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Application News

GC Nexis^{TM} GC-2030 FluxEdge^{TM}

High-Speed Gas Analysis Using the FluxEdge GC System

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User Benefits

◆ Simultaneous analysis of inorganic and organic gas components within a measurement cycle of 4 minutes.
◆ Achieves low carry over and high analytical repeatability with a small amount of sample.
◆ The backflush function that protects the column and the high-durability valve system provide long-term stability for the system.

■ Introduction

In recent years, there has been an urgent need to address climate change and to realize a decarbonized society, and research and development of various technologies have been promoted. Among these, technologies to generate renewable energy using catalytic reactions are attracting attention.

In the research and development of catalytic reactions, it is necessary to continuously grasp the changes in the composition of components before and after the reaction, so measurement in a short time is required. In addition, even when the gas produced after the reaction is limited to a small amount, it is necessary to be able to measure with high accuracy.

Taking methane generation technology as an example, here we introduce a system for simultaneous analysis of hydrogen (H_2), oxygen (O_2), nitrogen (N_2), carbon monoxide (CO), carbon dioxide (CO_2), methane (CH_4), and ethylene (C_2H_4), which are typical measurement targets in the research and development of such technologies.

■ Description of the system

Fig. 1 shows the appearance of the device. This system is an analyzer that combines the gas chromatograph Nexis GC-2030 with the latest technology, FluxEdge, achieving high-speed analysis and high analytical repeatability. The FluxEdge incorporates a microvalve that combines semiconductor manufacturing microfabrication technology with Shimadzu's cutting-edge passivation technology. This valve has very high durability and is essentially maintenance-free. The system is designed so that the entire analytical flow path, including this microvalve, is minimized, allowing for the use of industry-standard capillary columns. As a result, peaks are sharper, enabling high-speed analysis with trace samples.

Fig. 2 presents a schematic diagram illustrating the structure and mechanism of the microvalve. The microvalve is a pneumatic diaphragm valve. Inside the valve, there are two spaces separated by a diaphragm, with the lower space containing the analytical flow path. When pneumatic gas is applied to the upper space, the pressure deforms the diaphragm, blocking the analytical flow path in the lower space. When pneumatic gas is not applied, the diaphragm returns to a flat state, allowing the analytical flow path to open. The diaphragm is made of single crystal silicon, a material used in microstructures such as MEMS, and the optimized valve operation mechanism provides several hundred times better durability compared to conventional rotary valves.

Fig. 3 shows an overview of the FluxEdge main module. The FluxEdge main module, which plays a central role in the analysis conducted using this system. It is equipped with six microvalves and a sample loop, allowing efficient processing from pretreatment to analysis by switching each microvalve.

Fig. 1 Appearance of the FluxEdge™ GC System

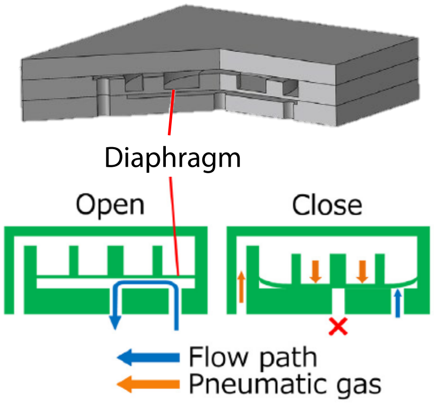

Fig. 2 Structure and Mechanism of the Microvalve

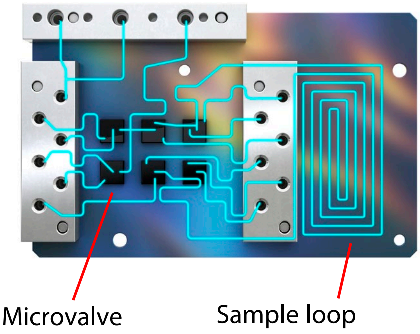

Fig. 3 Overview of the FluxEdge Main Module

Fig. 4 shows the flow path configuration. This system has three analytical lines (series) and automatically performs sampling of the sample gas, gas injection into the three series, and backflushing. Backflushing is a function that discharges components that are not the target of measurement outside the system. Each series is equipped with the FluxEdge main module and TCD, and by connecting the optimal separation column for each target component to each TCD, high-speed simultaneous analysis is made possible. The target components for each TCD are listed in Table 1.

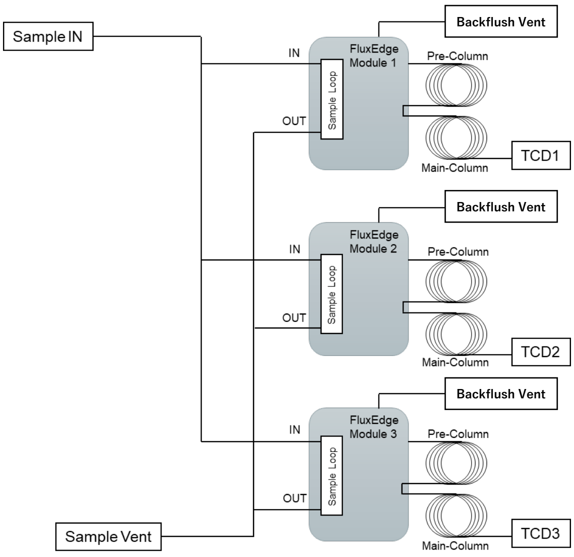

Fig. 4 Flow Path Configuration of the System

Table 1 Types of Carrier Gases and Target Components for Each Series

Detector	Target	Carrier Gas
TCD1	CO_2, C_2H_4	He
TCD2	O_2 , N_2, CH_4, CO	He
TCD3	H_2	N_2

■ Analysis Flow

The analysis proceeds mainly through the following four phases. Supplementary information with flow diagrams for each phase is included on the last page.

(1) Sampling

The sample gas is filled in the sample loop of the FluxEdge.

(2) Sample Injection

The sample gas is introduced from the sample loop into the pre-column.

(3) Backflushing

Backflushing starts when the target component to be measured passes through the pre-column. Components that are not the target of measurement are discharged through the backflush vent. Simultaneously, the target component transitions to the main column for separation.

Backflushing not only serves to discharge components with relatively long elution times, thereby shortening the analysis time, but it is also a highly effective method for column protection. For example, if CO_2 or moisture contaminates a molecular sieve column commonly used for separating O_2, N_2, and CO, its retention capacity significantly decreases, leading to poor analytical repeatability. However, by backflushing specific components out of the system, stable results can be achieved.

An example of backflushing CO_2 in the series of molecular sieve columns is presented in this paper.

(4) Detection

The target components separated by each column are detected by their respective TCDs.

■ Standard Gas Analysis Conditions

Table 2 presents the analysis conditions, and Table 3 shows the measured total flow rates of the gases used in the analysis. This system includes conditioned columns and adjusted methods, utilizing standard capillary columns. Table 4 indicates the concentrations of the standard gases used in the analysis and the concentration ranges that can be simultaneously analyzed by this system. The system is compatible with both gas bags using tubing and cylinders using metal piping, allowing for automatic sampling of the connected gas samples into the device. For this analysis here, the standard gas cylinder was connected to the system after being reduced to 100 kPa.

Table 2 Analysis Conditions *

Series (Detector)		1 (TCD1)	2 (TCD2)	3 (TCD3)
Sampling Time		15 sec		
Carrier Gas		He	He	N_2
Pre-Column		SH-Q-BOND PLOT 15m	SH-Q-BOND PLOT 15m	SH-Q-BOND PLOT 15m
Main Column		SH-Q-BOND PLOT 15m	SH-MS5A PLOT 5m	SH-MS5A PLOT 10m
Oven Temp.		40 °C		
TCD	Temp.	80 °C	80 °C	80 °C
	Current	90 mA	90 mA	40 mA
	Polarity	+	+	-
	Flow Rate	8 ml/min	8 ml/min	4 ml/min

Table 3 Measured Gas Flow Rate (ml/min) *

Gas Type	Phase	Series 1	Series 2	Series 3
Sample	Sampling	34		
Carrier Gas	Injection	48	32	33
Carrier Gas	Backflushing	52	34	36

* The analysis conditions not listed in Table 2 (such as sample injection and carrier gas control) and the flow rates in Table 3 may vary depending on the system configuration, such as the columns used and the pressure of the samples.

Table 4 Concentration of Target Components

Measured Components	Composition of Standard Gas	Concentration Range for Simultaneous Measurement
H_2	Balance	0.1~100%
O_2	1.02%	0.01~1%
N_2	1.01%	0.01~1%
CH_4	20.3%	0.01~20%
CO	0.051%	0.01~10%
CO_2	10.0%	0.1~10%
C_2H_4	0.049%	0.01~100%

■ Measurement Results of Standard Gas

Fig. 5 shows the results of the analysis of standard gases using this system. All target components were detected within 3 minutes. Additionally, the results of continuous analysis in the following section indicated that the measurement cycle, including the preprocessing time, was within 4 minutes. Repeated analysis was conducted five times using the same standard sample to confirm the repeatability of the area values and retention times. As shown in Table 5, good repeatability was obtained for the area values of all target components. The repeatability of retention times was less than 0.08% RSD, indicating that the chromatogram shown in Fig. 5 was obtained stably. Furthermore, based on the sampling times and flow rates from Tables 2 and 3 in the previous section, the total sample consumption per analysis was calculated to be 8.5 ml. This shows that the sample consumption of this system is less than one-tenth of conventional systems.

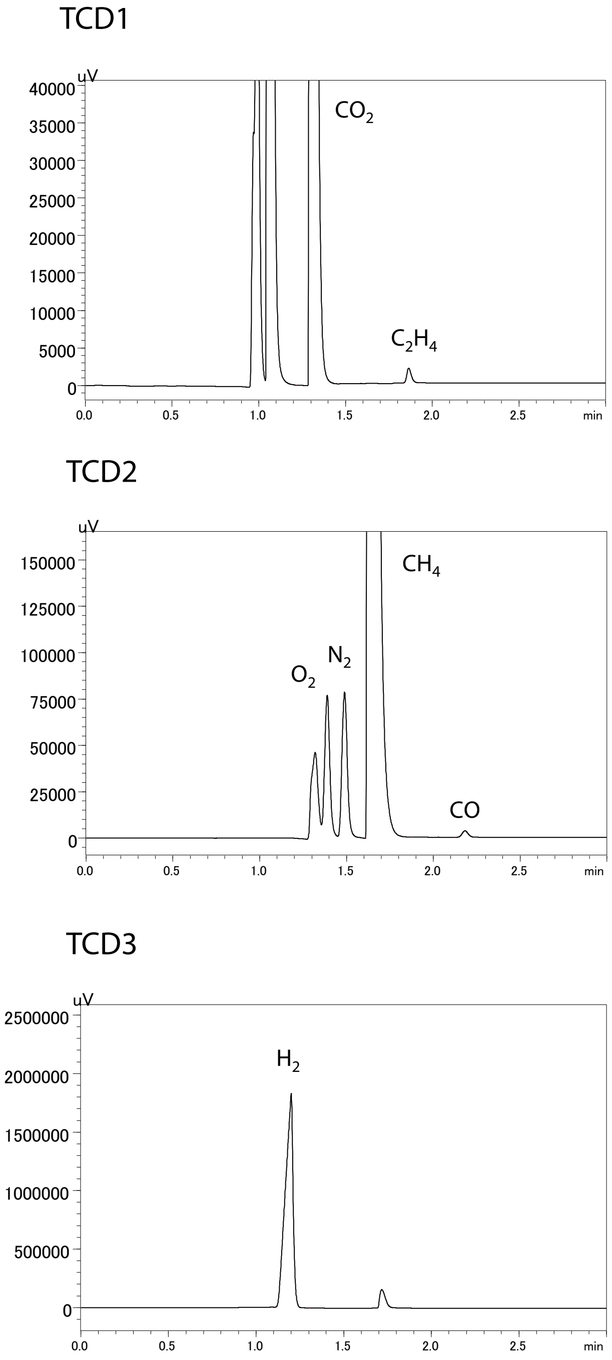

Fig. 5 Chromatogram of Mixed Standard Gas

Table 5 Analytical Repeatability (% RSD)

Series	1		2				3
Compound	CO_2	C_2H_4	O_2	N_2	CH_4	CO	H_2
Area %RSD	0.064	0.48	0.48	0.35	0.32	0.43	0.060
Rt %RSD	0.0013	0.0013	0.072	0.074	0.079	0.076	0.0026

■ Evaluation of Carry Over

The phenomenon in which components of a sample used in a previous analysis remain and affect the next analysis is referred to as carry over. It is particularly problematic when continuously analyzing samples with changing component compositions, so it is essential to have appropriate cleaning or sampling methods using suitable carrier gases or samples when analyzing gas samples. Here, we evaluated the degree of carry over in this system and the relationship between sampling time and carry over. Using Series 2 of this system, we analyzed pure CH_4 depressurized to 100 kPa under the conditions in Table 2, followed by the analysis of He (G1 grade) also depressurized to 100 kPa under the same conditions. Next, carry over was calculated from the area ratio of the obtained CH_4. Then, the sampling time was changed to 20 seconds from the conditions in Table 2, and similar analyses and calculations were performed. The results are presented in Table 6.

In the conditions of Table 2, with a sampling time of 15 seconds for standard gas analysis, carry over was confirmed to be less than 0.005%. This very low carry over indicates that sufficient sample cleaning inside the flow path can be achieved with a small amount of sample during the short preprocessing time. When the sampling time was changed to 20 seconds, even lower carry over results were observed. This result shows that extending the sampling time is an effective method to reduce carry over. This method is considered particularly effective for analyzing compressed gas samples at relatively low pressures or samples at atmospheric pressure, which tend to have increased carry over due to relatively low sampling flow rates.

Table 6 Evaluation Results of Carry Over

Sampling Time	Area value of CH_4		Carry Over
	Pure CH_4 Injection	He (G1 Grade) Injection	
15 sec	10,000,000	440	0.0044%
20 sec	10,000,000	290	0.0029%

■ Conclusion

The FluxEdge system detected peaks of H2, O2, N2, CO, CO2, CH4, and C2H4 within 3 minutes. The measurement cycle, including preprocessing time, was found to be under 4 minutes, demonstrating the capability for fast simultaneous analysis.

Good analytical repeatability was obtained for all target components mentioned above. Furthermore, the total sample consumption per cycle was measured to be 8.5 ml, which is less than one-tenth of conventional systems. Additionally, for the analysis of pure CH4 at 100 kPa, a low carry over of less than 0.005% was realized with a short sampling time of 15 seconds. These results indicate that sufficient sample cleaning and good analytical repeatability can be achieved with a small amount of sample.

The results showing good analytical repeatability with the molecular sieve column series indicated that the backflush function prevents the intrusion of components that degrade the retention capability of the column, thus protecting the column. Furthermore, this system employs microvalves with durability hundreds of times higher than conventional ones, contributing to the long-term stability of the entire system.

■ Supplementary Information

- Flow Diagram of the System -

Fig. 6 to 9 show the flow diagrams of this system during each phase from sampling to detection. The rectangles surrounded by dashed lines in the figures represent the FluxEdge main module, while V1 to V6 denote the microvalves installed in the same module.

(1) Sampling

Samples are filled into the sample loop. At this time, the pump located downstream of the sample loop is activated.

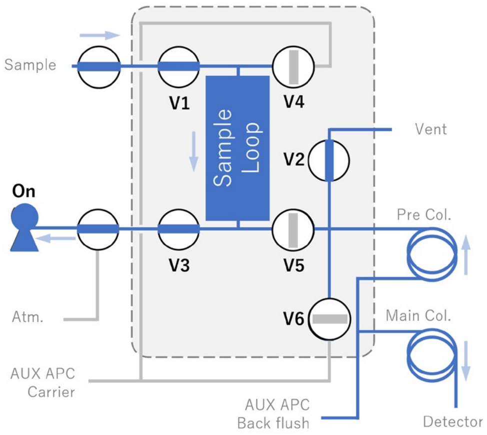

Fig. 6 Flow Diagram During Sampling

To achieve good analytical repeatability, the pressure inside the sample loop after sampling needs to remain constant. In this system, the pressure is stabilized by maintaining the state of the next flow path for a specified time after sampling, ensuring equilibrium with atmospheric pressure.

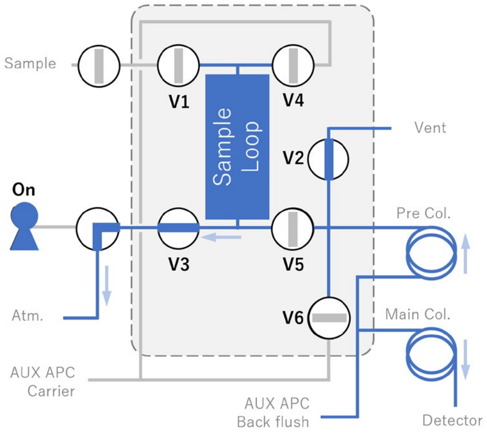

Fig. 7 Flow Diagram During Atmospheric Equilibrium *

* This system has two modes for atmospheric equilibrium.
Fig. 7 shows the flow path during atmospheric equilibrium for the analysis described in this document.

(2) Sample Injection

After opening V4 and V5 and injecting the sample for a specified time, V4 and V5 are closed, and V6 is opened to separate components in the injected sample using the pre-column.

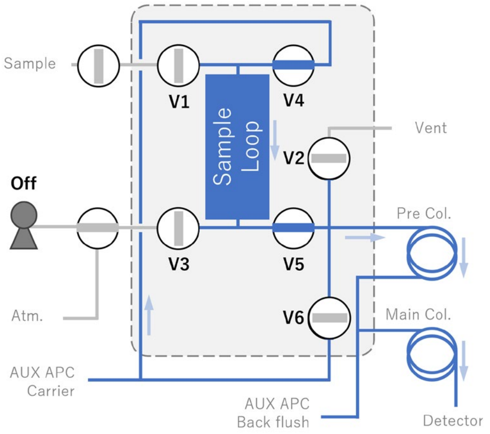

Fig. 8-1 Flow Diagram During Sample Injection

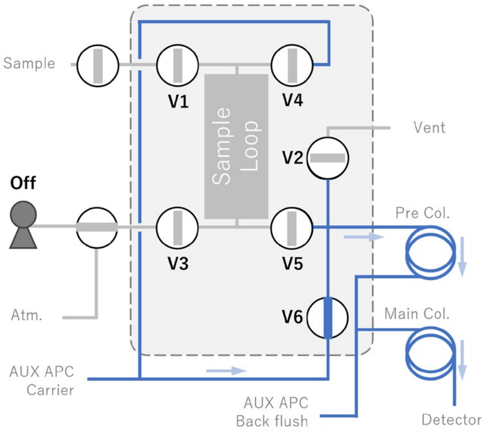

Fig. 8-2 Flow Diagram During Separation by Pre-column

(3) Backflush, (4) Detection

During backflushing of non-target components, the target components are separated and detected in the main column.

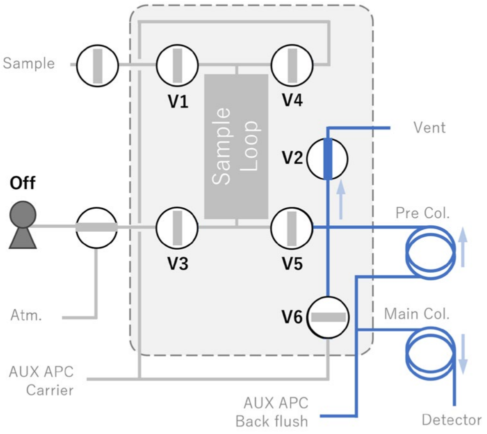

Fig. 9 Flow Diagram During Backflush and Detection

<Related Papers>

1) S. Shibamoto, W. Lu, A. Sato, A multifunction flow channel switching module for multi-column capillary gas chromatography using silicon pneumatic microvalves, Journal of Chromatography A 1696 (2023) 463961, doi.org/10.1016/j.chroma.2023.463961

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