

Application News

No. G298A

Gas Chromatography

Examples of Analyzing Organic Compound Species with Hydrogen Carrier Gas Using Nexis GC-2030

Hydrogen is lower in cost than helium, which is often used as a carrier gas for gas chromatography. In addition, it is known to allow favorable separation at higher linear velocities compared to helium. On the other hand, it is a combustible gas and therefore requires great care in handling.

The new Nexis GC-2030 gas chromatograph can be equipped with a hydrogen sensor for detecting the hydrogen concentration within the column oven (Fig. 1). When the hydrogen concentration inside the oven exceeds 0.4 %, an error message is displayed, all temperature controls are stopped and the oven flap is fully opened. When the concentration reaches 2 %, the instrument is forcibly stopped to prevent an accident. This sensor allows safe use of hydrogen as a carrier gas. This article introduces example analyses of a mixed solution containing typical organic compounds with hydrogen carrier gas using the new Nexis GC-2030 gas chromatograph.

Y. Nagao, T. Murata

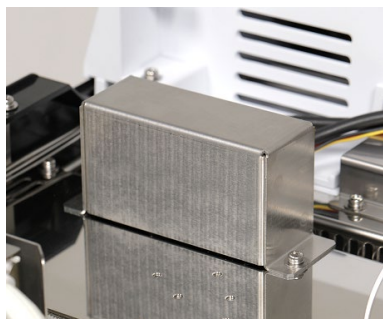


Fig. 1 Appearance of Hydrogen Sensor

Example Analysis 1

A mixed solution of organic compounds was prepared by dissolving 10 organic compound species in ethanol to contain 1 vol% of each compound. Table 1 lists the analysis conditions. A hydrogen flame ionization detector (FID) requires a constant flow rate of hydrogen for the detector gas. However, when using hydrogen for the carrier gas, the total volume of detector gas and carrier gas is supplied to the FID. This means that when the carrier gas flow rate of the column changes, the condition of the FID also changes. To avoid this, we employed the constant column flow rate mode in this analysis. Fig. 2 shows the obtained chromatograms.

We can see that analysis using hydrogen for the carrier gas achieved a shorter analysis time than that with helium and the degree of separation is of the same level. When using hydrogen, the linear velocity of carrier gas at the initial column temperature was 54.1 cm/s and when using helium the value was 45.3 cm/s.

Table 1 GC Analysis Conditions

Model	: Nexis GC-2030, AOC-20i
Injection Mode	: Split mode
Injection Volume	: 0.5 μ L
Split Ratio	: 1:50
Injection Temp.	: 260 $^{\circ}$ C
Carrier Gas	: H ₂ /He
Carrier Gas Control	: Constant column flow rate (3 mL/min)
Column	: SH-PolarWax (30 m \times 0.32 mm I.D., 0.50 μ m) ^{*1}
Column Temp.	: 50 $^{\circ}$ C (2 min) - 10 $^{\circ}$ C/min - 200 $^{\circ}$ C
Detector	: FID
Detector Temp.	: 260 $^{\circ}$ C
Detector Gas	: H ₂ 32.0 mL/min, Air 200 mL/min
Makeup Gas	: With H ₂ carrier gas: N ₂ (24 mL/min) With He carrier gas: He (24 mL/min)

*1 P/N: 227-36251-01

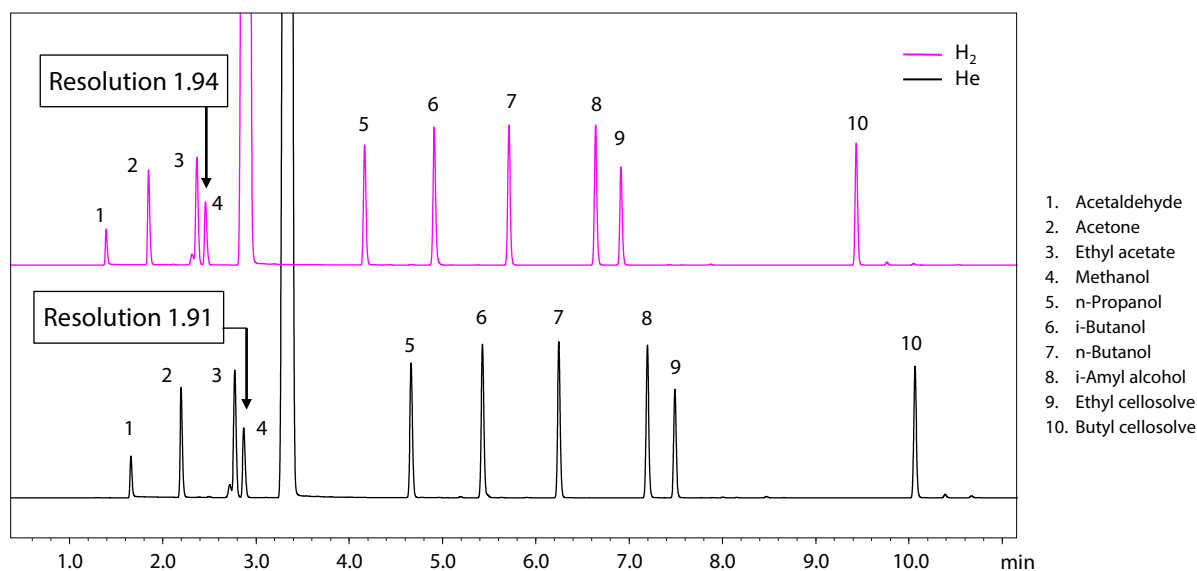


Fig. 2 Chromatograms of Mixed Solution of 10 Species (Pink: H₂ Carrier Gas, Black: He Carrier Gas)

Example Analysis 2

Narrow-bore columns with an inner diameter of 0.10 mm or 0.18 mm are known to produce higher theoretical plate numbers when compared to normal capillary columns because of their small inner diameter. On the other hand, narrow-bore columns have a large column resistance. This calls for an extremely high injection port pressure and at times may make pressure control difficult. Hydrogen has a lower viscosity compared to helium and nitrogen and a relatively lower injection port pressure is possible, enabling it to be used well together with narrow-bore columns which have a high theoretical plate number.

In this analysis, a narrow-bore column with an inner diameter of 0.10 mm and hydrogen carrier gas were used. Table 2 lists the detailed analysis conditions.

Table 2 GC Analysis Conditions

Model	: Nexis GC-2030, AOC-20i
Injection Mode	: Split mode
Injection Volume	: 0.5 µL
Split Ratio	: 1:100
Injection Temp.	: 260 °C
Carrier Gas	: H ₂ /He
Carrier Gas Control	: Constant column flow rate (0.8 mL/min)
Column	: SH-WAX (20 m × 0.10 mm I.D., 0.10 µm) ^{*1}
Column Temp.	: 40 °C - 4 °C/min - 50 °C (1 min) - 40 °C/min - 90 °C (2 min) ^{*2}
Detector	: FID
Detector Temp.	: 260 °C
Detector Gas	: H ₂ 32.0 mL/min, Air 200 mL/min
Makeup Gas	: With H ₂ carrier gas: N ₂ (24 mL/min) With He carrier gas: He (24 mL/min)

*1 P/N: 227-36356-01

*2 Only when using He

A mixed solution of organic solvents was prepared by dissolving nine organic solvent species in hexane to contain 1000 ppm (v/v) of each solvent. Fig. 3 shows the obtained chromatograms.

The analysis time was shortened when using hydrogen carrier gas. The linear velocity and the injection port pressure at the initial column temperature was 55.4 cm/s and 371.5 kPa respectively. When using helium, the values were 38.4 cm/s and 594.7 kPa respectively.

Measurement was performed five times with hydrogen carrier gas. Table 3 shows the repeatability in the obtained area values and retention times. Favorable repeatability was obtained with all compounds.

Table 3 Repeatability with each Compound Species (n = 5)

No.	Compound Name	Area Value %RSD	Retention Time %RSD
1	Acetone	0.725	0.007
2	Ethyl acetate	0.816	0.010
3	Isopropyl alcohol (IPA)	0.700	0.017
4	Methyl isobutyl ketone (MiBK)	0.835	0.014
5	Toluene	0.831	0.017
6	Butyl acetate	1.119	0.017
7	2-Hexanone (MBK)	0.667	0.018
8	Propylene glycol monomethyl ether	0.763	0.017
9	n-Butanol	0.835	0.014

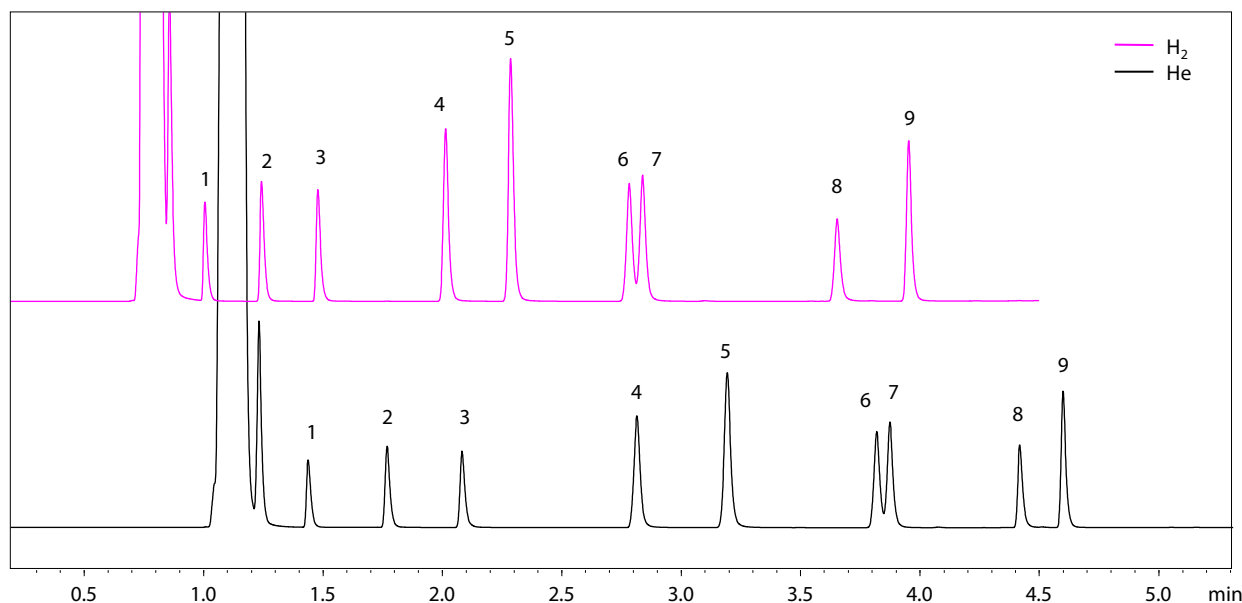


Fig. 3 Chromatograms of Mixed Solution of Organic Solvents (Pink: H₂ Carrier Gas, Black: He Carrier Gas)