METROLOGY for HYDROGEN VEHICLES 2

Deliverable D1

Report on the development of primary standards for heavy-duty vehicles (target uncertainty of < 0.5 %) and secondary standards for light-duty and heavy-duty vehicles (target uncertainty of < 2%) for measuring mass of hydrogen dispensed at HRS.

Project name: MetroHyVe 2

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https://www.sintef.no/projectweb/metrohyve-2/

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Summary

The partners achieved to develop the first primary standards for heavy-duty vehicles refuelling. The two standards developed were detailed in the report with emphasis on the uncertainty achieved which complies with uncertainty for type approval according to OIML R139:2018. The secondary standard for flow metering at hydrogen refuelling station was developed. The design and methodology were presented in the report. The uncertainty determined for the secondary standard were below the targeted uncertainty of 2%.

This report was written as part of activity 1.3.5 from the EMPIR Metrology for Hydrogen Vehicles 2 (MetroHyVe2) project. The three-year European project commenced on 1st August 2020 and focused on providing solutions to four measurement challenges faced by the hydrogen industry (flow metering, quality assurance, quality control, sampling and fuel cell stack testing). For more details about this project please visit https://www.sintef.no/projectweb/metrohyve-2/.

Confidentiality	Public

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EMPIR EURAMET

The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

Contents

1 Introduction

Inability to validate the measurements of hydrogen dispensed at hydrogen refuelling stations (HRS) is a major barrier to the uptake of hydrogen fuel cell vehicles (FCEVs). To address this, several portable primary flow standards were built in the previous MetroHyVe project. These flow standards can be taken to HRS to validate the dispenser measurements and are considered appropriate for "light-duty" vehicle applications, with maximum hydrogen capacities of 2.8 to 6 kg at 70 MPa and measurement uncertainty of approximately (3 to 5) g in mass. Filling times and flow rates using these standards are the same as for a car.

This is not the case for heavy-duty vehicles which store typically more than 30 kg of hydrogen at 35 MPa with collection volumes larger than 1000 L. Light-duty primary standards, with their limited collection vessels, would have the dispenser deliver hydrogen at much lower flow rates when filling the primary standards compared to a heavy-duty vehicle, this would not be a representative test of the HRS.

MetroHyVe 2 aims to further develop the traceability chain for FCEV refuelling, extending to "heavy-duty" vehicle applications with the introduction of new primary and secondary flow standards. The aim of this report is to present the development and measuring method of primary standards for heavy-duty hydrogen fuel cell road vehicles and secondary standards for light-duty and heavy-duty vehicles.

2 Requirements for calibration and verification of a HRS from OIML R139:2018

When a hydrogen station operator wishes to sell its product to customers, the station manufacturer must comply with the associated standards regarding resale of energy to individuals (legal metrology) to ensure reliability and fairness in the transaction. There is little information regarding the sale of hydrogen (HRS) to individuals. However, the "Organisation Internationale de Métrologie Légale - OIML" has a recommandation on compressed gas (natural gas mainly) which has recently been revised in order to integrate the technical constraints to the use of hydrogen. The most recent edition which was issued in October 2018 has 3 parts which are available on the OIML website. https://www.oiml.org/en/files/pdf_r/r139-p-e18.pdf/view

The text is divided into three parts, constituting three separate documents:

1. R139-1 - Metrological and Technical Requirements (52 pages) [1]
2. R139-2 - Metrological controls and performance tests (63 pages) [2]
3. R139-3 - Test Report Format (65 pages) [3]

2.1.1 Metrological and technical requirements (document R139-1)

This first section describes the elements that constitute the measurement system and therefore items that need to be tested for certification of the measuring system.

Figure 1 below summarizes the mandatory and optional elements.

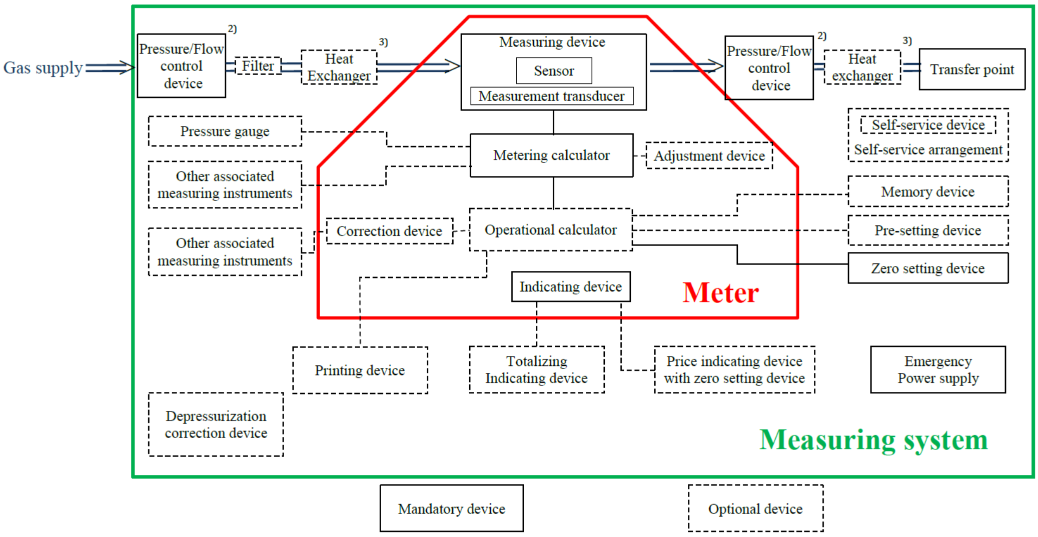


Figure 1: Constituents of a typical compressed gaseous fuel measuring system for vehicles.

<u>Metrological obligations for the measurement system:</u>

This section lists (in no order of importance) all mandatory metrological requirements to obtain OIML R139 certification: 2018 for a HRS. Metrological requirements not applicable to hydrogen are not noted (for example, those applying to CNG only).

1. The Maximum Permissible Error (MPE) is defined in the Table 1 below:

Accuracy class		MPE for the meter [in % of the measured quantity value]	MPE for the complete measuring system [in % of the measured quantity value]	
			at type evaluation, initial or subsequent verification	in-service inspection under rated operating conditions
For general application	1.5	1	1.5	2
For hydrogen only	2	1.5	2	3
	4	2	4	5

Table 1: MPE values

There are 2 main categories in this table, the MPE for the meter itself (on the left side) and the MPE for the complete measuring system.

It is important to note that there are 2 classes for hydrogen. Class 4 will be mainly accepted for existing stations whereas class 2 shall be chosen for new HRS. As an example, the MPE during a type approval of a new HRS (class 2) is 2%.

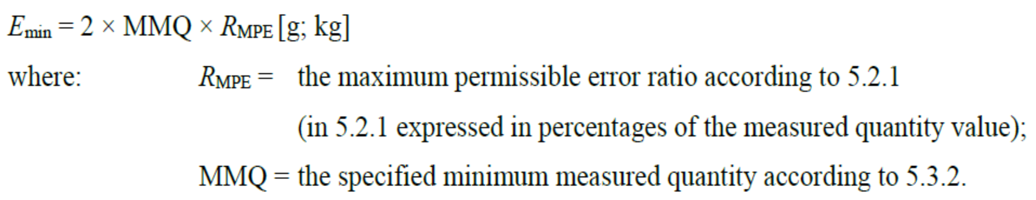
2. In the case of the measurement of the 'smallest measurable amount' (MMQ) (Minimum Measured Quantity) , the MPE is twice the value of the Table 1 above as shown in Section 5.2.3 of the OIML standard.

$$E_{min} = 2 \times MMQ \times R_{MPE}$$

Where:

R_{MPE} = The maximum permissible error ratio according to Section 5.2.1

MMQ = The specified minimum measured quantity accoridng to Section 5.3.2



$E_{\text{min}} = 2 \times \text{MMQ} \times R_{\text{MPE}}$ [g; kg]

where: R_{MPE} = the maximum permissible error ratio according to 5.2.1
(in 5.2.1 expressed in percentages of the measured quantity value);
MMQ = the specified minimum measured quantity according to 5.3.2.

The MMQ has been defined as a fixed value for all hydrogen applications whereas it was a function of the mass flow rate for other compressed gas fuels. It is stated in OIML R139:2018 that the maximum MMQ for all types of HRS is **1 kg**. Section 5.3.2.2.

For instance, for a class 2 HRS, the Maximum Permissible Error of the MMQ is 4% of 1 kg, yielding 40 g. This means that the MPE cannot be lower than this value for any measured quantity. Indeed, as long as the delivered quantity is smaller than twice the MMQ, the MPE will be 40 g. At twice the MMQ, the MPE for a class 2 HRS would be 2% and give a MPE of 2 % of 2 x MMQ, yielding 40 g. Going to higher delivered quantities will increase the MPE accordingly: 4 kg yields a MPE of 80 g.

2.1.2 OIML R139-2 Metrological control and performance tests

Part 2 of the OIML R139 recommendation relates to the metrological controls, instrument evaluation, type evaluation, and the initial and subsequent verifications of a HRS. For metrological controls, most of the section is devoted to the uncertainty calculations. The uncertainty requirements depend on the type of certification, and are related to the MPE of the HRS. For instrument evaluation, the recommendation details instrument specifications and meter capacity, as well as flow rate. The specific tests to be performed as part of an evaluation are detailed, and denoted as test # 4, #5 and # 7 as described in Table 2 and Table 2: Initial settings for tests on systems without sequential control

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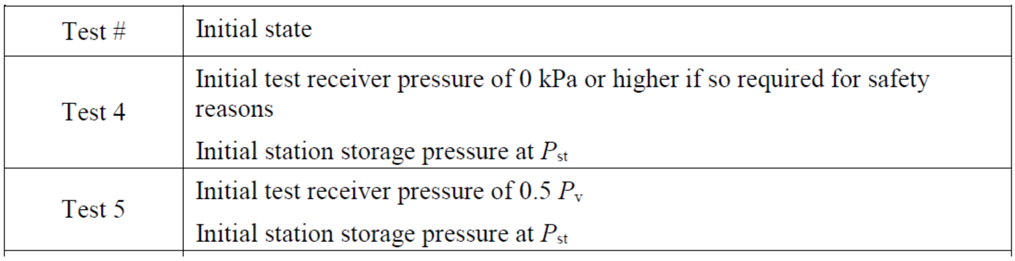
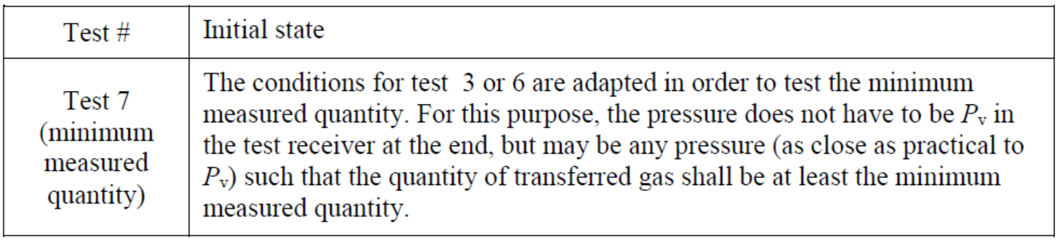


Test #	Initial state
Test 4	Initial test receiver pressure of 0 kPa or higher if so required for safety reasons Initial station storage pressure at P_{st}
Test 5	Initial test receiver pressure of 0.5 P_{v} Initial station storage pressure at P_{st}

Table 2: Initial settings for tests on systems without sequential control



Test #	Initial state
Test 7 (minimum measured quantity)	The conditions for test 3 or 6 are adapted in order to test the minimum measured quantity. For this purpose, the pressure does not have to be P_{v} in the test receiver at the end, but may be any pressure (as close as practical to P_{v}) such that the quantity of transferred gas shall be at least the minimum measured quantity.

Table 3: Initial settings for tests on systems with and without sequential control

P_{st}: maximum storage pressure

P_{v}: maximum allowable vehicle fast fill pressure

Test 4 is more commonly named as 'full fill', Test 5 as 'half fill' and Test 7 as 'MMQ'.

Section 1.3.2 gives the uncertainty associated with the test method. When a test is conducted, the expanded uncertainty on the determination of errors on indications of mass shall be:

- For type evaluation less than one-fifth of the applicable MPE;
- For verifications less than one-third of the applicable MPE.

If the above-mentioned criteria cannot be met, the test results can be approved alternatively by reducing the applied maximum permissible errors with the excess of the uncertainties. In this case the following acceptance criteria shall be used:

- For type evaluation ±(6/5 · MPE – U)
- for verifications ±(4/3 · MPE – U)

while U ≤ MPE.

A HRS of accuracy class 2 would require testing equipment with expanded uncertainties of:

- less than one fifth of 40 g → 8 g for type evaluation
- less than one third of 40 g → 13.3 g for verifications.

3 Requirements for the primary flow standards

3.1 Limitations of existing flow standards

The existing primary flow standards developed for light duty vehicles cannot be used for the vehicles in this range, for several reasons.

Due to limited tank capacity (up to 208 litres) of the existing flow standards, the refuelling station Coriolis meters operate at flow rates less than 3.6 kg/min during evaluations. Flow rates of up to 7.2 kg/min are expected for refuelling of buses and trucks, depending on if pre-cooling is applied or not. Like most flow meter types, the Coriolis meter response is more stable at medium to high flow rates. This could result in performance differences in the flow meter, depending on the size of vehicle being refuelled.

A major contribution to measurement error in HRS is the change in pressure and density in piping connection from the dispenser to a flow meter installed upstream. This ‘dead volume’ effect is unrelated to the flow meter behaviour and the relative contribution is reduced when filling larger vessels to higher hydrogen capacity. Therefore, using a light duty standard to assess the HRS accuracy for heavy duty refuelling could result in over-estimated errors.

Similarly, the HRS Coriolis meter can be greatly influenced by temperature effects if it is installed in the cold region of the HRS. From 16ENG01 MetroHyVe, it was observed that low temperature effects are only moderate if the temperature is stable and flow rates are relatively high. The real concern is transient temperature effects, as a meter which is initially at ambient temperature is cooled by the incoming stream of hydrogen at -40°C during the refuelling process.

This temperature effect will be reduced for heavy duty vehicle refuelling, for two reasons. Many heavy-duty vehicles are limited to 35 MPa filling pressures. There is a reduced requirement for pre-cooling compared to 70 MPa service. For refuelling of these vehicles, there will typically be less of an initial temperature difference between the flow meter and the incoming gas and therefore less opportunity for transient temperature effects to occur. Also, regardless of the vehicle filling pressure (35 MPa or 70 MPa) heavy duty refuelling occurs at higher flow rates and for longer durations than light duty. Therefore, a greater proportion of the hydrogen should be metered at high flow rates and stable temperatures compared to a light duty refuelling.

To conclude, using a light duty primary standard is likely to overestimate errors for heavy-duty vehicle refuelling, due to a greater influence of low flow rate, low temperature and dead volume effects. For a robust evaluation of HRS accuracy in heavy-duty vehicle refuelling, new flow standards are required.

3.2 Operating limits

The heavy-duty HRS primary standards need to have sufficient hydrogen capacity to represent the vehicles that the HRS will service. However, matching the vehicle capacities on a 1:1 basis is impractical. Currently, there is a greater prevalence of heavy-duty vehicles designed for 35 MPa service and hydrogen capacity of 30 to 40 kg in Europe. However other territories including North America and Asia favour the larger capacity HGVs based on 70 to 80 kg hydrogen storage at 70 MPa, and there are some early indications that Europe could follow suit.

If the primary standards built matched these vehicle sizes exactly, then two very different systems would be required. Even the smaller 40 kg version would be extremely expensive to build, difficult to transport and operate, and achieving a reasonable measurement uncertainty of less than 0.5% may not be feasible.

For context, a light duty (4 to 6 kg) primary standard with measurement uncertainty of about 3 g (0.3 % for 1 kg) weighs about 500 kg, could be transported by a small van and costs about 300k EUR to build. A heavy-duty standard with 40 kg hydrogen capacity could weigh more than 2000 kg and cost more than 1M EUR to build. The measurement uncertainty achievable will be higher compared to the light duty standards, as the available weighing scales and load cells suitable for this range have poorer resolution, while the MMQ for heavy duty vehicles is still 1 kg, despite the higher maximum capacity. Moreover, buoyancy correction has a larger contribution due to the larger size of the pressure tanks and the control of the temperature gradient around the scales will be key to limit the measurement uncertainty.

Instead, the project partners built flow standards which do not match the vehicle capacities on a 1:1 basis, but still operate at representative fuelling conditions and are suitable to perform OIML R 139 evaluation.

The flow standards produced from this project meet the following requirements:

- Maximum hydrogen capacity of at least 10 kg (SAE J601-2 specifies 10 kg and above for heavy-duty vehicles)
- Measurement uncertainty of 0.5% or better (required by JRP protocol)
- Average flow rate of 1.8 kg/min or higher (required by JRP protocol)
- At least 2 kg hydrogen transferred for a half fill, 17.5 to 35 MPa (required by OIML R 139)

3.3 Specifications of the primary standards

At the basis of this work package is an understanding that for the largest duty ranges, building primary flow standards which match the capacity of the vehicle on a 1:1 basis will not always be practical, and the metrological infrastructure may need to be scaled up using secondary flow standards and master meters which are traceable to smaller primary standards.

It was therefore decided to build two primary standards for heavy-duty vehicle service. Due to cost, weight, and measurement uncertainty considerations, both are in the 10 to 30 kg range. One was built for maximum pressures of 35 MPa, the other for 70 MPa.

Two portable primary flow standards were designed for heavy-duty refuelling:

					Average flow rate (kg/min)		
NMI/DI	Pressure Class	Tank Volume	H_2 capacity at 350 bar	H_2 capacity at 700 bar	3 MPa/min, no pre-cooling	20 MPa/min with pre-cooling	Expanded uncertainty
NEL	35 MPa	1050 L	25.2 kg	-	1.8	-	< 6 g
METAS	70 MPa	600 L	14.4 kg	24.5 kg	1.02	6.8	< 8 g

Table 4: Specifications of the primary standards

With the development of these new standards, it is possible to evaluate refuelling stations with both a light duty standard (up to 6 kg, 70 MPa) followed by a heavy-duty standard (>10 kg, 35 MPa). Comparing the results would allow the investigators to determine the sensitivity of various influences (influence of dead volume effect, average flow rate and temperature sensitivity), ultimately supporting the use of calibrated secondary standards for scale-up to very large applications (trains, marine transport, aircraft).

4 Design of the primary standards

4.1 METAS primary standard

4.1.1 Mechanical design

The METAS Hydrogen Field Test Standard 2 (HFTS2) consists of six 104 L pressure tanks mounted into an aluminium frame. The tanks are type 4 cylinders (carbon fibre-reinforced epoxy with a plastic liner) with a service pressure of 70 MPa at 15 °C corresponding to a capacity of 2.4 kg or 4 kg of H_2 each at 35 MPa or 70 MPa, respectively. Each tank weighs 87 kg with dimensions of 182 mm x 370 mm. The total weight is around 820 kg.

The HFTS2 is equipped with six 27 cm long Pt100 probes inserted at one end of each tank and six digital pressure transducers with a 1000 bar range. Additional Pt100 probes are mounted on the HFTS2 to monitor temperature around the tanks. All probes are equipped with Bluetooth transmitters, so no cabling from the HFTS2 to the data-acquisition system is needed.

The frame is mounted on three 300 kg scale with 0.1 g resolution for gravimetric measurements. Uncertainty requirements made such a design choice mandatory. Indeed, a larger single 1500 kg scale would not achieve the necessary accuracy. Three scales also ensure that the system is stable at all times during weighing. The weight of the frame can be lifted from the scales by a load removal system activated by hand using large levers. This system is only used once when starting the measurements, another more refined system allows a controlled lowering of the HFTS2 on the scales.

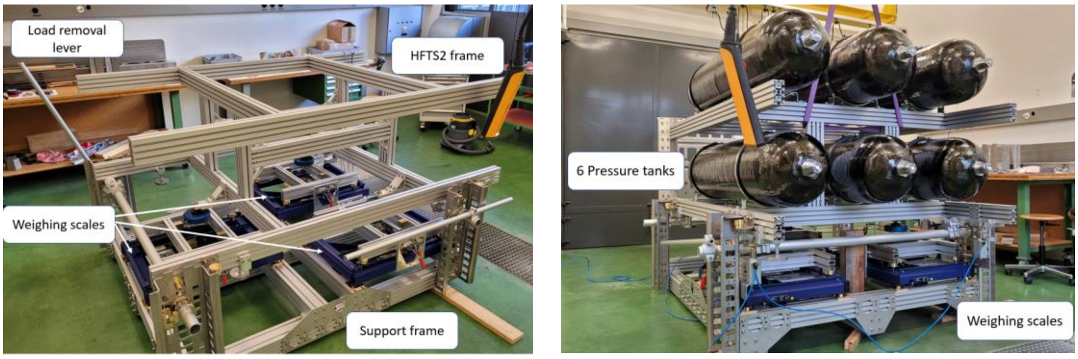


Figure 2: Left) Basic constituents of the HFTS2: support frame, HFTS2 frame and three scales. Right) Six pressure tanks mounted in the HFTS2 frame.

The challenge of such a design it to ensure that the positioning of the HFTS2 frame on the three weighing scales does not produce torquing, or if it does, that is it always withing tolerances. Moreover, all the scales should be levelled and lie in the same plane (have the same angle with respect to each other). This is done by first levelling the scales with respect to each other and then by mounting stones on each scale, which could be levelled again. Stones can be levelled to a much higher accuracy than metal, in this case 0.3 mm/m.

Now that the HFTS2 frame can be positioned on the three scales, special care has to be taken to ensure that lowering onto and raising the HFTS2 from the scale does not produce any strain on the scales.

The HFTS2 frame is equipped with three air bearings fed with nitrogen that separate the frame from the levelled stones on the scales, as shown in Figure 3. These air bearing, when switched on, allow the frame to hover a few microns above the scales and eliminate any strain on the scales. When switching off the air bearings, the frame will gently lower itself on the scales. By repeating this process several times in a row, one ensures that no strain is applied on the scales and that the frame is lowered in a controlled and repeatable way on the scales.

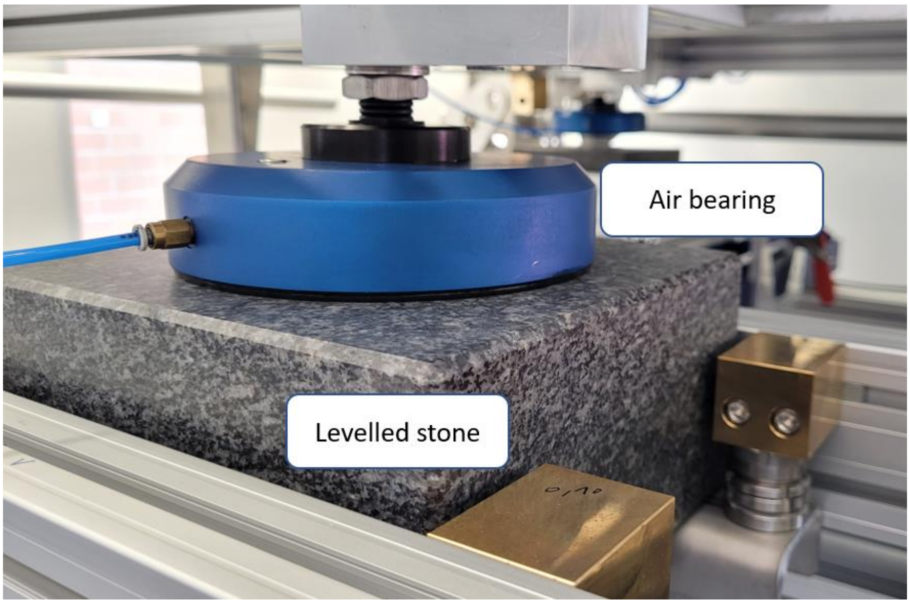


Figure 3: Air bearing on top of the levelled stone.

Centring actuators, shown in Figure 4, prevent the frame from sliding away from the scales when it is hovering, should the system not be levelled adequately.

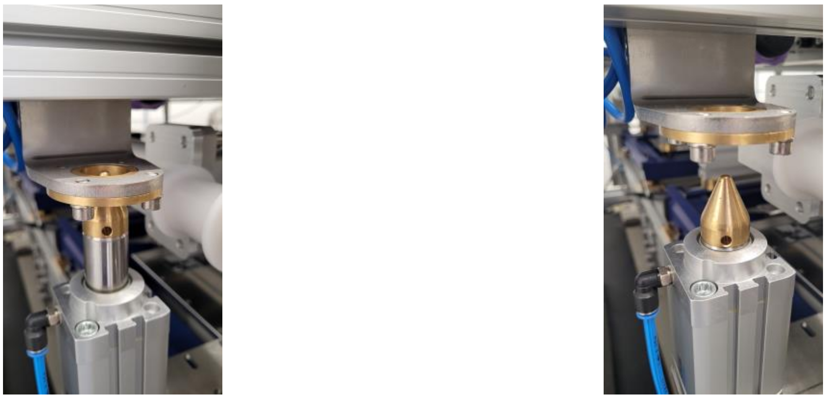


Figure 4: Actuators prevent the frame from sliding away when hovering.

This weighing method has been extensively tested and yielded a repeatability that is good enough for the required uncertainty. The uncertainty of the standard is addressed in a dedicated section.

There are also several mechanical transport safety systems to ensure that the frame remains immobile during transport and does not lead to an overload of the scales.

The complete system, installed in the trailer, in shown in Figure 5, where one recognises the pressure tanks behind the control panel with all the control valves, pressure reducers for venting the tanks after filling, the nozzles for connecting 70 MPa of 35 MPa dispensing lines.

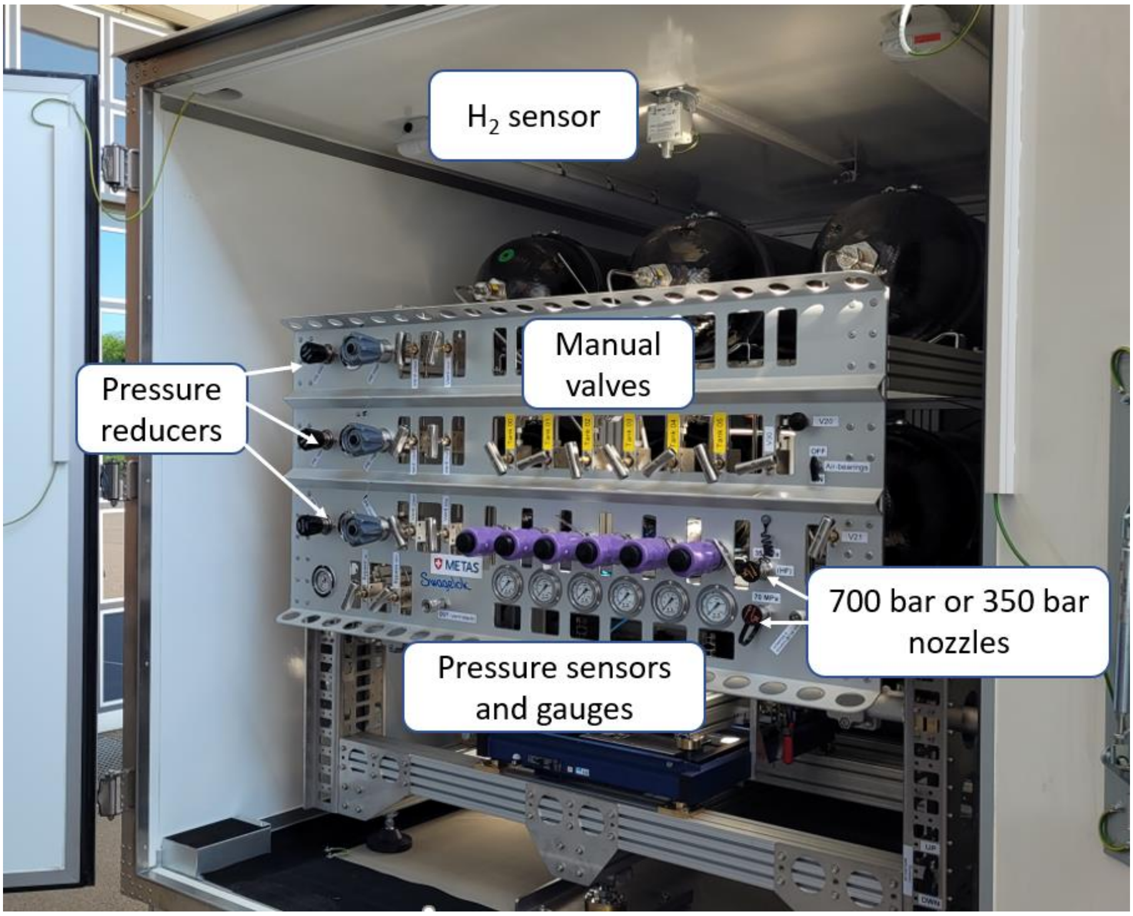


Figure 5: Complete HFTS2 standard installer in the trailer.

4.1.2 Flow scheme

The Piping and Instrumentation Diagram (P&ID) is shown in Figure 6. The schematic is divided into three parts, each delimited by a box. The components located in the blue box are all mounted on the HFTS2 frame and weighed by the scales. The components in the pink box are located in the trailer in the explosive atmosphere zone. The green box contains the components that are in the safe area in the trailer, where no explosive atmosphere constraints apply.

The system is composed of three lines: an inlet connected to the hydrogen dispenser (35 MPa HF or 70 MPa), a purge line to flush the system with N_2 and an outlet line for blowing the tanks down. Each pressure tank is equipped with a temperature and pressure sensor for monitoring purposes, a rupture disk for safety and a hand valve. The hydrogen from the dispenser enters the HFTS2 through a nozzle and is guided into one or several tanks, depending on the mass flow rate range that one wants to test. After filling, the filled tanks are emptied through a vent stack after passing through a cascade of pressure reducing valves.

An air conditioning unit is responsible for maintaining constant ambient conditions around the HFTS2. Ambient conditions are monitored by a barometer, hydrometer and several temperature sensors that are located around the scales and tanks. All the piping in contact with hydrogen is made of medium pressure 3/8" tubing FK fittings (inner section of 22 mm^2) in 316-stainless steel, except for the piping connecting the 70 MPa nozzle to the main piping, which is ¼" tubing FK (inner section of 7.3 mm^2) and the piping after the rupture disks, which are ½".

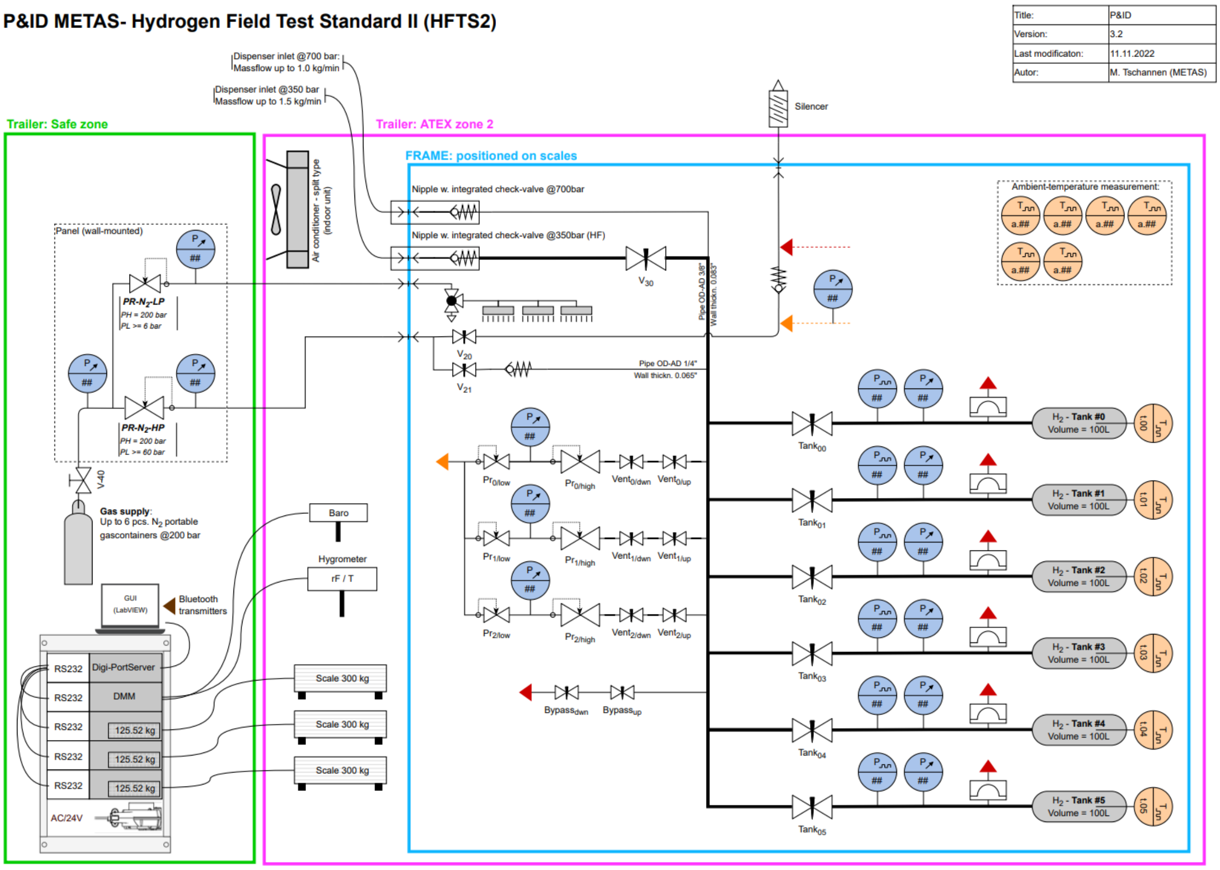


Figure 6: P&ID of the HFTS2 standard.

A N$_2$ supply, located in the non-explosive atmosphere zone of the trailer, allows flushing the lines and the tanks. Two hydrogen sensors are mounted around the HFTS2 for monitoring potential leaks and activate the safety system through a gas surveillance system.

All mechanical and electrical components are certified for functioning in an explosive atmosphere (ATEX Zone 2 at least). The data-acquisitions system is located in the safe zone from the trailer.

4.1.3 Trailer design

The HFTS2, its data-acquisition system and equipment are installed in a thermally isolated trailer (60 mm wall thickness) with dimensions 3980 mm x 2030 mm x 2000 mm with a maximum load capacity of 3500 kg. The trailer is separated in one explosive atmosphere zone housing the HFTS2 and a safe zone by a partition wall.

Figure 7 shows a side view of the trailer. The air-conditioning unit maintains constant ambient conditions in the Ex-zone of the trailer. An electrical generator will feed power to the three scales during travelling to limit warm-up time and therefore allow a faster deployment of the HFTS2 for on-site measurements. A hydraulic system allows levelling the trailer once on site.

Figure 7: Left) Side view of the trailer, Right) same as left but with doors open.

Several hatches, some of them are shown in Figure 7 and Figure 8, allow access to essential parts of the HFTS2.

Figure 8: Access doors to some parts of the HFTS2.

4.2 NEL primary standard

4.2.1 Mechanical design

The NEL Heavy Duty Hydrogen Field Test Standard (HD HFTS) consists of three 350 L pressure vessels mounted on a separate aluminium frame. The tanks are type 4 cylinders (carbon fibre-reinforced epoxy with a plastic liner) with a service pressure of 35 MPa at 15 °C corresponding to a capacity of 8.4 kg of H_2 each at 350 bar. The empty weight of each tank is 111 kg with outer diameter of 509 mm and 2342 mm length. The tanks have two thermal pressure relief valve devices each which activate in case of a fire. Figure 8 shows the general arrangement of the HD HFTS with three tanks.

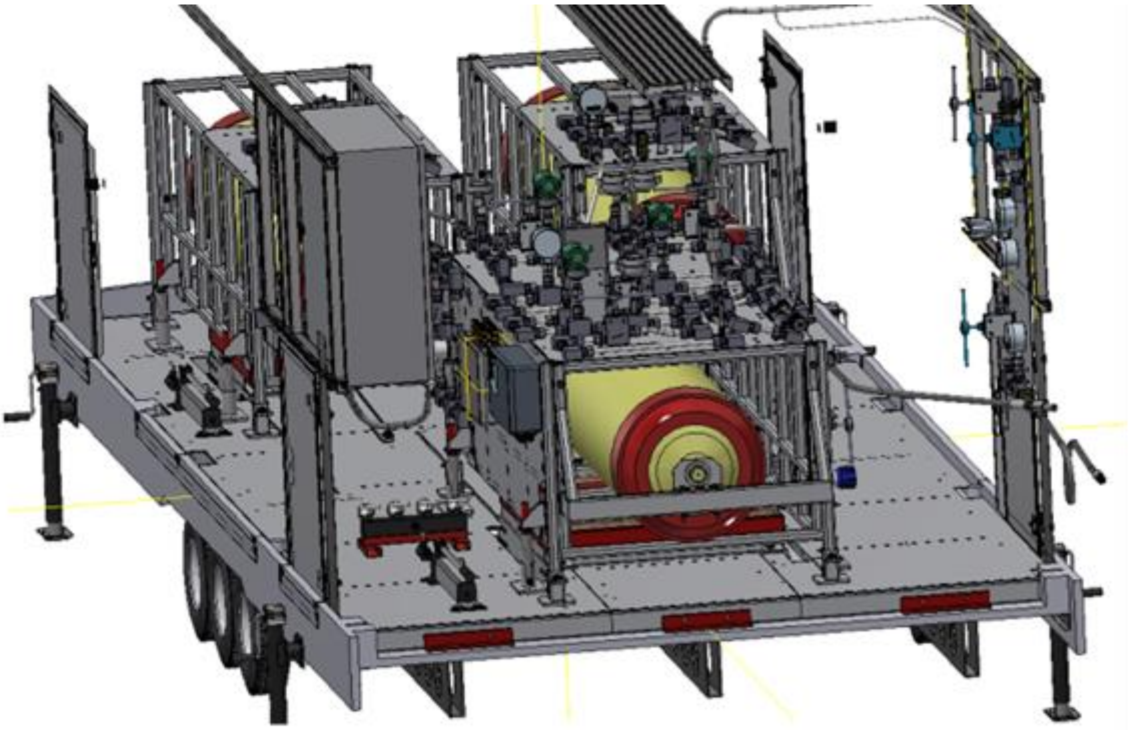


Figure 8: General arrangement of the HD HFTS.

The test standard operates by gravimetric measurement for the amount of hydrogen dispensed. Unlike the METAS HFTS2 which carefully balances the entire weight of the tanks, frame and instruments across three weigh-scales, the NEL system instead weighs the three tanks separately. Each tank is weighed by a different scale. A 600 kg capacity weigh scale WS002 is used for tank TK002's frame since it also supports most of the piping and instrumentation for HD HFTS. Frames for tanks TK001 and TK003 are each weighed using separate 300 kg capacity weigh scales WS001 and WS003. The two 300 kg weigh scales have a 0.05 g resolution by enabling 10x mode on the display terminals for each unit, whilst the 600 kg weigh scale has a resolution of 0.1 g.

The HD HFTS has temperature and pressure sensors installed on the inlet piping to the tanks. They are required for both metrological and safety purposes. A Coriolis flow meter is also installed on the frame for tank TK002. ATEX certified quick connectors are used on the electrical cabling to these sensors to allow the cables to be disconnected for weighing each frame before and after filling with hydrogen gas.

When the system is being filled, the inlet piping to the 3 tanks is connected by flexible hose. This allows the full 1050 L volume to be filled. Before weighing, the flexible hoses are isolated, vented and disconnected, to ensure separation between the weigh scales and prevent eccentric loading on the balances.

4.2.2 Flow scheme

The P&ID for the HD HFTS is shown in Figure 9. The schematic is divided into four parts, each delimited by a box. The components inside the green box are all mounted on the frame for tank TK001 and are weighed using scale WS001. The components in the blue box are on the frame for tank TK002 and weighed by scale WS002. The red box has the components fixed to the frame for tank TK003 and weighed by scale WS003. The nitrogen panel used for purging the tanks and piping after testing at a hydrogen refuelling station is shown in the orange box. All four parts shown in the P&ID are within the potentially explosive atmosphere zone of the trailer and therefore need to conform to ATEX regulations.

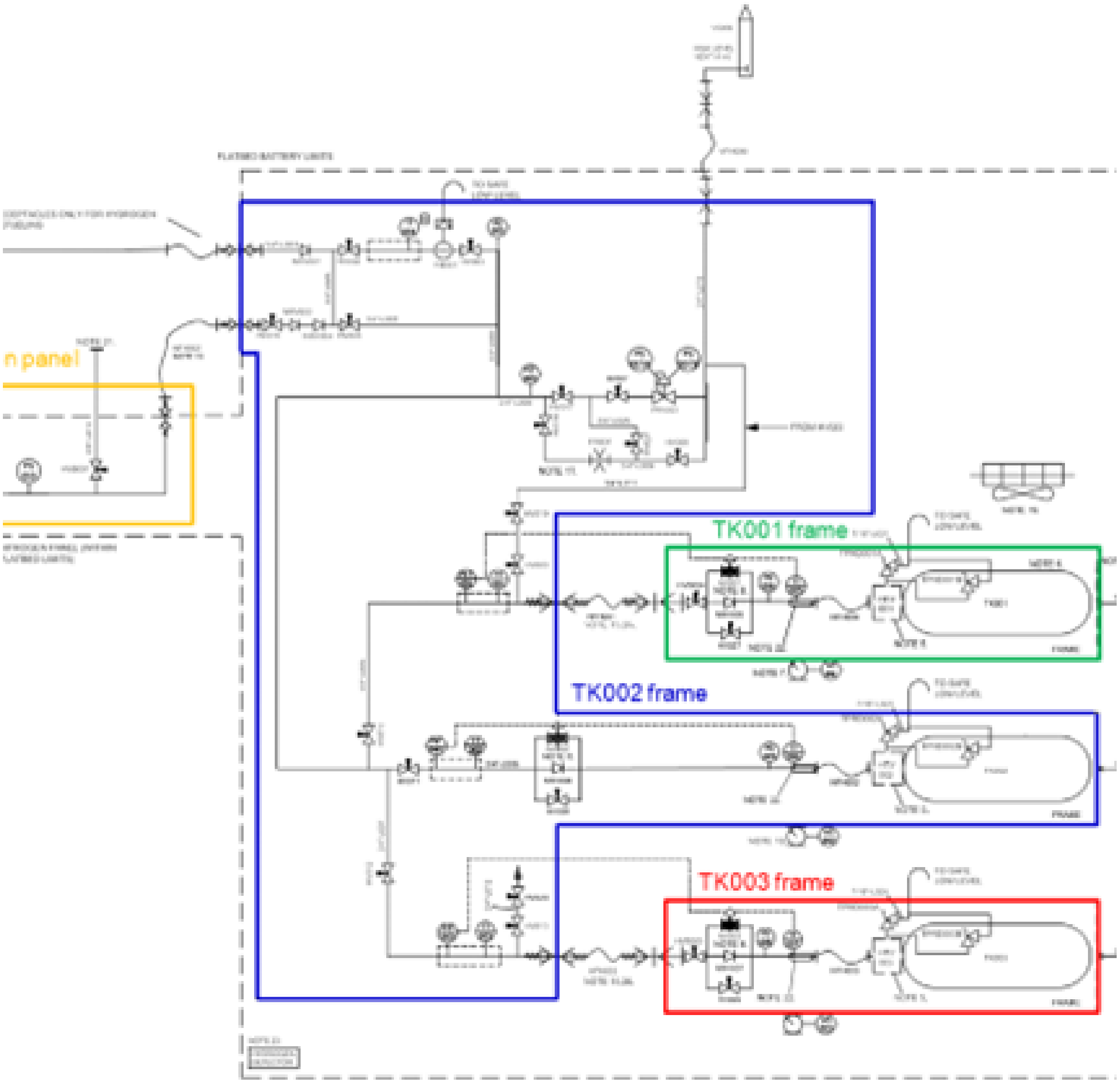


Figure 9: P&ID of the HD HFTS.

During filling, the dispenser nozzle connects to the 35 MPa receptacle on the frame for tank TK002. Depending on the type of test, either one tank (i.e., TK002) or all three tanks may be filled with hydrogen gas. Flexible hoses linking the piping on the frames for tanks TK001 and TK003 are disconnected before weighing. Hence the gas in this piping and hoses needs to be vented. It is therefore necessary to quantify the amount gas vented as it should be accounted for in the in the calculation of dispensed hydrogen. The mass of hydrogen vented from the hoses is determined from their estimated internal volume and measured temperature and pressure.

After filling, the tanks are emptied at a controlled rate by using a pressure regulator and a flow restrictor. A 4 m tall vent stack located in a safe area disperses the hydrogen gas.

Several sensors installed inside the HD HFTS trailer monitor the ambient pressure, temperature and relative humidity so appropriate buoyancy corrections can be applied to the mass measurement. All the piping in contact with hydrogen is made of medium pressure ¾-inch tubing in 316 stainless steel and therefore is suitable for hydrogen gas at 35 MPa pressure.

4.2.3 Trailer design

The HD HFTS is installed inside a trailer which has been modified for use in an ATEX environment. The floor of the trailer was strengthened with additional aluminium box sections to provide a stable surface for the gravimetric measurements. Three large doors allow entry into the trailer, while four side panels provide additional access. Four jacking posts are used to level the trailer. Figure 10 shows the trailer with some of the access doors and panels open, and the jacking posts.

Figure 10: HD HFTS Trailer with access doors and panels open.

For the HD HFTS to meet the ATEX Zone 2 classification, the trailer required an LEV system consisting of a duct, an ATEX rated fan and a flow sensor as shown in Figure 11. The weigh scales are placed beneath frames using separate hydraulic lift systems to raise each frame when required.

Figure 11: LEV duct with exhaust fan installed on the side of the trailer.

4.3 METAS uncertainty budget

The uncertainty calculation for the METAS primary standard is based on the uncertainty budget presented in [4].

The dispensed mass into the standard, corrected for buoyancy and apparent mass reading from the scale is given by:

$$m_{H2} = (W_2 - W_1) \cdot \left(1 - \frac{\rho_a}{\rho_N}\right) + V_0 \cdot [\rho_{air2} \cdot (1 + \lambda \Delta P_2) - \rho_{air1} \cdot (1 + \lambda \Delta P_1)] + V_{frame} \cdot (\rho_{air2} - \rho_{air1}) \, , \tag{1}$$

where W are the readings of the scale and the subscripts denote the reading before and after the filling, respectively, V_0 is the external volume of the tank or tanks at ambient conditions with no internal pressure, λ is the pressure expansion coefficient of the tanks and V_{frame} is the volume of the frame. The factor $\left(1 - \frac{\rho_a}{\rho_N}\right)$ turns apparent mass into true mass where $\rho_a = 1.2 \, \frac{kg}{m^3}$ and $\rho_N = 8000 \, \frac{kg}{m^3}$ are the densities of air and stainless steel at reference conditions, ρ_{air} is the density of the air around the scale and the tanks before and after the fill and is calculated using the formula by Giacomo [5].

The uncertainty of the gravimetric measurement can be calculated by

$$\left[\frac{u(m_{H2})}{m_{H2}}\right]^2 = \sum_i S_{x_i}^2 \cdot \left(\frac{u(x_i)}{x_i}\right)^2 \tag{2}$$

where x_i are the measurands from Equation (2) and S_{x_i} are the normalised sensitivity coefficients for each variable and can be calculated by

$$S_{x_i} = \frac{\partial m_{H2}}{\partial x_i} \cdot \frac{x_i}{m_{H2}}. \tag{3}$$

Each scale has a resolution of 0.1 g and the three scales are considered as a weighing system. It was characterised extensively up to 30 kg for:

Test	Description	Uncertainty contribution (k=1)
Extreme positioning	the HFTS2 can be lowered on the scale using the air bearings, one can move the HFTS2 to position itself on a different area of the scale	0.85 g
Asymmetric loading	the calibration masses are not loaded symmetrically with respect to the centre of mass, same as eccentricity for a single scale	0.75 g
Linearity and hysteresis	deviation of the scales with respect to the reference masses	0.35 g
Drift	Monitoring of the buoyancy corrected mass during 3.5 h	0.56 g
Repeatability		0.34 g
TOTAL		**1.36 g**

Table 5: Scales uncertainty assessment of the primary standard

We consider a conservative uncertainty of 1.5 g (k=1) for the weighing system. Air density depends mainly on the parameters pressure, temperature and humidity. These quantities are continuously logged with uncertainties of 0.25 mbar, 0.25 °C and 2.5 % and allow a determination of air density needed for the buoyancy correction of 0.1 % (k=1). The digital pressure transducers have a resolution of 0.1 bar and have been calibrated up to 1000 bar with an absolute error of at most 3 bar and an uncertainty of 0.2 bar. We consider here an uncertainty of 5 bar.

Table 6 presents a summary of the uncertainty components and their magnitude for the gravimetric method for a test collection of 1 kg of gas. The scales and the air density have the largest contribution. The values presented here yield an expanded uncertainty of 6.3 g for 1 kg, which is higher than the target value of 0.5 % but still fulfils the requirements for type approval testing of a HRS according to OIML R139:2018.

Uncertainty component	**Nominal value**	$u(x_i)$ **%**	**Contribution %**
Initial mass	850 kg	$1.8 \cdot 10^{-4}$	23
Final mass	851 kg	$1.8 \cdot 10^{-4}$	23
Tank volume	1.0 m^3	5.0	3.5
Frame volume	0.1 m^3	5.0	< 0.1
Initial air density	1.178 kg/m^3	0.1	24
Final air density	1.181 kg/m^3	0.1	24
Initial tank pressure	2 MPa	10	< 0.1
Final tank pressure	35 MPa	0.57	< 0.1
Pressure coefficient	$2.2 \cdot 10^{-10}$ Pa^{-1}	5	2

Table 6: Uncertainty budget for the METAS standard.

4.4 NEL uncertainty budget

Like the METAS system, the NEL uncertainty budget is determined from the propagation of the input uncertainties in each of the measurands of equation 2. Since the NEL standard is based on three separate weigh scales weighing separate tanks, there is an additional uncertainty to consider for the hydrogen vented from flexible hoses before weighing. This uncertainty source applies when at least two weigh scales are in use. The 600 kg balance is the main weigh scale, which is always in use, and can be used alone to collect up to 8 kg of hydrogen. Between 8 and 16 kg of hydrogen collected, one of the 300 kg weigh scales is also required, and both are required if collecting more than 16 kg of hydrogen. The uncertainty budget for the weigh scales is shown in Table 7 and Table 8.

Table 7: Uncertainty of 300 kg weigh balance

Rank	Uncertainty Source	Units	Value	Expanded Uncertainty U	Relative Uncertainty U* (%)	
6 - 0.4%	Resolution of display	kg	300	0.000050	0.00002	
5 - 2.1%	Calibration curve fit	kg	300	0.000087	0.00003	
3 - 3.0%	Repeatability	kg	300	0.000120	0.00004	
2 - 21.1%	Calibration weights	kg	300	0.000316	0.00011	
4 - 2.8%	Maximum deviation from calibration	kg	300	0.000100	0.00003	
1 - 70.5%	Drift (over 90 min)	kg	300	0.000500	0.00017	
	Overall Uncertainty	kg	300.0	**0.00069**	0.00023	

Table 8: Uncertainty of 600 kg weigh balance

Rank	Uncertainty Source	Units	Value	Expanded Uncertainty U	Relative Uncertainty U* (%)	
6 - 0.7%	Resolution of display	kg	600	0.0001	0.00002	
5 - 2.2%	Calibration curve fit	kg	600	0.000173	0.00003	
4 - 3.2%	Repeatability	kg	600	0.000240	0.00004	
1 - 56.3%	Calibration weights	kg	600	0.001001	0.00017	
2 - 18.8%	Maximum deviation from calibration	kg	600	0.0005	0.00008	
3 - 18.7%	Drift (over 90 min)	kg	600	0.0005	0.00008	
	Overall Uncertainty	kg	600.0	**0.00133**	0.00022	

The tables show that for the 600 kg weigh scale, the uncertainty for a 1 kg weight is about 1.33 g and for the 300 kg weigh scale it is about 0.69 g. The other important input sources for the uncertainty budget are shown in Table 9.

Table 9: Uncertainty budget of Heavy-Duty NEL test standard

Rank	Uncertainty Source	Unit	Value	Expanded Uncertainty U	Relative Uncertainty U* (%)	
1 - 44.0%	Initial mass, W_1	kg	400	2.668E-03	0.0007	
1 - 44.0%	Final mass, W_2	kg	401	2.668E-03	0.0007	
7 - 0.0%	Tank volume, Vo	m^3	0.35	1.000E-02	2.8571	
4 - 4.9%	Air density initial, ρ_{air1}	kg/m^3	1.183	1.613E-03	0.14	
3 - 4.9%	Air density final, ρ_{air2}	kg/m^3	1.179	1.611E-03	0.14	
6 - 0.0%	Frame volume, V_{frame}	m^3	0.2	1.000E-02	5.0000	
8 - 0.0%	Tank Initial pressure, P_1	Pa	2.00E+06	4.000E+05	20.00	
9 - 0.0%	Tank Final pressure, P_2	Pa	3.50E+07	4.000E+05	1.143	
5 - 2.2%	Pressure coefficient, λ	Pa^{-1}	2.20E-10	4.400E-11	20	
10 - 0.0%	H2 mass in inventory volume	kg	2.14E-09	5.38E-11	2.52	
	Overall Uncertainty	kg	1.000	4.023E-03	0.402	

The overall uncertainty of the NEL test standard for 1 kg hydrogen load is 0.4%. This value is based on using only the 600 kg weigh scale which will be pre-loaded to 400 kg (empty weight of tank, piping etc.).

5 Requirements for secondary standards

The requirements for secondary standards are best defined through the definition of technical specifications.

5.1 Scope and key performance goals

Building a mobile and fast deployable test rig for the field- verification of HRS (light-duty and heavy-duty vehicles) using calibrated secondary standards with a target uncertainty that is compliant with the OIMLR139:2018.

This mobile system shall not alter the operating procedure neither for the refilling station nor for the vehicle to be filled. In this case, the vehicle should not notice a difference compared to a normal refueling (vehicle could either be real cars or dummy tanks). Safety and protection of personnel and material take precedence over speed of refueling with the master meter method.

The key performances are reminded below:

- Better than 2% accuracy (goal of project).
- Uncertainty better than 0.67 % (1/3 class 2 MPE of OIML R139)
- Traceable to primary standards developed in 16ENG01 MetroHyVe or 19ENG04 MetroHyVe 2.
- Faster and easier to deploy than the primary standards.
- Operate on any flow rate, gas pressure and temperature provided by the HRS (in relation to SAE J2601/1 for LDV and SAE J2601/2 for HDV protocols see also PRHYDE for revisions of SAE J2602/2 https://prhyde.eu/).

5.2 Technical specifications of the secondary standard (master meter)

5.2.1 Reference meter

The reference meter of the secondary standard shall:

- Be a mass flow meter with ATEX certification and specified up to a pressure of at least 900 bar for hydrogen.
- Cover the mass flow rate range from (0.1 to 10) kg/min.
- Cover the gas temperature range (-40 °C to 50 °C).
- Have a transmitter with logging options of mass flow rate and tubing temperature.
- Have a proven track of successful application in hydrogen flow metering.
- have a stable zero flow condition (even at variable temperature).

5.2.2 Connections to and from the secondary standard

The secondary standard shall be placed between the dispenser and the final hydrogen container and not alter the refueling process from the dispenser. If the final hydrogen container is a vehicle, no contaminant from the secondary standard shall end up in the vehicle's tank.

The mobile secondary standard shall be usable for verifying HRS working at 35 MPa (also HF = High Flow) and 700 bar.

The mobile secondary standard shall allow data communication between the dispenser and the final hydrogen container if data communication is present.

5.2.3 Instrumentation

All instrumentation shall be specified for hydrogen in the relevant pressure range (ATEX IIC).

The mobile secondary standard shall allow logging of hydrogen temperature in the piping, before and/or after the meter.

The mobile secondary standard shall allow logging of pressure in the piping before and after the meter. It shall also be equipped with at least one dial pressure gauge to allow reading of the pressure value at every given moment during testing, operating independently of electrical supply.

5.2.4 Internal piping

Internal piping shall be specified for maximum operating pressures up to 1000 bar for hydrogen.

Internal piping volume shall be as small as possible to limit inventory (dead) volume but not cause significant flow restriction in the HRS. The piping length should be as short as possible, and the inner diameter should not be smaller than the inner piping diameter used in the HRS.

5.2.5 Safety

The secondary standard shall have an ATEX certification. Unwanted backflow shall not be possible. No leakage of hydrogen shall occur, regardless of whether inlet or outlet is disconnected first. The operator shall be able to vent the complete internal volume (piping + meter) from any pressure level into the environment. The secondary standard shall have connections to purge the internal volume (piping + meter) with nitrogen. All valves shall have a defined function that is clearly described. All measurements shall be done with as few actions required by the operator as possible. A complete handling procedure and documentation shall be always available to the operator.

5.2.6 Usability

The secondary standard shall be lighter than 60 kg and mobile (for instance the Swiss national accident insurance fund's requirements are 30 kg/person). The secondary standard shall be equipped with handles for easy lifting. All apparent surfaces shall be in a temperature range that the operator can touch them without risk of burn when wearing appropriate gloves or similar.

All fittings shall be placed to facilitate loosening/tightening, replacement and inspection. Standard fittings shall be used. The transmitter of the flow meter shall be located separately so that the operator can connect to it using a non–ATEX compliant system (or RS485 protocol can be used to acquires data during usage).

Buildup of condensate and subsequent icing on the piping shall be avoided where feasible. In any case, the influence of icing on the piping on the performance of the meter should be known.

6 Design of the secondary standard

The following system description presents a system that fulfills all the requirements from Section 5.2.

6.1 General description

The secondary standard consists of 2 mobile and transportable skids The first skid is ATEX certified and in an enclosure:

- A Coriolis mass flow meter designed for hydrogen flow metering up to 900 bar.
- A 35 MPa HF fitting and a 70 MPa fitting on the inlet side to allow the connection of the refueling hose from the dispenser to the secondary standard. Both fittings are equipped with check valves to prevent back flow. The fittings are isolated from each other by ball or needle valves.

- A 35 MPa HF fitting and a 70 MPa fitting on the outlet side to allow the connection of the refueling hose from the secondary standard to the final hydrogen container. The fittings are isolated from each other by ball or needle valves.
- Pipes and valves connecting the inlet and outlet fittings to the flow meter.
- A purging line and a venting line with their respective fittings, the former to purge either with nitrogen of hydrogen, the latter for venting the line after a measurement.
- Instrumentation for measuring pressure and temperature in the pipes.
- The transmitter from the Coriolis meter.

The second skid need not be ATEX certified and houses:

- Safety barriers to limit power delivery to the electrical equipment in the ATEX skid.
- Connections to the power supply.
- Connections to the data acquisition system.

A Piping and Instrumentation Diagram (P&ID) of a potential skid with the master meter is presented as an example in Figure 9.

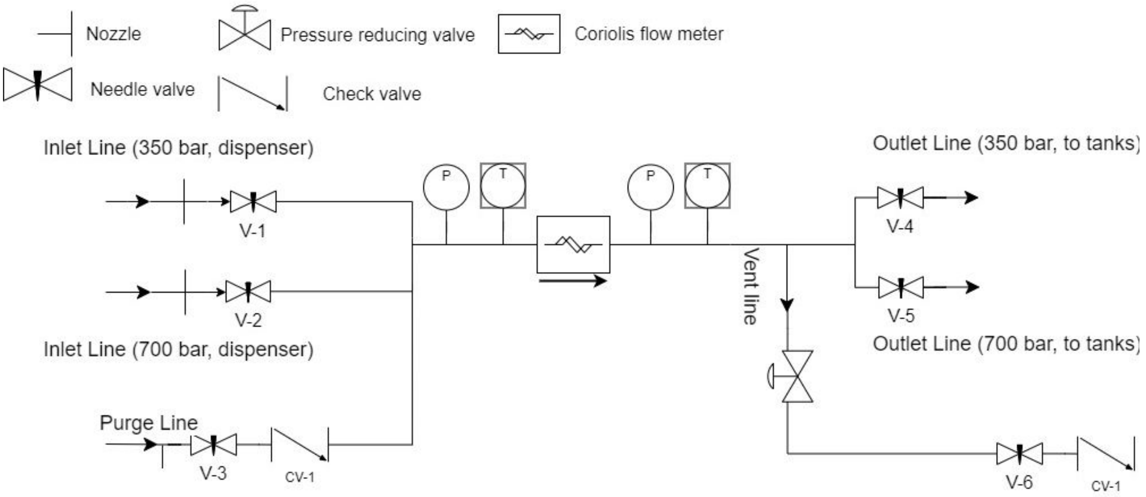


Figure 9: P&ID of the first skid housing the master meter.

Flow direction is from left to right. There are three inlet lines: one for 35 MPa HF, one for 70 MPa and one for purging the system with an inert gas or hydrogen. These three lines cannot be used simultaneously and lead to the Coriolis meter. Pressure and temperature are monitored upstream and downstream of the master meter using dedicated probes. Downstream of the meter, piping is again divided in three lines: two for dispensing hydrogen and one venting the line.

A typical layout of the secondary standard during field verification is indicated in Figure 10, flow direction is from left to right. One recognises the fuelling nozzle for the dispenser that is connected to the secondary standard composed of a receptable to connect the nozzle, some valves, the Coriolis meter and the exit hose connecting the secondary standard to either the fuel line or dummy tanks, where the hydrogen will be delivered to. The volume shown in red corresponds the part of the piping from the secondary standard which contains hydrogen that was counted by the process meter from the HRS but not counted by the meter from the transfer standard. This quantity must be accounted for.

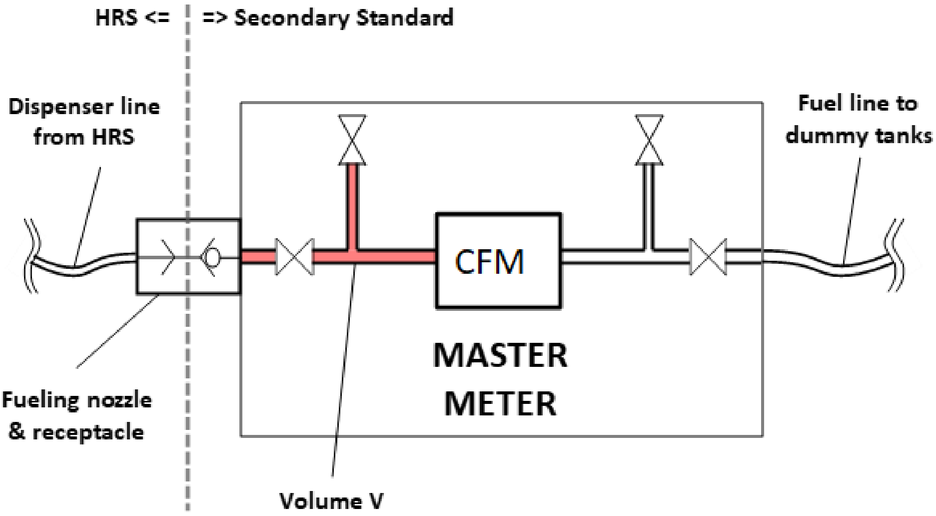


Figure 10: Schematic and layout of the secondary standard place between the dispenser and a fuel line or dummy tanks.

6.2 Purging, cooling and operation procedure

6.2.1 Purging procedure before starting a measurement.

Description:

The secondary standard may contain nitrogen at the beginning of the verification's procedure. In case of a complete system with dummy tanks, no purging is expected. On the other end, nitrogen should not end up in the tank of the hydrogen vehicle. A purging procedure with hydrogen of the portable set up must be applied. If the hydrogen vehicle tolerates a small amount of nitrogen, then purging before the measurement is not required.

1. Proposal 1: pressing the 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 using the upstream purge line as shown in Figure 9.

6.2.2 Precooling of the Master Meter

When the Master Meter is used on a 35 MPa or 70 MPa refilling process, the hydrogen passing the flow meter could be at a temperature of -40 °C (called T40 in the SAEJ2601 protocol). This temperature can be modified from (-40 °C to -30 °C or -20 °C). The gas temperature could also be at ambient condition when not pre-cooled or at -10 °C or 0 °C when the WENGER protocol is used.

The master meter will adjust its temperature after the initialization of the filling process to the temperature of the hydrogen. This unsteady working condition could potentially affect the accuracy of the flow meter. Precooling the flow meter can mitigate this problem. One currently used method is to pre-cool the flow meter by using dry ice. However, it is not self-evident that this method will provide the desired benefit. Dry ice sublimates at -78 °C and thus it is not clear to which temperature the flowmeter will cool down when surrounded by dry ice. It is also unclear if the same temperature conditions can be replicated on days with different ambient temperatures. Dry Ice sublimates at ambient conditions and therefore cannot be kept at (semi-)permanent stock. However, to stock it for several days is not problematic. This complex availability makes cooling by dry ice during the test phase of master meter disadvantageous. Cooling with a suitable fluid looks more promising as the temperature is controllable by adjusting the flux of nitrogen into the box with the master meter.

A promising way of keeping the flow meter cool would be to place it entirely into a cooling chamber in which the temperature is controlled. This guarantees that the complete meter is cooled down. The remaining space in the cooling chamber could be filled with ice or another material in order to increase the stability of the temperature due to the added mass. The setpoint of the climate chamber should be at -35 °C for T40 or other value close to the target gas temperature.

6.2.3 Proposed operation procedure with the secondary standard

1	Install the secondary standard in the vicinity of the dispenser and connect all signal cables to the transmitter and computer.
2	Connect the dispenser to the secondary standard.
3	Replace the nitrogen in the secondary standard with hydrogen (optional).
4	Connect the hose to the vehicle, primary standard or dummy tanks to be filled with the correct dispenser/nozzle.
5	Start the filling process.
6	Terminate the filling process.
7	Disconnect the line between the dispenser and the secondary standard.
8	Disconnect the connection to the vehicle (if needed) or apply correction for dead volume.
9	Get the corrected totalization of hydrogen mass on the Coriolis transmitter and compare it with the display of the dispenser.
10	Inflate the secondary standard with nitrogen and subsequently release the gas trapped inside, into the environment after the testing protocol

Table 10: Operation procedure when using a secondary standard for HRS verification.

6.3 CESAME secondary standard

The CESAME secondary standard Hydrogen Refuelling Station Secondary Reference (HRSmsr) meets the requirements from the previous sections and its main feature are presented below:

- 2 main containers: the “Rover” which holds the master meter in a thermally controlled enclosure and the trailer which holds dummy tanks, valves, safety equipment. Both are ATEX (zone II – IIC).
- The “Rover” is controlled in temperature and can be set from -30°C down to -10°C or at ambient condition.
- There are two dedicated inlet lines for H70 LDV and H35 HDV.

The 'Rover' is shown in Figure 11, connected between a HRS and the CESAME primary standard. The master mete, a pressure sensor and a hydrogen detector are mounted in the thermally controlled enclosure on the trolley. The dispensing hose of the HRS is connected to the Rover, which is then connected in this case to the CESAME primary standard. This is a typical setup for calibrating the master meter in the Rover.

Figure 11: The CESAME Rover connected to the CESAME primary standard at the Sorigny (37) HRS.

A close-up view of the Rover and the inside is shown in Figure 12. In the right image, one sees the inside of the Rover with the master meter, a pressure sensor and a H_2 detector.

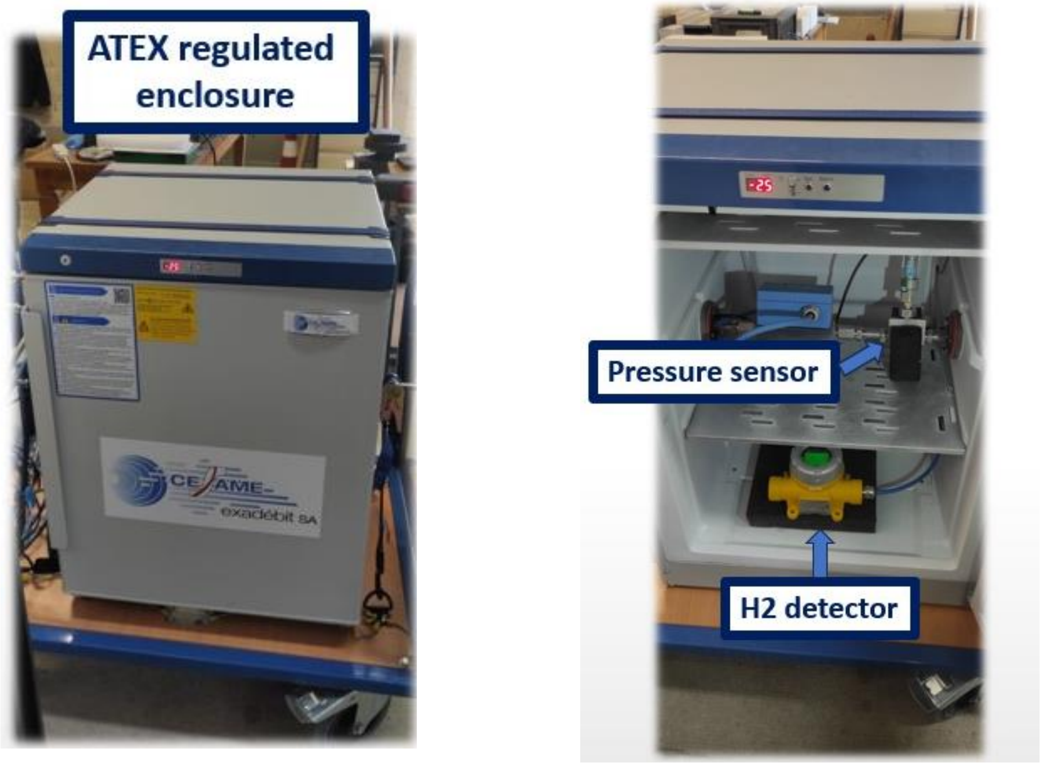


Figure 12: Left) Close-up view of the Rover, Right) Inside of the Rover.

6.4 Uncertainty and Corrections

One must be taken into consideration the difference between the mass delivered by the HRS and the mass measured by the secondary standard. Indeed, at the start of the refuelling, the piping indicated in red, the inventory volume, in Figure 10, located before the master meter from the secondary standard, contains gas that will be pushed through the master meter but was not measured by the process meter in the dispenser. The same applies to the end of the refuelling process, where this same volume will now contain gas metered by the dispenser but not by the master meter.

From the volume of this part of piping, pressure and temperature conditions as well as density, one can determine the mass of gas contained before and after the refuelling in this portion of piping.

This leads to a correction of the measured mass by the secondary standard and the corrected mass is given by Equation (4)

$$m_{std} = m_{meas} - m_{vol,ini} + m_{vol,final} \tag{4}$$

m_{std} is the corrected mass from the secondary standard that is to be considered as the reference value from the standard, m_{meas} is the mass as indicated by the master meter, $m_{vol,ini}$ and $mv_{ol,final}$ are the masses of gas in the inventory volume before and after the refuelling, respectively. One should keep in mind that this inventory volume is small and will lead to a correction of a few grams at most. As long as you correct for it, its uncertainty contribution will be a fraction of grams and negligible.

The uncertainty contributions come mainly from the master meter. The calibration of the master meter with a gravimetric standard is an important contribution. Zero flow stability of the master meter will contribute to the uncertainty and depends on the duration of the refuelling process. It is therefore important to perform a zeroing of the meter at the beginning of every measurement. The mean value should be recorded.

Two further main contributions are the repeatability of the meter, which can be determined from field-measurements and calibrations against a gravimetric standard, and the reproducibility. This latter quantity is difficult to determine because it would require monitoring the calibration of the master meter over an extended number of calibrations and there is no such data now. The current value is an estimate.

The uncertainty budget is summarised in Table 11.

Table 11: Uncertainty budget contributions for the secondary standard.

Uncertainty source	Uncertainty (k=1)	Comment
Traceability	2.5 g	Calibration with a gravimetric standard
Zero flow	1 g/min	Obtained from measurements
Repeatability	0.2 %	Calibration with gravimetric standard
Reproducibility	0.2 %	Estimation

Refuelling time for testing at MMQ (1 kg) is estimated to 1 min and yields an expanded uncertainty of 8 g or 0.8 %. A full fill of 4 kg and a refuelling time of 4 min yield an expanded uncertainty of 25 g or 0.61 %, which is close to the third of the MPEs allowed by the OIMLR139:2018.

Bibliography

[1] OIML, *OIML R139 - Compressed gaseous fule measuring systems for vehicles, Part 1 Metrological and technical Requirements,* OIML, 2018.

[2] OIML, *Compressed gaseous fuel measuring systems for vehicles - Part 2 Metrological controls and performance tests,* OIML, 2018.

[3] OIML, *Compressed gaseous fuel measuring systems for vehicles - Part 3 Test report format,* OIML, 2018.

[4] M. de Huu, M. Tschannen, H. Bissig, P. Stadelmann, O. Büker, M. MacDonald, R. Maury, P. T. Neuvonen, H.-T. Petter and K. Rasmussen, “Design of gravimetric primary standards for field-testing of hydrogen refuelling stations,” *Flow Measurement and Instrumentation,* vol. 73, p. 101747, 2020.

[5] P. Giacomo, “Equation for the Determination of the Density of Moist Air,” *Metrologia,* vol. 18, p. 33, 1982.

[6] *SAE J2601-1,* SAE International, 2020.

[7] *SAE J2601-2,* SAE International, 2014.

[8] E. W. Lemmon, M. L. Huber and J. W. Leachman, “Revised standardised equation for hydrogen gas densities for fuel comsumption applications,” *National Institute of Standards and Technology.*