SHIMADZU
Excellence in Science

Application News

No. G299

Gas Chromatography

Example Analysis Using a Highly Sensitive Trace Moisture Analysis System
Measurement of Moisture in Chlorofluorocarbon Gas and High-Purity Nitrogen Gas

The highly sensitive Trace Moisture Analysis System employs a sampling system for measuring trace moisture and therefore can prevent the inclusion of water at the time of sample injection. In addition, the system is equipped with an ionic liquid capillary column which enables separation of water and impurities, and Shimadzu's proprietary Barrier Ionization Discharge detector (BID-2030) which achieves detection of trace moisture with high sensitivity.

Chlorofluorocarbon gas and nitrogen gas are used widely in the fields of chemistry and semiconductors, but the presence of water in such gases in some cases hinders their use. It is therefore necessary to develop a system which can easily and accurately measure the amount of water contained in gas. Whereas other moisture meters sometimes cannot perform measurement at all due to the influence of impurities, the trace moisture analysis system can suppress the influence of impurities and achieve accurate measurements.

This article introduces an example of a high-sensitivity measurement of trace moisture contained in standard gases (chlorofluorocarbon gas and high-purity nitrogen gas) using the trace moisture analysis system.

T. Murata

Trace Moisture Analysis System

As shown in Fig. 1, the trace moisture analysis system is a gas chromatography system which consists of the Nexis™ GC-2030 equipped with the BID-2030 barrier ionization discharge detector and a sampling system equipped with gas and liquid sampling valves.

In addition, an ionic liquid capillary column appropriate for the analysis of moisture is employed to allow high-sensitivity analysis of moisture which was difficult until now.

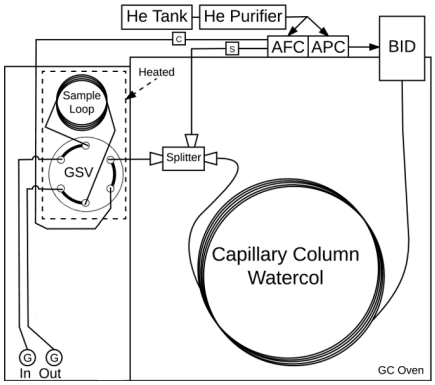

Fig. 1 Trace Moisture Analysis System

Analysis of Standard Gas Containing Trace Moisture

A calibration curve was created by the absolute calibration curve method for helium standard gas (purchased from Takachiho Trading) containing water at concentrations of 10 ppm and 100 ppm respectively.

Table 1 lists the analytical conditions and Fig. 2 shows the chromatograms of the standard gases.

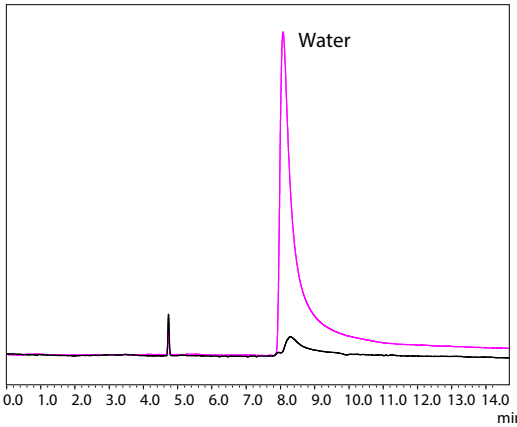

Fig. 2 Chromatograms of Helium Standard Gas Containing Trace Moisture (Black: 10 ppm, Red: 100 ppm)

Table 1 Analytical Conditions

Model	: Nexis™ GC-2030
Detector	: BID-2030 barrier discharge ionization detector
Column	: Watercol™-1910 (0.25 mm I.D. × 60 m, d.f. = 0.20 µm)
Column Temperature	: 100 °C iso-thermal Total 15 min
Injection Mode	: Split 1:25 (Splitter INJ)
Carrier Gas Controller	: Column flow rate 1.72 mL/min (He)
Linear Velocity	: 30 cm/sec
Detector Temperature	: 230 °C
BID Discharged Gas Flow Rate	: 50 mL/min (He)
Injection Volume	: 1 mL (MGS-2030 Sample Loop)

High-Sensitivity Measurement of Trace Moisture in Chlorofluorocarbon Gas and High-Purity Nitrogen Gas

Pure gases of CF_4 chlorofluorocarbon and high-purity nitrogen N_2 were analyzed using the trace moisture analysis system. Figs. 3 and 4 compare the obtained chromatograms with the chromatogram of the helium standard gas containing water at 10 ppm respectively.

The amount of water in the pure gases was obtained using the water peak detected from each pure gas according to the absolute calibration curve method.

Use of the trace moisture analysis system allowed measurement of trace moisture contained in gas at concentrations of 5 ppm and lower. The obtained results indicate that the detection limit (S/N ratio ≈ 3) of this system regarding water measurement is about 0.8 ppm showing that trace moisture in gas was measured successfully with high sensitivity.

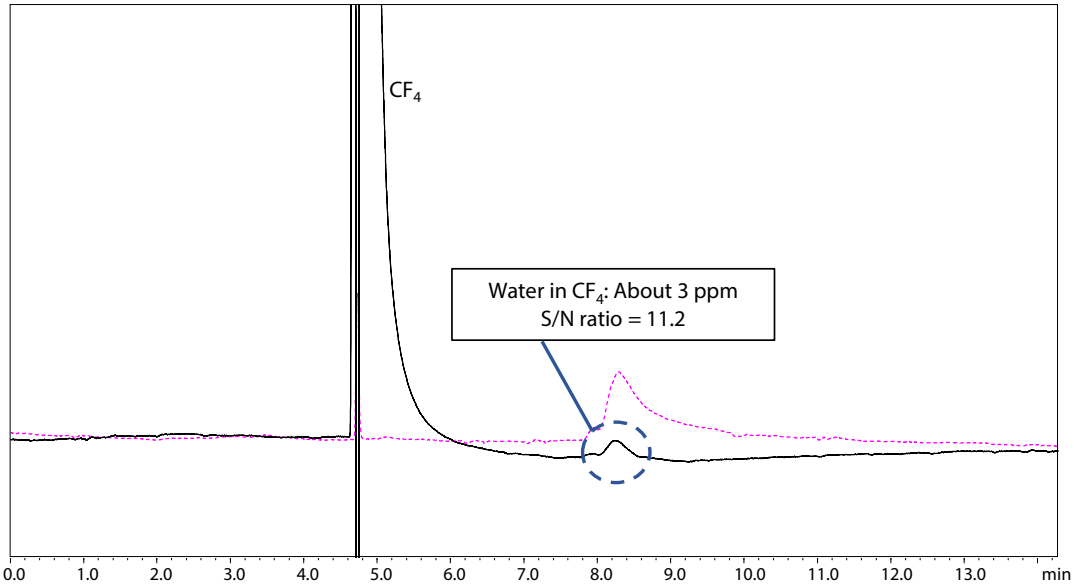

Fig. 3 Comparison of Chromatograms from CF_4 Chlorofluorocarbon Gas (Solid line) and Helium Standard Gas Containing Water at 10 ppm (Dotted line)

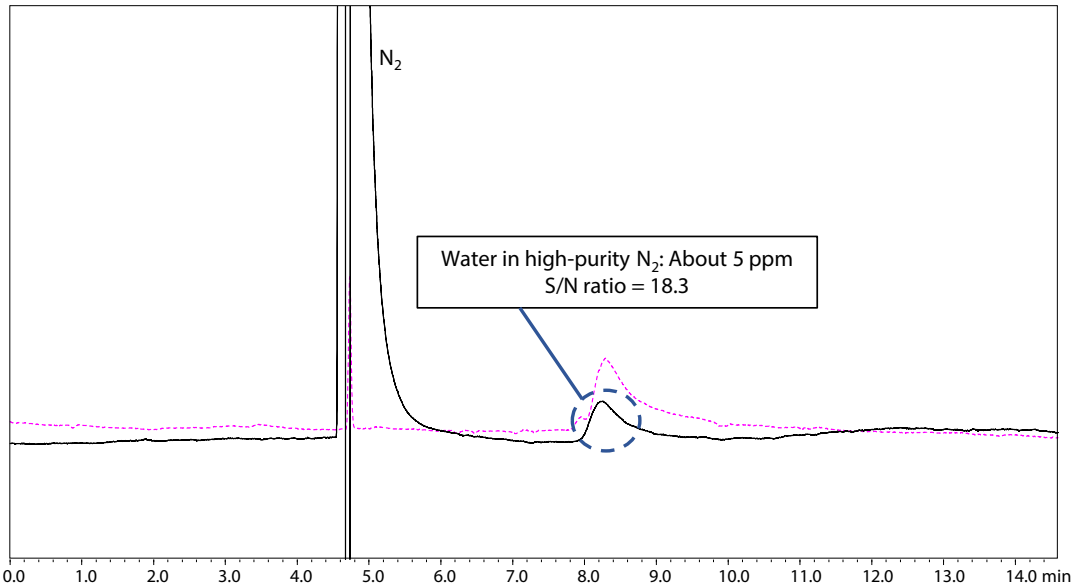

Fig. 4 Comparison of Chromatograms from High-Purity Nitrogen N_2 (Solid line) and Helium Standard Gas Containing Water at 10 ppm (Dotted line)

First Edition: Mar. 2018

SHIMADZU

Shimadzu Corporation

www.shimadzu.com/an/