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Application Data Sheet

No.82

GC-MS
Gas Chromatograph Mass Spectrometer

Analysis of Di(2-ethylhexyl)phthalate by GC-MS Using Hydrogen Carrier Gas

Helium gas is typically used as a carrier gas for GC-MS. However, in recent years, the insufficient supply of helium gas and its increase in price have caused problems, prompting global efforts to address these problems. The use of hydrogen gas as an alternative carrier gas holds promise for addressing the issue.

This data sheet presents the application of hydrogen carrier gas to the analysis of di(2-ethylhexyl)phthalate using the GCMS-QP2010 Ultra, which features a Dual Inlet Turbo Molecular Pump.

Experimental

A standard sample of di(2-ethylhexyl)phthalate was diluted with hexane for phthalate ester experimental use (from Kanto Chemical Co., Inc.) to prepare a 10 mg/L standard solution. Also, a standard sample of phenanthrene-d10 (from Wako Pure Chemical Industries, Ltd.) was diluted with hexane for phthalate ester experimental use to prepare a 10 mg/L internal standard solution. The 10 mg/L standard solution of di(2-ethylhexyl)phthalate was then diluted in stages to prepare 0.05, 0.1, 0.5, 1, and 5 mg/L standard solutions for creating calibration curves, and the internal standard substance was adjusted to 0.5 mg/L in all the standard solutions. Table 1 shows the analytical conditions.

Table 1 Analytical Conditions

GC-MS:	GCMS-QP2010 Ultra		
Column:	Rtx®-5MS (Length 30 m, 0.25 mm I.D., df = 1 μm) (Restek Corporation, P/N: 12653)		
Glass Liner:	Deactivated splitless glass liner with wool (P/N: 221-48876-03)		
[GC]		[MS]	
Injection Temp.:	250 °C	Interface Temp.:	280 °C
Column Oven Temp.:	100 °C (1 min) → (20 °C/min) → 230 °C → (10°C/min) → 320 °C (3min)	Ion Source Temp.:	200 °C
Injection Mode:	Splitless	[Scan Conditions]	
Flow Control Mode:	Linear Velocity (65 cm/sec)	Mass Range:	*m/z* 80 - 300
Injection Volume:	1 μL	Scan Event Time:	0.3 sec
		[SIM Conditions]	
		Monitoring *m/z*:	*m/z* 149, 167
		SIM Event Time:	0.3 sec

Analytical Results

The mass spectral patterns of some compounds are known to change when hydrogen carrier gas is used. To evaluate this, the 5 mg/L di(2-ethylhexyl)phthalate was measured in scan mode. The resulting mass spectra, shown in Fig. 1, indicate no change in the mass spectral pattern for di(2-ethylhexyl)phthalate.

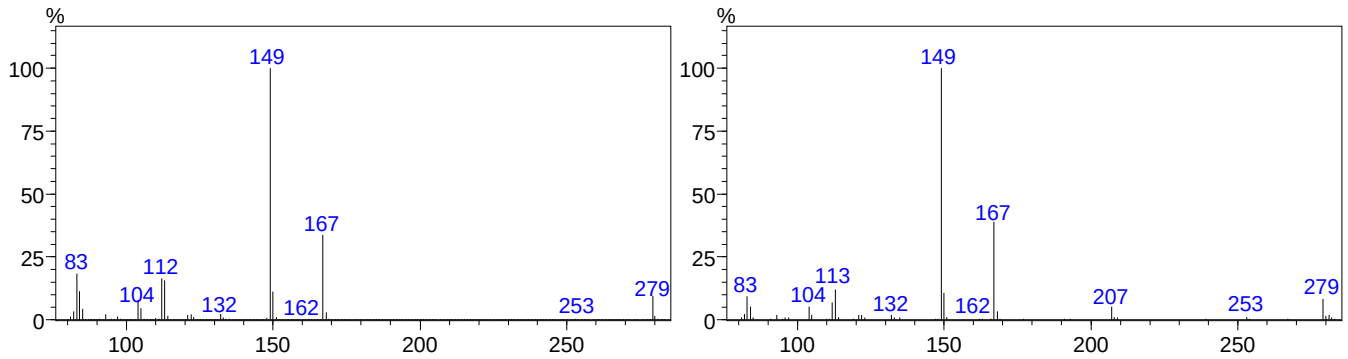

Fig. 1 Mass Spectra for Di(2-ethylhexyl)phthalate (left: hydrogen carrier gas, right: helium carrier gas)

In addition, it has been reported that using hydrogen carrier gas results in lower sensitivity than when helium is used. To test this, the 0.05 mg/L di(2-ethylhexyl)phthalate was measured in SIM mode. The resulting mass chromatograms, shown in Fig. 2, indicate that the S/N ratio was 94, and that even at 0.05 mg/L, it was detected with high sensitivity.

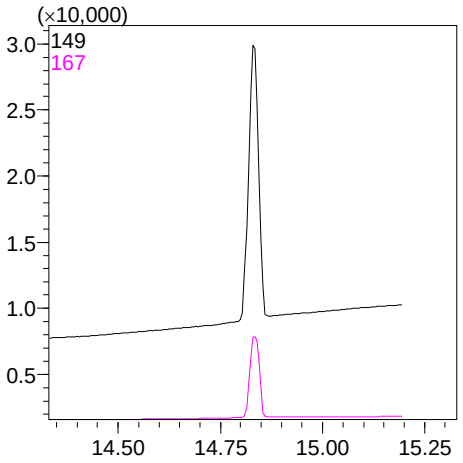

Fig. 2 SIM Mass Chromatograms for 0.05 mg/L Di(2-ethylhexyl)phthalate

The repeatability and calibration curve (concentration range of 0.05 to 5 mg/L) were checked for quantitative performance. The results are shown in Table 2 and Fig. 3. At the concentration of 0.05 mg/L, the repeatability (n = 5) was 2.63 %RSD and the coefficient of correlation of the calibration curve was 0.999, which were both satisfactory.

Table 2 Repeatability (n = 5, 0.05 mg/L)*

No.	Component Name	Data 1	Data 2	Data 3	Data 4	Data 5	%RSD
1	Di(2-ethylhexyl)phthalate	0.009251	0.008872	0.008944	0.008717	0.009248	2.63

* The data indicates the peak area ratio with respect to the internal standard.

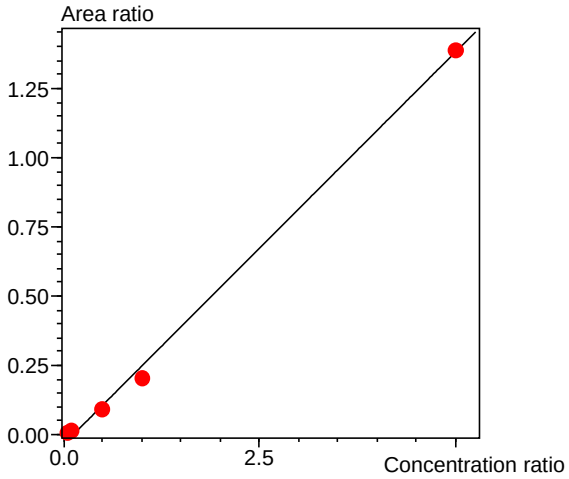

Fig. 3 Di(2-ethylhexyl)phthalate Calibration Curve

Summary

The application of hydrogen carrier gas to di(2-ethylhexyl)phthalate showed no change in the mass spectral pattern, thus demonstrating that the GCMS-QP2010 Ultra, featuring a Dual Inlet Turbo Molecular Pump, provides satisfactory sensitivity and quantitative performance.

Hydrogen gas is combustible, so it is necessary to handle it carefully. For safety measures, please refer to our web site: http://www.shimadzu.com/an/gc/support/faq/bombe/bombe1.html

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