



Development of an experimental setup for micro flow measurement using the front tracking method

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ABSTRACT

This work has the main objective to develop a method for microflow measurement using the front tracking principle under the project MeDD II – Metrology for Drug Delivery.

The experimental setup uses a high-resolution camera and an image processing software to track the distance travelled by the meniscus of a liquid in a capillary tube and calculate the flow rate. Experimental tests using a flow generator were carried out for different flow rates using two different cameras.

To validate the developed front tracking method, an internal comparison was made with the gravimetric method in LVC. The results obtained by this comparison were consistent. This developed method can be used to calibrate flow devices down to 1 $\mu\text{L/h}$ with an uncertainty value of 7% ($k = 2$).

1. Introduction

Nowadays, several industries work with devices capable of generating micro and nanoflow rates (ranging from 1000 $\mu\text{L/h}$ to 0.1 $\mu\text{L/h}$), e. g., precision syringe pumps and flow meters. The primary method for flow measurement is the gravimetric method, and it is used by most of the National Metrology Institutes for measuring and calibrating low flow rate devices [1]. Since there is a need to increase the capability of the laboratories to measure lower flow rates, there is active ongoing research is being carried out on methods involving different principles, such as optical methods.

In this work, an experimental setup for flow measurement using the front tracking principle was developed under the MEDDII project [2] in the Volume and Flow Laboratory (LVC) of the Portuguese Institute of Quality (IPQ) through a partnership between IPQ and the Department of Mechanical and Industrial Engineering (DEMI) of The New University of Lisbon (FCT/UNL). The MEDDII – Metrology for Drug Delivery is a project from EMPIR (European Metrology Programme for Innovation and Research) and one of its objectives is to develop new traceable methods to measure flow rates, from 0.3 $\mu\text{L/h}$ to 6 $\mu\text{L/min}$, using a Newtonian liquid, with an uncertainty of 1% (for steady flow rates) and 2% (for fast-changing flow rates).

The front tracking method for flow measurements is an optical method that consists of tracking the position of the meniscus of a liquid (liquid/air interface) inside a capillary tube over time. Knowing the displacement of the meniscus over time and the cross-section area of the capillary, it is possible to calculate the flow rate. A high-resolution camera connected to a computer was used to track the meniscus, together with an image processing software that identifies the meniscus and calculates its position over time. The software was developed in-house using the programming language Python and the open-source image processing library OpenCV.

The algorithm developed captures images from the camera, then

applies several segmentation techniques to identify the meniscus, determine its position, and calculate the average flow for a given time interval.

To validate the method developed, an internal comparison was performed with the LVC-IPQ gravimetric method [3].

2. Experimental setup

The experimental setup based on the front tracking method for flow measurements involved the use of the following components: a Chemyx Nexus 3000 syringe pump (flow generator), a glass syringe, a connection line, a USB camera, a capillary tube, a translucent paper, and a LED light.

The original setup used a lower resolution USB camera and a connection line made of Teflon, showed in Fig. 1. Nevertheless, improvements were identified, and a second setup was built, involving the use of a higher resolution camera and a connection line made of stainless steel, showed in Fig. 2.

As previously stated, the flow was generated by a high-performance syringe pump Chemyx Nexus 3000, designed to generate microflows down to 1.56 $\mu\text{L/min}$ (equivalent to approximately 0.094 nL/h). Glass syringes of 5 mL and 1 mL were used with the Nexus pump.

The syringe was then connected to a transparent capillary by a connection line. A high-resolution camera was manually positioned with its axis perpendicular to the capillary, with the field of view capturing a section of the capillary where the liquid meniscus was moving. By knowing the distance travelled by the meniscus over time, and the internal diameter of the capillary, it was possible to calculate the flow rate of the fluid and its associated uncertainty.

A USB camera connected to a computer was used to capture the movement of the meniscus. For the first setup, a microscope USB camera with 1.3 Mpx of resolution (Levenhuk DTX 50) was used, allowing the capture of images with a maximum size of 640x480. To improve the accuracy and lower the uncertainty in the lower flow rates, a high-

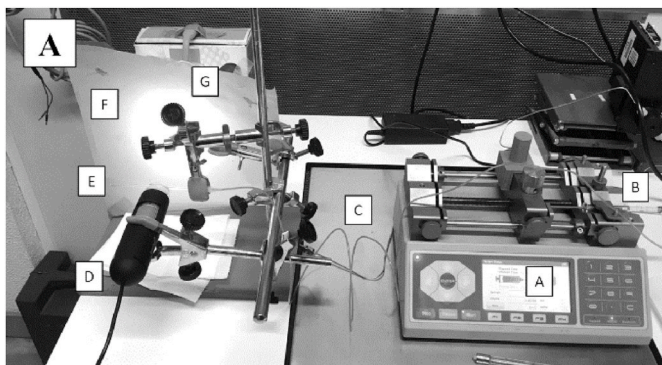


Fig. 1. Figure of the experimental setup, where A is the Nexus pump, B is the glass syringe, C is the connection line, D is the USB camera, E is the capillary tube, F is the translucent paper and G is the LED light.

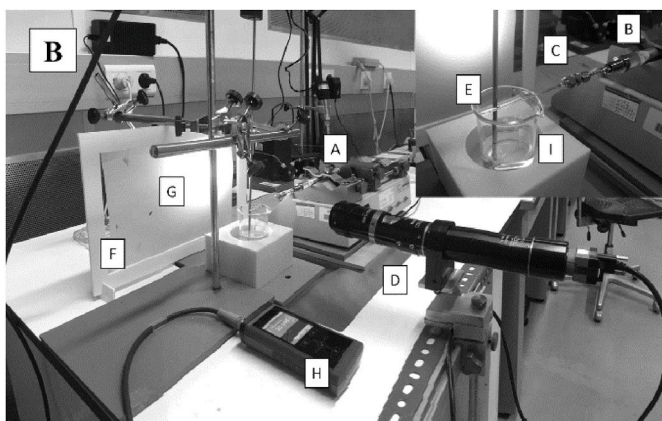


Fig. 2. Figure of the experimental setup, where A is the Nexus pump, B is the glass syringe, C is the connection line, D is the USB camera, E is the capillary tube, F is the translucent paper and G is the LED light; H is the thermometer, and I is the water reservoir.

resolution camera with 12 Mpx of resolution (Alvium 1800 U-1240) and a telecentric zoom lens (Qioptic Optem 7:1 zoom) were acquired. The camera was then mounted on an aluminum support system that allowed position adjustments in the three axes.

Moreover, in the first experimental setup, a translucent plastic (polypropylene) capillary and a Teflon connection line were used. The external diameter of 2.3 mm ($U = 0.03$ mm) was measured with a calibrated caliper while the inner diameter of 1.61 mm ($U = 0.003$ mm) was measured by an in-house gravimetric method. The higher resolution camera setup allowed for the reduction of the capillary diameter. So that, for the second experimental setup, a glass capillary was used, with an external diameter of 1.6 mm ($U = 0.03$ mm) and an inner diameter of 1.15 mm ($U = 0.005$ mm), also measured by an in-house gravimetric method. To improve the stability of the setup, the Teflon connection line was also replaced by a stainless-steel capillary tube (1/16 inch) with a smaller length.

A background illumination was chosen, allowing to decrease the reflection caused by ambient light, and obtain a good contrast between the background and the liquid meniscus. This was achieved by positioning a LED facing the camera. To smooth the light intensity, a translucent paper was placed between the capillary and the LED.

In the second setup, a thermometer was placed near the capillary tube, see Fig. 2B, submerged in the water, to monitor the temperature of the water inside the capillary since the laboratory conditions ($T = 20 \pm 3$ °C, humidity $>50\%$) and especially the light source could influence this value.

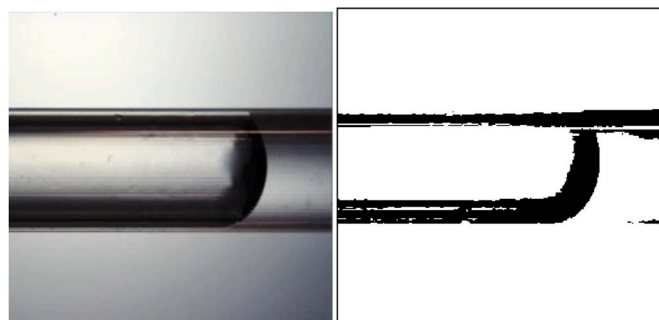


Fig. 3. a) Image before threshold; b) image after threshold.

3. Image processing software principles

The image processing software developed for applying the front tracking method had three main stages: scale definition, image segmentation, and determination of the meniscus position.

3.1. Scale definition

This step aims to establish a relation between the pixels of an image and the real dimensions of the physical world. The scale was defined by using the external diameter of the capillary as a reference. The external diameter of the capillary tube was measured with a calibrated calliper ($U = 0.03$ mm) on several sections over its length to determine an average value. This measurement gives traceability to the standard unity of length.

In the software developed during this research, the external diameter is manually selected using the *selectROI* function of the OpenCV library. This function returns the coordinates in pixels of the region of interest selected by the user. Knowing the real dimensions of the external diameter of the tube and the corresponding dimension in pixels permits the determination of a scale value. To validate this technique, a repeatability test was made, having been achieved a repeatability value of 8.6×10^{-6} mm/px.

3.2. Image segmentation

Image segmentation consists of subdividing an image into different regions to simplify and facilitate image analysis. In this work, the Thresholding technique was used [4].

The Thresholding technique consists of transforming a grayscale

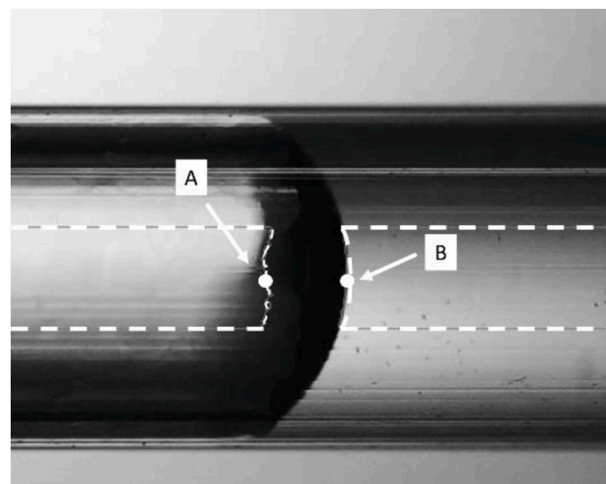


Fig. 4. Two points identified by *findContours* function library, where B is the reference point.

image into a binary image, facilitating the identification of contours. This technique was used to isolate the meniscus base, as shown in Fig. 3.

With the Alvium 1800 camera, the images obtained were larger (4024x3036) and made the algorithm slower when processing each image. To reduce the area for contour identification and meniscus position determination, a simplified solution was applied, cropping the image to the format of 1/30, in other words, a horizontal strip with 4024 px of width and a height of 100 px, containing the area where the central meniscus point is going to move, allowing time intervals between measurements to be reduced down to 0.5 s.

3.3. Determination of meniscus position

The contours are determined using the *findContours* function from the OpenCV library. This function returns the coordinates of the pixels on the contour line (dashed line in Fig. 4).

To determine the position of the meniscus over time, it was assumed as reference the central meniscus point that is the position of the point that is in the axis of the capillary and coincident with the meniscus (B in Fig. 3). The horizontal coordinate of this point is determined in pixels and converted to millimeters using the scale value.

4. Theoretical model and calculation of uncertainty

The instantaneous flow (Q) [5] is given by the equation:

$$Q = v \times A \quad (1)$$

where v is the velocity of the fluid and A is the internal cross-section area of the capillary (πr^2), assumed to have a circular section in all its range.

Velocity is obtained by dividing the displacement of the meniscus (d) through a time interval (Δt).

$$v = \frac{dx}{dt} = \frac{x_2 - x_1}{\Delta t} \quad (2)$$

The displacement of the meniscus is calculated between frames through the positions determined by the algorithm explained in 3.3. The time interval is defined by the user. The capillary section radius (r) is critical for this method; therefore, it was used an in-house method to measure the inside diameter of the capillary tube using the gravimetric approach. This method relies on determining the mass of the liquid inside a specific section of the capillary and converting this value to volume [1]. The length of the chosen section, d , is measured using a calliper, and the inner radius of the capillary, r , can be determined using equation (3).

$$r = \sqrt{\frac{V}{d \times \pi}} \quad (3)$$

For the presented setup, the instantaneous flow can be calculated through the equation (4).

$$Q = \frac{x_2 - x_1}{\Delta t} \times \pi \times r^2 \quad (4)$$

Since the liquid moves inside of a tube, the expansion of the capillary material due to temperature changes must be considered. The flow at 20 °C is then calculated using the equation (5).

$$Q = \frac{x_2 - x_1}{\Delta t} \times \pi \times r^2 \times [1 - \gamma(T - 20)] \quad (5)$$

where (γ) is the capillary material coefficient of expansion and (T) is calibration liquid temperature (°C).

The average flow is the mean value of the sum of the calculated instant flows for the duration of the test.

The main sources of uncertainty considered were the following:

- Meniscus displacement, $u_{\Delta x}$;
- Time interval between frames, $u_{\Delta t}$;

Table 1

Results FR Levenhuk – 1 mL syringe.

Measured flow (μL/h)	Reference Flow (μL/h)	Uncertainty (μL/h)	Error (%)	Uncertainty (%)
1	1.2	0.5	-16.7	41.7
10	11.1	1.1	-9.9	9.9
100	103.6	2.8	-3.5	2.7
500	520	13	-3.8	2.6
1000	1033	27	-3.2	2.6

Table 2

Results FR Alvium – 1 mL syringe.

Measured flow (μL/h)	Reference Flow (μL/h)	Uncertainty (μL/h)	Error (%)	Uncertainty (%)
1	0.99	0.07	0.9	6.9
10	9.97	0.30	0.3	3.0
100	99.6	2.1	0.4	2.1
500	499.8	6.8	0.0	1.4
1000	997	14	0.3	1.5

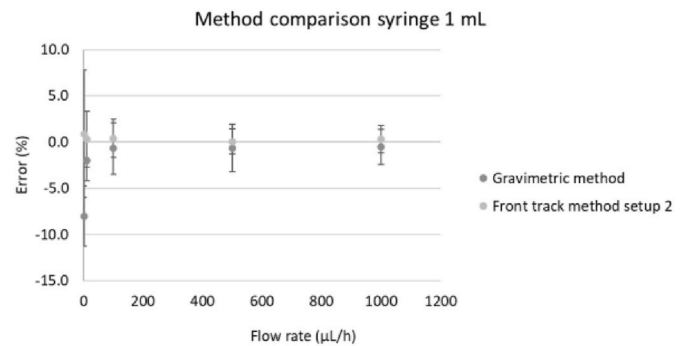


Fig. 5. Results of Nexus calibration.

- Capillary radius, u_{rg} ;
- Repeatability, u_{rep} ;
- Stability, u_{sta} ;
- Material expansion, u_{γ} ;
- Temperature, u_T ;

5. Setup implementation tests

For testing the method, five different flow rates were considered: 1000 μL/h, 500 μL/h, 100 μL/h, 10 μL/h, and 1 μL/h. Tests were performed in the Volume and Flow Laboratory of IPQ in controlled conditions (temperature = (20 ± 3) °C and humidity >50%). One glass syringe was used, with a volume of 1 mL. Tests were made with the two cameras to compare their performances, the Levenhuk DTX50 (Table 1) and the Alvium 1800 U-1240 (Table 2). Before any measurements, the cameras were aligned with the capillary, ensuring that the meniscus of the liquid was in their field of view. Ultra-pure water was used as a calibration liquid, with a conductivity of 0.05 μS/cm.

From the lower flow rates: 10 μL/h and 1 μL/h, with the second setup, it was possible to achieve much better results, with the uncertainty value for 1 μL/h below 10%.

6. Internal validation results

A Nexus 3000 pump was calibrated using the gravimetric method and the front track method using setup 2. The results presented in Fig. 5.

Also, a Sensirion meter was calibrated by the front track method and the interferometric method described in Ref. [6]; the results are in Fig. 6.

From Figs. 5 and 6, the results from all methods are consistent

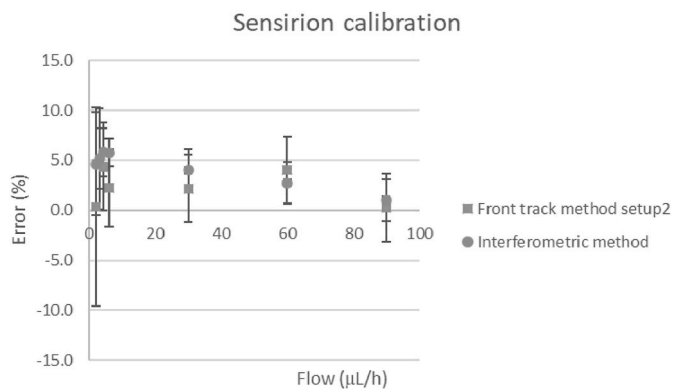


Fig. 6. Results of Sensirion meter calibration.

(within the mutual uncertainty) with each other. Also, it can be seen that the uncertainty values of the front tracking method are smaller when compared with the gravimetric method and very similar to the interferometer method.

7. Conclusions

A method for measuring microflow rates lower than 1 mL/h using the front tracking method was developed by the Volume and Flow Laboratory of the Portuguese Institute of Quality in cooperation with FCT/UNL - UNIDEMI.

Two cameras were tested, and improvements were made in the experimental setup to improve the accuracy and uncertainty of the front tracking method.

A higher resolution camera and zoom lens, together with a stainless-steel capillary connection line, allowed a significant improvement in the measurement accuracy and uncertainty value of the front tracking method developed. The uncertainty value associated with the setup stability becomes the second largest uncertainty component, behind the repeatability value.

From the internal comparison made with the gravimetric method, it was possible to conclude that this optical method is consistent with the results obtain by the gravimetric method (the values are with the mutual

uncertainty). This method can be used to calibrate flow devices down to 1 µL/h with an uncertainty value of 7% ($k = 2$). This value can be reduced if some improvements on the setup are applied, mainly the use of a capillary of smaller dimensions.

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