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Application News

Brevis™ GC-2050 Gas Chromatograph

Analysis of Trace Carbon Monoxide (CO) in Hydrogen Fuel Using Jetanizer™

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User Benefits

- Use of the Jetanizer, FID nozzle-type methanizer, makes it possible to detect carbon monoxide (CO) by FID, enabling analysis with a simple system configuration.
- Trace analysis of CO and CO_2 with excellent repeatability is possible, even with N_2 carrier gas.

■ Introduction

In recent years, accompanying the development of fuel cell technologies that generate electricity using hydrogen (H_2) fuel, popularization of fuel cell vehicles (FCVs) and home fuel cell systems is progressing. Because the main impurities that reduce the efficiency of hydrogen fuels are carbon monoxide (CO), carbon dioxide (CO_2), and methane (CH_4), the international standard ISO 14687-2 specifies allowable concentrations of these impurities.

One GC detector that can be used to analyze these impurities in hydrogen fuels is BID (Barrier discharge Ionization Detector), which uses helium (He) as the carrier gas. However, recent He supply problems have heightened demand for high-sensitivity analysis using an alternative carrier gas.

The Jetanizer is an FID nozzle-type methanizer developed by Activated Research Company, in which a catalyst is packed inside the nozzle. Although conventional FID is not sensitive to CO and CO_2, these compounds can be converted to CH_4, which can then be analyzed by FID, by replacing the FID nozzle with the Jetanizer and slight changes in the analysis conditions. Use of the Jetanizer enables highly sensitive simultaneous analysis of CO, CO_2, CH_4 and other compounds with N_2 carrier gas.

This Application News article introduces an example of analysis of CO (allowable concentration: 0.2 ppm), which is one of the impurities in hydrogen fuels, using the Jetanizer and a Shimadzu Brevis GC-2050 gas chromatograph. For details concerning the performance of the Jetanizer, please refer to Application News 01-00599.

This analysis was conducted with a Brevis GC-2050 (Fig. 1), which achieves uncompromising analysis performance in a space-saving instrument. In comparison with this company's existing GCs, the GC-2050 has an approximately 35 % smaller installation footprint, and also contributes to enhanced productivity.

Fig. 1 Appearance of Brevis™ GC-2050

Fig. 2 Appearance of Jetanizer™

■ Analysis Conditions

Table 1 shows the analysis conditions. The FID nozzle was replaced with the Jetanizer, and a Shimadzu MGS-2030 gas sampler was used to introduce the sample gas.

The analysis was conducted with a detector temperature of 400 ˚C and column insertion depth of 45 mm in the FID detector, and the air flow rate changed to 250 mL/min.

Table 1 Analysis Conditions

Model	: Brevis GC-2050
Gas sampler	: MGS-2030 + 1 mL loop
Injection temp.	: 100 ˚C
Injection mode	: Split 1 : 1
Carrier gas	: N_2, constant linear velocity mode (40 cm/s)
Column	: SH-Msieve 5A (30 m x 0.53 mm I.D., 50 µm) With particle trap, 2.5 m * When using the Jetanizer, connection of a metal column to the detector side is recommended.
Column temp.	: 40 ˚C (2.5 min) → 40 ˚C/min → 200 ˚C (3 min) → 25 ˚C/min → 250 ˚C → 15 ˚C/min → 270 ˚C (5 min) * Possible heating rate at 200 V.
Detector	: FID + Jetanizer (Column insertion depth in detector: 45 mm)
FID temp.	: 400 ˚C
Makeup gas	: N_2, 24 mL/min
H_2 flow	: 32 mL/min
Air flow	: 250 mL/min

■ Preparation of Calibration Curve of Carbon Monoxide in Hydrogen

Standard gases of CO were prepared for 4 points, 0.1 ppm, 0.5 ppm, 1 ppm, and 5 ppm (v/v%), using H_2 as the dilution gas. A calibration curve was prepared using these standard gases, and linearity was confirmed. Fig. 3 shows the calibration curve and chromatogram. Good results were obtained for the linearity of the calibration curve, as R^2 = 0.9999 or higher. Considering the possibility that samples cannot be quantified accurately if a gas other than H_2 (e.g., air or N_2) is used as the dilution gas for the standard gases when preparing the calibration curve, use of H_2 as the dilution gas is recommended.

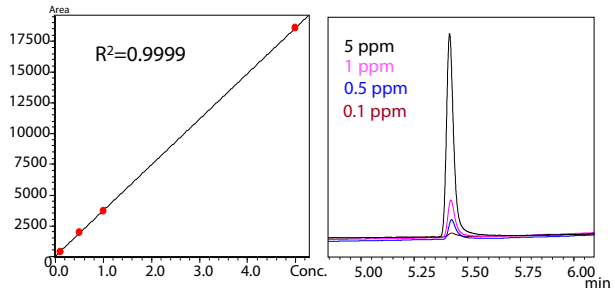

Fig. 3 Calibration Curve and Chromatogram of CO in H_2 Gas

■ Quantification of Trace CO in H_2 Gas and Confirmation of Repeatability

CO gas samples (CO concentration: 0.2 ppm) were prepared using H_2 as the dilution gas, and five sequential analyses were conducted. Fig. 4 and Fig. 5 show the entire obtained chromatogram and the overlaid chromatograms of the five sequential analyses, respectively.

Table 2 shows the results of the quantification values and area repeatability (n = 5) obtained from the results of the calibration curve described above.

The calculated Limit of Detection (S/N = 3) of CO was 0.04 ppm, demonstrating that trace analysis of CO is achieved easily by using the Jetanizer and SH-Msieve 5A column.

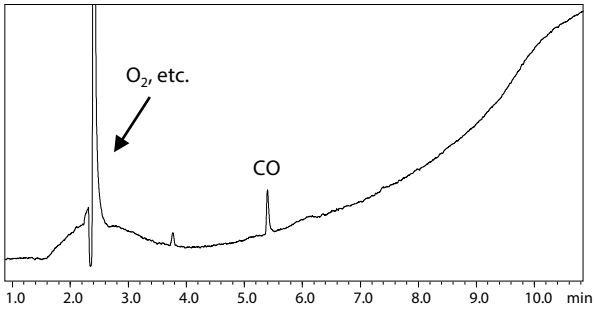

Fig. 4 Chromatogram of 0.2 ppm CO in H_2 Gas

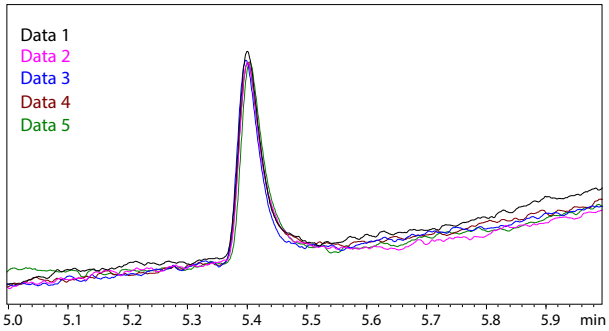

Fig. 5 Overlaid Chromatograms of 0.2 ppm CO in H_2 Gas (n=5)

Table 2 Quantitation Results of Prepared 0.2 ppm CO Gas Sample (ppm) and Area Repeatability %RSD (n = 5)

Quantitation value (ppm)					Area Repeatability (%RSD)
Data 1	Data 2	Data 3	Data 4	Data 5	
0.207	0.202	0.200	0.206	0.201	1.6

■ Conclusion

An analysis of CO, an impurity in hydrogen fuels, was conducted using a Brevis GC-2050 gas chromatograph and a Jetanizer. This method achieved high-sensitivity detection of CO at the 0.2 ppm level, which is the allowable concentration.

Good results were obtained for the linearity of the calibration curve of standard gas samples with CO concentrations of 0.1 ppm to 5 ppm, as the R^2 value was 0.9999 or higher. Satisfactory results were also obtained for the repeatability of five successive analyses.

By using the Jetanizer, it is possible to analyze impurities in H_2 fuels with high sensitivity and use N_2 as the carrier gas.

■ Notice

- Because the SH-Msieve 5A has high absorptivity for water and other components in the atmosphere, periodic conditioning of the column at a column oven temperature of 300 °C for approximately 30 minutes is recommended.
- When analyzing CO in H_2 gas, it is recommended that H_2 also be used for dilution when preparing the standard samples for the calibration curve.
- For other notices concerning the use of the Jetanizer, please refer to Application News 01-00599.

<Related Application>

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

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