Measuring inner diameter of capillary tubes

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Abstract. The dimensional characterization of capillary tubes is a recurring issue in microfluidic applications, since they are often used to connect microfluidic devices to injection and monitoring systems. In this work three experimental methods were applied and discussed for measuring the inner diameter of thin tubes, allowing the comparison and determination of the most accurate procedure, taking into account the application and involved measurement uncertainty. The methods analysed are based on (i) sizing by stereo microscope software, (ii) image analysis (binarization) and (iii) internal fluid mass. The results show that the three methods applied can provide accurate values, but the best choice in terms of simplicity and efficiency is that one based on image analysis.

Keywords: Inner diameter. Capillary tube. Image analysis. Internal fluid mass. Measurement uncertainty.

1. Introduction
Capillary tubes are small diameter inner tubes that are commonly used in microfluidic applications, especially when connecting microfluidic devices to injection and monitoring systems.

In this context, the determination of the inner diameter is essential for the correct prediction of the pressure drop along the tubes, allowing the indirect measurement of important transport properties (such as permeability). Also, the uncertainty of the measured diameter has to be quantified to allow the estimation of uncertainties associated to the microchannel system of interest.

Due to manufacturing imperfections the inner diameter may not be constant along its length. It is thus necessary to assess the variability of the diameter. Furthermore, an appropriate measurement system has to be selected, aiming at low uncertainty values.

This article addresses three methods for solving the aforementioned problem, namely: (i) sizing by stereo microscope software; (ii) measurement by image analysis using the free software ImageJ [1]; (iii) weighing of the fluid mass.

2. Materials and Methods
2.1. Scaling by Stereo Microscope

Thirteen (13) samples of a polymeric tube (Tygon) were cut with a new stylet to minimize any damage to its internal structure. A stereo microscope Olympus Model SZX10 featuring a DP26 5.0 MP digital camera with 2x objective lens and 6.3x focus magnification, enabled high-resolution images of the tube cross-section (Figure 1a).

After finding the approximate position of the center point of each tube by manually adjusting a circumference to the hole, assisted by the microscope software (Stream Essentials package, version 5.10), four lines were drawn, as illustrated in Figure 1b. The average inner diameter of tube sample \( i \) was calculated from the lengths of the lines 1 to 4: 
\[
\bar{d}_i = \frac{d_1 + d_2 + d_3 + d_4}{4},
\]
where \( d_1, d_2, d_3 \) and \( d_4 \) represent the diameters obtained from the lines. Such dimensions were measured by pixel counting.

The size of each pixel was calibrated using a known dimension provided by a calibration slide (1 DIV = 0.1 mm) with an expanded calibration uncertainty of 0.9 \( \mu \)m. Therefore, the average inner diameter, \( \bar{d}_{SMS} \), obtained by this method is given by
\[
\bar{d}_{SMS} = \frac{\sum_{i=1}^{13} d_i}{13},
\] (1)

![Figure 1](image1.png)

Figure 1 – In (a) it is displayed the original capillary tube cross-section photo and in (b) the same photo with the measurements.

2.2. Measurement by image analysis

The images taken as described in section 2.1 were analyzed using the free software ImageJ, version IJ 1.46r. Images were converted to 8-bit grayscale (Figure 2a). Afterward, an ellipse shape (Oval selections tool) was drawn and adjusted manually to that area that forms the hole. That step is crucial to get the best as possible fitting between the hole and ellipse shapes. Then, the ellipse was filled with the white color and the outside region was cleared (using the Fill and Clear Outside tools), what creates a digital binary image formed by black and white pixels.

In this case, the blank circle observed in the image represents the inner cross-sectional area. By counting the white pixels it was possible to calculate the internal diameter associated with the blank area
(Figure 2b) using the tools available in the software. From the value of the blank area of each sample, $A_i$, the internal diameter by image analysis, $d_{IA}$, was obtained from the following relation:

$$d_{IA} = \sqrt{\frac{4\bar{A}}{\pi}}, \quad (2)$$

where $\bar{A}$ is the arithmetic average of the internal blank areas.

2.3. Measurement of internal fluid masses
A 100.0±0.6 mm long tube was weighed on a digital balance SHIMADZU model ATX224 with a resolution of 0.1 mg and expanded calibration uncertainty of 0.1 mg up to the mass of 10 g. For this method 15 mass measurements were made with both the liquid-free and glycerin-filled tubes. The tube was cleaned with ultrapure water and was then blown dry with compressed air so that there was no trace of water particles. Glycerin was filled into the tube with glass syringes by controlling the inlet flow rate for getting a uniform filling without bubbles.

The difference between the filled and empty tube masses corresponds to the glycerin mass. Taking into account the laboratory temperature of 25 °C and the glycerin density of 1258.0±0.1 kg/m³ [2,3,4], the inner diameter $d_{IFM}$ of the tube is calculated according to equation 3

$$d_{IFM} = \sqrt{\frac{4\bar{m}}{\pi \rho l}}, \quad (3)$$

in which the average glycerin mass inside the tube is given by $\bar{m} = (2.400 \pm 0.014) \times 10^{-5}$ kg, $l$ is the tube length and $\rho$ is the glycerin density.

2.4. Estimation of measurement uncertainty
The last two methods are indirect measurement procedures of obtaining the internal diameter of the capillary tube. Thus, to obtain the combined standard uncertainty of both methods the following relationships were used [5]:

$$u_{d_{IA}}^2 = \left[ \left( \frac{\partial d_{IA}}{\partial A} \right) u_A \right]^2, \quad (4)$$
\[ u_{dIFM}^2 = \left[ \left( \frac{\partial d_{IFM}}{\partial \rho} \right) u_\rho \right]^2 + \left[ \left( \frac{\partial d_{IFM}}{\partial l} \right) u_l \right]^2 + \left[ \left( \frac{\partial d_{IFM}}{\partial m} \right) u_m \right]^2, \]  

(5)

where \( u_\rho, u_l, u_m \) and \( u_m \) represents the standard uncertainties associated with area, fluid density, tube length and internal mass measured in the balance, respectively. The standard uncertainty of the ruler was estimated from its resolution \((R = 1 \, \text{mm})\) according to Equation 6, assuming a rectangular probability distribution [5]:

\[ u_l = \frac{R}{2\sqrt{3}}. \]  

(6)

The standard uncertainty of the mass measurement was calculated by the combination of the standard deviation of the measured masses, \( u_\sigma \), the calibration uncertainty of the digital balance, \( u_{calB} \), and the uncertainty due to the limited scale resolution, \( u_{res} \), resulting in the following expression:

\[ u_m = \sqrt{u_\sigma^2 + u_{calB}^2 + u_{res}^2}. \]  

(7)

For the methods of the sections 2.1 and 2.2, the standard uncertainties \( u_{dSMS} \) and \( u_\rho \) were determined from the combination of the standard uncertainty of the mean, \( u_\sigma_i \), and the calibration uncertainty of the 0.1 mm ruler scale, \( u_{calR} \). In a general sense, the combined uncertainty of the method \( i \) is given by

\[ u_i = \sqrt{u_{\sigma_i}^2 + u_{calR}^2}, \]  

(8)

in which \( u_{\sigma_i} = \frac{u_\sigma_i}{\sqrt{n}} \) and \( u_\sigma_i \) is the calculated sample standard deviation of the \( n \) samples considered for the method \( i \). By proceeding in this way the expanded uncertainties of the three methods used in this paper could be directly compared. The expanded uncertainty, \( U \), for each method was calculated using

\[ U = u_c t, \]  

(9)

in which \( u_c \) is the combined standard uncertainty and \( t \) is the Student distribution coefficient obtained according to the number of degrees of freedom \((n - 1)\) of each method for a coverage probability of 95.45%.

3. Discussions and conclusions

As the method based on sizing by means of the microscope software consists on tracing some dimension lines to obtain the diameter value, the human error is an uncertainty factor, because the limiting circumference adjusted to the inner perimeter is defined by the operator. Besides that, the correct determination of the edges of the hole is difficult due to the low contrast of the polymer-air interface region, as illustrated in Figure 3. An automated numerical method for edge detection based on subpixel resolution [6] could provide better results.
Figure 3 – Averaged grayscale profile through a part of the polymer-air interface (from figure 1). Note that the correct position of the edge is difficult to determine since the interfacial area is thick (~35 pixels).

The method based on image analysis consists on the determination of the blank area by pixel counting after a manual binarization process. It was observed that such method is efficient, practical and easy to use because it requires less time, but it still suffers some influence of the operator. Additionally, it is important to mention that the performance of the binarization method also depends on the contrast of the material-air interface, being thus related to factors such as camera resolution, magnification, illumination and overall quality of the image. Generally, those pre-requirements are easy to achieve in laboratory conditions.

For the method based on the measurement of the internal fluid mass, uncertainty is related to the accuracy of the measurement of the tube length and mass, besides being limited to the uncertainty of the density value of the fluid.

The results obtained from the three methods are presented in Table 1.

| Method                        | Diameter [µm] | Standard uncertainty [µm] | t     | Expanded uncertainty [µm] |
|-------------------------------|---------------|--------------------------|-------|---------------------------|
| (i) Stereo microscope         | 489.1         | 0.86                     | 2.13  | 1.8                       |
| (ii) Image analysis           | 492.8         | 0.89                     | 2.13  | 1.9                       |
| (iii) Internal fluid mass     | 492.5         | 1.60                     | 2.00  | 3.2                       |

Methods (i) and (ii) resulted in similar uncertainty values. However, the diameter obtained by method (i) is slightly smaller than the diameter of method (ii). The result obtained by method (iii) is comparable to the results obtained by the other two methods. It must be pointed out that the results presented in this work were obtained by using well-calibrated instruments for which the calibration uncertainties are known and the presence of systematic errors can be neglected. That procedure guarantees the reliability and reproducibility of the results.

It is also interesting to note that the inner tube diameter provided by the manufacturer is 510.0 µm, resulting in an overestimated value of approximately 4% compared to the results obtained in this work.

The results presented in this paper will now be used to estimate the uncertainty of the experimental determination of transport properties of microfluidic systems.
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