METROLOGY for HYDROGEN VEHICLES

Comparison report

A1.4.2: Intercomparison of gravimetric standards

EMPIR EURAMET

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Activity A1.4.2 is a comparison between CESAME, JV, METAS and VSL for the gravimetric standards developed in A1.4.1. from the EMPIR Metrology for Hydrogen Vehicles (MetroHyVe) project. The three-year European project commenced on 1st June 2017 and focused on providing solutions to four measurement challenges faced by the hydrogen industry (flow metering, quality assurance, quality control and sampling). For more details about this project please visit www.metrohyve.eu.

This report was written by:

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Introduction

CESAME, JV, METAS and VSL organize this comparison to validate the method and the claimed uncertainties of their respective gravimetric standards developed in A1.4.1. The aim is to compare the standards and validate them for testing Hydrogen Refuelling Stations (HRS). This comparison also serves as preparation for handling the standards and making sure that the correct procedures are put in place. A Rheonik Coriolis mass flow meter and its transmitter, supplied by METAS, is used as transfer standard.

This report discussed the protocol as well as the results following the intercomparison. JV and METAS results are in good agreement and consistent with an En-value of 0.20.

Participants and planning

Table 1 lists the participants. It was scheduled that CESAME and VSL also participate in this comparison. Due to damage to the Air Liquide gravimetric standard, CESAME could not participate and the VSL gravimetric was not available yet for the comparison in Q1 of 2020. The transfer standard was then no more available. In the end, only METAS and JV participated in the comparison. METAS repeated measurements with two different setups to assess temperature effects.

Table 1: List of participants.

Institute	Country	Shipping address	Contact
METAS	Switzerland	Federal Institute of Metrology Laboratory for Flow Lindenweg 50 3084 Wabern Switzerland	Marc de Huu marc.dehuu@metas.ch
JV	Norway	Justervesenet Fetveien 99 N-2007 Kjeller Norway	Matthias Schrade msc@justervesenet.no

Transfer standard

A Coriolis mass flow meter with its transmitter is used for the intercomparison. See below for the specifications of the flow meter. The totalized mass has been read directly from the transmitter using the Rheonik software.

Flow meter type:	RHM 04L GET, SN RHM-22843
Transmitter	RHE 16, SN RHE-22735
Manufacturer:	Rheonik
Flow rate range	(0.2 – 10) kg/h
Pulses/kg	10'000
Connection type:	Autoclave 3/8" MP (9/16-18 UNF female thread)
Maximum pressure rating	1070 bar@50°C

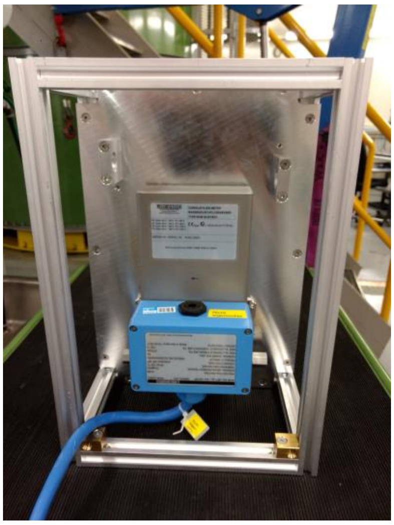

Figure 1: Rheonik Coriolis mass flow meter mounted in its frame.

The Coriolis flow meter was sent around partially equipped. The ball valve and the fitting had to be mounted before starting the measurements. The connections are shown in Figure 2.

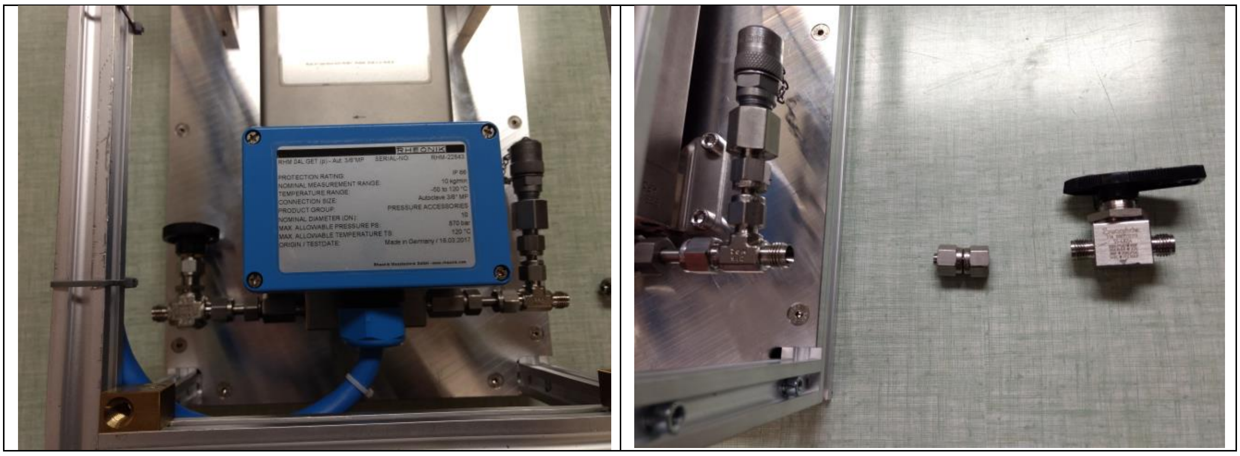

Figure 2: Left) CORIOLIS METER with needle valve and Minimess pressure fitting. Right) Ball valve with fitting. Flow direction is from right to left.

Measurement procedure

The relative error of the transfer standard ε in (%) is the quantity that will be used to compare the results. It is defined as the difference between the delivered mass indicated by the transfer standard and the delivered mass according to the reference:

$$\varepsilon = \frac{m_{CMF} - m_{ref}}{m_{ref}} \qquad (1)$$

where ε is the relative error of the transfer standard,
m_{CMF} is the delivered mass indicated by the transfer standard (kg),
m_{ref} is the delivered mass measured by the reference (kg).

The participants used their own gravimetric standard with some additional equipment (not part of the standard) to perform the measurements. Nitrogen is used as calibration gas and is supplied by a bundle of nitrogen bottles with a pressure reducer to limit the pressure of the incoming gas to a maximum of 50 bar. The transfer standard is then mounted in series between the nitrogen bundle and the gravimetric standard. A ball valve located before the Coriolis meter allows for starting and stopping the nitrogen flow. Mass flow rate was adjusted using the needle valve placed after the Coriolis meter. Once flow rate was adjusted, opening and closing the ball valve started or stopped the measurement. A diagram of a possible setup is shown in Figure 3, which shows also a heat exchanger between the bundle and the gravimetric standard. This heat exchanger has only been added to the METAS setup after noticing that the cooling of the delivered nitrogen led to a transient temperature profile of the Coriolis meter tube and affected the repeatability of the measurements. METAS results with and without the heat exchanger are presented here. JV performed measurements without heat exchanger.

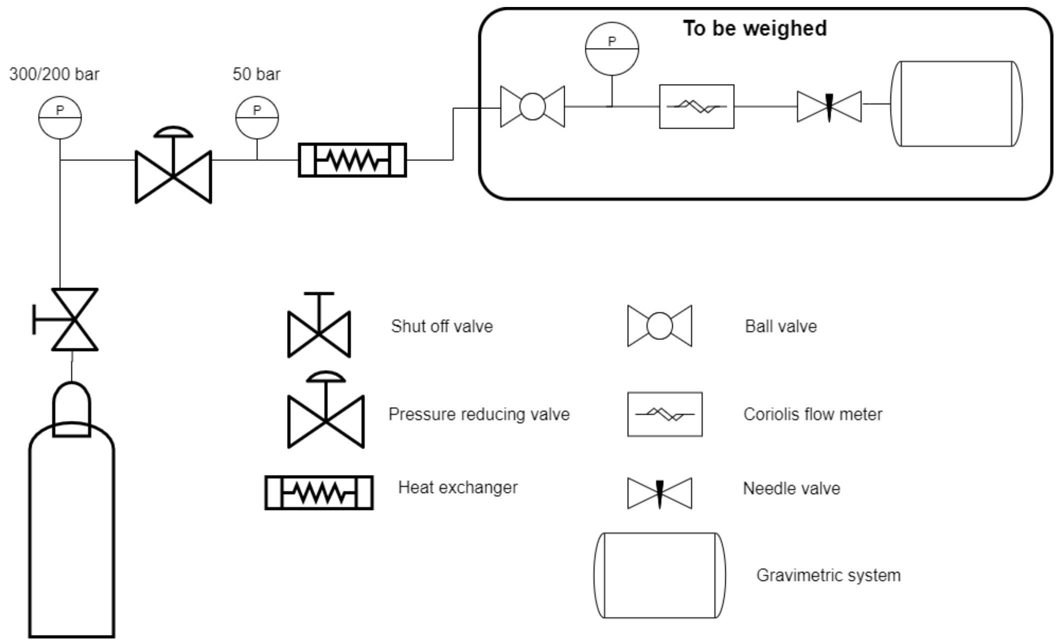

Figure 3: PID of the setup. METAS uses a 300 bar bundle, 200 bar is standard.

Calibration protocol

The participating NMI used the calibration protocol below:

- The initial pressure in the tank shall be 10 bar (+/- 1 bar).
- Zero the balance without any cable or filling line connected to it. The blue Coriolis meter cable and the Coriolis meter shall be part of the weighed system.
- Remove the load from the balance
- Connect the blue cable to the Coriolis meter, the needed cables of your setup and the filling line and switch on the Coriolis meter transmitter
- Fill the pipe from the bundle to the ball valve with N_2 up to 50 (+/- 2) bar.
- Zero the totalizer of the Coriolis meter.
- Open the ball valve to fill the high-pressure tank. The flow rate should be between 0.6 kg/min and 1.2 kg/min.
- Fill the tank up to 40 bar (+/- 1 bar) and close the ball valve located before the Coriolis meter.
- Write down the value of the totalizer (Fwd and Net value should be the same) and the tube temperature of the Coriolis meter. As soon as the filling is stopped, pressure in the tank will decrease due to temperature stabilization.
- Vent the line from the N_2 source to the ball valve.

- Switch off the Coriolis meter transmitter
- Disconnect all the cables (blue Coriolis meter + any others) and filling line from the gravimetric system and weight the amount of N_2 in the tanks. Your setup should be the same as during the initial weighing.
- Apply your calculations/corrections and report the calibration results.
- Wait until the tube temperature as indicated by the Coriolis meter has reached a value of at most 3 °C difference from the tube temperature of the first measurement
- Repeat 5 times

Based on the reported results, the following values are computed: the reference delivered mass (kg), the indicated mass from the Coriolis meter (kg) and the uncertainty of the calibration (k=2) (%).

Measurement results

The measurement results from METAS and JV, without the use of a passive heat exchanger, are presented in Figure 4 and Figure 5. The quoted expanded uncertainty does not contain any contribution from the transfer standard yet. Average mass flow rates for the METAS and JV data are around 0.75 kg/min and 0.65 kg/min, respectively. Average filling times are 180 s for METAS and 320 s for JV, respectively. Filling times for JV are longer because of a larger pressure drop through the piping of their gravimetric standard.

Partner: **METAS**

		Date [yyyy-mm-dd]	Time hh:mm:ss	Coriolis totalised mass [kg]	Coriolis temperature [°C]	Tank pressure [bar]	Tank temperature [°C]	Mass reading [kg]	Mass after corrections [kg]	Meter Error [%]	expanded measurement uncertainty (k=2) [%]
Run1	Initial			0	17.2	10.0		154.6286			
	Final			2.2497	8.5	38.0		156.9024			
	Increase		0	2.2497	-8.7	28.0	0	2.2738	2.2733	-1.04	0.30
Run2	Initial			0	20.4	10.0		154.5932			
	Final			2.2581	9.5	38.0		156.8754			
	Increase		0	2.2581	-10.9	28.0	0	2.2822	2.2816	-1.03	0.30
Run3	Initial			0	19	10.6		154.5948			
	Final			2.2658	9.7	38.0		156.889			
	Increase		0	2.2658	-9.3	27.4	0	2.2942	2.2858	-0.87	0.30
Run4	Initial			0	18.3	10.7		154.6037			
	Final			2.2872	9.7	39.0		156.9068			
	Increase		0	2.2872	-8.6	28.3	0	2.3031	2.3033	-0.70	0.30
Run5	Initial			0	18.7	10.6		154.5964			
	Final			2.2845	10.1	38.0		156.8995			
	Increase		0	2.2845	-8.6	27.4	0	2.3031	2.3024	-0.78	0.30

Figure 4: METAS results, without heat exchanger.

Partner: **Justervesenet (JV)**

		Date [yyyy-mm-dd]	Time hh:mm:ss	Coriolis totalised mass [kg]	Coriolis temperature [°C]	Tank pressure [bar]	Tank temperature [°C]	Mass reading [kg]	Mass after corrections [kg]	Meter Error [%]	expanded measurement uncertainty (k=2) [%]
Run1	Initial	07.03.2019	13:50:05	0	18.4	10.1	20.5	287.1333			
	Final		13:55:37	3.505756	7.94	38.2	29.85	290.6528			
	Increase		0.00384259	3.505756	-10.46	28.1	9.35	3.51955	3.51955	-0.39	0.30
Run2	Initial	07.03.2019	14:48:42	0	18.25	9.8	20	287.1068			
	Final		14:54:03	3.629359	2.36	38.4	29.2	290.7433			
	Increase		0.00371528	3.629359	-15.89	28.6	9.2	3.63655	3.63655	-0.20	0.29
Run3	Initial	08.03.2019	10:01:25	0	20.85	9.5	21.2	287.0353			
	Final		10:06:54	3.705044	7.92	38.7	31.05	290.7744			
	Increase		0.00380787	3.705044	-12.93	29.2	9.85	3.7391	3.7391	-0.91	0.29
Run4	Initial	08.03.1019	10:54:02	0	18.24	10	19.9	287.1346			
	Final		10:59:23	3.498008	3.41	38.3	31.1	290.6734			
	Increase		0.00371528	3.498008	-14.83	28.3	11.2	3.5388	3.5388	-1.15	0.30
Run5	Initial	08.03.1019	11:52:18	0	17.73	10.1	20	287.1446			
	Final		11:57:30	3.500098	5.23	38.3	31.8	290.6516			
	Increase		0.00361111	3.500098	-12.5	28.2	11.8	3.507	3.507	-0.20	0.31
Run6	Initial	08.03.1019	13:22:56	0	19.1	9.9	17.4	287.1168			
	Final		13:28:04	3.597664	1.91	38.3	30.8	290.7515			
	Increase		0.00356481	3.597664	-17.19	28.4	13.4	3.6347	3.6347	-1.02	0.30

Figure 5: JV results, without heat exchanger.

The measured error of the transfer standard is shown graphically in Figure 6. One notices a larger spread from the JV data compared to the METAS data. This is probably due to the larger temperature difference of the Coriolis meter tube between beginning and end of the filling, as can be seen by comparing the tubing temperature readings in Figure 4 and Figure 5. Indeed, the JV data show a larger and increasing difference as a function of run number, whereas the METAS temperature difference readings are rather constant.

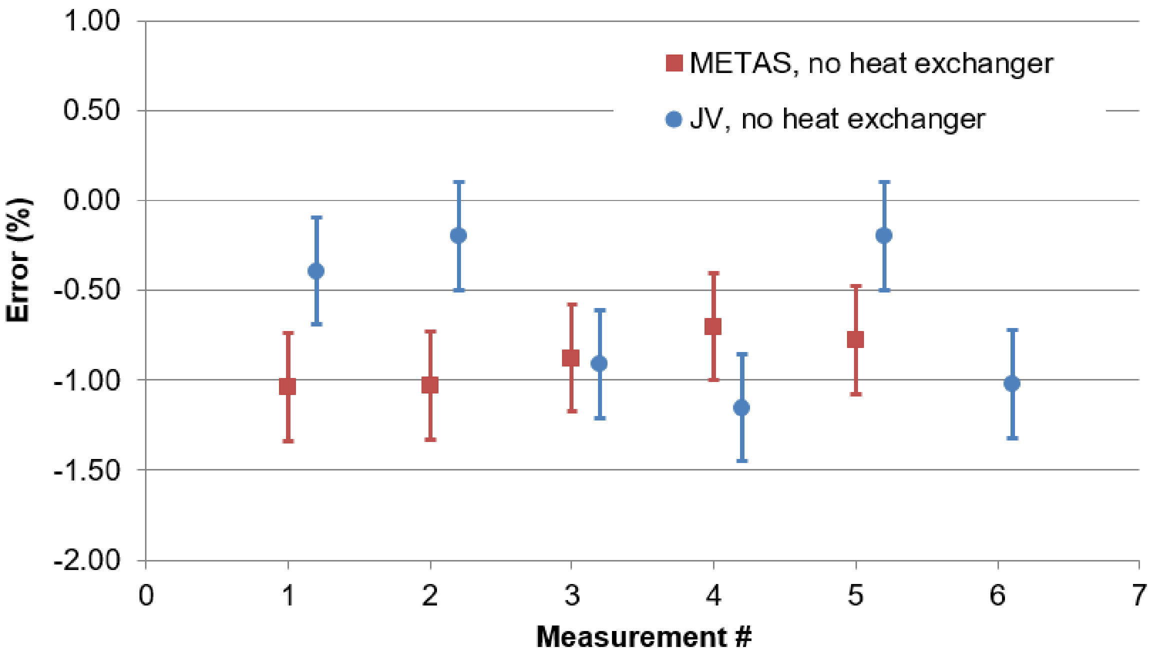

Figure 6: Error of the transfer standard for both laboratories.

Agreement between METAS and JV for the single measurements is reasonable; the measured error is always negative and ranges from -0.20 % to -1.15 %. Building an average from the runs for each laboratory and adding the standard deviation of the average coherently to the quoted expanded uncertainties yields the results presented in Table 2. One notices that the standard deviation from the JV measurements is about three times larger than the one from METAS. This indicates that the temperature variation of the nitrogen as it is flowing through the Coriolis meter definitely affects the repeatability of the instrument. The average errors determined by both laboratories are in excellent agreement, as indicated by the En-value, defined by

$$En = \frac{|\epsilon_{lab1} - \epsilon_{lab2}|}{\sqrt{U_{lab1} + U_{lab2}}}, \quad (2)$$

and which should be lower than 1 to have consistent results.

Table 2: Average error for both laboratories.

	Error (%)	Standard deviation (%)	U (k=2) (%)	En value
METAS	-0.88	0.13	0.40	0.20
JV	-0.70	0.41	0.88	

It should be noted that even if one reduces the expanded uncertainty of JV by a factor of two (by reducing the standard deviation for instance), results are still largely consistent.

METAS performed another round of measurements by placing a passive heat exchanger between the gas bundle and the transfer standard to stabilise the gas temperature. The heat exchanger, shown in Figure 7, consists of a 49 m copper tubing with outer diameter ¼" and inner diameter 4.35 mm, immersed completely in a water tank. The immersed volume is approximatively 0.73 L and the water temperature is at ambient laboratory temperature (22 °C). The gas temperature after the heat exchanger was also at the same ambient temperature.

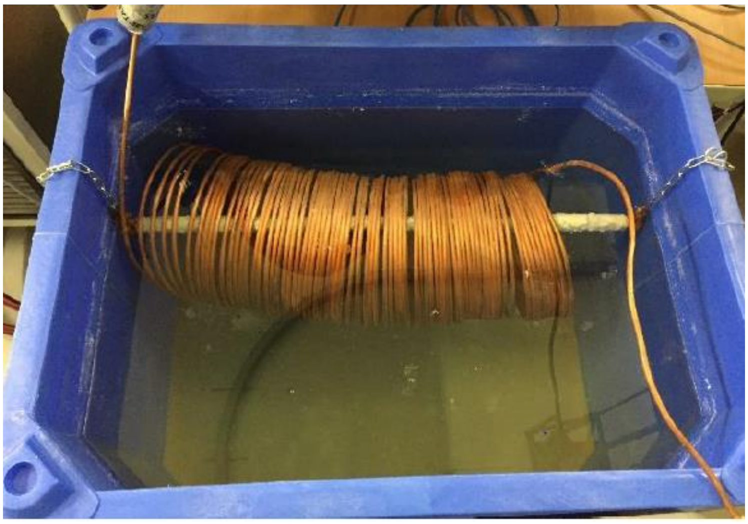

Figure 7: Passive heat exchanger used at METAS.

A comparison between the measured error with and without the heat exchanger with the METAS gravimetric standard is shown graphically in Figure 8 and summarised in Table 3. One notices an excellent agreement between both sets of measurements and a reduced spread (factor of 2) for the data taken with the heat exchanger located between the gas bundle and the transfer standard. Results are largely consistent. This clearly indicates that a heat exchanger must be used for future similar comparison measurements.

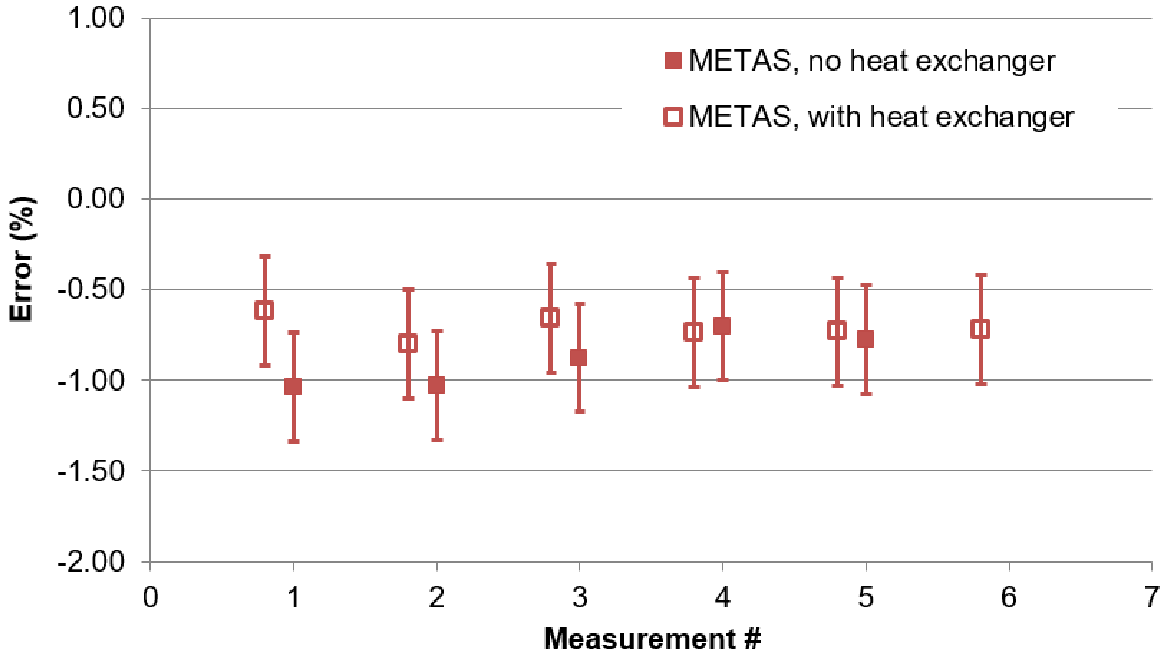

Figure 8: Error of the transfer standard with and without heat exchanger.

Table 3: METAS results with and without heat exchanger.

Run #	Error without heat exchanger (%)	Error with heat exchanger (%)
1	-1.04	-0.62
2	-1.03	-0.80
3	-0.87	-0.66
4	-0.70	-0.73
5	-0.78	-0.73
6	-	-0.72
Average (%)	-0.88	-0.71
Standard deviation (%)	0.13	0.06
Uncertainty (k=2)	0.40	0.32

Conclusion

Two laboratories participated in a comparison using their respective gravimetric standards and a Coriolis flow meter as transfer standard. The measurements results from both laboratories are consistent and show a very good agreement. This indicates that procedures and calculations developped by each laboratory are correct. Expanded uncertainty claims from both laboratories could also be confirmed, as indicated in Table 2.

It could also be shown that placing a heat exchanger between the gas bundle and the transfer standard reduces temperature gradients during measurement and greatly improves the repeatability of the measurements.