

ANALYSIS OF HYDROGEN QUALITY

REPORT 2015:177



Analysis of hydrogen quality

According to standard ISO/DIS 14687-2
Pre-Study

KARINE ARRHENIUS, ANNA ALEXANDERSSON,
HALEH YAGHOoby, JOHAN ENGELBREKTSSON,
NIKLAS STRÖMBERG

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Energiforsk AB | Telefon: 08-677 25 30 | E-post: kontakt@energiforsk.se | www.energiforsk.se

Förord

I syfte att koordinera teknikbevakning, samt sammanställa, analysera och sprida information om utvecklingen inom bränslecellsområdet till svenska intressenter, främst fordonsindustrin, finansierar Energimyndigheten ett projekt "Teknikbevakning av bränslecellsområdet". Projektet genomförs under 2014-2016 inom ramen för Svensk Hybridfordonscentrum (SHC) med Energiforsk som koordinator och projektledare.

Denna rapport är en förstudie som har tagits fram inom teknikbevakningsprojektet. Samtliga rapporter kommer att finnas publicerade och fritt nedladdningsbara på Energiforsks webbplats för bränslecellsbevakningen www.branslecell.se och på SHC:s webbplats www.hybridfordonscentrum.se.

Styrgruppen för projektet har bestått av följande ledamöter: Anders Hedebjörn Volvo Cars, Annika Ahlberg-Tidblad Scania, Azra Selimovic AB Volvo, Bengt Ridell Grontmij AB, Göran Lindbergh SHC/KTH, Elna Holmberg SHC och Bertil Wahlund Energiforsk AB. Energiforsk framför ett stort tack till styrgruppen för värdefulla insatser.

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Bertil Wahlund

Energiforsk AB

Sammanfattning

Om vätgas ska produceras i stor skala för nollemissionsfordon, finns det flera olika produktionsmetoder att välja på. Den största produktionen sker idag med ångreforming där ursprunget är naturgas. Denna vätgas används av oljeindustrin och incitament för att använda grön vätgas i denna industri existerar inte idag. Andra alternativ är att använda grön metan istället, att tillverka vätgasen mha elektrolys från vatten eller att använda restströmmar från kemisk industri. Om man vill använda ett annat produktionssätt än elektrolys behöver ett reningsteg tillsättas och man behöver ha god kontroll på vätgasens kvalitet.

Två standarder för ren vätgas finns:

ISO 14687-2 Hydrogen Fuel —Product Specification — Part 2: Proton exchange membrane (PEM) fuel cell applications for road vehicles.

ISO 14687-3 Hydrogen Fuel – Product Specification – Part 3: Proton exchange membrane (PEM) fuel cell applications for stationary appliances.

Eftersom ISO 14687-2 kräver högre renhet och kraven på noggrannhet är store fokuserar detta projekt på denna standard. SP har i detta projekt utvecklat mätmetoder för att kunna utföra mätningar enligt standarden. Ett problem är att standarden har extremt låga detektionsnivåer, vilket gör att det kräver extrema åtgärder för att nå dessa gränser. I slutet av rapporten återfinns också en kort diskussion om pris för vätgas sprunget ur industrins överskottsvätgas.

Att utveckla dessa mätmetoder var mer utmanande än vi trodde då projektet startade. Projektet har dock utmynnat i att SP nu med säkerhet kan utföra de flesta av mätningarna som krävs i standard ISO14687-2. Återstående kan vi utföra i samarbete med våra systerinstitut i andra länder. För att nå ända fram krävs en investering på 500 kSEK i ett speciellt instrument där matrisen är vätgas och därför inte används till något annat ändamål. Av de 14 parametrar som skulle verifieras, kan följande nio parametrar analyseras till specificerade detektionsnivåer: totala kolväteföreningar med bas metan, helium, kväve, argon, koldioxid, formaldehyd, myrsyra, ammoniak, total halogenföreningar (excl. HCl and Cl₂). Två parametrar (vatten och komonoxid) kan analyseras, men med högre detektionsgränser. Partikelkoncentrationen kräver ytterligare metodutveckling med ett instrument som redan finns och två parametrar (syrgas och svavelföreningar), kräver en investering i ett nytt instrument och att tillhörande metoder utvecklas.

Detektionsgränserna är troligen satta mycket lägre än vad som är nödvändigt för att inte skada bränslecellerna. I standarden står det nämnt att gränserna kan komma att justeras och detta tyder på att det redan var känt när man skrev standarden att gränserna var för låga. För att fortsätta detta arbete vore det intressant att pröva denna hypotes.

I nuvarande situation måste minst 10 olika analyser utföras för att täcka 12 av 14 parametrar. Vätgasen måste samplas in på minst 7 olika sätt som inkluderar att fylla gaspåsar, cylindrar, att sampla gasen på olika adsorbenter, att bubbla gasen genom impingers (gasprovtagare) exempelvis. Detta är mycket tidskrävande och således dyrt. Ett sätt att minska antalet olika analyser och samplingsmetoder är genom att använda ett instrument endast tillägnat att mäta orenheter i vätgas. Ett möjligt instrument är ett gasanalys-instrument som kallas OF-CEAS (optical feedback cavity enhancement

absorption spectroscopy). Detta tillåter simultana analyser av åtminstone fyra olika ämnen vid de specificerade detektionsnivåerna. Ämnena som kan analyseras är metan, ammoniak, kolmonoxid, koldioxid, formaldehyd, svavelväte, syre och vatten.

Vissa av detektionsnivåerna i ISO14687-2 är väldigt utmanande och eventuellt är de för lågt satta redan idag. Annars kan de komma att sänkas när materialutveckling för PEM-bränslecellerna går framåt och mer tåliga material används.

Estimeringen av priset för överskottsvätgas från svensk kemi industri ser lovande ut och vi rekommenderar att utreda detta vidare. För att energieffektivisera systemen, för att se på att minska vår miljöpåverkan borde dessa värdefulla strömmar kunna nyttjas på ett bättre sätt. Priset för överskottsvätgas estimerades till 15-20 kr/kg att jämföras mot vätgas från elektrolys för 40-50 kr/kg. Detta estimat bör utvärderas ordentligt och omvandlas till en full affärsmodell, innan några råd kan ges om investeringar. Siffrorna är dock en fingervisning att det finns potential att sänka bränslekostnaden för bränslecells-bilar i Sverige om industrierna hittar rätt affärsmodell.

Summary

If hydrogen should be produced in large scale to be used for zero emission vehicles there is different production methods available for the production. Today the largest hydrogen production is produced by steam reforming with origins from natural gas. This is cheap, but not green and the end consumers are the oil-industry. An alternative could be to use methane from a sustainable source or use an elektrolyzer to split water or to take left-over rest streams from already existing industries. If the production should come from biomass or industry streams it needs to be cleaned and the quality of the hydrogen must be under control. Two standards for clean hydrogen exist:

ISO 14687-2 Hydrogen Fuel —Product Specification — Part 2: Proton exchange membrane (PEM) fuel cell applications for road vehicles.

ISO 14687-3 Hydrogen Fuel – Product Specification – Part 3: Proton exchange membrane (PEM) fuel cell applications for stationary appliances.

The standard for road applications are the most challenging and therefore the one focused on. In this project SP developed methods for the required measurements in the standards above. Also a hydrogen price discussion follows at the end of the report.

Developing these methods was more challenging than we thought when the project started. This project has resulted in that SP now by certainty can carry out most of the required measurements required by the standard ISO14687-2. The remaining tests could be performed in cooperation with similar institutes in other countries. The required limits for this standard are probably lower than necessary meaning that a higher limit could be acceptable and still not harming the fuel cells. For a continuation of this work, it could be interesting to prove this hypothesis. The fact that it is mentioned in the standard that the limits could be readjusted, implies that it was already known when the standard was written. There is still more development to be done in this area and in order to lower the detection limits further an investment of 500 kSEK in advanced instruments that will be dedicated for this purpose only must be done.

Among the fourteen parameters to verify in the standard ISO14687-2, nine parameters; total hydrocarbons compounds as methane basis, helium, nitrogen, argon, carbon dioxide, formaldehyde, formic acid, ammonia, and total halogenated compounds (excl. HCl and Cl₂) can be analyzed at the required detection limit. Two parameters; water and carbon monoxide can be analyzed but with a higher detection limit than required in the standard. The particulate concentration will require a further method development on an instrument already available and two parameters (oxygen and total sulfur compounds) will require purchasing an analytical instrument and developing an analytical method.

In the current situation, at least 10 different analyses will need to be performed to cover 12 of the 14 parameters to verify. In the same way, the hydrogen will need to be sampled in at least 7 different methods including filling gas bags and cylinders, sampling the gas on different adsorbent tubes, bubbling the gas in impingers etc. In the circumstances, sampling and analyses will be time consuming and consequently expensive.

A solution to reduce the number of analyses and sampling methods is to use an instrument specific for the analysis of impurities in hydrogen. A possible instrument is a gas analyzer based on the principle of the optical feedback cavity enhancement absorption spectroscopy allowing the simultaneous analysis of at least four components at the required detection limit. The compounds that can be analyzed with this technique are

methane, ammonia, carbon monoxide, carbon dioxide, formaldehyde, hydrogen sulphide, oxygen and water.

Some of the detection limits specified in ISO14687-2 are very challenging, maybe these limits could be changed already today, but if improvement in the performance of fuel cells for transportation application goes towards more tolerant materials, then setting less restrictive detection limits may be possible.

The price estimation for left-over hydrogen from rest streams from Swedish chemical industries seems very promising and should be investigated further. Also to optimize the energy efficiency of the systems, to minimize the environmental effects, these valuable resources could be used wiser than today. The prize for left-over hydrogen was estimated to 15-20 SEK/kg compared to hydrogen from electrolyze that is about 40-50 SEK/kg. This estimation must be carefully evaluated into a full business model, before any advices on investments could be done. The figures means that there is a potential to lower the fuel cost for fuel cell cars if the chemical industry in Sweden can find a sustainable business model for this.

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1 Background

1.1 Introduction

Hydrogen as fuel is expected to play a growing role in a near future. In the report “Fuel cell and hydrogen technologies in Europe” published 2011, the authors highlighted that hydrogen provided an unique opportunity to contribute to all major European policy objectives in the transport and energy sectors and listed the following advantages:

- Hydrogen is a non-toxic gas with a high mass energy density
- It can easily be produced from water (hydrolysis), biomass, biogas, natural gas and from any other fossil fuel
- Hydrogen provides an efficient decentralized solution for combined heat and power generation when used in conjunction with fuel cells
- Hydrogen can be used as an alternative fuel using fuel cell vehicles (passenger cars, busses, light duty and material handling vehicles)
- Hydrogen offers a significant reduction of green-house gas emissions as hydrogen oxidation only produces water

The hydrogen energy chain includes production, distribution, retail and depot, on-board storage systems and conversion to energy (Roads2HyCom). Among the technologies for conversion, the fuel cell technology is one of the most promising.

A fuel cell works like a battery; it generates electricity from an electrochemical reaction. Fuel cells convert chemical energy into electrical energy and also, as a by-product of this process, into heat energy. A fuel cell uses an external supply of chemical energy (as hydrogen) and can run, as long as it is supplied with a source of hydrogen and a source of oxygen (usually air). Fuel cells are based around a central design using two electrodes separated by a solid or liquid electrolyte that carries electrically charged particles between them. A catalyst is often used to speed up the reactions at the electrodes. Fuel cells are generally classified according to the nature of the electrolyte.

The different kinds of fuel cells are:

- PEMFC: Proton exchange membrane fuel cell
- DMFC: Direct methanol fuel cell
- SOFC: Solid oxide fuel cell
- AFC: Alkaline fuel cell
- MCFC: Molten Carbonate fuel cell
- PAFC: Phosphoric acid fuel cell

There are several other kinds of fuel cells under development. For instance fuel cells running on different types of sugar, but the fuel cells above are the main types.

PEMFC are currently the leading technology for light duty vehicles and materials handling vehicles, and to a lesser extent for stationary and other applications. SOFC and PAFC are used in marine, rail and aerospace applications. Alongside MCFC technology and AFC, their field of application is mainly in the stationary sector. The third sector is the portable sector, with DMFC being one new technology.

The proton exchange membrane fuel cell (PEMFC) uses a water-based, acidic polymer membrane as its electrolyte, with platinum-based electrodes. They operate at relatively low temperatures (below 100°C) and can tailor electrical output to meet dynamic power requirements. Due to the relatively low temperatures and the use of precious metal-based electrodes, these cells must operate on pure hydrogen.

Naturally the hydrogen quality must be suitable for the technology that will use it.

Developing and implementing fuel quality specifications for hydrogen are prerequisites to the widespread deployment of hydrogen-fueled fuel cell vehicles. For this purpose, technical specification were developed and published:

ISO 14687-1:1999/ Cor. 1:2001/ Cor. 2:2008 Hydrogen fuel — Product specification —

Part 1: All applications except proton exchange membrane (PEM) fuel cells for road vehicles. This standard is no longer used.

ISO 14687-2 Hydrogen Fuel — Product Specification — Part 2: Proton exchange membrane (PEM) fuel cell applications for road vehicles.

ISO 14687-3 Hydrogen Fuel – Product Specification – Part 3: Proton exchange membrane (PEM) fuel cell applications for stationary appliances.

These standards contain the following specifications:

Component	Maximum impurity concentration (in µmol/mol unless stated)			
	Types I & II Grade D	Type I Grade E Cat. 1	Type I Grade E Cat. 2	Type I Grade E Cat. 3
Water	5	non-condensing at ambient conditions	non-condensing at ambient conditions	non-condensing at ambient conditions
Total hydrocarbons compounds as methane basis	2	10	2	2
Oxygen	5	200	200	5
Helium	300	400000	400000	1000
Nitrogen	100	400000	400000	1000
Argon	100	400000	400000	1000
Carbon dioxide	2	included in total non-hydrogen gases' (max 50 % mol/mol)	included in total non-hydrogen gases' (max 50 % mol/mol)	2
Carbon monoxide	0,2	10	10	0,2
Total sulphur compounds	0,004	0,004	0,004	0,004
Formaldehyde	0,01	3	0,01	0,01
Formic acid	0,2	12	0,2	0,2
Ammonia	0,1	0,1	0,1	0,1
Total halogenated compounds	0,05	0,05	0,05	0,05
Particulate concentration	1 mg/kg	1 mg/kg	1 mg/kg	1 mg/kg

Type I Grade D: Gaseous hydrogen fuel for PEM fuel cell road vehicle systems.

Type II Grade D: Liquid hydrogen fuel for PEM fuel cell road vehicle systems.

Type I Grade E Category 1: Gaseous hydrogen fuel for PEM fuel cell applications for stationary

appliance systems (for high efficiency applications: 50 % minimum hydrogen).
Type I Grade E Category 2: Gaseous hydrogen fuel for PEM fuel cell applications for stationary appliance systems (for high load applications: 50 % minimum hydrogen)
Type I Grade E Category 3: Gaseous hydrogen fuel for PEM fuel cell applications for stationary appliance systems (for high fuel index hydrogen applications: 99.9 % minimum hydrogen).

The requirements specified by the ISO 14687 standards present a series of analytical challenges, mostly due to the very low detection limit to be achieved. As a large number of components need to be quantified, it is not possible to analyse all the components using a single method, on the contrary, many methods are required. Moreover, a number of these components are unstable and/or reactive which implicates that sampling vessels need to be passivated and extreme care has to be taken during analysis to ensure that these components do not adsorb to sample lines and other analytical equipment. Another challenge is to minimize the contamination of the sample by air especially while measuring oxygen, nitrogen and water. This report proposes analytical methods that can be set up for each of the components named in ISO14687, together with a discussion about the detection limit that can be achieved and how hydrogen should be sampled.

1.2 Goal of the project

The goals of the project is to evaluate analytical methods to determine hydrogen gas quality with regard to impurities as required in the standard ISO 14687-2 “Hydrogen Fuel —Product Specification — Part 2: Proton exchange membrane (PEM) fuel cell applications for road vehicles” and to discuss possible developments in order to improve the detection limits and reduce the number of analyses required. The methods proposed here are based on the instrument park currently available at SP chemical laboratory and the possible developments indicates required investments in order to be able to perform all the analyses as required in ISO14687-2. Another goal is to discuss cost scenario for left-over hydrogen to be used as a vehicle gas compared to hydrogen produced from electrolysis.

1.3 Methods

In the next section “Parameters to analyse”, each of the components regulated in the standard ISO14687-2 are discussed in turn, firstly discussing the analytical challenges associated with the given compound, then describing some analytical methods that have been proposed for this compound before presenting and discussing the methods chosen during the course of this project, and comparing the limits of detection of these methods with the specifications in ISO/DIS 14687-2. Where relevant, the results are illustrated with chromatograms.

For the section “Price”, a small scale interview with different actors on the Nordic market was made. Information retrieved from the recent study “Study on development of water electrolysis in the EU” was also used for comparison.

2 Parameters to analyse

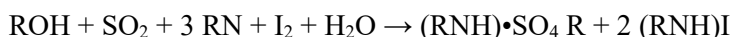
2.1 Water

The major difficulties for the analysis of water are the risk of contaminating the sample with water (from the surrounding air) when sampling, the risk of water diffusing through the sampling lines and therefore contaminating the sample when analyzing and the fact that stable gas mixtures with concentrations of water of less than 10 ppm are still under development (NPL).

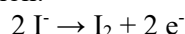
Analysis of water can be performed using different analysis techniques:

- Karl-Fischer titration (KF)
- Gas chromatography/mass spectrometry (GC/MS)
- Cavity-ring-down spectroscopy (CRDS) (NPL)
- Fourier transform infrared spectroscopy (FTIR)
- Optical feedback cavity-enhanced absorption spectroscopy (OFCEAS)

Water in gas can be analysed by the coulometric Karl-Fischer analysis which is suitable for samples where water is present in trace amounts. Coulometric KF determination of water is based on the standard reaction equation for the Karl Fischer reaction:



The iodine is generated electrochemically by anodic oxidation in the coulometric cell according to the following half-reaction:



The reproducible determination of the water content is challenging due to the low detection limit (5 $\mu\text{mol/mol}$) to achieve. This will require a volume of hydrogen of at least 3 liters. The problem is not the coulometric measurement itself, but the sample introduction. An optimal measuring setup and the exact determination of the gas volume are required for water determination in the lower ppm range. A stable gas flow is important for the exact determination of the gas volume. The tubing connections of the inlet line must be kept as short as possible to reduce absorption or desorption in the lines. The material of the inlet line also plays an important role, in order to achieve a determination limit of 5 ppm, metal tubing is essential.

A method based on GC-MS has been proposed by NPL as having the potential for measuring water in hydrogen with a limit of detection below 5 ppm (reporting detection limit is 0.8 ppm). The method requires fully passivation of all surfaces of the sample line, regulators, connectors etc.

NPL has also tested CRDS and achieved a very low detection limit of 0,01 ppm but the instrument needs a sample flow of 500 ml/min and 25 hours analysis time was necessary

to reach a stable reading, implying that the minimum required volume of sample was 750 liters.

Infrared spectroscopy using a gas cell can also be utilized as proposed in the ASTM standard Test Method D7653-10 (Determination of Trace Gaseous Contaminants in Hydrogen Fuel by Fourier Transform Infrared (FTIR) Spectroscopy). The analysis can be completed rapidly without pretreatment. Infrared (IR) spectroscopy measures the IR absorption spectrum of the measurement target gas introduced into the gas cell. Accurate quantitation can be obtained by introducing a standard gas of known concentration into the gas cell, measuring it under the actual analysis conditions and using the measured spectrum to create a calibration curve for the analysis. As infrared radiation has weaker energy than other electromagnetic radiation, very low detection limits can be challenging to reach.

SP has successfully developed a method to analyse water in methane or nitrogen using an Optical feedback cavity-enhanced absorption spectroscopy. Samples are collected in passivated gas cylinders of 1 liter with a pressure of 3-4 bars. The instrument is pre-calibrated in a chosen range. Prior to analysis, the instrument is dried by connecting a dryer in the inlet so the reading when starting the analysis is not more than 1-2 ppm-vol. Sampling lines is carefully flushed when the sample before the analysis. The analysis time necessary to reach a stable reading is not more than 45 min at a flow of 50 ml/min and a requiring totally not more than 3 liters of sample. Unfortunately, this instrument is not adequate for hydrogen as a matrix. It is therefore necessary to dilute the sample at least 10 times with dry nitrogen which lowers the detection limit to 30 ppm. Tests have been done to dilute a gas with dry nitrogen using a gas cylinder of 1 liter equipped with valves on both ends. The content of the gas can be analysed by GC/TCD in order to control on one hand, the dilution factor and on the other hand the contamination with surrounding air. The contamination with surrounding air was found to be less than 1500 ppm which implies that the water contamination in the diluted sample was less than 7 ppm (the surrounding air has 5000 ppm-vol water).

2.2 Total hydrocarbons compounds as methane basis

Methane and other light hydrocarbons (with 2 to 5 carbon atoms) can be analysed simultaneously by GC/FID (gas chromatography with flame ionization detector) using helium as a carrier gas and a Porous Layer Open Tubular (PLOT) columns which are well suited for the analysis of light hydrocarbons.

The gas can easily be sampled in a bag as contamination with air have been shown to be around 300 ppm-vol with a proper handling of the bag. This level of contamination will not significantly affect the concentration of methane and other hydrocarbons. A standard gas containing 10 ppm-vol of methane (CH₄) was used to develop the method.

Blank:

Area	Test 1 (He)	Test 2 (He)	Test 3 (H ₂)	Test 4 (H ₂)	Average	Rel. std*
CH ₄	1.1	0.9	1.1	0.9	1.0	11.5%
Ethylene	3.1	2.4	2.5	1.7	2.4	24%

Ethane	< 1	< 1	< 1	< 1	-	-
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* Rel. std: relative standard deviation

Standard gas:

Area	Test 1	Test 2	Test 3	Average	Rel. std*
CH ₄	30.2	29.9	29.9	30.0	0.6%
Ethylene	2.3	1.6	1.4	1.8	27%
Ethane	< 1	< 1	< 1	-	-

The response factor for methane is calculated to be 2.9 (area/amount in ppm-vol). The detection limit for methane (and for other hydrocarbons with 2 to 5 carbon atoms) is evaluated to be 1 ppm-vol.

Equivalent in methane basis	1 ppm
Ethane	1.6 ppm
Propane	2.9 ppm
Butane	4 ppm
Pentane	5 ppm

Propane and heavier hydrocarbons can be analysed by TD-GC-MS/FID (thermal desorption – gas chromatography with a mass spectrometer for identification and a flame ionization detector for quantification). The limit of detection is evaluated to be 0,05 ppm per hydrocarbon when sampling 100 ml hydrogen on sorbent tubes packed with a very strong molecular sieve adsorbent (as example CabosieveTM SIII) or an multisorbent tubes where one of the adsorbents is a strong adsorbent .

2.3 Oxygen

Oxygen can be analysed by GC/MS or by GC/TCD.

The most challenging difficulty for the analysis of oxygen is the high risk to contaminate the sample with surrounding air when sampling and/or analyzing. The sampling may be performed in gas cylinders (typically of 1 liter). In this case, it is important to carefully flush the cylinder with the hydrogen matrix to analyse. The pressure in the cylinder can be up to 3-4 bar.

GC/MS analysis:

Hydrogen can be analysed with regard to methane, oxygen, nitrogen, argon, carbon monoxide and carbon dioxide using GC/MS. In this study, the GC analysis of the sample has been performed on a Agilent gas chromatograph 7890 equipped with a Agilent mass spectrometer 5975C operated in the electron impact mode under standard conditions. GC/MS chromatograms of sample were obtained using the SIR (*Selected Ion Recording*) mode where the mass spectrometer is set to repeatedly scan a few selected ions ($m/z = 32$ for oxygen, $m/z = 16$ for methane, $m/z = 40$ for argon, $m/z = 28$ for carbon dioxide and nitrogen and $m/z = 44$ for carbon dioxide), which improves the detection limit. A standard gas containing 100 ppm-vol of nitrogen (N₂), 10 ppm-vol methane, 100 ppm-vol

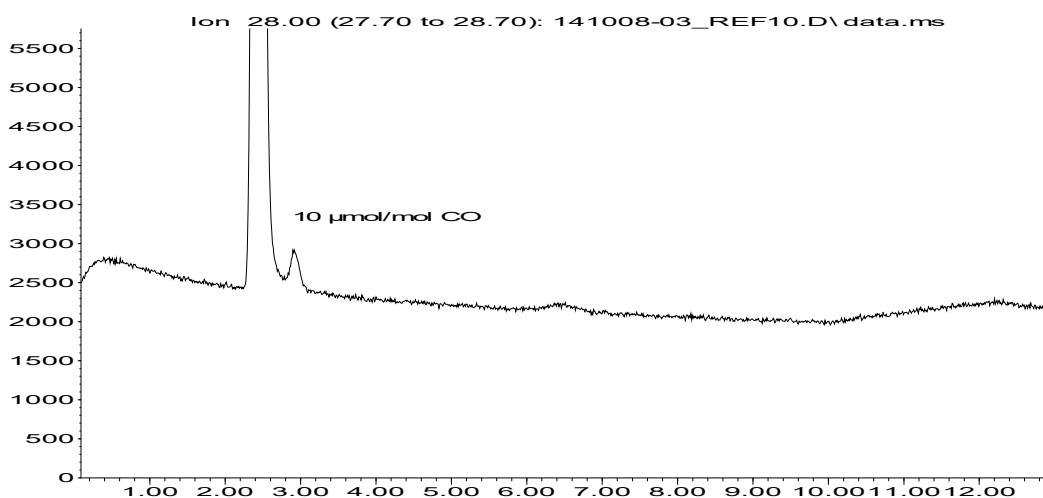
of argon (Ar), 10 ppm-vol of carbon dioxide (CO₂) and 10 ppm-vol oxygen O₂ was used to test the method.

The capillary column used is a Carboxen 1010 Plot, 30 m x 0.32 mm ID.

The column program temperature is monitored from 35°C (hold 10 min) to 170°C (hold 5 min.) at 12°C/min. Helium is used as a carrier gas at a flow rate of 1.25 ml/min. This instrument is equipped with an automated injector and the sample (compounds in a solvent) is introduced into a heated small chamber (called liner) via a syringe through a septum – the heat facilitates volatilization of the sample and sample matrix. The carrier gas then either sweeps the entirety (splitless mode) or a portion (split mode) of the sample into the GC column. In this study, the gas was injected manually using a 250 µl gas tight syringe into the liner. With this method, it was not possible to avoid a significant contamination of the gas sample with surrounding air. To solve the problem, the analysis instrument must be equipped with a proper system for gas introduction.

Because of the air contamination, it was not of course possible to analyse oxygen and nitrogen. However despite the air contamination, it was possible to detect 10 ppm-vol of CO (as shown on the chromatogram below), 10 ppm-vol methane, 100 ppm-vol of argon (Ar) and 10 ppm-vol of carbon dioxide (CO₂). With a proper gas introduction system and by minimizing air contamination of the sample, the detection limit specified in ISO14687-2 for methane (2 ppm-vol), carbon dioxide (2 ppm-vol), argon (100 ppm-vol), oxygen (5 ppm-vol) and nitrogen (100 ppm-vol) should not be challenging. The specified detection limit for carbon monoxide of 0.2 ppm-vol may however be difficult to achieve.

Abundance



GC/TCD analysis with helium as carrier gas:

The introduction of the sample is performed by using an injection system consisting of two 6 port-valves, a sampling loop and a pump. The loop is first filled with the gas to analyse. The gas in the loop is subsequently introduced into the GC column. The cylinder containing the gas to analyse is connected to the inlet port of the injection system.

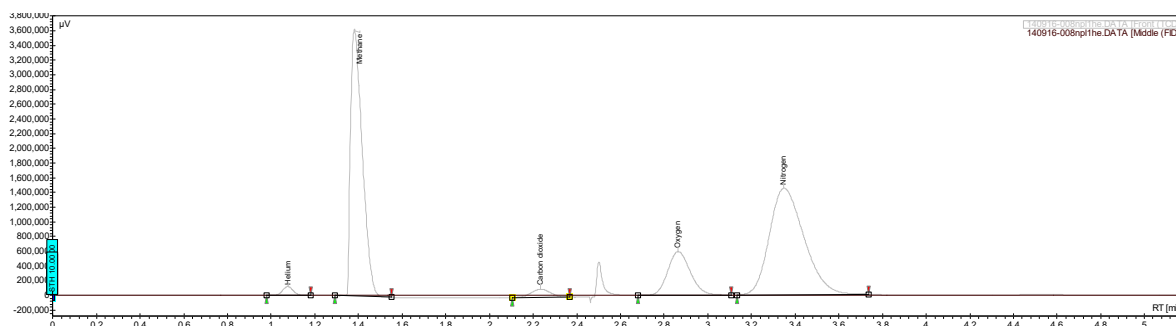
Using this system, the contamination of a sample of pure hydrogen by oxygen from air is estimated to be around 10 ppm-vol which also is the detection limit for oxygen. In these conditions, it is difficult to measure oxygen concentration as low as 5 ppm.

Another challenge is the difficulty to find analytical conditions where oxygen does not coelute with another compound of interest. Oxygen coelutes with argon on molecular sieve columns and with nitrogen on Hayesep columns.

2.4 Helium

Helium can be analysed by GC/TCD (gas chromatography with thermal conductivity detector) using hydrogen as a carrier gas.

The specified detection limit of 300 ppm-vol is not challenging and the gas can be sampled either in a gas bag or in a cylinder as the contamination with air will not significantly influence the concentration of helium. Helium is stable in both gas bags and in gas cylinders.



2.5 Nitrogen, argon and carbon dioxide

Nitrogen, argon and carbon dioxide can be analysed simultaneously by GC/TCD (gas chromatography with thermal conductivity detector) using helium as a carrier gas. Normally, gases are sampled in special gas bags and a known volume of gas is withdrawn from the bag by a pump to fill a loop with the gas to analyse which is subsequently introduced in the columns of the GC/TCD. The challenge with this analysis method consists to not contaminate the sample with surrounding air in order to avoid overestimating the concentration of nitrogen in hydrogen. By using gas bags, the level of air contamination could not be lowered as required and the background of air in the blank was varying significantly. For the purpose to not contaminate the sample, a cylinder must be used to sample the gas to analyse.

A standard gas containing 100 ppm-vol of nitrogen (N_2), 100 ppm-vol of argon (Ar), 10 ppm-vol of carbon dioxide (CO_2) and 10 ppm-vol oxygen O_2 was used to develop the method. A 300 ml-cylinder with a valve at each end was filled with the standard gas. The gas was allowed to flow through the cylinder for some minutes. The valve at the end of the sampling set up was closed and the pressure in the cylinder was set up at 3 bar. Helium (He) blanks were prepared in the same way (Max 2 ppm-vol O_2 , max 5 ppm-vol N_2 in helium). With this method, argon cannot be separated from oxygen. So the result is the sum argon+oxygen.

Blank:

	Test 1	Test 2	Test 3	Average	Rel. std*
CO ₂	< 3	< 3	< 3	-	-
Ar + O ₂	<3	<3	<3	-	-
N ₂	408	440	382	410	7%

* Rel. std: relative standard deviation

Standard gas:

	Test 1	Test 2	Test 3	Average	Rel. std
CO ₂	125	118	115	119	4%
Ar + O ₂	969	968	985	974	1%
N ₂	1589	1465	1601	1552	5%

The results show that the response factor (Area/amount) of nitrogen is 11.4. By using this factor, the contamination of the sample by nitrogen from the surrounding air is evaluated to be around 35 ppm-vol. This value needs to be withdrawn to the amount measured for real samples. The detection limit of 100 ppm-vol specified in ISO 14687 Grade D for nitrogen is then not challenging (calculated detection limit: 50 ppm-vol).

No background of oxygen, argon or carbon dioxide was found in the blank. The expected detection limit of 300 ppm-vol for argon is not challenging but the result from this analysis is the sum of oxygen and argon. If possible, oxygen measured by another analytical method must be withdrawn from the result of argon+oxygen to obtain the concentration of argon. The expected detection limit of 2 ppm-vol for carbon dioxide can be reached by this method but without any marginal.

The cylinder was also filled with hydrogen in order to control the background level of O₂, Ar, CO₂ and N₂ in this matrix. The pump on the GC works harder when the cylinder is filled with H₂ compared to He because of the properties of hydrogen. A possible solution is to disconnect the pump and allow the loop to be filled with the gas to analyse using the pressure in the cylinder.

These compounds can also be analysed simultaneously by GC/MS (see the section of oxygen)

2.6 Carbon monoxide

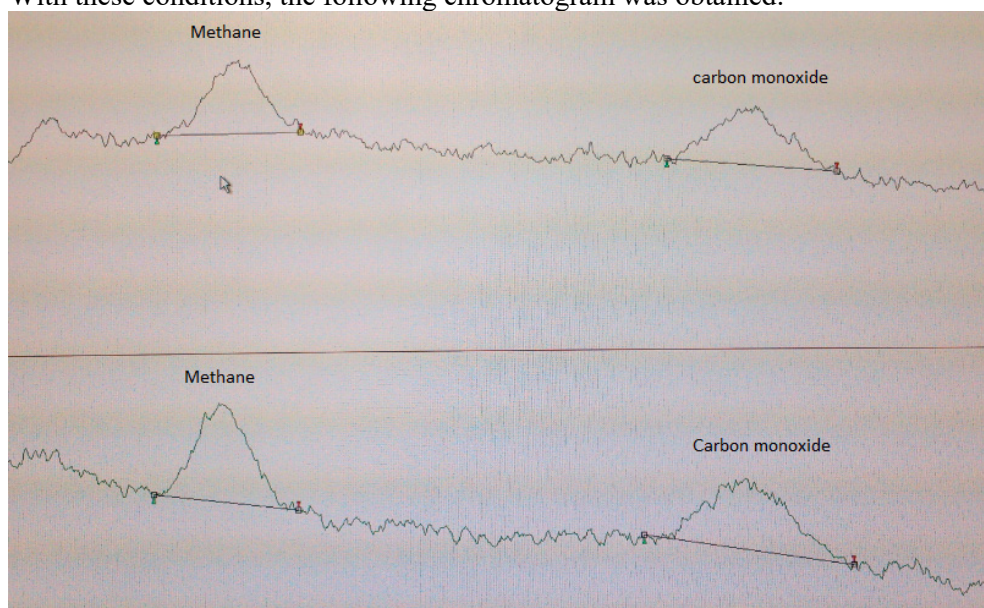
GC/TCD:

Carbon monoxide can be analysed by GC/TCD (gas chromatography with thermal conductivity detector) using helium as a carrier gas. Gas can be sampled in special gas bags and a known volume of gas is withdrawn from the bag by a pump to fill a loop with the gas to analyse which is subsequently introduced in the columns of the GC/TCD. The challenge with this analysis method is the low detection limit of 0.2 ppm.

A standard gas containing 100 ppm-vol of nitrogen (N₂), 100 ppm-vol of argon (Ar), 10 ppm-vol of carbon dioxide (CO₂) and 10 ppm-vol oxygen O₂ was used to develop the method. The parameters must be selected to insure a detection limit as low as possible:

- Optimized column for this analysis: Molecular Sieve 13X
- Detector temperature > 115 °C with a filament temperature at 350°C
- Oven temperature: 95°C
- Loop of 500 µl

With these conditions, the following chromatogram was obtained:



The ratio signal/noise was measured to be around 10 which imply that the detection limit is 5 ppm (for both CO and CH₄). The analysis was performed three times:

Standard gas:

	Conc.	Test 1	Test 2	Test 3	Average	Rel. std	Response factor
Unit	ppm	Area	Area	Area	Area	%	Area/ppm
O ₂ +Ar							
N ₂							
CH ₄	9.42	161	163	160	161	0.9	17.1
CO	9.28	163	161	172	165	3.5	17.8

Carbon monoxide can also be analysed by GC/MS (see the section of oxygen).

2.7 Total sulphur compounds

The analysis of total sulphur compounds to a limit of detection of less than 4 ppb is the most challenging specifications given by ISO/DIS 14687-2. In the report AS6, NPL proposed a method based on gas chromatography – sulphur chemiluminescence detection (GC-SCD) equipped with a loop of 1 ml and samples are introduced in the GC from cylinders. They reached a limit of detection of about 1 ppb.

Hydrogen sulphide can be analysed using optical feedback cavity-enhanced absorption spectroscopy. Samples would be collected in passivated gas cylinders of 1 liter with a pressure of 3-4 atm. The instrument is pre-calibrated in a chosen range and a detection limit of 2 ppb can be achieved. The remaining challenge is that sulphur compounds are very unstable at these low concentrations; the risk of adsorption of the cylinder wall needs to be assessed at these levels.

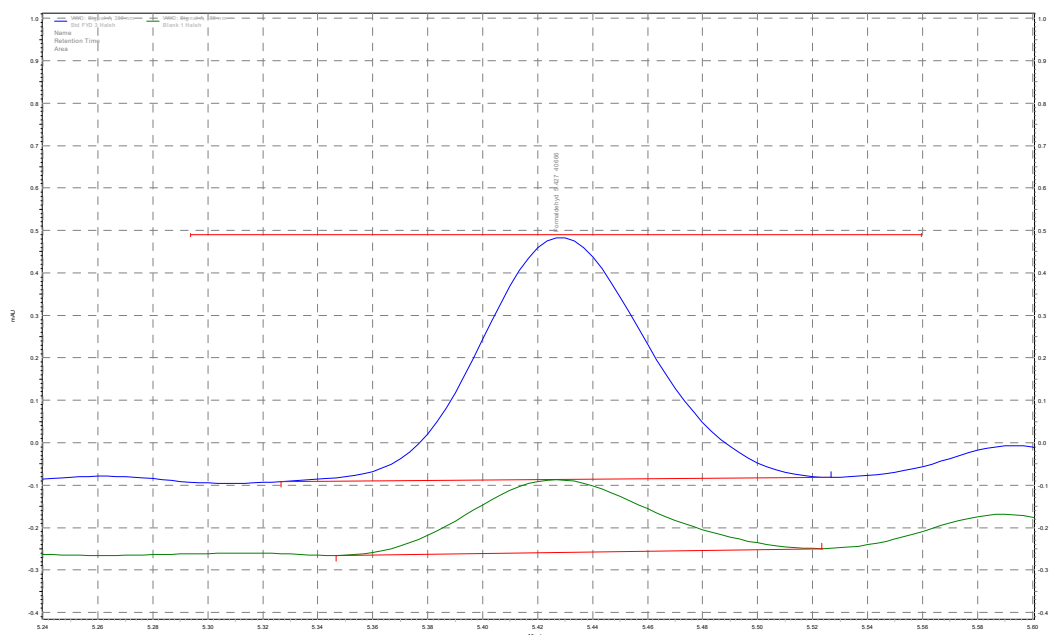
For organic sulphur compounds, thermal desorption/gas chromatography-mass spectrometry may be used. The main advantage is the same method than for hydrocarbons (with 3 or more carbon atoms), and halogenated compounds. The detection limit of 4 ppb with this method is still very challenging.

2.8 Formaldehyde

The sampling of formaldehyde can be carried out by adsorption on cartridge type column containing silica gel coated with 2,4-dinitrophenylhydrazine (DNPH). The sampling is performed onsite at low and controlled flow (typically 0.5 l/min) for a short period of time (6 minutes so the volume is 3 l). As the detection limit for the analytical method is 0.03 µg formaldehyde per cartridge/sample, the sampling of 2.5 liters of hydrogen is enough to reach the detection limit of 0.01 ppm-vol specified in ISO 14687 Grade D. At the laboratory, the cartridge is eluted with acetonitrile and the analysis is performed on a high performance liquid chromatograph with absorbance detector (HPLC/UV-Vis).

In order to test the method, a known amount of formaldehyde was introduced in a gas bag containing pure hydrogen. The hydrogen containing formaldehyde was then passed through a DNHP cartridge. The cartridge subsequently underwent the analytical procedure described above. Pure hydrogen was also passed through DNPH cartridges in order to check that the hydrogen will not interfere with the measurement.

The chromatograms obtained for the hydrogen containing formaldehyde (in blue) and for the pure hydrogen (in green) are presented on the Figure below. The concentration of formaldehyde in the pure hydrogen was found to be less than 0.002 ppm-vol (average of three blanks) and the concentration of formaldehyde in the spiked hydrogen was found to be 0.016 ppm (the spiked concentration being 0.036 ppm). The difference can be explained by the fact that some formaldehyde may adsorb on the walls of the bag.



2.9 Formic acid

Formic acid and other small organic acids in gases can be analysed by purge and trap ion exclusion chromatography. The gas sample is purged through an alkaline buffer solution (15 ml) made of e.g. CAPS (N-cyclohexyl-3-aminopropanesulfonic acid; PKa 10.4) and ultra-purified water that is injected on the ion exclusion chromatography system. A minimum of 1 liter of gas is required to detect the required 0, 2 μmol formic acid /mole hydrogen gas. The sampling flow can be up to 100 ml/min.

Another method can be to use thermal desorption/gas chromatography-mass spectrometry. The main advantage is the same method than for hydrocarbons (with 3 or more carbon atoms), sulphur compounds (except hydrogen sulphide) and halogenated compounds.

2.10 Ammonia

The sampling of ammonia can be carried out on silicagel treated adsorbent tubes. The sampling is performed onsite at a controlled flow (typically 1 l/min) for a determined period of time (15 minutes so the volume is 15 l). The quantification of ammonia is subsequently performed by high performance liquid chromatograph with conductivity detector (HPLC/conductivity detector).

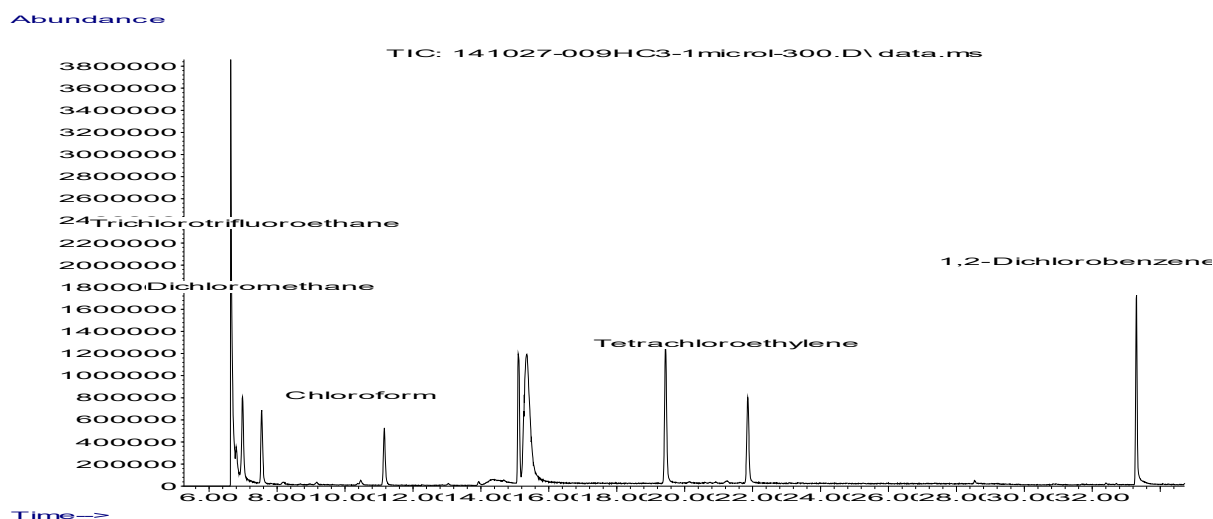
In order to test the method, a known amount of ammonia was introduced in a gas bag containing pure hydrogen. The hydrogen containing ammonia was then passed through a silica gel treated adsorbent tube. The tube subsequently underwent the analytical

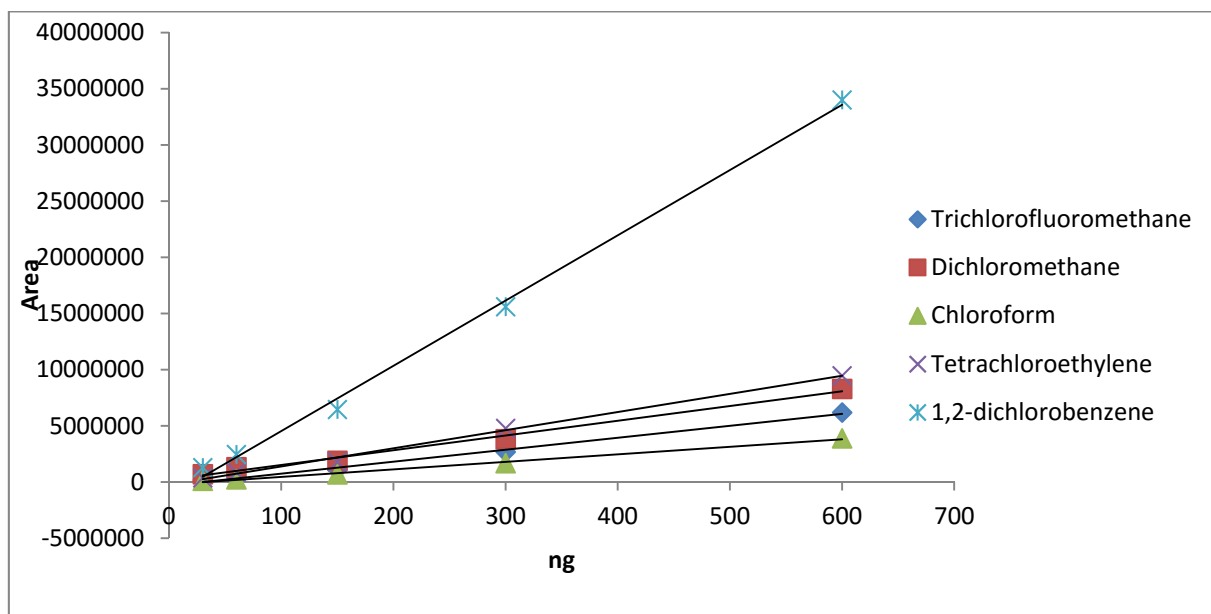
procedure described above. Pure hydrogen was also passed through a silica gel treated adsorbent in order to check that the hydrogen will not interfere with the measurement. The amount of ammonia in the pure hydrogen was found to be less than 0.9 µg per tube. The concentration of ammonia in the spiked hydrogen was found to be 0.8 ppm in good agreement with the spiked concentration of 1.2 ppm.

A simpler and faster method to analyse ammonia would be to use an Optical feedback cavity-enhanced absorption spectroscopy. In this case, samples would be collected in passivated gas cylinders of 1 liter with a pressure of 3-4 liters. The instrument is pre-calibrated in a chosen range and can analyse ammonia down to 0.1 ppm. Prior to analysis, the instrument is passivated with a gas containing ammonia. The analysis time necessary to reach a stable reading is not more than 45 min at a flow of 50 ml/min and a requiring totally not more than 3 liters of sample.

2.11 Total halogenated compounds

Analysis of total halogenated compounds (exclusive HCl and Cl₂) can be performed using TD-GC-MS when collecting the gas to analyse onsite on a multisorbent (ex Carbotrap 300). To reach the detection limit of 0.05 ppm, the required sampling volume is typically 100-200 ml. An example of chromatogram obtained for the analysis of 30 ng of trichlorotrifluoroethane, dichloromethane, chloroform, 1,2-dichlorobenzene and tetrachloroethylene is presented on the figure below. The standard curves for these compounds (from 30 to 600 ng) are reported below.





2.12 Particulate concentration

The particulates in hydrogen fuel for hydrogen fuel cell vehicles may adversely affect pneumatic control components such as valves or other critical system components. Therefore the concentration of particulates in the hydrogen fuel should be limited as specified by ISO14687-2 to 1 mg/kg.

There are two ASTM standards describing how to measure the particulate concentration: ASTM D7651-10: Standard test method for gravimetric measurement of particulate concentration of hydrogen fuel and D7634-10: standard test method for visualizing particulate sizes and morphology of particles contained in hydrogen fuel by microscopy.

The D7651-10 test method uses filters and requires a meticulous handling of these filters. Filters (47 mm diameter polytetrafluoroethylene, PTFE with one side composed of PTFE and the reverse side composed of polypropylene) held in a mini clean room (with HEPA filters and moisture of less than 30%) are transferred to a glove box containing a balance (measuring 10^{-5} g) inside and equipped with a HEPA filter. The glove box is equipped with a HEPA filter and is flushed with clean dry nitrogen (0.5 l/min) and the moisture is maintained at less than 30%. The output of the balance can be downloaded. The weighing is repeated 10 times and the relative standard deviation of the 10 weights should be less than 0.02%. An unused filter is kept inside the glove box and weighed according to the same procedure than sampled filter. The reason for monitoring the weight of an unused filter is to make sure the environment of the glove box does not affect weight of the PTFE filter. The balance is calibrated with 0.05 g and 0.2 g standard weights.

3 Discussion

Multiple analytical techniques are required to cover the large number of components regulated in the ISO 14687 specifications. In the table below, we reported for each compound or group of compounds, the detection limit we can reach with the method (including sampling and analysis) mentioned together with possible developments in order to improve the detection limits and reduce the number of analyses required. The limits of detection (LOD) are expressed in ppm unless specified.

Compounds	Current situation				Possible developments			
	LOD	Analysis Method	Sampling method	LOD	Development	Analysis Method	Sampling method	LOD
Water	5	OFCEAS	In gas cylinders and 10 times dilution in nitrogen	30	Acquire an instrument as Proceas Multigas	OFCEAS	in pressurized cylinder	5
Total hydrocarbons compounds as methane basis	2	GC/FID +ev. TD/GC/MS	Gas bags +ev. on adsorbent C300	2	-	-	-	-
Oxygen	5	GC/TCD with He as carrier gas	Gas cylinders	30 (but coeluates with Argon)	-	-	Evaluate methods in order to avoid contamination with air	-
Helium	300	GC/TCD with H2 as carrier gas	Gas bags or gas cylinders	300	-	-	-	-
Nitrogen	100	GC/TCD with He as carrier gas	Gas cylinders	100	-	-	-	-

Argon	100	GC/TCD with He as carrier gas	gas cylinders	100	-	-	-	-
Carbon dioxide	2	GC/TCD with He as carrier gas	gas cylinders	2	-	-	-	-
Carbon monoxide	0,2	GC/TCD	Gas bags or gas cylinders	5	Acquire an instrument as Proceas Multigas	OFCE AS	in pressuri zed cylinder	
Total sulphur compounds	0,004	TD/GC/MS	on adsorbent	0.02 0 exkl. H ₂ S	Acquire an instrument as Proceas Multigas	OFCE AS	in pressuri zed cylinder	0,00 4 (H ₂ S)
Formaldehyde	0,01	HPLC/UV- Vis	DNPH cartridge	0,01	Acquire an instrument as Proceas Multigas	OFCE AS	in pressuri zed and treated gas cylinder	0,01
Formic acid	0,2	Ion exclusion chromatography	Purge and trap in alkaline buffer solution	0,2	-	-	-	
Ammonia	0,1	HPLC/conductivity detector	Silica gel treated tube	0,1	Acquire an instrument as Proceas Multigas	OFCE AS	in pressuri zed and treated gas cylinder	0,1
Total halogenated compounds (excl. HCl and Cl ₂)	0,05	TD/GC/MS	Carbotrap 300	0,05	-	-	-	
Particulate concentration	1 mg/kg	-	-	-	Method development needed	-	-	-

4 Price

Today the price for refueling hydrogen is often 9 EUR/kg (84,4 SEK) by the refueling station. This price is set by companies providing hydrogen compressed to 700 bars. The cost for compression, storage and dispensing (CSD) is included and this adds about 1,00-2,80 dollar per kg hydrogen (8,4-23,6 SEK per kg) and depends on the delivery system and the construction of the HRS (NREL, G. Parks, R. Boyd, J Cornish and R. Remick, 2014). The companies owning the HRS, needs to either produce hydrogen or buy hydrogen from an industrial production. Also the cost for the investment of the Hydrogen Refueling station (HRS) needs to be covered and today the HRS is expected to be profitable, but only after several years (Heiko Ammermann, 2013). The first investments need some sort of support or at least economical risk sharing between partners. This report is focused on the production price, before the costs for CSD occurs in the system and without any costs for return of investment in an HRS.

Some chemical industries in Sweden get hydrogen as a rest stream when producing their main products. In some locations the hydrogen is burned and used for heat and in some it is burned over the roof and not used at all. This is a resource that could be used in a better way, but in order to be able to use it for vehicles, it needs to be cleaned from toxic substances and also transported to an HRS. Depending on the production the hydrogen contains different substances and the step for cleaning could differ between industries. The easiest streams to clean are from chloralkali industry, but there are also other larger streams available in Sweden.

In Stenungsund for instance, there is an existing hydrogen network in the ground used by several industries in the Chemical Cluster. Some of the hydrogen comes from chloralkali processes but most of it has its origins in natural gas. Some industries use this hydrogen, but most of it is burned and used for heating. There is also a question of greening this facility and use bio-methane instead. In this case the cost scenario could change, but instead you get a green product. The issue to produce green hydrogen and how to certify this to Swedish standards (that today not exists) needs to be investigated more and may be the course for a whole project. It has also been discussions about scaling up the facility, then the streams will dramatically be larger than today. Today the mass of the hydrogen stream is 6 000 tonnes yearly and this could provide 40 000 – 50 000 fuel cell cars due to the report “Förstudie om vätgasanvändning i Stenungsund” written by, Vätgas Sverige.

In Sundsvall t1/5 of the hydrogen is just let out and not used at all. This stream could refuel 3 000 cars due to calculations made in the report “Vätgasinfrastruktur för transporter i svenska regioner”, written by Sweco, For this hydrogen the business case may be easier to set up.

4/5 is used internally for heating, but could be used better if combined with Fuel Cells or made into another product. If this hydrogen is substituted by another heat source, the hydrogen could supply 15 000 fuel cell cars.

It is very hard to estimate a price for this Swedish left-over-hydrogen; the price should involve some kind of estimated basic value, cost for cleaning, cost for transport, cost for measurements and also a profit. On top of that taxes could be added, depending on what we guess about the political set-ups for clean fuels in the future.

To estimate the price for hydrogen for vehicles produced from left-over-hydrogen from industry, a small scale interview with different actors on the Nordic market was made. The persons chosen sits on the right position in the company to be able to estimate this price. One actor comes from another Nordic country and they are today producing hydrogen from left-over-streams. Because these persons have great knowledge of their streams, their internal costs (that could not be shared with us) they were asked to estimate a price based on the discussion above. The cost for producing the hydrogen is of course a business secret, but without mentioning sources or companies we can get an approximate price for the product. This price is estimated without energy taxes and can be seen as a benchmark for further calculations and estimations. The estimated price for left-over-hydrogen from industry upgraded to vehicle standard is 15-20 SEK/kg.

To put this price in a context, it can compare to the price of hydrogen produced from electrolysis. In the recent study from Fuel Cells and Hydrogen Joint Undertaking called "Study on development of water electrolysis in the EU" some calculations are presented for different countries and situations. According to this report, the price depends on the levies/charges/taxes of the country, network charges and electricity price, if the electrolyzer is set on 100% load or run by price minimization. The price could also be adjusted if the electrolyzer could be used for balancing services which therefore could lead to some price reduction. Another case would be if the electrolyzer was installed off-grid, today it would require a higher cost but 2030 the report is estimating a lower cost for off-grid-cases.

In this case, we base the price below on the assumption, that of the countries examined in the report, Finland is the most similar to Sweden, because of the Nordic market that has a strong interconnected electricity grid. Also we assume that the electrolyzer is run on full load or on price minimization.

These numbers are taken from Fig 6, 7, 8 on pages 17-18 in the report.

Price for clean hydrogen for PEMFC	
Hydrogen from electrolysis 2012	38-47SEK /kg
Hydrogen from electrolysis 2030	33-38 SEK/kg
Left over hydrogen from Swedish Chemical Industry (estimation from interviews)	15-20 SEK/kg

As can be seen, this could be a very interesting opportunity for hydrogen production, because it could be produced to a low cost. Before this should be decided, every facility needs to make an own business model to see the whole cost scenario.

To compare the hydrogen production from electrolyze or from left-over hydrogen for the purpose to advice which production method that is preferable cannot be done. Today the

hydrogen is mainly produced by steam reforming with origins from natural gas. This is cheap, but not green. The end consumer for this hydrogen is the oil industry and the incentive for buying green hydrogen does not exist in this industry. If we talk about a sustainable future, electrolysis is of course the cleanest way of making hydrogen if green electricity is used. When we talk about rest-streams from industry, some part of it may be green and some grey. We could see a greening of these streams, but today there is no regulation on how to estimate how green a hydrogen production is. We could see on the regulation for how green electricity is reviewed and also on how biogas-certificate is assessed, but more work needs to be done in this area. If we however see a valuable stream that is not used, the simple most sustainable thing to do is to use it or in some cases use more than the heat value.

5 Conclusion

Developing these methods was more challenging than we thought when the project started. This project has resulted in that SP now by certainty can carry out most of the required measurements required by the standard ISO14687-2. The remaining tests could be performed in cooperation with similar institutes in other countries. The required limits for this standard are probably lower than necessary meaning that a higher limit could be acceptable and still not harming the fuel cells. For a continuation of this work, it could be interesting to prove this hypothesis. The fact that it is mentioned in the standard that the limits could be readjusted, implies that it was already known when the standard was written that the detection limits are too low. There is still more development to be done in this area and in order to lower the detection limits further an investment of 500 kSEK in advanced instruments that will be dedicated for this purpose only must be done.

Among the fourteen parameters to verify in the standard ISO14687-2, nine parameters; total hydrocarbons compounds as methane basis, helium, nitrogen, argon, carbon dioxide, formaldehyde, formic acid, ammonia, and total halogenated compounds (excl. HCl and Cl₂) can be analyzed at the required detection limit. Two parameters; water and carbon monoxide can be analyzed but with a higher detection limit than required in the standard. The particulate concentration will require a further method development on an instrument already available and two parameters (oxygen and total sulfur compounds) will require purchasing an analytical instrument and developing an analytical method.

In the current situation, at least 10 different analyses will need to be performed to cover 12 of the 14 parameters to verify. In the same way, the hydrogen will need to be sampled in at least 7 different methods including filling gas bags and cylinders, sampling the gas on different adsorbent tubes, bubbling the gas in impingers etc. In the circumstances, sampling and analyses will be time consuming and consequently expensive. A solution to reduce the number of analyses and sampling methods is to use an instrument specific for the analysis of impurities in hydrogen. A possible instrument is a gas analyzer based on the principle of the optical feedback cavity enhancement absorption spectroscopy allowing the simultaneous analysis of at least four components at the required detection limit. The compounds that can be analyzed with this technique are methane, ammonia, carbon monoxide, carbon dioxide, formaldehyde, hydrogen sulphide, oxygen and water.

Some of the detection limits specified in ISO14687-2 are very challenging. If improvement in the performance of fuel cells for transportation application goes towards more tolerant materials, then setting less restrictive detection limits may be possible.

The price estimation for left-over hydrogen from rest streams from Swedish chemical industries seems very promising and should be investigated further. Also to optimize the energy efficiency of the systems, to minimize the environmental effects, these valuable resources could be used wiser than today. The prize for left-over hydrogen was estimated to 15-20 SEK/kg compared to hydrogen from electrolyze that is about 40-50 SEK/kg. This estimation must be carefully evaluated into a full business model, before any advices

on investments could be done. The figures mean that there is a potential to lower the fuel cost for fuel cell cars if the chemical industry in Sweden can find a sustainable business model for this.

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ASTM D7651-10: Standard test method for gravimetric measurement of particulate concentration of hydrogen fuel

ASTM D7634-10: standard test method for visualizing particulate sizes and morphology of particles contained in hydrogen fuel by microscopy.

ASTM D7653-10 Standard Test Method for Determination of Trace Gaseous Contaminants in Hydrogen Fuel by Fourier Transform Infrared (FTIR) Spectroscopy

ANALYSIS OF HYDROGEN QUALITY

Kvalitet på ren vätgas ställs enligt de två standarderna: ISO 14687-2 och ISO 14687-3.

Projektet valde att fokusera på standarden ISO 14687-2 då denna kräver högre renhet och har större krav på noggrannhet. Rapporten presenterar de mätmetoder som utvecklats för att kunna utföra mätningar enligt standarden. En utmaning var att denna standard har extremt låga detektionsnivåer, vilket gör att det krävs extrema åtgärder för att nå dessa gränser. I slutet av rapporten återfinns en kort diskussion om pris för vätgas sprunget ur industrins överskottsvätgas. Siffrorna är en fingervisning att det finns potential att sänka bränslekostnaden för bränslecellsbilar i Sverige.

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