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Application News

No. G323

Gas Chromatography

Packed Column Analysis of Gases Using Nexis™ GC-2030 (TCD)

The Nexis GC-2030 gas chromatograph has begun to support packed columns. The detectors compatible with packed columns are a flame ionization detector (FID) and a thermal conductivity detector (TCD).

As an example of SUS packed column TCD analysis using the Nexis GC-2030, this article introduces gas analysis using a Molecular Sieve 5A (MS-5A) column.

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Packed Column Support System

An SUS column which can be used for the Nexis GC-2030, a system compatible with packed columns, is the same as that used for the GC-14, GC-17 or GC-2014, and it can be shared between these instruments.

For the TCD for packed columns (PTCD-2030), a column is connected to the sample vaporization chamber (SINJ) and the carrier gas flow rate is controlled by the Advanced Flow Controller (AFC).

Fig. 1 shows the appearance of the instrument equipped with the PTCD-2030 and an example of connecting an SUS column and an empty pipe to the analysis line and reference line, respectively. Also, it is possible to eliminate the effect of baseline drift identified in a column temperature program analysis by connecting the same type of column as that for the analysis line on the reference side.

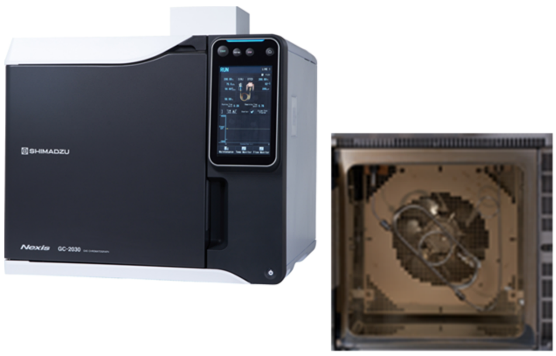


Fig. 1 Appearance of Instrument with PTCD-2030 and Example of SUS Column Installation

In gas analysis using a TCD, as the mount of sample injected increases, this may cause greater baseline drift associated with sample injection, and the peak of the compound whose retention time is short is affected by the drifting baseline. To reduce baseline drift, an effective countermeasure is to change the carrier gas control from flow rate control to pressure control, as is the case with analysis using the GC-2014. In this situation, we recommend to install an advanced pressure controller (AUX APC) for pressure control.

Gas Analysis

Gas samples were analyzed using an SUS packed column (MS-5A). As samples to be analyzed, approximately 1000 ppm (v/v) each of H_2, CH_4, CO and C_2H_6 were prepared using He as the base gas. The samples were prepared by repeatedly taking a certain volume of the target ingredient using a gas-tight syringe and adding it to a bag filled with He, which served as the blank. In the analysis, 1 mL of each gas sample was aspirated by a gas-tight syringe and injected manually. Since ambient gas was mixed into the bag or syringe (needle) during these operations, N_2 or O_2, which was not added intentionally, was also analyzed.

Analysis Conditions

Table 1 lists the configuration of the instrument used for analysis and the analysis conditions.

Table 1 Instrument Configuration and Analysis Conditions

Model	: Nexis GC-2030APT
Injection Mode	: Direct
Injection Volume	: 1 mL (gas-tight syringe)
Injection Temp.	: 120 °C
Carrier Gas	: He
Carrier Gas Control	: 40 mL/min
Column	: MS-5A 60/80 (2 m × 3 mm I.D.) * Reference line measurement was performed via a directly connected single column
Detector	: Thermal Conductivity Detector (TCD)
Detector Temp.	: 220 °C
Current	: 100 mA
Column Oven Temp.	: 60 °C (5min) - 10 °C/min – 200 °C (6min)

Results of Gas Analysis

The chromatogram of gas analysis is shown in Fig. 2.

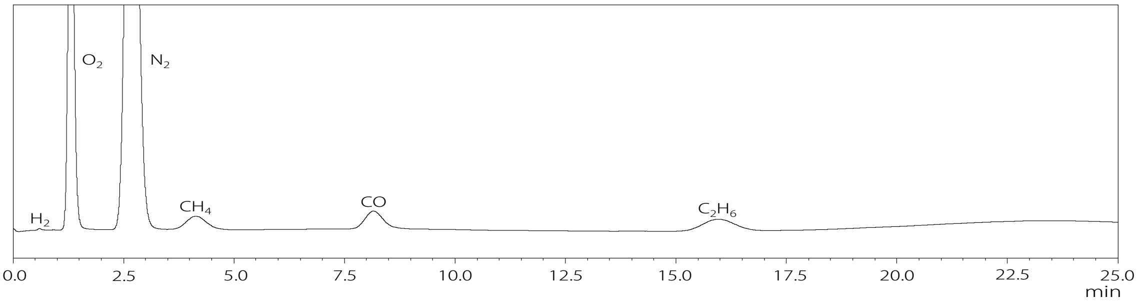


Fig. 2 Chromatogram of Gas Analysis

Baseline Drifts Associated with Sample Injection

Fig. 3 shows the baseline drifts caused by injection of 0.5 mL, 1 mL and 2 mL of the sample gas while controlling the flow rate of the carrier gas. As shown in the figure, as more sample is injected, this is associated with a lower baseline, and these baseline drifts are still detected at 0.6 minutes when the H_2 peak is eluted. As a comparison, Fig. 4 shows the baseline drifts while the pressure of the carrier gas was controlled using an APC. When the pressure of the carrier gas is controlled, the result is baseline drifts associated with sample injection, but the baselines return to the original level at approximately 0.4 minutes after 2 mL of sample is injected, indicating that the baselines return to their original level earlier than when the flow rate of carrier gas is controlled.

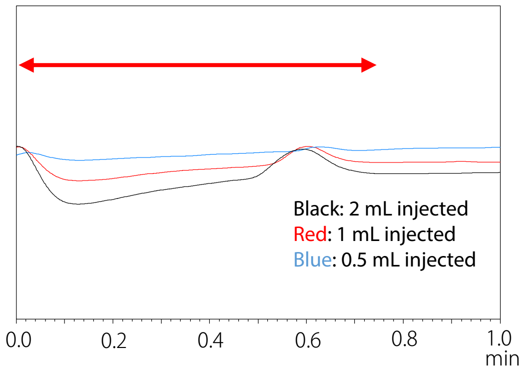


Fig. 3 Baseline Drifts at Sample Injection (Flow Control)

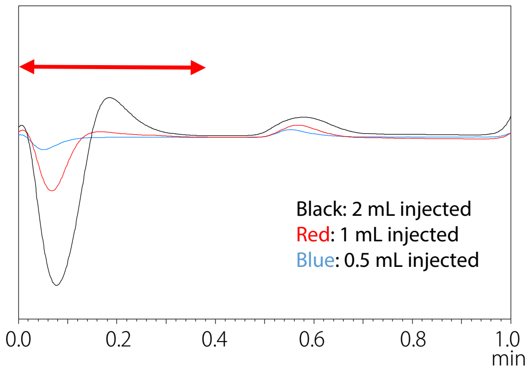


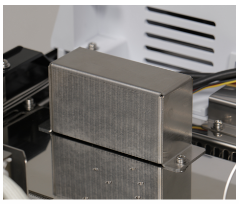
Fig. 4 Baseline Drifts at He Gas Injection (Pressure Control)

Conclusion

With an SUS packed column connected to a TCD for packed columns of the Nexis GC-2030, gas analysis was performed. The peaks of compounds were favorably separated. Additionally, the analysis results reconfirmed that pressure control using an APC is an effective technique for gas analysis when a large quantity of sample injected.

Optional Information on Nexis GC-2030

Nexis GC-2030 system makes a wide variety of useful options available.



<Hydrogen sensor>

For customers who are considering the use of H_2 as carrier gas in a situation where it is difficult to obtain helium, installation of a hydrogen sensor is recommended to ensure the safe operation of the instrument.

This sensor monitors the hydrogen concentration in the gas chromatograph oven and promptly detects potential leakages. When a leakage is detected, it stops the temperature control operation and automatically switches the mode to the safe standby mode. If the hydrogen concentration increases further to exceed 2%, the main power to the gas chromatograph is cut to prevent accidents.

First Edition: Jun. 2020

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Gas Chromatograph

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