EXTENDING THE ACCREDITED LOW FLOW LIQUID CALIBRATION RANGE

T.H. Platenkamp[1], *J.C. Lötters*[1,2]
[1] Bronkhorst®, Ruurlo, The Netherlands
[2] University of Twente, Transducers Science and Technology, Enschede, The Netherlands

ABSTRACT

There is an increasing demand for ISO/IEC 17025:2005 accredited liquid flow calibrations in the range of 1 g/h to 30 kg/h. The accredited Low Flow liquid Calibration Setup [1] (LFCS) at Bronkhorst® covers a flow range of 1 to 200 g/h, leaving a traceability gap in the flow range of 0.2 to 30 kg/h. By extending the calibration setup with two new balances it can perform calibrations from 1 g/h to 30 kg/h, thus covering the traceability gap. Crosschecks between references show good consistency and the setup extension was successfully accredited for ISO/IEC 17025:2005 calibrations. By participating in an intercomparison with National Metrology Institutes (NMI's) the uncertainty of the LFCS extension is traceable to European NMI's.

KEYWORDS

Calibration, gravimetric, micro flow, uncertainty

INTRODUCTION

The importance of ISO/IEC 17025:2005 accredited calibrations cannot be overstated. The ISO/IEC 17025:2005 standard ensures the calibrations and reference equipment is traceable to its primary standard, which represents the fundamental unit. Low flow instruments depend on a traceable uncertainty to perform well in critical applications were knowledge of the exact flow rate is crucial, e.g. critical applications like drug delivery systems and pharmaceutical processes in which the stable delivery and exact amount of drugs plays a key role in successful treatment.

By extending our LFCS to flow rates up to 30 kg/h, we can calibrate flowmeters in a wider flow range that are used in applications were accredited traceability of the flow rate is required. For flow rates above 200 g/h the LFCS is expanded with two balances. Some critical parts needed to be added to the setup in terms of flow generation and evaporation prevention. An uncertainty budget is made to provide the extension with a traceable uncertainty on mass flow.

PRINCIPLE

Both the LFCS for flow rates below 200 g/h and the extension of the setup for flow rates above 200 g/h are based on the gravimetric principle of mass flow measurement, where a balance is used as reference.

By differentiating the measured mass (Δm) to measured time (Δt) the resulting output is mass flow ($\dot{m}$), as shown in equation 1 below:

$$\dot{m} = \lim_{\Delta t \to 0} \frac{\Delta m}{\Delta t} = \frac{m_r}{t_r} \qquad (1)$$

where m_r and t_r are respectively the reference mass and reference time. With a RS232 balance interface between balance and data acquisition each collected mass sample on the balance is continuously measured and combined with the correct time sample, resulting in a reference mass flow. The continuous collection of mass samples during a flow measurement is called "dynamic weighing" method [2] and enables the direct comparison between reference- and DUT (Device Under Test) mass flow.

SETUP

The extension of the LFCS consists of a pump, filter, degasser and pressure controller for generation of a pure liquid flow (Figure 2). Upstream from the pure liquid flow generation, the DUT controls a stable flow. Downstream from the DUT the flow rate is compared with one of two reference balances, depending on the flow rate (see Figure 1). Reference 1 is a balance used to measure flow rates between 0.1 kg/h and 2 kg/h. Reference 2 is used to measure flow rates between 1 kg/h and 30 kg/h. The flow range is divided over two balances because of the limitations on readability versus weight capacity of the balance.

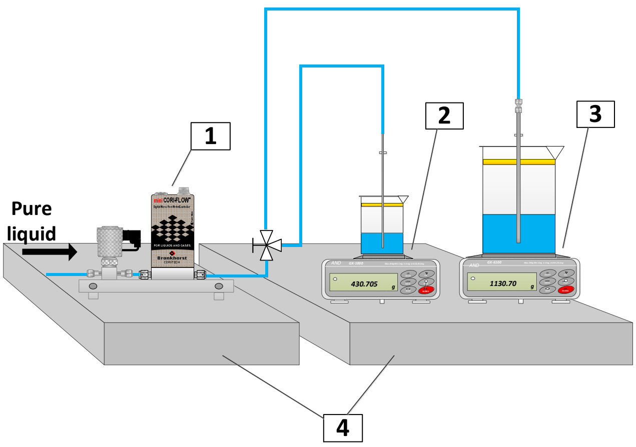

Figure 1. Schematic illustration of the extension setup for comparison between the DUT with control valve(1), balance reference 1(2) and -2(3). Balance and DUT are placed on a granite table(4).

Both the balances and DUT are placed on a granite table with shock absorbing blocks to reduce vibration interference from the environment. The balances are placed in a box to prevent influences of draft and fast temperature changes on the mass flow measurement.

Although the LFCS and its extension are similar, in terms of mass collection and data processing, there is a difference in stable flow generation and evaporation prevention.

Micro pump flow generation

First, in the LFCS (for flow rates <200 g/h) flow is generated by a pressurized liquid tank in an open loop system. In the extension of the setup (for flow rates >200 g/h) pumps are used to generate flow in a closed loop system, making it easier- and faster to handle the amount of water displaced. Figure 2 shows a schematic of the pump driven flow generation. The pump builds up pressure at a constant pump frequency. Downstream from the pump the liquid is filtered and the flow path splits in a flow path for flow measurement and a bypass. The bypass leads back to the water storage tank and acts as a flow stabilizer, by actively controlling the bypass valve to the demanded pressure. Downstream from the pressure sensor the water is pushed through a degasser for flow rates up to 2 kg/h, removing dissolved gas from the liquid.

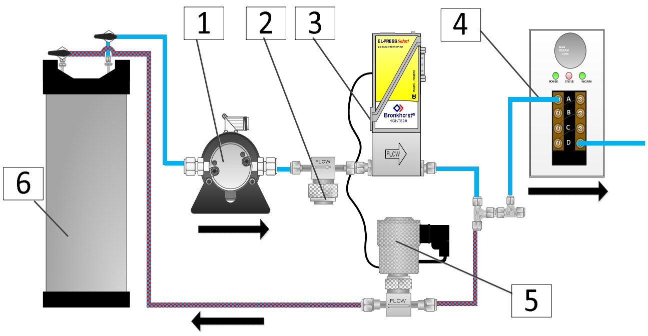

Figure 2. Schematic of pump driven flow generation with pump(1), filter(2), pressure sensor(3), degasser(4), bypass control valve(5) and liquid storage tank(6).

Evaporation prevention

Second, in the LFCS a layer of oil is used on top of the water surface to prevent evaporation of the collected water in the measurement beaker on the balance. Though prevention by a layer of oil on the water surface is suitable for flow rates below 200 g/h, it causes irregularities in the reference mass flow at flow rates above 200 g/h. By using a sealed cover on the measurement beaker (Figure 3) in the setup extension, instead of an oil layer on the water surface, these irregularities are not present. The tube that dispenses the liquid in the measurement beaker runs freely through a small hole in the cover. The use of a sealed cover results in a nearly saturated air between cover and water surface in the measurement beaker, thus reducing and stabilizing the evaporation rate.

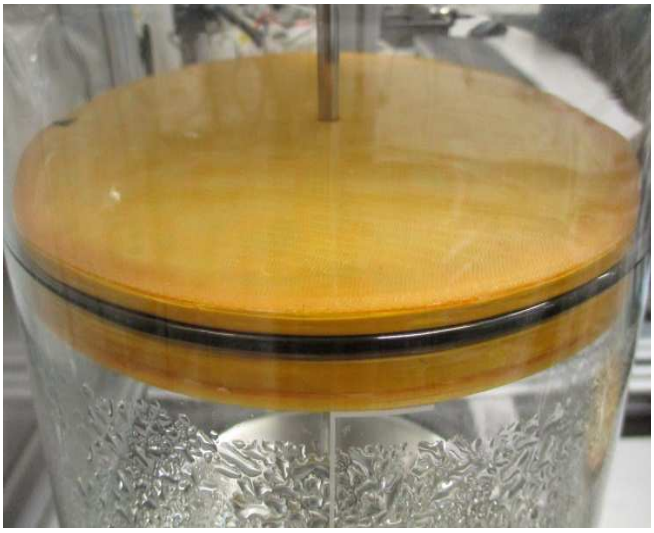

Figure 3. Sealed cover on the measurement beaker to prevent evaporation of the collected water on the balance.

Because the evaporation rate is reduced to a minimum it is considered an uncertainty on the reference flow rate and needs no correction.

UNCERTAINTIES

By extending the LFCS to higher flow rates new parts are added. Some of these new parts add new uncertainties to the uncertainty budget. Components like contact angle force fluctuations and standard Buoyancy corrections change, becoming more dominant in the calculation of the total uncertainty of the extension setup.

Contact angle forces

When water is delivered through the submerged tube in the beaker, the balance measures the mass increase. While the water level in the beaker rises the the water front moves along the tube. While moving along the tube, the contact angle between water and tube depends on the tube properties and other possible environmental effects. Irregularity in the contact angle results in irregular vertical forces [3], thus influencing the measured mass flow on the balance.

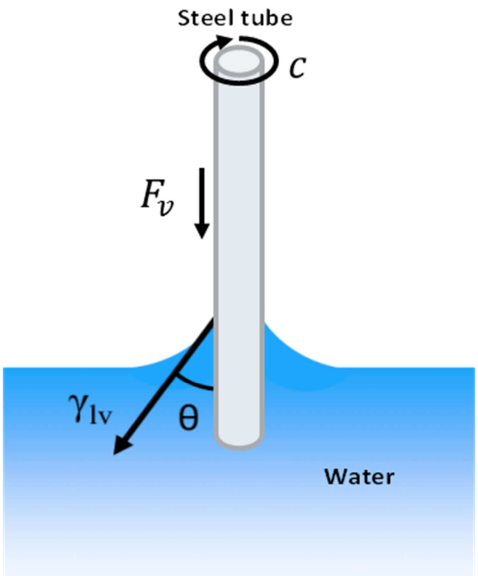

Figure 4. This schematic shows the meniscus between a steel tube and waterinterface. The contact angle (θ) results in a vertical force (F_v).

Figure 4 with equation 2 show that the vertical force (F_v), between water and tube, is dependent on the contact angle (θ), surface tension (γ_{lv}) and circumference (c) of the tube.

$$F_v = \gamma_{lv} c \cos\theta \qquad (2)$$

Nonlinear Buoyancy mass flow correction

The standard Buoyancy correction due to density differences between calibration and measurement [4] on the balance (m_{cor1}) is shown in equation 3 below. Besides the initial mass (m_r), The correction depends on the difference in weighted mass- and air density during calibration (ρ_{mass_cal} and ρ_{air_cal}) and measurement (ρ_{object} and ρ_{air}). During calibration the weighted mass is a calibrated weight. During flow measurement the weighted mass is a beaker with liquid; the object.

$$m_{cor1} = \frac{m_r * \left(1 - \frac{\rho_{air\_cal}}{\rho_{mass\_cal}}\right)}{\left(1 - \frac{\rho_{air}}{\rho_{object}}\right)} - m_r \qquad (3)$$

Because the extension of the LFCS uses larger beakers of borosilicate glass the density of the object varies, thus changing the Buoyancy correction as the beaker collects more water. An example of this nonlinear relation between object density and amount of collected water in a 1000 ml beaker is shown in Figure 5.

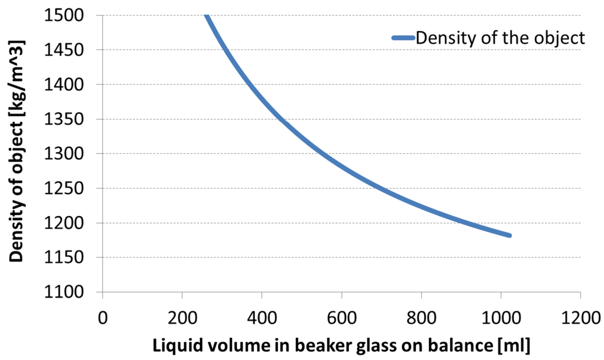

Figure 5. Nonlinear relation between density of the object and the amount of water collected in the beaker. Density of the borosilicate glass is 2230 kg/m^3. Water density at 21 °C and 1 bar(g) is 998.2 kg/m^3.

RESULTS

Crosschecks

Crosschecks between the LFCS and the extension setup confirm good consistency. The crosschecks were performed with a Bronkhorst® ML120- and M13 mini-Cori flowmeter as transfer standards. The ML120 was used to measure the deviation on 100, 150 and 200 g/h points between the LFCS and reference 1 of the extension setup (Figure 1). The M13 was used to measure the deviation on 1000, 1500 and 2000 g/h between reference 1 and -2 in the extension setup of the LFCS.

Table 1 and Figure 6 show that the measured deviation is within the combined uncertainty. To judge whether the results are consistent the E_n value is used. The E_n value should be below 1 in order to confirm consistency between the references.

Table 1. Crosscheck results between references.

Bronkhorst results

REF flow rate (g/h)	Deviation measured by TS (%)	Combined uncertainty (%)	E_n value (-)
100	-0.04	0.14	0.28
150	-0.05	0.14	0.35
200	-0.02	0.14	0.14
1000	0.01	0.14	0.07
1500	0.02	0.14	0.14
2000	0.01	0.14	0.07

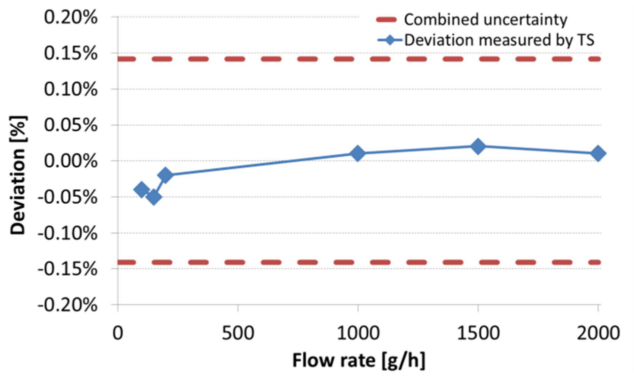

Figure 6. Graphical display of the results presented in Table 1.

Intercomparison

Bronkhorst® participated in an intercomparison on liquid flow rates of 0.5 to 10 kg/h between European NMI's. The intercomparison was coordinated by VSL under EURAMET project 1379, in which liquid micro flow rates from 0.5 kg/h to 10 kg/h are compared between the primary standards of the participants. The participating NMI's are VSL (Netherlands), DTI (Denmark), CMI (Czechia), CETIAT (France), METAS (Switzerland) and LEI (Italy). A M14 mini-Cori from Bronkhorst® was used as the transfer standard in the comparison. Though the results are not yet published at the time of writing, preliminary results show good consistency ($E_n < 1$) between Bronkhorst® and the average of the participating NMI's. When published on https://www.euramet.org/, the results show the traceability of the calibration setup towards the primary standards of European NMI's.

Previous intercomparisons, in which Bronkhorst® participated with the LFCS for liquid flow rates below 200 g/h, already showed good consistency [5][6].

CONCLUSION

The LFCS extension closes the traceability gap for micro flows above 200 g/h. Parts are added to the LFCS extension to provide pure and stable water flows up to 30 kg/h. A new uncertainty budget was made, including the new- and changed uncertainty component contributions to the total uncertainty of the extension setup. By performing crosschecks the extension setup was validated. The results show good consistency. When published, the results of the intercomparison show the traceability towards European NMI's. The LFCS extension setup was ISO/IEC 17025:2005 accredited at the end of July 2017.

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