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GCMS-QP™2020 NX TD-30 GC-MS

Application News

Measurement of VOCs by Thermal Desorption GC-MS Using Hydrogen Carrier Gas

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User Benefits

- Analysis is possible using hydrogen, which is economical and stably available, as the carrier gas as an alternative to helium gas.
- Thermal desorption GC/MS enables high throughput analysis of VOCs because solvent extraction work is not required.

■ Introduction

In GC/MS analysis, helium is generally used as the carrier gas, but instability of helium gas supplies has become a problem in recent years. Since the analysis itself must be stopped if this carrier gas cannot be obtained, analysis using hydrogen as a carrier gas in place of helium has attracted attention. When hydrogen gas is used, sensitivity to specific compounds may be inferior to that with helium gas, but hydrogen gas has the advantages of low cost and stable availability.

The Shimadzu TD-30 Series thermal desorption system supports the use of hydrogen as a carrier gas in addition to the helium and nitrogen. Therefore, in this experiment, volatile organic compounds (VOCs) were measured with a TD-30R+GCMS-QP2020 NX system using hydrogen as the carrier gas.

The VOCs are harmful volatile organic compounds that exist in air and are monitored under various environments, such as factories, urban areas, and indoor environments, as one index of the level of atmospheric pollution. Efficient thermal desorption (TD)-GC/MS, which does not require solvent extraction, was used in this VOC measurement. Here, an analysis of the VOCs which are frequently analyzed using hydrogen as the carrier gas was conducted by TD-GC/MS, and linearity and repeatability were confirmed.

The reader may also refer to Application News 01-00366-EN in which nitrogen was used as the carrier gas in place of helium in the VOC measurement.

Fig. 1 TD-30R+GCMS-QP™2020 NX

■ Experiment

In this experiment, mixed standard samples of toluene, ethylbenzene, *o, m, p*-xylene, styrene, *p*-dichlorobenzene, and tetradecane were prepared by dilution with methanol to obtain concentrations of 10 mg/L, 50 mg/L, 100 mg/L, 500 mg/L, and 1000 mg/L, and 1 µL of each sample was added to a TENAX-TA trap tube for the analysis. Table 1 shows the analysis conditions.

Table 1 Analysis Conditions

Model	: GCMS-QP2020 NX
Autosampler	: TD-30R
[TD-30R]	
Tube desorb temp.	: 280 ˚C (10 min)
Tube desorb flow	: 60 mL/min (N_2)
Trap cooling temp.	: -20 ˚C *
Trap desorb temp.	: 250 ˚C (10 min)
Joint temp.	: 250 ˚C
Valve temp.	: 250 ˚C
Transfer line temp.	: 250 ˚C
Dry Purge	: 25 ˚C (20 mL/min, 3 min)
[GC]	
Injection Mode	: Split
Split Ratio	: 20
Carrier Gas	: H_2
Carrier Gas Control	: Pressure (80.0 kPa)
Column	: SH-I-5Sil MS (P/N 221-75954-30) (30 m × 0.25 mm I.D., 0.25 µm)
Column temp.	: 40 ˚C (2 min) - 20 ˚C/min - 300 ˚C (3 min)
[MS]	
Ion source temp.	: 200 ˚C
Interface temp.	: 230 ˚C
Acquisition mode	: Scan
Event time	: 0.3 s
m/z range	: *m/z*=20-600

* If nitrogen is used as the purge gas, the trap cooling temperature may not decrease to -20 ˚ C in some cases, depending on the room temperature.

■ Repeatability and Calibration Curve Results

The mixed standard samples of toluene, ethylbenzene, *o, m, p*-xylene, styrene, *p*-dichlorobenzene, and tetradecane were measured. Fig. 2 shows the mass chromatograms, and Table 2 shows the results of the linearity of the calibration curve and repeatability. In the repeatability results for 100 ng of each compound, good repeatability of 5 % or less was obtained for all target compounds.

Regarding linearity, a calibration curve was prepared for the range of 10 ng to 1000 ng, and a satisfactory result of R^2>0.998 was obtained for toluene (Fig. 3). Good results of R^2>0.996 were also obtained for the other compounds, that is, ethylbenzene, *o, m, p*-xylene, styrene, *p*-dichlorobenzene, and tetradecane.

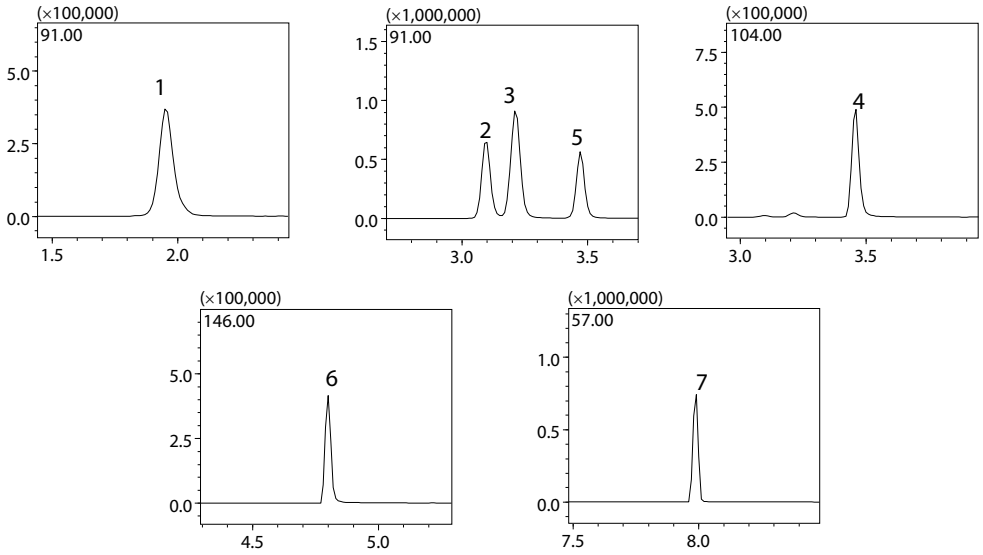

Fig. 2 Mass Chromatograms (100 ng)

Table 2 Linearity of Calibration Curve and Repeatability at 100 ng

ID	Compound	Retention time	*m/z*	Linearity (R^2)	Linearity (R)	100 ng peak area %RSD (n=5)
1	Toluene	1.943	91	0.9989474	0.9994736	2.0
2	Ethylbenzene	3.091	91	0.9986216	0.9993105	3.6
3	*m*, *p*-Xylene	3.201	91	0.9969509	0.9984743	2.9
4	Styrene	3.446	104	0.9992695	0.9996347	3.9
5	*o*-Xylene	3.472	91	0.9983703	0.9991848	3.7
6	*p*-Dichlorobenzene	4.793	146	0.9992117	0.9996058	3.5
7	Tetradecane	7.982	57	0.9999323	0.9999662	4.9

■ Conclusion

In this article volatile organic compounds (VOCs) were analyzed with a TD-30R+GCMS-QP2020 NX system using hydrogen as the carrier gas. Satisfactory results were obtained for linearity and repeatability, demonstrating that it is possible to measure the VOCs (toluene, ethylbenzene, *o, m, p*-xylene, styrene, *p*-dichlorobenzene, and tetradecane) by this technique with hydrogen as the carrier gas.

It should be noted that identical data will not necessarily be obtained in all analyses if the analysis is performed after changing the carrier gas from helium or nitrogen to hydrogen. Therefore, when the carrier gas is changed, it is important to study the analysis conditions and target compounds to be measured in advance.

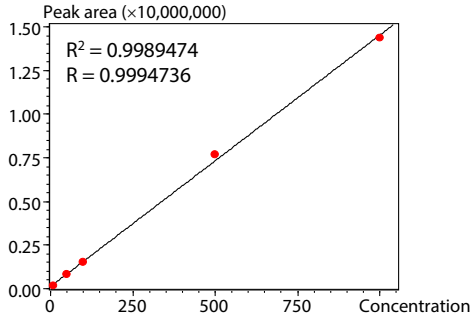

Fig. 3 Calibration Curve of Toluene

■ Points to Note when Using Hydrogen Gas

Hydrogen is a dangerous gas that explodes easily if mishandled. When using hydrogen gas, the analyst should fully understand the nature of the gas and handle it properly.

■ Proposal for Helium Gas Supply Shortages

Shimadzu provides a special site which summarizes functions for reducing helium gas consumption and points to note when switching to another carrier gas. Please be sure to refer to this site.

Countermeasure and Solutions for Helium Gas Supply Shortages

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