

# Application News

## No. G300

### Gas Chromatography

## Analysis of SF<sub>6</sub> Insulation Gas Using a GC-BID System

Sulfur hexafluoride (SF<sub>6</sub>) 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<sub>6</sub> 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<sub>6</sub> 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<sub>6</sub> and SF<sub>6</sub> decomposition products.

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### ■ Analysis of Impurities in SF<sub>6</sub>

SF<sub>6</sub> 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<sub>6</sub>

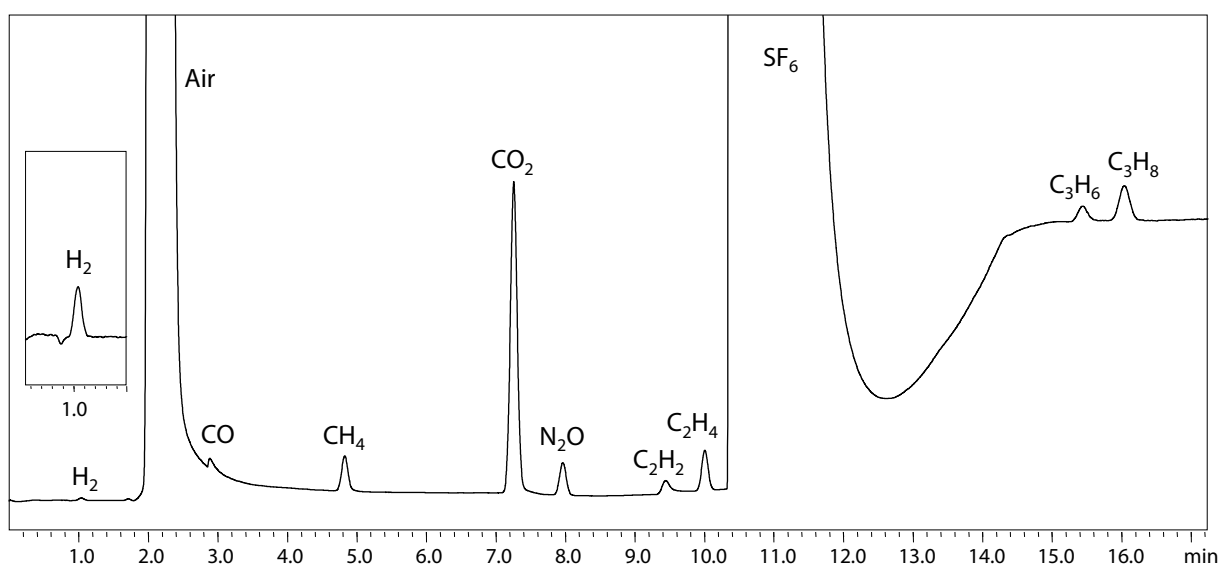
using a BID. The primary component SF<sub>6</sub> is saturated, but did not influence the quantitation accuracy of surrounding components, showing that high-sensitivity batch analysis of impurities including inorganic gas and lower hydrocarbons was successful. (Under the analytical conditions using this type of column, C<sub>2</sub>H<sub>6</sub> is overlapped by SF<sub>6</sub>.)

The concentrations of impurities found in the SF<sub>6</sub> sample are as follows.

|   |   |   |
|---|---|---|
| H <sub>2</sub> : 0.9 ppm                | CO: 0.9 ppm                             | CH <sub>4</sub> : 1.7 ppm               |
| CO <sub>2</sub> : 21 ppm                | N <sub>2</sub> O: 2.0 ppm               | C <sub>2</sub> H <sub>2</sub> : 2.4 ppm |
| C <sub>2</sub> H <sub>4</sub> : 1.4 ppm | C <sub>3</sub> H <sub>6</sub> : 1.0 ppm | C <sub>3</sub> H <sub>8</sub> : 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.<br>(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) –<br>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<sub>6</sub>**

### ■ Analysis of Trace SF<sub>6</sub> in the Atmosphere

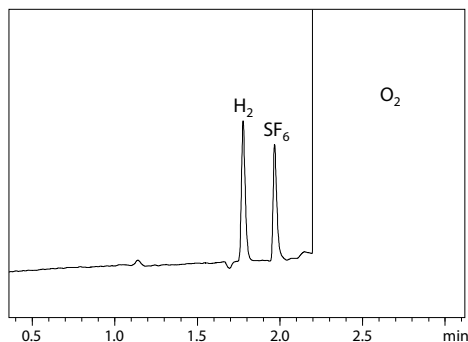
Since SF<sub>6</sub> is a potent greenhouse gas and its emission into the atmosphere must be avoided, leak tests for insulation gas from electrical equipment and residue tests after collecting gas may require analysis of trace amounts of SF<sub>6</sub>. Table 2 lists the measurement conditions and Fig. 2 shows the result of analyzing trace SF<sub>6</sub> in the atmosphere.

SF<sub>6</sub> at a concentration of 0.1 ppm was detected (S/N = 24<sup>\*1</sup>) and favorable linearity was obtained in the range from 0.1 to 50 ppm (R<sup>2</sup> = 0.9998).

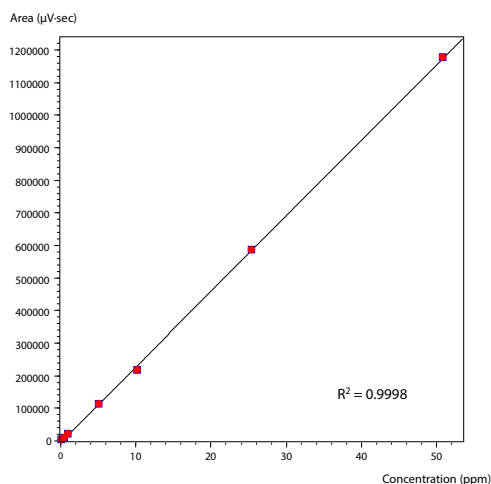
\*1 Determined by calculating noise from the baseline between 0.5 and 1.5 min.

**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<sub>6</sub> (0.1 ppm) in the Atmosphere**



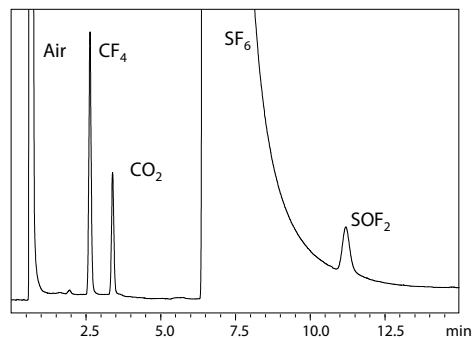
**Fig. 3 Linearity of SF<sub>6</sub> in the Atmosphere (0.1 to 50 ppm)**

### ■ Analysis of SF<sub>6</sub> Decomposition Gas

SF<sub>6</sub> 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<sub>6</sub> 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<sub>4</sub> and SOF<sub>2</sub> which are SF<sub>6</sub> 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<sub>6</sub> Decomposition Gases (CF<sub>4</sub>: 310 ppm, SOF<sub>2</sub>: 107 ppm)**

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