

Measurement of VOCs in Vehicle Interiors Using Thermal Desorption GC-MS with Nitrogen as the Carrier Gas

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User Benefits

- ◆ Analysis can be performed using nitrogen as the carrier gas, which is readily available compared to helium gas.
- ◆ Thermal desorption GC-MS facilitates high throughput VOC analysis because it does not require solvent extraction.
- ◆ Analysis efficiency is heightened by an automatic internal standard addition function and a retrapping function of sample.

Introduction

The amount of toxic volatile organic compounds (VOC) in the air is one index of the extent of air pollution and is used for environmental monitoring in factories, urban areas, and indoor environments. VOCs can be measured efficiently using TD-GC/MS, which does not require solvent extraction.

Helium is commonly used as the carrier gas for GC/MS analysis, but in recent years there have been supply problems. If this carrier gas cannot be obtained, the analysis itself must stop. In this article, analysis was performed using nitrogen rather than helium as the carrier gas. Nitrogen gas can be less sensitive than helium gas, but it is inexpensive and readily available.

An analysis was performed of VOCs typically analyzed in voluntary assessments of vehicle interiors and vehicle interior materials, using thermal desorption GC/MS with nitrogen as the carrier gas. The calibration curve, repeatability and efficiency of retrapping were also tested.

Analysis System

In the TD-30R thermal desorption instrument, the sample gas collected within a sample tube is thermally desorbed and then concentrated in a cold trap before injection into the GC-MS. In the TD-30R, there is a retrapping function that collects the split sample gas again in a tube, and a function that automatically adds the internal standard. Using this retrapping function reduces the risk of analysis failure. In addition, the value calculated by the retrapping function can be corrected by using this function together with the internal standard auto addition.

Measurement

The standard mixture of toluene, benzene, ethylbenzene, *m,p,o*-xylene, styrene, 1,4-dichlorobenzene, and tetradecane diluted with methanol were prepared with concentrations of 10 ppm, 40 ppm, 100 ppm, 400 ppm, and 1000 ppm. These standard mixtures were added 1 μ L to each TENAX-TA sample tube and then analyzed. The analytical conditions are shown in Table 1. During the analysis, toluene-d₈ was added by the TD-30R's internal standard automatic addition function.

Table 1 Analytical Conditions

Model	GCMS-QP 2020 NX
Autosampler:	TD-30R
[TD-30R]	
Tube Desorb. Temp.:	280 °C (10 min)
Tube Desorb. Flow:	60 mL/min
Trap Cooling Temp.:	-20 °C
Trap Desorb Temp.:	280 °C (10 min)
Joint Temp.:	250 °C
Valve Temp.:	250 °C
Transfer Line Temp.:	250 °C
Internal STD:	Variable volume (20 mL/min, 0.1 min)
Restore:	ON
Dry Purge:	-20 °C (20 mL/min, 3 min)
[GC]	
Injection Mode:	Split
Split Ratio:	50
Carrier Gas:	N ₂
Carrier Gas Control:	Linear velocity (40 cm/sec)
Column:	SH-5MS (P/N 221-758555-30) (30 m × 0.25 mm I.D., 0.25 μ m)
Column Temp.:	40 °C (1 min) – 10 °C/min – 100 °C -40 °C/min – 200 °C -20 °C/min – 300 °C (5 min)
[MS]	
Ion Source Temp.:	230 °C
Interface Temp.:	280 °C
Acquisition Mode:	Scan
Event Time:	0.3 sec
<i>m/z</i> Range:	<i>m/z</i> = 45 – 600



Fig. 1 TD-30R + GCMS-QP2020 NX

■ The Calibration Curve, Repeatability, and Retrapping Results

The standard mixture of benzene, toluene, ethylbenzene, *m,p*-xylene, *o*-xylene, styrene, 1,4-dichlorobenzene, and tetradecane were measured. The TIC chromatogram is shown in Fig. 1, and the calibration curve linearity, repeatability, and retrapping results are shown in Table 1. The range of the calibration curve created was 10 ng to 1000 ng, and for all compounds, a good result was obtained, with $R > 0.999$.

As an example, the calibration curve for toluene is shown in Fig. 3. In addition, when the repeatability was checked at 10 ng, the minimum concentration in the calibration curve, the %RSD ($n = 5$) for all compounds was found to be 5 % or less. Furthermore, when the measurement repeatability was checked by retrapping at 10 ng using the TD-30R retrapping (Restore) function, the %RSD ($n = 5$) for all compounds was 6 % or less.

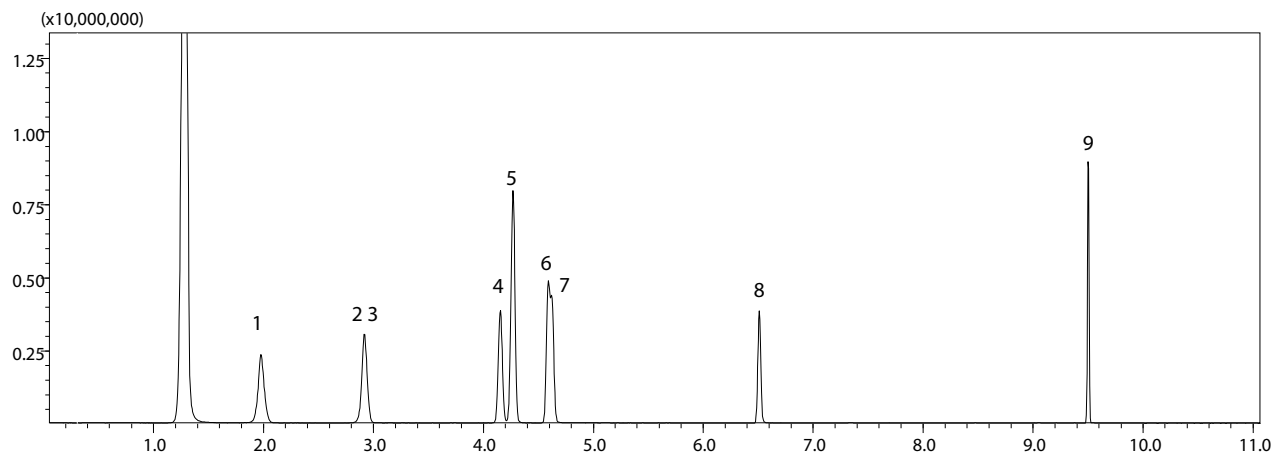


Fig. 2 TIC Chromatogram for the 1000 ng Standard Sample

Table 2 Results for Calibration Curve Linearity, Repeatability at 10 ng, and Retrapping Results

ID	Compound	Retention Time	<i>m/z</i>	Linearity (R)	10 ng Average Concentration (n = 5)		10 ng Concentration %RSD (n = 5)	
					1st time	Retrapping	1st time	Retrapping
1	Benzene	1.978	78	0.9997903	8.9	9.0	2.6	2.1
2	Toluene-D8	2.871	98	-	-	-	-	-
3	Toluene	2.918	91	0.9998511	9.3	9.2	4.6	3.6
4	Ethylbenzene	4.154	91	0.9997918	8.4	8.4	3.9	1.7
5	<i>m,p</i> -Xylene	4.270	91	0.9998336	9.3	9.3	2.3	3.6
6	Styrene	4.587	104	0.999927	10.7	10.5	3.8	3.5
7	<i>o</i> -Xylene	4.624	91	0.9997498	9.4	9.3	3.7	5.9
8	Benzene, 1,4-dichloro-	6.510	146	0.999958	9.7	9.7	3.9	5.9
9	Tetradecane	9.502	57	0.9999466	11.1	10.8	2.1	2.3

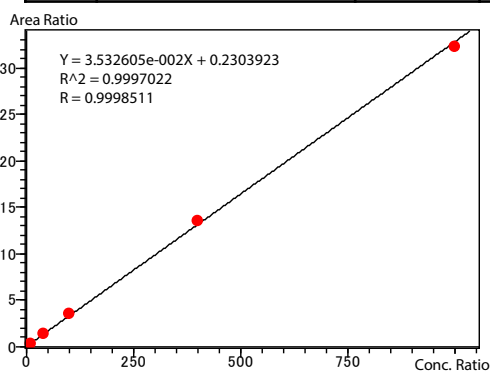


Fig. 3 Toluene Calibration Curve Analyzed with Nitrogen as the Carrier Gas (10 ng, 40 ng, 100 ng, 400 ng, 1000 ng)

■ Conclusion

This article described an analysis of VOCs frequently analyzed in voluntary assessments of vehicle interiors and vehicle interior materials using the TD-30R + GCMS QP2020 NX with nitrogen as the carrier gas. Favorable values were obtained for the calibration curve, repeatability, and Retrapping results.

Using this method it was possible to measure nine VOCs (toluene, benzene, ethylbenzene, *m,p,o*-xylene, styrene, 1,4-dichlorobenzene, and tetradecane) within a vehicle interior using nitrogen as the carrier gas.