# RESEARCH INTER-COMPARISON FOR SMALL LIQUID FLOW RATES (2 g/h to 600 g/h)

**Christopher DAVID*, Claus MELVAD, Hugo BISSIG, Elsa BATISTA**

**LNE-CETIAT (Centre Technique des Industries Aérauliques et Thermiques)**
**Domaine Scientifique de la Doua – 25 avenue des arts – B.P.52042, FR-69603 Villeurbanne, FRANCE,**
**christopher.david@cetiat.fr,**

**DTI (Danish Technological Institute)**
**TeknologiparkenKongsvangAllé 29, DK-8000 Arhus, DENMARK**
**cmd@teknologisk.dk**

**Federal Institute of Metrology METAS**
**Lindenweg 50, CH-3003 Bern-Wabern, SWITZERLAND**
**Hugo.bissig@metas.ch**

**IPQ (Instituto Português da Qualidade)**
**Rua António Gião, 2, 2829-513 Caparica, PORTUGAL**
**ebatista@ipq.pt**

**Key words: Water flow, comparison**

## Abstract

This paper presents the results obtained in a research inter-comparison organized in the framework of the development of traceable micro flow facilities for liquids within the EMRP project "HLT07 Metrology for Drug Delivery" [1].

The paper presents the protocol of the comparison and the first results revealing some discrepancies between the laboratories.

## Introduction

The comparison was organized within the scope of the task 1.1. of the EMRP project HLT07 Metrology for Drug Delivery (MeDD). The aim of this task was to validate the calibration facilities with their measurement uncertainties. Four European primary standards (LNE-CETIAT, DTI, IPQ, METAS) were involved for flow rate ranging from 10 ml/min down to 10 µl/min.

The comparison has been performed by means of the calibration of 2 transfer standards (TS) by each laboratory using water at approximately 20°C. The 2 transfer standards were Coriolis Flowmeter M12P and M13 from Bronkhorst High Tech. The M12P was calibrated at 2 g/h; 6 g/h; 20 g/h; 60 g/h and 200 g/h and the M13 was calibrated at 200 g/h and 600 g/h. Calibrations were performed using the individual procedures and flow generators of each laboratory. The pilot laboratory was LNE-CETIAT (France).

## Organisation of the comparison

### Transfer standards

The two Coriolis flowmeter M12P and M13 from Bronkhorst High Tech were sent around by road traffic and not by flight traffic to avoid possible vacuum influences.

The flow meters were equipped with 1/8" stainless steel tubing and fast connecting valves (Upchurch) on the upstream and downstream connectors to easily plug 1/8" or 1/16" tubing. The flowmeters were fixed on a mass block to damp the influence of vibrations as shown in the figure below.

Figure 1 - One of the flowmeters equipped with the 1/8" stainless steel tubing and fast connecting valves (Upchurch) on the upstream and downstream connectors.

**Protocol**

The calibration parameters were the following:
- Upstream pressure: 0,5 bar to 2,5 bar
- Water temperature: 20°C +/- 1°C
- Water flow : 600 g/h and 200 g/h for the M13 (at least 3 points for each flow value)
- Water flow : 200 g/h; 60 g/h; 20 g/h; 6 g/h and 2 g/h for the M12P (at least 3 points for each flow value)

Prior to their individual calibration procedure each laboratory had to perform 3 specific tasks after installing the flow meter on the facility. First, the different laboratories had to verify if the evacuation of possible air bubbles in the piping was successful with their own procedure. Then, water had to flow through the meter at its maximum flow rate (200 g/h for M12P and 600 g/h for M13) for at least 30 minutes to warm up the flow meter electronics. And finally, a "zero" procedure had to be performed.

To check the quality of the flushing procedure, participant were encouraged to open and close a fast valve in their circuit (see figure). If the curve from the meter presented a sharp change in flow rate (at least as quick as the opening and closing) with no oscillation of the flow rate, participants could expect that their degassing procedure succeeded.

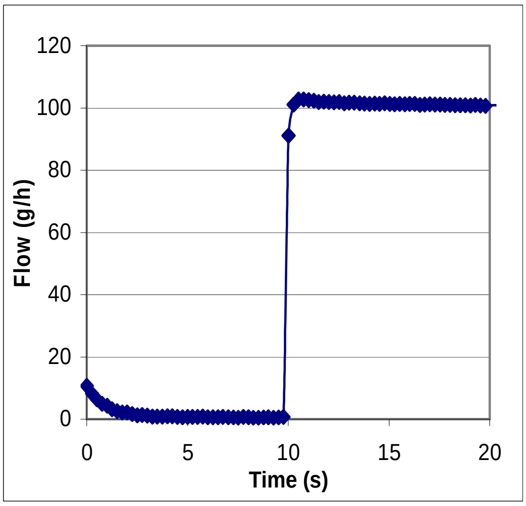


Figure 2 - Example of the fast increase of the flow (data acquisition: every 100 ms)

The "zero" procedure was performed under measurement conditions without flow.

Table 1 - Time schedule of the comparison

| Step n° | Laboratory | Date |
|---|---|---|
| 1-1 | LNE-CETIAT | 16th August 2012 to 3rd September 2012 |
| 1-2 | DTI | 7th September 2012 to 2nd November 2012 |
| 1-3 | METAS | 19th November 2012 to 18th December 2012 |
| 1-4 | IPQ | 8th January to 29th January 2013 |
| 1-5 | LNE-CETIAT | 29th January 2013 to 3rd February 2013 |

**Determination of the reference value**

To determine the reference value of this comparison the weighted mean was selected. To calculate the reference value, the second results obtained at LNE-CETIAT were used to avoid dominance in the calculation of the reference value. The weighted mean value (1) is determined using the inverses of the squares of the associated standard uncertainties as the weights [2], according to the recommendations given by the BIPM:

$$y = \frac{x_1/u^2(x_1) + ... + x_n/u^2(x_n)}{1/u^2(x_1) + .... + 1/u^2(x_n)} \quad (1)$$

To calculate the standard deviation u(y) associated with the flow rate y, equation (2) was used:

$$u(y) = \sqrt{\frac{1}{1/u^2(x_1) + ... + 1/u^2(x_n)}} \quad (2)$$

To identify eventual inconsistent results, a chi-square test can be applied to all n calibration results of each experimental test:

$$\chi^2_{obs} = \frac{(x_1 - y)^2}{u^2(x_1)} + \ldots + \frac{(x_n - y)^2}{u^2(x_n)} \qquad (3)$$

where the corresponding degree of freedom is: $\nu = n - 1$

The consistency check is regarded as failed at a significance level $\alpha = 5\%$ if:

$$\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0{,}05$$

## Results

### Coriolis flow meter M12P

The individual results of each laboratory, the comparison reference value and the uncertainty of the reference value are presented in the following figures:

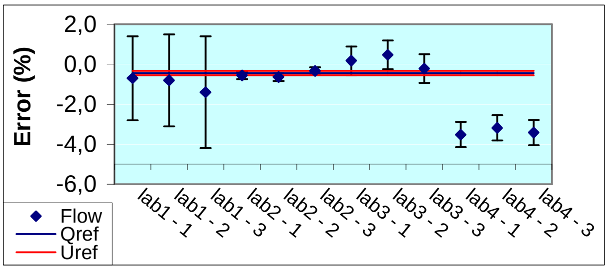


Figure 3 - Results with reference value 2 g/h with M12P

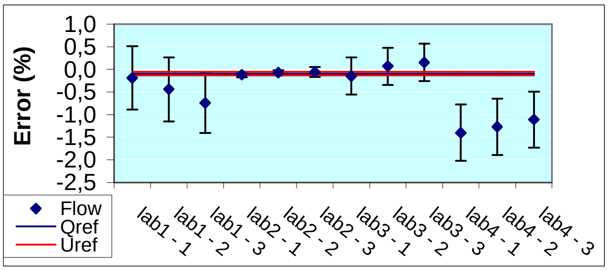


Figure 4 - Results with reference value 6 g/h with M12P

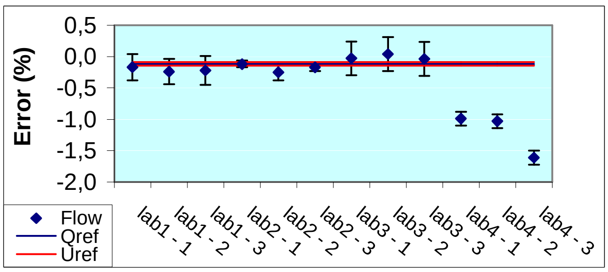


Figure 5 - Results with reference value 20 g/h with M12P

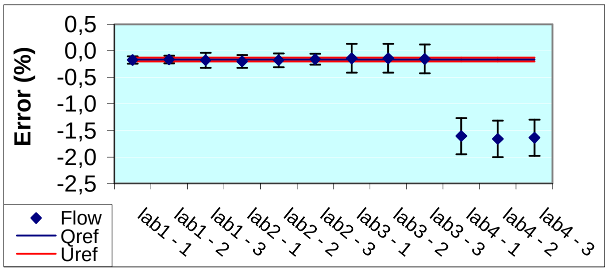


Figure 6 - Results with reference value 60 g/h with M12P

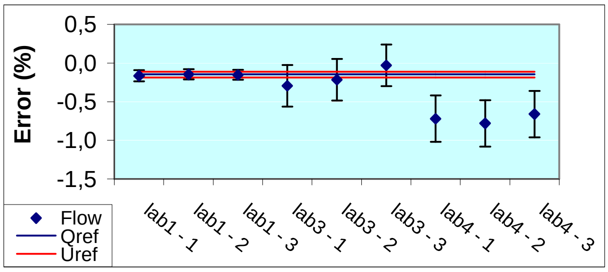


Figure 7 - Results with reference value 200 g/h with M12P

### Coriolis flow meter M13

The individual results of each laboratory, the comparison reference value and the uncertainty of the reference value are presented in the following figures:

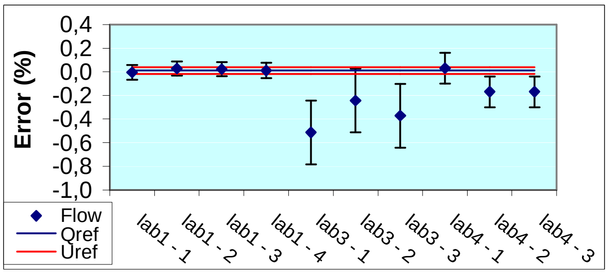


Figure 8 - Results with reference value 200 g/h with M13

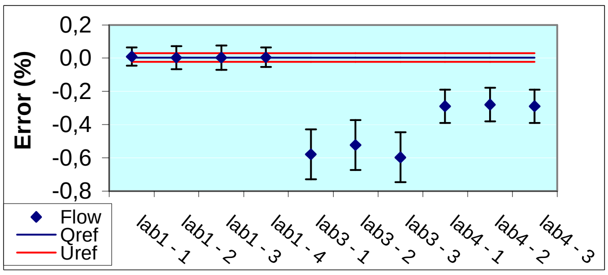


Figure 9 - Results with reference value 600 g/h with M13

## Analysis

Based on the weighted mean analyses, the measured calibration curves with the claimed uncertainties by some of the laboratories were not in agreement with the results obtained. The variability of results was also larger for the M13 meter than for the M12P meter

The drift and the reproducibility of the flow meters over month have been measured by 2 laboratories. The results obtained in one laboratory with the M12P are shown below and we can clearly see that the reproducibility of the coriolis flow meter is very good. The second laboratory presented larger discrepancies between the measurements. A poor reproducibility of the laboratory is suspected. For both laboratories, a good repeatability of the meter was identify.

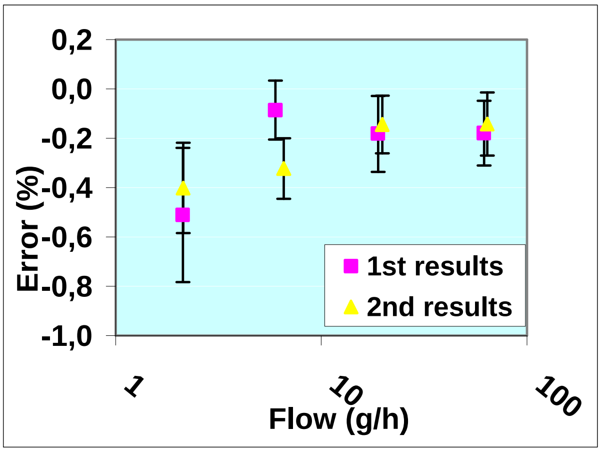


Figure 10 - Reproducibility tests with M12P

Two laboratories are now investigating their methods and facilities to determine the reasons which cause these discrepancies. Once the methods and facilities will be improved, additional measurements with the flow meters will be performed. An additional crosscheck for the reproducibility of the coriolis flow meters will also be performed to link the 2 parts of the research inter-comparison.

Technical assessments of the different facilities are also planned to identify potential sources of inconsistency with the claimed uncertainties or the reproducibility of the facilities.

## Conclusion

This research inter-comparison of the micro flow calibrations facilities in the flow rate range from 2 g/h to 600 g/h has raised new issues regarding the handling of the transfer standards and calibration procedures to ensure good and reproducible results. This information will help us to improve the reproducibility of our calibrations facilities.

Another issue is that the uncertainty budgets of the different laboratories can be harmonized to some extent in this frame work.

The two main conclusions that we can point out with respect to the results obtained are:

- The good reproducibility of both coriolis flow meters which makes them suitable as transfer standards for inter-comparison,
- The measured calibration curves with the claimed uncertainties of some of the participating laboratories are not in agreement with the results obtained. Possible reasons might also be found in the calibration procedures.

Two laboratories are now investigating the pressure and temperature dependence, as well as the short term and long term reproducibility of the meters. This knowledge will help us to improve our comparison procedures and take into account some deviations from the predefined measurement conditions in the different laboratories and facilities.

This project is funded by the EMRP (European Metrology Research Program). The EMRP is jointly funded by the EMRP participating countries within EURAMET (European Association of National Metrology Institutes) and the European Union.

## References


[1] http://www.drugmetrology.com/

[2] Cox M.G., The evaluation of key comparison data, Metrologia, 2002, Vol. 39, 589-595