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Application News

No. G297

Gas Chromatography

Comparison of Separation by MICROPACKED-ST Columns

Barrier discharge ionization detectors (BID) are capable of detecting almost all compounds, excluding helium and neon, with a higher sensitivity compared to general detectors such as thermal conductivity detectors (TCD) and flame-ionization detectors (FID). When analyzing inorganic gases together with small hydrocarbons, simultaneous analysis with high sensitivity is possible by using a BID as the detector and the MICROPACKED-ST as the column. Until now we have introduced a number of example analyses using MICROPACKED-ST columns and now recently, 1.0 m and 3.0 m long columns have become available, enhancing the applicability of the columns.

This article introduces examples of separation using MICROPACKED-ST columns of different lengths: 1.0 m, 2.0 m, and 3.0 m.

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Column Information and Analysis Conditions

A MICROPACKED-ST column is a stainless steel tube with an inner diameter of 1.0 mm and is packed with SHINCARBON ST. Therefore, carrier gas can be controlled by inputting information of a capillary column with an approximated flow resistance.

Table 1 shows the common analysis conditions and Tables 2 to 4 show the analysis conditions for MICROPACKED-ST columns 1.0 m, 2.0 m, and 3.0 m long respectively. Although BIDs are easily influenced by changes in the carrier gas flow rate, the Nexis GC-2030 is capable of constant flow rate control, making it possible to maintain a constant flow rate with no need to create a pressure program.

Table 1 Common Analysis Conditions

Model	:	Nexis GC-2030
Detector	:	BID-2030
Inj. Mode	:	Split 1:4
Inj.Temp.	:	150 °C
Carrier Gas	:	7 mL/min
Purge flow	:	3 mL/min
Det. Temp.	:	280 °C
Discharge Gas	:	50 mL/min (He)
Inj. Volume	:	1.0 mL (Using MGS-2030) 50 µL (Using a gas-tight syringe)

Table 2 Analysis Conditions for the 1.0 m MICROPACKED-ST

Column	:	MICROPACKED-ST 1.0 m × 1.0 mm I.D. (Input 125 m × 0.50 mm I.D. and df = 15 µm for flow rate calculation)
Carrier Gas *	:	He, 152.8 kPa (2.0 min) - 21.7 kPa/min - 242.2 kPa (0 min) - 13.5 kPa/min - 269.3.6 kPa (2.87 min) (Pressure Mode)
Column Temp.	:	35 °C (2.0 min) - 40 °C/min - 200 °C (0 min) - 25 °C/min - 250 °C (2.87 min)

Table 3 Analysis Conditions for the 2.0 m MICROPACKED-ST

Column	:	MICROPACKED-ST 2.0 m × 1.0 mm I.D. (Input 250 m × 0.50 mm I.D. and df = 10 µm for flow rate calculation)
Carrier Gas *	:	He, 226.8 kPa (2.5 min) - 15.2 kPa/min - 390.1 kPa (5.95 min) - 11.2 kPa/min - 405.1 kPa (5.42 min) (Pressure Mode)
Column Temp.	:	35 °C (2.5 min) - 20 °C/min - 250 °C (0 min) - 15 °C/min - 270 °C (5.42 min)

Table 4 Analysis Conditions for the 3.0 m MICROPACKED-ST

Column	:	MICROPACKED-ST 3.0 m × 1.0 mm I.D. (Input 250 m × 0.50 mm I.D. and df = 30 µm for flow rate calculation)
Carrier Gas *	:	He, 282.0 kPa (4.5 min) - 9.1 kPa/min - 450 kPa (9.98 min) (Pressure Mode)
Column Temp.	:	35 °C (4.5 min) - 10 °C/min - 270 °C (10 min)

* With GC-2010 Plus, approximated to a constant flow rate using a pressure program

Checking Separation of Oxygen and Nitrogen

Fig. 1 shows the chromatograms obtained by analyses after injecting 50 µL of indoor air using a gas-tight syringe. With the 1.0 m and 2.0 m columns, separation was incomplete with a resolution of under 1.5. On the other hand, the 3.0 m column achieved complete separation of oxygen and nitrogen with a resolution of over 1.5.

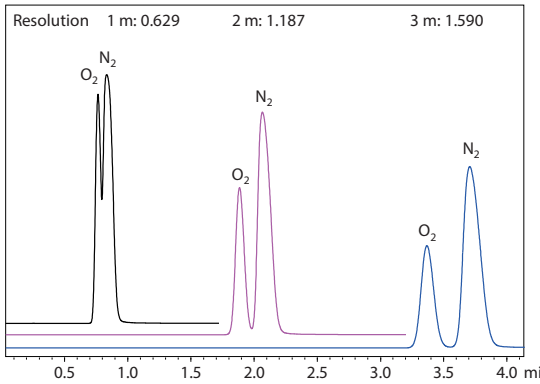

Fig. 1 Comparison of Separation of Oxygen and Nitrogen

Standard Gas Analysis of Inorganic Gases and Small Hydrocarbons of Low and High Concentrations

Low-concentration standard gas (1.0 mL) of inorganic gases and small hydrocarbons was analyzed by using the MGS-2030 manual gas sampler (P/N: 221-78990-41) and SPLITTER-INJ (P/N: 221-78280-41). The obtained chromatograms are indicated in Fig. 2. The analysis time with the 1.0 m column is almost half the analysis time with the 2.0 m column, showing that reduction of analysis time is possible. In addition, the rise in the baseline is small compared to that with the 2.0 m column. This contributes to noise suppression during heating and therefore enables high-sensitivity analysis of propane and propylene. On the other hand, with the 3.0 m column, propylene of 5 ppm could not be detected due to the characteristics of the packing material used for MICROPACKED-ST columns; propylene easily adsorbs to the packing material.

We then analyzed high-concentration standard gas of inorganic gases and small hydrocarbons by injecting 50 µL using a gas-tight syringe. The obtained chromatograms are indicated in Fig. 3. The chromatograms indicate that by reducing the injection volume, analysis of high-concentration samples is possible.

The following are guidelines for selecting each column.

1.0 m : Where separation of oxygen and nitrogen is not necessary
For high-speed analysis, quantification of trace amounts of propylene

2.0 m : General analysis of inorganic gases and small hydrocarbons

3.0 m : Where separation of oxygen and nitrogen is necessary
Measurement of impurities in high-concentration substances

Analysis of inorganic gases and small hydrocarbons can be optimized by selecting a column length appropriate for the purpose of analysis.

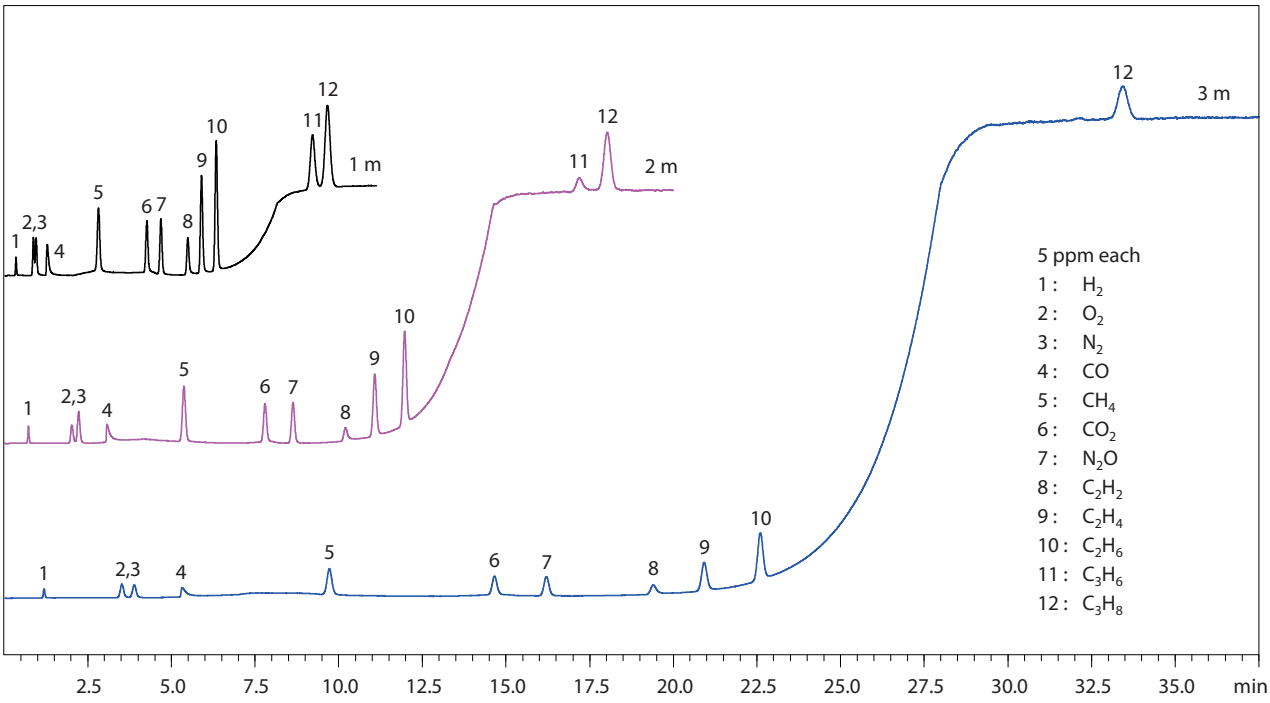

Fig. 2 Comparison of Chromatograms of Low-concentration Standard Gas of Inorganic Gases and Small Hydrocarbons

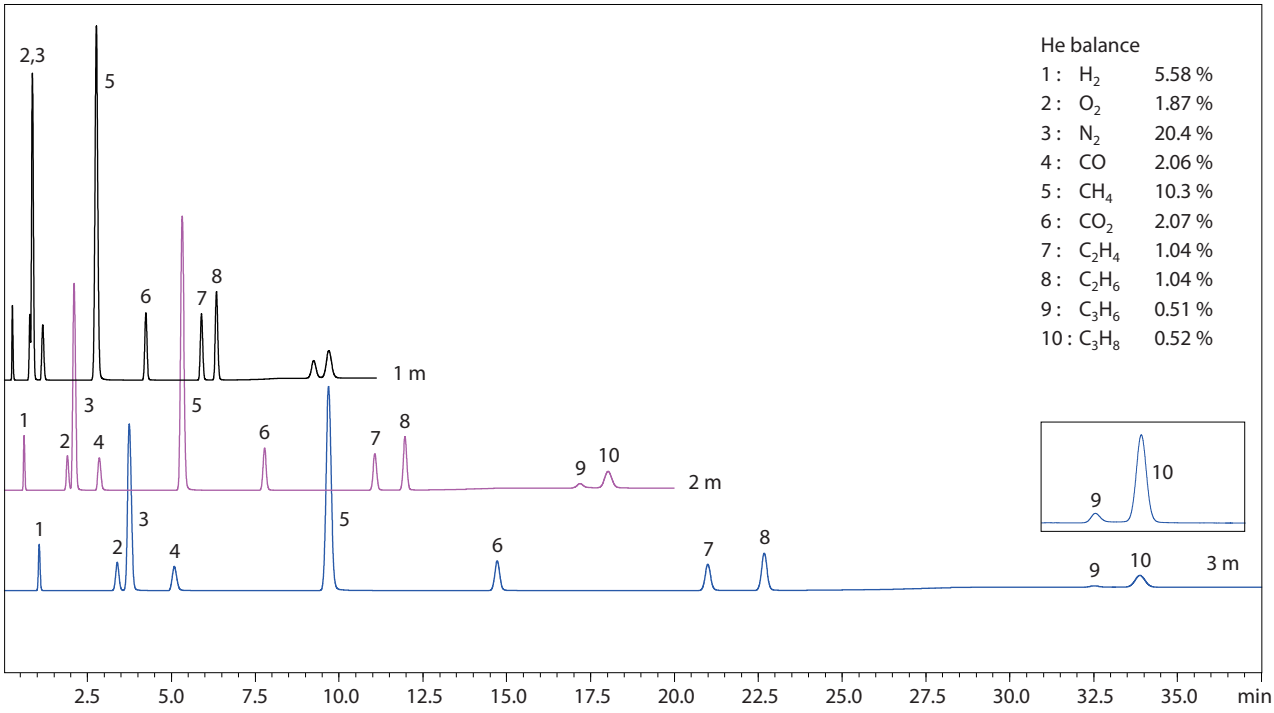

Fig. 3 Comparison of Chromatograms of High-concentration Standard Gas of Inorganic Gases and Small Hydrocarbons

First Edition: Nov. 2017

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