THE PROGRESS OF GRAVIMETRIC PRIMARY STANDARDS FOR LIQUID FLOW CALIBRATION AT THE DANISH TECHNOLOGICAL INSTITUTE FROM 500 m^3/h TO 1E-9 m^3/h

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Abstract

The Danish Technological Institute has extended their primary lab liquid calibration range from 5 l/h and down to 1 µl/h using gravimetric set-ups. The calibration uncertainty is between 0.05 % and 0.5 %, but the uncertainty still needs to be validated by an intercomparison. A secondary objective when building the standards was to enable dynamic measurement, enabling characterization of varying or pulsating flows.
In the paper the design details of the systems going from large flow rates to small flow rates are discussed. Evaluation measurements made to assess the performance of the set-ups as well as the uncertainties are presented.

Introduction

Back in the 1980s flow meters were calibrated by the manufacturers and no service for independent evaluation and calibration were available. With the introduction of ISO 9000 in 1987 and the growth in certifications, a demand for independent accredited calibration of water flow in Denmark arose. The Danish Technological Institute (DTI) built the flow standard in 1989 ranging 270 m^3/h to 5 l/h, targeting both industrial needs as well as utilities. DTI became the designated institute for water flow in 2000. Since then the flow range has been extended to 500 m^3/h with a water temperature range from 4 to 85 °C and with a upcoming increase up to 2000 m^3/h, this to satisfy customer demand especially for water and district heating meters calibration and type testing.
In 2008 DTI started extending their flow range downward, expecting an upcoming similar demand for independent calibration from the medical industry and intensive care units as was the case for the industry in the 1980s. A large amount of devices used in the medical industry work with liquid flow rates lower than what is supported by the current metrological infrastructure. For bioresearch purposes syringe pumps working down to a claimed liquid flow rate down to 1 pl/h are available [1].
The first target was to be able to calibrate flow meters and medical pumps down to flow rates of 100 ml/h (completed in 2009), the second step to extend the range down to 1 ml/h (completed in 2010) [2] and most recent extending the range to below 1 µl/h (completed in 2013), which currently seems to be the limit for gravimetric set-ups using microbalances.

Design considerations

In this publication the calibration facilities are divided into three parts.

Ultra

This type of setup covers flow rates that are too high for a gravimetric setup to be feasible, which at DTI is decided to be 500 m^3/h. Here DTI uses a series of reference flow meters in parallel to extend the gravimetric range.

To achieve low uncertainties with the parallel reference flow meter setup for the ultra-range, it is important to have stable and reliable reference flow meters. The design of the set-up with the reference flow meters in the test rig is made with fixed in- and outlet piping as a package to be used also for the frequent calibration of the flow meters at our 'intermediate' gravimetric test rig. For the separation of the total flow into the parallel reference flow meters and to combine the flow after the meters a special manifold system is made.

The actual flow profile into the section for meter under test will be documented by LDV measurements.

Intermediate

This type of setup covers flow rates from 500 m3/h to 100 ml/h using gravimetric methods for flow measurement e.g. ISO 4185 [3].

For the intermediate range, besides an accurate weighing system the focus is on accurate determination of the calibration time, by reducing the diverter timing error using the flying start/stop method.

To calibrate at different water temperatures than ambient temperature, homogenization and insulation is important to minimize spatial thermal gradients throughout the system.

Micro

This type of setup covers flow rates from 100 ml/h to 1 µl/h which has been considered too demanding for gravimetric flow calibration due to the high evaporation of 2-3 mg/h pr. cm^2, and surface forces.

To be able to measure in the micro range, it is first of all necessary to limit the evaporation. At the lowest flow rate, the evaporation is, if left untreated, two decades higher than the flow rate. At DTI a liquid oil cover is used as evaporation trap, which is very efficient (9 nl/h evaporation remaining). However the flow needs to be delivered below the oil surface through a tube and this leads to a number of challenges, e.g. capillary forces, buoyancy, inertia, stiction and friction, absorption, adsorption, stick/slip and vibration transferal. To limit these effects, the tube is fixed, made of 1/32" stainless steel tubing and surface coated to become oleophobic. Further to equalize any heating supplied by the device under test, a water-to-water heat exchanger is mounted around the tubing, this enables a more stable flow as well as makes the density and buoyancy corrections more accurate. The measurement is dynamic, which means that each mass measurement (10 Hz) is time stamped and using Deming regression [4] the dynamic flow and regression uncertainty is calculated.

Theory

Gravimetric calibration of water mass flow is determined by measuring the delivered or removed mass from a vessel during a measured time period.

$$Q = \frac{m}{t}$$

Where Q is mass flow, m is mass and t is time. For volume flow rates the density of the liquid is included in the equation.
For the three calibration facilities the measurement of time can be quite similar, using e.g. a start-stop method or time-stamping a series of mass measurements to determine the dynamic flow. The dominance of this uncertainty component only arises during very short calibration times e.g. less than 5 minutes.
The main difference of the set-ups lies in the method for determining the delivered mass, and the dominant uncertainties of the measurement.

Uncertainties

A number of uncertainties are briefly summarized and then the most significant uncertainties of each range is detailed afterwards.

The environment of the calibration setup is considered. The range, measurement uncertainty as well as temporal and spatial gradients of temperature, humidity, pressure, air density, drafts, gravity, vibrations, electrostatic and RF noise all can produce errors in the mass measured.

The medium is often water, but can also be other fluids. The medium density, purity and temperature are considered. The medium temperature affects the viscosity and thus the flow stability and a temperature difference compared to the surrounding air will produce a convection draft.

The mass measurement using weighing cells or microbalances have beside a calibration uncertainty further uncertainties from the resolution, nonlinearity, repeatability, reproducibility, eccentricity, zero point stability, response time and lag and drift since last calibration. Changes in buoyancy during calibration of both the offset masses (e.g. the vessel) as well as of the medium will affect the mass measurement. For the smallest flow rates, water adsorption changes can also be relevant.

The calibration time with respect to the beginning and the end of the gravimetric flow measurement needs to be simultaneous and with fully developed flows. At DTI either a diverter is used or the flow is measured continuously to ensure measurement on a stable flow. Further the time measurement needs to be calibrated to become traceable.

The connections from the device under test to the reference setup needs to be leak tight and preferably not water absorbing. It is also good especially for small tubing to minimize air-traps, dead volumes and clogging.

The outflow pipe into the vessel yields as described in the design considerations a number of challenges.

The flow generator has flow stability drift and might produce flow pulsations.

The device under test has a resolution, repeatability but might also be temperature, pressure and pulsation dependent.

After evaluating these uncertainties, it is a good idea to check the theory for the possibility of unconsidered uncertainties. This is done by cross checking, predicting the effect of variations in the set-up.
Examples of such variations are changing the calibration and stabilization time, changing the tube diameter going into the vessel, changing the vessel dimensions etc.

The dominating uncertainties for the DTI gravimetric primary liquid flow standard, excluding the device under test, for each range are summarized here:

Ultra

The measurement uncertainty in this flow range is expected to be 0.5 % (k=2).

Calibration uncertainty for the reference flow meters are expected to be in the range from 0.1% up to 0.3% depending on the actual number of reference flow meters used for achieving the desired flow rate.

Drift/stability between calibrations of the reference flow meters are expected to be in the range 0.1% to 0.2%.

Temperature influence on the reference flow meters will be determined and corrected and the uncertainty of the correction is expected to be in the range 0.1% to 0.2%.

Intermediate

The measurement uncertainty in this flow range is between 0.05 % and 0.1 % (k=2).

Diverter timing error (up to 0.02 s) is dominant for the flow rates 100 – 500 m^3/h.

Below 100 m3/h uncertainties from the scale become significant.

For flow rates below 20 m3/h at water temperatures between 50 and 85 °C determination of the temperature at the meter under test (up to 1 °C) becomes the dominant uncertainty contribution.

Micro

The main uncertainty (>90 %) comes from the uncertainty on the correction for the forces between the tube and the water and oil in the measurement beaker. The measurement uncertainty is about 0.05 % (k=2) in this flow range.

Below 300 µl/h flow rates, the balance uncertainty and uncertainty on the correction for buoyancy become dominant (90 % at 5 µl/h), as the level of water rise becomes insignificant (about 20 µm). The measurement uncertainty is rising to about 0.5 % (k=2) at the lowest flow rate of 1 µl/h.

Measurements

Using dynamic measurement the stability of flow generators such as syringe pumps, peristaltic pumps etc. can be determined.

A 50 µl glass syringe was mounted into a Tecan Cavro XLP 6000 Syringe pump and set the pump to empty the syringe at a rate of 10 ml/h and 5 ml/h. The dynamic, gravimetric measurement of the output is shown in figure 1 and 2.

Figure 1

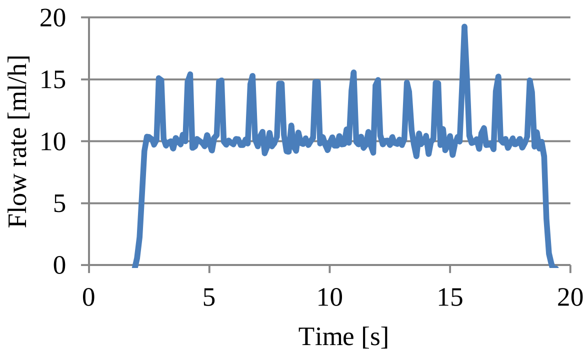
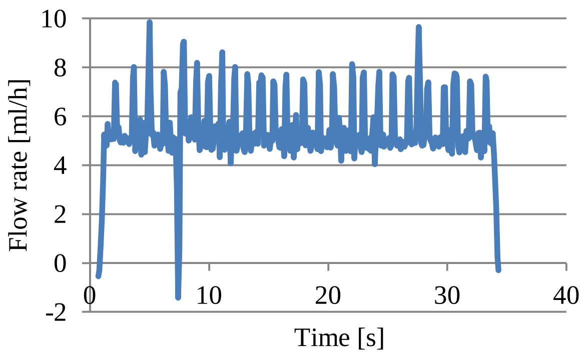


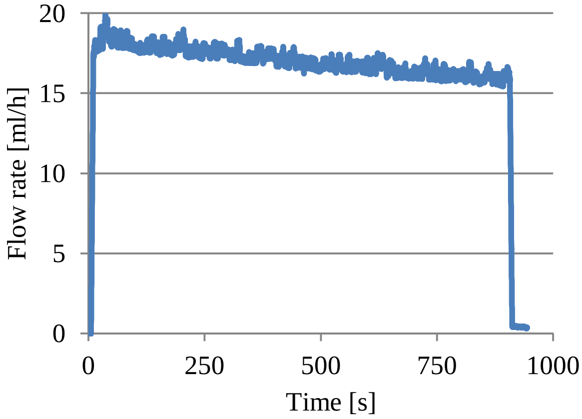
Figure 2



The resolution of the balance is 1 µg, the output stability is below 10 µg, the 99.9999 % step response time is 0.5 sec and the readout is 10 Hz, window size is 0.2 sec. So the pulsating behaviour seen in the figures is due to mechanical instability of the very thin plunger, step size of the step motor and stick/slip in the syringe.

Measuring on a pressure driven pump it is possible to see if the flow rate is slowly dropping which could lead to a calibration error in not treated properly, see figure 3.

Figure 3



Discussion

For the Ultra test rig it will be necessary in the future to perform intercomparisons to prove the claimed uncertainty, especially for flow rates above 500 m^3/h.

The Intermediate test rig has since 1990 participated in several intercomparisonswith very fine results [5], [6]. For all European flow laboratories time has shown difficulties to get good results in the lower range of flow (5 - 25 l/h). The problem to make these intercomparisons in this flow range is both in the difficulty to find reliable and stable transfer standards but also that there are problems in realizing these flow rates in the test rigs with the claimed low uncertainties.

The flow rates below 5 l/h still have to be validated with an intercomparison. Currently a research based intercomparison for the flow rates 2 – 600 g/h is currently running. Intercomparisons for flow rates down to 1 µl/h are planned and are expected executed during 2013/2014.

Conclusion

The Danish Technological Institute now covers liquid flow rate calibration in the range from 500 m3/h to 1 µl/h with accredited calibration uncertainties between 0.5 % and 0.05 % (k=2). The intercomparison results are good and the market for the expanded flow range is large.

References

[1] Harvard Apparatus Syringe pump with 0.5 µl syringe, http://www.harvardapparatus.com

[2] C. Melvad, U. Krühne, J. Frederiksen, "Design considerations and initial validation of a liquid microflow calibration setup using parallel operated syringe pumps" Measurement Science and Technology, Vol 21, no. 7, 2010

[3] ISO 4185 Measurement of liquid flow in closed conduits - Weighing method

[4] R.F. Martin, "General Deming regression for estimating systematic bias and its confidence interval in method-comparison studies", Clinical chemistry, 46(1), 100–4, 2000.
[5] EURAMET Project 863; Intercomparison on water-/heatmeters at 50 °C and 1-20 m³/h
[6] BCR Project MTR166: Intercomparison of Hot Water Calibration Test Facilities; DN15-DN100; 20-70 °C