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Application News

No. G300

Gas Chromatography

Analysis of SF_6 Insulation Gas Using a GC-BID System

Sulfur hexafluoride (SF_6) is an extremely stable gas with excellent dielectric properties and is used in various fields. Applications include tracer gas as well as insulation gas used in electrical equipment such as gas-insulated transformers and circuit breakers. On the other hand, SF_6 is also known to be a highly potent greenhouse gas and was identified as an emission reduction target in the Kyoto Protocol at the COP3 meeting. Conventional analysis of SF_6 employs an electron capture detector (ECD) which is capable of detecting electrophilic compounds with high sensitivity. However, the quantitation accuracy in the high-concentration range and the hassle of procedures necessary due to the use of radioisotopes have been an issue. Shimadzu's barrier discharge ionization detector (BID) offers high-sensitivity detection of almost all components. Utilizing proprietary barrier discharge technology, the detector achieves both the same stability as general conventional detectors and high sensitivity.

This article introduces example analyses of SF_6 and SF_6 decomposition products.

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Analysis of Impurities in SF_6

SF_6 is used as an insulation gas in various electrical equipment and requires purity analysis for quality control to maintain insulation properties and when recycling the gas. Fig. 1 shows the result of analyzing impurities in SF_6 using a BID. The primary component SF_6 is saturated, but did not influence the quantitation accuracy of surrounding components, showing that high-sensitivity batch analysis of impurities including inorganic gas and lower hydrocarbons was successful. (Under the analytical conditions using this type of column, C_2H_6 is overlapped by SF_6.)

The concentrations of impurities found in the SF_6 sample are as follows.

H_2 :	0.9 ppm	CO :	0.9 ppm	CH_4 :	1.7 ppm
CO_2 :	21 ppm	N_2O :	2.0 ppm	C_2H_2 :	2.4 ppm
C_2H_4 :	1.4 ppm	C_3H_6 :	1.0 ppm	C_3H_8 :	1.0 ppm

Table 1 Measurement Conditions

Model	: Nexis™ GC-2030
Detector	: BID-2030
Inj. Mode	: Split 1:4
Inj.Temp.	: 150 °C
Carrier Gas	: 7 mL/min (constant flow rate)
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)
Column Temp.	: 35 °C (2.5 min) – 20 °C/min – 250 °C (0 min) – 15 °C/min – 265 °C (3.0 min)
Purge flow	: 3 mL/min
Det. Temp.	: 280 °C
Discharge Gas	: 50 mL/min (He)
Inj. Volume	: 3.0 mL (MGS-2030)

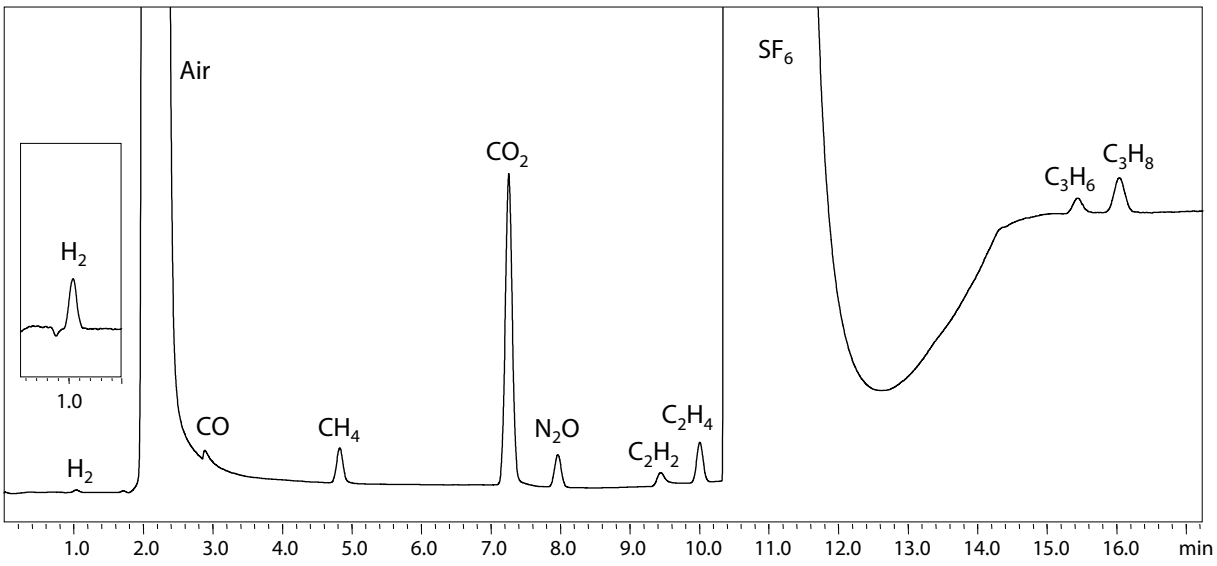

Fig. 1 Analysis of Impurities in SF_6

Analysis of Trace SF_6 in the Atmosphere

Since SF_6 is a potent greenhouse gas and its emission into the atmosphere must be avoided, leak tests for insulation gas from electrical equipment and residue tests after collecting gas may require analysis of trace amounts of SF_6. Table 2 lists the measurement conditions and Fig. 2 shows the result of analyzing trace SF_6 in the atmosphere.

SF_6 at a concentration of 0.1 ppm was detected (S/N = 24[*1]) and favorable linearity was obtained in the range from 0.1 to 50 ppm (R^2 = 0.9998).

*1 Determined by calculating noise from the baseline between 0.5 and 1.5 min.

Table 2 Measurement Conditions

Model	: Nexis™ GC-2030
Detector	: BID-2030
Inj. Mode	: Split 1:7
Inj.Temp.	: 150 °C
Carrier Gas	: 45 cm/sec (constant linear velocity)
Column	: SH-Rt™-Msieve 5A (0.53 mm I.D.×30 m, d.f.50 µm)
Column Temp.	: 35 °C (2.5 min) – 20 °C/min – 250 °C (0 min) – 15 °C/min – 270 °C (3.42 min)
Purge flow	: 3 mL/min
Det. Temp.	: 280 ˚C
Discharge Gas	: 50 mL/min (He)
Inj. Volume	: 3.0 mL (MGS-2030)

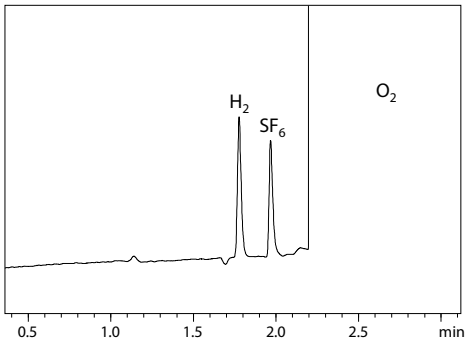

Fig. 2 Analysis of Trace SF_6 (0.1 ppm) in the Atmosphere

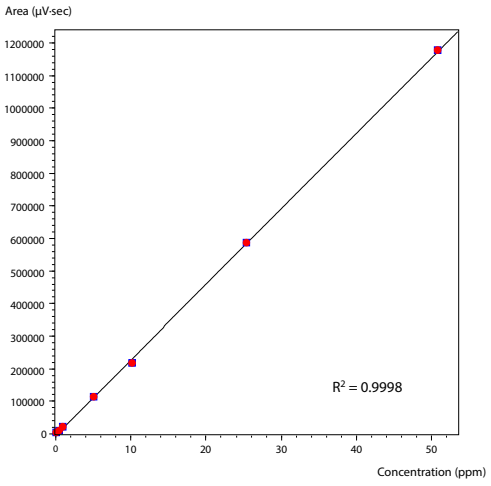

Fig. 3 Linearity of SF_6 in the Atmosphere (0.1 to 50 ppm)

Analysis of SF_6 Decomposition Gas

SF_6 is used as an insulation gas in circuit breakers. Breakers are installed on electrical grids to shut off high voltages that may occur due to causes such as lightning strikes. When performing maintenance on such breakers, SF_6 decomposition gases are analyzed to judge the degradation level of insulation gas. Table 3 lists the measurement conditions and Fig. 4 shows the results of an example analysis of CF_4 and SOF_2 which are SF_6 decomposition gases.

Sample injection using a gas sampler and a gas-tight syringe is possible on the same system, allowing analysis of gas samples in various forms and concentrations.

Table 3 Measurement Conditions

Model	: Nexis™ GC-2030
Detector	: BID-2030
Inj. Mode	: Split 1:4
Inj.Temp.	: 150 °C
Carrier Gas	: 7 mL/min (constant flow rate)
Column	: MICROPACKED-ST 1.0 m × 1.0 mm I.D. (Input 1,250 m × 0.50 mm I.D. and df = 15 µm for flow rate calculation)
Column Temp.	: 50 °C (1.0 min)– 25 °C/min – 150 °C (0 min) – 5 °C/min – 200 °C (0 min)
Purge flow	: 3 mL/min
Det. Temp.	: 280 ˚C
Discharge Gas	: 50 mL/min (He)
Inj. Volume	: 200 µL (gas-tight syringe)

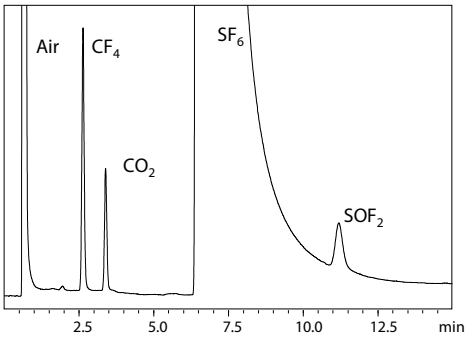

Fig. 4 Analysis of SF_6 Decomposition Gases (CF_4: 310 ppm, SOF_2: 107 ppm)

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