HyCoRA – Hydrogen Contaminant Risk Assessment Grant agreement no: 621223

Deliverable 3.1 Hydrogen sampling unit tested and certified

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Confidentiality: Public

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Summary

Commercial instrumentation has been acquired for sampling of gas and particles from HRS at 700 bar. These instruments require proper training of operators in order to ensure HSE compliance. Sampling is performed by using a FCHEV as hydrogen sink. Sampling could be performed from public HRS without assistance from HRS operator.

SINTEF has received and tested the particulate sampler from HYDAC. The gas sampler from Linde is manufactured and SINTEF will receive it with operator training during a test sampling from Økern HRS on October 31. 2014.

The instruments will be deployed for the first sampling campaign in Germany in November 2014 by assistance from CEP.

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Contents

1. Introduction

Sampling of gas and particulate samples from 700 bar Hydrogen Refuelling Stations is challenging mainly due to the high pressure involved. The fuelling protocol[1] dictates a test pulse of 875 bar to precede fuelling. This implies that sampling instrumentation must be pressure rated for 1034 bar (15,000 psi). These requirements limit, and severely increase the cost of, hardware to be used for sampling.

ASTM D7606[2] describes a sampling apparatus for hydrogen fuel at 700 bar. The apparatus purges large amounts of hydrogen through the sampler and the sample gas cylinders to an exhaust gas tube prior to a sample being collected. SINTEF has used this kind of instrumentation previously[3] for HRS quality monitoring. Whereas the purging option is beneficial, there is a HSE aspect of venting large amounts of hydrogen from the HRS.

At the beginning of the HyCoRA project, a commercially available hydrogen gas sampler from Linde had become available for purchase. This instrument is allows for sampling of hydrogen from the HRS without safety override or excessive venting of hydrogen from purging. The sampler is principally a T-piece where a sample is collected while a FCHEV is refuelled. A requirement for this strategy is of course the availability of a fuel cell vehicle that is close to empty on fuel.

ASTM D7650[4] describes a filter holder for sampling of particulates from HRS 350 bar nozzles. SINTEF has successfully applied this instrument on HRS QC measurements previsously[3]. At the start of the HyCoRA project, a 700 bar sampler was available from HYDAC. This instrument was acquired for use in HyCoRA sampling campaigns.

2. Health, Safety and Environment

The safety data for hydrogen is well documented. Of particular interest to this application is the fact that hydrogen has a negative Joule-Thompson coefficient. To counteract the

[1] SAE J2601, Fueling protocols for light duty gaseous hydrogen surface vehicles.
[2] ASTM D7606, Standard practice for sampling of high pressure hydrogen and related fuel cell gases.
[3] FCH-JU H2Moves Scandinavia (Grant Agreement NO 245101), Deliverable D7.4 Results from H2-gas quality monitoring at various HRSs in the Oslo area.
[4] ASTM D7650, Standard test method for sampling of particulate matter in high pressure hydrogen used as a gasesous fuel with an instream filter.

temperature increase by hydrogen relaxation, hydrogen is often pre-cooled down to -40 °C in order to achieve fast (down to 3 minutes per vehicle) fuelling rates. Freezing on sampling adapters while sampling hydrogen does occur, and this could affect the functionality of connections (quick connects and nozzle adapters).

High-pressure hydrogen releases are known to ignite by static discharge. Discharges can originate from the high velocity flow over surfaces. Arching could also originate from mechanical operations: non-sparking tools (ie. brass spanners) should be used. Pressure release from bleed valves should be performed with caution.

High pressure sampling instrumentation requires frequent maintenance. Whereas leak testing should be performed continuously while operating the instrument, the instrument requires inspection by manufacturer every 6 months. It is referred to the instrument documentation for further details on maintenance.

Operators of the sampling instrumentation should receive training from manufacturer prior to use. Personal safety equipment like glasses, hearing protection and gloves should be worn at all times.

3. Instrument descriptions

Gas sampler: Linde Qualitizer

The Linde Qualitizer is based on a Tescom reduction valve, rated for 1034 bar. A maximum pressure of 875 bar is allowed for a temperature range of -40 to 85 °C. A sealed safety valve is installed for prevention of exposure to higher pressures. The maximum reduced pressure is 160 bar. The reduction valve is illustrated in Figure 1.

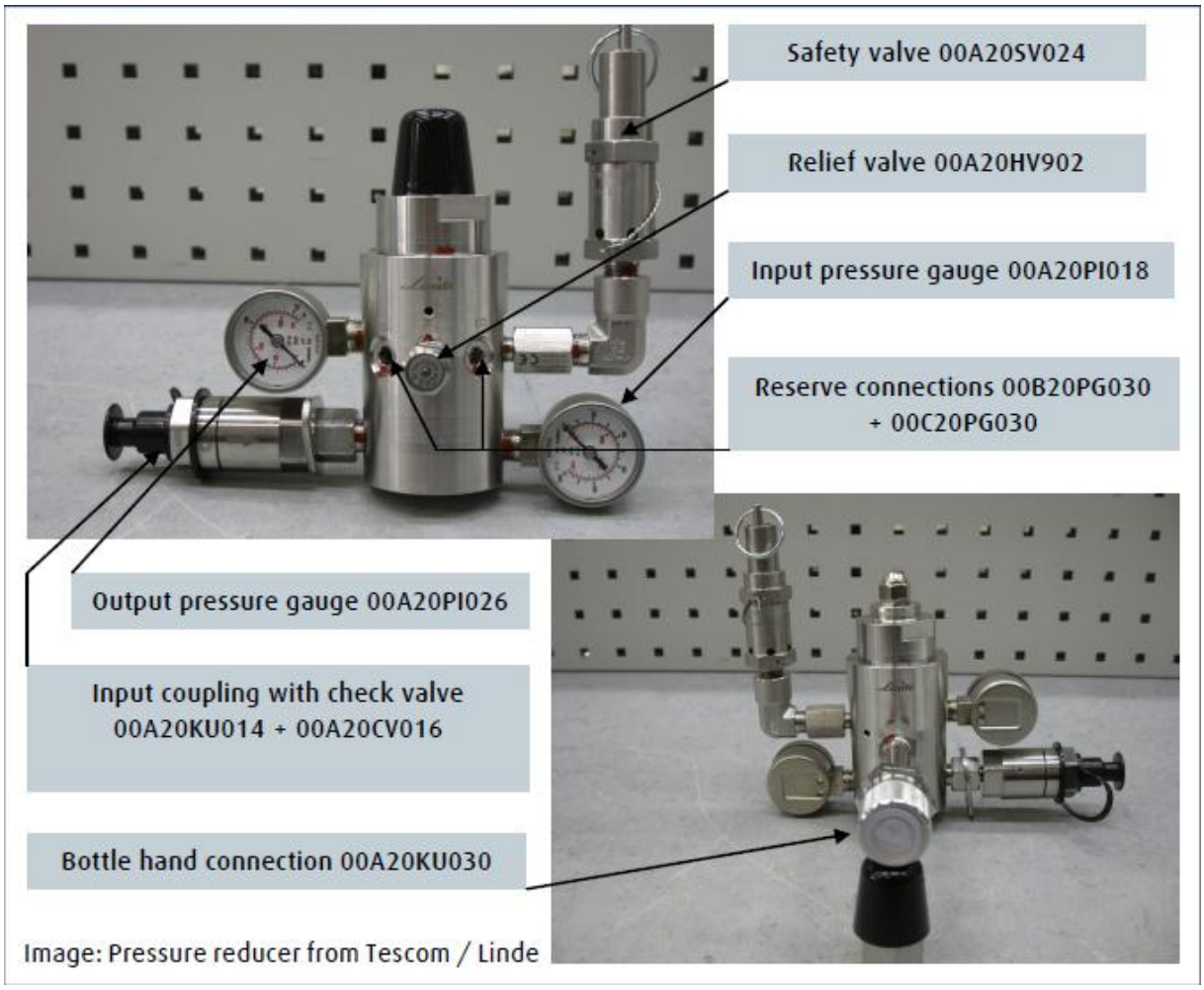

Figure 1. Tescom reduction valve.

The reduction valve is connected to a sampling cylinder, typically a 10 L aluminium canister with a DIN477/1 connector.

The sampling adaptor itself is a "T-piece" inserted between the HRS nozzle and the FCHEV. The adaptor is depicted in Figure 2.

Figure 2. Sample adaptor "T-piece".

The adaptor is connected to the Tescom reduction valve through a high pressure hose with quick connects. The adaptor is not equipped with IR communication interface. This implies that fuelling will be slower and in most cases limited to 600 bar due to safety reasons (no temperature feedback from vehicle hydrogen cylinders).

Operational procedure for gas sampling:

1) Installation/fastening of 10L sampling cylinder near HRS dispenser.
2) Connection of Tescom reduction valve to sample gas cylinder
3) Connection of high pressure hose to Tescom reduction valve
4) Connection of high pressure hose to sampling adapter
5) Connection of sampling adapter to FCHEV receptacle
6) Connection and locking of HRS nozzle to sampling adapter
7) Start filling of FCHEV
8) Taking sample by opening gas sample valve slowly
9) Close gas sample valve after end of fuelling
10) Slowly depressurize sample hose through bleed valve of Tescom reduction valve
11) Disconnect HRS nozzle from sampling adapter
12) Disconnect high pressure hose from Tescom reduction valve and sampling adapter
13) Disconnect Tescom reduction valve from gas sample cylinder
14) Disconnect sampling adapter from FCHEV receptacle
15) Label gas sample cylinder appropriately

Purging

According to the manufacturer, purging of the instrument prior to use is not neccessary. The argument for this is that the internal volume of the sampling unit is several orders of magnitude lower than the pressurized volume of the gas sample cylinder, which is maximum 1600 L.

The sampling unit has check valves in both ends, meaning that if the bleed valve is closed after depressurization then the sampler will be filled with hydrogen from the HRS. In the event of sampler not being filled with H_2 prior to use, there are two ways of performing an intital purge:

- Initiate sampling as normal but abort (press dispenser 'STOP' button) within 15 seconds in order to isolate test pulse. Depressurize with bleed valve
- Perform operational procedure without connecting sampler to FCHEV. HRS safety will shut off H2 dispensing. Depressurize with bleed valve, before attaching to FCHEV receptacle and performing operational procedure for gas sampling.

The second alternative is adapted from the operational procedure for the HYDAC particle sampler. Linde does not endorse this method and suggests to use the first alternative for purging.

Initial sampling and subsequent analysis will further map the need for purging of the sampler.

Test sampling

SINTEF has tested the gas sampling instrument at the Økern HRS in Oslo, Norway. The instrument function was successfully verified and samples were collected in two 10L aluminium cylinders.

One possible weakness of the sampling instrument was discovered: when the HRS switches bank due to H_2 supply, safety was triggered. HRS needed reset after this even, which could cause a problem for unassisted sampling of H_2. It was belived that this event was caused by the following: when the HRS shifts bank, the filling is temporarily stopped. As gas flow into the sampling cylinder commences as long as the pressure in the sampling unit is higher than 160 bar, the HRS safety perceives this as a leak and shuts of the filling. This could be technology specific for the Økern HRS, but for unassisted sampling with the Qualitizer this event should be further evaluated.

Figur 1. Linde Qualitizer sampling at Økern HRS.

<u>Particle sampler: HYDAC PSA-H70</u>

The particulate sampler is a solid filter holder to be placed in line with the fuelling line. The HRS nozzle connects on the top of the sampler whereas the bottom end is connected to the FCHEV through a high pressure hose and a TK17 700 bar hydrogen nozzle. A bleed valve allows of purging of the instrument prior to sampling.

In order to prevent the 875 bar pressure test pulse rupture the filter, a rotary valve is used to reduce the bore from 4 to 1 mm at the beginning of the sampling. Full flow is required in order to have a representative particulate concentration collected onto the filter.

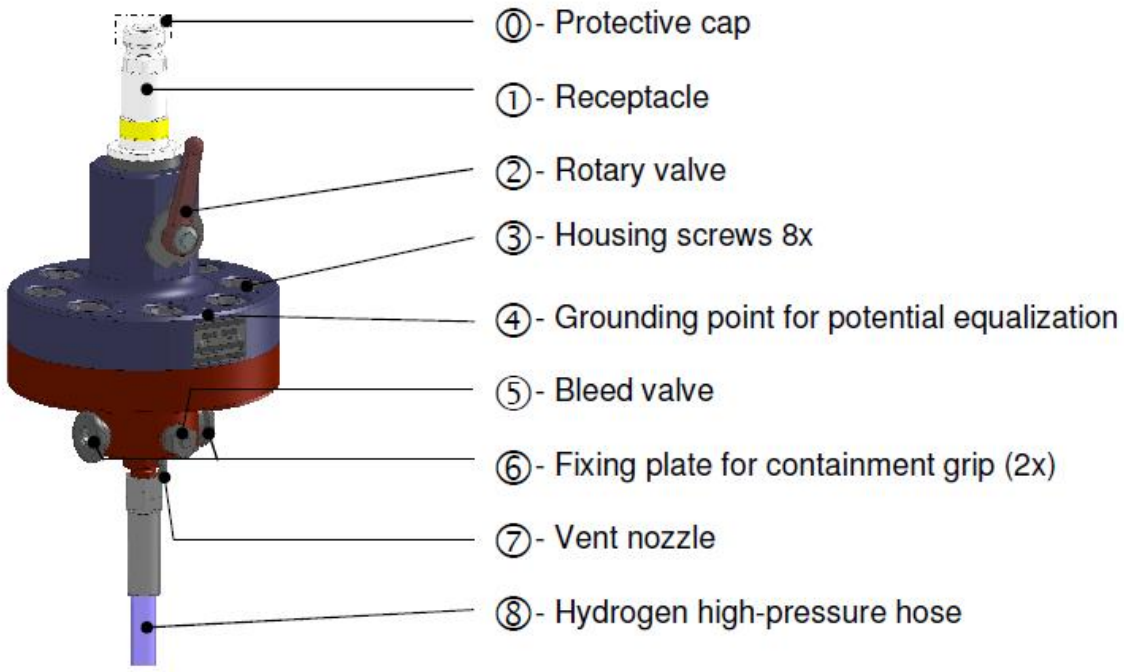

Figure 3. HYDAC PSA-H70 particulate sampler.

The sampler takes ∅ 47 mm filters. In accordance with standard procedure D7650, 0.2 µm filters should be used.

Particulate sampler assembly

According to HYDAC procedures, the assembly of the sampler must be performed in a clean room or by use of a glove box in order to avoid possible contamination of the filter. This is complicating for the application of the instrument on sampling campaigns where said facilities is not necessary available.

In HyCoRA, SINTEF will investigate the significance of applying clean room and/or glovebox to the assembly of the particulate sampler.

The sampler has been tested by SINTEF at a HRS in Oslo, Norway. The installation is shown in Figure 4

Figure 4. PSA-H70 sampler installed at HRS Økern in Oslo, Norway.

Operational procedure for collecting particles onto filter:

1) Connect and locl high pressure hose to PSA
2) Close bleed valve
3) Ground PSA with cable
4) Attach HRS nozzle to PSA
5) Put rotary valve in Clock wise (∅ 1 mm bore) position
6) Start filling procedure
7) When HRS safety triggers, conduct leak test
8) Carefully depressurize PSA through bleed valve. Close bleed valve after
9) Connect PSA to FCHEV receptacle
10) Remove HRS nozzle from PSA
11) Reconnect and lock HRS nozzle to PSA
12) Start filling of FCHEV procedure
13) Switch rotary valve to Counter Clock wise (∅ 4 mm bore) position
14) At end of fuelling disconnect HRS nozzle
15) Carefully depressurize PSA through bleed valve
16) Detach PSA from FCHEV receptacle
17) Close bleed valve

4. Sampling filters and cylinders

Sampling filters
Two types of filters are specified by HYDAC:

Millipore PTFE Porex B 5 µm ∅ 47 mm
ALBET Lab Science PTFE hydrophob blanca 0.2 µm ∅ 47 mm

Only the last filter complies with the D7659 standard protocol.

Sampling cylinders

Linde spectra-seal aluminium cylinders, 10 L will be used for sampling. The cylinders is equipped with a stainless steel valve in order to minimize contamination from gas. Sylinders is evacuated prior to use, and valve should remain closed until start of sampling.