Key comparison of gravimetric standards for hydrogen refuelling stations

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Abstract

There are currently several mobile gravimetric standards for testing and calibrating hydrogen refuelling stations in operation. These standards are compliant for fuel delivery to light-duty vehicles according to the SAE J2601 protocol and provide measurement uncertainties that are low enough to use them for type-approval testing according to OMIL R139 recommendation. An international field comparison under real conditions is required to ensure a complete acceptance of test results by operators and notified bodies.
In this paper, we will present details on a currently running comparison to settle this issue, and how it differs from standard comparisons. Specific strategies had to be adopted to make the experimental results consistent and comparable. The refuelling operating conditions can affect the outcome from the comparison and need to be taken into account. All these points are being addressed in this paper and are part of the comparison protocol. The aim is that in the end, based on the comparison results and the applied strategy, participants will be able to declare certified measurement capabilities to the BIPM.

1. Introduction

In a previous EMPIR project entitled MetroHyVe 1, partners developed several primary standards for testing the amount of hydrogen delivered by hydrogen refuelling stations (HRS) up to 700 bar [1]. The standards are compliant for fuel delivery to light-duty vehicles according to the SAE J2601 [2] protocol and provide measurements uncertainties that are low enough to use them for type-approval testing of HRS according to the OIML R139 [3] recommendation. An international field comparison under real conditions for clear acceptance by the HRS operators and notified bodies is missing. A previous unregistered comparison under laboratory conditions using nitrogen up to 40 bar took place within the MetroHyVe 1 project and yielded very positive results, alas not under real conditions.
To settle this issue, a EURAMET comparison was registered to compare and validate the existing gravimetric standards used for calibrating HRS under real conditions, i.e. by measuring the quantity of hydrogen delivered by a HRS. The outcome from this comparison should allow the participants (CESAME Exadebit from France, Justervesenet from Norway, METAS from Switzerland, BEV from Austria and NEL from the UK) to apply for Calibration and Measurement Capabilities (CMC) to the BIPM (Bureau International des Poids et Mesures) for this new quantity. Additional participants (KRISS from Korea and VSL from the Netherlands) will also take part but are not formally registered in the comparison protocol.
In this paper, we will present how this comparison is different to a standard comparison and which strategies have been adopted to make the experimental results consistent and comparable. The way the refuelling process from the HRS can affect the outcome from the comparison will be explained in detail, as well as the uncertainty considerations due to the refuelling process. An option has also been devised should some participants not be able to attend the measurement campaign scheduled at the HRS.

2. Comparison strategy

In standard comparisons, a transfer standard is shipped to participants and calibrated by each participant in its laboratory using its primary or secondary standard according to a comparison protocol. The pilot of the comparison performs several additional measurements to ensure that the transfer standard meets the required stability criteria over time or over certain testing condition ranges like pressure or temperature and that the contribution from the transfer standard can be accounted for in the uncertainty calculation of the comparison.
In this comparison, the primary standards have been designed for performing a very specific type of measurements, namely measuring the quantity of hydrogen delivered by a HRS under real refuelling

conditions, and on site. This leads to several experimental considerations, due to the design and functioning of HRS, which have to be taken into account to make sure that comparable data can be acquired by the different participants and eventually used as basis data for comparing and evaluating the standards. This is an important point to make sure that the comparison results can be used for declaring CMCs. One obvious condition is that the comparison measurements are to take place at a HRS.

One option would be to gather all the standards at a refuelling station and perform all comparison measurements within a reasonably short time span (say two weeks) using the built-in flow meter of the HRS as transfer standard. This would probably be the easiest solution but would put severe constraints on planning as mobilising five to six standards that need to be moved on trailers for performing measurements at a HRS over a limited time span would be an organisational challenge. Moreover, shipping distance could be an obstacle and would limit the participants to neighbouring countries to where the HRS is located.

To forego such constraints, it was decided to aim for flexibility: 1) the measurements are to take place at a HRS but over a much longer time span (several months), 2) instead of using the meter mounted in the HRS, another meter with a known history is mounted in series with the existing process meter of the HRS and used as transfer standard, 3) the transfer standard can now be regarded as a true transfer standard as it could be mounted in another HRS if a participant cannot attend the measurement campaign at the main HRS. It is hoped that with such a procedure, measurement results from the different participants can be compared and used to generate a reference value for the transfer standard. As this is not a standard comparison, several experimental aspects need to be explained in more detail for a better acceptance of the measurement results.

The comparison will take place at the Empa HRS, shown in Figure 1, close to Zurich in Switzerland. Empa is an interdisciplinary research institute that is part of the Swiss federal institutes of technology. The HRS is a public installation for customers to fill up their vehicles at 350 bar or 700 bar but used mainly as a demonstration facility.

3. The transfer standard

3.1 Description of the transfer standard

The transfer standard (TS) is a Coriolis meter, type RHM04L, from the manufacturer Rheonik, with its transmitter (type RHE 16) and is supplied by METAS. This is a Coriolis meter that is typically mounted in HRS in Europe and covers a flow rate range of (0.2 to 10) kg/min with a repeatability of 0.1 %. These performances relate to water as calibration fluid and are taken directly for the technical datasheet issued by the manufacturer.

Figure 1: Top) General view of the Empa HRS, the hydrogen dispenser is at the extreme right, bottom) METAS gravimetric standard installed at the Empa HRS.

The TS will be provided with the proprietary software used to communicate with the flow meter and record the measurements results.

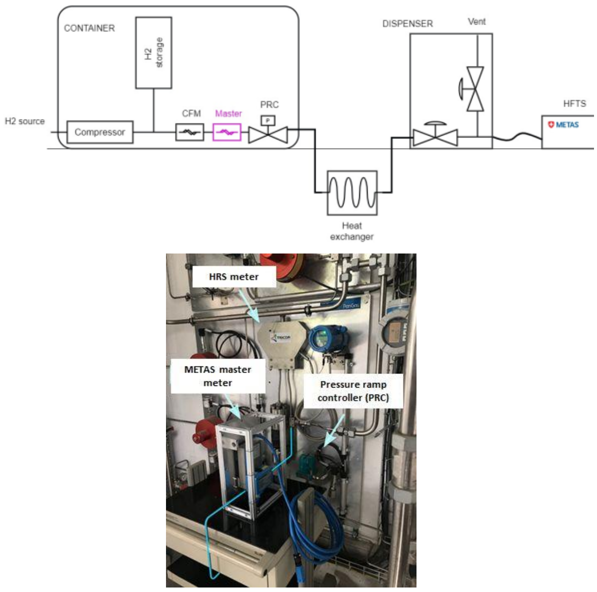

Figure 2: Top) Schematic of the experimental setup, both process and TS meter (here labelled 'Master') are located in the main container of the HRS. The gravimetric standard, labelled here HFTS, is connected to the dispenser, Bottom) Transfer standard and process meter connected in series before the pressure ramp controller.

3.2 Location of the transfer standard

In the first MetroHyVe project, it was observed that the performance of a Coriolis meter is sensitive to temperature effects and large variable errors occur if the internal temperature of the meter changes quickly. To ensure the best stability, reliability and accuracy of the flow meter, it should be operated at stable temperatures, ideally near ambient temperature.

Therefore, the TS is mounted in the HRS upstream of the heat exchanger and close to the process Coriolis flow meter from the Empa HRS. A schematic of the experimental setup and the location of the transfer standard in the Empa HRS are shown in Figure 2. The TS is located before the heat exchanger that cools down the hydrogen to -40 °C and is therefore in relatively stable ambient conditions that will not affect its performances. Indeed, if the TS was located after the heat exchanger, it would be submitted to transient temperature conditions as its tubing temperature would be close to ambient temperature at the beginning of the refuelling process and close to -40 °C at the end.

4. The refuelling process and its associated uncertainties

4.1 Mass flow rate and Average Pressure Ramp Rate

The mass flow rate in a HRS is generated by an average pressure ramp rate (APRR) as defined in SAE J2601 protocol and is not constant during a refuelling process as can be seen in Figure 3 where the mass flow rate as a function on time is shown as a continuous line. One notices a first bump due to the pressure pulse from the dispenser to check for any leaks, followed by the delivery of hydrogen during the steady refuelling process. The bump at 250 s is due to a change in pressure banks in the HRS. The dotted line indicates the integrated mass.

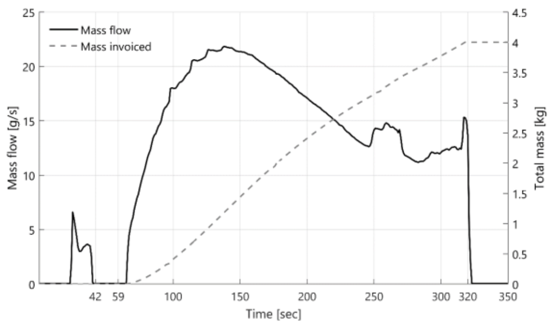

Figure 3: Mass flow rate as function of time during a refuelling process.

The average mass flow rate during delivery to the vehicle can be estimated by

$$\frac{dm}{dt} = \frac{V_{tank}}{\Delta t} \cdot (\rho_{H2,f} - \rho_{H2,i}), \tag{1}$$

where V_{tank} is the tank volume, Δt the refuelling time and $\rho_{H2,x}$ the final and initial hydrogen density in the tank at the end and the beginning of the refuelling. This equation indicates that the average mass flow rate depends directly on tank volume.

The fuelling time can be estimated using tabulated APRR value from SAE J2601. Figure 4 shows a fuelling table for a HRS working at 700 bar and delivering precooled hydrogen close to -40 °C. The table relates ambient conditions to initial tank pressure in the vehicle and establishes an APRR in MPa/min and an end pressure in the vehicle's tank. SAE J2601 contains several such tables depending on tank size ranges (here 2 kg to 4 kg hydrogen content at 700 bar and 15 °C), precooling temperature and whether there is a communication established between the dispenser and the vehicle being refuelled.

The primary standards from the different participants have different tank volumes, meaning that the average mass flow rate during a refuelling will be different for some of the participants. Moreover, ambient temperature also affects the average mass flow rate.

Table D19 - H70-T40 2-4kg Non-Communications

H70-T40 2-4kg non-comm	APRR [MPa/min]	Target Pressure, P_{target} [MPa]											
		Initial Tank Pressure, P_0 [MPa]											
Ambient Temperature, T_{amb} [°C]		0,5	2	5	10	15	20	30	40	50	60	70	> 70
> 50	no fueling	no fueling	no fueling	no fueling	no fueling	no fueling	no fueling	no fueling	no fueling	no fueling	no fueling	no fueling	no fueling
50	3,2	no fueling	77,2	76,8	76,4	76,1	75,7	75,2	74,8	74,3	73,7	72,7	no fueling
45	5,8	75,3	76,5	76,2	75,8	75,8	75,5	75,1	74,9	74,3	73,7	72,7	no fueling
40	8,6	72,2	74,9	76,0	75,7	75,7	75,5	75,1	74,9	74,3	73,6	72,6	no fueling
35	9,7	71,3	74,2	75,8	75,4	75,5	75,4	74,9	74,7	74,1	73,5	72,6	no fueling
30	12,5	69,1	73,2	75,1	74,6	74,8	74,6	73,9	73,7	72,9	72,2	71,3	no fueling
25	15,7	66,4	71,2	74,5	73,9	74,2	73,8	72,9	72,6	71,6	70,9	no fueling	no fueling
20	19,3	66,2	70,9	74,0	73,1	73,4	73,0	71,8	71,5	70,3	69,5	no fueling	no fueling
10	27,0	66,1	70,9	73,1	71,7	72,0	71,2	70,4	69,2	68,0	66,7	no fueling	no fueling
0	28,5	73,8	73,3	72,3	70,4	70,5	69,4	68,1	66,6	65,3	63,9	no fueling	no fueling
-10	28,5	73,1	72,6	71,6	71,2	69,7	68,2	66,2	63,9	62,5	61,2	no fueling	no fueling
-20	28,5	72,4	71,9	70,9	70,6	69,1	67,6	65,4	62,2	59,7	no fueling	no fueling	no fueling
-30	28,5	71,4	71,0	70,1	69,8	68,4	67,0	64,8	61,7	58,6	no fueling	no fueling	no fueling
-40	28,5	70,9	70,5	69,6	69,4	68,0	66,5	64,4	61,4	58,4	no fueling	no fueling	no fueling
< -40	no fueling	no fueling	no fueling	no fueling	no fueling	no fueling	no fueling	no fueling	no fueling	no fueling	no fueling	no fueling	no fueling

Figure 4: Fuelling table from SAE J2601 (2016).

Based on these considerations, one can estimate average mass flow rates under different ambient temperatures and tank sizes of the standards. The idea in this comparison is to obtain a list of measurement conditions that would yield similar average mass flow rates in the TS during the comparison measurements using the different standards from the participants. **Table 1** presents estimates of average mass flow rates for different ambient conditions and tank volumes. The initial tank pressure is taken as 40 bar, the final tank temperature is 70 °C. Final pressure conditions are taken from SAE J2601 fuelling tables. Field measurements with a 72 L gravimetric standard yielded results that were within 10 % of the present estimates for the average mass flow rates.

One notices that ambient temperature affects the average mass flow rate by around 25 % per step of 10 °C and that similar mass flow rates can be attained at different ambient conditions for different tank volumes.

Table 1: Mass flow rate estimates for various refuelling scenarios.

T_{amb} (°C)	V_{tank} (L)	APRR (MPa/min)	Mass H_2 (kg)	Time (min)	q_m (kg/min)
10	72	27.0	2.578	2.55	0.963
20	72	19.3	2.578	3.57	0.689
30	72	12.5	2.637	5.70	0.443
10	104	28.0	3.732	2.47	1.441
20	104	21.8	3.732	3.22	1.107
30	104	15.3	3.834	4.70	0.781
10	148	28.0	5.310	2.47	2.050
20	148	21.8	5.310	3.22	1.576
30	148	15.3	5.457	4.70	1.111

4.2 Dead volume and vented quantity

An important consideration for the comparison is that there are factors others than the flow meter performances that can affect the accuracy of the dispenser mass of the HRS. For instance, the dispenser hose is vented after a refuelling for safety reasons. This vented quantity has been measured by the TS but not delivered to the primary standard. Moreover, as the TS is located in the region upstream of the pre-cooler, there can be a significant length of piping between the outlet of the TS and the primary standard. The density of the hydrogen contained in this interconnecting piping or 'dead volume' can vary depending on the final pressure of the previous refuelling and the amount of hydrogen that has been replaced and is not necessarily the same. This represents another source of error or measurement uncertainty. Depending on the filling sequence followed, this can result in positive or negative errors of several tens of grams.

Therefore, to ensure a successful comparison, the refuelling process will always be a full fill where the HRS stops the refuelling process automatically. This will ensure that the amount of hydrogen replaced in the dead volume between the TS and the delivery point will be very similar and will not have a large contribution as a source of uncertainty. If the comparison measurements are not performed at the Empa HRS, the participants must accurately quantify the amount of hydrogen vented from the dispenser hose and the difference in the amount of hydrogen contained in the dead volume.

5. Measurement procedure

Each participant will follow a calibration procedure that is part of the comparison protocol and which will be shortly described here:

- Initial pressure in the tank shall be 30 bar for the first measurement and (50 ± 5) bar for the following measurements,
- Zero the balance after having disconnected all the cable connected to it and lowered the frame,
- Set the cut off value of the TS to 0.0 g/min and zero the meter twice,
- Set the cut off the value of the TS to 3 g/min,
- Connect the nozzle, zero the totalizer and start logging the data (mass flow rate and tubing temperature) of the TS
- Start the refuelling and wait until the HRS stop automatically, this corresponds to a full fill,
- Record the values of the totalizer and save the logged data,
- Weigh the amount of delivered hydrogen using the participant's procedure,
- Report the calibration results,
- Repeat the process 5 fives.

A cut-off is applied to the meter to ensure that participants can read properly the integrated mass as recorded by the transmitter as it is not electronically synchronized with the end of delivery signal of the dispenser. Before every measurement, a zeroing of the sensor of the TS is performed to limit the effect of zero flow on the result. Indeed, a refuelling lasts about 3 minutes and it is assumed, that the zero flow of the TS is stable during this period. This also aims to guarantee that all participants use the TS in almost identical initial conditions.

The participants report the relative error of the TS and this value will be used to compare the different standards. It is defined as the difference in delivered mass indicated by the TS and the delivered mass according to the reference. The method of determination of the reference value will correspond to procedure A presented by M. G. Cox [4] and will accompanied by a consistency check.

6. Conclusion

A BIPM-registered comparison to compare and validate existing gravimetric standards used for calibrating HRS under real conditions is ongoing. The specificities of the hydrogen delivery process imply that a comparison cannot be performed in a standard way. A particular strategy needed to be devised to make sure that all participants can perform comparable measurements, even if measurements take place at another HRS. In this paper, the participants proposed a measurement procedure that will hopefully allow them to apply for CMCs. This comparison is currently ongoing and should be finished in spring 2023.

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