Micromachines **2015**, *6*, 473-486; doi:10.3390/mi6040473

OPEN ACCESS

micromachines

ISSN 2072-666X

www.mdpi.com/journal/micromachines

Article

Low Flow Liquid Calibration Setup †

Tom H. Platenkamp [1,*], Wouter Sparreboom [1], Gijs H. J. M. Ratering [1], Marcel R. Katerberg [1] and Joost Lötters [1,2]

[1] Bronkhorst High-Tech BV, Nijverheidsstraat 1A, 7261 AK Ruurlo, The Netherlands; E-Mails: w.sparreboom@bronkhorst.com (W.S.); g.ratering@bronkhorst.com (G.H.J.M.R.); m.katerberg@bronkhorst.com (M.R.K.); j.c.lotters@bronkhorst.com (J.L.)

[2] MESA+ Institute for Nanotechnology, University of Twente, P.O. Box 217, 7500 AE Enschede, The Netherlands

† This paper is an extended version of our paper published in the 2nd International Conference on Microfluidic Handling Systems, Freiburg, Germany, 8–10 October 2014.

* Author to whom correspondence should be addressed; E-Mail: t.platenkamp@bronkhorst.com; Tel.: +31-573-45-8800; Fax: +31-573-45-8808.

Academic Editor: Nam-Trung Nguyen

Received: 27 February 2015 / Accepted: 17 April 2015 / Published: 22 April 2015

Abstract: This article describes a primary calibration setup, and its uncertainty, for low flow liquid calibrations at Bronkhorst High-Tech. It will be used to calibrate reference flow meters from 1 to 200 g/h. By setting up an uncertainty budget for this setup, the calibration of the instruments can be compared to that of NMI's (National Metrology Institutes). The uncertainty budget consists of mass, time and mass flow uncertainties/corrections that need to be taken in to account for determining the traceable mass flow. Tests results of different flow meters/actuators measured on the setup support the calculated uncertainty. By participating in an intercomparison with NMI's the measurement and uncertainty of this setup is traceable to European NMI's.

Keywords: liquid mass flow; calibration; uncertainty budget

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1. Introduction

Worldwide there is an increasing demand for low flow liquid mass flow meters. Because applications require traceable measurement of these instruments, they need to be calibrated by a reference that is traceable to a primary standard. At the time of writing, there is no primary standard for low liquid mass flows smaller than 200 g/h [1]. Therefore, a setup that is able to calibrate mass flow meters in the range of 1 to 200 g/h was developed.

In this article a primary low flow liquid calibration setup is introduced, based upon the gravimetric method of measuring mass flow. A balance is used as reference for its traceability towards higher standards on mass. A unique feature in this setup is that the DUT (device under test) itself establishes a stable flow. With stable flow a mass flow that is constant for several minutes or more is meant.

In Section 2 the setup and uncertainties are described. Details about the procedure and measurements on the setup are given in Section 3. The results of the measurements can be found in Section 4 and the conclusions are given in Section 5.

2. Description of the Setup and Uncertainties

This section describes the low flow calibration setup and its uncertainties on mass, time, and mass flow.

2.1. Setup

The low flow liquid calibration setup that was built consists of a pressurized liquid tank, filter, degasser, pressure sensor, DUT with control valve, balance, and valves with small internal volume. All parts in the setup are connected by 1/16" tubes. In Figure 1 a schematic of the setup is shown. It is placed on a granite table with shock absorbing blocks to reduce vibration interference from the environment. The complete setup with table is installed in a box to reduce fast temperature changes and draught.

The uncertainty budget is based on the flow path downstream from the control valve, where the liquid is led by a small metal tube, to a beaker on the balance. In the following subsections the theoretical background of a high precision balance used as mass flow reference and typical uncertainties involved with gravimetric calibrations is described.

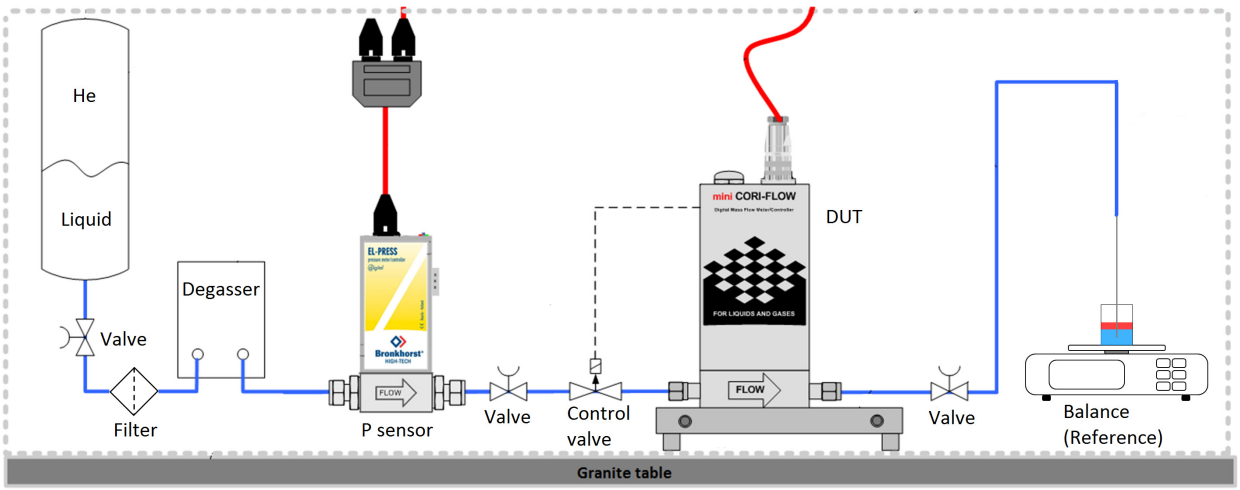

Figure 1. A schematic of the setup with all its components.

2.2. From Mass-to-Mass Flow

In the calibration setup a high precision balance is used as mass flow reference. This is done by differentiating its measured mass (Δm) to measured time (Δt). The resulting output is mass flow ($\dot{m}$), as shown in Equation (1) below:

$$\dot{m} = \lim_{\Delta t \to 0} \frac{\Delta m}{\Delta t} = \frac{\mathrm{m}_r}{t_r} \quad (1)$$

where m_r and t_r are, respectively, the reference mass and time.

An RS232 balance interface is used between the balance and the data acquisition computer. This interface combines each mass sample with the correct time sample, resulting in a mass flow. This way the flow of the DUT can be directly compared to the flow indicated by the reference (balance). The uncertainty on mass and time of the balance is characterized and used to determine the total uncertainty of the setup.

2.3. Description of Uncertainties

Besides the mass- and time- (m_r and t_r) uncertainties of the reference, there are also extra mass flow uncertainties ($\dot{m}_{unc}$) due to environmental effects and interactions between tube and fluid. An absolute correction on mass is needed due to buoyant interaction forces [2] (m_{cor}). The actual mass flow is then given by Equation (2):

$$\dot{m} = \left(\frac{m_r + m_{cor}}{t_r}\right) \pm \dot{m}_{unc} \quad (2)$$

As can be seen in the equation above, the actual mass flow consists of four components: m_r, m_{cor}, t_r and $\dot{m}_{unc}$. Therefore, the uncertainty budget is divided into four groups. Each group has its own uncertainties. The uncertainties are combined into a total uncertainty [3] on mass flow with a confidence level of $k = 2$ (95%).

2.3.1. Mass Uncertainties

Mass uncertainties are caused by various possible differences in mass. The total contribution of the balance to the mass uncertainty in the budget can be divided into six individual uncertainties. These uncertainties influence the reference mass (m_r). The reference mass is obtained by subtracting the mass at the start of the calibration (m_0) from the mass at the end of the calibration (m_1), shown in Equation (3) below. $u(xi)$ represents the standard uncertainty, which is, in this case, a mass uncertainty but can be a time or mass flow uncertainty as well.

$$m_r = (m_1 - m_0) \pm u(xi) \quad (3)$$

The reference mass uncertainties are listed below:

- Calibration;
- Drift;
- Readability;
- Linearity;

- Repeatability;
- Temperature sensitivity.

2.3.2. Mass Correction and Uncertainty

The standard buoyancy correction due to density differences between calibration and measurement (m_{cor1}) is shown in the Equation (4). In addition to the raw scale reading of the balance (m_r), the correction depends on the difference in 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 measurement the weighted mass is a beaker with liquid, called "object" in Equation (4):

$$m_{cor1} = \frac{m_r \times \left(1 - \frac{\rho_{air\_cal}}{\rho_{mass\_cal}}\right)}{\left(1 - \frac{\rho_{air}}{\rho_{object}}\right)} - m_r \quad (4)$$

Because a tube is used to deliver the liquid in the beaker on the balance and is partly submerged in the liquid, another buoyant force will make the weighted object appear heavier. For a submerged body, the buoyancy force of the fluid is equal to the weight of displaced fluid [2]. Because the tube is fixed and the cross sectional area of beaker and tube are constant, the extra weight will rise constantly when the liquid rises in the beaker. This results in a second mass correction (m_{cor2}) needed to determine the actual mass, shown in Equation (5). The correction depends on the difference between the two cross sectional areas (A_{tube} and A_{beaker}) and m_r.

$$m_{cor2} = (m_r \times (1 - \frac{A_{tube}}{A_{beaker}})) - m_r \quad (5)$$

Summing the two corrections on mass, m_{cor1} and m_{cor2}, results in a total mass correction (m_{cor}) for the collected mass shown in Equation (6):

$$m_{cor} = m_{cor1} + m_{cor2} \quad (6)$$

2.3.3. Time Uncertainties

The RS232 balance interface determines the mass flow by calculating the difference of the received mass samples divided by the time between each sample. The time uncertainty between the samples depends on the absolute accuracy of the internal clock of balance interface and the round-off error of the clock implemented in the firmware. Irregularities in the clock, or jitter, will not influence the time uncertainty in the budget. Jitter only causes noise in the measured flow, but will not influence the average measured flow. The reference time is obtained by subtracting the time at the start of the calibration (t_1) from the time at the end of the calibration (t_2), shown in Equation (7):

$$t_r = (t_2 - t_1) \pm u(xi) \quad (7)$$

2.3.4. Mass Flow Uncertainties

Environmental Influences

Fast environmental temperature changes can have a big influence on measurements during calibration. Liquid and tube volumes that shrink or expand within calibration time will create flow fluctuations, resulting in flow errors between DUT and reference. Because fast temperature changes have a big influence, the setup is fitted in a box that prevents this. The allowed temperature range is 21 ± 2 °C. Typical temperature changes are between 0 and 0.2 °C/h. Besides keeping the temperature stable, the liquid volumes in the setup should be kept as small as possible to reduce the temperature influence on the mass flow measurement even further. This is done by using PTFE 1/16" tubing with an inner diameter of 0.5 mm. At the end of the flow path a stainless steel tube (outer diameter of 0.64 mm and inner diameter of ~0.6 mm) is used to deliver the water in the beaker on the balance, because of its smooth outer surface.

The environmental changes also affect the evaporation rate of the water in the beaker. To reduce and stabilize the evaporation rate, a layer of low viscous oil (red colored Shell morlina 10 oil) is put on top of the water surface, as shown in Figure 2.

The evaporation rate of water through the oil layer was measured over 48 h. This is shown in Figure 3.

Figure 2. Griffin 100 mL PP beaker filled with water and red colored oil. The oil layer covers the water surface.

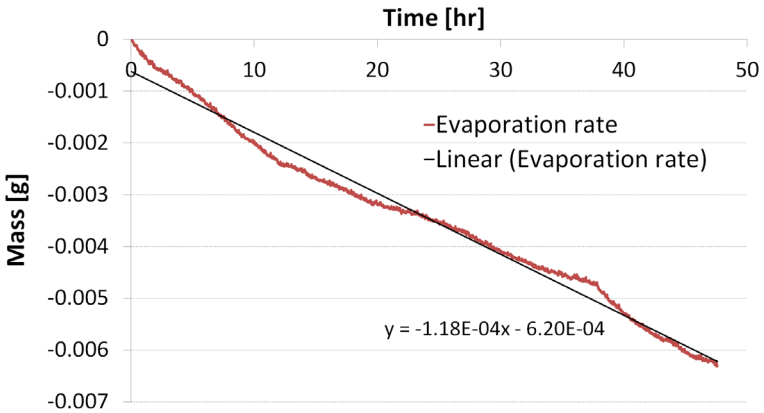

Figure 3. Shows the evaporation rate measured over 2 days with a layer of oil on top of the water surface. The linear trend line shows an average evaporation rate of ~0.118 mg/h.

The evaporation rate of ~0.118 mg/h was measured inside the box of the setup. During this measurement, the environmental humidity and temperature in the box was logged. The humidity and temperature behaviors are shown in Figure 4.

The influence of these environmental effects on used materials and mass flow measurement is accounted for in the uncertainty budget.

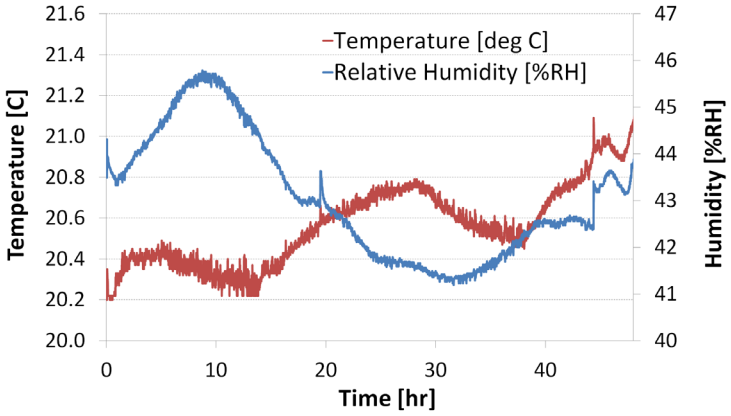

Figure 4. Temperature and humidity behavior inside the box during evaporation measurement.

Surface Tension Effects between Tube and Fluid

Because of this oil layer on top of the weighted mass (water), another effect appears that influences the mass flow uncertainty. Due to the change of the water volume in the beaker during measurements, the water with the oil front moves along the metal tube. As the oil is pushed along the tube, the surface tension between tube, water, and oil varies due to environmental changes and possible irregularities on the outer surface of the tube. These variations in surface tension can cause an irregularity in mass flow. The contribution of this effect to the mass flow uncertainty is difficult to predict by theoretical analysis, because of experimental variability. Therefore, the uncertainty of this effect was empirically determined. While moving the tube up and down in the fluid with a linear motor the fluctuations in mass flow are measured. These fluctuations are used to determine its worst case uncertainty contribution to the total uncertainty.

2.4. Typical Mass Flow Uncertainty

For the lowest flow rates 1–15 g/h the surface tension uncertainty between tube and fluid is the most dominant uncertainty in the budget. For higher flow rates (15–200 g/h), the uncertainty of the mass correction becomes dominant. The total uncertainty depends on the combination between flowrate and reference time. The reference time needs to be at least 200 s at high flow rates, and can go up to 3600 s at low flow rates. An example of the uncertainty budget for a flow rate of approximately 15 g/h is given in Table 1.

Typical uncertainty of the reference mass flow;

- For flow rates between 1 and 15 g/h the reference uncertainty is approximately 0.6 to 0.06%Rd (percentage of reading).
- For flow rates between 15 and 200 g/h the reference uncertainty is approximately 0.06 to 0.04%Rd.

Table 1. An example of the reference uncertainty budget for a flow rate of approximately 15 g/h.

Component	*xi* value	Standard uncertainty ($u(xi)$)	Sourse number	Source name	Sensitity coeficient (ci)	Ci × $u(xi)$	Absolute	Influence total uncertainty
M_r (g)	7.50	4.50×10^{-6}	1	Scale Calibration	2.00	9.01×10^{-6}	9.01×10^{-6}	0.08%
		1.55×10^{-4}	2	Scale_drift	2.00	3.11×10^{-4}	3.11×10^{-4}	2.80%
		1.00×10^{-5}	3	Scale_readability	2.00	2.00×10^{-5}	2.00×10^{-5}	0.18%
		1.00×10^{-4}	4	Scale_linearity	2.00	2.00×10^{-4}	2.00×10^{-4}	1.80%
		1.20×10^{-4}	5	Scale_repeatability	2.00	2.40×10^{-4}	2.40×10^{-4}	2.16%
		3.00×10^{-5}	6	Sacle_drift temperature	2.00	6.01×10^{-5}	6.01×10^{-5}	0.54%
M_{cor} (g)	7.51	1.88×10^{-4}	7	Mass-cor_rho air at calibration	2.00	3.75×10^{-4}	3.75×10^{-4}	3.38%
		1.13×10^{-4}	8	Mass-cor_rho mass at calibration	2.00	2.26×10^{-4}	2.26×10^{-4}	2.03%
		1.55×10^{-3}	9	Mass-cor_rho air at measurement	2.00	3.10×10^{-3}	3.10×10^{-3}	27.92%
		5.58×10^{-4}	10	Mass-cor_rho object at measurement	2.00	1.12×10^{-3}	1.12×10^{-3}	10.04%
		1.60×10^{-4}	11	Mass-cor_surface variation of tube	2.00	3.20×10^{-4}	3.20×10^{-4}	2.88%
		2.10×10^{-4}	12	Mass-cor_surface variation of breaker	2.00	4.20×10^{-4}	4.20×10^{-4}	3.78%
t_r (s)	1.80×10^{3}	5.40×10^{-2}	13	FBI_crystal	-8.34×10^{-3}	-4.50×10^{-4}	4.50×10^{-4}	4.05%
		6.19×10^{-2}	14	FBI_roundings error clock	-8.34×10^{-3}	-5.16×10^{-4}	5.16×10^{-4}	4.64%
$\dot{m}_{unc}$ (g/h)	1.50×10^{1}	3.54×10^{-4}	15	Massflow_evaporation rate	1.00	3.54×10^{-4}	3.54×10^{-4}	3.19%
		3.00×10^{-3}	16	Massflow_surfacetension/sticky	1.00	3.00×10^{-3}	3.00×10^{-3}	27.00%
		3.93×10^{-4}	17	Massflow_volume expansion medium	1.00	3.93×10^{-4}	3.93×10^{-4}	3.54%
$\dot{m}$ (g/h)	1.50×10^{1}				$u(\dot{m})$ (g/h)	9.23×10^{-3}		100%
					$u(\dot{m})$ (%)	0.06%		

3. Experimental Section

3.1. Procedures

In the following subsections, the procedures for generating a pure and stable liquid flow to perform a calibration are described.

3.1.1. Pure Liquid

The flow path situated before the control valve of the DUT consists of parts to create a pure liquid flow; this is needed to create a stable flow. Particle contamination in the liquid is prevented by using demineralized water (laboratory grade 3) and a 0.5 μm SS filter. The filter has a large surface to reduce pressure drop (Figure 5).

In addition to possible contamination of the liquid by particles, it is also contaminated by dissolved gas. As the dissolved gas undergoes a pressure drop the gas bubbles will expand and could get stuck in the flow path acting as a hydraulic spring damper. This causes major flow errors between DUT and reference. By pressurizing the liquid with Helium (low solubility in water) and the use of a degasser, the amount of dissolved gas in the liquid is reduced to a minimum and a fast and responsive system is obtained. The degasser used is a 4 × 480 μL chamber mini degasser from Systech (BioTech).

Figure 5. Low delta p SS filter with a pore size of 0.5 μm.

3.1.2. Stable Mass Flow

A stable mass flow is created by using the DUT to control a piezo electric control valve. With stable flow a mass flow that stays within the specifications of the DUT is meant. The control valve is situated upstream from the DUT to eliminate its influence on the comparison between DUT and balance. This is a unique way of establishing a stable low liquid flow during calibration. To prevent control instability of the flow, pressure fluctuations may not exceed 5 mbar/min. The flow path downstream from the control valve is led, by a small metal tube, to the balance. The tube transports the liquid into a beaker on the balance. The outlet of the tube must stay under the water surface in the beaker to prevent droplet formation. Doing so results in a continuous and stable flow towards the balance.

3.1.3. Calibration

A measurement is started by controlling the flow from zero to the given set point by the DUT. If the DUT reaches the given set point and stabilizes, the reference is held stable within the required

accuracy and stability. The balance interface sampler uses a moving average filter with a filter length of 60 s. Therefore, a stable flow on the reference is reached after a minimum of 60 s. During calibration the flow on the reference must be kept stable. This ensures a comparison between DUT and reference during calibration with two stable flows. The calibration duration, or reference time (t_r), depends on the flowrate; the higher the flowrate, the shorter the duration of the calibration. Calibration durations for different flow ranges are described in Section 2.4. For comparison, at least 50 samples of the average flowrates (of reference and DUT) are used. An example of calibration with duration of 600 s is given in Figure 6.

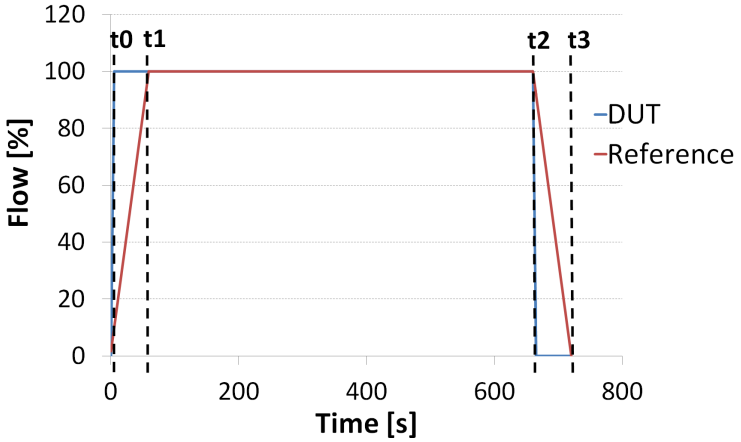

Figure 6. Example of calibration between DUT and reference. At t_0 a flow set point is sent to the DUT, that controls the flow to 100%. After approximately 60 s the reference becomes stable at t_1. If both flows are stable the calibration can start. After 600 s of sampling, the calibration is stopped and the DUT controls the flow to 0% (t_2). The reference is at 0% again after approximately 60 s (t_3).

3.2. Measurements

Every part in the setup was chosen to provide repeatable, reproducible, and accurate calibrations. The test details in this subsection show how repeatable and accurate these calibrations are and whether the results are within the stated uncertainty.

By performing measurements with a mass flow meter and syringe pump as DUT the repeatability, reproducibility and insensitivity for different liquid flow meters/actuators is tested. Stated measurement and uncertainty of the setup is supported by the results of an intercomparison between national labs, in which Bronkhorst High-Tech participated.

3.2.1. Mass Flow Meter as Flow Controller

A calibrated mass flow meter was used to test our setup on measuring mass flows. The meter that was used is a M12p Coriolis mass flow meter from Bronkhorst Cori-Tech (Ruurlo, The Netherlands, shown in Figure 7). This meter has a 0.2%Rd (percentage of reading) accuracy with a 0.05 g/h zero stability and covers a flow range of 0–200 g/h. It covers the same flow range as the calibration setup and has a good stability. Due to these specifications, it is used as DUT in the first measurements performed on this setup. Details of the flow control by the DUT in the setup can be found in

Section 3.1.2. Five different flow points were measured over the entire flow range in the following order; 200, 100, 20, 5, and 2 g/h. Each flow point was measured four times to check its repeatability.

Figure 7. The M12p Coriolis mass flow meter.

3.2.2. Syringe Pump as Flow Generator

In addition to the previous tests with a mass flow meter as DUT (see Section 3.2.1.), additional measurements with a volumetric flow actuator as DUT were performed. The actuator used was a Nexus 3000 syringe pump, shown in Figure 8a. The pump has an accuracy of ±<0.35% and operates within a flow range of 0.001 µL/h up to 90 mL/min. Using water as medium, this is equal to a mass flow range of approximately 0.001 mg/h to 5400 g/h. Measurements on four flow points were performed; 333, 100, 33 and 10 µL/min. Flow points 333 and 100 µL/min are repeated 3 times using a calibrated 25 ml glass syringe. Flow points 33 and 10 µL/min are repeated 4 times respectively using a calibrated 5 and 2.5 mL glass syringe. The syringes are filled with extra pure DI water through the degasser fitted in the setup. The measurements are carried out with the syringe pump directly connected to the tube that leads the water to the beaker on the balance as shown in the Figure 8b. To compare the results of the DUT to the reference, the volumetric dosage flow rates (µL/min) of the DUT are converted to mass flow (g/h) using the actual density.

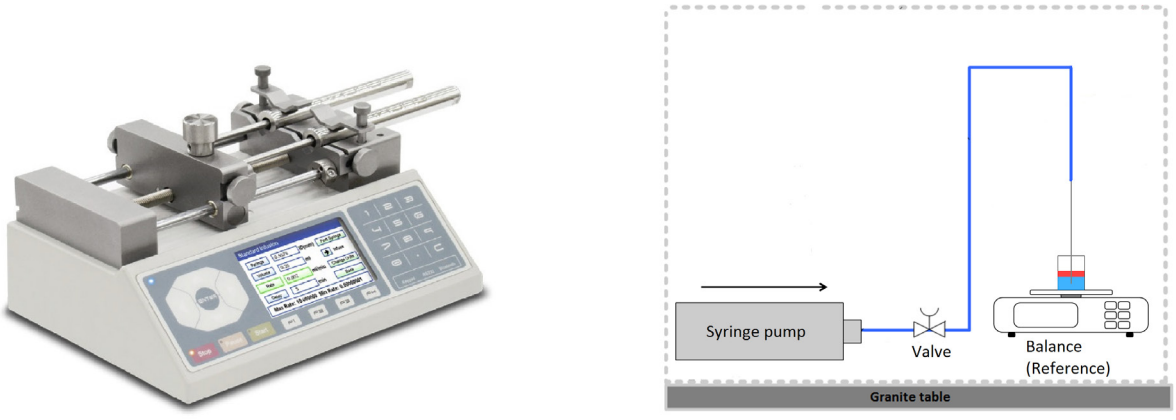

Figure 8. (**a**) The Nexus 3000 syringe pump. (**b**) Test setup with the syringe pump as DUT. It is directly connected to the tube that leads the water to the beaker on the balance.

3.2.3. Intercomparison

By participation in an intercomparison between NMI's, the setup and method for calibrating low liquid flows was validated [4]. In this comparison two Bronkhorst Coriolis mass flow meters where used as transfer standard, the M12p and M13. The M12p was used to calibrate 200, 60, 20, 6, and 2 g/h, the M13 to calibrate 200 and 600 g/h. The M13 has a 0.2%Rd accuracy with a 0.2 g/h zero stability and covers a flow range of 0 g/h to 2000 g/h. M12p specifications can be found in Section 3.2.1. The average result of three participating labs is compared with the average result of Bronkhorst and its trumpet curve. The tests were performed with the setup as shown in Figure 1 for the flow rates up to 200 g/h. The M12p used in the intercomparison differs from the M12p used in the first measurements on the setup, described in Section 3.2.1. In Section 4.3, the results of the M12p in this comparison are given. Only the results of the M12p are presented since it covers the entire flow range. The M13 results are omitted in this article, since it does not give additional data to verify this setup. Other participants in the intercomparison described the results of the M13 in their reports [4,5].

4. Results

In this section the results of the measurements carried out on the low flow liquid calibration setup are presented. Details on the measurements can be found in Section 3.

4.1. Mass Flow Meter

A calibrated M12p mass flow meter was used as DUT to test the low flow liquid calibration setup on mass flow. Test details can be found in Section 3.2.1. The test results are shown in Figure 9.

All flow points measured are within the uncertainty of the reference. As the deviation of the measurements increases by lowering the flow rate, the repeatability decreases. The measured deviation and repeatability is within the accuracy ± zero stability of the M12p.

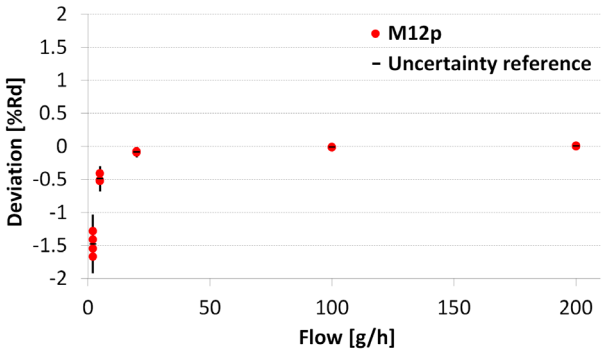

Figure 9. Mass flow deviation between the M12p as DUT in the setup and the reference. Flow rates 20, 100, and 200 g/h are very repeatable making the four measurements per flow point indistinguishable from each other in this chart.

4.2. Syringe Pump

Aside to previous tests with a mass flow meter as DUT extra measurements were performed with a syringe pump as DUT. By using a syringe pump as DUT the reference in the setup on volumetric flow

was tested. Test details can be found in Section 3.2.2. After converting the dosed volume flow to mass flow, the results are compared to the reference in the setup. Figure 10 shows the results with the uncertainty of the reference on mass flow.

Average deviation between DUT and reference is close to zero for all measured flow points. The repeatability is within 0.5%Rd over the entire range measured. The uncertainty shown in Figure 10 is the uncertainty of the reference on mass flow, neglecting the uncertainty contribution of the volume-to-mass flow conversion.

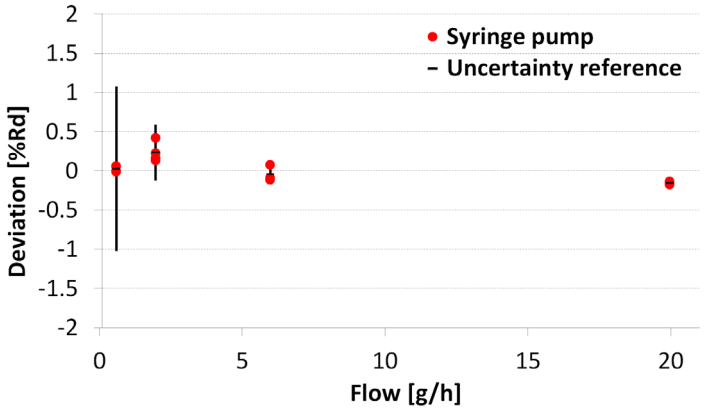

Figure 10. Mass flow deviation between the syringe pump as DUT and the reference.

4.3. Intercomparison

By participation in an intercomparison between NMI's, the setup and the method for calibrating low liquid flows was validated. Details of this intercomparison can be found in Section 3.2.3 and in the supplement report [4]. The trumpet curve in Figure 11 shows the boundaries of the error ± zero stability. All points within it meet the stated maximum allowed error and zero stability.

The intercomparison shows that the measurements and accompanying uncertainty budget are consistent (Table 2). The E_n values are the result of comparing the mean value obtained by this setup to the mean value of all labs. This value should be <1 to be consistent.

By comparing our measured points on each flow it is shown that the repeatability is well within the uncertainty of the setup and trumpet curve of 0.1%Rd ± 0.005 g/h (Figure 12).

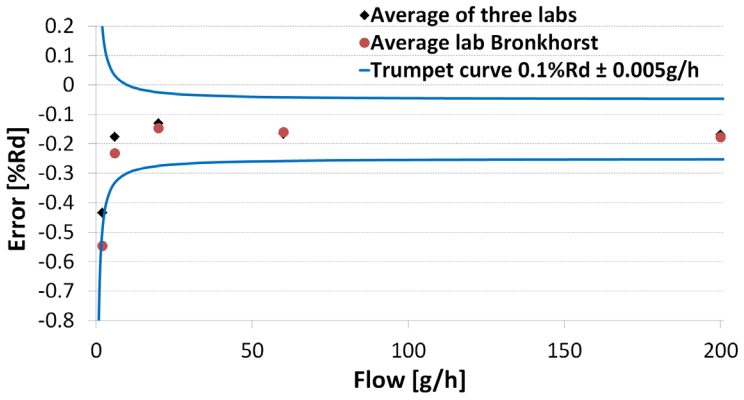

Figure 11. Measured average deviation of the M12p by three labs and the average deviation measured by Bronkhorst. The trumpet curve shows the boundaries of the error ± zero stability.

Table 2. Results of the comparison between the low flow liquid calibration setup at Bronkhorst and the primary standard of three national metrology labs in Europe.

Bronkhorst results		Uncertainty setup (%)	E_n value (-)
Flow rate (g/h)	Error (%)		
2	−0.55	0.31	0.05
6	−0.23	0.11	0.21
20	−0.14	0.06	0.05
60	−0.16	0.06	0.10
200	−0.17	0.10	0.14

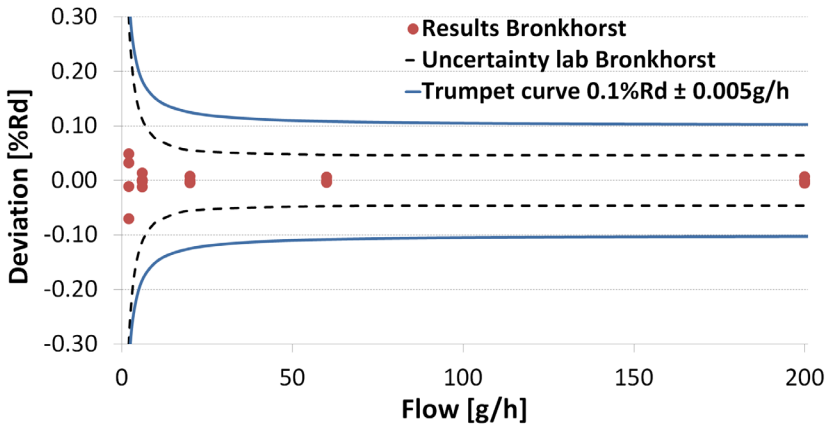

Figure 12. Repeatability of the measured points on each flow rate in the intercomparison measurements of Bronkhorst. All points are within the uncertainty of the reference setup and trumpet curve of 0.1%Rd ± 0.005g/h.

The uncertainty is within the trumpet curve of 0.1%Rd ± 0.005 g/h, which is used as reference for calibrations. Both flow meters (M12p and M13) are found repeatable and reproducible enough as a transfer standard [5].

5. Conclusions

A low flow liquid calibration setup is built and validated. With this setup one is able to perform calibrations on mass flow meters in the range from 1 to 200 g/h. Tests with the M12p and syringe pump show that the repeatability is well within the calculated uncertainty of the reference. It is found that the uncertainty of this setup is within a 0.1%Rd ± 0.005 g/h trumpet curve with a 95% confidence level. This is validated by the results of the intercomparison, in which Bronkhorst participated. The M12p and M13 are both found repeatable and reproducible enough as transfer standards [5].

Acknowledgments

The authors would like to thank all the participating NMI's in the EMRP project: metrology for drug delivery. Special thanks to Peter Lucas and Harm Tido Petter from VSL for their support and fruitful discussions. The authors would also like to thank Sander Smeenk for his software support.

Author Contributions

Tom Platenkamp and Gijs Ratering designed and built the setup. Tom Platenkamp conducted most experiments and analyzed the results. Wouter Sparreboom conducted the larger part of the syringe pump experiments and participated in several discussions regarding the experiments. The uncertainty budget was designed by Tom Platenkamp and was reviewed by the other authors. The manuscript was designed and mainly written by Tom Platenkamp. Tom Platenkamp and Wouter Sparreboom finalized the manuscript. Marcel Katerberg supervised the project together with Joost Lötters who was also the principle investigator.

Conflicts of Interest

The authors declare no conflict of interest.

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