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

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Abstract. The necessity of standardizing and traceability to SI in micro scale flow has been subject of intense discuss in the metrology community. The Bureau International of Poids et Mesures (BIPM) has encouraged National Metrology Institutes to be prepared to participate, in 2024, of the first Key Comparison event planned in this area. With this aim, the present work describes a standard system based on gravimetric method for fluid delivery and respective quantification, which was developed by the National Institute of Metrology, Quality and Technology (INMETRO) in order to provide traceability for microflow rate measurement. The system performance and capability were evaluated. An important goal of the work is contribute to the establishment and advancement of researches on microfluidic issues in the Institute.

Keywords: micro flow rate; microfluidics; metrological traceability; gravimetric method

1. Introduction

Microfluidics can be applied to several fields of the technology. This science and their applications can optimize and improve processes as those found in areas such as biomedical, pharmaceutical, chemical, environmental, aerospace and others. However, to ensure reliability on quantifying fluid flow rate in micro scale is a challenging task, due to the need of controlling variables that influence in the measurement process. Nowadays, there is a gap in the metrological traceability chain for very low flow rate. Due to this gap, the Bureau International des Poids et Mesures – BIPM [1] has encouraged National Metrology Institutes to offer systems for providing metrological traceability on micro flow rate measurement up to 2024, when will run the first international key comparison event in microfluidic.

The metrological community has discussed about the microscale flow rate measurement. Several techniques and methods have been suggested by researchers, whom has characterized and calibrated microfluidic systems by using such techniques and methods [2-11].

The present work describes the new standard system for metrological traceability on liquid micro flow rate measurement which was developed in the Fluid Dynamics Division of the National Metrology Institute of Quality and Technology (INMETRO). The system capability is evaluated under different ranges of low flow rates. Since with this system implementation will be possible to study flow in micro channels with traceability to national standards, the results can be used as reference to fluid velocity measurement by applying the microPIV (micro Particle Image Velocimetry) technique.
2. Methods and Instrumentation

The measurement technique of the Inmetro’s standard system is based on gravimetric method because the good accuracy in conversion of measured mass to volume. This method consists on measuring the mass which is contained or delivered to a recipient, and the conversion from mass to volume of liquid is performed according to the mathematical model (Equation 1) based on the equation described in ISO 4787 [13].

2.1. Setting up the system

The system assembling and setting is very relevant, since are aspects that strongly influence the measurement results. There are several types of gravimetric setups, syringes and infusion pumps. 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

In the system capacity evaluation were considered important effects such as evaporation, thermal and environmental influences. The evaporation can be a critical influence in gravimetric systems and the difficulty for controlling it is high. In these experiments, a plastic film was placed on the upper edge of the glassware for reducing water evaporation. Before receiving the liquid, the plastic film was crossed by the syringe needle (see “2” on figure 1). On the thermal influence, the water temperature variation can cause thermal expansion of the syringe material and then, change its volume. Therefore, it is advisable to create and to maintain a system with temperature, pressure and humidity monitored by calibrated instruments.

![Figure 1. System for microflow rate measurement.](image)

2.2. Techniques and measurement procedures

The apparatus schematic is shown in figure 1. The syringe (6) was attached to the infusion pump (5) and connected to a hose (7). With a three way valve, through hoses the syringe pump was connected to a glassware receiver (2) and a water reservoir (8). A camera (9) was used to read the volume of fluid (according to the syringe piston position) on the syringe scale. A flow rate was set in the pump, and before infusion, the initial water temperature was measured. The reference temperature was 20°C. While transferring water from the syringe (6) to the glassware (2), the air temperature, humidity and pressure were monitored. The mass of the glassware (before and after collecting water) was measured by the balance (1) at each run. There were performed six measurements for each flow rate, in order to assess the system repeatability. Also, at each run it were registered the volume of fluid read on the
syringe scale by using the camera (figure 1). After that, the conversion from mass to volume at reference temperature 20ºC, as described at the mathematical model (equation 1) was made. So, based on the calculated volume by gravimetric method and on the study of the setting up of the gravimetric system, including associated uncertainty components analysis, it was possible to ensure the traceability of micro flow rate to SI (International System of Units).

Considering the previously quoted variables, the flowrate capacity of the system under different syringe volumes were:

| Syringe Volume | 100 µl      | 1 ml       | 10 ml      |
|----------------|-------------|------------|------------|
| Highest Flow rate | 266.447µl/min | 2.47528 ml/min | 25.5609ml/min |
| Lowest Flow rate   | 0.33µl/min  | 3.3µl/min  | 0.0323ml/min |

After identifying the flowrate ranges, it was chosen the syringe of 1ml in capacity for running tests and evaluating the uncertainties by using the gravimetric method. The syringe capacity was 1 ml, but in each run the totalized volume was 0.8 ml in order to visualize appropriately the delivered volume of water, and avoid barrel end effects. In the experiments, to improve the visualization of the syringe scale and avoid parallax error in reading it, a camera was installed as shown on figure 1.

During the system assembly additional care was taken, for instance, environmental influence was controlled (causing a thermal equilibrium), the system was insulated of vibration (for stability and equilibrium during the measurements), air entrance in the system was prevented and air bubble trapping was minimized, etc. The syringe and hose were filled with deionized water very carefully before starting the first run. The basic sequence for filling the hydraulic system with water and delivering this fluid was: after delivering liquid to the glassware, the syringe was filled again by collecting water from the reservoir. The water density was determined using an equation recommended by BIPM, the Tanaka Equation [13].

3. Mathematical model

The following equation was used for volume of liquid determination:

\[
\Delta V_e = \frac{M_c - M_v + M_E}{\rho_L[T_L] + \delta\rho_L[T_L] - \rho_{ar}} \cdot \left(1 - \frac{\rho_{ab}}{\rho_b}\right) \cdot \left(1 + \alpha_v \cdot (T_v - T_v)\right) + \Delta R \tag{1}\]

Where:
- \(\Delta V_e\) is the volume of liquid at the reference temperature \((T_{ref} = 20^\circ C)\)
- \(M_c\) is the apparent mass (glassware), after receiving the liquid
- \(M_v\) is the apparent mass (glassware), before receiving the liquid
- \(M_E\) is the evaporated mass
- \(\rho_L\) is the liquid density at temperature \(T_L\)
- \(T_L\) is the liquid temperature
- \(\delta T_L\) is the error due to the liquid temperature variation in space and time
- \(\rho_{ar}\) is the air density during the apparent water mass measurement
- \(\rho_{ab}\) is the air density during balance calibration
- \(\rho_b\) is the density of the standard weight used in balance calibration
- \(\alpha_v\) is the volumetric thermal expansion coefficient
- $T_d$ - is the syringe(barrel) temperature
- $\delta R$ - is the error due to repeatability of volume measurement

4. Analysis of results
Table 2 shows the results for different flow rate levels (mean of six measurement at each level). The reading correction is the difference between the calculated volume by equation 1 and the indicated volume on the syringe scale. The relative and expanded uncertainties were calculated considering all factors of the mathematical model [14, 15].

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

The relative contributions to the uncertainty are presented in table 3.

| Contribution to uncertainty | Relative Contribution To 0.013 ml/min | Relative Contribution To 6.6 \(\mu\)l/min | Relative Contribution To 4.4 \(\mu\)l/min | Relative Contribution To 3.3 \(\mu\)l/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                                    |

In table 3 can be seen that the read volume on the barrel scale represents the major contribution for uncertainties at all tested flow rates. While at the flow rate 6.6 \(\mu\)l/min the contribution due to the repetition is in the same range of that due to each mass evaluated, for other experimented flow rate
levels there is not this tendency. More investigation has to be done in order to explore in details this aspect.

5. Conclusion
In this work, a system developed for providing metrological traceability in microflowrate measurement was evaluated.

The main sources for uncertainty of the bench were identified. It was found that the system performance could be improved if a syringe and a balance with better resolution were used, and also the way of reading the syringe scale was refined. However, as first studies of the bench, the results were considered satisfactory, and this system could be used as reference in experiments using the microPIV (micro Particle Image Velocity) technique, for example. The system development and validation for the bench improvement are in progress, and in this way, some points will be explored in more details, as e.g.:

- Evaporation effects
- Uncertainty components
- Aspects of the gravimetric configuration

The National Institute of Metrology, Quality and Technology (Inmetro) aims to improve this system and, in the future, participate of intercomparison.

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