

FCH-JU:HyCoRA

# Performing hydrogen fuel quality control - sampling strategies and challenges

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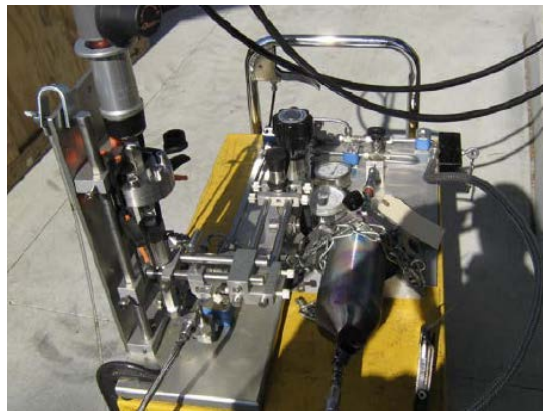
Brussels, 2014-09-30

# Outline

- Previous experience: H<sub>2</sub>Moves Scandinavia
- Prevailing standards relevant for Quality Control
- Requirements for quality control
  - Analysis
  - Gas cylinders
  - HRS maintenance schedule
- Strategy for sampling campaigns in HyCoRA
  - Availability of HRS's
  - Feedstock
  - Sampling instrumentation

# Previous experience with H<sub>2</sub> fuel QC

- H<sub>2</sub>Moves Scandinavia project 2012
- Rented sampling instrumentation from Smart Chemistry (.us)
  - Manual HRS safety override required
  - Venting of hydrogen through 2x 1/2" tubes
  - Sampling at 350 bar only



## H2Moves Scandinavia results (D7.4/PU)

Constituent	Tolerance	Porsgrunn	Gaustad	Økern
Total Hydrocarbons (C1 basis)	2	0.80	0.035	0.049
Nitrogen	100*	<b>2800</b>	7.7	< 5
Argon	100*	0.77	< 0.5	1.9
Carbon Dioxide	2	<b>3.3</b>	< 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 <sup>-1</sup>	0.042	0.14	0.21
Number of particles		11	48	7
Hydrogen Fuel Index	99.97 %	<b>99.7195%</b>	99.9992 %	99.9998%

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

- Gaustad: On-site electrolysis (alkali)
- Porsgrunn: Hydrogen by-product (chlorine-alkali)
- Økern: Trucked-in electrolytic (alkaline)

# Protocols and standards relevant for Quality Control

## Refuelling

- [SAE J2601](#) Fueling Protocols for Light Duty Gaseous Hydrogen Surface Vehicles
- [SAE J2600](#) Compressed Hydrogen Surface Vehicle Fueling Connection Devices
- [SAE J2799](#) Hydrogen Surface Vehicle to Station Communications Hardware and Software

## Sampling

- [ASTM D7606](#) Standard Practice for Sampling of High Pressure Hydrogen and Related Fuel Cell Feed Gases
- [ASTM D7650](#) Standard Test Method for Sampling of Particulate Matter in High Pressure Hydrogen used as a Gaseous Fuel with an In-Stream Filter

## Analysis

- [SAE J2719](#) Hydrogen Fuel Quality for Fuel Cell Vehicles
- [ISO 14687-\[2,3\]](#) Hydrogen Fuel – Product specification

# Analysis

Species	Tolerance ( $\mu\text{mol/mol}$ )	Methods
H <sub>2</sub> O	5	JIS K0225, ASTM D-7649, KIS K0123, NPL AS64, JIS K0225, ASTM D-7653, JIS K0117
THC (C1)	2	ASTM D7675, JIS K0114, JIS K0117
O <sub>2</sub>	5	JIS K0225, ASTM D-7649, NPL AS64, ASTM D7607
He	300	ASTM D-1945, JIS K0114, JIS K0123
N <sub>2</sub> , Ar	100	ASTM D-7649, JIS K0123, JIS K0114
CO <sub>2</sub>	2	ASTM D-7649, JIS K0123, JIS K0114, ASTM D-7653, JIS K0117
CO	0.2	JIS K0114, ASTM D-7653, JIS K0117
Total Sulphur	0.004	JIS K0127, ASTM D-7652, JIS K0114, NPL AS64
HCHO	0.01	JIS K0124, JIS K0114, ASTM D-7653, JIS K0117
HCOOH	0.2	ASTM D-7550, JIS K0127, ASTM D-7653, JIS K0117
NH <sub>3</sub>	0.1	JIS K0127, ASTM D-7653, JIS K0117
Total Halogenated Pariculate	0.05 1 mg/kg	JIS K0101, K0127, WK34574, WK23815 ASTM D7651, JIS Z8813

FTIR

GC-MSD

IC

GC-PDD

# Sample cylinders

- Analysis volume requirements
  - Pre-concentration
- Cylinder lining requirement
  - Feedback from VSL, NPL and Linde
  - Different types of linings
    - Spectraseal
    - Sulfinert
    - Silonite
- All sample loops and transfer lines lines

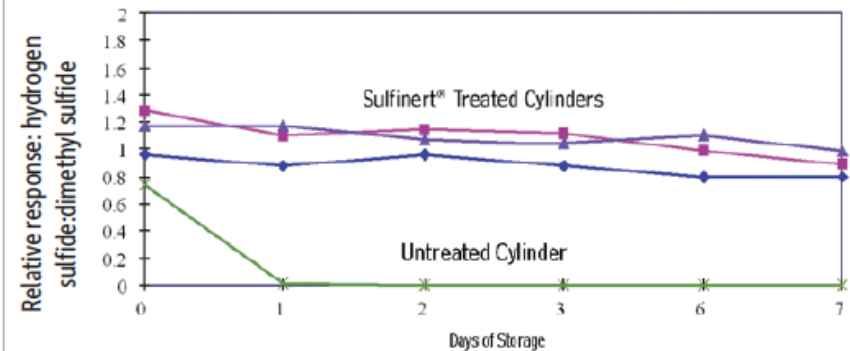


Silonite® High Pressure Cylinders



Sulfur compounds are stable in Sulfinert® treated stainless steel systems.

17 ppbv hydrogen sulfide in 500 mL cylinders



# HRS maintenance

- $N_2$  not easily flushed out of the system
  - Vacuum more efficient
- SS coating degradation
  - $C_4Cl_4Cl_6$  isomers found (halogenate)
- Particulates
  - Introduced by new parts
  - SS coating
  - Removed by purging
- Important to know maintenance schedule

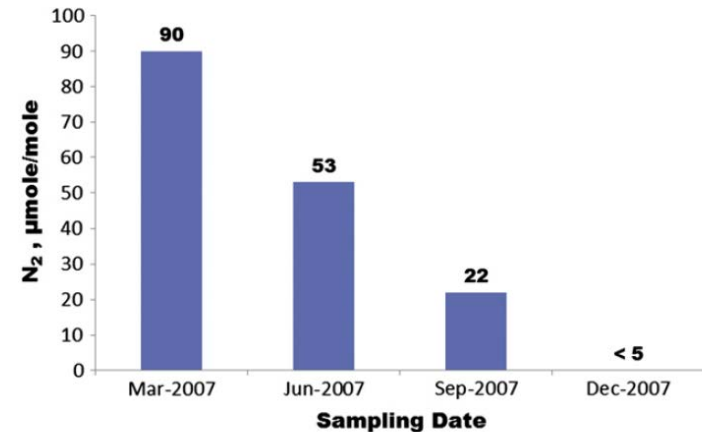
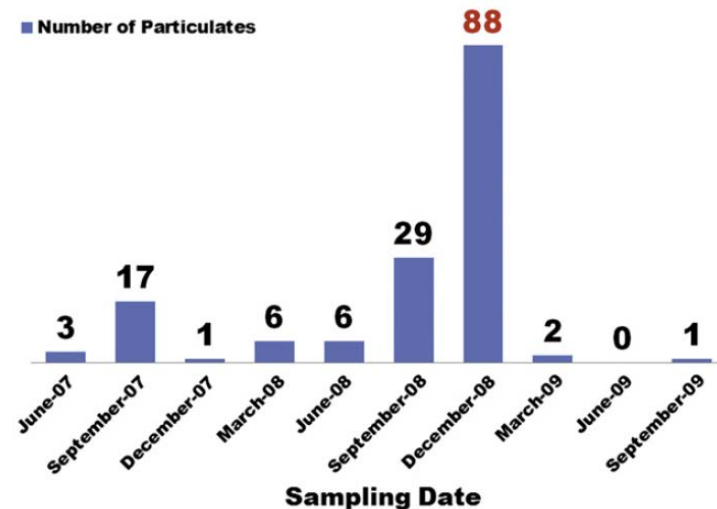


Fig. 10 –  $N_2$  in Station #7  $H_2$ .



Hsu, J.P., Int. J. Hydrogen Energy, 37 (2012) pp. 1770-1780



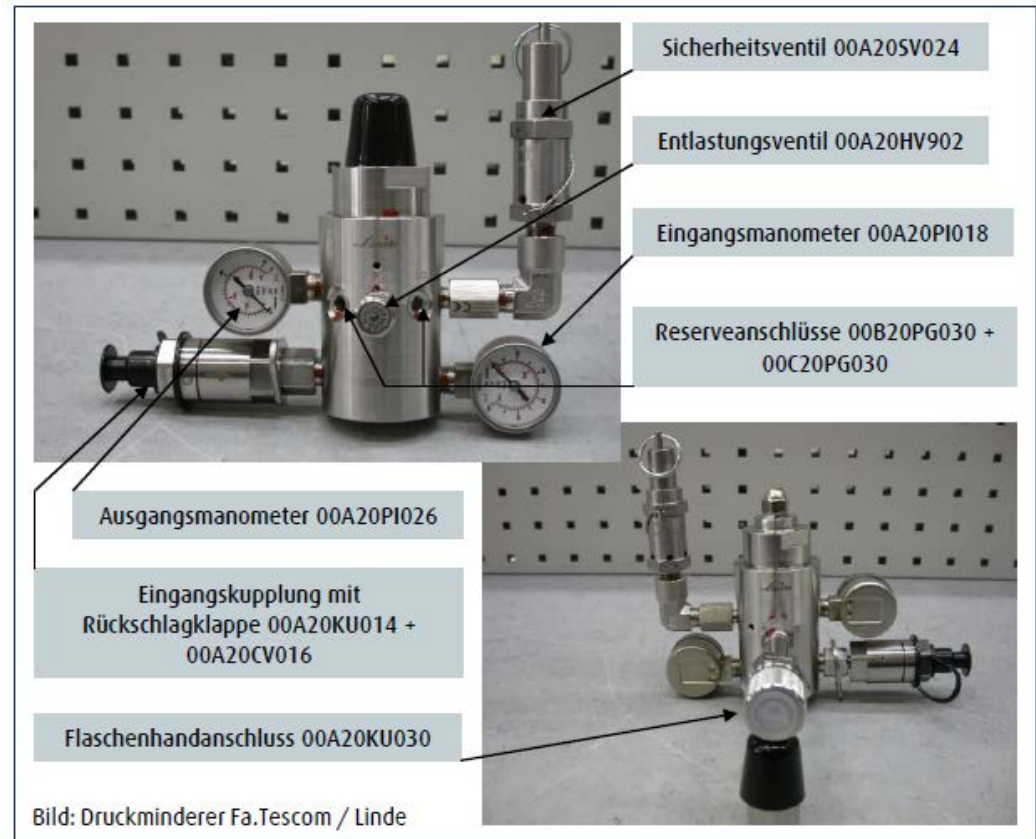
# Strategy for sampling in HyCoRA

- Assistance and manual HRS safety override should be avoided
- Commercial instrumentation for 700 bar sampling
  - Particle sampler (HyDAC)
  - Gas sampler (Linde)
- Health safety and environment
  - Use FCHEV to avoid venting of hydrogen
  - Large sample cylinders (10 L Al Spectraseal)
    - Extra volume for pre-concentration efforts
    - Single ended
    - SS valve



# Linde Gas sampling instrumentation ('Qualitizer')

- Max 875/160 bar reduction valve
- Non IrDA: 600 bar max
- 10L @ 160 bar sample per FCHEV
- Safety release valve
- Bleed valve
- No purging required
  - 10 mL / 1600 L = 5 ppm N<sub>2</sub>
- Purge evaluation
  - Test pulse > bleed
- Instrument tested by CEP in .de
- Delivery in October



# Qualitizer



# Particle sampler

- HyDAC PSA-H70
- Purging by test pulse
- Filter installation requires clean room
  - Portable glove box
  - HyDAC portable laboratory service
- Received and tested by SINTEF



# HRS selection

- HRS available to public
- FCHEV vehicle range
- Hydrogen feedstock
  - SMR (Berlin/Hamburg)
  - Pyroreforming of glycerine (Berlin)
  - By-product (Norway)
- Assistance from CEP offered
  - Sampling
  - FCHEV vehicles (Oslo-Kiel ferry does not allow FCHEV)



# HRS selection

- Berlin (5)
  - Bio/LH<sub>2</sub>
  - SMR
  - Electrolysis
- Hamburg (3)
  - SMR
  - Electrolysis
- Subject to change
  - Maintenance schedule



# Challenges summarized

- Sampling of low tolerance constituents uncertain
  - Sulphur, Halogenates
- Purging of sampling instrumentation
- Sampling rate
- FCHEV vehicle H<sub>2</sub> consumption
  - Efficiency of sampling
- Particle sampling
  - Clean room requirement for filter holder assembly
- HRS technologies are different
  - Adaptation required



Thank you!

