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Application News

No. G319

Gas Chromatography

Example of Analysis of Moisture in Nitrogen Using Highly Sensitive Trace Moisture Analysis System

The Shimadzu highly sensitive trace moisture analysis system is a dedicated system GC for moisture analysis using a Nexis™ GC-2030 equipped with Shimadzu proprietary high-sensitivity detector, BID (barrier discharge ionization detector), and a combination of an ionic liquid capillary column and a dedicated sampling valve. These features enable highly accurate measurement of trace moisture in gases at the ppm level. In this experiment, moisture present at a low concentration moisture in a standard gas was analyzed by using the highly sensitive trace moisture analysis system.

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Highly Sensitive Trace Moisture Analysis System

Fig. 1 shows the appearance of the highly sensitive trace moisture analysis system. In the past, high-sensitivity analysis of moisture by GC was considered to be extremely difficult due to two main reasons, namely, the lack of a column capable of selective separation of water and the lack of a detector that could detect water with high-sensitivity. However, more accurate quantitation of the moisture component than with conventional methods such a Karl Fischer titration is now possible by using an ionic liquid capillary column in combination with a BID.

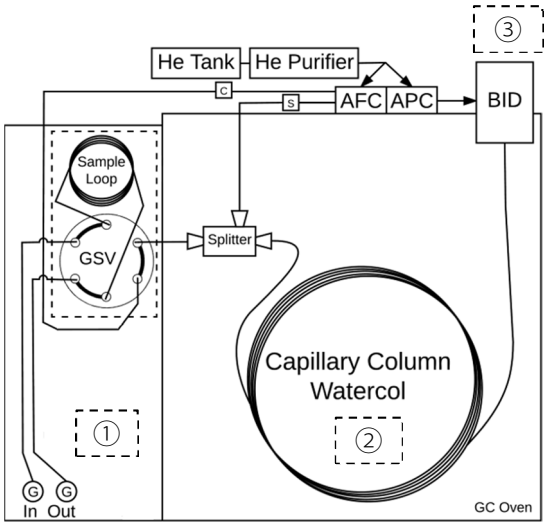

Fig. 1 Highly Sensitive Trace Moisture Analysis System

Composition of Highly Sensitive Trace Moisture Analysis System

① Dedicated sampling system

In sample injection using a syringe, the analysis is affected by trace moisture in the atmosphere. Therefore, in this system, a dedicated gas sampling system is used in place of a sample injection unit. Injection of an accurate amount of gas into the GC is possible by using this gas sampling system.

② Ionic liquid capillary column (Watercol™: Sigma-Aldrich)

Ionic liquids are liquids that consist of only ions and salts that have melting points of no more than approximately 100 °C. These liquids have the following distinctive features when used as the liquid phase of a column.

- Low column bleed
- High maximum use temperature
- Durability against moisture and oxygen

This column system enables measurement of moisture at low concentration levels which had been considered impossible in the past. In particular, although impurities had also been detected as moisture in conventional moisture measurement technologies not accompanied by separation, this system can realize high-sensitivity/accuracy quantitation by separation, utilizing its strong point as a chromatograph.

③ BID (barrier discharge ionization detector)

BID is a universal detector that includes a photoionization technique using low-frequency dielectric barrier discharge plasma and offers high-sensitivity analysis. This new Shimadzu proprietary and general-purpose detector is capable of ionizing and detecting virtually all compounds except helium and neon with higher-sensitivity than other general-purpose detectors such as TCD and FID.

Instrument Composition and Analysis Conditions

Table 1 shows the instrument composition and analysis conditions. It should be noted that this system is a special order product.

Table 1 Analysis Conditions

GC model	: Nexis GC-2030
Injection system	: Dedicated sampling system (valve with helium purge function)
Injection mode	: Split (splitter injection)
Injection volume	: 1 mL (Sulfinert-type piping)
Carrier gas	: He (helium purifier: HP2 connection)
Split ratio	: 1 : 20
Purge flow	: 3 mL/min
Carrier gas control	: Linear velocity 40 cm/s (column flow rate: 1.77 mL/min)
Column	: Watercol-1910 (0.25 mm I.D. × 30 m, d.f. = 0.20 µm)
Column temp.	: 120 °C
Detector	: BID-2030
Detector temp.	: 150 °C
Detector gas	: He 50 mL/min

Analysis of Standard Gas (Moisture in N_2)

The calibration curve was prepared by the absolute calibration curve method using nitrogen standard gas containing approximately 5 ppm, 25 ppm, or 100 ppm of moisture. Fig. 2 shows the chromatogram, Fig. 3 shows the calibration curve, and Table 2 shows the peak area and repeatability (N = 5) at each moisture concentration level. Although trace moisture analysis by GC had been considered difficult until now, a trace moisture analysis was carried out at the ppm level, and good separation, linearity, and repeatability were obtained. The peak of water separated by the ionic liquid capillary column was sharper than those obtained with conventional columns and could also be obtained without tailing. Under these analysis conditions, a shorter analysis cycle time was achieved by setting a constant oven temperature (120 °C) without GC oven cooling time. (Although a sharper peak shape is possible by programmed temperature GC, the analysis cycle time becomes long.)

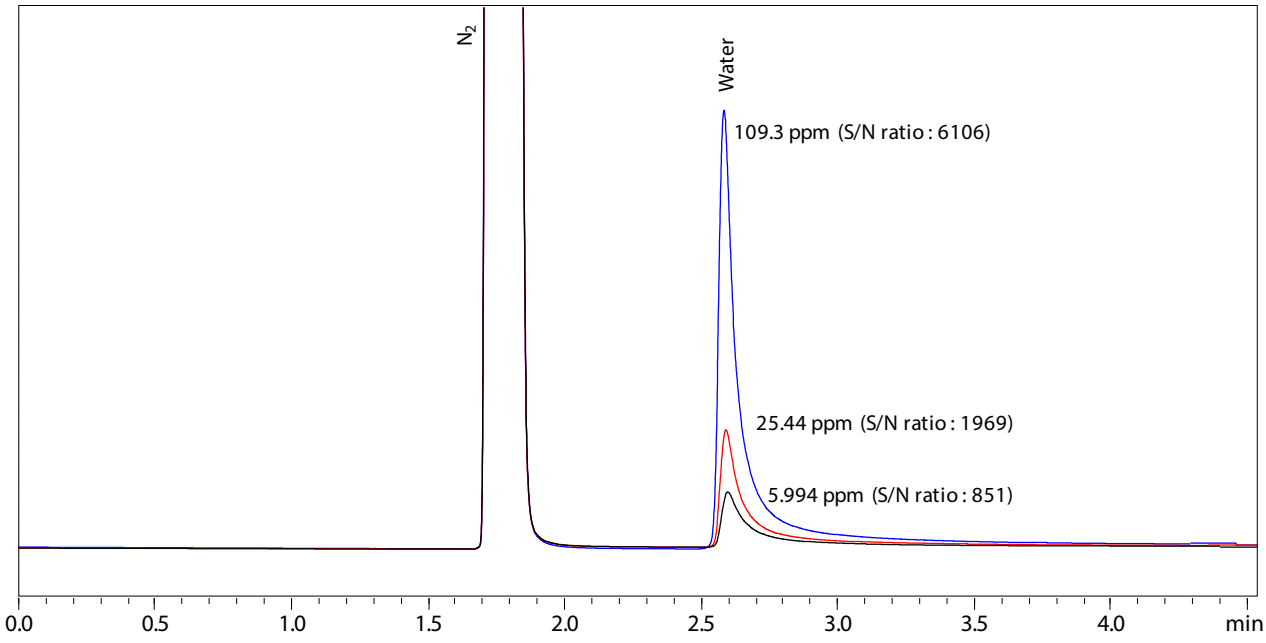

Fig. 2 Chromatogram of Nitrogen Standard Gas Containing Trace Moisture

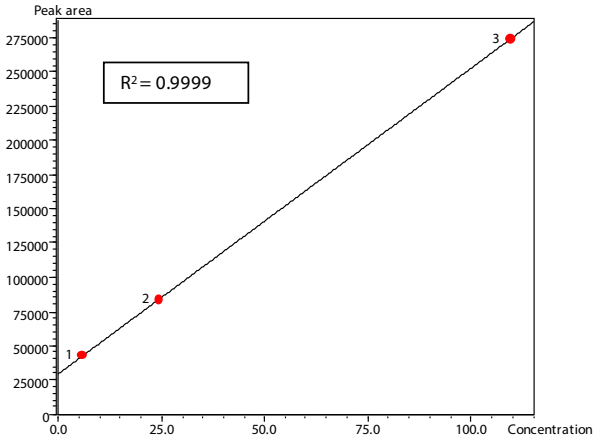

Fig. 3 Calibration Curve

Table 2 Peak Area and Repeatability at Each Moisture Concentration

	Moisture concentration (ppm)		
	5.994	24.44	109.3
Peak area 1	42431	85912	272693
Peak area 2	43249	85077	274031
Peak area 3	42832	83731	272912
Peak area 4	42395	82438	274313
Peak area 5	42373	82360	272680
Average peak area	42656	83904	273326
%RSD	0.894	1.881	0.287

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