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Continuous measurement of reactive ammonia in hydrogen fuel by online dilution module coupled with Fourier transform infrared spectrometer

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ABSTRACT

Fuel cell electric vehicles hold great promise for a diverse range of applications in reducing greenhouse gas emissions. In power fuel cell systems, hydrogen fuel serves as an energy vector. To ensure its suitability, it is necessary for the quality of hydrogen to adhere to the standards set by ISO 14687:2019, which sets maximum limits for 14 impurities in hydrogen, aiming to prevent any degradation of fuel cell performance. Ammonia (NH_3) is a prominent pollutant in fuel cells, and accurate measurements of its concentration are crucial for hydrogen fuel cell quantity. In this study, a novel detection platform was developed for determining NH_3 in real hydrogen samples. The online analysis platform integrates a self-developed online dilution module with a Fourier transform infrared spectrometer (ODM-FTIR). The ODM-FTIR can be operated fully automatically with remote operation. Under the optimum conditions, this method achieved a wide linear range between (50~1000) nmol/mol. The limit of detection (LOD) was as low as 2 nmol/mol with a relative standard deviation (RSD, $n = 7$) of 3.6% at a content of 50 nmol/mol. To ensure that the quality of the hydrogen products meets the requirement of proton exchange membrane fuel cell vehicles (PEMFCV), the developed ODM-FTIR system was applied to monitor the NH_3 content in Chengdu Hydrogen Energy Co., Ltd. for 21 days during Chengdu 2021 FISU World University Games. The proposed method retains several unique advantages, including a low detection limit, excellent repeatability, high accuracy, high speed, good stability, and calibration flexibility. It is an effective analytical method for accurately quantifying NH_3 in hydrogen, especially suitable for online analysis. It also provides a new idea for the analysis of other impurity components in hydrogen.

The global greenhouse effect has led to the recognition of hydrogen as an excellent alternative energy source in the 21st century, especially for vehicles [1-3]. In a hydrogen vehicle, the fuel cell system requires high purity and quality hydrogen, as even small amounts of contaminants can have irreversible effects on the performance and lifespan of the fuel cells [4-9]. To mitigate the detrimental effects of pollutants, several international standards have been established including ISO 14687:2019 [10], EN 17124:2018 [11], and SAE J2719:2020 [12], which sets the amount fractions of 14 contaminants and their respective maximum thresholds. Sensitive analytical methods have been developed for measuring ISO 14687:2019 impurities in fuel cell graded hydrogen. In recent years, Heleen Meuzelaar used laser-based spectroscopic methods for HCHO, HCOOH, HCl, and HF analysis in hydrogen fuel to promote traceable hydrogen purity analysis. These methods have sufficient high sensitivity and low uncertainty [13]. A new system by combining online cryogenic trapping of trace sulfur compounds and subsequent GC-SCD was developed by Pan's group for sulfur compound detection in hydrogen. This method obtained a much lower LOD than the minimum concentration level of total SCs by ISO 14687:2019, which shows great promise for practical applications [14]. Furthermore, Sang Woo Kim's group developed a hydrogen impurity analyzer (HIA) based on mobile gas chromatography with a thermal conductivity detector for the quality assurance of hydrogen fuel, where O_2, Ar, N_2, and CH_4 were selected as target impurities [15]. Cavity-enhanced Raman Spectroscopy technology was used for the detection of gaseous impurities in hydrogen for fuel cells. At a total pressure of 0.1 and 2.5 MPa, the LOD can reach sub-ppm- and ppb-levels. This method has excellent application prospects in gaseous impurity analysis in

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hydrogen [16]. In addition to the above research, other analytical methods have also been introduced [17-23]. From the reported research methods, it can be seen that currently, most analysis methods focus on the analysis of inorganic components and sulfides, with relatively little research on other active impurity components in hydrogen.

As one of the key impurities, NH_3 can readily react with the proton in the proton exchange membrane to generate NH_4^+. This reaction reduces the proton transfer capacity of the electrolyte membrane, leading to a significant degradation in fuel cell performance [24-27]. NH_3 has a high reaction active and low threshold (100 nmol/mol), which immaterially improves the difficulty of accurate quantitative analysis. To date, a gas chromatography-mass spectrometry (GC–MS) method was employed to analyze the concentration of NH_3 in nitrogen. The method utilized single ion monitoring ($m/z = 17$) with a LOD of 0.5 μmol/mol. It is important to note that this LOD is higher than the defined threshold for NH_3 [28]. ASTM D7550:2009 describes an ion chromatography method for measuring NH_3 in hydrogen with sensitivities in the low nmol/mol. This method must involve dissolving NH_3 in a solution and analyzing it using ion chromatography. However, it should be noted that this process is complex and can be influenced by environmental conditions [29]. One ASTM D7941/D7941M:2023 method using cavity ring-down spectroscopy (CRDS) was proposed for the determination of contaminants in fuel-cell-grade hydrogen, which proposes that the LOD of NH_3 is 0.86 nmol/mol [30]. A Fourier transform infrared (FTIR) spectroscopy was developed in ASTM D7653:2018 for measuring several impurities in hydrogen. It provides an LOD of 20 nmol/mol for NH_3 analysis [31]. Although the LOD of CRDS and FTIR methods meet the requirements, they have not yet been validated. The GB/T 37244:2018 [32] recommends the use of the ion-selective electrode method [33], which has some disadvantages including the need to prepare multiple standard solutions, susceptibility to interference from manual operation, and limited applicability to air and industrial waste gas. To sum up, there is still a lack of effective methods for monitoring NH_3 in hydrogen.

FTIR, as a mature precise spectroscopy method, has remarkable advantages of simple operation, fast analysis speed, high accuracy, low detection limit, *etc*. It was widely used in the qualitative and quantitative analysis of various micro and trace gases [34-37]. Here a novel method based on an online dilution module (ODM-FTIR) was developed for qualitative and quantitative analysis of NH_3 in hydrogen. In contrast to traditional FTIR, an online dilution module was integrated with FTIR. Additionally, a multi-point calibration for the FTIR instrument can be easily achieved by using a single bottle of standard gas at any time. The ODM-FTIR can be operated fully automatic automatically with remote operation. The schematic diagram is shown in Fig. 1. The performance of the analytical system was verified using NH_3 in hydrogen as an example. NH_3 standard gas was diluted to different amount fractions using a dynamic dilution module and then detected using FTIR. This ODM-FTIR analysis platform has a good linear relationship, low detection limit, and good accuracy in an amount fraction range, which can meet the requirements of accurate analysis of trace NH_3 in hydrogen with good long-term stability. This work provides an idea for the analysis of other impurities in hydrogen.

In this work, NH_3 in hydrogen is prepared at our laboratory in NIMTT (National Institute of Measurement and Testing Technology of China, Chengdu, China) according to ISO 6142–1:2015 [38]. An aluminum gas cylinder (internal water volume: 8 L) is purchased from Shenyang Zhongfu Kejin Pressure Vessels, Co., Ltd. and is further modified in NIMTT by a proprietary technique to passivate the internal surface. This gas cylinder is equipped with a stainless diaphragm valve with minimum dead volume. High purity H_2 was purchased from Jiangsu Wujiang Messer Industrial Gas Co., Ltd.

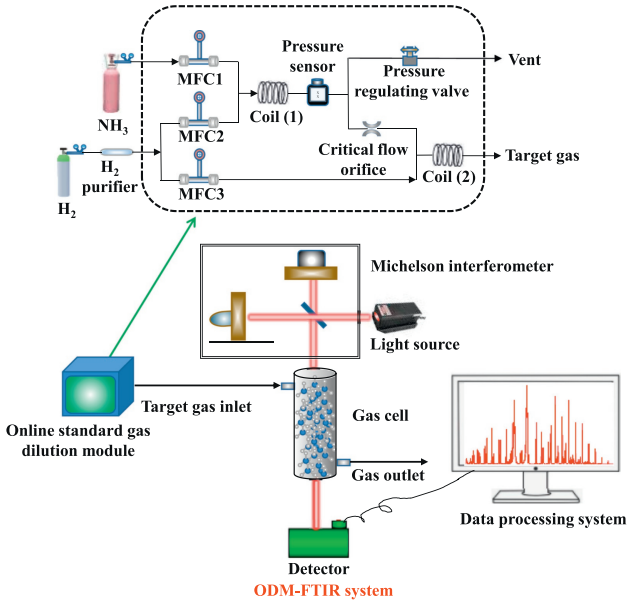

Fig. 1. Schematic diagram based on ODM-FTIR for NH_3.

Pure NH_3 comes from Zhonghao Guangming Research & Design Institute of Chemical Industry Co., Ltd.

The gas cylinder is evacuated in advance using a turbo molecular pump. In terms of the preparation of NH_3 samples in hydrogen, the "master" standards of NH_3 with the concentration of 500 μmol/mol are prepared in the gas cylinder. NH_3 samples with amount fraction of 10 μmol/mol are further prepared by using the H_2 dilution "Master" standard, detailed principles and procedures were referenced in the paper published by NIMTT in 2020 [39].

The fourier transform infrared spectrometer was used for measuring the sampling signal (Thermo Fisher Scientific Inc., ANTARIS IGS Analyzer, USA). The gas cell had a 10-meter path long and was operated at 40 °C to minimize the inhomogeneity of the gas in the cell. The mirrors within the gas cell were coated with gold to limit the deposition of carbon fines introduced in the H_2 sample. Background spectra (found by scanning with the gas cell filled by H_2) were subtracted from the sample spectrum and their ported value was processed from the average value of the 128 scans, with NH_3 discriminated at 1047.15-1045.14 $^{-1}$.

Dynamic dilution module is used for dilution of standard gas to provide different amounts of fraction trace NH_3, which plays a significant role in calibrating instruments. A two-stage dilution system is designed with dilutions ranging from 30 times to 10,000 times, the design principle is shown in Fig. 1. The flow rates of NH_3 reference standard and H_2 were controlled by mass-flow controllers (MFC1, MFC2, and MFC3) respectively. According to the dilution ratio, a certain flow of NH_3 standard gas (MFC1) and H_2 (MFC2) are mixed and diluted at the coil (1), the obtained diluted gas reaches high-precision pressure sensor, which can accurately monitor the pressure of the mix gas. Under the control of the secondary dilution ratio, the excess diluted mix gas is vented. Moreover, the flow rate of the remaining gas was further adjusted by a critical flow orifice and mixed with H_2 (MFC3) in coil (2) to obtain the target amount of fraction gas. All gas lines are SilcoTek® passivated with 1/16 inch external diameter stainless steel tubing in order to reduce physics or chemistry adsorption.

For online analysis, the dynamic dilution module was integrated with the FTIR and the NH_3 reference standard was connected directly to the inlet of the dynamic dilution module, so that gases with different amount fractions constantly flowed through the gas cell respectively.

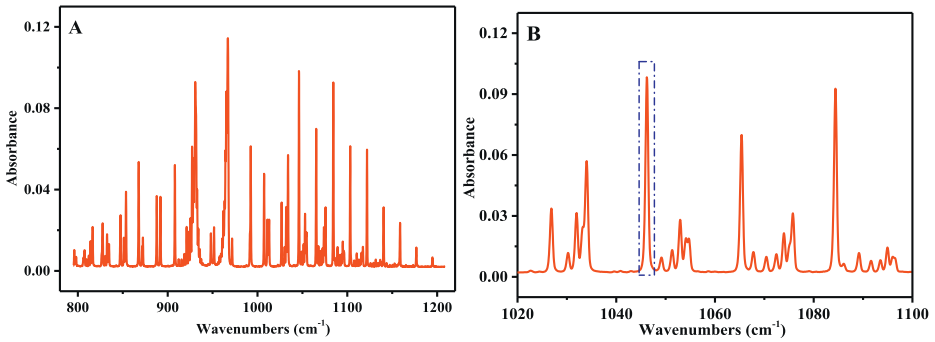

Fig. 2. (A, B) The characteristic absorption peak of NH_3.

The gas cell should be continuously purged with H_2 until it is cleaned. During this process, a background spectrum should be collected using H_2 gas. A vacuum pump is used to extract the H_2 from the gas cell. When the pressure of the gas cell is slightly higher than atmospheric pressure, open the outlet valve of the gas cell to make the gas keep flowing state. Subsequently, the instrument reaches stable state, the target gas is measured, and measurement is repeated 7 times for each amount fraction point.

To evaluate the FTIR spectrometer on real hydrogen gas, the ODM-FTIR analysis platform was installed on-site in a natural gas hydrogen production plant for nearly a month. The hydrogen keeps purging the sampling line during online analysis. When undertaking measurements, sample gas went through the gas cell with 128 scans taken over 210 s. The characteristics of analytical methods were investigated to make sure this method meets the requirements of the ISO 21087:2019 [40].

The quantitative method of NH_3 is established by the TQ analysis software of FTIR. In accordance with the characteristic absorption peak of NH_3, select an appropriate wavenumber range to integrate the area of the characteristic absorption peak, and establish the standard curve by using the integrated area of the absorption peak and amount fraction of NH_3.

The LOD was given by (Eq. 1) [40]:

$$\mathrm{LOD} = 3 \times S_0' = 3 \times \frac{\sqrt{\sum (x_i - x_{\mathrm{mean}})^2/(m-1)}}{\sqrt{n}} \quad (1)$$

where x_i is the FTIR reading for sample i; x_{mean} is the average value; m is the number of replicates used during validation of the analytical method. n is the number of replicates in the routine use.

The limit of quantification (LOQ) was given by (Eq. 2) [40]:

$$x_{\mathrm{LOQ}} = 5 \times S_0' = 5 \times \frac{\sqrt{\sum (x_i - x_{\mathrm{mean}})^2/(m-1)}}{\sqrt{n}} \quad (2)$$

For determine the quantitative wavenumber range more accurately, the characteristic absorption peak of NH_3 was initially determined by measuring a standard substance of NH_3 (Fig. 2A). This standard substance was selected for peak area integration. Given the necessity for the selected absorption peak to exhibit both high signal value and stability without interference from other components, the wavenumber range of 1047.15–1045.14 cm^{-1} was chosen for area integration in this study (Fig. 2B).

The parameters such as injection pipeline, resolution and temperature are the key parameters that affect the performance of the analysis system, which are optimized in this work. NH_3 is a highly active and strongly adsorptive alkaline gas. However, its low content (nmol/mol) can easily get adsorbed on the sampling pipe, leading to uncontrollable measurement results. In order to obtain the best suitable pipeline with smallest adsorption, different pipelines with PFA pipe, Peek pipe, and stainless steel passivation pipe with the same length were investigated using NH_3 standard gas (800 nmol/mol). The measurement results indicate that the response values of the PFA pipe are slightly higher compared to the Peek pipe and stainless steel passivation pipe (Fig. 3). Therefore, the PFA pipe was chosen for the subsequent experiment.

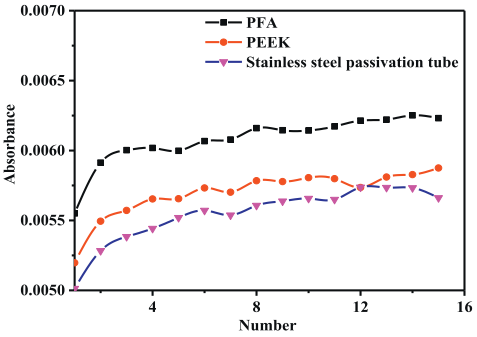

Fig. 3. The effect of injection line with the same length.

Resolution has a great influence on the spectral signal and precision. In order to obtain smaller spectral noise and finer spectra, spectral signals with resolution from 0.5 cm^{-1} to 4 cm^{-1} were investigated using NH_3 standard gas (1.00 μmol/mol). With the increase of the resolution, the fine structure of the spectrum gradually disappears, and the spectral signal also shows a significant decreasing trend (Fig. 4). To ensure the analytical spectrum can accurately and reliably reflect the fine structure and response signal of the object, 0.5 cm^{-1} was selected for the follow-up experiments.

Temperature is another key factor affecting the response value. In the actual analysis process, in addition to considering the signal of the component to be measured, we should also fully consider the condensation of the component to be measured and the possible adverse effects of environmental temperature changes. The effect of temperature on the response value of NH_3 (10.18 μmol/mol) is analyzed at four different temperatures: 40 °C, 50 °C, 60 °C, and 70 °C. Fig. 5A shows the corresponding absorption spectra at different temperatures. It can be clearly seen that the absolute peak area of the response spectral decreases with increasing temperature, which is consistent with the conclusion derived from the ideal state equation of gas (Fig. 5B). To obtain a lower LOD, 40 °C is selected for the follow-up experiments.

Under optimal conditions, the analytical performance of this method is demonstrated using NH_3 as a reference. Figs. 6A and B illustrates the calibration curve and spectral response obtained for contents ranging from 50 nmol/mol to 1000 nmol/mol, respectively. The regression equation is $y = 0.0000074x + 0.000024$ (where x is the content of NH_3). The linear coefficient was better than 0.999, the LOD of NH_3 was calculated to be 2 nmol/mol and the LOQ was 3.3 nmol/mol.

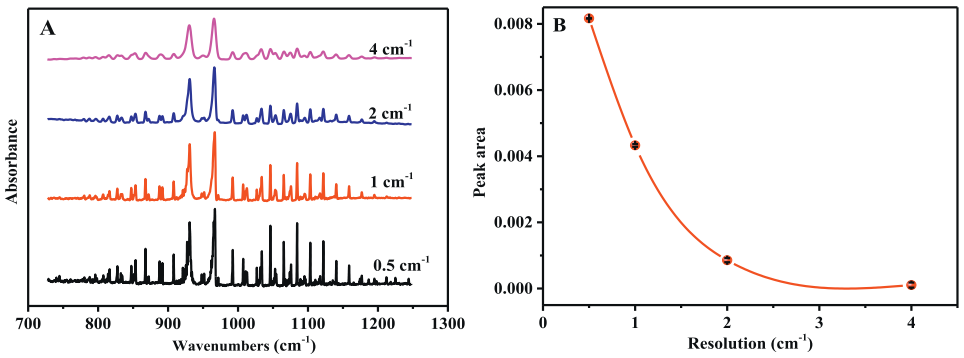

Fig. 4. The influence of resolution on the fine structure of spectrum (A) and response signal (B).

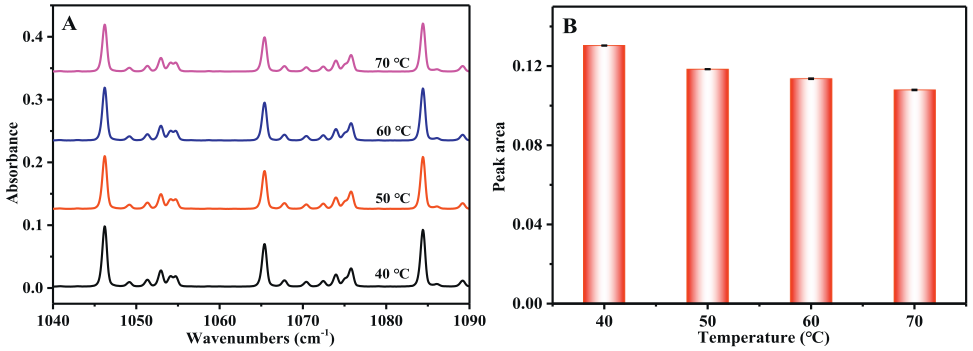

Fig. 5. The influence of gas cell temperature on the response signal spectrum (A) and the response signal comparison of different temperature in terms of the peak area (B).

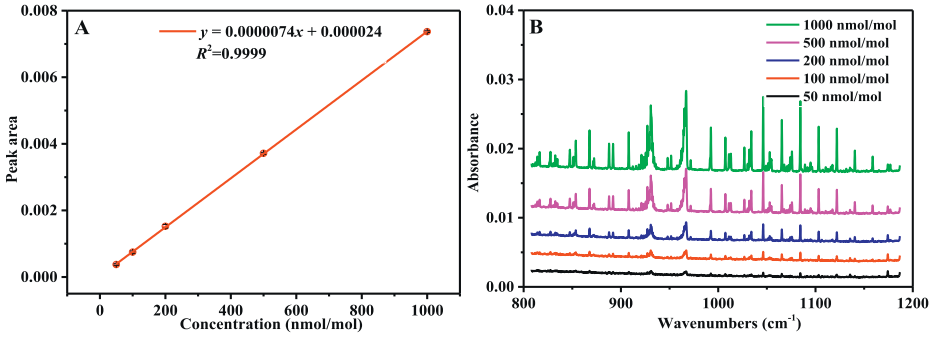

Fig. 6. The analytical performance of this method for NH_3. (A) The linear relationship with various contents of NH_3 ranging from 50 nmol/mol to 1000 nmol/mol. (B) Spectral response of NH_3.

Table 1
Investigation of analytical accuracy and precision.

Content (nmol/mol)	Calculated value (nmol/mol)							Average	Accuracy (%)	Repeatability (%)
210	205	209	208	207	207	209	207	207	−1.2	0.7
	206	199	200	204	197	199	201	201	−4.3	1.5
	203	202	207	205	205	206	213	206	−2.0	1.7
510	518	519	530	523	525	523	521	523	2.5	0.8
	506	508	507	509	508	512	515	509	−0.1	0.6
	516	513	516	511	517	508	509	513	0.6	0.7
790	818	814	815	809	818	810	815	814	3.0	0.4
	805	804	806	804	809	813	811	807	2.2	0.4
	806	806	810	809	812	812	810	809	2.4	0.3

The accuracy and repeatability were further assessed through analysis of reference material at 210 nmol/mol, 510 nmol/mol, and 790 nmol/mol. Measurement was performed over three days (seven replicate analysis each day for each content). Statistical analysis was performed to determine the accuracy and repeatability of the measurement, as shown in Table 1. Test results indicate that this method has satisfactory accuracy and reproducibility.

The long-term stability of this method was investigated by ODM-FTIR. Three content points in the linear range, high, medium, and low, were selected and analyzed for day 33 under the same analytical conditions. As shown in Table 2, the relative standard deviation (RSD) of the analysis results ranged from 1.2% to 2.7%, indicating that the ODM-FTIR method holds great long-term stability for sample detection application.

Table 2
Investigation of long-term stability.

Content (nmol/mol)	Calculated value (nmol/mol)								RSD (%)
	Day 1	Day 5	Day 7	Day 8	Day 11	Day 18	Day 25	Day 33	
210	211	214	207	201	205	203	218	208	2.7
510	508	538	522	509	513	512	522	512	1.9
790	786	792	814	807	809	801	807	792	1.2

Table 3
Analytical figures of merit compared with other methods for NH_3 analysis in hydrogen.

Method	LOD (nmol/mol)	Linear range (nmol/mol)	Accuracy (RSD%)	Repeatability (RSD%)	Long-term stability (RSD%)	Ref.
HPLC-CD	100	/	/	/	/	[41]
OPO based CRDS	100	/	/	/	/	[42]
OFCEAS	100	/	/	/	/	[41]
QEPAS sensor	/	0∼100,000	/	/	/	[43]
This method	2	50∼1000	<4.3%	<1.7%	<2.7%	/

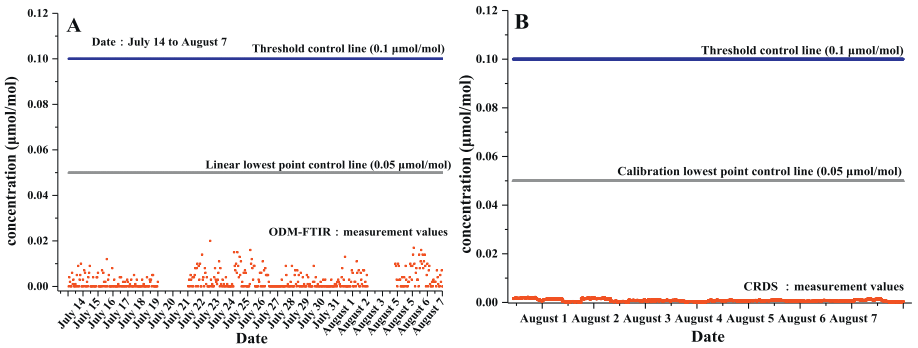

Fig. 7. (A, B) Distribution map of online measurement data of hydrogen fuel samples in pipelines of hydrogen production plant.

Table 3 summarizes the analytical figures of merit characterizing this method and compares its performance to other methods [41-43]. The LOD of NH_3 obtained by this method is comparable to those obtained by other methods. In addition, the LOD might be further decreased by simply increasing the length of the gas cell. The ISO 21087:2019 addresses the validation of analytical methodology for the analysis of hydrogen in the laboratory. Here, detailed standardized procedures for analyzing NH_3 in hydrogen samples are given and the limit of detection and limit of quantification, working range, trueness, and precision are fit for purpose criteria in ISO 21087:2019.

The demonstrated results revealed the great potential of the developed system in determining NH_3 in H_2 with high sensitivity, precision and accuracy. To verify the practical utility of this analysis system, it was applied to the determination of trace of NH_3 in real H_2 samples.

"Low-carbon energy, low-carbon transportation" is the concept of hosting the Chengdu 2021 FISU World University Games 80 hydrogen buses and 100 food refrigerated logistics vehicles were deployed. Chengdu Hydrogen Energy Co., Ltd. is responsible for providing hydrogen energy during the games, and they produce hydrogen gas from natural gas using the steam methane reforming (SMR) process. It is important to monitor the quality of hydrogen as the lifetime and performance of hydrogen fuel cells are dependent on it.

To ensure that the quality of the hydrogen products meets the requirement of PEMFCV, the developed ODM-FTIR system was applied to monitor the NH_3 content in Chengdu Hydrogen Energy Co., Ltd. for 21 days. The ODM-FTIR system can also analyze methane, carbon dioxide, carbon monoxide, formaldehyde, formic acid, and hydrogen chloride in the hydrogen gas simultaneously. An online hydrogen quality monitoring data software was developed, which can display the measured values of the ODM-FTIR analysis system in real-time (Fig. S1 in Supporting information).

Table 4
Investigation of analytical accuracy in HRS samples determined by ODM-FTIR online analysis.

Date	ISO 14,687:2019 (nmol/mol)	Average measured value (nmol/mol) ($n=7$)	Accuracy (%)	Repeatability (%) ($n=7$)
2023/07/13	100	0.086	−14.3	6.2
2023/07/20		0.083	−17.1	9.1
2023/08/04		0.099	−1.4	7.0

During the 21 days, the correctness of the threshold concentration of NH_3 was verified in the pre-operation, mid-operation, and post-operation respectively, to ensure the reliability of online monitoring data. Verification results show that the accuracy is less than 20%, which meets the requirements for the determination of NH_3 in hydrogen (Table 4). The data is collected every hour and the distribution of online measurement data of hydrogen fuel samples is shown in Fig. 7A. Measurement results show that the content of NH_3 in hydrogen samples is lower than the ISO 14687:2019 threshold, indicating that the NH_3 impurity content in hydrogen sample meets the requirements of PEMFCV. The measurement results of the ODM-FTIR method are consistent with the conclusions obtained from the on-site CRDS method, which is validated by NH_3 standard gas, further indicating that this method can be used for accurate monitoring of ammonia content in hydrogen samples (Fig. 7B).

In this work, a sensitive and accurate method for determining NH_3 content in hydrogen using the ODM-FTIR system is developed. The method demonstrates a linear correlation for NH_3 in the con-

tent range of (50–1000) nmol/mol, with a correlation coefficient greater than 0.999. The LOD was as low as 2 nmol/mol and the LOQ was 3.3 nmol/mol. This method offers high precision, easy operation, high speed, good stability, and high selectivity, making it an ideal choice for determining NH_3 content in hydrogen samples. Additionally, it provides insights for onsite quantitatively analyzing other impurity components in hydrogen.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Wenqing Deng: Data curation, Writing – original draft. **Fanfeng Deng:** Formal analysis. **Ting Zhang:** Investigation. **Junjie Lin:** Software. **Liang Zhao:** Resources. **Gang Li:** Resources. **Yi Pan:** Conceptualization, Project administration, Supervision, Validation, Writing – review & editing, Methodology. **Jiebin Yang:** Project administration, Resources, Validation.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2024.110085.

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