



D7.4

RESULTS FROM H₂-FUEL GAS QUALITY MONITORING AT VARIOUS HRSS IN OSLO AREA

Final Version

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ACRONYMS AND ABBREVIATIONS

BOP	Balance Of Plant
BTL	Biomass-to-Liquid
CTL	Coal-to-Liquid
GTL	Gas-to-Liquid
FC	Fuel Cell
FTIR	Fourier-Transform Infrared Spectroscopy
GC	Gas Chromatography
GHG	Greenhouse Gas
GT	Gas Turbine
H ₂	Hydrogen
ICE	Internal Combustion Engine
LBST	Ludwig-Bölkow-Systemtechnik
LCA	Life-Cycle Assessment
LOD	Limit Of Detection
LOQ	Limit Of Quantification
MEA	Membrane Electrode Assembly
MS	Mass Spectrometry
N ₂	Nitrogen
PEM	Polymer Electrolyte Membrane
PM	Particulate Matter
PSA	Pressure Swing Adsorption
QA	Quality Assurance
QC	Quality Control
SDD	Sustainability Due Diligence
WtT	Well-to-Tank
WtW	Well-to-Wheel
yr	Year

EXECUTIVE SUMMARY

Hydrogen fuel quality control has been conducted for three Hydrogen Refuelling Stations in the Oslo area. The sampling of gas and particles were conducted by SINTEF. Sampling was limited to 350 bar for several reasons. The samples were analysed by a subcontracting laboratory, fully capable of performing quality control in accordance with prevailing standards on hydrogen fuel for fuel cells. The main results are summarized in the table:

Constituent	Tolerance	Porsgrunn	Gaustad	Økern
Total Hydrocarbons (C1 basis)	2	0.80	0.035	0.049
Nitrogen	100*	2800	7.7	< 5
Argon	100*	0.77	< 0.5	1.9
Carbon Dioxide	2	3.3	< 0.5	< 0.5
Carbon Monoxide	0.2	0.0097	0.0047	0.0010
Total Sulphur	0.004	0.00022	< 0.0001	< 0.0001
Total Halogenates	0.05	< 0.002	0.0042	0.014
Particulate Concentration	1 mg kg ⁻¹	0.042	0.14	0.21
Number of particles		11	48	7
Hydrogen Fuel Index	99.97 %	99.7195%	99.9992 %	99.9998%

* Sum of N, Ar should be less than 100 ppm.

One HRS failed the hydrogen QC on several criteria. For one of HRSs a large deviation from the nitrogen tolerance limit resulted in a significant reduction in the fuel index. All particle concentrations were lower than the tolerance limit, although each and every particle had a larger diameter than the accepted 10 µm.

In addition to the subcontracted analysis, SINTEF used the opportunity to evaluate the feasibility of FTIR and GC-MS for fuel quality analysis. Because of the limited number of impurities found in the samples, evaluation of feasibility of these techniques for an extended range of impurities was difficult. It is suggested that a pre-concentration techniques is applied to sample before analysis.

1 INTRODUCTION AND MOTIVATION

As PEM technology is still trying to attain its durability targets, the tolerance for potentially degrading conditions is strict. It is well established that the tolerance for sulfur for platinum catalysts are limited as the element adsorbs irreversibly on the surface. In PEM fuel quality standards, this is reflected in a maximum tolerance level of 4 ppb total sulphur. A list of tolerance levels for impurities is shown in Figure 1.

Characteristics		Fuel Cell*	ICE
		ISO/TS14687-2 Type 1, Grade D	ISO14687:1999 Type 1, Grade A
Hydrogen Fuel Index		99.99 %	98 %
Impurities	Total hydrocarbons (C ₁ basis)	2 ppm	100 ppm
	Water(H ₂ O)	5 ppm	1,900 ppm
	Oxygen(O ₂)	5 ppm	
	He, N ₂ , Ar	100 ppm	
	Carbon dioxide (CO ₂)	2 ppm	-
	Carbon monoxide (CO)	0.2 ppm*	1 ppm
	Total sulfur compounds	0.004 ppm*	2.0 ppm
	Formaldehyde	0.01 ppm*	-
	Formic acid	0.2 ppm*	-
	Ammonia	0.1 ppm	-
Particulates	1 µg/L(10 µm max)	Not contain in an amount sufficient to damage	

*He now allowed at 300 ppm, total halogenates: 50 ppb

Figure 1 ISO Technical Standards for hydrogen fuel quality for PEM fuel cells and Internal Combustion Engines (ICE).

It is not difficult to foresee that hydrogen fuel quality control could be cost driving for hydrogen fuel production and delivery.

Whether the tolerance levels of inerts and water are too stringent could be debated, as these components are mainly associated with Balance of Plant (BOP) issues. It has also been argued that the nitrogen gas cross-over from an air-fed cathode is higher than the tolerance levels set in the standards.

In the case of sulphur, the impact on durability is unquestionable. Sulphur, in the form of a sticky gas such as H₂S, is almost impossible to quantify at a level of 4 ppb. Verification of the impact of 4 ppb H₂S fed to a fuel cell is also difficult to evaluate, as a significant part of the molecules would stick to the gas feed line and never reach the catalyst. For hydrogen produced from Steam Methane Reforming (SMR), Pressure Swing Adsorption (PSA) is always applied to purify hydrogen. It has been argued that for the adsorbents normally applied, the selectivity of H₂S is much higher than that of CO. Thus, breakthrough of H₂S should only be possible in the case of saturation of the PSA. This condition could, however, be evaluated by monitoring the CO concentration.

Currently, fuel quality standards do not reflect the hydrogen fuel feedstock. For onsite, electrolytically produced hydrogen fuel the sources of sulphur or halogenates are very limited. In the case of trucked-in hydrogen, using hydrogen fuel of various feedstocks might complicate the impurity situation at an HRS. It is expected that as more experience data from HRS of various feedstock is reported, it is possible to differentiate the standards.

A proposed approach to simplify the quality control of hydrogen fuel is by the use of canary constituents. By establishing well know correlations between impurities, the canary could be used as an indicator of impurities present in the fuel. Although thermochemical calculation has been performed to evaluate equilibria relevant for the composition of the gas, most of the co-variance between impurity species requires an experimental verification as they do not react with each other. It could be argued by taking feedstock into consideration, this might simplify the evaluation of co-varying species.

A pre-requisite for the use of canary constituents is that the co-variance between constituents is verified. This requires execution of quality control and collection of data from several HRS based on different feedstock. Another challenge for the evaluation of co-variance is the low concentration of some of these constituents. If the concentration is lower than the Limit of Quantification (LOQ), analytical error is high.

There are currently two approaches to pre-concentration of impurities in hydrogen fuel. The first approach reported by Papadias [Papadias 2010] was to use hydrogen selective membranes. By using a palladium membrane allowing for hydrogen permeation, the impurities are thus accumulated. The schematic setup is shown in Figure 2.

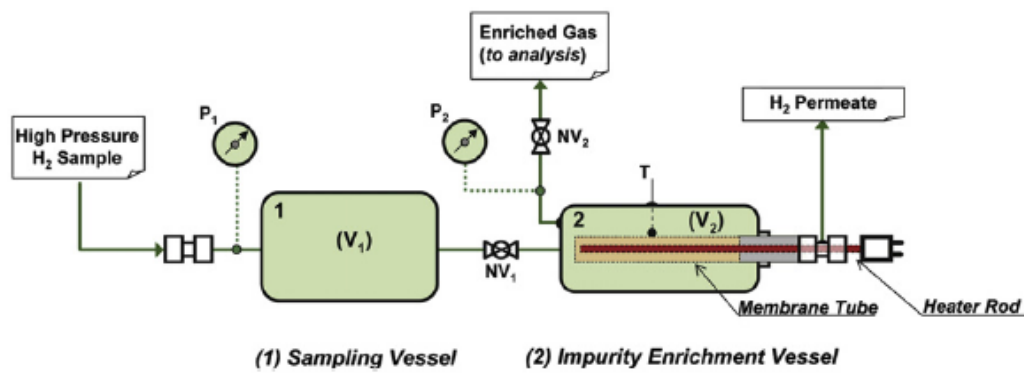


Figure 2 Pre-concentration of impurities by use of a hydrogen selective membrane.

Silicon lined sample vessels are commonly used for sampling of hydrogen fuel in order to avoid losses of "sticky gases" to the vessel walls. This is also a challenge for the hydrogen selective membrane: Not only is gas molecules lost to the membrane, they also block the hydrogen permeation rate. There is, however, possible to tailor the membrane alloying composition but also to use temperature to reduce membrane surface adsorption.

Figure 3 shows the result of enrichment experiments for five fuel impurity constituents.

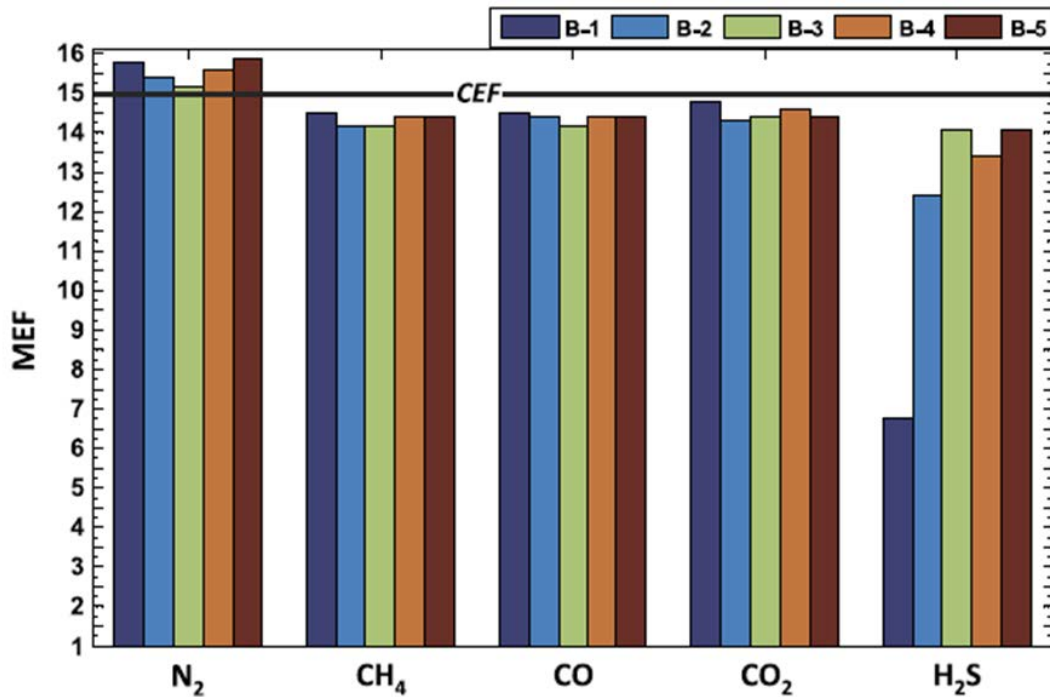


Figure 3 Enrichment experiment containing five fuel impurities with a Calculated Enrichment Factor (CEF) of 15. B-1 was performed at temperatures between 220-250 °C while B-2 to B-5 was performed at 270 °C.

can be pre-concentrated by a desorption step by reducing the pressure. The enrichment efficiency is a function of adsorbent type and the desorption pressure. For a given adsorbent, the enrichment efficiency is a non-linear function of the pressure ratio of adsorption and desorption pressure. Generally, an as-low-as possible desorption pressure is beneficial, but the volume and/or pressure requirements for the analytical techniques normally limits desorption from being close to ambient pressure.

As higher temperature decreases the efficiency of the adsorbent, compression work as well as head of adsorption must be minimized by slow pressurization. Enrichment efficiency is shown in Figure 4.

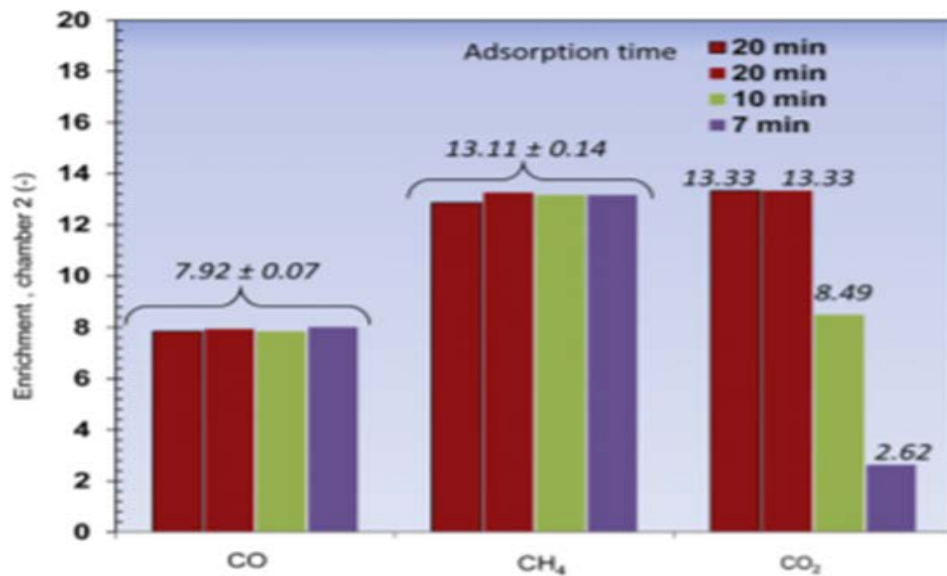


Figure 4 Enrichment factors for selected impurities for an experiment where a gas flow rate of 2.2 NLPM, adsorption at 75 atm and desorption at 2.42 atm.

ent factor is obtained for all constituents, enrichment factors vary with the adsorption affinity. Most impurities, hydrogen often being the exception, have higher affinities than hydrogen.

Both approaches have their pros and cons. The hydrogen selective membrane approach has the highest enrichment potential. The drawbacks are the long sampling time required as well as membrane temperature control requirement. The PSA approach is more limited when it comes to enrichment potential. However, tenfold enrichment can be obtained in less than 20 minutes.

Neither work discusses the applicability of pre-concentration with respect to the full constituent list. From the Dynamis EU project [Besancon 2009], more information on impurity adsorption strengths are available. These data are shown in Figure 5.

RELATIVE STRENGTH OF ADSORPTION

+	++	+++	++++
H ₂	Ar	CO	C ₃ H ₈
	O ₂	CH ₄	C ₄ H ₁₀
	N ₂	CO ₂	C ₅ +
		C ₂ H ₆	H ₂ S
		C ₂ H ₄	NH ₃
		C ₃ H ₈	H ₂ O

- Strength

+ Strength

Alumina
Carbon Prefilter
Activated Carbon
Molecular Sieve

Figure 5 Impurity adsorption strengths for impurities for a selection of adsorbents.

Deliverable D7.4

SINTEF's approach to simplification of hydrogen fuel QC has so far been to evaluate a possible reduction in the number of analytical techniques required to perform QC in accordance with the standards proposed. The two analytical techniques that could potentially cover most analytes are FTIR and GC-MS. The goal for task 7.2.1 of the H2Moves Scandinavia project was to ensure the quality hydrogen fuel supply. SINTEF approached this goal by subcontracting a laboratory capable of performing QC in accordance with standards. In addition, SINTEF collected samples to be analysed with FTIR and GC-MS. In this way, the findings by SINTEF could be reference to the results from the professional laboratory.

Another approach of interest for simplification of hydrogen fuel control QC is evaluation of co-variance between impurities in the fuel. By establishing knowledge of correlation between impurities, although most likely hydrogen feedstock dependent, QC could be simplified. The main challenge for this strategy is that the co-variance study is challenging due to low concentration levels, often near or below Limit of Quantification (LOQ). With low analytical performance, the co-variance study requires a large population of samples in order to establish knowledge about known correlations. One possible approach to remedy this is to make use of pre-concentration techniques in order to simplify analysis.

2 SAMPLING

2.1 Methodology

Sampling instrumentation for gases at 700 bar was not commercially available at the start of the project activities for Task 7.2.1. In collaboration with Hochdruck Reduziertechnik GmbH, a sampling unit was designed. The unit is shown in Figure 6.

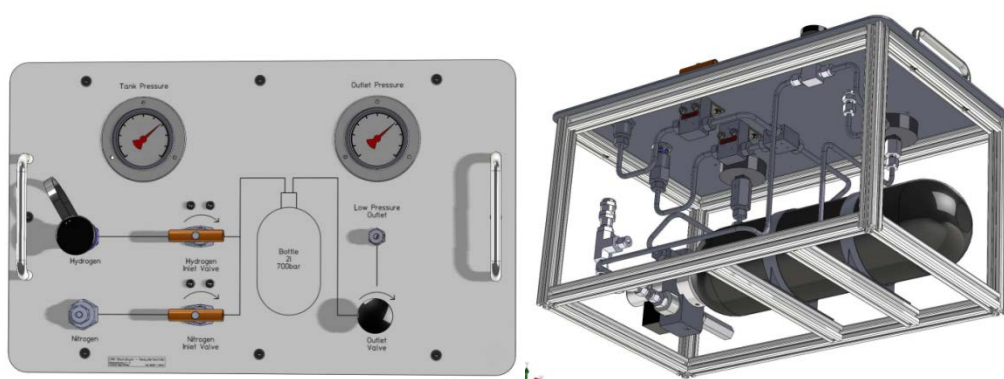


Figure 6 High-pressure sampling unit.

Although a contract was made for 12 weeks delivery time, due to technical difficulties with components during pressure verification at approximately 1000 bar. It was then resolved to rent 350 bar gas sampling instrumentation from Smart Chemistry. The unit is shown with a 700 bar receptacle in Figure 7.

For sampling of particles, a 700 bar sampling unit was available for purchase, but not for rent, from Wenger Engineering, GMBH. The purchase of gas sampling unit(s) was not an option given the task budget for direct expenses. It was therefore resolved to rent particle sampling units from Smart Chemistry. The PSU is shown in Figure 7.

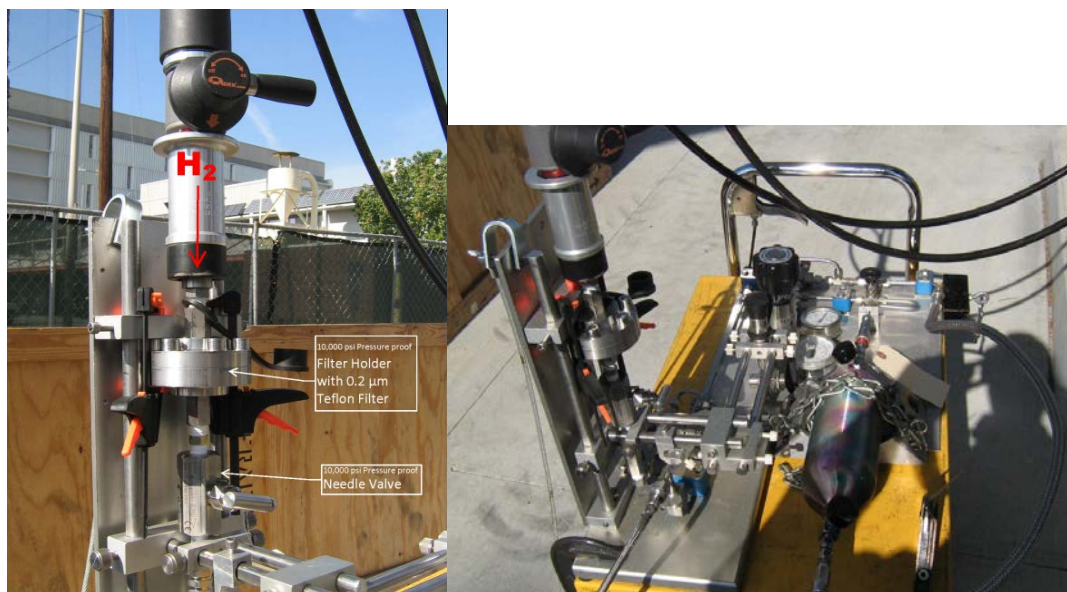


Figure 7 Smart Chemistry Particle Sampling Unit (left) and gas sampling instrumentation (right).

As sampling was to be conducted at 350 bar, the available fuel cell vehicles could not be used as sink for the sampled hydrogen. The hydrogen fuel was therefore vented through two ½” tubes. This approach also made manual override of the HRS safety system required. With no pressure detected at the nozzle (like in a completely empty hydrogen gas powered vehicle or at ambient condition) the HRS safety system shuts down the hydrogen flow.

2.1.1 HRS Økern: 2012-11-06

The HRS at Økern has Hydrogen fuel from alkaline electrolysis feedstock. It is not produced at site, but trucked-in from Rjukan in Norway. The HRS has separate nozzles for 350 and 700 bar. As particle sampling instrumentation was only available for 350 bar, all sampling was performed at this nozzle. The HRS operator calculated the mass of hydrogen from the buffer tank pressure drop. However, for particle sampling, the requested 2 kg of fuel turned out to be 0.95 kg. Gaseous samples were collected in silicon lined containers at 70 bar for subsequent analysis at Smart Chemistry and at SINTEF. All hydrogen sampled through gas containers and particle filters was vented to air.

2.1.2 HRS Gaustad: 2012-11-06

The HRS at Gaustad (SINTEF) delivers hydrogen fuel from onsite electrolytic production. It is only equipped with a 700 bar nozzle so the operators from H2 Logic had to reduce delivery pressure to fit 350 bar certified sampling instrumentation. The operators used software to gauge 2.1 kg of hydrogen through the PSU. As there were no 700 bar nozzle available for the gas sampling unit, an additional 0.3 kg of hydrogen was passed through the PSU connected upstream the gas sampling unit. The gas samples were collected in silicon lined containers at 70 bar for subsequent analysis at Smart Chemistry and at SINTEF. All hydrogen sampled through gas containers and particle filters was vented to air.

2.1.3 HRS Herøya: 2012-11-07

The HRS on Herøya, Porsgrunn delivers by-product hydrogen from chlorine-alkaline industry at 350 bar. The operators used software to gauge 2.4 kg of hydrogen through the PSU. Gaseous samples were collected in silicon lined containers at 70 bar for subsequent gas analysis. All hydrogen sampled through gas containers and particle filters was vented to air.

2.1.4 HRS Økern: 2013-01-16

SINTEF received the 700 bar sampling equipment from Germany after the QC sampling campaign was over. Even if the task activity was ended in December 2013, SINTEF arranged for one sampling campaign in January 2013 in order to make use of the sampling unit. Upon arrival at HRS Økern, SINTEF representatives were informed that the instrument specification was insufficient for sampling at 700 bar. Apparently, the pressure testing at around 1025 bar was insufficient for this application. The reason given was that the initial pressure pulse at the 700 bar nozzle could be higher due to temperature effects. SINTEF was therefore unable to sample hydrogen fuel at 700 bar as planned.

Detailed description is given in Appendix 3.

3 RESULTS FROM HYDROGEN FUEL QC

3.1 Results from SmartChemistry

The full report from SmartChemistry is given in Appendix 1. The main results and results of interest are shown in Table 1.

Table 1: SmartChemistry QC results. All results in ppmv unless specifically noted. Tolerance levels are in accordance with SAE J2719. Results in red are in conflict with the standard.

Constituent	Tolerance	Porsgrunn	Gaustad	Økern
Total Hydrocarbons (C1 basis)	2	0.80	0.035	0.049
Nitrogen	100*	2800	7.7	< 5
Argon	100*	0.77	< 0.5	1.9
Carbon Dioxide	2	3.3	< 0.5	< 0.5
Carbon Monoxide	0.2	0.0097	0.0047	0.0010
Total Sulphur	0.004	0.00022	< 0.0001	< 0.0001
Total Halogenates	0.05	< 0.002	0.0042	0.014
Particulate Concentration	1 mg kg ⁻¹	0.042	0.14	0.21
Number of particles		11	48	7
Hydrogen Fuel Index	99.97 %	99.7195%	99.9992 %	99.9998%

*The sum N and Ar required to be less than 100 ppm.

The individual results are discussed in the following paragraphs.

3.1.1 Total Hydrocarbons

The total hydrocarbon level in Porsgrunn was higher than for the other HRSs, although well below the tolerance level. The main hydrocarbons found were Methane (0.39 ppmv) and Ethane (0.19 ppmv). Traces of acetone, heptane and propane were also reported.

3.1.2 Nitrogen

The nitrogen level of 0.28 % found in Porsgrunn lowers the fuel index well outside the tolerance level of 99.97 %.

3.1.3 Argon

For the HRS in Porsgrunn, a significant concentration of 0.77 ppmv was found. The sum of argon and nitrogen should be less than 100 ppm.

3.1.4 Carbon Dioxide

Carbon dioxide was only detected at Porsgrunn at a level slightly higher than the tolerance limit.

3.1.5 Carbon Monoxide

All three HRSs were well within the tolerance limit. Again, the HRS in Porsgrunn has the highest level, as the case is for other impurities containing carbon listed in Table 1.

3.1.6 Total Sulphur

For Porsgrunn a total sulphur level of 0.22 ppbv was recorded. This is well below the tolerance limit of 4 ppbv. 0.18 ppbv of this was found in the form of carbon disulphide. The rest was made up by carbonyl sulphide.

For Gaustad and Økern, hydrogen sulphide and carbonyl disulfide were detected in concentrations just above the detection limit of 0.02 ppbv.

3.1.7 Total Halogenates

For the Porsgrunn HRS, with hydrogen sourcing from chlor-alkali electrolysis, this result was of particular interest. The results show that all three HRSs are well below the 50 ppbv tolerance limit. For both Gaustad and Økern, traces of tetrachlorohexafluorobutane were found. These $C_4Cl_4F_6$ isomers are frequently found and are believed to be sourcing from stainless steel tubing [Hsu 2012].

3.2 Particles

For all HRSs, the particle concentration was found to be well below the tolerance limit of 1 mg kg⁻¹. The HRS at Økern had the highest concentration with 0.21 mg kg⁻¹ while the lowest result was found for Porsgrunn with 0.042 mg kg⁻¹. Both prevailing standards, SAE J2719 and ISO/TS 14687-2 sets maximum allowable particle to be less than 10 µm. All particles counted by SmartChemistry is larger than 10 µm.

For the Gaustad HRS visual inspection of the filter revealed several oily stains, green of colour. The total particle count was 48 and the largest particle found was 1.4 mm.

For the HRS at Porsgrunn, 11 discrete particles was found. The largest particle was measured to be 0.22 mm.

For the Økern HRS seven particles were found. The largest two, at 0.47 and 0.51, appeared in polarized light to be of a metallic nature.

3.3 Results from SINTEF

3.3.1 Long Path FTIR

An extensive study on applicability of FTIR to hydrogen has been done by SINTEF. The scope of this work is too extensive for this report, so only the analytical results are presented. The full-length MEMO can be found in Appendix 2.

As the study has been based on real samples, collected from three HRSs in the Oslo area, the study is limited to the species found in these samples. This does not mean that FTIR could be applied to other impurities listed in the hydrogen fuel for fuel cells standards. In order to evaluate the full potential of FTIR, synthetic gas compositions have to be analysed. Optionally, pre-concentration of impurities would also extend the list of quantifiable impurities in the fuel.

The result from the SINTEF FTIR analysis is shown in Table 2.

Table 2 Analytical results from Long Path FTIR quantification of impurities.

Analyte	CO	CO ₂	H ₂ O	CH ₄	C ₃ H ₈	C ₄ H ₁₀	Heptane	Acetone
<i>LOD</i>	<i>5 ppb</i>	<i>100 ppb</i>	<i>1 ppm</i>	<i>5 ppb</i>	<i>10 ppb</i>	<i>20 ppb</i>	<i>10 ppb</i>	<i>40 ppb</i>
Gaustad	12 ppb	< 500 ppb	< 1 ppm	8 ppb	<10 ppb	< 20 ppb	< 10 ppb	< 40 ppb
Porsg.	21 ppb	3150 ppb	< 1 ppm	22 ppb	45 ppb	460 ppb	< 40 ppb	< 80 ppb
Økern	11 ppb	< 500 ppb	< 1 ppm	5 ppb	<10 ppb	< 20 ppb	< 10 ppb	< 40 ppb

The estimated detection limits are better than required by the fuel standards. Especially the LOD for CO is very good. This is important with respect to the fact that CO has been proposed as a potential canary constituent.

The SINTEF results verify the trends in SmartChemistry data for several impurities. The high CO₂ level found for the Porsgrunn HRS is this way verified. The trend for CO is also similar for the two analyses, although the concentration levels are slightly different. The higher level of hydrocarbons is also verified by the SINTEF analysis.

3.3.2 GC-MS

SINTEF approach to QC simplification has been to cover the list of impurities by as few analytical techniques as possible. An inherent limitation to IR spectroscopy is the ability to analyse homonuclear molecules (ie. N₂, O₂, Cl₂ etc.) as well as helium and argon. One of the more versatile instruments available to SINTEF was a GC-MS from agilent: 5975T LTM. Upon adaptation to the standard method used and developed by Smart Chemistry (ASTM D7649-10), several challenges were faced:

- Due to laboratory safety regulations, the GC-MS was not allowed to use hydrogen as carrier gas
- Multivalve, pressurized sample and standard injection instrumentation was not available
- Standards with hydrogen as balance gas could not be made by gas mixing in the laboratory

As the proposed instrument setup is quite complex, SINTEF attempted a simplified approach. Using a gas tight syringe for sample injection is not feasible when trying to quantify components in air. However, a manual multivalve with a 100 µL sample loop made sample injection from the canisters possible after pressure reduction.

Quantification was set up for helium carrier gas and for gas mixtures balanced by helium. Examples on calibration curves are given in Figure 8.

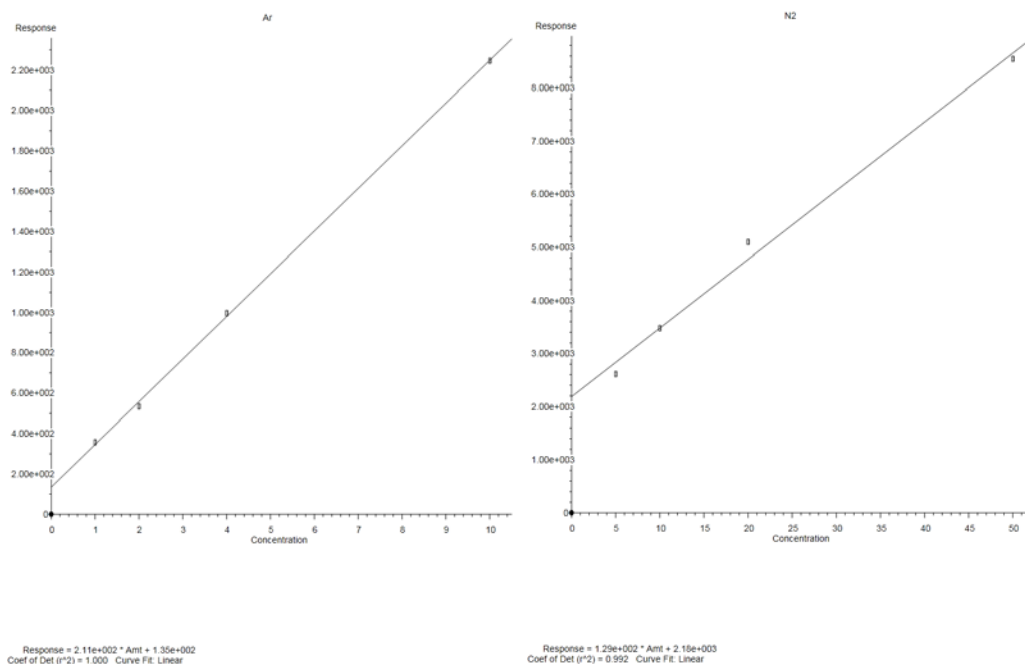


Figure 8 GC-MS calibration curves for Ar (left, 1,2,4 and 10 ppmv) and N₂ (right, 5, 10, 20 and 50 ppmv).

The curves show that there is scope for analysis of these impurities in the low ppmv concentration range.

After analysing samples from the three HRSs, the results were compared with those from SmartChemistry. The correlations were not good. After comparing the calibrations with samples of hydrogen 5.0, it became clear that the ionization of analytes is significantly different in hydrogen and helium gas.

A direct comparison between hydrogen 5.0 and the Porsgrunn samples is shown in Figure 9.

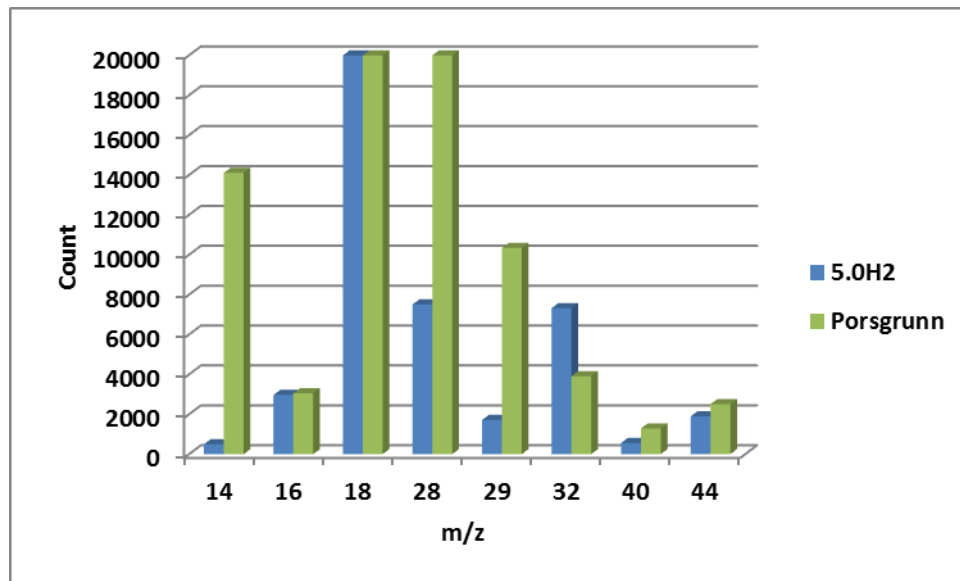


Figure 9 Comparison between H2 5.0 and Porsgrunn sample.

The high nitrogen level found in the Porsgrunn sample is seen in contrast to the hydrogen 5.0 sample (m/z 14, 29 and partly 28). A lower oxygen level is indicated by m/z 32 whereas argon (m/z 40) and carbon dioxide (m/z 44). The water concentration appears to be fairly similar for the two samples (m/z 18).

SINTEF has experienced that for the application of GC-MS to the fuel quality assessment, hydrogen carrier and gas balancing is required. The current investigation used a limited mass range for investigation (10-50 m/z). In order to investigate the full potential of the GC-MS, a wider mass range should be applied in order to evaluate heavier mass fragments.

4 LITERATURE

- [Papadias 2010] International Journal of Hydrogen Energy, 35 (2010) 12480-12490.
- [Papadias 2012] International Journal of Hydrogen Energy, 37 (2012) 14413-14426
- [Besancon 2009] International Journal of Hydrogen Energy, 34 (2009) 2350-2360.
- [Hsu 2012] International Journal of Hydrogen Energy, 37 (2012) 1770-1780.

5 LIST OF APPENDICES

1. SmartChemistry analytical report
2. SINTEF report on FTIR analysis of fuel samples
3. SINTEF note on high temperature (700 bar) sampling

Report Summary

	Porsgrunn		Gaustad		Økern	
	12SIN001-01		12SIN001-02		12SIN001-03	
Constituent	SAE Limits ($\mu\text{mol/mol}$)	Smart Chemistry Detection Limits ($\mu\text{mol/mol}$)	Concentration ($\mu\text{mol/mol}$)	Concentration ($\mu\text{mol/mol}$)	Concentration ($\mu\text{mol/mol}$)	Analytical Method
Water	5	1	< 1	< 1	< 1	ASTM D7649-10
Total Hydrocarbons (C₁ Basis)	2		0.80	0.035	0.049	
Methane		0.001	0.39	0.022	0.033	ASTM WK34574
Acetone		0.001	0.0010	0.0012	0.0018	ASTM WK34574
Ethane			0.19	0.0047	0.0027	
Heptane			0.0011	< 0.001	< 0.001	
Propane			0.0099	< 0.002	< 0.002	
Isopropyl Alcohol			< 0.001	< 0.001	0.0016	
Oxygen	5	2	< 2	< 2	< 2	ASTM D7649-10
Helium	300	10	< 10	< 10	< 10	ASTM D1946
Nitrogen, Argon	100					
Nitrogen		5	2800	7.7	< 5	ASTM D7649-10
Argon		0.5	0.77	< 0.5	1.9	ASTM D7649-10
Carbon Dioxide	2	0.5	3.3	< 0.5	< 0.5	ASTM D7649-10
Carbon Monoxide	0.2	0.0009	0.0097	0.0047	0.0010	ASTM WK34574
Total Sulfur	0.004	0.0001	0.00022	< 0.0001	< 0.0001	ASTM D7652-11
Hydrogen Sulfide		0.00002	< 0.00002	0.000021	0.000024	ASTM D7652-11
Carbonyl Sulfide		0.00002	0.000034	< 0.00002	< 0.00002	ASTM D7652-11
Methyl Mercaptan		0.00002	< 0.00002	< 0.00002	< 0.00002	ASTM D7652-11
Carbon Disulfide		0.00002	0.00018	0.000029	0.000023	ASTM D7652-11
Formaldehyde	0.01	0.001	< 0.001	< 0.001	< 0.001	ASTM WK34574
Formic Acid	0.2	0.001	< 0.001	< 0.001	< 0.001	ASTM WK34574
Ammonia	0.1	0.04	< 0.04	< 0.04	< 0.04	ASTM WK34574
Total halogenates	0.05			0.0042	0.014	
Chlorine		0.004	< 0.002	< 0.004	< 0.004	ASTM WK34574
Hydrogen Chloride		0.001	< 0.002	< 0.002	< 0.002	ASTM WK34574
Hydrogen Bromide		0.001	< 0.001	< 0.001	< 0.001	ASTM WK34574
Organic Halides (32 compounds in red and bold listed in "Other Hydrocarbons"). Smart Chemistry limit is for each individual organic halide.				0.0042	0.014	
C₄Cl₄F₆		0.001	< 0.001	0.0042	0.014	ASTM WK34574
Particulate Concentration	1mg/Kg		0.042 mg/kg	0.14 mg/kg	0.21 mg/kg	ASTM D7651-10
Particulates Found & Size (ASTM D7634-10) - Images of particulates and pinholes found are in Table 1			Particulate 1 – 0.18mm Particulate 2 – 0.11mm Particulate 3 – 0.15mm Particulate 4 – 0.13mm Particulate 5 – 0.15mm Particulate 6 – 0.24mm Particulate 7 – 0.13mm Particulate 8 – 0.22mm Particulate 9 – 0.14mm Particulate 10 – 0.13mm Particulate 11 – 0.11mm	There are total 48 particulates found with their sizes over 0.03mm. The particulate size distribution is listed below. # of Particulate @ 0.12mm – 1 # of Particulate @ 0.07mm – 3 # of Particulate @ 0.06mm – 1 # of Particulate @ 0.05mm – 6 # of Particulate @ 0.04mm – 22 # of Particulate @ 0.03mm – 15	Particulate 1 - 0.47mm Particulate 2 - 0.51mm Particulate 3 – 0.06mm Particulate 4 – 0.26mm Particulate 5 – 0.30mm Particulate 6 - 0.15mm Particulate 7 – 0.41mm	
Hydrogen Fuel Index The hydrogen fuel index is the value obtained when the amount of aggregate impurities, as, expressed as percent ($\mu\text{mole}/\mu\text{mole}$), is subtracted from 100%. (Section 3.5 of SAE J2719) The particulate is NOT included in Hydrogen Fuel Index.			99.7195%	99.9992%	99.9998%	

Analytical Data for Non-Hydrogen Gaseous Constituents

The analytical data is tabulated for each non-hydrogen gaseous constituent, required by Chevron, listed in the sequence below. The analytical data includes calibration standards, detection limit study, sample analysis, spike, or duplicate.

Non-Hydrogen Gaseous Constituents	
1.	Water
2.	Total Hydrocarbons
2.1	Methane
2.2	Other Hydrocarbons
3.	Oxygen
4.	Helium
5.	Nitrogen
6.	Argon
7.	Carbon Dioxide
8.	Carbon Monoxide
9.	Sulfur
9.1	H ₂ S (Only); COS, CH ₃ SH & CS ₂ (Not Required)
9.2	Total Sulfurs, H ₂ S, COS, CH ₃ SH, CH ₃ CH ₂ SH, CS ₂ , (CH ₃) ₃ CSH and Tetrahydrothiophene are analyzed (Not Required)
10.	Formaldehyde (Not Required)
11.	Formic Acid (Not Required)
12.	Ammonia
13.	Total Organic Halogenates (Not Required)
13.1	Chlorine (Not Required)
13.2	Hydrogen Chloride (Not Required)
13.3	Hydrogen Bromide (Not Required)
13.4	Organic Halides (32 compounds in red and bold listed in "Other Hydrocarbons")

Analytical Data	1. H ₂ O ASTM 7649-10						
File Name, Sample, Sample Loop Pressure (psi)	H2O Sample RT or BAD RF in Pink	H2O Sample Area	Co-Injected Peak RT	Co-Injected Peak AREA	Conc. (PPMV) of H2O in G0793	RF	H2O CONC. (ppmv)
21005B.D - PRAXAIR UHP H2;ENTECH SPLIT 0.01;3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 214.7	0.00	0	0 0 0	874012 489084 152421	0 0 0	1.4E-09 1.3E-09 1.6E-09	0.00
12122001.D - G0860,4PPM CO2,3.2PPM AR,4PPM N2 & O2;ES0.01 3 10UL G0793;10,5&1UL AIR;ES1:100;CF=1.5ML/MN 162.7	0.00	0	0 0 0	1629882 899700 267282	0 0 0	7.6E-10 6.9E-10 9.2E-10	0.00
12122002.D - G0860,4PPM CO2,3.2PPM AR,4PPM N2 & O2;ES0.01 3 10UL G0793;10,5&1UL AIR;ES1:100;CF=1.5ML/MN 158.7	0.00	0	0 0 0	1631681 917381 271572	0 0 0	7.6E-10 6.7E-10 9.1E-10	0.00
12122003.D - G0860,4PPM CO2,3.2PPM AR,4PPM N2 & O2;ES0.01 3 10UL G0793;10,5&1UL AIR;ES1:100;CF=1.5ML/MN 154.8	0.00	0	0 0 0	1685953 887162 287202	0 0 0	7.3E-10 7.0E-10 8.6E-10	0.00
12122004.D - PRAXAIR UHP H2;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 218.8	0.00	0	0 0 0	1604737 908840 289649	0 0 0	7.7E-10 6.8E-10 8.5E-10	0.00
12122005.D - 12SIN00101#5,PORSGRUNN I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 173.9	0.00	0	0 0 0	1680346 909931 294825	0 0 0	7.3E-10 6.8E-10 8.4E-10	0.00
12122006.D - 12SIN00101#5,PORSGRUNN I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 312.5	0.00	0	0 0 0.00	1758473 858392 254121	0 0 0	7.0E-10 7.2E-10 9.7E-10	0.00
12122007.D - 12SIN00101#5,PORSGRUNN I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 263.9	0.00	0	0 0 0	1620567 866575 270546	0 0 0	7.6E-10 7.1E-10 9.1E-10	0.00
12122008.D - 12SIN00102#13,GAUSTAD I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 348	0.00	0	0 0 0	1635131 898412 282882	0 0 0	7.5E-10 6.9E-10 8.7E-10	0.00
12122009.D - 12SIN00102#13,GAUSTAD I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 318.8	0.00	0	0 0 0	1541051 869310 283131	0 0 0	8.0E-10 7.1E-10 8.7E-10	0.00
12122010.D - 12SIN00102#13,GAUSTAD I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 376	0.00	0	0 0 0	1760689 940462 274811	0 0 0	7.0E-10 6.6E-10 9.0E-10	0.00
12122011.D - 12SIN00103#8,OKEM I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 148.2	0.00	0	0 0 0	1711405 930398 324346	0 0 0	7.2E-10 6.6E-10 7.6E-10	0.00
12122012.D - 12SIN00103#8,OKEM I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 137.7	0.00	0	0 0 0	1723569 837966 318215	0 0 0	7.2E-10 7.4E-10 7.8E-10	0.00
12122013.D - 12SIN00103#8,OKEM I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 127.6	0.00	0	0 0 0	1618989 928553 213801	0 0 0	7.6E-10 6.6E-10 1.2E-09	0.00

2.1

CH₄

12/21/2012

Analytical Data

Date

File Name	Injection	Standard Conc. (PPMV)	Volume of CH ₄ in Standard (μL)	Ret. Time (MIN) of CH ₄	Area of CH ₄	CH ₄ Response Factor	Sample CH ₄ Conc. (ppmv)
001F0101.D	G0909,0.0010 PPMV CO, CO2, N2, O2 & 0.00077 PPMV CH4 (INITIAL: SZ 860 MTORR) [SAMPLING: 1L: 55 TORR] in 1MN03SEC (QPZ: 0 TORR) (WAIT 5MIN AFTER SWITCHING TO ANALYTICAL MODE AND START GC) (OT=80C) [PRE-COL IS @LIQ. N2]	0.00077	0.000056	5.469	6679	8.34E-09	
001F0201.D	G0900,0.24 PPMV CO, CO2, N2, O2 & 0.20 PPMV CH4 (INITIAL: SZ 1100 MTORR) [SAMPLING: 1L: 55TORR] IN 3MIN12SEC (QPZ: 0 TORR) (WAIT 5MIN AFTER SWITCHING TO ANALYTICAL MODE AND START GC) (OT=80C) [PRE-COL IS @LIQ. N2]	0.20	0.014474	5.489	102143	1.42E-07	
001F0301.D	PRE-COL IN LIQ. N2 IN ANALYTICAL MODE AND START GC) (OT=80C) [PRE-COL IS @LIQ. N2]			5.503	411		2.82E-04
001F0401.D	12SIN001-03,OKERN I (INITIAL: SZ 425 MTORR) [SAMPLING: 1L: 57TORR] in 27SEC (WAIT 5MIN AFTER SWITCHING TO ANALYTICAL MODE AND START GC) (OT=80C) [PRE-COL IS @LIQ. N2]			5.377	17435		3.29E-02
001F0501.D	12SIN001-01,PORSGRUNN I (INITIAL: SZ 485 MTORR, 270MTORR QPZ:) [SAMPLING: 1L: 58TORR] in 60SEC (WAIT 20MIN AFTER SWITCHING TO ANALYTICAL MODE AND START GC) (OT= 80C) [PRE-COL IS @LIQ. N2]			5.38	210366		3.91E-01
001F0601.D	12SIN001-02#13,GAUSTAD I (INITIAL: SZ 305 MTORR, 300MTORR QPZ:) [SAMPLING: 1L: 57TORR] in 58SEC (QPZ: 0 TORR) (WAIT 5MIN AFTER SWITCHING TO ANALYTICAL MODE AND START GC) (OT=80C) [PRE-COL IS @LIQ. N2]			5.359	11708		2.21E-02
001F0701.D	G0909,0.0010 PPMV CO, CO2, N2, O2 & 0.00077 PPMV CH4 (INITIAL: SZ 395MTORR, S-QSZ:230MTORR) [SAMPLING: 1L: 121TORR] in 8SEC, (QPZ: 0 TORR) (WAIT 5MIN AFTER SWITCHING TO ANALYTICAL MODE AND START GC) (OT=80C) [PRE-COL IS @LIQ. N2]	0.00077	0.000123	5.36	2483	4.94E-08	

2.3 Analytical Laboratory Report for "Other Hydrocarbons"
ASTM 5466

Client: SINTEF
Hydrogen Station at: Porsgrunn
Sample Type: Hydrogen Fuel
Date Sampled: 11092012,12:00
Date Received: 1109/2012
Date Analyzed: 12042012
Time Analyzed: 12:43 pm

Field ID #: 400ML 12SIN001-01#5
Lab Sample ID: 12SIN00101
Concentration Units: PPBV
Date File Location [12SIN001TPH.pdf](#)
Data Filename: 12122303.D
Dilution Factor: 1.0

Analytes	MW	CASNUM	MQL (PPBV)	Results (PPBV)	Qualifier	MQL (ug/L)	Results (ug/L)
1,1,1-Trichloroethane	132	71-55-6	1	0	U	0.005	0
1,1,2,2-Tetrachloroethane	166	79-34-5	1	0	U	0.007	0
1,1,2-Trichloroethane	132	79-00-5	1	0	U	0.005	0
1,2-Dibromoethane	186	106-93-4	1	0	U	0.008	0
1,1-Dichloroethane	98	75-34-3	1	0	U	0.004	0
1,1-Dichloroethene	96	75-35-4	1	0	U	0.004	0
1,2,4-Trichlorobenzene	180	120-82-1	1	0	U	0.007	0
1,2,4-Trimethylbenzene	120	95-63-6	1	0	U	0.005	0
1,2-Dichloroethane	98	107-06-2	1	0	U	0.004	0
1,2-Dichloropropane	112	78-87-5	1	0	U	0.005	0
1,3,5-Trimethylbenzene	120	108-67-8	1	0	U	0.005	0
1,3-Butadiene	54	106-99-0	1	0	U	0.002	0
1,2-Dichlorobenzene	146	95-50-1	1	0	U	0.006	0
1,3-Dichlorobenzene	146	541-73-1	1	0	U	0.006	0
1,4-Dichlorobenzene	146	106-46-7	1	0	U	0.006	0
1,4-Dioxane	88	123-91-1	1	0	U	0.004	0
2-Butanone	72	78-93-3	1	0	U	0.003	0
2-Hexanone	100	591-78-6	1	0	U	0.004	0
4-Ethyltoluene	120	622-96-8	1	0	U	0.005	0
4-Methyl-2-Pentanone	100	108-10-1	1	0	U	0.004	0
Acetone	58	67-64-1	1	1	=	0.002	0.0024
Ethene	28	9002-88-4	2	0	U	0.002	0
Benzene	78	71-43-2	1	0	U	0.003	0
Benzyl Chloride	126	100-44-7	1	0	U	0.005	0
Bromodichloromethane	162	75-27-4	1	0	U	0.007	0
Bromoforn	250	75-25-2	1	0	U	0.01	0
Bromomethane	94	74-83-9	1	0	U	0.004	0
Carbon Disulfide	76	75-15-0	1	0	U	0.003	0
Carbon tetrachloride	152	56-23-5	1	0	U	0.006	0
Chlorobenzene	112	108-90-7	1	0	U	0.005	0
Chloroethane	64	75-00-3	1	0	U	0.003	0
Chloroform	118	67-66-3	1	0	U	0.005	0
Chloromethane	50	74-87-3	1	0	U	0.002	0
cis-1,2-dichloroethene	96	156-59-2	1	0	U	0.004	0
cis-1,3-Dichloropropene	110	10061-01-5	1	0	U	0.005	0
Cyclohexane	84	110-82-7	1	0	U	0.003	0
Dibromochloromethane	206	124-48-1	1	0	U	0.008	0
Dichlorodifluoromethane	120	75-71-8	1	0	U	0.005	0
Ethane	30	74-84-0	2	186	E	0.002	0.23
Ethanol	46	64-17-5	1	0	U	0.002	0
Ethyl Acetate	88	141-78-6	1	0	U	0.004	0
Ethylbenzene	106	100-41-4	1	0	U	0.004	0
Freon113	186	76-13-1	1	0	U	0.008	0
Freon114	170	76-14-2	1	0	U	0.007	0
Heptane	100	142-82-5	1	1.1	=	0.004	0.0045
Hexane	86	110-54-3	1	0	U	0.004	0
Hexachlorobutadiene	258	87-68-3	1	0	U	0.01	0
Isopropyl Alcohol	60	67-63-0	1	0	U	0.002	0
Methylene chloride	84	75-09-2	1	0	U	0.003	0
Methyl tert-Butyl Ether	88	1634-04-4	1	0	U	0.004	0
Propane	44	74-98-6	2	9.9	=	0.004	0.018
Propene	36	115-07-1	1	0	U	0.001	0
Styrene	104	100-42-5	1	0	U	0.004	0
Tetrachloroethene	164	127-18-4	1	0	U	0.007	0
Tetrahydrofuran	72	109-99-9	1	0	U	0.003	0
Toluene	92	108-88-3	1	0	U	0.004	0
trans-1,2-dichloroethene	96	156-60-5	1	0	U	0.004	0
trans-1,3-Dichloropropene	110	10061-02-6	1	0	U	0.005	0
Trichloroethene	130	79-01-6	1	0	U	0.005	0
Trichlorofluoromethane	136	75-69-4	1	0	U	0.006	0
Vinyl acetate	86	108-05-4	1	0	U	0.004	0
Vinyl chloride	62	75-01-4	1	0	U	0.003	0
Xylenes, m&p-	106	108-38-3 & 106-42-3	1	0	U	0.004	0
Xylenes, o-	106	95-47-6	1	0	U	0.004	0
1,1,3,4-Tetrachlorohexafluorobutane	334	423-38-1	1	0	U	0.01	0
Bromochloromethane (surrogate)	128	74-97-5		70	=		
4-BFB(surrogate)	174	460-00-4		103	=		

NOTES:

U - Analytes not detected at, or above the stated detection limit.

0 - A result of zero represents an undetected result at the MQL reported and does not imply an actual value.

PPBV - Parts per billion volume.

MQL - Method quantitation limit.

Surrogate results are in units of percent recovery with control limits: 65 to 135%.

The compounds in red and bold are organic halides.

Tentatively Identified Compound Estimated Concentration (PPBV)

None

12SIN001.xlsx

2.3 Analytical Laboratory Report for "Other Hydrocarbons"

ASTM 5466

Client: SINTEF
 Hydrogen Station at: Gaustad
 Sample Type: Hydrogen Fuel
 Date Sampled: 11092012,12:00
 Date Received: 12202012
 Date Analyzed: 12042012
 Time Analyzed: 1:27 pm

Field ID #: 400ML 12SIN001-02#13
 Lab Sample ID: 12SIN00102
 Concentration Units: PPBV
 Date File Location
 Data Filename: 12122304.D
 Dilution Factor: 1.0

Analytes	MW	CASNUM	ML (PPBV)	Results (PPBV)	Qualifier	ML (ug/L)	Results (ug/L)
1,1,1-Trichloroethane	132	71-55-6	1	0	U	0.005	0
1,1,2,2-Tetrachloroethane	166	79-34-5	1	0	U	0.007	0
1,1,2-Trichloroethane	132	79-00-5	1	0	U	0.005	0
1,2-Dibromoethane	186	106-93-4	1	0	U	0.008	0
1,1-Dichloroethane	98	75-34-3	1	0	U	0.004	0
1,1-Dichloroethene	96	75-35-4	1	0	U	0.004	0
1,2,4-Trichlorobenzene	180	120-82-1	1	0	U	0.007	0
1,2,4-Trimethylbenzene	120	95-63-6	1	0	U	0.005	0
1,2-Dichloroethane	98	107-06-2	1	0	U	0.004	0
1,2-Dichloropropane	112	78-87-5	1	0	U	0.005	0
1,3,5-Trimethylbenzene	120	108-67-8	1	0	U	0.005	0
1,3-Butadiene	54	106-99-0	1	0	U	0.002	0
1,2-Dichlorobenzene	146	95-50-1	1	0	U	0.006	0
1,3-Dichlorobenzene	146	541-73-1	1	0	U	0.006	0
1,4-Dichlorobenzene	146	106-46-7	1	0	U	0.006	0
1,4-Dioxane	88	123-91-1	1	0	U	0.004	0
2-Butanone	72	78-93-3	1	0	U	0.003	0
2-Hexanone	100	591-78-6	1	0	U	0.004	0
4-Ethyltoluene	120	622-96-8	1	0	U	0.005	0
4-Methyl-2-Pentanone	100	108-10-1	1	0	U	0.004	0
Acetone	58	67-64-1	1	1.2	=	0.002	0.0028
Ethene	28	9002-88-4	2	0	U	0.002	0
Benzene	78	71-43-2	1	0	U	0.003	0
Benzyl Chloride	126	100-44-7	1	0	U	0.005	0
Bromodichloromethane	162	75-27-4	1	0	U	0.007	0
Bromoform	250	75-25-2	1	0	U	0.01	0
Bromomethane	94	74-83-9	1	0	U	0.004	0
Carbon Disulfide	76	75-15-0	1	0	U	0.003	0
Carbon tetrachloride	152	56-23-5	1	0	U	0.006	0
Chlorobenzene	112	108-90-7	1	0	U	0.005	0
Chloroethane	64	75-00-3	1	0	U	0.003	0
Chloroform	118	67-66-3	1	0	U	0.005	0
Chloromethane	50	74-87-3	1	0	U	0.002	0
cis-1,2-dichloroethene	96	156-59-2	1	0	U	0.004	0
cis-1,3-Dichloropropene	110	10061-01-5	1	0	U	0.005	0
Cyclohexane	84	110-82-7	1	0	U	0.003	0
Dibromochloromethane	206	124-48-1	1	0	U	0.008	0
Dichlorodifluoromethane	120	75-71-8	1	0	U	0.005	0
Ethane	30	74-84-0	2	4.7	=	0.002	0.0058
Ethanol	46	64-17-5	1	0	U	0.002	0
Ethyl Acetate	88	141-78-6	1	0	U	0.004	0
Ethylbenzene	106	100-41-4	1	0	U	0.004	0
Freon113	186	76-13-1	1	0	U	0.008	0
Freon114	170	76-14-2	1	0	U	0.007	0
Heptane	100	142-82-5	1	0	U	0.004	0
Hexane	86	110-54-3	1	0	U	0.004	0
Hexachlorobutadiene	258	87-68-3	1	0	U	0.01	0
Isopropyl Alcohol	60	67-63-0	1	0	U	0.002	0
Methylene chloride	84	75-09-2	1	0	U	0.003	0
Methyl tert-Butyl Ether	88	1634-04-4	1	0	U	0.004	0
Propane	44	74-98-6	2	0	U	0.004	0
Propene	36	115-07-1	1	0	U	0.001	0
Styrene	104	100-42-5	1	0	U	0.004	0
Tetrachloroethene	164	127-18-4	1	0	U	0.007	0
Tetrahydrofuran	72	109-99-9	1	0	U	0.003	0
Toluene	92	108-88-3	1	0	U	0.004	0
trans-1,2-dichloroethene	96	156-60-5	1	0	U	0.004	0
trans-1,3-Dichloropropene	110	10061-02-6	1	0	U	0.005	0
Trichloroethene	130	79-01-6	1	0	U	0.005	0
Trichlorofluoromethane	136	75-69-4	1	0	U	0.006	0
Vinyl acetate	86	108-05-4	1	0	U	0.004	0
Vinyl chloride	62	75-01-4	1	0	U	0.003	0
Xylenes, m&p-	106	108-38-3 & 106-42-3	1	0	U	0.004	0
Xylenes, o-	106	95-47-6	1	0	U	0.004	0
1,1,3,4-Tetrachlorohexafluorobutane	334	423-38-1	1	4.22	=	0.01	0.058
Bromochloromethane (surrogate)	128	74-97-5		70	=		
4-BFB(surrogate)	174	460-00-4		104	=		

NOTES:

U - Analytes not detected at, or above the stated detection limit.

0 - A result of zero represents an undetected result at the MQL reported and does not imply an actual value.

PPBV - Parts per billion volume.

MQL - Method quantitation limit.

Surrogate results are in units of percent recovery with control limits: 65 to 135%.

The compounds in red and bold are organic halides.

Tentatively Identified Compound Estimated Concentration (PPBV)

None

12SIN001.xlsx

2.3 Analytical Laboratory Report for "Other Hydrocarbons"

ASTM 5466

Client: SINTEF
 Hydrogen Station at: Økern
 Sample Type: Hydrogen Fuel
 Date Sampled: 11092012,12:00
 Date Received: 12202012
 Date Analyzed: 12042012
 Time Analyzed: 2:07 pm

Field ID #: 400ML 12SIN001-03M8
 Lab Sample ID: 12SIN00103
 Concentration Units: PPBV
 Date File Location: 12SIN001TPH.pdf
 Data Filename: 12122305.D
 Dilution Factor: 1.0

Analytes	MW	CASNUM	ML (PPBV)	Results (PPBV)	Qualifier	MQL (ug/L)	Results (ug/L)
1,1,1-Trichloroethane	132	71-55-6	1	0	U	0.005	0
1,1,2,2-Tetrachloroethane	166	79-34-5	1	0	U	0.007	0
1,1,2-Trichloroethane	132	79-00-5	1	0	U	0.005	0
1,2-Dibromoethane	186	106-93-4	1	0	U	0.008	0
1,1-Dichloroethane	98	75-34-3	1	0	U	0.004	0
1,1-Dichloroethene	96	75-35-4	1	0	U	0.004	0
1,2,4-Trichlorobenzene	180	120-82-1	1	0	U	0.007	0
1,2,4-Trimethylbenzene	120	95-63-6	1	0	U	0.005	0
1,2-Dichloroethane	98	107-06-2	1	0	U	0.004	0
1,2-Dichloropropane	112	78-87-5	1	0	U	0.005	0
1,3,5-Trimethylbenzene	120	108-67-8	1	0	U	0.005	0
1,3-Butadiene	54	106-99-0	1	0	U	0.002	0
1,2-Dichlorobenzene	146	95-50-1	1	0	U	0.006	0
1,3-Dichlorobenzene	146	541-73-1	1	0	U	0.006	0
1,4-Dichlorobenzene	146	106-46-7	1	0	U	0.006	0
1,4-Dioxane	88	123-91-1	1	0	U	0.004	0
2-Butanone	72	78-93-3	1	0	U	0.003	0
2-Hexanone	100	591-78-6	1	0	U	0.004	0
4-Ethyltoluene	120	622-96-8	1	0	U	0.005	0
4-Methyl-2-Pentanone	100	108-10-1	1	0	U	0.004	0
Acetone	58	67-64-1	1	1.8	=	0.002	0.0043
Ethene	28	9002-88-4	2	0	U	0.002	0
Benzene	78	71-43-2	1	0	U	0.003	0
Benzyl Chloride	126	100-44-7	1	0	U	0.005	0
Bromodichloromethane	162	75-27-4	1	0	U	0.007	0
Bromoform	250	75-25-2	1	0	U	0.01	0
Bromomethane	94	74-83-9	1	0	U	0.004	0
Carbon Disulfide	76	75-15-0	1	0	U	0.003	0
Carbon tetrachloride	152	56-23-5	1	0	U	0.006	0
Chlorobenzene	112	108-90-7	1	0	U	0.005	0
Chloroethane	64	75-00-3	1	0	U	0.003	0
Chloroform	118	67-66-3	1	0	U	0.005	0
Chloromethane	50	74-87-3	1	0	U	0.002	0
cis-1,2-dichloroethene	96	156-59-2	1	0	U	0.004	0
cis-1,3-Dichloropropene	110	10061-01-5	1	0	U	0.005	0
Cyclohexane	84	110-82-7	1	0	U	0.003	0
Dibromochloromethane	206	124-48-1	1	0	U	0.008	0
Dichlorodifluoromethane	120	75-71-8	1	0	U	0.005	0
Ethane	30	74-84-0	2	2.7	=	0.002	0.0033
Ethanol	46	64-17-5	1	0	U	0.002	0
Ethyl Acetate	88	141-78-6	1	0	U	0.004	0
Ethylbenzene	106	100-41-4	1	0	U	0.004	0
Freon113	186	76-13-1	1	0	U	0.008	0
Freon114	170	76-14-2	1	0	U	0.007	0
Heptane	100	142-82-5	1	0	U	0.004	0
Hexane	86	110-54-3	1	0	U	0.004	0
Hexachlorobutadiene	258	87-68-3	1	0	U	0.01	0
Isopropyl Alcohol	60	67-63-0	1	1.6	=	0.002	0.0039
Methylene chloride	84	75-09-2	1	0	U	0.003	0
Methyl tert-Butyl Ether	88	1634-04-4	1	0	U	0.004	0
Propane	44	74-98-6	2	0	U	0.004	0
Propene	36	115-07-1	1	0	U	0.001	0
Styrene	104	100-42-5	1	0	U	0.004	0
Tetrachloroethene	164	127-18-4	1	0	U	0.007	0
Tetrahydrofuran	72	109-99-9	1	0	U	0.003	0
Toluene	92	108-88-3	1	0	U	0.004	0
trans-1,2-dichloroethene	96	156-60-5	1	0	U	0.004	0
trans-1,3-Dichloropropene	110	10061-02-6	1	0	U	0.005	0
Trichloroethene	130	79-01-6	1	0	U	0.005	0
Trichlorofluoromethane	136	75-69-4	1	0	U	0.006	0
Vinyl acetate	86	108-05-4	1	0	U	0.004	0
Vinyl chloride	62	75-01-4	1	0	U	0.003	0
Xylenes, m&p-	106	106-38-3 & 106-42-3	1	0	U	0.004	0
Xylenes, o-	106	95-47-6	1	0	U	0.004	0
1,1,3,4-Tetrachlorohexafluorobutane	334	423-38-1	1	14	=	0.01	0.19
Bromochloromethane (surrogate)	128	74-97-5		74	=		
4-BFB(surrogate)	174	460-00-4		105	=		

NOTES:

U - Analytes not detected at, or above the stated detection limit.

0 - A result of zero represents an undetected result at the MQL reported and does not imply an actual value.

PPBV - Parts per billion volume.

MQL - Method quantitation limit.

Surrogate results are in units of percent recovery with control limits: 65 to 135%.

The compounds in red and bold are organic halides.

Tentatively Identified Compound Estimated Concentration (PPBV)

None

12SIN001.xlsx

Analytical Data	3. O ₂ ASTM 7649-10						
File Name, Sample, Sample Loop Pressure (psi)	O2 Sample RT OR Appraent Injection Vol. (uL)	O2 Sample Area	Co-Injected Peak RT, or O2 Conc. (ppmv) in P0136	Co-Injected Peak AREA	O2 RF at Low Conc.	O2 RF at High Conc.	O2 CONC. (ppmv)
21005B.D - PRAXAIR UHP H2;ENTECH SPLIT 0.01;3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 214.7	0.00	0	61804 56881 53274	1263083 662761 253016	1.5E-08 1.6E-08 1.7E-08	3.1E-08 3.0E-08 3.1E-08	0.0
12122001.D - G0860,4PPM CO2,3.2PPM AR,4PPM N2 & O2;ES0.01 3 10UL G0793;10,5&1UL AIR;ES1:100;CF=1.5ML/MN 162.7	0.85	27360	77480 72572 69535	1916893 989257 390089	1.2E-08 1.3E-08 1.3E-08	2.1E-08 2.0E-08 2.0E-08	4.3
12122002.D - G0860,4PPM CO2,3.2PPM AR,4PPM N2 & O2;ES0.01 3 10UL G0793;10,5&1UL AIR;ES1:100;CF=1.5ML/MN 158.7	0.85	23080	79936 75620 69258	1877270 972082 383448	1.1E-08 1.2E-08 1.3E-08	2.1E-08 2.0E-08 2.1E-08	3.7
12122003.D - G0860,4PPM CO2,3.2PPM AR,4PPM N2 & O2;ES0.01 3 10UL G0793;10,5&1UL AIR;ES1:100;CF=1.5ML/MN 154.8	0.85	18072	73989 65422 66923	1879959 982498 383680	1.2E-08 1.4E-08 1.4E-08	2.1E-08 2.0E-08 2.1E-08	2.9
12122004.D - PRAXAIR UHP H2;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 218.8	0.00	0	67768 71721 68519	1917229 977788 378295	1.3E-08 1.3E-08 1.3E-08	2.1E-08 2.0E-08 2.1E-08	0.0
12122005.D - 12SIN00101#5,PORSGRUNN I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 173.9	0.00	0	72026 71375 74936	1912719 987581 376468	1.3E-08 1.3E-08 1.2E-08	2.1E-08 2.0E-08 2.1E-08	0.0
12122006.D - 12SIN00101#5,PORSGRUNN I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 312.5	0.00	0	75048 66549 66976	1916331 973145 380460	1.2E-08 1.4E-08 1.4E-08	2.1E-08 2.0E-08 2.1E-08	0.0
12122007.D - 12SIN00101#5,PORSGRUNN I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 263.9	0.00	0	70755 65172 71326	1937211 983473 373575	1.3E-08 1.4E-08 1.3E-08	2.0E-08 2.0E-08 2.1E-08	0.0
12122008.D - 12SIN00102#13,GAUSTAD I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 348	0.00	0	71736 73339 71332	1937150 980636 379657	1.3E-08 1.2E-08 1.3E-08	2.0E-08 2.0E-08 2.1E-08	0.0
12122009.D - 12SIN00102#13,GAUSTAD I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 318.8	0.85	22722	68882 67507 71030	1922504 987637 367405	1.3E-08 1.3E-08 1.3E-08	2.1E-08 2.0E-08 2.1E-08	3.6
12122010.D - 12SIN00102#13,GAUSTAD I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 376	0.83	22838	81312 66718 68057	1915026 995156 381699	1.1E-08 1.4E-08 1.3E-08	2.1E-08 2.0E-08 2.1E-08	3.6
12122011.D - 12SIN00103#8,OKEM I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 148.2	0.00	0	62825 60305 64423	1906145 979810 392228	1.4E-08 1.5E-08 1.4E-08	2.1E-08 2.0E-08 2.0E-08	0.0
12122012.D - 12SIN00103#8,OKEM I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 137.7	0.00	0	71913 66607 67207	1966190 910643 377370	1.3E-08 1.4E-08 1.4E-08	2.0E-08 2.2E-08 2.1E-08	0.0
12122013.D - 12SIN00103#8,OKEM I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 127.6	0.00	0	69698 64800 53561	1828267 991944 317784	1.3E-08 1.4E-08 1.7E-08	2.2E-08 2.0E-08 2.5E-08	0.0

Smartchemistry.com

Analytical Data

4. He

Date of Analysis

12/23/2012

Instrumentation

GC/TCD

File Name	Helium Retention Time (min)	Helium Retention Time (min)	Helium Standard Conc. (PPMV)	Area	Response Factor (RF)	Sample Concentration (PPMV)
001F0101.D	G0812 (164.2 PPMV HE IN HYDROGEN). OT=40C.	3.798	164.2	1.31E+01	12.6	
001F0201.D	G0813 (58.9 PPMV HE IN HYDROGEN). OT=40C.	3.743	58.9	4.11E+00	14.3	
001F0301.D	G0814 (9.1 PPMV HE IN HYDROGEN). OT=40C.	3.801	9.1	6.40E-01	14.2	
001F0401.D	12SIN001-01#5,PORSGRUNN I. R121220. OT=40C.	3.801		0.00E+00		0
001F0501.D	12SIN001-02#13,GAUSTAD I. R121220. OT=40C.	3.989		5.84E-01		8.5
001F0601.D	12SIN001-03#8,OKERN I. R121220. OT=40C.	3.801		0.00E+00		0.0
001F0701.D	G0814 (9.1 PPMV HE IN HYDROGEN). OT=40C.	3.78	9.1	6.14E-01	14.8	

Analytical Data	5. N2 ASTM 7649-10							
File Name, Sample, Sample Loop Pressure (psi)	N2 Sample RT OR Appraent Injection Vol. (uL)	N2 Peak 1 Area	Co-Injected Peak RT	Co-Injected Peak AREA	N2 RF at Low Conc.	N2 RF at High Conc.	N2 CONC. (ppmv)	Appraent Injection Vol. (uL)
21005B.D - PRAXAIR UHP H2;ENTECH SPLIT 0.01;3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 214.7	0.00	0	200531 191082 177408	4663013 2583950 1052164	4.5E-09 4.8E-09 5.1E-09	3.1E-08 2.8E-08 2.8E-08	0.0	
12122001.D - G0860,4PPM CO2,3.2PPM AR,4PPM N2 & O2;ES0.01 3 10UL G0793;10,5&1UL AIR;ES1:100;CF=1.5ML/MN 162.7	0.85	89066	270803 255348 252116	8121014 4474867 1853865	3.4E-09 3.6E-09 3.6E-09	1.8E-08 1.6E-08 1.6E-08	4.8	78
12122002.D - G0860,4PPM CO2,3.2PPM AR,4PPM N2 & O2;ES0.01 3 10UL G0793;10,5&1UL AIR;ES1:100;CF=1.5ML/MN 158.7	0.85	66584	302250 266067 257219	8158035 4506845 1868272	3.0E-09 3.4E-09 3.5E-09	1.8E-08 1.6E-08 1.6E-08	3.6	59
12122003.D - G0860,4PPM CO2,3.2PPM AR,4PPM N2 & O2;ES0.01 3 10UL G0793;10,5&1UL AIR;ES1:100;CF=1.5ML/MN 154.8	0.86	68960	287002 267498 265451	8191852 4536160 948716	3.2E-09 3.4E-09 3.4E-09	1.8E-08 1.6E-08 3.1E-08	3.7	61
12122004.D - PRAXAIR UHP H2;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 218.8	0.00	0	250878 234932 256716	8367474 4551275 1905084	3.6E-09 3.9E-09 3.5E-09	1.7E-08 1.6E-08 1.5E-08	0.0	
12122005.D - 12SIN00101#5,PORSGRUNN I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 173.9	0.88	6371338	274142 245736 292861	8431867 4574391 1884294	3.3E-09 3.7E-09 3.1E-09	1.7E-08 1.6E-08 1.6E-08	340	2203
12122006.D - 12SIN00101#5,PORSGRUNN I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 312.5	0.86	9606674	252463 247424 268926	8411693 4557868 1885036	3.6E-09 3.7E-09 3.4E-09	1.7E-08 1.6E-08 1.6E-08	513	3322
12122007.D - 12SIN00101#5,PORSGRUNN I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 263.9	0.86	8219032	265060 247713 270684	8428170 4587131 1897626	3.4E-09 3.7E-09 3.4E-09	1.7E-08 1.6E-08 1.5E-08	439	2842
12122008.D - 12SIN00102#13,GAUSTAD I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 348	0.85	111714	269049 265845 243215	8387270 4578387 1899629	3.4E-09 3.4E-09 3.7E-09	1.7E-08 1.6E-08 1.5E-08	6.0	
12122009.D - 12SIN00102#13,GAUSTAD I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 318.8	0.87	144274	270711 257728 260732	8446965 4621490 1829358	3.4E-09 3.5E-09 3.5E-09	1.7E-08 1.6E-08 1.6E-08	7.7	
12122010.D - 12SIN00102#13,GAUSTAD I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 376	0.85	174373	313897 247116 259354	8478125 4667175 1922357	2.9E-09 3.7E-09 3.5E-09	1.7E-08 1.6E-08 1.5E-08	9.3	
12122011.D - 12SIN00103#8,OKEM I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 148.2	0.88	30274	257516 244178 231019	8332688 4628505 1920280	3.5E-09 3.7E-09 3.9E-09	1.8E-08 1.6E-08 1.5E-08	1.6	
12122012.D - 12SIN00103#8,OKEM I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 137.7	0.87	32100	262949 240360 274055	8521322 4283403 1873679	3.5E-09 3.8E-09 3.3E-09	1.7E-08 1.7E-08 1.6E-08	1.7	
12122013.D - 12SIN00103#8,OKEM I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 127.6	0.88	31867.79	259302 249271 197322	8106489 4629139 1630077	3.5E-09 3.6E-09 4.6E-09	1.8E-08 1.6E-08 1.8E-08	1.7	

Analytical Data	6. Ar ASTM 7649-10 Date of Analysis 12/20/2012						
File Name, Sample, Sample Loop Pressure (psi)	Ar Sample RT or BAD RF	Ar Sample Area	Co-Injected Peak RT	Co-Injected Peak AREA	RF	RF	Ar CONC. (ppmv)
21005B.D - PRAXAIR UHP H2;ENTECH SPLIT 0.01;3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 214.7	0.00	0	46113.51 44217.56 45841.79	110626 60249 23898	1.6E-08 1.6E-08 1.6E-08	1.6E-08 1.5E-08 1.5E-08	0.0
12122001.D - G0860,4PPM CO2,3.2PPM AR,4PPM N2 & O2;ES0.01 3 10UL G0793;10,5&1UL AIR;ES1:100;CF=1.5ML/MN 162.7	0.86	17206	76337.54 77542.89 77035.66	173450 92967 40027	9.5E-09 9.3E-09 9.4E-09	1.0E-08 9.4E-09 8.8E-09	3.4
12122002.D - G0860,4PPM CO2,3.2PPM AR,4PPM N2 & O2;ES0.01 3 10UL G0793;10,5&1UL AIR;ES1:100;CF=1.5ML/MN 158.7	0.85	15121	75239.41 77076.48 75858.87	170537 92407 40309	9.6E-09 9.4E-09 9.5E-09	1.0E-08 9.5E-09 8.7E-09	3.0
12122003.D - G0860,4PPM CO2,3.2PPM AR,4PPM N2 & O2;ES0.01 3 10UL G0793;10,5&1UL AIR;ES1:100;CF=1.5ML/MN 154.8	0.86	16547	78258.15 79238.17 170945.72	90896 34770 34576	9.2E-09 9.1E-09 4.2E-09	1.9E-08 2.5E-08 1.0E-08	3.3
12122004.D - PRAXAIR UHP H2;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 218.8	0.00	0	79518.07 78449.60 78093.64	180215 92384 38574	9.1E-09 9.2E-09 9.3E-09	9.7E-09 9.5E-09 9.1E-09	0.0
12122005.D - 12SIN00101#5,PORSGRUNN I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 173.9	0.89	3846	79743.56 79855.78 79215.85	173494 92227 37905	9.1E-09 9.0E-09 9.1E-09	1.0E-08 9.5E-09 9.2E-09	0.76
12122006.D - 12SIN00101#5,PORSGRUNN I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 312.5	0.86	3956	76332.20 77870.50 77856.17	180136 93403 36812	9.5E-09 9.3E-09 9.3E-09	9.7E-09 9.4E-09 9.5E-09	0.78
12122007.D - 12SIN00101#5,PORSGRUNN I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 263.9	0.87	3903.8	80528 79532 78571	177721 94908 35879	9.0E-09 9.1E-09 9.2E-09	9.9E-09 9.2E-09 9.8E-09	0.77
12122008.D - 12SIN00102#13,GAUSTAD I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 348	0.00	0	79243 78857 80011	178483 95070 37912	9.1E-09 9.2E-09 9.0E-09	9.8E-09 9.2E-09 9.2E-09	0.00
12122009.D - 12SIN00102#13,GAUSTAD I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 318.8	0.85	3246	77669 77680 79716	177370 93672 35316	9.3E-09 9.3E-09 9.1E-09	9.9E-09 9.4E-09 9.9E-09	0.64
12122010.D - 12SIN00102#13,GAUSTAD I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 376	0.00	0	83739 80780 81105	182825 94329 37654	8.6E-09 8.9E-09 8.9E-09	9.6E-09 9.3E-09 9.3E-09	0.00
12122011.D - 12SIN00103#8,OKEM I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 148.2	0.88	8494	77393 77984 79029	178454 94459 37305	9.3E-09 9.3E-09 9.1E-09	9.8E-09 9.3E-09 9.4E-09	1.67
12122012.D - 12SIN00103#8,OKEM I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 137.7	0.85	10216	77642 80314 79501	185842 89016 37874	9.3E-09 9.0E-09 9.1E-09	9.4E-09 9.8E-09 9.3E-09	2.01
12122013.D - 12SIN00103#8,OKEM I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 127.6	0.87	9776.716	82268 73432 79961	172859 93988 32953	8.8E-09 9.8E-09 9.0E-09	1.0E-08 9.3E-09 1.1E-08	1.92

Analytical Data	7. CO ₂ ASTM 7649-10							Date of Analysis 12/20/2012
File Name, Sample, Sample Loop Pressure (psi)	CO2 Sample RT	CO2 Sample Area	Co-Injected Peak RT	Co-Injected Peak AREA	RF	Conc. CO2 in Air (PPMV)	CO2 CONC. (ppmv)	Appraent Injection Vol. (uL)
21005B.D - PRAXAIR UHP H2;ENTECH SPLIT 0.01;3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 214.7	0.00	0	180981 178550 175483	74122 15420 6579	5.0E-09 5.1E-09 5.2E-09	20 8 9	0.0	
12122001.D - G0860,4PPM CO2,3.2PPM AR,4PPM N2 & O2;ES0.01 3 10UL G0793;10,5&1UL AIR;ES1:100;CF=1.5ML/MN 162.7	0.87	72269	298399 305468 308557	79599 26091 10052	3.0E-09 3.0E-09 2.9E-09	22 14 14	4.0	49
12122002.D - G0860,4PPM CO2,3.2PPM AR,4PPM N2 & O2;ES0.01 3 10UL G0793;10,5&1UL AIR;ES1:100;CF=1.5ML/MN 158.7	0.87	71362	301622 299359 302175	110366 41638 20695	3.0E-09 3.0E-09 3.0E-09	30 23 28	4.0	49
12122003.D - G0860,4PPM CO2,3.2PPM AR,4PPM N2 & O2;ES0.01 3 10UL G0793;10,5&1UL AIR;ES1:100;CF=1.5ML/MN 154.8	0.86	70712	322853 332290 333249	100151 22117 12032	2.8E-09 2.7E-09 2.7E-09	27 12 16	4.0	48
12122004.D - PRAXAIR UHP H2;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 218.8	0.00	0	334436 342323 339651	102093 55177 12394	2.7E-09 2.7E-09 2.7E-09	28 30 17	0.0	
12122005.D - 12SIN00101#5,PORSGRUNN I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 173.9	0.88	48302	339199 343367 341649	95555 28148 10809	2.7E-09 2.6E-09 2.7E-09	26 15 15	2.7	
12122006.D - 12SIN00101#5,PORSGRUNN I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 312.5	0.86	70760	331458 335541 342541	90853 40317 13658	2.7E-09 2.7E-09 2.7E-09	25 22 19	4.0	
12122007.D - 12SIN00101#5,PORSGRUNN I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 263.9	0.86	57727	330285 337025 341584	92970 39386 13310	2.8E-09 2.7E-09 2.7E-09	25 21 18	3.2	
12122008.D - 12SIN00102#13,GAUSTAD I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 348	0.00	0	335791 341168 343699	70913 35615 9566	2.7E-09 2.7E-09 2.6E-09	19 19 13	0.0	
12122009.D - 12SIN00102#13,GAUSTAD I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 318.8	0.00	0	340720 341380 345318	118370 45577 12015	2.7E-09 2.7E-09 2.6E-09	32 25 16	0.0	
12122010.D - 12SIN00102#13,GAUSTAD I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 376	0.00	0	349987 349718 349696	130761 28602 14575	2.6E-09 2.6E-09 2.6E-09	36 16 20	0.0	
12122011.D - 12SIN00103#8,OKEM I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 148.2	0.00	0	334009 355403 347817	72929 25713 11742	2.7E-09 2.6E-09 2.6E-09	20 14 16	0.0	
12122012.D - 12SIN00103#8,OKEM I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 137.7	0.00	0	339342 342943 340471	76521 22501 10315	2.7E-09 2.7E-09 2.7E-09	21 12 14	0.0	
12122013.D - 12SIN00103#8,OKEM I,R121220;ES0.01 3 10UL G0793;10,5&2UL AIR;ES1:100;CF=1.5ML/MN 127.6	0	0	347597 312588 351133	114426 36365 8398	2.6E-09 2.9E-09 2.6E-09	31 20 11	0.0	

Analytical Data

8. CO

12/21/2012

Date

File Name	Injection	Standard Conc. (PPMV)	Volume of CO in Standard (μL)	Ret. Time (MIN) of CO	Area of CO	CO Response Factor	Sample CO Conc. (ppmv)
001F0101.D	G0909,0.0010 PPMV CO, CO2, N2, O2 & 0.00077 PPMV CH4 (INITIAL: SZ 860 MTORR) [SAMPLING: 1L: 55 TORR] in 1MN03SEC (QPZ: 0 TORR) (WAIT 5MIN AFTER SWITCHING TO ANALYTICAL MODE AND START GC) (OT=80C) [PRE-COL IS @LIQ. N2]	0.0010	0.000072	6.316	506	1.43E-07	
001F0201.D	G0900,0.24 PPMV CO, CO2, N2, O2 & 0.20 PPMV CH4 (INITIAL: SZ 1100 MTORR) [SAMPLING: 1L: 55TORR] IN 3MIN12SEC (QPZ: 0 TORR) (WAIT 5MIN AFTER SWITCHING TO ANALYTICAL MODE AND START GC) (OT=80C) [PRE-COL IS @LIQ. N2]	0.24	0.017368	6.317	70252	2.47E-07	
001F0301.D	PRE-COL IN LIQ. N2 IN ANALYTICAL MODE AND START GC) (OT=80C) [PRE-COL IS @LIQ. N2]			6.317	0		0.00E+00
001F0401.D	12SIN001-03,OKERN I (INITIAL: SZ 425 MTORR) [SAMPLING: 1L: 57TORR] in 27SEC (WAIT 5MIN AFTER SWITCHING TO ANALYTICAL MODE AND START GC) (OT=80C) [PRE-COL IS @LIQ. N2]			6.21	508		9.68E-04
001F0501.D	12SIN001-01,PORSGRUNN I (INITIAL: SZ 485 MTORR, 270MTORR QPZ:) [SAMPLING: 1L: 58TORR] in 60SEC (WAIT 20MIN AFTER SWITCHING TO ANALYTICAL MODE AND START GC) (OT= 80C) [PRE-COL IS @LIQ. N2]			6.204	5156		9.66E-03
001F0601.D	12SIN001-02#13,GAUSTAD I (INITIAL: SZ 305 MTORR, 300MTORR QPZ:) [SAMPLING: 1L: 57TORR] in 58SEC (QPZ: 0 TORR) (WAIT 5MIN AFTER SWITCHING TO ANALYTICAL MODE AND START GC) (OT=80C) [PRE-COL IS @LIQ. N2]			6.182	2451		4.67E-03
001F0701.D	G0909,0.0010 PPMV CO, CO2, N2, O2 & 0.00077 PPMV CH4 (INITIAL: SZ 395MTORR, S-QSZ:230MTORR) [SAMPLING: 1L: 121TORR] in 8SEC, (QPZ: 0 TORR) (WAIT 5MIN AFTER SWITCHING TO ANALYTICAL MODE AND START GC) (OT=80C) [PRE-COL IS @LIQ. N2]	0.0010	0.000159	6.191	457	3.48E-07	

9.1 S		Date of Analysis 12/21/2012			H ₂ S		COS			CH ₃ SH		C ₂ H ₅ SH		CS ₂		(CH ₃) ₃ CSH			Tetrahydrothiophene							
File Names	Injection	H2 VOLUME (uL)	Volume of Standard Injected (uL)	Injected Standard Conc (ppbv)	Recovery or Spiked Conc. (ppbv)	H ₂ S RT	H ₂ S Area	H ₂ S RF, H ₂ S Conc. (PPBV) or % Spike Recovery	COS RT	COS Area	COS RF, COS Conc. (PPBV) or % Spike Recovery	CH ₃ SH RT	CH ₃ SH Area	CH ₃ SH Conc. (PPBV) or % Spike Recovery	C ₂ H ₅ SH RT	C ₂ H ₅ SH Area	C ₂ H ₅ SH Conc. (PPBV) or % Spike Recovery	CS ₂ RT	CS ₂ Area	CS ₂ RF, CS ₂ Conc. (PPBV) or % Spike Recovery	TBM RT	TBM Area	TBM RF, TBM Conc. (PPBV) or % Spike Recovery	THT RT	THT Area	THT RF, THT Conc. (PPBV) or % Spike Recovery
12122101.D	200UL G0915,1PPMV H2S,COS,CH3SH,CS2,DB@H2O,LN, 1PPMV ETHYLMERCAPTAN,TBM & THT	200	1000	2.00E-04		1.37	4862	4.11E-08	1.48	13471	1.48E-08	2.07	4433	4.51E-08	3.10E+00	4.47E+03	4.48E-08	3.85E+00	1.38E+04	1.45E-08	4.84	9806	2.04E-08			
12122102.D	50UL G0915,1PPMV H2S,COS,CH3SH,CS2,DB@H2O,LN, 1PPMV ETHYLMERCAPTAN,TBM & THT	50	1000	5.00E-05		1.37	1559	3.21E-08	1.48	3809	1.31E-08	2.07	1624	3.08E-08	3.10E+00	1.74E+03	2.88E-08	3.85E+00	5.68E+03	8.81E-09	4.83	4306	1.16E-08			
12122103.D	200UL G0915,1PPMV H2S,COS,CH3SH,CS2,DB@H2O,LN, 1PPMV ETHYLMERCAPTAN,TBM & THT	200	1000	2.00E-04		1.37	3461	5.78E-08	1.48	12399	1.61E-08	2.07	4102	4.88E-08	3.10E+00	4.47E+03	4.47E-08	3.85E+00	1.43E+04	1.40E-08	4.84	10516	1.90E-08	8.17	18597	1.08E-08
12122104.D	100UL G0915,1PPMV H2S,COS,CH3SH,CS2,DB@H2O,LN, 1PPMV ETHYLMERCAPTAN,TBM & THT	100	1000	1.00E-04		1.37	2138	4.68E-08	1.48	7499	1.33E-08	2.07	2328	4.29E-08	3.11E+00	2.61E+03	3.83E-08	3.85E+00	8.44E+03	1.19E-08	4.83	6935	1.44E-08	8.16	7427	1.35E-08
12122105.D	50UL G0915,1PPMV H2S,COS,CH3SH,CS2,DB@H2O,LN, 1PPMV ETHYLMERCAPTAN,TBM & THT	50	1000	5.00E-05		1.37	1523	3.28E-08	1.48	3772	1.33E-08	2.07	1537	3.25E-08	3.09E+00	1.98E+03	2.52E-08	3.84E+00	5.73E+03	8.73E-09	4.84	4399	1.14E-08	8.17	7756	6.45E-09
12122106.D	20UL G0915,1PPMV H2S,COS,CH3SH,CS2,DB@H2O,LN, 1PPMV ETHYLMERCAPTAN,TBM & THT	20	1000	2.00E-05		1.38	579	3.45E-08	1.49	1396	1.43E-08	2.07	573	3.49E-08	3.11E+00	9.13E+02	2.19E-08	3.85E+00	1.94E+03	1.03E-08	4.82	1433	1.40E-08	8.17	2163	9.24E-09
12122107.D	10UL G0915,1PPMV H2S,COS,CH3SH,CS2,DB@H2O,LN, 1PPMV ETHYLMERCAPTAN,TBM & THT	10	1000	1.00E-05		1.36	244	4.10E-08	1.47	677	1.48E-08	2.06	303	3.30E-08	3.09E+00	3.36E+02	2.97E-08	3.84E+00	1.09E+03	9.15E-09	4.84	986	1.01E-08	8.17	1349	7.41E-09
12122108.D	500ML 12SIN00101#5_PORSGRUNN I,DB@H2O,LN, R121220	500				1.34	60	0.0049	1.45	1176	0.034	2.05	68	0	3.10E+00	0.00E+00	0	3.83E+00	7.97E+03	0.18	4.84	164	0.0047	8.34	389	0.0075
12122109.D	500ML 12SIN00102#13_GAUSTAD I,DB@H2O,LN, R121220	500				1.35	309	0.0251	1.45	492	0.014	2.03	141	0.011	3.10E+00	0.00E+00	0	3.83E+00	1.51E+03	0.034	4.82	0	0	8.16	0	0
12122110.D	500ML 12SIN00103#8_OKERN I,DB@H2O,LN, R121220	500				1.34	297	0.0242	1.44	312	0.009	2.05	163	0.012	3.10E+00	0.00E+00	0	3.82E+00	1.06E+03	0.023	4.82	0	0	8.16	0	0.0000
12122111.D	500ML 12HYF001#11,DB@H2O,LN,	500				1.34	220	0.0179	1.45	1522	0.044	2.04	455	0	3.10E+00	0.00E+00	0	3.82E+00	6.91E+03	0.15	4.81	19052	0.55	8.16	5350	0.1036
12122112.D	500ML 12SIN00101#9_PORSGRUNN II,DB@H2O,LN, R121220	500				1.34	264	0.0215	1.45	588	0.017	2.02	150	0	3.10E+00	0.00E+00	0	3.82E+00	4.82E+03	0.11	4.82	0	0	8.28	183	0.0036
12122113.D	500ML 12SIN00102#1_GAUSTAD II,DB@H2O,LN, R121220	500				1.34	218	0.0177	1.45	374	0.011	2.06	0	0.00	3.10E+00	0.00E+00	0	3.83E+00	1.13E+03	0.025	4.82	0	0	8.16	0	0
12122114.D	500ML 12SIN00103#12_OKERN II,DB@H2O,LN, R121220	500				1.34	698	0.0567	1.45	942	0.027	2.06	0	0	3.10E+00	0.00E+00	0	3.82E+00	1.99E+04	0.44	4.82	0	0	8.16	0	0.0000
12122115.D	10UL G0915,1PPMV H2S,COS,CH3SH,CS2,DB@H2O,LN, 1PPMV ETHYLMERCAPTAN,TBM & THT	10	1000	1.00E-05		1.36	255	3.92E-08	1.47	680	1.47E-08	2.06	316	3.16E-08	3.10E+00	3.50E+02	2.85E-08	3.83E+00	8.92E+02	1.12E-08	4.82	677	1.48E-08	8.16	926	1.08E-08

10.

HCHO

Date of Analysis 12/28/2012

FILE NAME	Standards or Sample	RT OF M/E30	HCHO AREA OF M/E30	H2 VOLUME (ML)	EJ INJECTION HCHO VOLUME (UL)	CONC OF HCHO STANDARD (PPMV)	SPIKED HCHO STD CONC (PPBV)	HCHO RESPONSE FACTOR	SAMPLE HCHO CONC (PPBV)
12122801.D	EJ132UL G0905 (3.8PPMV HCHO),500ML H2 G0862-3.8PPMV HCHO	2.984	2687	500	132	3.8	1.0	1.9E-07	
12122802.D	500ML H2	3.016	269	500					0.10
12122803.D	500ML 12SIN001-01#5,PORGRUNN I,R121220 12SIN001-01#5	0	0	500					0.00
12122804.D	500ML 12SIN001-02#13,GAUSTAD I,R121220 12SIN001-02#13	0	0	500					0.00
12122805.D	500ML 12SIN001-03#8,OKERN I,R121220 12SIN001-032#8	0	0	500					0.00
12122806.D	EJ132UL G0905 (3.8PPMV HCHO),500ML H2 G0862-3.8PPMV HCHO	2.974	1328	500	132	3.8	1.0	3.8E-07	

11.
HCOOH

Date of Analysis 12/12/2029

FILE NAME	Injection	Volume of Hydrogen (mL)	Volume Injected (uL) of HCOOH Standard @ 43 PPMV	Volume of HCOOH (uL) Injected in Standard & Spike Analysis	RET TIME OF HCOOH	AREA OF HCOOH	Response Factor of Sample Conc in ppmv, Spike Recovery
001F0701.D	12UL G0918 (43 PPMV HCOOH IN H2) WITH LN2 AND FAST MODE. CF=14.3MLK/MIN AND MAKE-UP+CF=100ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). PUMP=5 TURNS. INJ. T= 100C. OT=50C. DESORBED @RT H2O.		12	0.000516	0.779	6.70E+05	7.7E-10
001F0801.D	12UL G0918 (43 PPMV HCOOH IN H2) WITH LN2 AND FAST MODE. CF=14.3MLK/MIN AND MAKE-UP+CF=100ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). PUMP=5 TURNS. INJ. T= 100C. OT=50C. DESORBED @RT H2O.		12	0.000516	0.77	8.68E+05	5.9E-10
001F0901.D	12UL G0918 (43 PPMV HCOOH IN H2) WITH LN2 AND FAST MODE. CF=14.3MLK/MIN AND MAKE-UP+CF=100ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). PUMP=5 TURNS. INJ. T= 100C. OT=50C. DESORBED @RT H2O.		12	0.000516	0.778	6.67E+05	7.7E-10
001F1001.D	12UL G0918 (43 PPMV HCOOH IN H2) WITH LN2 AND FAST MODE. CF=14.3MLK/MIN AND MAKE-UP+CF=100ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). PUMP=5 TURNS. INJ. T= 120C. OT=50C. DESORBED @RT H2O.		12	0.000516	0.78	5.21E+05	9.9E-10
001F1101.D	12UL G0918 (43 PPMV HCOOH IN H2) WITH LN2 AND FAST MODE. CF=14.3MLK/MIN AND MAKE-UP+CF=100ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). PUMP=5 TURNS. INJ. T= 100C. OT=50C. DESORBED @RT H2O.		12	0.000516	0.776	4.29E+05	1.2E-09
001F1201.D	12UL G0918 (43 PPMV HCOOH IN H2) WITH LN2 AND FAST MODE. CF=14.3MLK/MIN AND MAKE-UP+CF=100ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). PUMP=5 TURNS. INJ. T= 100C. OT=50C. DESORBED @RT H2O.		12	0.000516	0.775	6.78E+05	7.6E-10
001F1301.D	12UL G0918 (43 PPMV HCOOH IN H2) WITH LN2 AND FAST MODE. CF=14.3MLK/MIN AND MAKE-UP+CF=100ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). PUMP=5 TURNS. INJ. T= 100C. OT=50C. DESORBED @RT H2O.		12	0.000516	0.773	5.13E+05	1.0E-09
001F1701.D	500ML 12SIN001-01#5,PORSGRUNN I, R121220. WITH LN2 AND FAST MODE. CF=14.3MLK/MIN AND MAKE-UP+CF=100ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). PUMP=5 TURNS. INJ. T=100C. OT=50C. DESORBED @RT H2O.	500			0.773	0.00E+00	0.000000
001F1901.D	12UL G0918 (43 PPMV HCOOH IN H2) WITH LN2 AND FAST MODE. CF=14.3MLK/MIN AND MAKE-UP+CF=100ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). PUMP=5 TURNS. INJ. T= 100C. OT=50C. DESORBED @RT H2O.		12	0.000516	0.78	4.72E+05	1.1E-09
001F2101.D	12UL G0918 (43 PPMV HCOOH IN H2)+500ML 12SIN001-01#5,PORSGRUNN I, R121220. WITH LN2 AND FAST MODE. CF=14.3MLK/MIN AND MAKE-UP+CF=100ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). PUMP=5 TURNS. INJ. T=100C. OT=50C. DESORBED @RT H2O.	500	12	0.000516	1.153	3.42E+05	Percent Spike Recovery of HCOOH @0.001 PPMV is 57%
001F2401.D	500ML 12SIN001-02#13,GAUSTAD I, R121220. WITH LN2 AND FAST MODE. CF=14.3MLK/MIN AND MAKE-UP+CF=100ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). PUMP=5 TURNS. INJ. T=100C. OT=50C. DESORBED @RT H2O.	500			1.226	3.85E+04	0.000066
001F2501.D	12UL G0918 (43 PPMV HCOOH IN H2)+500ML 12SIN001-02#13,GAUSTAD I, R121220. WITH LN2 AND FAST MODE. CF=14.3MLK/MIN AND MAKE-UP+CF=100ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). PUMP=5 TURNS. INJ. T=100C. OT=50C. DESORBED @RT H2O.	500	12	0.000516	1.085	1.99E+05	Percent Spike Recovery of HCOOH @0.001 PPMV is 33%
001F2701.D	500ML 12SIN001-03#8,OKERN I, R121220. WITH LN2 AND FAST MODE. CF=14.3MLK/MIN AND MAKE-UP+CF=100ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). PUMP=5 TURNS. INJ. T=100C. OT=50C. DESORBED @RT H2O.	500			1.077	3.82E+04	0.000065
001F2801.D	12UL G0918 (43 PPMV HCOOH IN H2)+500ML 12SIN001-03#8,OKERN I, R121220. WITH LN2 AND FAST MODE. CF=14.3MLK/MIN AND MAKE-UP+CF=100ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). PUMP=5 TURNS. INJ. T=100C. OT=50C. DESORBED @RT H2O.	500	12	0.000516	1.035	3.74E+05	Percent Spike Recovery of HCOOH @0.001 PPMV is 62%
001F2901.D	12UL G0918 (43 PPMV HCOOH IN H2) WITH LN2 AND FAST MODE. CF=14.3MLK/MIN AND MAKE-UP+CF=100ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). PUMP=5 TURNS. INJ. T= 100C. OT=50C. DESORBED @RT H2O.		12	0.000516	0.778	9.89E+05	5.2E-10

12.

NH₃

Date of Analysis 12/12/2028

FILE NAME	Injection	H2 VOLUME (ML)	Volume Injected (uL) of 12. NH3 Standard @ 4976 PPMV	Injected Standard Volume (uL)	RET TIME OF 12. NH3	AREA OF 12. NH3	Response Factor or <i>Sample Conc in ppmv, Spike Recovery</i>
001F0201.D	4UL G0808 (4976PPMV NH3 IN H2). WITH LIQ. N2. CF=30ML/MN AND MAKE-UP=88ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). N MODE. PUMP AT 5 BACK TURNS RATE. DESORBED @RT. NO LIQ. N2 CRYOFUCUSSING.		4	0.0199040	0.542	1.06E+06	1.9E-08
001F0301.D	4UL G0808 (4976PPMV NH3 IN H2). WITH LIQ. N2. CF=30ML/MN AND MAKE-UP=88ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). N MODE. PUMP AT 5 BACK TURNS RATE. DESORBED @RT. NO LIQ. N2 CRYOFUCUSSING.		4	0.0199040	0.54	1.22E+06	1.6E-08
001F0401.D	4UL G0808 (4976PPMV NH3 IN H2). WITH LIQ. N2. CF=30ML/MN AND MAKE-UP=88ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). N MODE. PUMP AT 5 BACK TURNS RATE. DESORBED @RT. NO LIQ. N2 CRYOFUCUSSING.		4	0.0199040	0.548	8.13E+05	2.4E-08
001F0501.D	4UL G0808 (4976PPMV NH3 IN H2). WITH LIQ. N2. CF=30ML/MN AND MAKE-UP=88ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). N MODE. PUMP AT 5 BACK TURNS RATE. DESORBED @RT. NO LIQ. N2 CRYOFUCUSSING.		4	0.0199040	0.545	6.41E+05	3.1E-08
001F0601.D	4UL G0808 (4976PPMV NH3 IN H2). WITH LIQ. N2. CF=30ML/MN AND MAKE-UP=88ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). N MODE. PUMP AT 5 BACK TURNS RATE. DESORBED @RT. NO LIQ. N2 CRYOFUCUSSING.		4	0.0199040	0.545	1.48E+06	1.3E-08
001F0701.D	4UL G0808 (4976PPMV NH3 IN H2). WITH LIQ. N2. CF=30ML/MN AND MAKE-UP=88ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). N MODE. PUMP AT 5 BACK TURNS RATE. DESORBED @RT. NO LIQ. N2 CRYOFUCUSSING.		4	0.0199040	0.541	1.78E+06	1.1E-08
001F1001.D	500ML 12SIN001-01#5,PORSGRUNN I,R121220. WITH LIQ. N2. CF=30ML/MN AND MAKE-UP= 88ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). N MODE. PUMP AT 5 BACK TURNS RATE. DESORBED @RT. NO LIQ. N2 CRYOFUCUSSING.	500			0.541	0.00E+00	0
001F1401.D	4UL G0808 (4976PPMV NH3 IN H2)+500ML 12SIN001-01#5,PORSGRUNN I,R121220. WITH LIQ. N2. CF=30ML/MN AND MAKE-UP=88ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). N MODE. PUMP AT 5 BACK TURNS RATE. DESORBED @RT. NO LIQ. N2 CRYOFUCUSSING.	500	4	0.0199040	0.556	7.41E+05	<i>Percent Spike Recovery of NH3 @0.040 PPMV is 74%</i>
001F1501.D	500ML 12SIN001-02#13,GAUSTAD I,R121220. WITH LIQ. N2. CF=30ML/MN AND MAKE-UP= 88ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). N MODE. PUMP AT 5 BACK TURNS RATE. DESORBED @RT. NO LIQ. N2 CRYOFUCUSSING.	500			0.541	0.00E+00	0
001F1601.D	4UL G0808 (4976PPMV NH3 IN H2)+500ML 12SIN001-02#13,GAUSTAD I,R121220. WITH LIQ. N2. CF=30ML/MN AND MAKE-UP=88ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). N MODE. PUMP AT 5 BACK TURNS RATE. DESORBED @RT. NO LIQ. N2 CRYOFUCUSSING.	500	4	0.0199040	0.569	6.20E+05	<i>Percent Spike Recovery of NH3 @0.040 PPMV is 62%</i>
001F1701.D	500ML 12SIN001-03#8,OKERN I,R121220. WITH LIQ. N2. CF=30ML/MN AND MAKE-UP=88ML/ MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). N MODE. PUMP AT 5 BACK TURNS RATE. DESORBED @RT. NO LIQ. N2 CRYOFUCUSSING.	500			0.569	0.00E+00	0
001F1801.D	4UL G0808 (4976PPMV NH3 IN H2)+500ML 12SIN001-03#8,OKERN I,R121220. WITH LIQ. N2. CF=30ML/MN AND MAKE-UP=88ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). N MODE. PUMP AT 5 BACK TURNS RATE. DESORBED @RT. NO LIQ. N2 CRYOFUCUSSING.	500	4	0.0199040	0.545	8.49E+05	<i>Percent Spike Recovery of NH3 @0.040 PPMV is 85%</i>
001F1901.D	4UL G0808 (4976PPMV NH3 IN H2). WITH LIQ. N2. CF=30ML/MN AND MAKE-UP=88ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). N MODE. PUMP AT 5 BACK TURNS RATE. DESORBED @RT. NO LIQ. N2 CRYOFUCUSSING.		4	0.0199040	0.55	9.25E+05	2.2E-08
001F2001.D	4UL G0808 (4976PPMV NH3 IN H2)+500ML 12SIN001-03#8,OKERN I,R121220. WITH LIQ. N2. CF=30ML/MN AND MAKE-UP=88ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). N MODE. PUMP AT 5 BACK TURNS RATE. DESORBED @RT. NO LIQ. N2 CRYOFUCUSSING.		4	0.0199040	0.538	1.28E+06	1.6E-08
001F2101.D	4UL G0808 (4976PPMV NH3 IN H2). WITH LIQ. N2. CF=30ML/MN AND MAKE-UP=88ML/MIN. 30M 0.53MM ID 3.0UM DB5 FSCC (SC0CC 001). N MODE. PUMP AT 5 BACK TURNS RATE. DESORBED @RT. NO LIQ. N2 CRYOFUCUSSING.		4	0.0199040	0.547	7.53E+05	2.6E-08

Analytical Data

13.1

Cl₂

Date of Analysis 01/04/2013

FILE NAME	Injection	Volume of Hydrogen (mL)	Volume Injected (uL) of Cl ₂ Standard @ 100.0 PPMV	Volume of Cl ₂ (uL) Injected in Standard & Spike Analysis	RET TIME OF Cl ₂	AREA OF Cl ₂	Response Factor or <i>Sample Conc in ppmv, Spike Recovery</i>
001F0501.D	5UL G0922 (100 PPMV CL2 IN H2) WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND		5	0.0005	0.464	1.23E+06	4.1E-10
001F0601.D	5UL G0922 (100 PPMV CL2 IN H2) WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND		5	0.0005	0.445	1.38E+06	3.6E-10
001F0701.D	5UL G0922 (100 PPMV CL2 IN H2) WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND		5	0.0005	0.489	1.11E+06	4.5E-10
001F0801.D	5UL G0922 (100 PPMV CL2 IN H2) WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND		5	0.0005	0.52	1.26E+06	4.0E-10
001F0901.D	5UL G0922 (100 PPMV CL2 IN H2) WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND		5	0.0005	0.496	9.48E+05	5.3E-10
001F1001.D	5UL G0922 (100 PPMV CL2 IN H2) WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND		5	0.0005	0.515	9.04E+05	5.5E-10
001F1101.D	5UL G0922 (100 PPMV CL2 IN H2) WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND		5	0.0005	0.511	9.80E+05	5.1E-10
001F1201.D	500ML 12ALA001-01#5,PORSGRUNN I, R121220 WITH LIQ. N2. N AND FAST MODE. CF=32.	500		0	0.511	0.00E+00	0.0E+00
001F1401.D	5UL G0922 (100 PPMV CL2 IN H2) WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND		5	0.0005	0.534	6.29E+05	7.9E-10
001F1801.D	5UL G0922 (100 PPMV CL2 IN H2) WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND		5	0.0005	0.573	7.74E+05	6.5E-10
001F1901.D	5UL G0922 (100 PPMV CL2 IN H2)+500ML 12ALA001-01#5,PORSGRUNN I, R121220 WITH	500	5	0.0005	0.858	2.54E+05	<i>Percent Spike Recovery of Cl₂ @0.001 PPMV is 23%</i>
001F2001.D	5UL G0922 (100 PPMV CL2 IN H2)+500ML 12ALA001-01#5,PORSGRUNN I, R121220 WITH	500	5	0.0005	0.925	1.72E+05	<i>Percent Spike Recovery of Cl₂ @0.001 PPMV is 16%</i>
001F2101.D	10UL G0922 (100 PPMV CL2 IN H2)+500ML 12ALA001-01#5,PORSGRUNN I, R121220 WITH	500	10	0.001	0.761	1.53E+06	<i>Percent Spike Recovery of Cl₂ @0.002 PPMV is 71%</i>
001F2201.D	500ML 12ALA001-02#13,GAUSTAD I, R121220 WITH LIQ. N2. N AND FAST MODE. CF=32.	500		0	0.7	0.00E+00	0.0E+00
001F2501.D	10UL G0922 (100 PPMV CL2 IN H2)+500ML 12ALA001-02#13,GAUSTAD I, R121220 WITH	500	10	0.001	0.678	3.73E+05	<i>Percent Spike Recovery of Cl₂ @0.002 PPMV is 17%</i>
001F2701.D	20UL G0922 (100 PPMV CL2 IN H2)+500ML 12ALA001-02#13,GAUSTAD I, R121220 WITH	500	20	0.002	0.617	7.46E+05	<i>Percent Spike Recovery of Cl₂ @0.004 PPMV is 17%</i>
001F2801.D	500ML 12ALA001-03#8,OKERN I, R121220 WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/	500		0	0.619	0.00E+00	0.0E+00
001F2901.D	10UL G0922 (100 PPMV CL2 IN H2)+500ML 12ALA001-03#8,OKERN I, R121220 WITH LIQ.	500	10	0.001	0.7	2.85E+05	<i>Percent Spike Recovery of Cl₂ @0.002 PPMV is 13%</i>
001F3001.D	20UL G0922 (100 PPMV CL2 IN H2)+500ML 12ALA001-03#8,OKERN I, R121220 WITH LIQ.	500	20	0.002	0.675	8.99E+05	<i>Percent Spike Recovery of Cl₂ @0.004 PPMV is 21%</i>
001F3101.D	5UL G0922 (100 PPMV CL2 IN H2) WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND		5	0.0005	0.416	1.05E+06	4.7E-10

FILE NAME	Injection	Volume of Hydrogen (mL)	Volume Injected (uL) of HCl Standard @ 64 PPMV	Volume of HCl (uL) Injected in Standard & Spike Analysis	RET TIME OF HCl	AREA OF HCl	Response Factor of Sample Conc in ppmv, Spike Recovery
001F0101.D	7.8UL H2 (CARRIER GAS). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	7.8			0.463	0.00E+00	0.0E+00
001F0201.D	7.8UL G0923 (64 PPMV HCL IN H2). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.		7.8	0.0004992	0.403	5.80E+05	8.6E-10
001F0301.D	7.8UL G0923 (64 PPMV HCL IN H2). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.		7.8	0.0004992	0.518	9.74E+05	5.1E-10
001F0401.D	7.8UL G0923 (64 PPMV HCL IN H2). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.		7.8	0.0004992	0.508	5.25E+05	9.5E-10
001F0501.D	7.8UL G0923 (64 PPMV HCL IN H2). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.		7.8	0.0004992	0.459	1.46E+06	3.4E-10
001F0601.D	7.8UL G0923 (64 PPMV HCL IN H2). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.		7.8	0.0004992	0.394	6.06E+05	8.2E-10
001F0701.D	7.8UL G0923 (64 PPMV HCL IN H2). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.		7.8	0.0004992	0.38	5.98E+05	8.3E-10
001F0801.D	7.8UL G0923 (64 PPMV HCL IN H2). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.		7.8	0.0004992	0.525	6.59E+05	7.6E-10
001F0901.D	7.8UL H2 (CARRIER GAS). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	7.8			0.467	3.02E+05	28
001F1001.D	500ML 12SIN001-01#13,PORSGRUNNER I. WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	500			0.525	0.00E+00	0.0E+00
001F1201.D	15.6UL G0923 (64 PPMV HCL IN H2)+500ML 12SIN001-01#13,PORSGRUNNER I. WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	500	15.6	0.0009984	0.691	6.78E+05	Percent Spike Recovery of HCl @0.002 PPMV is 49%
001F1301.D	500ML 12SIN001-02#5,GAUSTAD I. WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	500			0.691	0.00E+00	0.0E+00
001F1401.D	15.6UL G0923 (64 PPMV HCL IN H2)+500ML 12SIN001-02#5,GAUSTAD I. WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	500	15.6	0.0009984	0.647	6.93E+05	Percent Spike Recovery of HCl @0.002 PPMV is 50%
001F1501.D	500ML 12SIN001-03#8,OKERN I. WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	500			0.691	0.00E+00	0.0E+00
001F1601.D	15.6UL G0923 (64 PPMV HCL IN H2)+500ML 12SIN001-03#8,OKERN I. WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	500	15.6	0.0009984	0.658	7.04E+05	Percent Spike Recovery of HCl @0.002 PPMV is 51%
001F1701.D	7.8UL G0923 (64 PPMV HCL IN H2). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.		7.8	0.0004992	0.467	7.56E+05	6.6E-10

FILE NAME	Injection	Volume of Hydrogen (mL)	Volume Injected (uL) of HBr Standard @ 120 PPMV	Volume of HBr (uL) Injected in Standard & Spike Analysis	RET TIME OF HBr	AREA OF HBr	Response Factor or Sample Conc in ppmv, Spike Recovery
001F0101.D	8.4UL H2 (CARRIER GAS). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF= 116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	8.4			0.509	1.45E+05	11
001F0201.D	8.4UL G0924 (120 PPMV HCL IN H2). WITH LIQ. N2. N AND FAST MODE. CCF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.		8.4	0.001008	0.511	2.95E+06	3.4E-10
001F0301.D	4.2UL G0924 (120 PPMV HCL IN H2). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.		4.2	0.000504	0.412	1.10E+06	4.6E-10
001F0401.D	4.2UL G0924 (120 PPMV HCL IN H2). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.		4.2	0.000504	0.442	8.77E+05	5.7E-10
001F0501.D	4.2UL G0924 (120 PPMV HCL IN H2). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.		4.2	0.000504	0.439	8.64E+05	5.8E-10
001F0601.D	4.2UL G0924 (120 PPMV HCL IN H2). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.		4.2	0.000504	0.476	5.82E+05	8.7E-10
001F0701.D	4.2UL G0924 (120 PPMV HCL IN H2). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.		4.2	0.000504	0.418	7.41E+05	6.8E-10
001F0801.D	4.2UL G0924 (120 PPMV HCL IN H2). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.		4.2	0.000504	0.498	6.35E+05	7.9E-10
001F0901.D	4.2UL G0924 (120 PPMV HCL IN H2). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.		4.2	0.000504	0.405	6.56E+05	7.7E-10
001F1001.D	4.2UL H2 (CARRIER GAS). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF= 116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	4.2			0.473	2.73E+05	41
001F1101.D	500ML 12SIN001-01#13,PORSGRUNNER I. WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	500			0.448	1.74E+04	2.2E-05
001F1201.D	500ML 12SIN001-01#13,PORSGRUNNER I. WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	500			0.405	0.00E+00	0.0E+00
001F1301.D	8.4UL G0924 (120 PPMV HBR IN H2)+500ML 12SIN001-01#13,PORSGRUNNER I. WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	500	8.4	0.001008	0.652	2.76E+06	Percent Spike Recovery of HBr @0.002 PPMV is 173%
001F1401.D	500ML 12SIN001-02#5,GAUSTAD I. WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	500			0.76	6.66E+04	8.4E-05
001F1501.D	8.4UL G0924 (120 PPMV HBR IN H2)+500ML 12SIN001-02#5,GAUSTAD I. WITH LIQ. N2. N AND FAST MODE. CCF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	500	8.4	0.001008	0.6	1.73E+06	Percent Spike Recovery of HBr @0.002 PPMV is 109%
001F1601.D	500ML 12SIN001-03#8,OKERN I. WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	500			0.783	1.13E+05	1.4E-04
001F1701.D	500ML 12SIN001-03#8,OKERN I. WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	500			0.652	0.00E+00	0.0E+00
001F1801.D	8.4UL G0924 (120 PPMV HBR IN H2)+500ML 12SIN001-03#8,OKERN I. WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	500	8.4	0.001008	0.595	1.88E+06	Percent Spike Recovery of HBr @0.002 PPMV is 118%
001F1901.D	4.2UL G0924 (120 PPMV HCL IN H2). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.		4.2	0.000504	0.398	3.63E+06	1.4E-10
001F2001.D	4.2UL G0924 (120 PPMV HBR IN H2)+500ML 12SIN001-01#13,PORSGRUNNER I. WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	500	4.2	0.000504	0.638	2.10E+06	Percent Spike Recovery of HBr @0.001 PPMV is 65%
001F2101.D	4.2UL G0924 (120 PPMV HBR IN H2)+500ML 12SIN001-02#5,GAUSTAD I. WITH LIQ. N2. N AND FAST MODE. CCF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	500	4.2	0.000504	0.685	3.93E+06	Percent Spike Recovery of HBr @0.001 PPMV is 122%
001F2201.D	4.2UL G0924 (120 PPMV HBR IN H2)+500ML 12SIN001-03#8,OKERN I. WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	500	4.2	0.000504	0.719	4.30E+06	Percent Spike Recovery of HBr @0.001 PPMV is 133%
001F2301.D	4.2UL H2 (CARRIER GAS). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF= 116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.	4.2			0.431	6.58E+05	22
001F2401.D	4.2UL G0924 (120 PPMV HCL IN H2). WITH LIQ. N2. N AND FAST MODE. CF=32.6ML/MIN AND MUF+CF=116ML/MIN. 30M 0.53MM ID 1.0UM DB5 FSCC (SCCC 036). PUMP=FS.TRAP DESORBED @RT H2O.		4.2	0.000504	0.451	2.90E+06	1.7E-10

Report Summary for Particulates

	Porsgrunn		Gaustad		Økern	
		12SIN001-01		12SIN001-02		12SIN001-03
Constituent	SAE Limits (µmol/mol)	Smart Chemistry Detection Limits (µmol/mol)	Concentration (µmol/mol)	Concentration (µmol/mol)	Concentration (µmol/mol)	Analytical Method
Particulate Concentration	1mg/Kg		0.042 mg/kg	0.14 mg/kg	0.21 mg/kg	ASTM D7651-10
Particulates Found & Size (ASTM D7634-10) - Images of particulates and pinholes found are in Table 1			Particulate 1 – 0.18mm Particulate 2 – 0.11mm Particulate 3 – 0.15mm Particulate 4 – 0.13mm Particulate 5 – 0.15mm Particulate 6 – 0.24mm Particulate 7 – 0.13mm Particulate 8 – 0.22mm Particulate 9 – 0.14mm Particulate 10 – 0.13mm Particulate 11 – 0.11mm	There are total 48 particulates found with their sizes over 0.03mm. The particulate size distribution is listed below. # of Particulate @ 0.12mm – 1 # of Particulate @ 0.07mm – 3 # of Particulate @ 0.06mm – 1 # of Particulate @ 0.05mm – 6 # of Particulate @ 0.04mm – 22 # of Particulate @ 0.03mm – 15	Particulate 1 - 0.47mm Particulate 2 - 0.51mm Particulate 3 – 0.06mm Particulate 4 – 0.26mm Particulate 5 – 0.30mm Particulate 6 - 0.15mm Particulate 7 – 0.41mm	

Table 1 Particulate Report

Porsgrunn: Particulate Concentration - 0.042 mg/kg

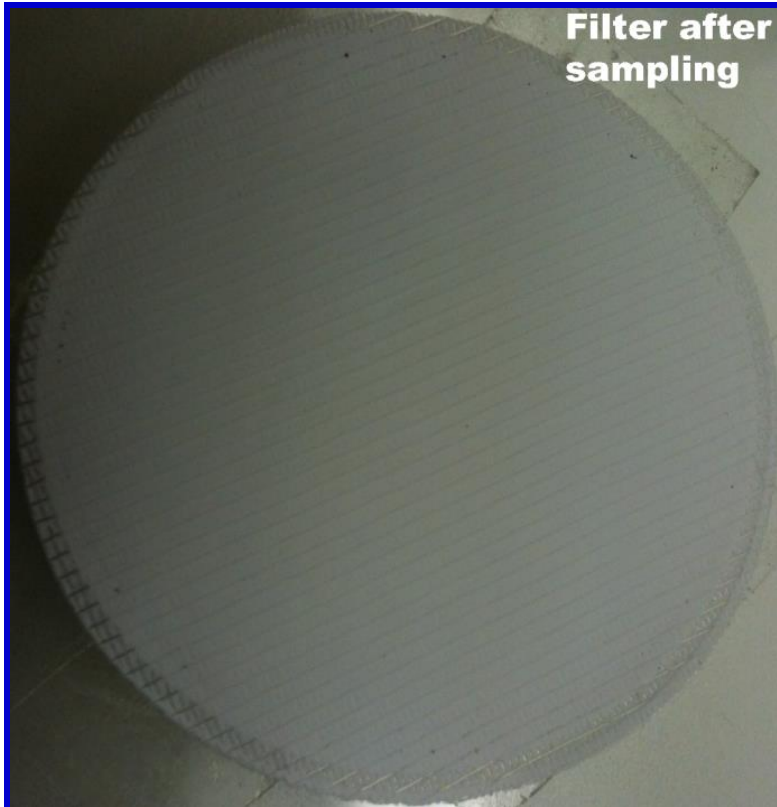

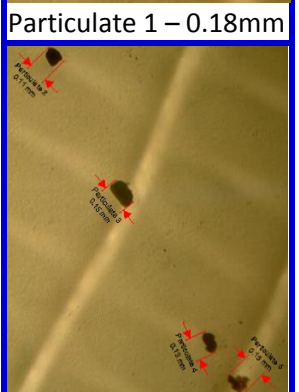

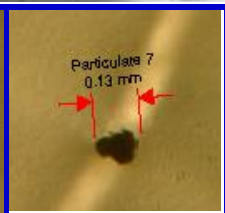
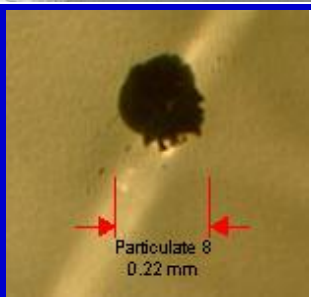
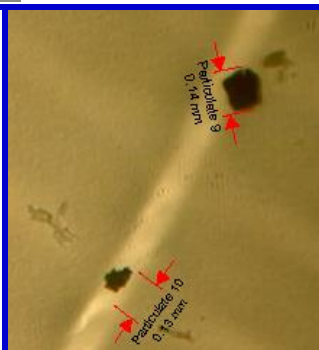

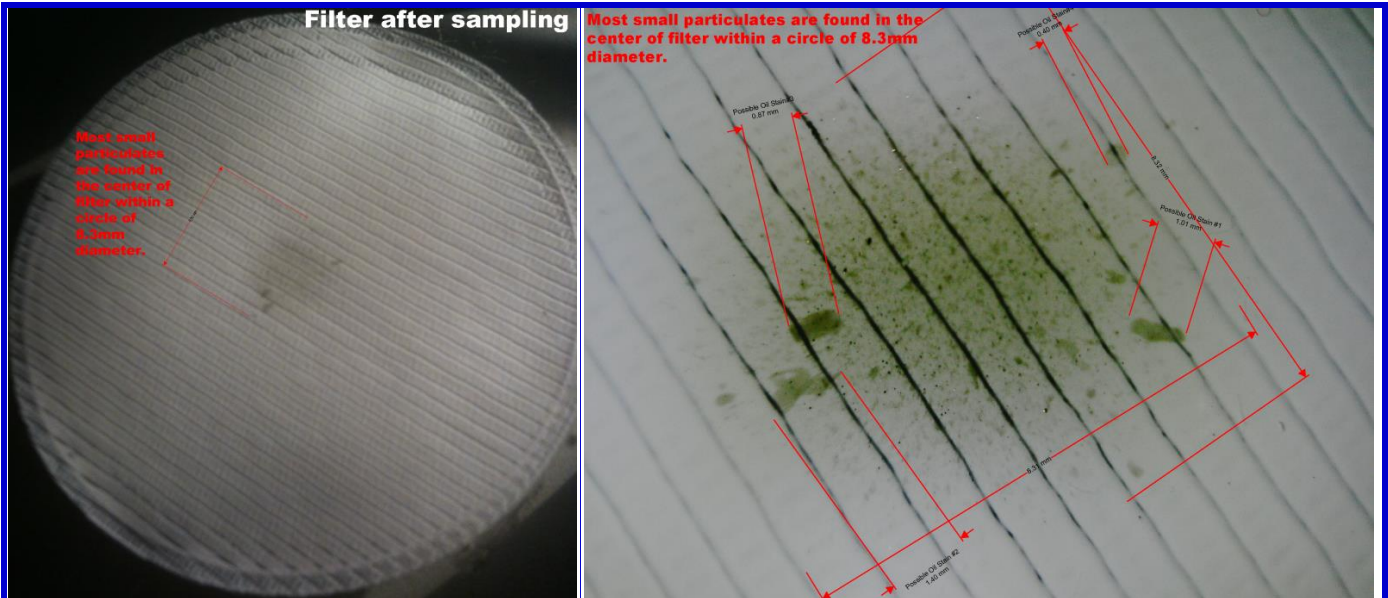
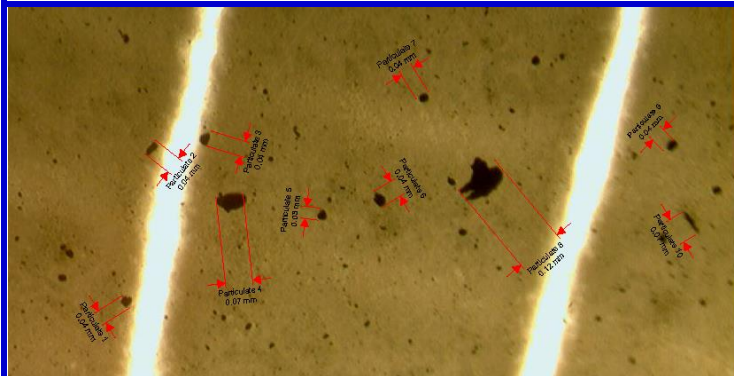
 <p>Filter after sampling</p>		<p>This is particulate filter after sampling, in which 0.086 mg particulates are found in 2.07 kilogram hydrogen. Eleven particulates are found and shown on the left and below along with images and sizes.</p>	 <p>Particulate 1 – 0.18mm</p>  <p>Particulate 2 – 0.11mm Particulate 3 – 0.15mm Particulate 4 – 0.13mm Particulate 5 – 0.15mm</p>	
 <p>Particulate 6 – 0.24mm</p>	 <p>Particulate 7 – 0.13mm</p>	 <p>Particulate 8 – 0.22mm</p>	 <p>Particulate 9 – 0.14mm Particulate 10 – 0.13mm</p>	 <p>Particulate 11 – 0.11mm</p>

Table 1 Particulate Report

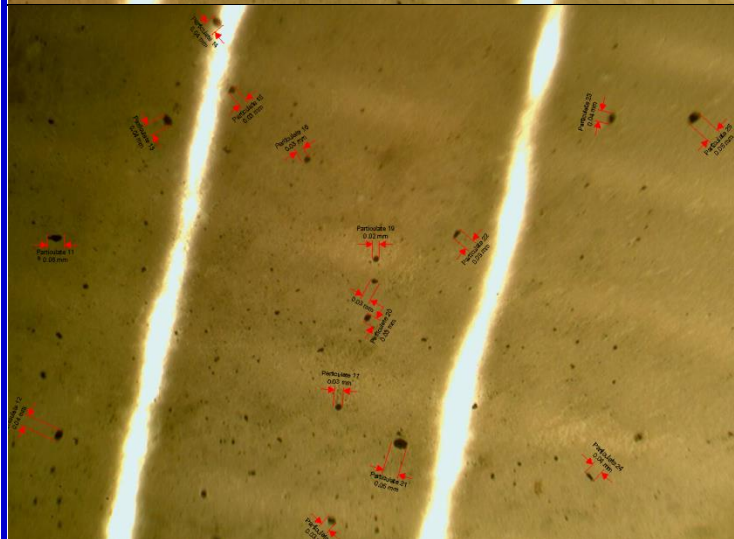
Gaustad: Particulate Concentration - 0.14 mg/kg



This is **particulate filter after sampling**, in which **0.346 mg particulates** are found in **2.4 kilogram hydrogen**. The picture on the right with 27 times magnification shows many oily stains in the filter center within diameter of 8.3mm, in which four large spots with the sizes 1.4, 1.0, 0.87 and 0.40 mm length are found. Forty eight particulates with size larger than 0.02mm are found in the central 8.3mm OD circle. The images and sizes of the 48 particulates are shown below.



- Particulate 1 – 0.04mm
- Particulate 2 – 0.04mm
- Particulate 3 – 0.04mm
- Particulate 4 – 0.07mm
- Particulate 5 – 0.03mm
- Particulate 6 – 0.04mm
- Particulate 7 – 0.04mm
- Particulate 8 – 0.12mm
- Particulate 9 – 0.04mm
- Particulate 10 – 0.07mm



- Particulate 11 – 0.06mm
- Particulate 12 – 0.04mm
- Particulate 13 – 0.04mm
- Particulate 14 – 0.04mm
- Particulate 15 – 0.03mm
- Particulate 16 – 0.03mm
- Particulate 17 – 0.03mm
- Particulate 18 – 0.03mm
- Particulate 19 – 0.03mm
- Particulate 20 – 0.03mm
- Particulate 21 – 0.05mm
- Particulate 22 – 0.03mm
- Particulate 23 – 0.03mm
- Particulate 24 – 0.04mm
- Particulate 25 – 0.05mm

Table 1 Particulate Report

Gaustad: Particulate Concentration - 0.14 mg/kg

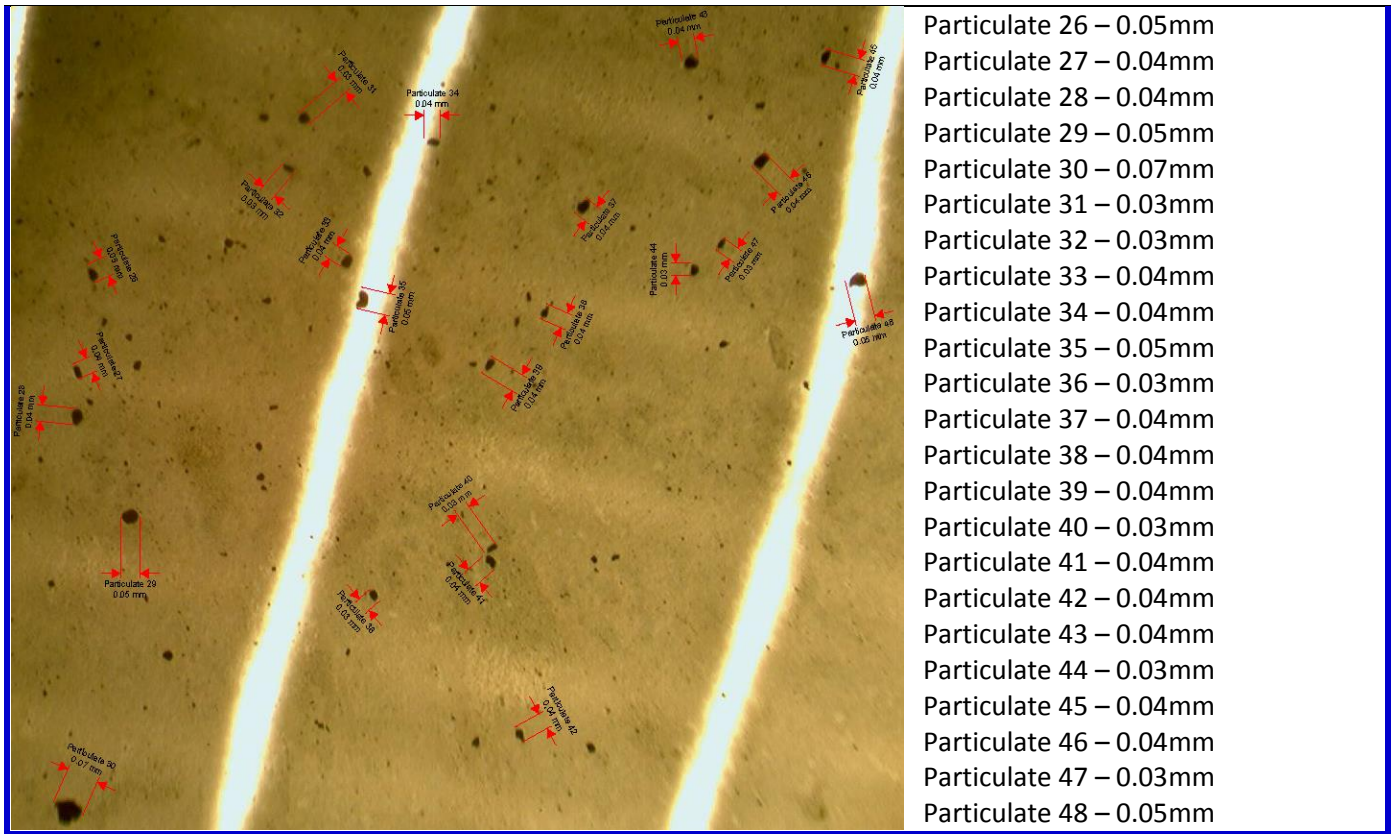


Table 1 Particulate Report

Økern: Particulate Concentration - 0.21 mg/kg



Filter after sampling

This is **particulate filter after sampling**, in which **0.195 mg particulates** are found in **0.950 kilogram hydrogen**. Seven particulates are found and shown below along with images and sizes.

<p>Particulate 1 under polarized light</p>	<p>Particulate 2 under polarized light</p>	<p>Particulate 3 0.06 mm</p>	<p>Particulate 4 0.26 mm</p>	<p>Particulate 5 0.30 mm</p>	<p>Particulate 6 0.15 mm</p>	<p>Particulate 7 0.41 mm</p>
<p>Particulate 1 0.47 mm</p>	<p>Particulate 2 0.51 mm</p>	<p>Particulate 3 – 0.06mm</p>	<p>Particulate 4 – 0.26mm</p>	<p>Particulate 5 – 0.30mm</p>	<p>Particulate 6 - 0.15mm</p>	<p>Particulate 7 – 0.41mm</p>
<p>Particulate 1 (0.47mm) looks metallic under polarized light.</p>	<p>Particulate 2 (0.51mm) looks metallic under polarized light.</p>					

Particulate Concentration Calculation Sheet

Date	WMF (Weight Monitoring Filter) Weight (g)	WMF (Filter#88) (Weight Monitoring Filter) Weight (g)	#129, PORGRUNN:		#130, GAUSTAD:		#131, ΦKERN: 950G - #131, ΦKERN: 950G	
			2.07KG - Filter Weight (g) before Sampling	#129, PORGRUNN: 2.07KG - Filter Weight (g) after Sampling	2.04KG - Filter Weight (g) before Sampling	#130, GAUSTAD: 2.04KG - Filter Weight (g) after Sampling	Filter Weight (g) before Sampling	- Filter Weight (g) after Sampling
2012-10-24time 12.45.58	0.09605		0.09745		0.09773		0.09713	
			0.09745		0.09773		0.09713	
			0.09746		0.09778		0.09714	
			0.09745		0.09779		0.09715	
			0.09746		0.09775		0.09715	
			0.09743		0.09777		0.09717	
			0.09742		0.09777		0.09714	
			0.09745		0.09776		0.09715	
			0.09744		0.09778		0.09716	
			0.09745		0.09779		0.09714	
2012-10-24time 16.18.57		0.09259		0.09754		0.09809		0.09732
2012-11-13time 15.11.08	0.0961			0.09753		0.0981		0.09731
				0.09754		0.0981		0.09736
				0.09754		0.09813		0.09732
				0.09754		0.09812		0.09738
				0.09755		0.09813		0.09732
				0.09754		0.09811		0.09734
				0.09754		0.0981		0.09735
				0.09752		0.09811		0.09737
2012-11-13time 17.55.34		0.09265		0.09748		0.09812		0.09734
Number of Measurement	2	2	10	10	10	10	10	10
Average Filter Weight	0.09608	0.09262	0.09745	0.09753	0.09777	0.09811	0.09715	0.09734
Average Standard Deviation of Weight (g)	3.54E-05	4.24E-05	1.26E-05	1.99E-05	2.22E-05	1.37E-05	1.26E-05	2.38E-05
Relative Standard Deviation of Weight (g)	0.0368%	0.0458%	0.0130%	0.0204%	0.0227%	0.0140%	0.0130%	0.0244%
			Average Weight of Particulates (g) on Filter		Average Weight of Particulates (g) on Filter		Average Weight of Particulates (g) on Filter	
			0.000086		0.000346		0.000195	
Sampling								
Event	Sampling Duration (second)	Inlet Pressure (psi)	Sampling Flow Rate	Hydrogen Sampled (kg)	Sampling Flow Rate	Hydrogen Sampled (kg)	Sampling Flow Rate	Hydrogen Sampled (kg)
Particulate Sampling				2.07		2.4		0.95
H₂ Sampled for Particulate (m³)				25.3		29.3		11.6
Particulate Concentration								
Average Particulate Concentration (mg/kg)			0.042 mg/kg		0.14 mg/kg		0.21 mg/kg	
Average Particulate Concentration (µg/L)			0.0034 µg/Liter		0.0137 µg/Liter		0.0077 µg/Liter	

FTIR measurement of Hydrogen fuel gas impurities at 3 different H₂ refuelling stations in Norway

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1. Objectives

The main objective is to assess the performance of the FTIR system for Hydrogen quality control. The sub-objectives are threefold:

- Demonstrate the quantitative estimation of carbon monoxide concentrations down to 10 ppb with an accuracy of ± 5 ppb.
- Investigate whether other gases, as carbon dioxide, hydrocarbons and water can be measured with the required accuracy.
- Establish the minimum specifications of a FTIR based system and a long path gas cell able to perform the measurements with the required accuracy.

2. Equipment

The instrumentation consists of a FTIR instrument and a long path gas cell.

We have tested 2 different FTIR instruments.

- FTIR Vertex 70 from Bruker Optics.
 - o Resolution: 0.5 cm^{-1}
 - o Spectral range: 500-17000 cm^{-1}
 - o Broadband detectors: DTGS and MCT
 - o Nitrogen flashed optics
- FTIR IFS 66v/S from Bruker
 - o Resolution: 0.1 cm^{-1}
 - o Spectral range: 55-55000 cm^{-1}
 - o Broadband detectors: DTGS and MCT
 - o Vacuum Optics (down to 2 mbar)

The selected gas cell is a 35 m cell from Infrared Analysis which has been acquired specially for this project (see Figure 1)

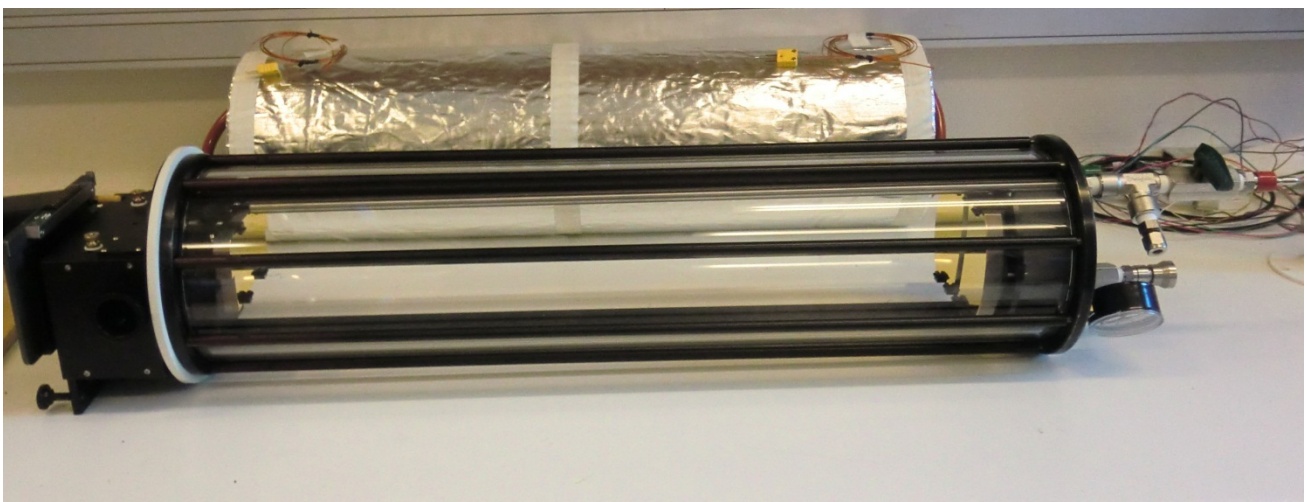


Figure 1: Long path cell and heating jacket

Measurement Gas cell specifications

The long path gas cell has the following specifications:

- Path length: 35 meters
- Body material: Borosilicate glass
- Mirror coating: Protected gold
- Body dimensions: Length, 60 cm; diameter, 12.5 cm.
- Volume: 8.5 liters.
- Window material: Potassium chloride

Others: Two stainless steel plug valves, pressure release valve, compound gauge (0-2 bar abs.) and septum holder for gas mixing.

The heating jacket is temperature controlled and may provide heating up to 200 °C

3. System preparations and tests

Instrument selection

The gas cell was first tested with the Vertex 70 FTIR from Bruker. This spectrometer has a fast scanning frequency, but the instrument optics and the transfer optics of the gas cell must be purged continuously if low levels of carbon monoxide and water have to be measured. Figure 2 is showing the 35 m gas cell mounted in the sample compartment of the Vertex 70 FTIR.

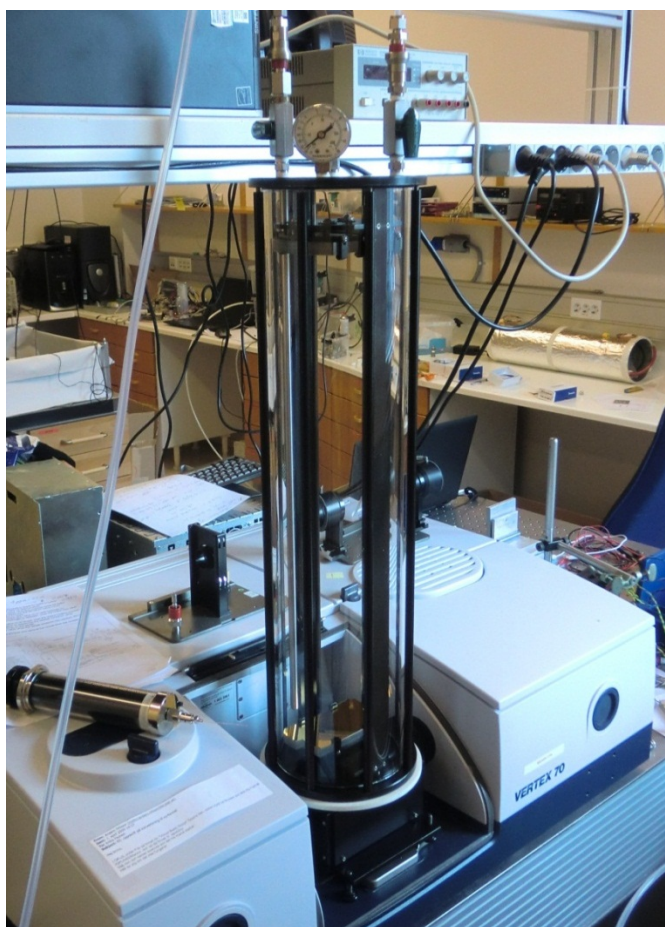


Figure 2: 35 m gas cell mounted in the sample compartment of the Vertex 70

When averaging over 250 scans at a resolution of 1 cm^{-1} , we were able to detect 100 ± 50 ppb CO with continuous nitrogen purging and a DTGS detector. The signal to noise ratio (SNR) was about 4000 RMS (800 peak to peak)¹.

Using a liquid nitrogen cooled MCT detector can usually enhance the signal to noise ratio (SNR) by a factor of 5 or better, but this would only make us able to detect 20 ± 10 ppb CO.

Furthermore it appeared that it could become difficult to detect low concentrations of CO₂ and H₂O without a high nitrogen flow through the optics and/or a better evacuation system for the sampling compartment.

We therefore decided to test a FTIR instrument with vacuumed optics.

Professor Claus Jørgen Nielsen, at the Chemical Institute of the University of Oslo, made the IFS 66v/S FTIR instrument available for our use and has contributed with its FTIR expertise.

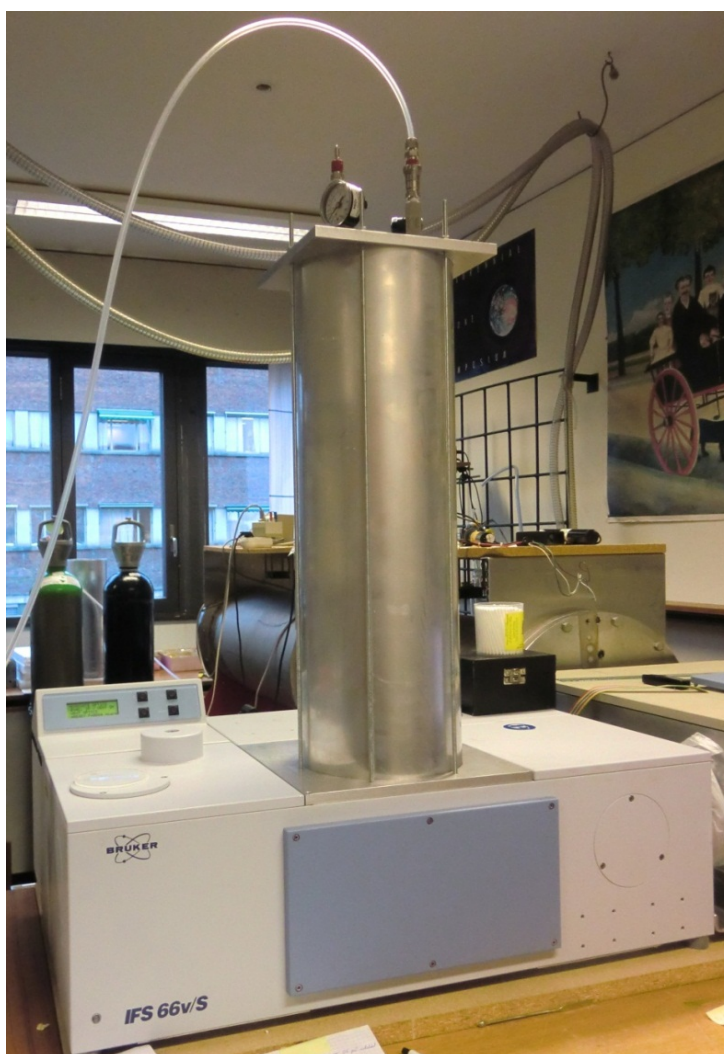


Figure 3: 35 m long path cell with vacuum housing mounted in the IFS 66v/S

The IFS 66v/S FTIR instrument is a relatively old brand with vacuumed optics and high spectral resolution. Its main weakness was the MCT detector which was negatively affected by a non-original focusing.

¹ During the adjustment of the gas cell inside the IFS 66v/S it was found that the cell mirrors were not optimally adjusted and that the throughput could easily be enhanced by about 50%.

To make use of the vacuuming capability of the IFS 66V/S with the 35 m gas cell, we had to build a sealed housing around the gas cell and the sampling compartment of the FTIR. The gas cell, with its vacuum sealed housing, mounted in the sample compartment of the IFS 66v/S is shown in Figure 3.

It was unfortunately not possible to integrate the heating jacket inside the sealed housing of the gas cell.

This instrument configuration has been used for both the collection of calibration spectra and the acquisition of absorption spectra for the hydrogen samples collected at the Hydrogen station of Gaustad, Økern and Porsgrunn.

It should be noted that the Vertex 70 FTIR or equivalent may still be an option if an efficient arid zone is fitted between the transfer optics of the cell and the instrument.

System tests

We have used a MCT detector (Mercury-Cadmium Telluride) and obtained a signal to noise ratio (SNR) of nearly 18000 RMS (amplitude ratio of 3200) at a wavenumber of 2600 cm^{-1} . This is 40% below the expected SNR. This sensitivity shortage was probably due to a non-optimal evacuation of the detector Dewar and the previous mentioned non-original focusing mirror. In comparison we did obtain a SNR of nearly 6000 for the same conditions with the DTGS detector.

An optimal, healthy, MCT detector will therefore enhance the Limit Of Detection (LOD) and the resolution significantly.

Another observation we made was a small, but significant, instrument baseline drift and a substantial baseline deformation that we later found was due to the incomplete evacuation of the liquid nitrogen Dewar. These discrepancies have complicated the processing of the baseline compensation/correction and introduced an additional inaccuracy.

4. Measurements

Preliminary vacuuming and flashing

Before starting the measurements, the cell must be vacuumed and optionally flashed with nitrogen. This procedure depends highly upon the quality of the vacuum equipment. Optimally, the nitrogen flashing is not absolutely required when a high capacity vacuum pump and a moderate heating of the gas cell are applied over a significant number of hours (e.g. over one night). However, since our vacuum pump was not very efficient and the gas cell could not be heated, we decided to perform three subsequent vacuuming-flashing cycles with each vacuuming period lasting 1 hour.

In a future commercial system the vacuuming procedure will be significantly enhanced by heating up the gas-cell moderately in order to release the remaining gas molecules sticking more or less to the cell's walls.

Medium quality Nitrogen 4.6 (purity 99.996%) was used during the flushing procedure.

Background spectra

The quality of the background spectra is decisive to obtain a reliable and low noise measurement. The background spectra may be collected with either a vacuumed or nitrogen filled cell. A vacuumed cell is preferred but is only useful when high vacuum ($\leq 2\text{ mbar}$) can be confirmed in both the cell and the optics (transferring optics and FTIR optics). In the present case we had 2 mbar inside the optics, but unfortunately

we were not able to validate a vacuum better than 20 mbar in the cell because of a relatively inefficient vacuum pump and a low resolution manometer².

The background spectra have therefore been collected with the cell filled with high quality Nitrogen 6.0. (Purity $\geq 99.9999\%$, $H_2O \leq 1\text{ppm}$ hydrocarbons (C_nH_m) $\leq 100\text{ppb}$, $CO \leq 100\text{ppb}$, $CO_2 \leq 500\text{ppb}$). This quality is necessary when low concentrations of H_2O , CO , CO_2 and hydrocarbons are to be estimated.

Calibration spectra

Calibration spectra were collected for carbon monoxide, methane, ethane and propane. Only two concentrations for each gas species have been used, since the absorbencies were expected to be low and a linear behavior was therefore ensured.

The following calibration spectra have been collected and used in a PLS model:

- Carbon monoxide (CO): 100 ppb and 400 ppb
- Methane (CH_4): 100 ppb and 400 ppb
- Ethane (C_2H_6): 200 ppb and 1 ppm
- Propane (C_3H_8): 100 ppb and 400 ppb (doubtful quality)

For gas with overlapping absorption features (e.g. hydrocarbons like methane, ethane, propane and heptane) it is recommended to collect calibration spectra of mixtures in order to generate reliable calibration models that can be used to predict the individual components. Such gas mixing process must be performed with high accuracy and is time-consuming. Unfortunately, we had not enough time and resources to prepare the mixtures of hydrocarbons and collect their calibration spectra.

However, as long as the total absorbance is low (<0.5) and the calibration spectra of single gases are of high quality it is possible to handle mixture by adding scaled and pressure corrected calibration spectra from different gases to each other. One should be careful and remember that although total simulated pressure is correctly adjusted to 1 bar, scaling also affect the final noise. Furthermore, addition of spectra (scaled or not) from different sources is impaired by instrument transfer function differences and should be avoided.

However, this was not a real hindrance for the estimation of methane concentration because of the clear line structure of its absorption spectrum and the possibility to select a spectral range outside the main C-H stretching band. Ethane, propane and heptane were more affected by this omission, but the concentration of Ethane in the Porsgrunn sample was so high that a measurement accuracy of $\pm 15\%$ could be confirmed.

The calibration was performed with a relatively low precision manometer and without controlling the temperature which, however, however not fluctuating by more than $5\text{ }^\circ\text{C}$. Furthermore, the non-optimal dilution of the reference gases used to generate the calibration samples may introduce an inaccuracy of about $\pm 15\%$. As we can assume that this inaccuracy is normal distributed we can conclude that the standard deviation for the calibration gas concentration is not far from 15%.

An optimal procedure for calibration gas preparation, like the one we have used for NASA, can bring this value down to 2.5%. Such a procedure is tedious and will only be used for a final calibration.

² We have subsequently acquired a high capacity vacuum pump and a Pirani vacuum manometer, and have tested the 35m gas cell down to 1 mbar. The next calibration will therefore be simplified and much more accurate.

Synthetic spectra generated from HITRAN2008 by SpectralCalc software (SC) have been used to model carbon dioxide (CO₂) and water, while spectra from the Infrared Analysis database have been used for Heptane and Acetone.

Noise in the collected spectra is a limiting factor for both calibration and sample measurements. A main purpose of the PLS modeling is to assign optimal weights on the measured values to balance information against noise. The synthetic spectra must therefore reflect the actual noise in the collected spectra. We have therefore added spectral dependent, but stochastically independent noise, generated by the baseline corrected ratio of two subsequent background spectra to the simulated spectra.

Acquisition of spectra

The acquisition of the absorption spectra has been performed at 1 cm⁻¹ (unapodised) and averaged over 250 scans. It should be noted that the real apodised resolution is in reality 1.25 cm⁻¹.

5. Data analysis

The analysis has been performed on two different platforms:

- Essential FTIR software (Partial Least Square – PLS modeling and prediction)
- Our one Matlab program (Baseline correction, PLS modeling and prediction).

To simplify PLS modeling we have assumed relatively low absorbencies (<0.5) and concentrations < 1 ppm.

The two platforms have issued nearly the same results and the results from the Essential FTIR PLS prediction are listed in Table 1.

	CO	CO2	H2O	Methane	Propane	Ethane	Heptane	Acetone
<i>LOD</i>	<i>5 ppb</i>	<i>100 ppb</i>	<i>1 ppm</i>	<i>5 ppb</i>	<i>10 ppb</i>	<i>20 ppb</i>	<i>10 ppb</i>	<i>40 ppb</i>
Gaustad	12 ppb	< 500 ppb	< 1 ppm	8 ppb	<10 ppb	< 20 ppb	< 10 ppb	< 40 ppb
Porsgrunn	21 ppb	3150 ppb	< 1 ppm	22 ppb	45 ppb	460 ppb	< 40 ppb	< 80 ppb
Økern	11 ppb	< 500 ppb	< 1 ppm	5 ppb	<10 ppb	< 20 ppb	< 10 ppb	< 40 ppb

Table 1: Measurement results based upon our own calibration (the value in italic are doubtful)

Only carbon monoxide, carbon dioxide and methane have been detected above their estimated Limit of Detection (LOD) in the samples from Gaustad and Økern. Furthermore, the 5 ppb methane concentration found in Økern's sample is doubtful because this sample was too small, resulting in a cell pressure of only 650 mbar (absolute pressure) which had to be corrected for during the analysis.

Additionally; carbon dioxide, ethane and propane have been detected in the Porsgrunn's sample. Carbon monoxide is easy to estimate, methane is relatively easy although other hydrocarbon were interfering and the sensitivity of our MCT detector is lower at 3000 cm⁻¹ than at 2000 cm⁻¹. Figure 7 is showing the absorption by methane from the Porsgrunn's sample in the range 3060-3110 cm⁻¹.

Ethane is absorbing through a broad absorption range (2800-3100 cm⁻¹) and has many narrow lines superimposed the broad absorption (see Figure 5 where the ethane reference is matching nicely with the absorption in our sample). The broad feature means that other gases, absorbing in the same range, like

propane and heptane (also shown in the same figure) can be partially masked and difficult to estimate separately with non-optimal modeling. This uncertainty/doubtfulness is pertinent regarding the concentration of propane. It should be noted that a more thorough calibration and modeling will tackle this superposition.

The reliability of these results has been checked, for Carbon monoxide (CO) and methane (CH₄), against synthetic spectra generated from the HITRAN-2008 database by making use of the SpectraCalc software (SC). Carbon monoxide, carbon dioxide, methane, propane and ethane have also been checked with spectra from the Infrared-Analysis database (IR-A). The results obtained when applying these spectra are listed in Table 2

	CO <i>ppb</i>		CO ₂ <i>ppb</i>		Methane <i>ppb</i>		Ethane <i>ppb</i>	Propane <i>ppb</i>
	SC	IR-A	SC	IR-A	SC	IR-A	IR-A	IR-A
Gaustad	13	18	< 500	< 500	<50	<10	<50	<40
Porsgrunn	23	32	3150	1400	20	17	510	<40
Økern	12	16	< 500	< 500	<50	<10	<50	<40

Table 2: Comparison with results estimated with calibration spectra obtained from HITRAN2008 (SC) and Infrared-Analysis database (IR-A)

Inaccuracy related to the data-analysis and the non-optimal calibration task may also be significant when estimating hydrocarbons which are largely overlapping each other's in the 3000 cm⁻¹ C-H stretching band. This last remark does not apply for methane which usually is easy to quantify using prediction based on PLS-modeling because of its fine line absorption spectrum (see Figure 7).

All the remarks about accuracy mean that an overall inaccuracy of up to ± 30% is undoubtedly realistic a decade above the LOD for some gas species. This inaccuracy will definitely be lowered to less than 10% by a thorough calibration making use of a precision manometer, temperature monitoring and professional gas dilution equipment.

Our calibration with 100 ppb CO, together with a 100 ppb CO spectrum from IR-A database and the synthetic spectrum for 100 ppb CO generated with the HITRAN2008 database are plotted in Figure 4. It is easy to see that our calibration spectrum is comparable with the HITRAN2008 simulation, while the spectrum from IR-A has significant lower amplitude. The comparison also appears for methane estimation in Figure 7.

Characteristics	ISO/TS 14687-2 Type 1, Grade D
Hydrogen Fuel Index	99.99%
Total hydrocarbons (C1 basis)	2ppm
Water (H ₂ O)	5 ppm
Carbon dioxide (CO ₂)	2 ppm
Carbon monoxide (CO)	200 ppb

Table 3: Fuel specification according to ISO/TS14687-2

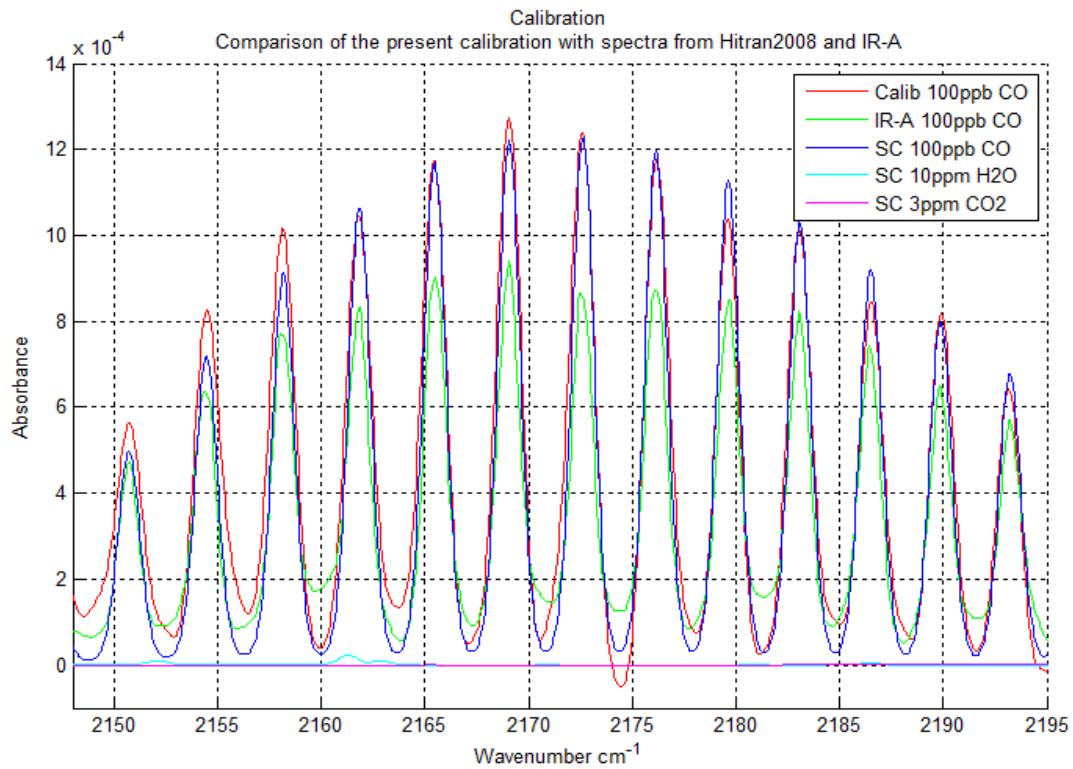


Figure 4: Comparison of the present calibration for 100 ppb CO (256 scans) with 100 ppb CO spectrum from Infrared-Analysis's database and 100 ppb CO synthetic spectrum generated by the SpectraCalc software from the HITRAN2008 database.

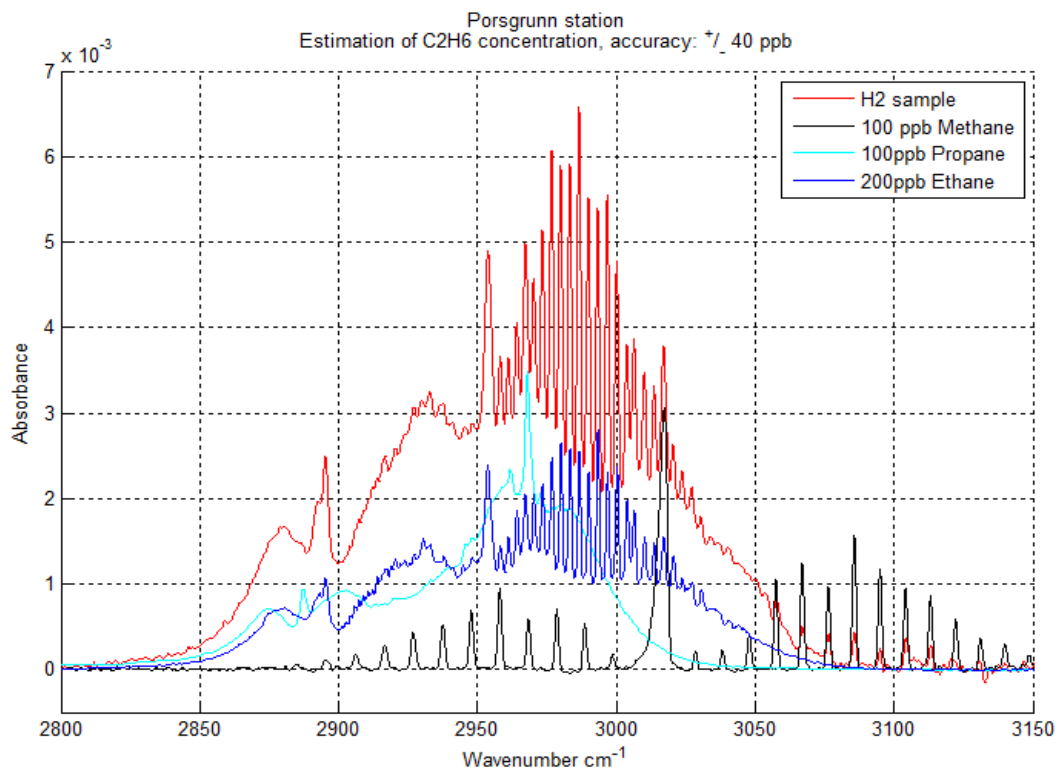


Figure 5: Hydrogen sample from the Porsgrunn's station and main hydrocarbon's absorbency associated with the fundamental C-H stretching vibration around 3000 cm^{-1}

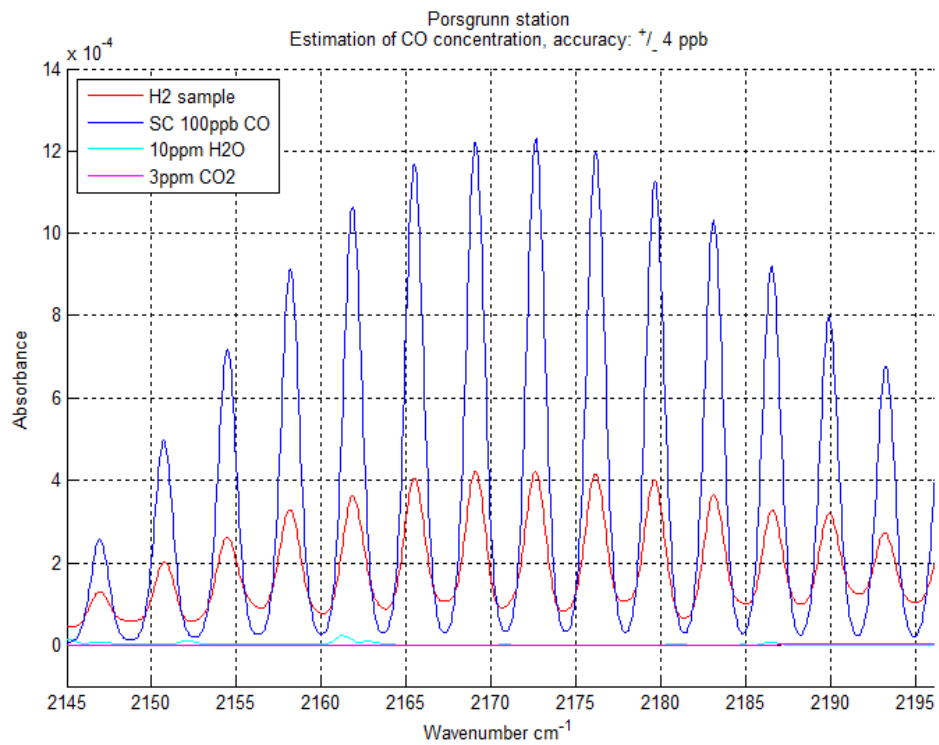


Figure 6: Selected spectral range and interfering gases for the estimation of CO. (23 ppb CO)

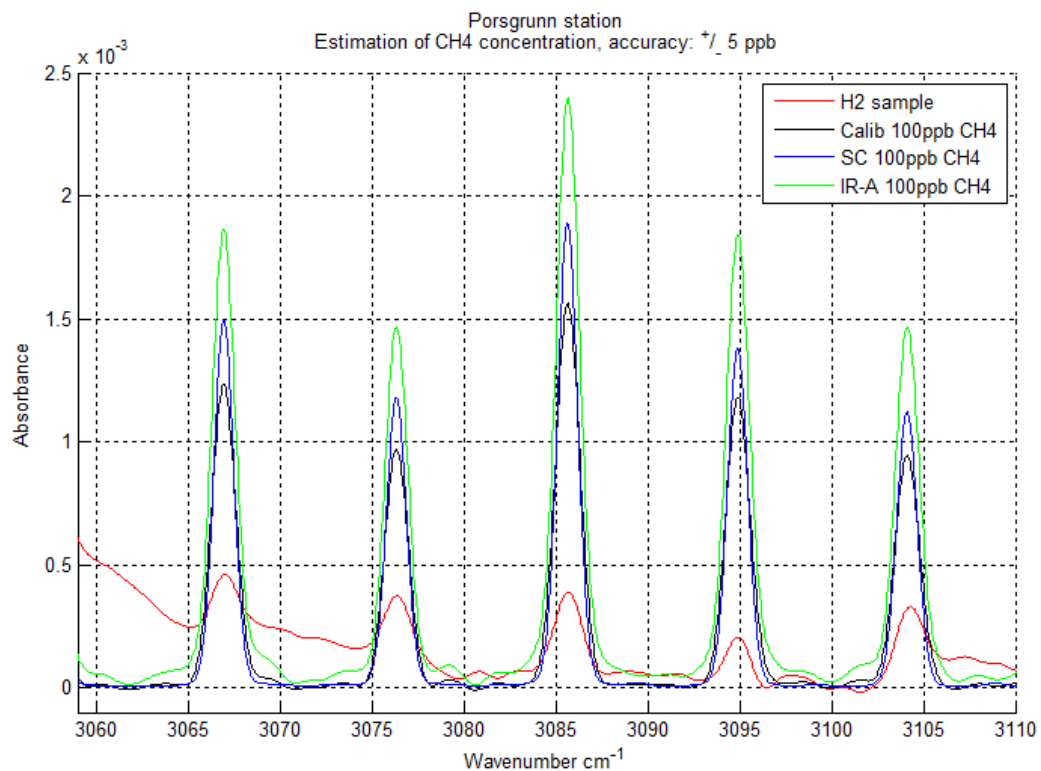


Figure 7: Selected estimation range for CH4. The 100ppb calibration spectrum (black) has been averaged through 1000 scans. The reference spectrum from the IR-A database and the synthetic generated spectrum from the HITRAN database are also plotted.

6. Required system specifications

The following specifications should be followed when designing a FTIR based system able to perform the Hydrogen Quality Control with the requested specifications (see Table 3)

FTIR instrument

- Resolution: 1 cm^{-1}
- Spectral range: $600\text{-}4000 \text{ cm}^{-1}$ (this is standard range for commercial FTIR)
- Broadband detectors: Liquid nitrogen MCT (or Stirling Cycle Cryo-cooler)
- Vacuum Optics: Preferably down to 2 mbar for very low concentrations of CO₂ and H₂O.
Vacuum optics is not required for carbon monoxide and hydrocarbons. A properly sealed arid zone flashed with high quality nitrogen will also be sufficient if the SAE limits of 5ppm H₂O and 2 ppm CO₂ are maintained.

Gas cell

A 10m long path cell will be sufficient for CO, CO₂, CH₄ and H₂O. The cell should sustain high vacuum (≤ 2 mbar) and preferably include a heating function in order to ensure efficient removing of gases. Even hydrocarbons could probably be measured with a 10 m long path cell with heating functionality and high efficiency vacuum equipment for purging.

Vacuum pump

A high efficiency vacuum pump and a high precision pressure gauge are required.

Software

The PLS-modeling software must be optimized for the Hydrogen Quality Control task and instrument dependent variables must be considered to ensure system robustness.

7. Conclusion

We have demonstrated the quantitative estimation of carbon monoxide concentrations down to 5 ppb with an accuracy of ± 4 ppb (conservative estimation). We have also shown that both the limit of detection and accuracy can be significantly enhanced by making use of simple improvements.

We were also able to quantify relatively low concentrations of carbon dioxide, methane and ethane. A thoroughly scrupulous calibration of hydrocarbons like propane, heptane, acetone and others will absolutely allow us to quantify these species with the accuracy required for Hydrogen Quality Control (QC).

Water may also be measured with the required accuracy if efficient vacuuming equipment is used and the optics parts of the system are nitrogen purged continuously or a vacuum FTIR is used.

A lot of work remains, especially concerning the calibration procedure and the PLS-modeling, before an optimal and robust quantification of the target gases for QC can be routinely quantified with a commercially available FTIR instrument. But this work is manageable and can lead to a commercially feasible cost for hydrogen quality assurance (QA).

Furthermore, if CO is selected as a canary constituent and the concentration of CO for the H₂ fuel specification is maintained at 200 ppb (or even 100ppb), then the calibration and the PLS-modeling will be

tremendously simplified. The system requirements, and by this the instrumentation cost will also be lowered significantly.

Memo

Hydrogen Sampling equipment

PERSON RESPONSIBLE / AUTHOR

Alain Marc C Ferber

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CLASSIFICATION

Restricted

1 Background

At the beginning of the H2-moves project we realised that special equipment was necessary to collect hydrogen samples from Hydrogen station. We found that the best way to do this was to design and order a sampling system with the same receptical as the one used for the filling of the car's reservoir and able to withstand the working pressure of 700 bar. This sampling system should also be able to be flashed with nitrogen for security reasons and flashed with hydrogen from the station for cleanliness of the sample.

2 Design of the sampling system

We have tried to order the system from Svafas i Stavanger and Dynetek in Denmark, but they were both unable to deliver the sampling system for 700 bar.

The system required by Sintef is shown in Figure 1.

We have then contacted GHR in Germany which was ready to cooperate with Dynetek Europe to build our sampling system.

They had, however, to modify our layout and used a single side opening cylinder. GHR has built the system according to the design shown in Figure 2.

Sampling cylinder for high pressure H2 tank

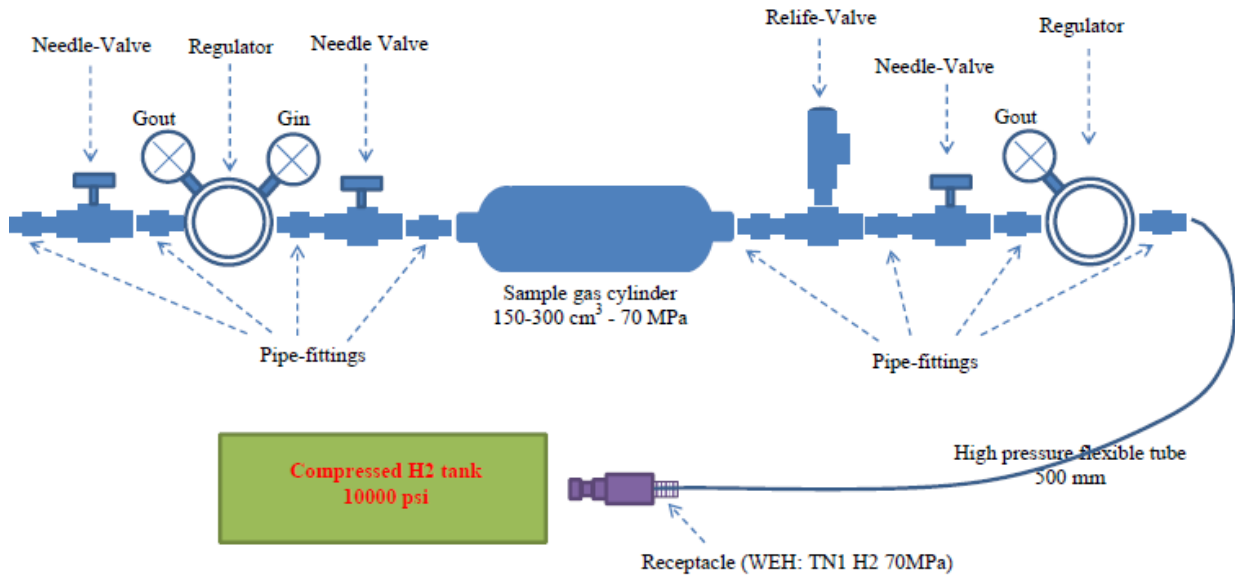


Figure 1: High pressure sampling system (Sintef's design)

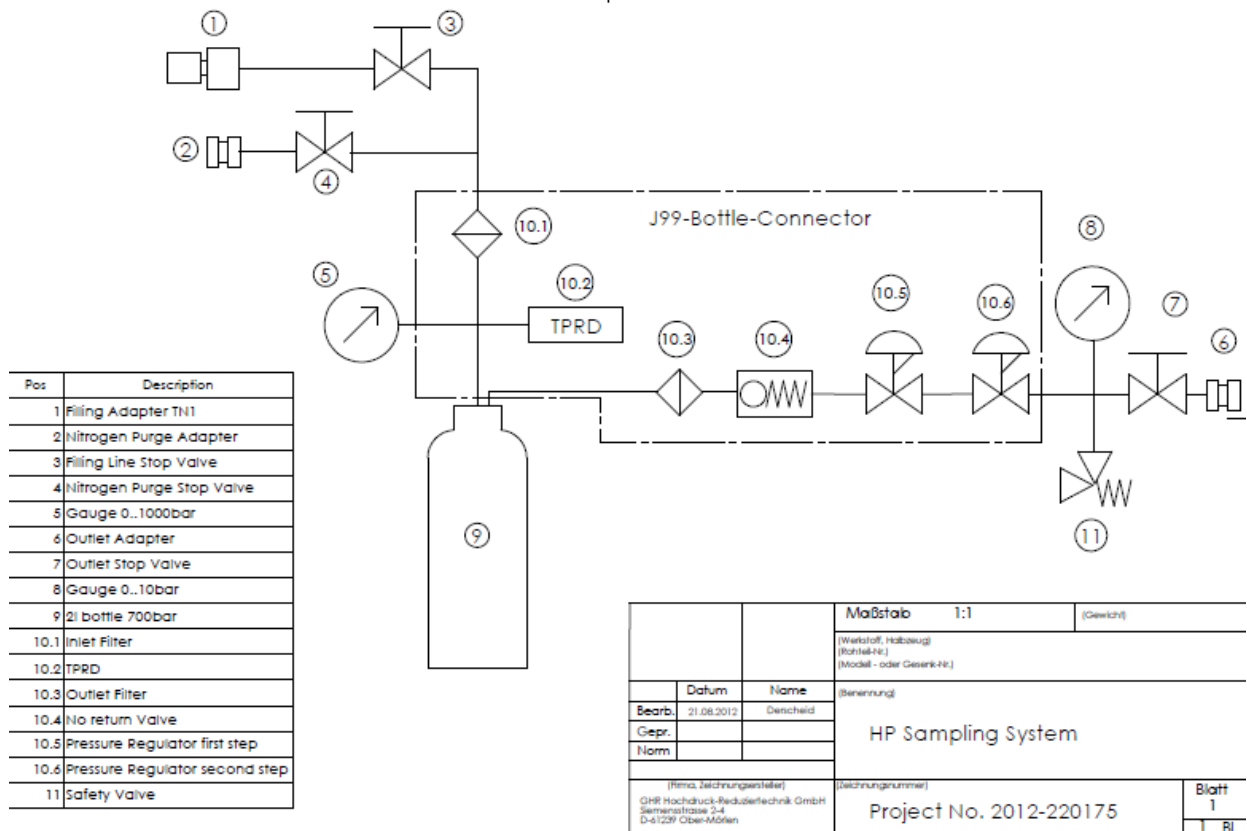


Figure 2: High pressure sampling system (GHR's update)

3 Test of the sampling system.

We have tried to test the sampling system at the Økern hydrogen station, but the responsible for the station was concerned about the aptitude of the system to withstand the pressure of 700 bar and was questioning about the system's ability to survive the filling procedure.

The main concern was the lack of documentation about the system specification and certification. We have now recovered test information and certifications from the cylinder manufacturer and are waiting for additional documentation and certification from GHR.

Another important concern was the heat-transfer issue. Hydrogen is undergoing a temperature increase during fast tank-filling processes. This because hydrogen exhibits a reverse Joule-Thomson effect at room temperature, i.e., throttling processes from a high pressure stationary tank to a tank being filled cause heating of the gas instead of cooling.

A third concern was related to the pressure pulse used for measuring the initial vehicle tank pressure and the available volume in the tanks. This pulse is very short ($< 1s$) but is completed at 875 bar and may represent a mechanical chock for the system. During this time, the pressure at the nozzle should not be less than 6.7 bar and not exceed 670 bar. The recommended fuelling process is drafted in Figure 3

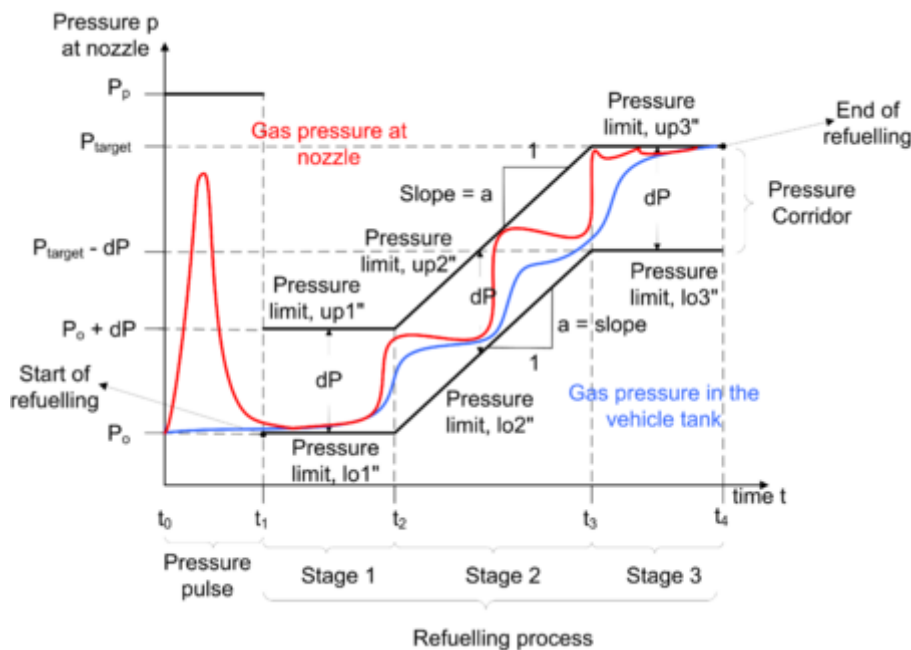


Figure 3: Fuelling process (with "pressure corridor" and start pulse) which satisfy the fuelling protocol.

The system has therefore only been tested at lower pressure (60 bar) with manual control of the station output pressure and, during this test, we have found that hydrogen flashing was not optimal and should be enhanced by installing an additional high pressure regulator and a stop valve between the input valve and the main regulator.