Capacity assessment of a system for metrological traceability on liquid micro flow rate measurement

Brenda Lopes Dias[1]. Marcos Henrique Aquino [2], Maria Helena Farias[3], Jair Koiller[4], Priscila Costa Gabriel[5]

[1]National Institute of Metrology, Quality and Technology, brendaldias@yahoo.com.br
[2] National Institute of Metrology, Quality and Technology ,mhaquino@inmetro.gov.br
[3] National Institute of Metrology, Quality and Technology, mhfarias@inmetro.gov.br
[4]National Institute of Metrology, Quality and Technology , jkoiller-pronametro@inmetro.gov.br
[5]National Institute of Metrology, Quality and Technology, pcgabriel@inmetro.gov.br

Abstract: The necessity of standardizing and traceability to SI in micro scale flow has been subject of intense discuss in the metrology. The aims contribute to establishment and advancement of research in microfluidics at National Institute of Metrology, Quality and Technology (INMETRO). The system is based on the gravimetric method for fluid delivered quantification. This work describes the evaluated capability of bench, in order to provide traceability in microflow rate, encouraged by Bureau International of Poids et Mesures (BIPM) to 2024, when is planned to run the first Key Comparison event in this area.

Keywords: micro flow rate; microfluidica; metrology traceability; gravimetric method

1. INTRODUCTION

The microfluidic can be applied to several fields of the technology. This science and their applications can optimize and improve processes in the areas of science, technology, biomedical, pharmaceutical, chemical, environmental, aerospace and others. However, ensure reliability on quantifying fluid flow rate in micro scale is a challenging task, due the need to control of variable that can influence in the measurement process. Nowadays, there is a gap in the chain of metrological traceability for very flow rate.

Because of this gap, the Bureau International des Poids et Mesures –BIPM [1] has encouraged National Institutes of Metrology to offer systems for providing of metrological traceability on micro flow rate measurement up to 2024, when will run the first international key comparison event in microfluidic.

The metrology community has discussed about the microscale measurement and several techniques and methods have been used by researchers, as who has characterized and has calibrated microfluidic system with different techniques and methods. [2-11]

The present work describes the new standard system for metrological traceability on liquid micro flow rate measurement developed in the Division of Fluid Dynamics at the National Institute of Metrology, Quality and Technology (INMETRO).The system capability is evaluated or different low flow rates. Through the implementation of this system, will be possible to study the flow in micro channels, traceable at national standards and the results will be used as reference to velocity measurement by microPIV (micro Particle Image Velocimetry).

2. METHODS AND INSRUMENTANTION

The measurement technique in the Inmetro´s standard system is based on gravimetric method because its accuracy in conversion of mass measured to volume. This method consists on measuring the mass delivered or contained in recipient whose volume need be determined. From this mass (traceable to SI) the volume is determined and the conversion from mass to volume of liquid is performed describe in the mathematical model (Equation1)based on the equation described in ISO 4787.[13]

2.1Setting up the system

The setting of system is very relevant and the assembly of system can influence in the measurement. There are several types of gravimetric setups, syringes and infusion pump. In this work the system was composed by:

- Syringe: 1 ml with resolution: 1µl
- Balance: 610g with resolution: 0.1mg
- Micro Pump with accuracy: ± 0.5% and Resolution: 0.00001 ml/min
- Glassware to receive water from syringe

To evaluating of the system flow capacity were considered important effects as evaporation and thermal and environmental influences. The evaporation effects can be a critical influence in gravimetric systems and the degree of difficulty of controlling evaporation is relevant. In the experiment was possible controlling evaporation using a plastic film on the glassware for reduces the evaporation of water. Before the measurement, the plastic film was crossed by the needle of the syringe. Other effect considered was thermal influence, once the water temperature variation can cause thermal expansion of the syringe material changing its volume. These variables may to influence the measurement results by gravimetric method. Therefore, it is advisable creating a system with temperature, pressure and humidity monitored by calibrated instruments.

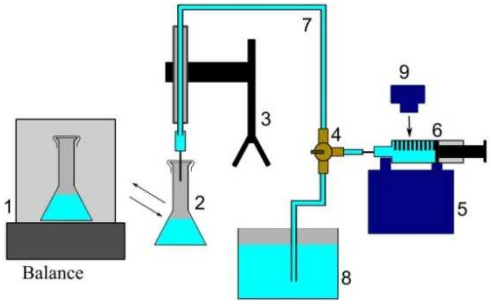


Figure 1 – System of microfluidic measurement.

2.2 Techniques and procedures **measurement**

There were performed six measurements for each flow rate, in order to assess the repeatability of the system. The reference temperature was 20ºC and the design of the apparatus is showed in figure 1. The syringe (6) was connected at the pump infuse (5) and a hose (7).Both of them was connected a glassware (2) and a reservoir with water (8). Before infusion, the initial water temperature was measured. The mass of the glassware with water is measured at the balance (1). During the transfer of water from syringe (6) to the glassware (2) the air temperature, humidity and pressure were be monitored. After infusion, the final mass of the glassware with water was measured. Finally, the water temperature was measured. After this, the conversion from mass to volume at reference temperature at 20ºC is described at mathematical model 1 and was realized reading syringe piston with camera. From the calculated volume by gravimetric method and with a study on setting up the gravimetric system including associated uncertainty components, it is possible to ensure the traceability of micro flow rate traceable to SI.

Considering the variables quoted previously, the flow capacity of system to a each volume syringe is:

Table 1. Capacity of system for each volume syringe

Volume Syringe	100 µl	1 ml	10 ml
Higher Flow rate	266.447 µl/min	2.47528 ml/min	25.5609 ml/min
Low Flow rate	0.33 µl/min	3.3 µl/min	0.0323 ml/min

In this present work, the uncertainty evaluated was used the syringe of 1 ml. However, it is possible determine the capacity of system to measurement micro flow rate, from the volume and the diameter of syringe.

So, to realize the assembly of system, others some care has been taken. The environmental influence was controlled, causing a thermal equilibrium. The system was assembled free the vibration, causing the instability and equilibrium in the measurement. To minimize of air bubble in the system, the syringe and hose were filled with deionizer water and it's without air. The fluid used in the experiment was deionized water and the water density was determined using an equation recommended by BIPM, the Tanaka Equation [13]. To minimize the measurement procedure was used an on/off valve with entrance to a water reservoir, a glassware and a syringe, in order that. After injection of the liquid in the glassware, the syringe is filled again via the water present in the reservoir, making the continuous process. The syringe capacity was 1 ml, but in each run the totalized volume was 0.8 ml to obtain a best visualization of the delivered volume of water and to avert accidental problems. In the experiment, to read a best visualization of the resolution syringe and avoid parallax error was installed a camera.

3. MATHEMATICAL MODEL

$$\Delta Ve = \frac{Mc - Mv + M_E}{(\rho_L[T_{Lt}] + \delta\rho_L[T_{Lt}] - \rho_{ar})} \cdot \left(1 - \frac{\rho_{ab}}{\rho_b}\right) \cdot (1 + \alpha_v \cdot (T_r - T_t)) + \delta R \quad (1)$$

- ΔV_e- is the volume at its reference temperature $T_{r=}\,20ºC$
- M_c - The apparent mass, after the transference of liquid
- M_{a2} -The apparent mass, before the transference of liquid
- M_E – Evaporation mass
- ρ_L- is the liquid density at temperature T_L
- T_L-Liquid Temperature
- δT_L - error due to the variation of liquid temperature in space and time
- ρ_A- is the air density during the apparent water mass measurement
- ρ_{ab}– is the air density during balance calibration
- ρ_b – is the density of the standard weight used in balance calibration
- α_V _ is the thermal expansion volumetric coefficient
- T_d - is the syringe temperature
- δR – is the reference temperature at 20ºC

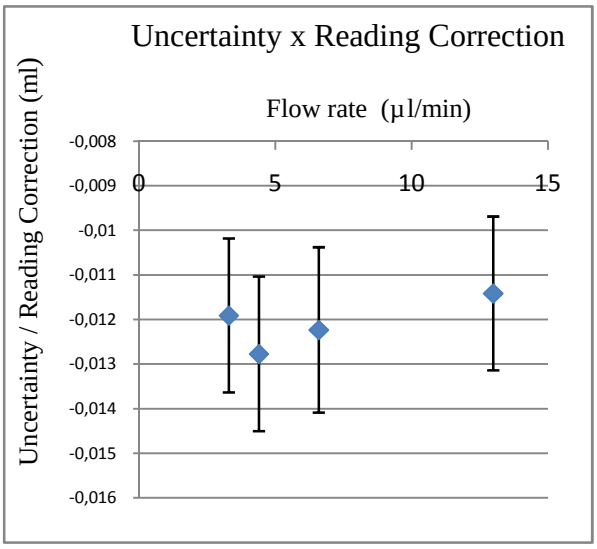
4. ANALYSES OF RESULTS

The flow rate capacity defined to syringe of 1ml are 0.013 ml/min, 6.6µl/min, 4.4 µl/min and 3.3 µl/min, as described above For each flow rate was realized six measurements and the results are present in next table. The reading correction is the difference between the calculated volume and the indicated volume at the syringe. The Relative Uncertainty and Expanded Uncertainty were calculated considering all factors of the mathematical model. [14,15]

Table 2. Results

Calculated Volume (ml)	0.7988	0.7971	0.7924	0.7958
Nominal Flow rate	0.013 ml/ min	6.6 µl/min	4.4 µl/ min	3.3 µl/ min
Reading Correction (%)	0.0312	0.0859	0.0382	0.0330
Expanded Uncertainty (ml)	0.00173	0.00185	0.00174	0.00173
Relative Uncertainty (%)	0.22	0.23	0.22	0.22

Figure 1. Uncertainty x Reading Correction



The relative contributions to the uncertainty are presented in the following table:

Table **3.**Contribution to Uncertainty

Contribution to uncertainty	Relative Contribution To 0,013 ml/min	Relative Contribution To 0,013 ml/min	Relative Contribution To 0,013 ml/min	Relative Contribution To 0,013 ml/min
Read Volume with camera	32.206	28.627	31.680	32,060
Evaporation Mass	20.108	17.874	19.780	20.016
Mass before transferred	20.108	17.874	19.780	20.016
Mass delivered	20.108	17.874	19.780	20.016
Repetition	7.101	17.394	8.556	7.484
Others	0.370	0.358	0.414	0.406

The results present in table 2 are initial results, realized to investigate the capacity in the bench of micro flow rate. The results described obtained and described above are considered satisfactory and it was obtained a value for uncertainty within expectations, however, some aspects can be more investigation, as the evaporation effects or thermal influence. Besides, the results can be improvedusing instruments with better resolution as syringe and balance.

5. CONCLUSION

The results presents in this work are considered satisfactory to first studies of bench and it could be satisfactory for using as reference in experiments with the microPIV (micro Particle Image Velocimetry).

In this article was evaluated the main sources of uncertainty. The result can will be improved with better resolution syringe and balance, and the improvement of measurement system to reading of syringe piston.

However, the system development and validation are in progress and some points still need to be more evaluated, as e.g.:

- Evaporation effects
- Research of uncertainty components
- Continue investigation to improve gravimetric configuration

To provide traceability in the proposed method and uncertainty, in the future will be realized an intercomparison. However, the results presents in this work are considered satisfactory.

7. ACKNOWLEDGEMENT

The authors are grateful to CNPq, FAPERJ, FINEP, PETROBRAS, PFRH-PETROBRAS and INMETRO for their support and sponsorship which have become possible the development of this research

7. REFERENCES

[1] Bureau International des Poids et Mesures– BIPM.; strategy 2014-2024, *consultative committee for mass and related quantities (ccm).*

[2]Hannu a. Sairanen, Marti, O. Heinonem.; 2014. *Hygrometer-based calibration method for syringe pumps providing ultra-low liquid flows.Microfluidicnanofluidic*, **16:**187-193.

[3]Haswell, S.J., 1997. *Development and operating characteristics of micro flow injection analysis systems based on eletroosmotic flow, Analyst.*;**122 (1R-10R).**

[4]Marinozzi, F., Bini, F. andCappa, P., 2005. *Calibrator for micro flow delivery systems, Rev.Sci*. Instr., 76.

[5]Nath, P., Roy, S., Conlisk, T. and Fleischman, A.J., 2005. *A system for micro/nano fluidic flow diagnostics, Biomedical Devices*, 7.

[6] Nuno Miguel Matos Pires, Tao Dong, UlrikHanke and Nils Hoivik.; 2014.*Recent Developments in Optical Detection - Technologies in Lab-on-a-Chip Devices for Biosensing Applications, Sensors*, **14, pp15458-15479**

[7] Reitz, S., Kummrow, A., Kammel, M. And Neukammer, J., 2010. *Determination of micro-litre volumes with high accuracy for flow cytometric blood cell counting, Meas. Sci. Technol.*, **21**

[8] Wolf, H., 2010. *Microflow meets metrology, meas. Sci. Technol.*,**21.**

[9]Bruss, Henrik., 2008. *Theoretical Microfluidics*.**1**

[10]Yi-Jui Liu , Juin-Yi Yang , Yung-Mau Nie, Chun-Hung Lu, Eric Dowkon Huang, Chow-Shing Shin, Patrice Baldeck, Chih-Lang Lin.; 2015. *A simple and direct reading flow meter fabricated by Two-photonpolymerization for microfluidic channel* **,** *MicrofluidNanofluid* **18:**427–431

[11]König, J., Voigt, A., Büttner, L. and Czarske, J., 2010. *Precise micro flow rate measurements by a laser Doppler velocity profile sensor with time division multiplexing, Meas. Sci. Technol.*,**21**.

[12] ISO 4787:2010 – *laboratory glassware – volumetric instruments methods for testing of capacity and for use.*

[13] M. Tanaka, G. Giard, R. Davis, A. Peuto and N. Begnell, 2001.*Recommended table for the density of water between 0ºC and 40ºC based on recent experimental reports.*

[14] Evaluation of measurement data,; 2008. *Guide to the expression of uncertainty in measurement – GUM.*

[15] International Vocabulary of Metrology.2008. *Basic and General concepts and associated terms (VIM)* **3rd edition**.