

LNG FLOW CALIBRATION FACILITIES

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1. Introduction

Reliable, accurate and commonly agreed measurement methods are a first requirement for the trade of goods. For the large scale LNG distribution chain there is a commonly agreed measurement practice as described in various ISO standards and in the GIIGNL "Custody transfer handbook". In comparison with other commodities like natural gas or gasoline the total uncertainty on measured energy is very high for LNG. The LNG industry is therefore committed to improve their measurement accuracies to reduce their financial risks and to optimize their mass and energy balances.

As an extension of the traditional distribution chain for LNG a new market of professional consumers is developing with LNG as transport fuel. In this case there is no commonly agreed measurement practice and the metrological framework is not yet in place. This will be a showstopper for the roll-out of the LNG as transport fuel because laws will be enforcing protection of (professional) consumers. For small scale LNG the legal metrology framework will lay down requirements on traceability to national standards and will define maximum permissible measurement errors. This is not the case for large scale LNG where the industrial players are assumed to take care of their own business interests.

For this reason the development of an LNG calibration facility is a crucial step and urgently required for the development of the small scale LNG distribution chain. The results obtained with this research facility will however impact the business of the large scale LNG distribution chain as well. The development of a primary calibration standard and a mid-scale calibration facility could be the stepping stones towards the development of larger scale calibration facilities in the more distant future.

2. Objectives

Together with industry and other metrological institutes VSL has been working on the development of new LNG standards and calibration facilities for the past 3-5 years. The end goal is a facility available for R&D, testing and calibration of LNG flow metering and LNG quality measurement systems. This paper will show the roadmap for the development of this facility and the results obtained so far with the development of flow calibration standards.

The first step is the development and building of a primary mass flow standard. In this paper, we present the results of the primary mass flow standard validation with liquid nitrogen.

The second step will be the construction of a mid-scale mass flow standard, traceable to the primary standard. This project has newly started in 2013.

Other elements of the roadmap are a calibration standard for volume flow measurement and an LNG composition standard. Those elements are to be realized in new R&D projects.

Figure 1 is showing the roadmap towards a metrological framework for LNG custody transfer with a prominent role for the use of flow metering. The mass flow metering route is a three step approach consisting of a primary standard, a mid-scale standard traceable to the primary standard and a large-scale standard. The large scale standard may not be needed if the mid-scale standard leads to validated extrapolation schemes from small to higher flows and perhaps even from water to LNG. The volume flow route is needed to accomplish traceability for large scale ultrasonic flow meters. This requires the development and validation of cryogenic piston prover technology or the conversion from mass to volume flow through an accurate density measurement/calculation. Accurate and reliable measurement of LNG composition is needed for quality measurements and density calculations. A test and calibration standard will be of great value in validating various types and brands of measurement systems. These can be either the traditional sampling based systems or the new kind of Raman spectroscopy based systems. A primary density standard is being developed in order to produce reference data with improved accuracy. The new reference data can be used to validate and perhaps to improve the accuracy of the equations of state used to calculate the LNG density based on composition.

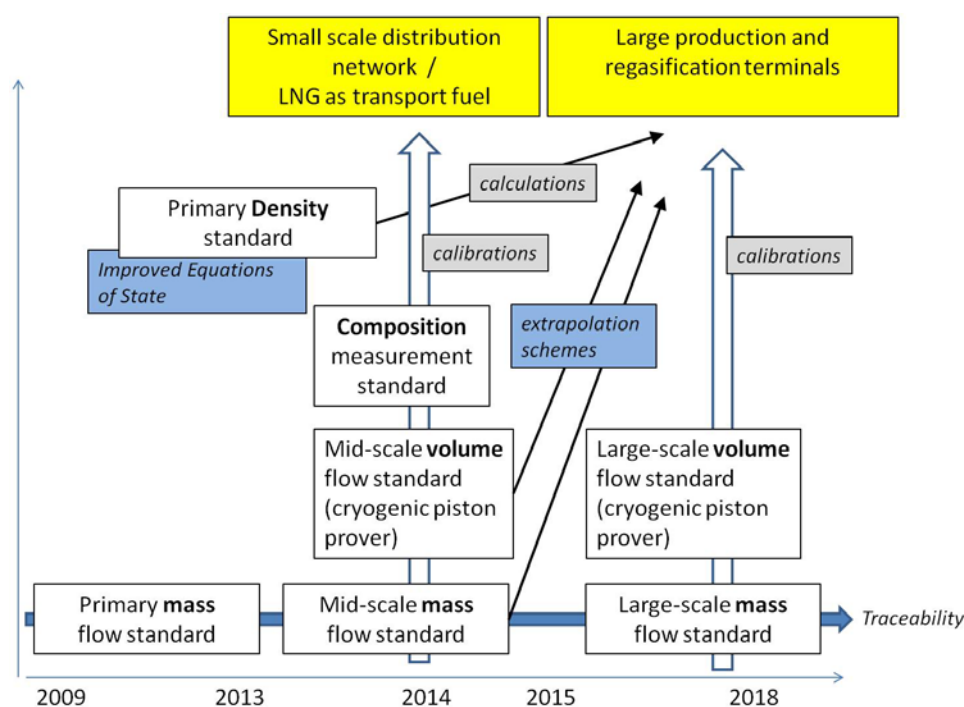


Figure 1 Roadmap towards a sound metrological framework for LNG custody transfer.

3. Development of primary mass flow standard

3.1 Measurement principle

The primary cryogenic mass flow calibration standard, like any primary standard, is designed to provide direct traceability to the SI units. In this case it provides traceability to the SI unit of mass, the kilogram. The primary standard is not to be used for regular calibrations of flow meters but for the periodic calibration of a limited set of flow meters, which are often referred to as master meters. Those meters can then be used to calibrate other flow meters in a specially designed calibration set-up.

A schematic picture of the measurement set-up is displayed in Figure 2. It consists of a 1 m³ storage tank, a pump, the flow meter under test and a 0,5 m³ weight tank placed on a mass balance. The principle of operation is as follows: the pump generates a flow of cryogenic liquid that passes the meter under test (MuT) and is collected in the weighing tank resting on top of the mass balance. In order to obtain stable conditions at the position of the MuT (temperature, pressure) the flow is initially circulated through the MuT while bypassing the weighing tank. When the conditions are stable the flow

is diverted to the weighing tank using a set of fast switches. When the weighing tank is nearly full, the flow is diverted back. The integrated flow over the period between the start and stop time stamps is compared with the increase of weight on the mass balance. A correction is made for the weight of the vapor that is displaced and evaporated from the weighing tank during filling. This amount of vapor is measured using a specially designed gas flow meter in the vapor return line.

The cryogenic liquid is kept under subcooled conditions to prevent two-phase flows. This is accomplished by pressurizing the system up to 3 bar(g) to raise the boiling point. After some time of operation the liquid temperature will have increased and be in equilibrium with the operating pressure. The liquid will be subcooled again by depressurizing the system, waiting until the liquid has cooled down and quickly pressurizing the system.

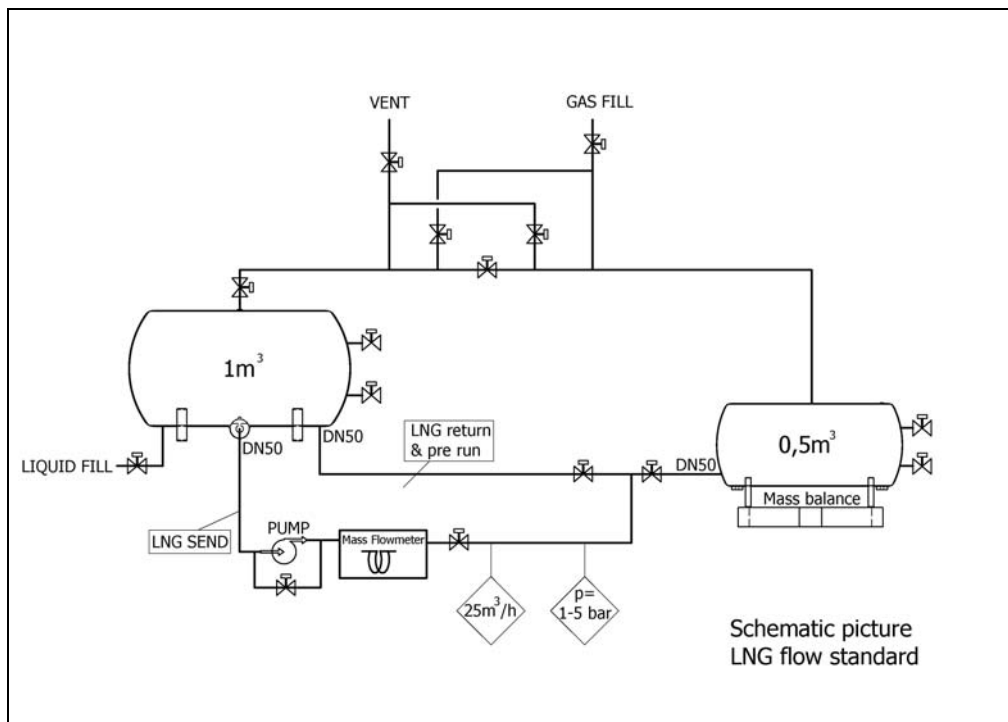


Figure 2 Schematic picture of the primary LNG flow standard

3.2 Critical elements

There are a couple of elements in the system which will influence the measurement result and uncertainty. The most critical ones are *hysteresis in the mass balance system*, *displaced vapor correction*, *linepack volume corrections* and *time stamping*. A qualitative description and some measures to minimize the influence of these effects are discussed below. The experimental results quantifying these effects are described in more detail in the results paragraph

Hysteresis in the mass balance system

As shown in Figure 2, the weighing tank has fixed connections to the outer world through the filling line at the bottom and the vapor return line at the top. This will automatically cause strain on the weighing system potentially leading to unpredictable effects. The variation of strain on the weighing tank is minimized by using flexible tubing and an L-shaped configuration (not shown in the schematic picture). The flexible tube however does expand as a function of pressure. This sets requirements on the stability of the operating pressure throughout the calibration sequence. A simple on/off pressure control system has been implemented to this purpose. These measures cannot completely avoid variations in the residual force applied on the weighing system, leading to hysteresis effects and occasional jumps in the balance response. A semi-automatic recalibration system has been developed to facilitate frequent recalibration of the weighing system. This allows comparing the linearity of the balance before and after the calibration.

Displaced vapor correction

In the case that there is no evaporation and no pressure collapse, the theoretical vapor gas volume flow rate will be equal to the displaced liquid volume entering the weighing tank. Note that there would be no displaced vapor if the weighing tank were to be closed. Experiments with closing the vapor return line however showed that the pressure was not under control. This is due to the fact that the liquid is injected from the bottom of the tank instead of the top. The reason for that is that we need to push the liquid back to the storage tank by applying a pressure difference between the weighing tank and storage tank.

The correction for the displaced vapor proved to be on the order of 1% which is significant compared to the target measurement uncertainty of 0.1% for the liquid flow rate. A vapor flow meter has been designed with a target measurement uncertainty of better than 5%. Details on this flowmeter are provided in the experimental results sections.

Linepack volume corrections

The linepack volume is defined as the liquid volume between the MuT and the entrance of the weighing tank. Any weight change of the linepack volume between the start and end of the calibration must be corrected. For example, if the density has decreased, this means that some extra mass has been moved into the weighing tank that is not being accounted for in the MuT however. In order to monitor the density variation the temperature is being measured both at the position of the filling line and at the MuT.

Time stamping

The calibration result is based on the comparison of the collected weight on the balance and the flow counted by the MuT. In the so-called dynamic or 'flying' start-stop mode there is a constant flow that is passing through the MuT and being measured continuously. The counting must start exactly at the moment that the flow is diverted to the weighing tank and stops when the flow is diverted away from the weighing tank at the end. This requires accurate time stamping. The diverting is accomplished by closing and opening two fast switched valves simultaneously. The complete switching process takes some time (time window less than 0.05 s) during which the flow direction is not defined exactly. The switching valve design is such that they are making a symmetrical movement related to each other. However the crossing point repeatability is a few 0.01 s.

The switching and time stamp challenge is avoided in the so-called static start-stop method. In this case the pump is switched off after stable temperature conditions have been obtained in the pre-run mode. The diverter switches are then positioned in filling mode and the pump is switched on. After the weighing tank is nearly filled the pump is switched off. The calibration interval is simply defined as the period between switching the pump on and off. The main disadvantage of the start-stop method is that the meter output has to cross potentially non linear regions over its range. A comparison between the results of the dynamic and start-stop method is shown in the results paragraph.

4. Results

The calibration results in terms of the deviation of the mass flow meter under test (MuT) is defined as follows:

$$\phi_{MuT} = \frac{(\text{Indicated mass MuT} - \text{Reference Mass})}{\text{Reference Mass}} * 100\%$$

In which the Reference Mass is calculated from:

$$\text{Reference mass} = \text{Mass}_{LN2} + \text{Mass}_{Vapor} + \text{Mass}_{Linepack}$$

In which

Mass_{Vapor} = Vapor mass vented from the weighing tank during calibration time window.

$Mass_{Linepack}$ = The change in trapped liquid mass between MuT and weighing tank.

$Mass_{LNG}$ = Accumulated cryogenic liquid in the weighing tank during the test time window.

It is calculated according to:

$$Mass_{LNG} = AvgReading_{\tau_1} - AvgReading_{\tau_0}$$

In which:

$AvgReading$ = Average of all balance readings just before and after the test time window that fulfill the requirements defined below. Number of readings is mostly between 3 and 20

τ_0, τ_1 = time at start and stop moment respectively.

The system is designed to run in dynamic or 'flying' start-stop mode, although it is also suitable for static start-stop mode. The static mode is not applicable for some flow meter types.

The balance readings just before τ_0 and after τ_1 are valid under the following prerequisites:

1. The difference between subsequent balance readouts (updated once per second) is less than 0.003 kg;
2. The difference between the actual linepressure in the system and the linepressure setpoint is less than 30 mbar. (Linepressure was set at 3 bar gauge and controlled by a on/off control system).

The average is calculated from a minimum of 3 balance readings that fulfilled these criteria.

During the pre-run, a small purge batch is generated to flush out any gas bubbles in the linepack volume. The minimum purge amount is equal to the linepack volume and is approximately 0.03 cubic meter.

The following prerequisites need to be fulfilled as well in order to produce a valid calibration result:

1. The temperature of the cryogenic liquid should be lower than the saturation temperature at the process pressure condition. The 'subcool margin' is calculated from

$$Subcool\ margin = LiquidTemperature - SaturationTemperature_{at\ P_{process}} \text{ } ^\circ\text{C};$$

2. The process temperature variations (expressed as standard deviation) should be smaller than 1°C;
3. The reading of the ultrasonic meter (installed upstream of the MuT) should be smooth without spikes. Spikes are indications of vapor bubbles in the process.

4.1 Physical characteristics of a calibration run

In Figure 3, a number of measurands are plotted against time. From the figure we can see what is happening:

- A small pre-run test shows that there were still some bubbles in the lines. This can be observed from the spiky output of the USM (ultrasonic meter);
- Bubble formation seems logic at the start: the liquid temperature just crosses the saturation temperature. After a few moments the Sub cool margin reaches a safe level up to -4°C during the calibration run (dashed line). After an initial filling line purge of approximately 20 kg, the balance was loaded with 140 kg LIN during the actual calibration run;
- Apparently the flow rate in the pre-run loop is 0.5 kg/s higher than during the calibration run. This is caused by the fact that the pre-run piping configuration has a different pipeline resistance

compared to the calibration loop. Keep in mind that the calibration loop also consists of the vapor return line (including a gas flow meter and several valves) contributing to the total calibration flow resistance. The control valve at the weighing tank entrance can be used for tuning the line resistance to get a smooth transition from pre-run flow to calibration flow. Measurements however show that a smooth transition is not essential for this type of mass flow meter under test given the fast response of the meter, the linearity and the long test time;

- During the calibration run, the flow rate keeps varying a little. The USM and CFM (coriolis flow meter) are trending in the same way. The variation is caused by the pressure variations in the system. The pipeline resistance in the vapor return line is dependent on the gas pressure and thus affects the working point of the LNG pump. The unstable equilibrium between evaporation, saturation and condensation in the weighing tank causes the smallest movements of the liquid to disturb temperature stratifications leading to pressure collapse. The pressure control system will immediately respond by adding Nitrogen gas into the system.
- The balance reading is carried out at the points where all criteria earlier mentioned are fulfilled. These reading moments are indicated with red crosses in the graph;
- In the case that there is no evaporation and no pressure collapse, the theoretical vapor gas volume flow rate will be equal to the displaced liquid volume entering the weighing tank. The assumption that the vapor return gas temperature is equal to the saturation temperature in the tank at 3 bar(g) leads to a density of the nitrogen vapor of 16.7 kg/m^3 and a liquid density of 738.6 kg/m^3 at $T = -181.8 \text{ }^\circ\text{C}$. So the relative mass loss from the weighing tank due to vapor would be 2.3%. Actual measurements indicate that the vapor temperature is at minimum $-80 \text{ }^\circ\text{C}$ which leads to a relative mass loss of 1%. This quantification sets the requirement of vapor gas meter accuracy to be better than 5% in order to contribute 0.05% on the accuracy of the calibration result.

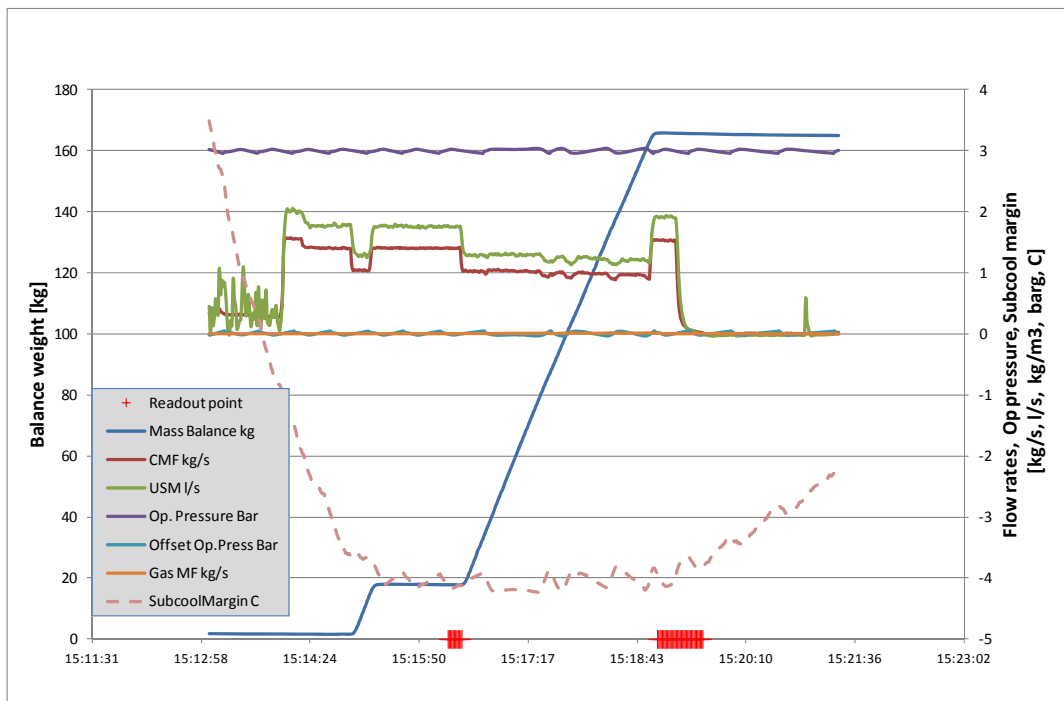


Figure 3 Graph showing a number of relevant parameters before and during a calibration run

Error! Reference source not found. Figure 4 gives more details about the system behavior during a calibration run:

- Return vapor gas: The oscillating behavior of the return vapor flow rate (light blue line) is caused by the on/off pattern of the pressure control.(light green line). During active gas venting and releasing, the vapor generation inside the weighing tank increases and vice versa;

- The surface beneath the vapor mass flow line is representative for the total vented vapor released from the weighing tank ($\int Q_{vapor(t)} * dt$). The calculations support the assumption of a relative mass loss of 1% due to vapor displacement and venting;
- Stability of density: The (fast response) density output of the CFM is a measure for the stability of the liquid condition. The dark blue line shows the delta between $\rho_{average}$ and ρ_{actual} within the time window of the calibration;
- Stability of process pressure: The light green line depicts a control band within 30 mbar
- The derivative of the balance mass output cannot be used directly as reference in the mass flow rate domain. The balance output electronics cause a delay together with a non uniform readout. However for calibration purpose of the gas meter it can be used prevailing some statistic processing is used (See next sections).

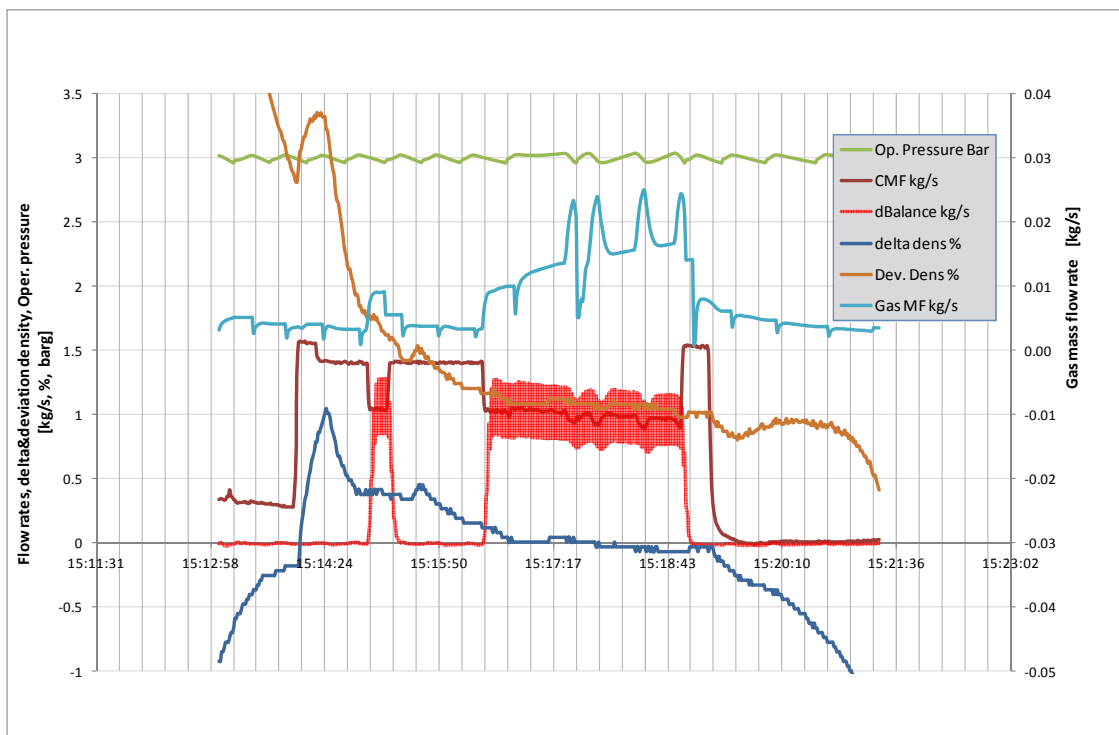


Figure 4 Graph showing dynamics of some essential parameters.

4.2 Results of a mass flow meter calibration

Two different types of 2" coriolis mass flow meters were calibrated with LIN (Liquid Nitrogen). The CMF was adjusted to read 'zero deviation' when calibrated with ambient water.

The results of the extensive calibration of a CMF are depicted as a function of mass flow rate in the following graph. Keep in mind that the indicated mass readout from the MuT is based upon a fixed pulse factor which was based upon the 'zero deviation water calibration' for comparison.

In 2009 this meter was also tested at NIST, Boulder Colorado. At NIST, the meter was tested in open air (no extra thermal insulation) whereas at VSL, the meter was mounted inside of a cold box filled with insulating perlite powder. According to the meter manufacturer these differences in set-up should not generate any difference. The VSL standard is not provided with an extra active cool-unit to maintain the liquid subcooled like in Boulder. Subcooling at VSL is achieved by increasing process pressure prior to calibration run.

Most of the tests are carried out in 'flying start and stop' mode (purple diamonds), where the MuT was stabilized in a pre-run at approximately the calibration flow rate setpoint. Data output collection is achieved with a fast counter and accumulated mass is related to the time window between the diverter switch trigger moments.

Some tests are done in 'Static start and stop mode' (blue squares.) In this case, the MuT is ramped up from zero flow, kept a while at the setpoint flow and then ramped down to zero again. The timing issue does not play a role of importance, it is only the accumulated mass that counts and that will be directly compared to the balance mass increase. The main disadvantage is that the meter output has to cross potentially non linear regions over its range.

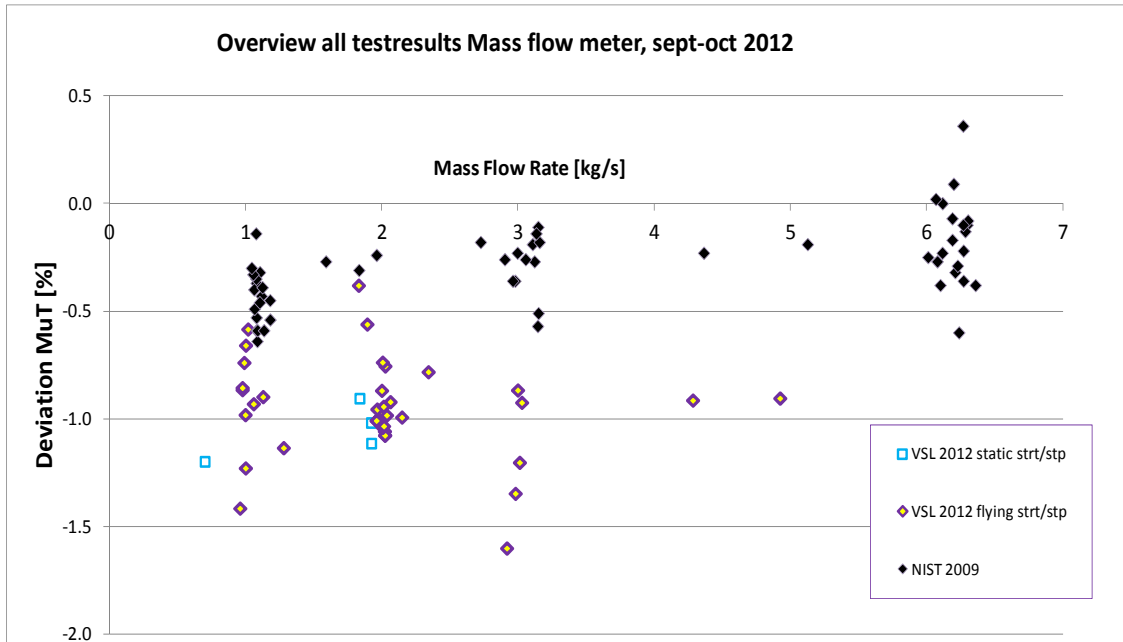


Figure 5 Calibration results using the new VSL primary LNG flow standard and the NIST liquid nitrogen flow standard.

The linepack volume correction can have a significant impact on the calculated deviation and it compensates for the instabilities caused by the (small) changes in density between the connections of MuT and weighing tank during the calibration time window.

Linepack correction (C_{Linepack}) is calculated as follows:

$$C_{\text{Linepack}} = - \frac{(\rho_{\tau 1} - \rho_{\tau 0}) * V_{\text{linepack}}}{\text{Mass}_{\text{Liquid}}} * 100\%$$

In which:

- $\rho_{\tau 1}$ = density of the liquid in the linepack volume at time $\tau 1$ (end of calibration)
- $\rho_{\tau 0}$ = density of the liquid in the linepack volume at time $\tau 0$ (start of calibration)
- V_{linepack} =connected volume between flanges of CFM and weighing tank
- $\text{Mass}_{\text{Liquid}}$ =accumulated amount of liquid during the calibration.

The sensitivity of density change due to variations in temperature is very high close to the saturation temperature: approximately 0.5%/°C. While the liquid density is slowly decreasing over time due to temperature rise, the corrections are in the order of 0 to +0.2%. It is however crucial to determine the representative temperature change in the connected volume. If linepack effects would not be considered, the results end up as depicted in Figure 6:

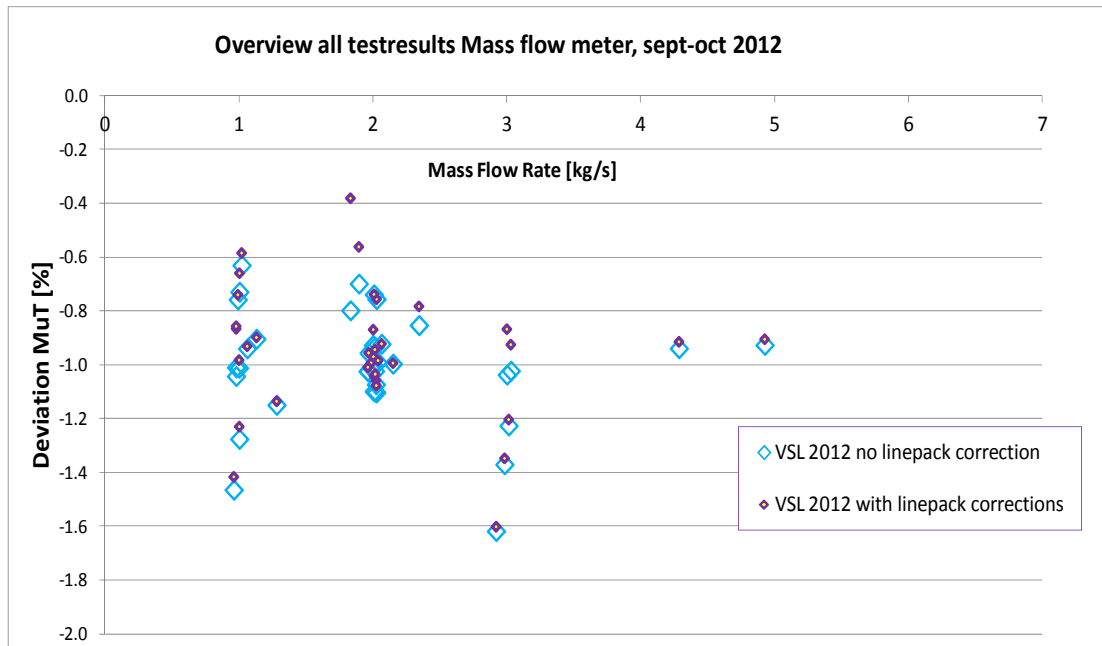


Figure 6 Calibration results using the new VSL primary LNG flow standard with and without linepack corrections applied.

In this case, the standard deviations appears to increase slightly if the corrections are applied. In other words, the results are not correlated with the linepack correction. The conclusion may be drawn that either:

1. The model for linepack correction is incorrect or not applicable or,
2. The measured density change at the position of the CMF is not representative for the actual density change in the linepack volume.

From VSL experience scenario 2 is most likely and more efforts should be taken to determine the mixed cup temperature drift of the linepack volume.

In any case, on average the density during the test run is slightly decreasing as expected.

4.3 Results of balance calibrations, effects of flexible hose connection

The balance is calibrated numerous times with the deadweight disk semi automatic manifold prior and after calibration series.

The flexible filling hose is constructed from two bellow-shaped tubes with vacuum insulation in between. The exterior of the hose is protected with steel wire braiding. The braiding is essential to prevent the hose from stretching during pressurization. This is however disadvantageous for the parasitic forces onto the weighing tank. Stable force 'offsets' to the weighing tank are not of any concern because we are interested in the weight change after filling. The liquid flowing through the filling hose may however cause shocks, vibrations and mechanical hysteresis. The parasitic force changes caused by this are partly non-reversible and will attribute significantly to the normal hysteresis loop of the balance. The typical loop amplitude of a 'normally used' weighing device of this quality level amounts to a few gram. In our configuration the maximum observed hysteresis was 280 grams.

Figure 7 shows the balance deviation as a function of loading and for different process pressure.

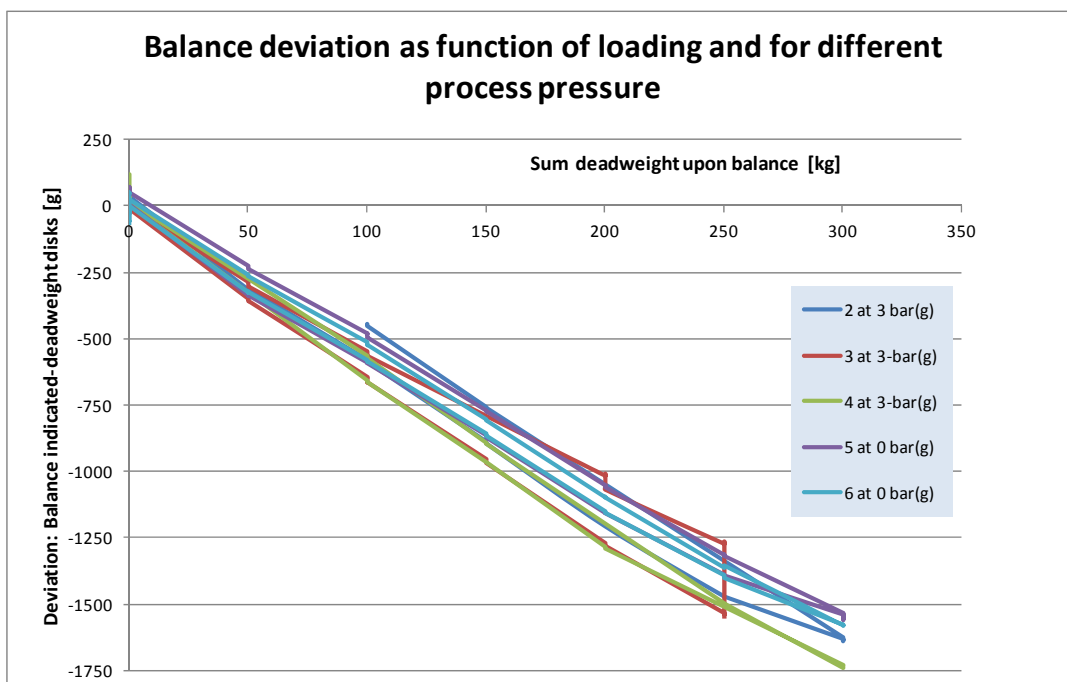


Figure 7 Balance deviation as function of loading showing hysteresis

In Table 1 the influence for each separate calibration hysteresis loop on the measurement uncertainty has been estimated. For the uncertainty model the hysteresis is conservatively described with a rectangular distributed probability function.

batch [kg]	U balance due to hysteresis in % (batch measurement) [%] 2s equivalent				
	series 2	series 3	series 4	series 5	series 6
50	0.03	0.23	0.26	0.31	0.30
100	0.16	0.16	0.18	0.15	0.15
150	0.11	0.05	0.10	0.09	0.10
200	0.01	0.16	0.07	0.07	0.07
250	0.00	0.13	0.05	0.05	0.06
300	0.01	0.03	0.04	0.03	0.04

Table 1 Uncertainty of balance reading due to hysteresis

Series 2, 3 and 4 were subject to 3 bar(g), series 5 and 6 to atmospheric pressure. The preliminary conclusion could be drawn that different operating pressures do not affect hysteresis amplitude and/or deviation curve significantly. Furthermore the uncertainty evaluation of batch mass determination shows that batches less than 150 kg should be avoided.

4.4 Displaced vapor correction

The displaced vapour is measured using a specially designed vapor return gas flow meter. The uncertainty requirement was set at 5% of reading under normal test conditions. The design is partly based on the ISO 5167 standard for orifice plates. Several designs were considered to find the best possible solution for gas metering under these demanding conditions:

- Temperature variations of more than 40 °C during the test run;

- Non stationary flow conditions;
- Fast changes of gas flow rate;
- Possible damage due to liquid overflow of the weighing tank;
- Suitable for cold Nitrogen as well as Methane gas;

The gas temperature sensor is placed in axial direction in the centreline of the orifice throat and outlet piping. Since this is not in accordance to the ISO 5167 a laboratory or field calibration should be carried out.

In the next figure, the configuration of this vapour gas meter is explained.

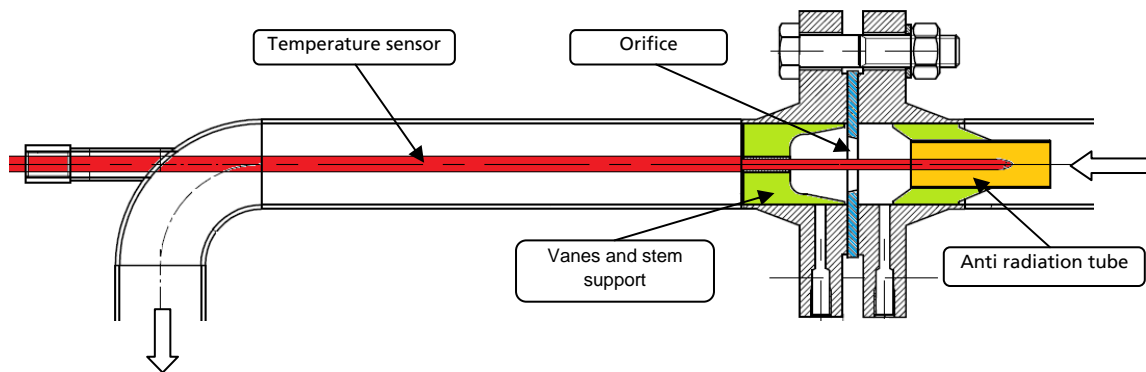


Figure 8 Schematic drawing of the return vapour flow meter

To improve the representativeness of the measured gas temperature, the sensor is provided with an anti-radiation baffle and is in contact with the gas over 50 times stem diameter-length to prevent heat in-leak from the pipe wall to the sensor tip.

The orifice plate armature is calibrated with air under atmospheric conditions and the correction factors are being fine tuned to match with the Reynolds number of the expected cold gas conditions for N_2 and CH_4 .

The model for the gas meter is derived from ISO 5167 and simplified to:

$$Q_{gas\ mass\ flow} = (C_0 - C_1 * Q_{gas\ mass\ flow}) * \sqrt{\Delta P * P_{upstream} / T_{throat}} \quad [gs^{-1}]$$

In which

- ΔP = differential pressure over the orifice [bar]
- $P_{upstream}$ = Pressure just upstream of the orifice [bar]
- T_{throat} = Gas temperature in the orifice throat [K]
- $C_{0,1}$ = constants depending on gas type N_2 or CH_4

The solution is solved by iteration after which the returned vapour mass is totalized according to:

$$Total\ gas\ mass = \sum_{i=1}^{i=run\ duration} Q(i) dt$$

Where dt = reading interval (1 second)

With a balance resolution of 1 g the vapour gas flow meter could be calibrated 'in the field' by comparison with the derivate of the balance weight. Figure 9 shows the results when cold gas is vented from the weighing tank through the vapor gas meter:

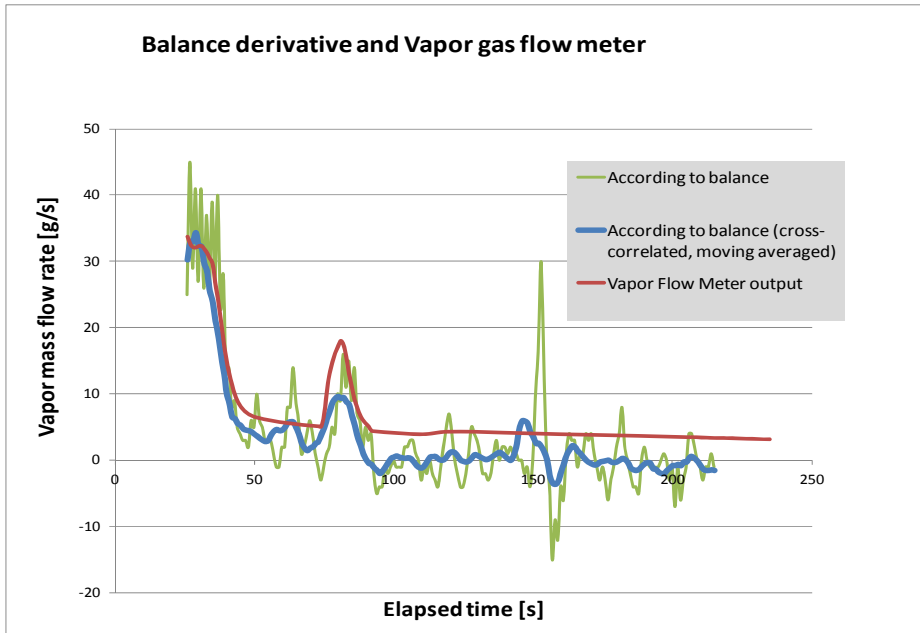


Figure 9 Comparison of the balance weight derivative and the vapor gas flow meter readings

The mass loss in the weighing tank is determined based on the balance readings at 1 second interval. Due to mechanical vibrations and time delay in the balance electronics, the calculated instantaneous mass loss cannot be directly used as a reference value for calibration purpose. Therefore, the data are processed by moving average filtering of the data and time lag compensation. The time lag is determined by a cross correlation technique. The processed data are used for the calibration of the vapor gas flow meter.

Figure 10 shows that the vapor gas flow meter is over reading below 30 g/s. The origin of the offset of the gas meter at zero gas flow is probably caused by a ΔP transmitter zero shift.

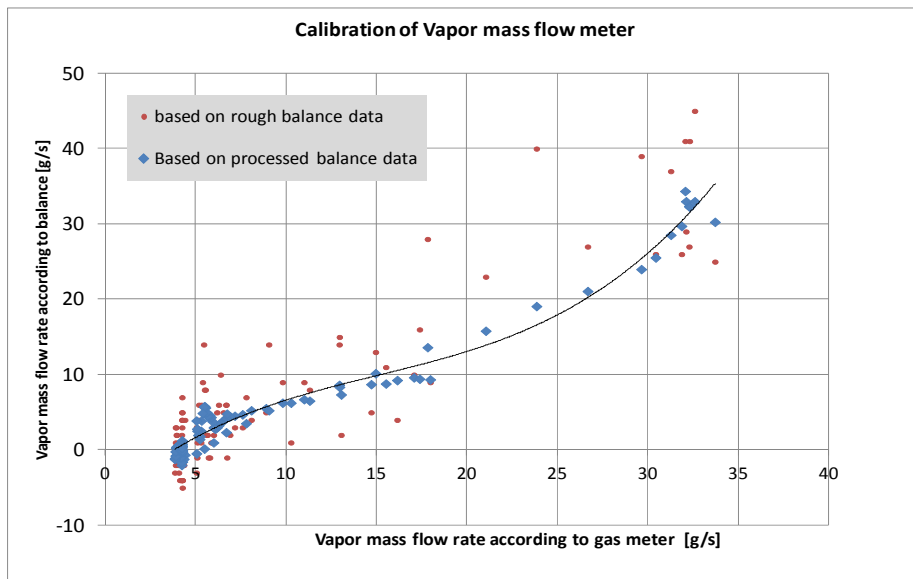


Figure 10 Correlation Vapor gas flow meter related to Balance reading derivatives

The next step is to operate the primary standard with LNG. Nitrogen gas will still be used to pressurize the system. Although this is expected to have a stabilizing effect on the operating pressure it will also cause additional uncertainty on the density of the displaced vapor being a mixture of nitrogen and natural gas. The vapor in the return line will need to be sampled to study the variations in composition.

5. Conclusions

This paper reports on the liquid nitrogen validation of the primary LNG mass flow standard. This is a fundamental and first step on the roadmap towards sound metrological support for the new as well as the traditional LNG markets.

The validation results show reasonable reproducibility figures. At flow rates of 1, 2 and 3 kg/s the reproducibility is respectively: 0.14%, 0.07% and 0.28% (2 times standard deviation of average). The main contributions to the irreproducibility come from *balance hysteresis* (nonreversible parasitic forces from the flex-tubing connection), *vapor gas flow measurement* and *timing*. It is believed that the coriolis meter under test was not significantly contributing; however this has to be confirmed with e.g. Youden analyses. The potential for further improvements of the reproducibility is realistic.

The deviations of the VSL results compared to the results at NIST Cryogenic Flow measurement Facility (CFMF) are subject to further investigations in the near future.

Further research / LNG validation

Further work is required to finalize the uncertainty analyses for liquid Nitrogen.

In the first half of 2013 the calibration standard will be validated with LNG with a focus on:

- Displacement measurements of flex-tubing to gain better understanding of possible parasitic forces and torques behavior to balance;
- Optimization of vapor gas flow measurements;
- Sampling of return vapor to determine the maximum variation in composition (natural gas + nitrogen gas) and density.
- Feed-forward control of pump speed for flow stability. Modeling of characteristic behavior of the system on basis of experience gained from previous Nitrogen tests;
- Youden analyses based on two meters under test to find out attributors;
- Calibrating different brands flow meters for LNG (Coriolis- and Ultrasonic meters);
- Improving response time and representativeness of temperature metering especially for better i.e. more accurate linepack determination, check for correlation improvement;

VSL has, as a national standards institute, not only a responsibility to metrology as a science but also to the origin of metrology, viz. the need of society for reliable and traceable measurements as a base for fair trade, quality control and process metering. Development, continuity and stability of Reference Values is an important element in the function and service of a National Metrology Institute to society. Realization of reference values for LNG flow metering is one of VSL's focal points for the past and coming years.

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