Development of analytical methods for H_2 purity analysis

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There is a strong need to move away from the conventional fossil fuels used today. Hydrogen is a promising alternative and its use will significantly reduce harmful emissions if produced by renewable energy sources. However, the performance of PEM fuel cells is strongly dependent on the presence of impurities in the hydrogen gas. To ensure that the hydrogen quality meets the criteria specified in ISO 14687-2: 2012, it has to be analyzed for the presence of impurities.

Many of the impurities present in the hydrogen gas strongly adsorb and have maximum allowable levels at nmol/mol level posing stringent requirements to the analytical instrumentation and sampling system. An overview is given of the development of the analytical capabilities at VSL with respect to the analysis of selected analytes in hydrogen.

Introduction

The performance of PEM (Proton Exchange Membrane) fuel cells is strongly dependent on the presence of impurities in the H_2 gas. Table 1 shows some of the specifications in ISO 14687-2: 2012.

Table 1 *Specifications for impurities in ISO 14687-2*

Impurity	Max. amount fraction (µmol/mol)
Total hydrocarbons (C_1 basis)	2
Carbon dioxide	2
Carbon monoxide	0.2
Total sulphur compounds	0.004
Formaldehyde	0.01
Formic acid	0.2
Ammonia	0.1
Total halogenated compounds	0.05

VSL has developed analytical capabilities at or below the specifications of ISO 14687-2 for the analysis of most of the specified analytes in hydrogen.

Measurement instrumentation & methods

For the analysis of ammonia, formaldehyde, formic acid and hydrogen chloride a mid-infrared CRDS spectrometer is used.

Figure 1 *Mid-infrared light source tunable from 2.4-5.1 µm*

The spectrometer is based on a tunable infrared source (Figure 1). To enable the analysis of often only small ("real") sample volumes, and to also shorten the time of analysis, the measurement cell and flow system are coated with SilcoNert 2000. The analysis of all 4 analytes requires less than 60 L of H_2 gas.

Figure 2 *Analysis of a H_2 sample.*

For the analysis of sulphur compounds, a GC-SCD (model Agilent 6890) is used equipped with a DB-1 capillary column and helium as carrier gas. All tubings, including the tubing inside the GC, and devices used were Silcosteel® passivated.

Results

Figures 3 and 4 depict measurements of the analysis of formic acid, formaldehyde and ammonia.

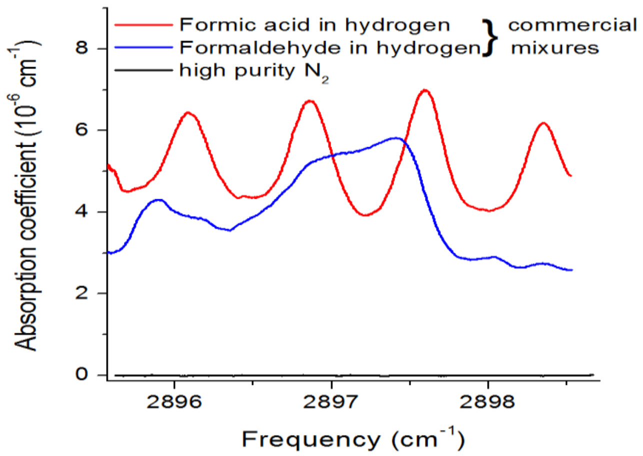

Figure 3 *Measurement of formic acid and formaldehyde in H_2 (nominal 10 µmol/mol and 1.3 µmol/mol, respectively).*

Detection limits for the different compounds are at or below the specifications in ISO 14687-2.

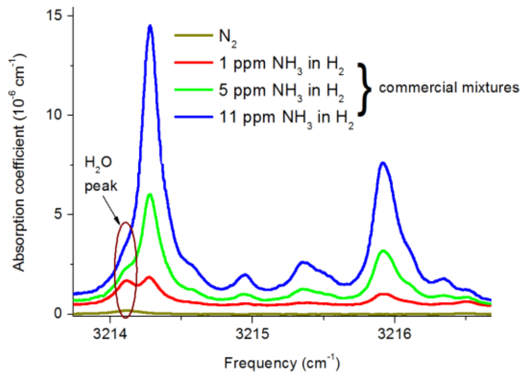

Figure 4 *Measurement of 3 ammonia in hydrogen mixtures*

VSL is currently using the developed methods to analyze H_2 produced via different methods like steam methane reforming and electrolysis right at the hydrogen production sites, using especially selected sample containers for the transfer of these samples.

Discussion

VSL now has measurement methods available for the most challenging impurities specified in ISO 14687-2. A dedicated sampling and measurement system ensures that analysis can be performed at or even below the high-demanding specifications of ISO 14687-2.

Acknowledgement

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