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Application News

Gas Chromatograph Nexis™ GC-2030

Simultaneous Analysis of Greenhouse Gases Using Nitrogen Carrier Gas

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User Benefits

- The main greenhouse gases CO_2, CH_4, and N_2O can be simultaneously analyzed.
- The Jetanizer™ can detect CO_2 with a flame ionization detector and perform analysis with a simple equipment configuration.
- Reasonable and accessible N_2 can be used as the carrier gas.

■ Introduction

There is a global desire to combat global warming by reducing levels of greenhouse gases. Hence, many fields of industry and science are pursuing research and development aimed at achieving carbon neutrality, a state of net zero greenhouse gas emissions.

The main greenhouse gases are carbon dioxide (CO_2), methane (CH_4), and dinitrogen monoxide (N_2O). Because CO_2, CH_4, and N_2O are present at very different concentrations in the atmosphere, a thermal conductivity detector (TCD), a flame ionization detector (FID), and an electron capture detector (ECD) are needed to measure levels of all three greenhouse gases in the atmosphere simultaneously. The Jetanizer™ can detect CO_2 with an FID, thus enabling the simultaneous analysis of greenhouse gases with a simpler equipment configuration. More information on the performance of the Jetanizer can be found in Application News 01-00599. This article describes using a Jetanizer-FID and ECD to perform a simultaneous analysis of CO_2, CH_4, and N_2O.

■ Equipment Configuration and Analytical Conditions

First, the FID nozzle was replaced with a Jetanizer. The capillary adapter on the lower part of the injection port was replaced with a two-way branched capillary adapter,*1 and two columns were connected to the adapter. The insertion lengths of the columns were 51 and 61 mm. Gas samples were introduced using the MGS-2030 gas sampler, and samples were divided and introduced to both columns through the lower part of the injection port and detected by the Jetanizer-FID and the ECD. A plumbing diagram of the system is shown in Fig. 1, and the analytical conditions are shown in Table 1.

*1 Requires an INJ2-way branch unit (S221-75231-41).
The unit consists of a two-way branched adapter (a multi-column hanger and two INJ nuts).

Table 1 Analytical Conditions

Model:	Nexis GC-2030
Gas Sampler:	MGS-2030 + 1 mL Loop
Inj. Temp.:	100 °C
Inj. Mode:	Split
Split Ratio:	1:9
Carrier Gas:	N_2, fixed column flowrate mode (5 mL/min)
Purge Flow:	0 mL/min
Column:	MICROPACKED-ST 2 m × 1 mm I.D. 2 columns (250 m × 0.50 mm I.D. , df = 10 µm entered to calculate flowrates) (Column insertion lengths on injection port side: 51 mm, 61 mm)
Column Temp.:	50 °C (2 min) → 40 °C/min → 200 °C → 25 °C/min → 250 °C → 15 °C/min → 275 °C (5 min)
Detector:	FID＋Jetanizer*2 (Column insertion length: 45 mm) ECD-2010 Exceed*3 (Column insertion length: 32 mm)
ECD Temp.:	340 °C
ECD Gas:	Ar＋5 %CH_4
ECD Gas Prog.*4:	30 mL/min (0 min) → 400 mL/min^2 → 200 mL/min (2 min) → -400 mL/min^2→ 30 mL/min (11.57 min)
ECD Current:	1 nA
FID Temp.:	400 °C
Makeup Gas:	N_2, 20 mL/min
H_2 Flow:	32 mL/min
Air Flow:	250 mL/min

*2 A normal FID can only be used when analyzing just CH_4 and not CO_2. The parameters for the column insertion length, detector temperature, and gas flowrate for the FID should be optimal.

*3 Flow lines changed using ECD-2010 Exceed contamination prevention kit NX (S221-89320-41). The flow lines were designed for durability and linearity. The column insertion length, ECD gas flowrate, and electric current differed from those of normal flow lines.

*4 The flowrate program was configured to increase the ECD gas flowrate when eluting O_2 and then return to the normal flowrate.

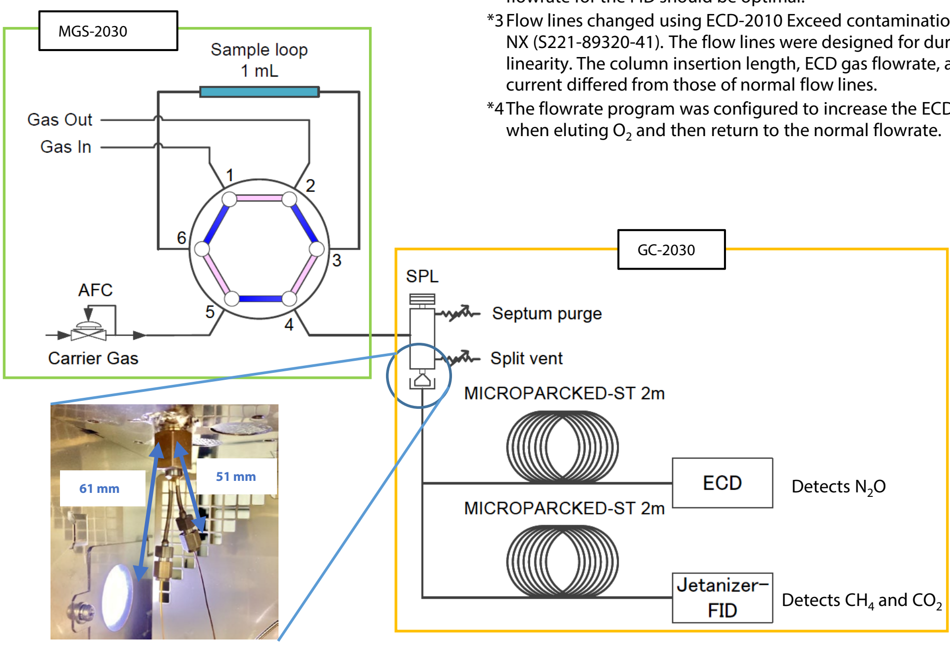

Fig. 1 Plumbing Diagram of System

■ Confirming Linearity

Calibration curves were prepared by diluting CH_4, CO_2, and N_2O with N_2. The calibration curve ranges were 1-100 ppm for CH_4, 10-1000 ppm for CO_2, and 0.05-2.5 for N_2O. The calibration curves for each compound are shown in Figs. 2 and 3. Good linearity was obtained with all three compounds.

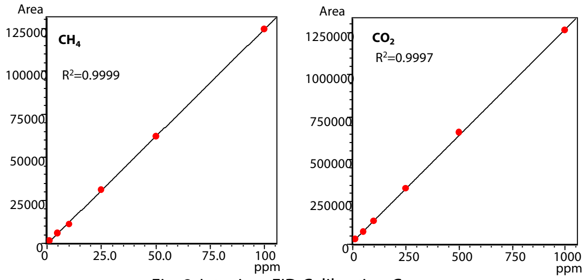

Fig. 2 Jetanizer-FID Calibration Curves

CH_4: 1, 5, 10, 25, 50, 100 ppm CO_2: 10, 50, 100, 250, 500, 1000 ppm

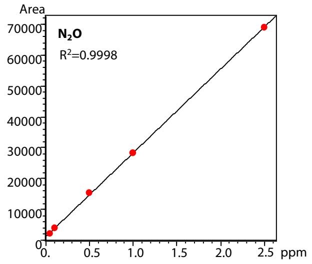

Fig. 3 ECD Calibration Curve

N_2O: 0.05, 0.1, 0.5, 1, 2.5 ppm

■ Quantitative Analysis of Atmosphere and Repeatability

Samples of atmosphere were analyzed five times consecutively. The resulting chromatograms are shown in Fig. 4, and the levels in the atmosphere, which were determined using the calibration curves and the peak area repeatability, are shown in Table 2. The peak area repeatability was good for all three compounds.

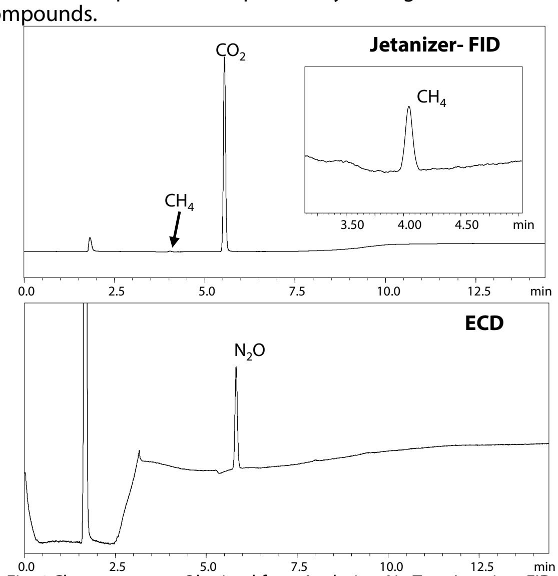

Fig. 4 Chromatograms Obtained from Analyzing Air: Top: Jetanizer-FID, Bottom: ECD

Table 2 Amount of Each Compound Measured in Air (ppm) and Peak Area Repeatability (%RSD, n = 5)

	CH_4	CO_2	N_2O
Quantified amount (ppm)	2.2	413	0.36
Peak area repeatability (%RSD)	1.1	0.38	0.55

■ Assessing Long-Term Stability of ECD

Measuring N_2O with an ECD normally requires using Ar+5 % CH_4 as the ECD gas and a pre-cut of O_2. Since this analytical method introduces O_2 to the ECD, the stability of the ECD during the extended introduction of air was assessed to compare with using Ar+5 % CH_4 and N_2 as the ECD gas. An HS-20 headspace sampler was used for consecutive analysis, air was used for vial pressurization,*5 and analysis was performed on atmosphere sealed in vials. The area value reproducibility and sensitivity variation were evaluated by continuously analyzing N_2O in the atmosphere at every 100 mL air introduction volume. Table 3 shows N_2O peak area repeatability of measurements taken every 100 mL of introduced air. Good repeatability with all %RSD results below 0.5 was obtained using Ar+5 % CH_4 as the ECD gas, while almost all %RSD results were above 5 % when N_2 was used as the ECD gas. Fig. 5 shows the change in peak area ratio with the peak area measured by the first analysis as 1. There was almost no variation in sensitivity and measurements were stable when Ar+5 % CH_4 was used as the ECD gas. However, the peak area decreased after introducing 100 mL of air when N_2 was used as the ECD gas. These results show that Ar+5 % CH_4 must be used as the ECD gas to obtain stable measurements with good repeatability.

*5 He or N_2 is normally used as the vial pressurization gas.

Table 3 N_2O Peak Area Repeatability (%RSD) Every 100 mL of Introduced Air

Total Air Introduced (mL)	0	100	200	300	400	500	600	700	800	900	1000
Ar+5 %CH_4 (%RSD)	0.40	0.25	0.29	0.20	0.42	0.45	0.46	0.41	0.40	0.40	0.43
N_2 (%RSD)	2.29	5.29	6.42	5.15	7.38	6.10	5.48	6.38	5.13	6.25	4.96

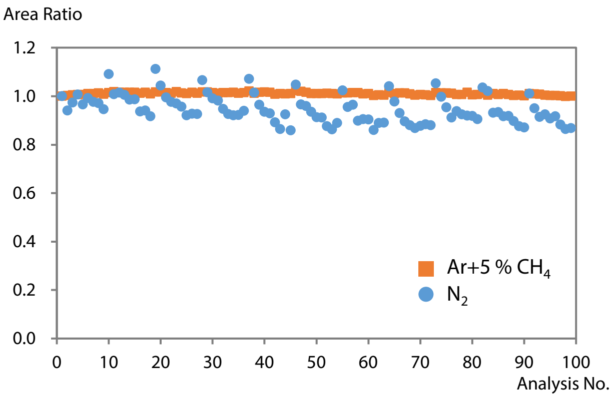

Fig. 5 Change in Peak Area Ratio Taking First Peak Area as 1

■ Conclusion

A Jetanizer-FID and ECD were successfully used to perform a simultaneous analysis of the main greenhouse gases CO_2, CH_4, and N_2O.

Analyzing standard gases confirmed good linearity, which enabled the quantitative analysis of atmospheric CO_2, CH_4, and N_2O. Furthermore, the long-term stability of the ECD was also confirmed.

The analytical method described in this article allows greenhouse gases to be measured using a simple equipment configuration that does not require helium gas.

< Related Application News Articles >

1. Assessment of Jetanizer™ and Quantitative Analysis of CO_2 and CH_4 in the Atmosphere: Application News No. 01-00599-EN

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