Application Note

Energy & Chemicals

Total Hydrocarbon Impurity Analysis in PEM Fuel Cell Grade Hydrogen Using the Agilent 8890 GC-FID System

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Abstract

In this application note, an analytical solution for total hydrocarbons (THC) analysis in proton exchange membrane (PEM) fuel cell grade hydrogen was developed using the Agilent 8890 gas chromatograph (GC) with FID system (GC-FID). The developed method measures THCs including methane. The specifications listed in SAE J2719 and ISO 14687 standards require a nonmethane THC measurement. So this method is suitable when combined with comprehensive impurity analytical methods for fuel cell grade hydrogen analysis. The methane number was obtained by a separate method and then subtracted from the THC number from this method to obtain a nonmethane THC measurement. Method precision, accuracy, and detection limit were evaluated. The evaluation results demonstrate the method can accurately analyze the THC impurity in hydrogen and identify whether it meets the corresponding quality specification.

Introduction

High-purity hydrogen is crucial for the optimal and long-term performance of fuel cells. Different than the industry grade hydrogen used for chemicals, refining, and metal processing, where small amounts of impurities are acceptable. Fuel cell hydrogen is required to meet the species-specific impurity limits described in the specifications. Such impurities include hydrocarbons, sulfur-containing compounds, ammonia, permanent gases, halogens, and formaldehyde. Each group of compounds has its own regulation limit. The limits are defined based on their impact on the fuel cell lifetime and performance. ISO 14687 and SAE J2719 are the most recognized standards for regulation of fuel cell hydrogen quality.

Multiple testing points are implemented throughout the whole supply chain of fuel cell grade hydrogen—from its production, storage, and distribution to its final application. The analytical approaches employed depend on the impurity type and the corresponding limit. Among the varied contaminants, hydrocarbon impurity is controlled based on THC amount (excluding methane because it has a separate specific limit) and the quality limit as specified in ISO 14687 and SAE J2719 (2 ppm).

There are two ways to analyze THCs in hydrogen. One is to separate and detect each hydrocarbon, then add their concentrations to get the THC amount (excluding methane). This analytical approach is recommended by the GB/T 44238-2024 test method.[1] The other way is by eluting all hydrocarbons as a single peak and quantitating the peak according to methane's response factor (RF). The total THC is then obtained by subtracting the methane amount, which is analyzed by a separate method.

In this application note, THC impurity was tested using the second approach. An Agilent 8890 GC with FID system were used for THC analysis, and the Agilent 990 Micro GC was used for methane measurement. The methane analysis solution is published in application note 5994-8830EN.[2] Here, we will showcase THC analysis using the 8890 GC-FID system.

Experimental

The 8890 GC is configured with one gas sampling valve, a split/splitless (S/SL) inlet, and a flame ionization detector (FID), as shown in Figure 1. The fixed volume of gas sample is injected into the S/SL inlet and part of the sample flows into an uncoated capillary column, then elutes off as a single peak for detection. Table 1 lists experimental conditions for the analysis. The standard gases shown in Table 2 are used to verify system precision, generate methane's RF, and calculate method detection limit (MDL).

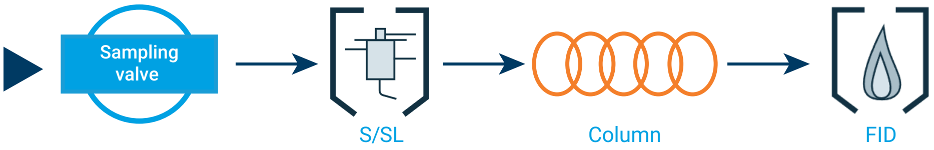

Figure 1. GC flow path schematic.

Table 1. Experimental conditions on the Agilent 8890 GC for THC analysis.

Parameter	Setting
Carrier Gas	Hydrogen
Valve Box	90 °C
Sample Loop	0.25 mL
Inlet Temperature	250 °C
Split Ratio	40:1
Column Flow Rate	2 mL/min
FID Temperature	250 °C
Air	400 mL/min
Fuel Gas/H_2	30 mL/min
Make Up Gas/N_2	25 mL/min
Valve 1 On	0.01 min
Valve 1 Off	0.4 min
Oven Program	40 °C (3.5 min), 30 °C/min to 180 °C, no hold
Column	10 m × 320 µm, 0 µm, Uncoated (p/n 160-2325-10)

Table 2. Standard gases composition.

Components	Calibration Gas	Sample for MDL Calculation
CH_4	10 ppm	2 ppm
H_2	Balance	Balance

Results and discussion

Chromatograms

The uncoated column does not separate the hydrocarbons, allowing them to elute as a single peak. The 10 ppm methane was analyzed to find the hydrocarbon peak retention time (RT) and calibrate the system, as the SAE J2719 specification requires the THC quantified as methane equivalent. The chromatogram is shown in Figure 2. Without interaction with the stationary phase, methane eluted at 0.5 minutes. The hydrogen blank shows no peak in the RT window of methane using hydrogen carrier gas (Figure 3), indicating that the hydrogen matrix in the sample did not interfere with the hydrocarbon quantitation.

Method precision and accuracy

Method repeatability was established by running eight injections of 10 ppm methane consecutively. Site reproducibility was evaluated based on eight injections of 10 ppm methane on the same day and an additional eight injections in 20 days.

Both repeatability and reproducibility tests are precision tests. The repeatability achieved was 2.09%. The system used 0.25 mL sample loop and the method split ratio was set at 40:1. In the repeatability test, the actual methane mass loaded on the column was less than 0.05 ng. With such a low sample amount, the repeatability result was excellent. The reproducibility of 16 injections was 2.12%. Comparable precision obtained within 1 day and 20 days by different analysts demonstrated the stability of the analytical system.

The method accuracy was evaluated by comparing the measured concentration of 10 ppm methane to its nominal value. The quantitation of target analytes was based on methane's average RF, which was calculated according to Equation 1. The THC amount was calculated using Equation 2.

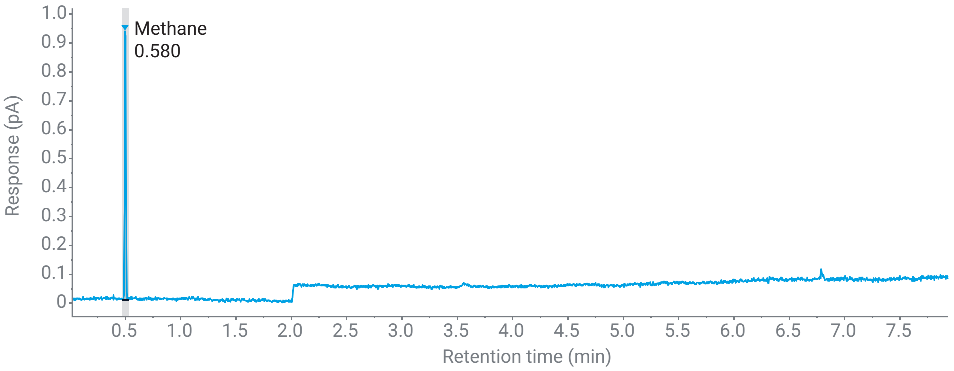

Figure 2. The 10 ppm methane in hydrogen.

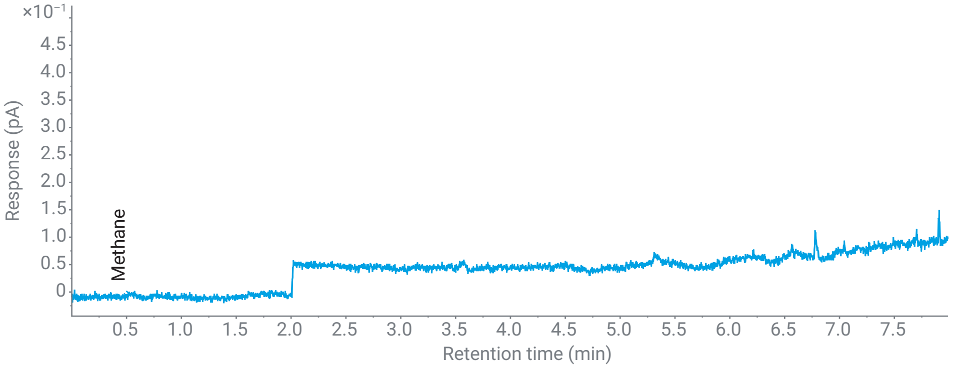

Figure 3. Chromatogram of hydrogen blank.

Equation 1.

RF = Area/Amount (or concentration)

Equation 2.

THC amount = Area of hydrocarbon peak/RF

The average method accuracy for 10 ppm methane (n = 8) was 102.3%. The average accuracy for 2 ppm methane (n = 10) was 111%.

Method detection limit

MDL was derived from 10 injections of 2 ppm menthane (test concentration equals to THC quality limit) according to EPA CFR-2011-40 (Equation 3). The calculated MDL was 0.97 ppm. As required by SAE J2719, the specification limit of 2 ppm for THC excludes methane.

Equation 3.

MDL = t(n – 1, 0.99) × S

Where:

n = Number of measurements (10)

t = t-distribution value (one-sided) with degrees of freedom of n – 1 at a 99% confidence level; in this method, t = 2.82

S = Standard deviation of 10 parallel measurements

In our collaboration lab, a variety of hydrogen impurities were analyzed on GC platforms with different configurations. Hydrocarbons were analyzed by the system as described in this application note. Methane was measured on the 990 Micro GC using highly sensitive µTCD and then subtracted from the total hydrocarbon results to report nonmethane THC. If methane and other hydrocarbons are expected to test on the same platform, another THC test approach like analyzing the individual hydrocarbon on a coated column can be utilized as described in previous work.[3]

Table 3 provides a summary of the test results, showing that all results met the analytical requirements of regulating the THC in fuel cell grade hydrogen according to SAE J2719 and ISO 14687.

Table 3. Test results summary.

Component	Diluted Certified Concentration (ppm)	Recovery (%)	MDL (ppm)	Repeatability (%)	Site Precision (%)	Specification Limit (ppm)*
THC (Methane Equivalent)	10	102.3	0.97	2.09	2.13	2

*SAE J2719

Conclusion

This application note demonstrates THC analysis in fuel cell hydrogen using the Agilent 8890 GC-FID system.

The hydrocarbons eluted as a single peak on an uncoated column and were quantitated using methane's RF. The method repeatability and reproducibility were around 2%. Method accuracy was better than 120%. The MDL was 0.97 ppm. All results met the analytical requirements of regulating THC in fuel cell grade hydrogen according to SAE J2719 and ISO 14687.

The applied analytical approach requires methane analysis by a separate method, which can be realized using the Agilent 990 Micro GC or using a coated column followed by FID detection on another channel of the 8890 GC system.

References

1. GB/T 44238-2024, Hydrogen for Proton Exchange Membrane Fuel Cell Vehicles-Determination of Helium, Argon, Nitrogen and Total Hydrocarbons-Gas Chromatography Method.
2. Coleman, S.; de Jong, R.; Jones, B.; Beard, K.; Zhang, J.; McPherson, S.; Gatzke, A. Micro GC Analysis of Permanent Gas Impurities in PEM Fuel Cell Grade Hydrogen according to SAE J2719 (and ISO 14687, EN 17124 and ISO 19880-8), *Agilent Technologies application note*, publication number 5994-8830EN, **2025**.
3. Jie, Z. Helium, Argon, Nitrogen, and Hydrocarbon Impurity Analysis in Hydrogen Using an Agilent 8890 GC and TCD/FID System, *Agilent Technologies application note*, publication number 5994-7590EN, **2024**.

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