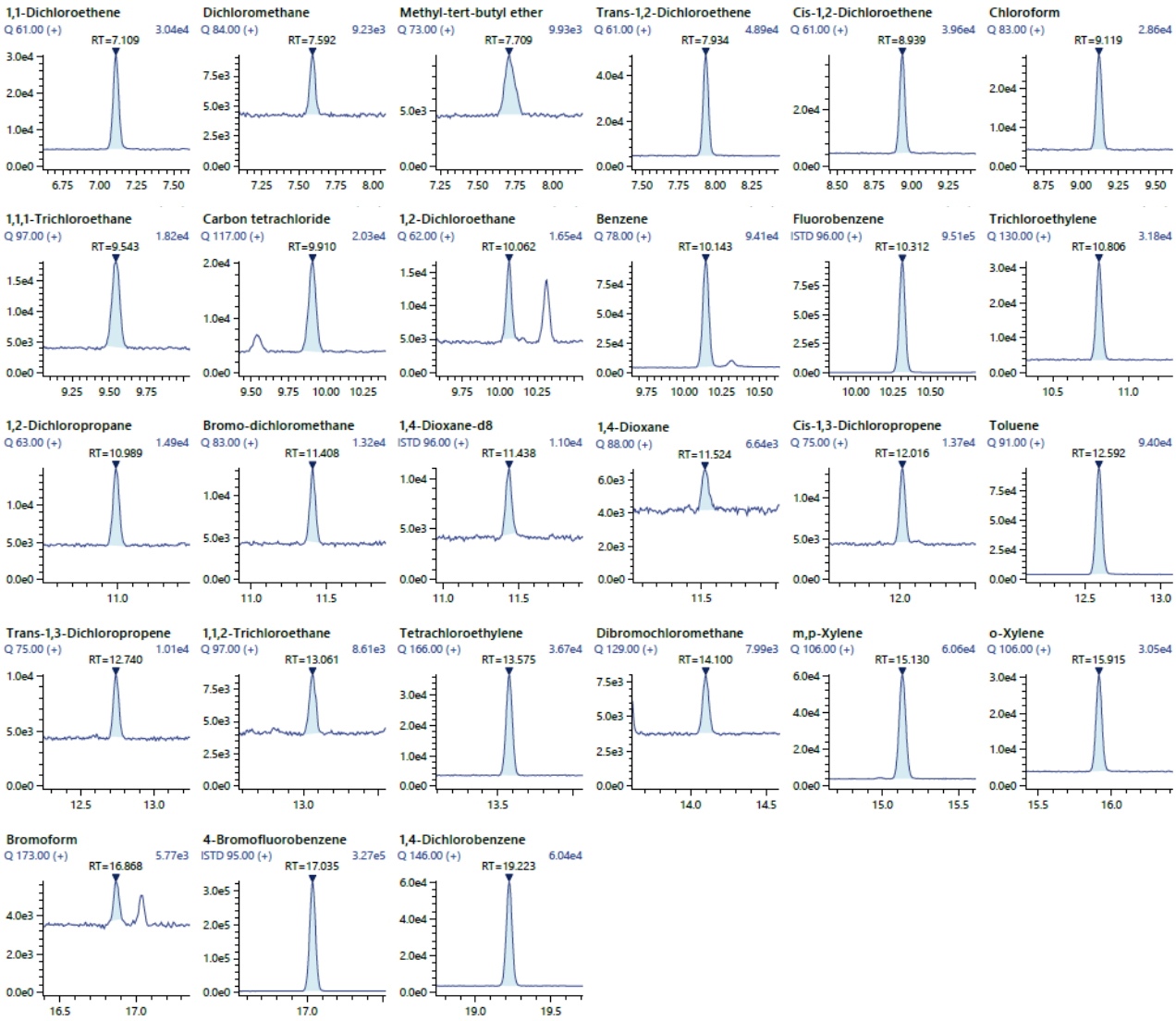

Fig. 3 Mass Chromatograms from 0.2 µg/L Calibration Standards (1,4-dioxane at 5 µg/L)
Smoothing: standard; smoothing frequency: once; and smoothing width: 1 sec

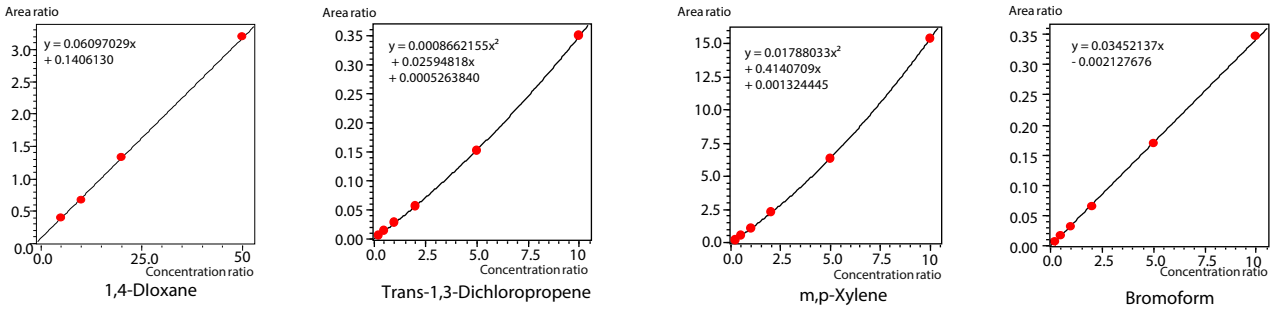

Fig. 4 Calibration Curves of Selected Compounds

Table 2 Analysis Parameters, Quantitative Results, and Calibration Curve Data

#	Compound Name	ISTD Group	Quantifier Ion	Qualifier Ion	Quantitative Result (µg/L)	Repeatability of Quantitative Result (%)	Accuracy (%)	Line Type	Weighting	R^2
1	1,1-Dichloroethylene	1	61	98	0.180	1.5	90.0	Linear	1/C	0.99817
2	Dichloromethane	1	84	86	0.193	3.8	96.7	Linear	1/C	0.99915
3	Methyl-tert-butyl ether	1	73	57	0.222	2.2	111.2	Linear	1/C	0.99880
4	Trans-1,2-Dichloroethylene	1	61	98	0.196	1.0	97.9	Linear	1/C	0.99965
5	Cis-1,2-Dichloroethylene	1	61	98	0.207	0.7	103.4	Linear	1/C	0.99931
6	Chloroform	1	83	85	0.221	1.4	110.3	Linear	1/C	0.99899
7	1,1,1-Trichloroethane	1	97	99	0.213	2.4	106.5	Linear	1/C	0.99861
8	Carbon tetrachloride	1	117	119	0.226	2.9	113.2	Linear	1/C	0.99879
9	1,2-Dichloroethane	1	62	64	0.222	3.2	111.1	Linear	1/C	0.99861
10	Benzene	1	78	77	0.217	0.4	108.5	Linear	1/C	0.99911
11	Fluorobenzene (IS)	1	96	70	-	-	-	-	-	-
12	Trichloroethylene	1	130	132	0.218	1.7	109.2	Linear	1/C	0.99906
13	1,2-Dichloropropane	1	63	62	0.185	3.4	92.5	Quadratic	1/C	0.99995
14	Bromo-dichloromethane	1	83	85	0.221	2.2	110.6	Linear	1/C	0.99764
15	1,4-Dioxane-d8 (IS)	2	96	64	-	-	-	-	-	-
16	1,4-Dioxane	2	88	58	5.068	4.7	101.4	Linear	1/C	0.99913
17	Cis-1,3-Dichloropropene	1	75	49	0.170	3.6	84.9	Quadratic	1/C	0.99979
18	Toluene	1	91	92	0.198	0.8	99.2	Quadratic	1/C	0.99993
19	Trans-1,3-Dichloropropene	1	75	49	0.167	3.1	83.3	Quadratic	1/C	0.99984
20	1,1,2-Trichloroethane	1	97	83	0.181	3.4	90.3	Quadratic	1/C	0.99994
21	Tetrachloroethylene	3	166	164	0.219	1.4	109.6	Linear	1/C	0.99949
22	Dibromochloromethane	3	129	127	0.215	4.3	107.7	Linear	1/C	0.99816
23	m,p-Xylene	3	106	105	0.204	2.2	102.0	Quadratic	1/C	0.99999
24	o-Xylene	3	106	105	0.197	2.2	98.5	Quadratic	1/C	0.99992
25	Bromoform	3	173	171	0.229	2.0	114.5	Linear	1/C	0.99924
26	4-Bromofluorobenzene (IS)	3	95	174	-	-	-	-	-	-
27	1,4-Dichlorobenzene	3	146	148	0.227	0.7	113.4	Linear	1/C	0.99767

■ Conclusion

VOC levels in water were measured using an HS-20 NX in trap mode and a GCMS-QP2050 with nitrogen carrier gas. Trap mode allowed VOCs to be measured in water with a high degree of sensitivity, even with nitrogen carrier gas.

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