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Application News

Non-Suppressor Ion Chromatograph

Analysis of Ammonia as an Impurity in Fuel Cell Grade Hydrogen According to ISO 14687 Grade D Using a Gas Generator

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User Benefits

- The non-suppressor method provides good linearity of ammonium ions and reduces maintenance costs.
- Since organic solvents are not used for extraction in pretreatment, environmental impact can be reduced.
- Using a gas generator ensures the validity of the method including pretreatment.

■ Introduction

Hydrogen is expected to be a new power and heat source to reduce greenhouse gas emissions. Hydrogen is an easily available substance that can be produced from the electrolysis of water in our daily lives. However, when used in fuel cells, it is necessary to control impurities in hydrogen gas because certain impurities introduced during the manufacturing process deteriorate the electrode and electrolyte membrane, reducing the performance of the fuel cell or shortening its life. When ammonia is used as a carrier of hydrogen gas due to its distribution as a chemical, there is a possibility that ammonia may be introduced into hydrogen. This article presents an example of analysis of ammonia in hydrogen using an ion chromatograph (IC) based on the standard values described in ISO 14687 Grade D.[1)]

■ Hydrogen Impurity Standard under ISO 14687 Grade D

ISO 14687 Grade D specifies impurities in hydrogen that affect the quality of fuel cell vehicles. Ammonia is specified to be 0.1 µmol/mol or less, and the concentration converted to ammonium ion (NH_4) by IC is 26 µg/L.

■ Analysis of Ammonium Ion by IC

A non-suppressor IC (Fig.1) was used to analyze NH_4. Since the IC can selectively measure ions dissolved in the sample, it is less affected by contaminants than other methods and can measure NH_4 with high accuracy. In addition, by using the same device together with a suppressor IC, it is possible to measure inorganic halogens and formic acid, which are other impurities listed in ISO 14687. Table 1 shows the analytical conditions for NH_4, Fig. 2 shows the chromatogram of a standard sample at a concentration near the specified value (25 µg/L), and Fig. 3 shows the calibration curve in the range from 2.5 µg/L to 200 µg/L. As shown in Fig. 3, the linearity was greater than 0.9999. Table 2 shows the reproducibility of the sample at 10 µg/L, which is around 1/2 of the specified concentration, repeated 7 times.

Fig.1 Non-suppression ion chromatograph HIC-NS

Table 1 Analytical conditions

System	: HIC-NS
Column	: Shim-pack™ IC-C4 (150 mm × 4.6 mm I.D., 7 µm) *1
Guard Column	: Shim-pack IC-GC4 (10 mm × 4.6 mm I.D., 7 µm) *2
Mobile Phase	: 2.5 mM Methane sulfonic acid
Flow Rate	: 1.0 mL/min
Column Temp.	: 40 °C
Injection Vol.	: 100 µL
Vial	: 4 mL, Polypropylene *3
Detection	: Conductivity

*1 P/N : 228-41616-91, *2 P/N : 228-59900-91, *3 P/N : 228-31537-91

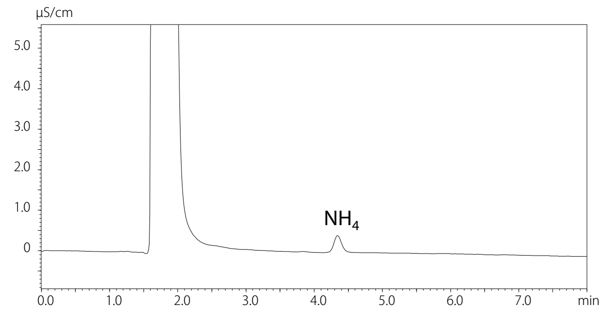

Fig.2 Chromatogram of standard sample (25 µg/L= equivalent to 0.1 µmol/mol)

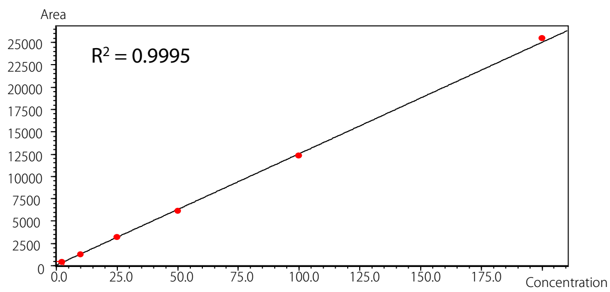

Fig.3 Calibration curve

Table 2 Repeatability (10 µg/L n=7)

	Retention time (min)	Peak area	Concentration (µg/L)
1	4.36	1315	9.99
2	4.36	1355	10.3
3	4.37	1380	10.5
4	4.37	1284	9.74
5	4.37	1292	9.80
6	4.37	1246	9.43
7	4.36	1318	10.0
Average	4.36	1313	9.97
%RSD	0.04	3.41	3.61

■ Verification of Concentration Using a Gas Generator

Next, using a gas generator 491 Flex[2], we mixed ammonia standard gas near the standard value into hydrogen and measured NH_4 collected in ultrapure water by IC. The gas collection conditions and flow are shown in Fig.4. Fig.5 shows the chromatograms of ultrapure water collected from 1st and 2nd impinger, and Table 3 shows the quantitative values and recovery rates of 3 analyses.

When the ammonia concentration was 0.1 μmol/mol, which is the standard value, the concentration of ammonia collected by 2nd impinger was N/D, indicating that ammonia was properly collected by 2nd impinger. Figure 6 shows the sum of the concentrations of the first and second impingers and the correlation between the quantitative value of IC and the concentration of gas produced by the gas generator.

1. Standard gas production by gas generator

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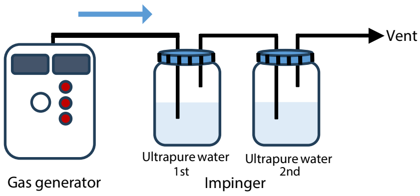
2. Ammonia is trapped in ultrapure water in the impinger.



Collection conditions

Flow Rate	:	500 mL/min
Sampling Time	:	30 min
Water Amount in Impinger	:	40 mL for each
Material of Impinger	:	Perfluoroalkoxy alkanes 100mL volume

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3. The collected water is analyzed by ion chromatography.

Fig.4 Gas collection conditions and flow

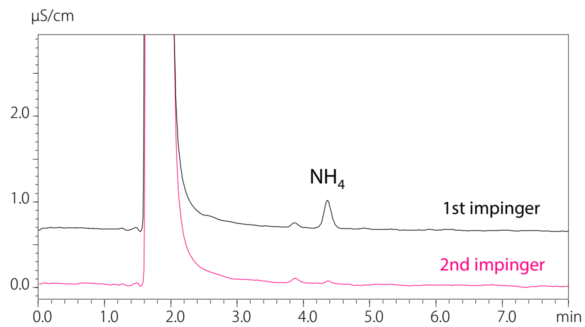

Fig.5 Chromatogram of NH_4 discharged from gas generator (0.1 μmol/mol)

Table 3 At ammonia standard gas concentration of 0.1 μmol/mol
Analysis results of NH_4 quantitative value and recovery rate (3 times)

	NH4 Assay value (μmol/mol)		Total NH_4 Assay value (μmol/mol)	Recovery (%)
	1st Impinger	2nd Impinger		
1st	0.091	N/D *	0.091	91
2nd	0.092	N/D	0.092	92
3rd	0.084	N/D	0.084	84

* S/N=3 or less is defined as N/D.

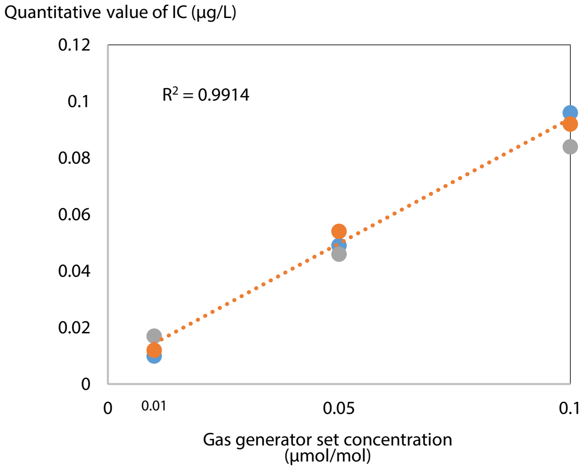

Fig.6 Correlation between quantitative value of IC and gas concentration produced by gas generator

■ Conclusion

Ammonia, an impurity in hydrogen, was analyzed using a non-suppressor IC, and it was confirmed that the method met the standard value specified by ISO 14687 Grade D. In addition, when ammonia standard gas near the standard value was mixed into hydrogen using a gas generator, a good recovery rate was obtained, which confirmed the validity of the method including the collection flow of pretreatment.

< References >

1) ISO 14687 Hydrogen fuel quality-product specifications (2025)
2) A product of KIN-TEK in the United States. https://kin-tek.com/

<Related Applications>

1. Analysis of Inorganic Halogen, Formic Acid, Ammonia, and Formaldehyde as Impurities in Fuel Cell Grade Hydrogen According to ISO14687 Grade D, Application News No. 01-01071

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