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Secondary standard for hydrogen refuelling station verification: Method and requirements

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Abstract

The requirements for the refuelling process to minimise overheating and overfilling have a considerable influence on HRS design and have a substantial impact on prospective fuelling performance. Type approval of HRS is a complex, expensive and time-consuming process whereas on-field inspection should be quick, cost efficient, reliable, and easy to perform. This paper describes a secondary flow standard which could be used to meet those objectives. Through the implementation of secondary standard verification, type approval time will drastically decrease from 2 days of testing (using the primary standard) to half a day. Thus, decreasing operation time and cost which will appeal to investors (OPEX reduction). This will pave the way for the development of new hydrogen infrastructure to facilitate the increase in hydrogen cars.

1. Introduction

The decarbonization of transportation is one of the EU's primary goals. Since it contributes to 25% of greenhouse gas (GHG) emissions, the aim is to reduce them by 95% by 2050. Efficient driving solutions such as fuel cells and clean alternative technologies are required to meet this long-term climate objective. Hydrogen offers the greatest long-term potential to radically reduce the many problems inherent in fuel used for transportation, used on a global scale it could produce almost zero greenhouse gas emissions and reduce air pollutant emissions. In support of this, increases in the development of hydrogen infrastructure, including a network of HRS (for more information regarding HRS metrological performances see [1]) are necessary for the adoption of hydrogen cars.

Trends show a record year for hydrogen vehicle sales in 2021, with the hydrogen transportation market expected to reach $42,038.9 million by 2026 [2]. Factors for this market's expansion include: a growing increase in environmental concern, government initiatives, investment in infrastructure and technological advancement. At the end of 2021, Europe had 200 hydrogen stations, 100 of which are in Germany and 34 in France.

However, the industry technological advancements still need to be made so the aim of this work is to establish a field-verification using calibrated secondary standards.

2. Technical specifications for the master meter method

2.1 Project Scope

Building a mobile and quickly deployable test rig for field-verification of HRS (light-duty and heavy-duty vehicles) using calibrated secondary standards for assessment to Accuracy Class 2. This system shouldn't alter any part of the refilling procedure and the vehicle shouldn't detect any difference from a normal refuelling. It will contain a standard gas meter for measuring the flow passing from the HRS to the pressure vessel, or the vehicle connected at the outlet of the mobile meter. It will be mounted in between and will have a small internal volume (see Figure 1).

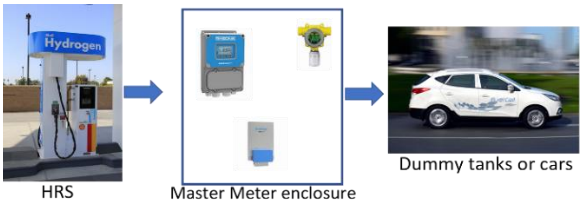

Figure 1: Master meter method for on-field verification of HRS

A suitable method of field-verification must be devised with proper measurement, accuracy, and repeatability to assure this. This method prioritises personnel and material safety over speed of refuelling.

2.2 Key Performance goals

The mobile test rig should be traceable to primary standards but faster and easier to deploy. It should be able to operate at any flowrate, gas pressure and temperature provided by the HRS [3, 4] with < 0.67 % uncertainty (related to one third of the MPE (OIML R139-2:2018 1.3.2) for class 2 in OIML R139-1:2018 5.2.1) [3]

2.3 Main technical specifications

2.3.1 Reference meter

The reference meter of the test rig will: be an ATEX certified mass flow meter, be designed up to pressure of at least 900 bar for hydrogen, cover the mass flow rate range from (0.1 to 7.2) kg/min (SAEJ2601 [4]) and a temperature range of -40 °C to 85 °C, and have a transmitter that can log mass flow rate and tubing temperature

2.3.2 Connections to and from the mobile test rig

To avoid interference with the dispenser's refuelling process, the test rig must be located between the dispenser and the final hydrogen container. If the final hydrogen container is a vehicle, no contaminant from the test rig shall end up in the vehicle's tank. It must be able to verify HRS working at 350 bar (also HF = High Flow) and 700 bar. If data communication is present, the rig will allow data transmission between the dispenser and the final hydrogen container.

2.3.3 Instrumentation

All instrumentation will be calibrated for hydrogen's relevant pressure range. The test rig will log the hydrogen's temperature and pressure in the piping, before and/or after the meter. Therefore, it must include at least one dial pressure gauge, independent of the electrical supply that can always read the pressure value during testing.

2.3.4 Internal piping

Internal piping shall be designed for maximum operating pressure up to 1000 bar for hydrogen. Its volume must be kept as minimal as possible to limit inventory (dead) volume while not causing significant pressure loss at maximum flow rate.

2.3.4 Safety

For safety, ATEX certification is required for the test rig and all measurements must be performed with as little human intervention as feasible. There must be no unwanted backflow or hydrogen leakage, regardless of whether the inlet or outlet is disconnected first. A H2 sensor inserted into the enclosure, will monitor the potential hydrogen leakage during operations. In case of detection, the system's power is switched off to prevent hydrogen ignition. Additionally, the operator must be able to vent the complete internal volume (piping + meter) into the environment from any pressure level. The test rig will have connections to purge the internal volume (piping + meter) with nitrogen. The operator must be able to safely touch all visible surfaces without risk of burn while wearing appropriate apparatus.

2.3.5 Usability

To be user-friendly, the test rig will be mobile and lighter than 100 kg with handles for easy lifting. All fittings will be positioned for easy loosening / tightening, replacement, and inspection. Where possible, fixtures should be from Europe for quick replacement. For flexibility, a remote flow meter transmitter will be installed so that the operator can connect to it using a non–ATEX compliant system.

2.4 System description

The following system description presents a system that fulfils all the requirements from the previous section.

2.4.1 General description

The test rig consists of 2 mobile and transportable skids.

The first skid is ATEX certified and houses a Coriolis mass flow meter; a transmitter from the Coriolis meter; a 350 bar HF fitting and a 700-bar fitting on the inlet side that connects the refuelling hose from the dispenser to the test rig with check valves to prevent backflow; the same fitting on the outlet side connecting the refuelling hose from the test rig to the final hydrogen container with check valves to isolate the fittings; a purging line, to purge with either nitrogen or hydrogen; a venting line, for venting the line after a measurement; instruments for measuring pressure and temperature in the pipes; connections to the power supply and the data acquisition system.

The second frame could be partially ATEX certified and houses with dummy tanks (ATEX zone 2 inside the trailer), ATEX Instruments for measuring pressure and temperature in the pipes and safety barriers to limit power delivery to the electrical equipment in the ATEX frame.

2.5 More reflections on certain functions

2.5.1 Purging procedure before starting a measurement.

If nitrogen is present in the secondary meter, perform a hydrogen purging procedure. If the hydrogen vehicle tolerates a small amount of nitrogen, then it's not required.

1. Proposal 1: introduce nitrogen into a dummy tank, or the primary standard. With this

solution no problems from the HRS side are expected.

2. Proposal 2: releasing hydrogen directly from the HRS through the vent stack. The HRS will likely have problems with this solution because the secondary standard has no internal volume.
3. Proposal 3: Filling the secondary standard with hydrogen from a bottle.

2.5.2 Precooling of the Master Meter

When the Master Meter is used on a 70MPa refilling process the hydrogen passes through the flow meter at -35°C. Following the start of the filling process the master meter adjusts its temperature to that of the hydrogen. This unsteady working condition could affect the accuracy of the flow meter but can be mitigated by precooling it.

Three options for precooling are presented below:

- Dry ice
- Climate chamber (ATEX)
- ATEX cooling enclosure designed by Cesame Exadebit (ATEX zone 2 certified)

However, with dry ice, the temperature isn't easy to control, it sublimates at temperatures of -78°C and it can't be kept as (semi)-permanent stock. Furthermore, it hasn't yet been tested at the temperature the flowmeter will cool down while surrounded by dry ice and it's difficult to predict how varying outside temperatures would affect it. This complex availability makes cooling by dry ice during the test phase of master meter disadvantageous.

The most promising method of keeping the flow meter cold would be to completely immerse it in a temperature-controlled cooling chamber. To enhance temperature stability, the remaining space in the cooling chamber will be filled with ice or similar substance. The climate chamber's setpoint will be -35°C. This method, however, is unlikely to be used due to the initial expenditure, the scarcity of ATEX-proofed cooling chambers, and the cooling chamber's inconvenient size.

Selected approach:

Cooling the flow meter and the pipe section upstream of the flowmeter with a dedicated ATEX cooling enclosure designed, developed, and built by Cesame Exadebit.

2.5.3 Heat exchanger upstream of measurement device

The hydrogen is fuelled at -35°C for H70 fillings, the transfer standard must be at the same temperature to avoid any unexpected behaviour and a potential modification in accuracy.

2.5.4 Proposed operation procedure with the secondary standard

1	Install the secondary standard with both skids in the vicinity of the dispenser and connect all signal cables to the transmitter and computer.
2	Connect the dispenser to the inlet of the secondary standard skid.
3	Replace the nitrogen in the secondary standard with hydrogen (optional).
4	Connect the hose to the vehicle or the dummy tanks with the appropriate hose and / or nozzle.
5	Start the filling process.
6	Terminate the filling process.
7	Disconnect the line between the dispenser and the secondary standard skid.
8	Disconnect from the vehicle or dummy tanks.
9	Purge the secondary standard with nitrogen, and subsequently release the gas trapped inside, into the environment.

3. Method for field-verification for HRS using calibrated master meter

3.1 Uncertainty and Corrections

The primary source of uncertainty in the secondary standard is the Type B uncertainty (U_B) given by the Coriolis flow meter manufacturer. It's usually expressed as a flow uncertainty that changes with flow magnitude. There's a negative correlation between the uncertainty and the flow because the target quantity for measurement is not mass flow rate but invoiced totalized amount of hydrogen (in mass).

U_B may be calculated using the following formula:

$$U_{meas} = \frac{\int U_{flow}(Q(t)) \cdot Q(t) \cdot dt}{\int Q(t) \cdot dt} = \frac{\int U_{flow}(Q(t)) \cdot Q(t) \cdot dt}{m_{meas}}$$

Where $U_{flow}(Q(t))$ is the type B uncertainty of the mass flow and Q(t) is the mass flow. The difference between the mass supplied and the mass measured by the Coriolis flow meter must be measured in addition to the primary uncertainty contribution. If we specify the filling coupling as the interface at which hydrogen mass counting should be assessed, we must account for a modest volume of secondary standard pipework between the filling receptacle and the Coriolis flow meter. On one hand the amount of gas already contained ahead of the filling process must be discounted. On the other hand, one must account for gas in the same volume which would have been invoiced into a hydrogen vehicle but does not reach the Coriolis flow meter.

For the calculation of these small amounts of gas one has to know a) the volume of this section of the piping, b) the temperature, c) the pressure and d) the compressibility factor of hydrogen (and/or other gas if this section of the piping is filled with for example nitrogen prior to the filling process, which is only possible if the invoiced gas is stored in tanks and not in hydrogen vehicles). The user interface should ask whether hydrogen or nitrogen is used. The amount to be discounted for can be calculated by:

$$m_{disc} = \frac{p_{in} \cdot V \cdot M_{gas}}{Z_{gas}(p_{in}, T_{in}) \cdot R \cdot T_{in}}$$

where p_{in} is the pressure and T_{in} is the temperature of the gas before the filling process, V is the volume of the section of the piping between the refuelling station receptacle and the inlet of the Coriolis meter, M_{gas} is the molar mass of the gas $Z_{gas}(p_{in},T_{in})$ is the compressibility factor of the gas (taken from for example REFPROB) and R is the gas constant. The amount to be accounted for can be calculated by:

$$m_{acc} = \frac{p_{fin} \cdot V \cdot M_{H_2}}{Z_{H_2}(p_{fin}, T_{fin}) \cdot R \cdot T_{fin}}$$

where p_{fin} is the pressure and T_{fin} is the temperature of the gas after the filling process, M_{H2} is the molar mass of hydrogen and $Z_{H2}(p_{fin},T_{fin})$ is the compressibility factor of hydrogen.

The corrected invoiced mass:

$$\begin{aligned} m_{cor} = m_{meas} - (m_{disc} - m_{acc}) \\ = m_{meas} \\ - \left(\frac{p_{in} \cdot M_{gas} \cdot V}{Z_{gas}(p_{in}, T_{in}) \cdot T_{in} \cdot R} \right. \\ \left. - \frac{p_{fin} \cdot M_{H_2} \cdot V}{Z_{H_2}(p_{fin}, T_{fin}) \cdot T_{fin} \cdot R} \right) \\ = m_{meas} - \left(\frac{M_{gas} \cdot p_{in}}{Z_{gas}(p_{in}, T_{in}) \cdot T_{in}} \right. \\ \left. - \frac{M_{H_2} \cdot p_{fin}}{Z_{H_2}(p_{fin}, T_{fin}) \cdot T_{fin}} \right) \cdot \frac{V}{R} \end{aligned}$$

where m_{meas} is the measured mass.

Each of these quantities comes with its associated uncertainty ($u_i(x_i)$) which needs to be considered for the total uncertainty. The combined uncertainty is given by:

$$u_c^2(m_{corr}) = \sum_i \left(\frac{\partial m_{corr}}{\partial x_i} \right)^2 \cdot u_i(x_i)$$

Where $u_i(x_i)$ are uncertainties of the individual quantities necessary to correct the measured mass.

3.2 Test procedures according to OIML R 139 [3]

OIML R 139-2 Edition 2018 (E) contains the relevant test programs. *4.6.6 Alternative Procedure* and *4.6.7 Alternative procedure for hydrogen CGF measuring systems* are considered in this case. Different conditions apply for the different test procedures (e.g., for the maximum observed flow rate, specifications for the maximum permissible flow rate, etc.) but the key aspect is the type of tests to be performed:

3.2.1 Alternative Procedure 4.6.6

"Tests sufficiently representing the real conditions of use are performed." Which means: two tests of filling a test receiver from empty to maximum pressure ("full" in blue) and two tests of filling a test receiver from half pressure to full pressure ("half full" in orange).

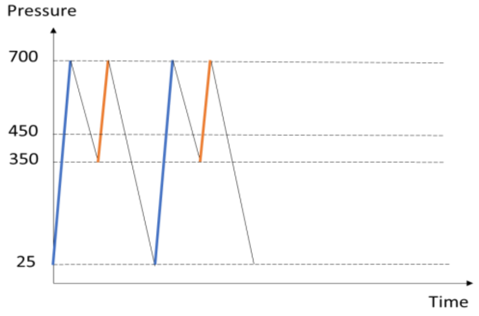

Figure 2: Procedure 4.6.6 for one storage tank

The sequence of measurements should be alternating (i.e., full – half full – full – half full). According to OIML R 139 4.6.6, the maximum permissible error (MPE) *"shall fulfil the requirement on MPEs specified in R 139-1, 5.2.3."* [3]. This is an error because 5.2.3 only references MPE for MMQ. It is meant to say, *"shall fulfil the requirement on MPEs specified in R 139-1,* ***5.2.1 and*** *5.2.3."*

3.2.2 Alternative procedure 4.6.7 for hydrogen CGF measuring systems

Three tests of filling a test receiver from empty to maximum pressure ("full" in blue, *Test 4* in *2.2.7.3*) and three tests of filling a test receiver from half pressure to full pressure ("half full" in orange, *Test 5* in *2.2.7.3*). Additionally, two Minimum Measured Quantities (MMQs in green, *Test 7* in *2.2.7.4)* are to be measured. Each test shall be performed consecutively under the same conditions or in a cyclic consecutive order.

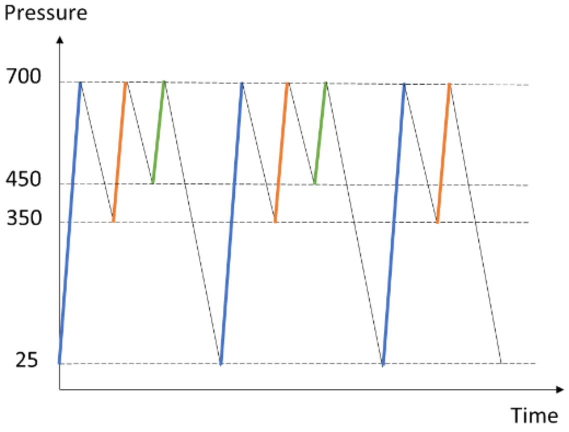

Figure 3: Procedure 4.6.7 for one storage tank

The MPEs are given *by OIML R 139-1 5.2:* [3]- 2 % for accuracy class 2 of hydrogen for full and half fills 4% for accuracy class 4 and for MMQ. To verify these MPEs the goal is to reach an expanded uncertainty of one third of the MPE, i.e., for accuracy class 2 this means 0.67% (and 1.33% for accuracy class 4) for periodic verifications.

3.6 Procedure for verification

Various options for storing hydrogen are used, the most common of which being fuel cell vehicles (FCVs) and dummy tanks (see SAE J2601/1 8.2 Table 4) [4]. For this application, we prepared both procedures for dummy tanks since FCVs appear difficult to implement. As it would require a large fleet of expensive FCVs and demand more crew. Furthermore, the use of dummy tanks eliminates the possibility of contamination with gases other than hydrogen. They are placed in a dummy tank assembly (DTA), which enables a) individual tank filling and b) individual and collective venting. The fact that several tens of kilograms (for heavy duty even more) of hydrogen are vented into the atmosphere may indicate an alternative course for subsequent verifications, both environmentally and economically (maybe an assembly for a closed loop test setup where the invoiced hydrogen is transferred back into the HRS).

3.6.1 Preparation

We presume that a master meter has previously been calibrated against a primary standard and under similar conditions as an HRS. After the DTA and master meter arrive at the HRS to be checked, the system is installed and set up near the dispenser to be tested. All signal cables are linked to the computer and transmitter. Following that, the dispenser is attached to the master meter, which is connected to the DTA.

3.6.2 Zeroing

The zero procedure for the flow meter is performed at -35°C three times in a row.

3.6.3 Measurement

The tables outline the operations required for the relevant tests in the shortest amount of time. Therefore, venting one tank occurs concurrently with fuelling another. The tables provide an approximate time estimate but *handling times have not yet been considered*. Time is a crucial factor venting, and we estimate 15 minutes for venting a full tank to 450 bar (1-MMQ), 20 minutes for venting to 350 bar and 90 minutes for full venting. The times listed are exclusively for light duty vehicles. Each tank must be flushed with nitrogen at completion.

3.6.3.1 Procedure 4.6.7 for 3 tanks

This test procedure lasted an hour (excluding venting at the end).

Tank #1			Tank #2			Tank #3			Timing	
Operation	Pressure [bar]	Target Pressure [bar]	Operation	Pressure [bar]	Target Pressure [bar]	Operation	Pressure [bar]	Target Pressure [bar]	Start time [min]	Duration [min]
Full Fill #1 (Test4)	20	700							0	5
vent start	700	350							5	20
			Full Fill #2 (Test4)	20	700				5	5
			vent start	700	350				10	20
						Full Fill #3 (Test4)	20	700	10	5
						vent start	700	350	15	20
vent stop	350								25	
Half Fill #1 (Test5)	350	700							25	3
vent start	700	450							28	15
			vent stop	350					30	
			Half Fill #2 (Test5)	350	700				30	3
			vent start	700	450				33	15
						vent stop	350		35	
						Half Fill #3 (Test5)	350	700	35	3
						vent start	700	3	38	90
vent stop	450								43	
MMQ #1 (Test6)	450	700							43	2
vent start	700	3							45	90
			vent stop	450					48	
			MMQ #2 (Test6)	450	700				48	2
			vent start	700	3				50	90
						vent stop	3		128	
						Flush start	3	24	128	5
						Flush end	24		133	
vent stop	3								135	
Flush start	3	24							135	5
Flush end	24								140	
			vent stop	3					140	
			Flush start	3	24				140	5
			Flush end	24					145	

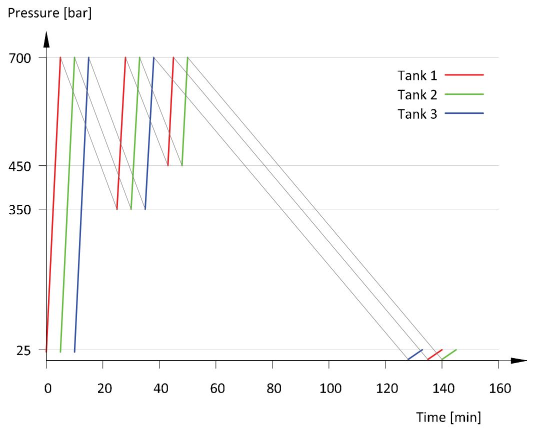

Figure 4: Representation of test sequencing during verification when procedure 4.6.7 with 3 tanks is used.

3.6.3.2 Filling Procedure 4.6.6 for 2 tanks

Tank #1			Tank #2			Timing	
Operation	Pressure [bar]	Target Pressure [bar]	Operation	Pressure [bar]	Target Pressure [bar]	Start time [min]	Duration [min]
Full Fill #1 (Test4)	20	700				0	5
vent start	700	350				5	20
			Full Fill #2 (Test4)	20	700	5	5
			vent start	700	350	10	20
vent stop	350					25	
Half Fill #1 (Test5)	350	700				25	3
vent start	700	3				28	90
			vent stop	350		30	
			Half Fill #2 (Test5)	350	700	30	3
			vent start	700	3	33	90
vent stop	3					118	
Flush start	3	24				118	5
Flush end	24					123	
			vent stop	3		123	
			Flush start	3	24	123	5
			Flush end	24		128	

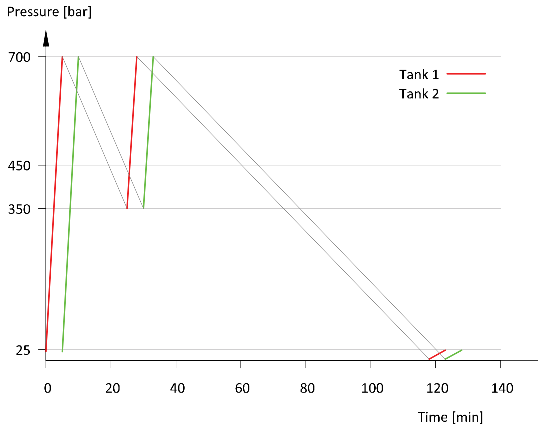

Figure 5: Representation of test sequencing during verification when procedure 4.6.6 with 2 tanks is used.

3.6.4 Post measurement procedure

The dispenser line is disconnected from the master meter, as is the master meter from the dummy tank assembly. To limit the inhibition of HRS the dummy tank assembly can then be moved to a less frequented location at the HRS for venting.

3.6.5 Venting

As stated in the test procedure tables at the end of the measurement, the filled tanks need venting. Each tank requires its own venting stack, reducing the number of venting stacks (i.e., venting two tanks through one venting stack) requires further examination.

7. Conclusion

Through MetroHyVe key stakeholders including hydrogen producers, HRS operators, automotive manufacturers and standardisation bodies, the consortium has identified unmet measurement challenges to better and quicker deploy hydrogen for transport in Europe:

There is currently no method using secondary standards calibrated by the European primary standards. The development of secondary standards for hydrogen flow dispensed at HRS traceable to the European NMIs primary standard (developed in MetroHyVe) would allow quicker, cheaper and more frequent HRS testing.

To mitigate this issue, a secondary standard has been designed, developed, and built. The target uncertainty should be met in the upcoming tests campaign. Cesame Exadebit developed a unique ATEX mobile secondary standard with controlled measuring conditions which allows to perform quick and affordable on field verifications for HRS owners. The verification period will be reduced by using this technique (especially if 4.6.6 of OIMLR139 can be used – 4 tests only). It will attract more investors, since they will be more maintainable and lose less time during periodic verifications, which will increase the number of HRS. Quick verification of the HRS will make them more maintainable, cheaper and … which will help the market grow.

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