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Presentation of the METAS pipe viscometer

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Abstract

Calibration of flow devices is important in several areas of pharmaceutical, flow chemistry and HPLC applications where dosage of process liquids or accurate measurement of the flow rate are important. The process-oriented liquid itself might influence the performance of the flow device. Therefore, the calibration of the flow meter or microfluidic device with the process-oriented liquid is important and the simultaneous determination of the dynamic viscosity under flow conditions is a valuable information for viscosity dependent flow metering methods or other process parameters. To offer the simultaneous calibration of the dynamic viscosity of the process-oriented liquid at the corresponding flowrate, METAS has built a pipe viscometer for the traceable in-line measurement of the dynamic viscosity in the current flow facilities for low flow rates from 1 µL/min to 150 mL/min and pressure drops up to 10 bar. To guarantee the tracability, the most challenging part remain the determination of the inner diameter of the micro tube. This can be determined by measuring the pressure drop as a function of flow rate and applying the law of Hagen-Poiseuille with a well known liquid (water) or perform the measurements with the µ-CT at METAS, which determines the inner diameter by x-ray diffraction. The setup of the facility, the uncertainty calculation for the in-line measurement of the dynamic viscosity and the validation measurements are discussed in this paper.

1. Introduction

Calibration of flow devices is important in several areas of pharmaceutical, flow chemistry and microfluidic applications where dosage of process liquids or accurate measurement of the flow rate are important. The process-oriented liquid itself might influence the performance of the flow device. Therefore, the calibration of the flow meter or microfluidic device with the process-oriented liquid is important and the simultaneous determination of the dynamic viscosity under flow conditions is a valuable information for viscosity dependent flow metering methods or other process parameters. METAS has built a pipe viscometer for the traceable in-line measurement of the dynamic viscosity in the current flow facilities for low flow rates from 1 µL/min to 150 mL/min and pressure drops up to 10 bar. The traceability of the pipe viscometer includes the traceability of the flow rate, the pressure measurement, the temperature measurement and the geometrical dimensions of the micro tube. Therefore, the length and the inner diameter of the micro tube have to be determined in a first step to guarantee traceability. The length of the soda glass micro tube is calibrated by the length metrology laboratory. The calibration of the inner diameter of the soda glass micro tube has been performed with the µ-CT at METAS, which determines the inner diameter by x-ray diffraction. Additionally, the average inner diameter has been also calibrated with the flow calibration method, which consists of the measurements of the pressure drop as a function of the flow rate with the liquid water. The dynamic viscosity of water can be calculated with the measured temperature and pressure of the water [1]. Applying the law of Hagen-Poiseuille allows then to determine the average inner diameter of the micro tube. These two methods for the calibration of the inner diameter of the micro tube and the METAS pipe viscometer are described in the paper. A description of the measurement uncertainty and its validation with calibrations of reference liquids for dynamic viscosity are also presented.

2. Experimental setup of pipe viscometer

The Milliflow facility at METAS [2,3] has been modified to include a section with a pipe viscometer as can be seen in Figure 1. The pipe viscometer consists of a micro tube with temperature and pressure sensors upstream and downstream of the micro tube. The tubing, the internal volume of the pressure sensor and the connectors to the micro tube have much larger inner diameters (more than 2 mm) than the diameter of the micro tube being approximately 0.13 mm. This guarantees that the recorded pressure drop between the two pressure sensors is mainly due to the pressure drop over the micro tube for pressure drops larger than 0.5 bar.

The flow rate is generated by the piston prover, where a high precision linear stage with a linear measuring system moves the plunger. The position of the plunger and the inner diameter of the piston are calibrated by the length metrology lab. Therefore, the speed, the cross-section and thus the volume flow rate are traceable to

length and time. The pressure sensors are also calibrated over the measurement range.

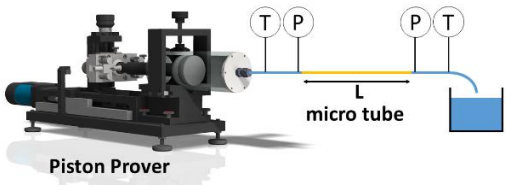

Figure 1: Schematic setup of the pipe viscometer. Temperature and pressure sensors upstream and downstream of the micro tube.

Knowing the calibrated length and the inner diameter of the micro tube, the measured pressure drop as a function of the flow rate allows determining the dynamic viscosity according to the Hagen-Poisseuille equation (1):

$$\eta = \frac{\Delta P \cdot \pi \cdot r^4}{8 \cdot Q \cdot L} \qquad (1)$$

, where η is the dynamic viscosity, ΔP is the pressure drop over the micro tube, Q is the volumetric flow rate, L is the length of the micro tube and r is the inner radius of the micro tube.

Obviously, the dimensions of the micro tubes have to be calibrated first to guarantee the traceability. The lengths are easily accessible for calibration by the length metrology, but the inner diameter is somewhat more challenging.

3. Determination of the inner diameter

The calibration of the inner diameter of the micro tubes can either be obtained by means of a µ-CT system [4] or by means of the flow calibration in the laminar regime. For the flow calibration a reference liquid with known dynamic viscosity is used to determine the inner diameter of the micro tube in the laminar flow regime according to equation (2).

$$r = \sqrt[4]{\frac{8 \cdot \eta \cdot Q \cdot L}{\pi \cdot \Delta P}} \qquad (2)$$

, where η is the dynamic viscosity, ΔP is the pressure drop over the micro tube, Q is the volumetric flow rate, L is the length of the micro tube and r is the inner radius of the micro tube.

3.1 Flow calibration method

For the flow calibration method pure water is chosen as reference liquid as the dynamic viscosity and the density formulas as a function of temperature are well established. These properties of the pure water are calculated according to the equations from the NIST database [1] with the average of the measured temperatures in the tubing upstream and downstream the micro tube. Flow rate measurements are performed in the range to obtain pressure drops in the range from 0.5 bar to 8.0 bar. The linear fit with forced zero intercept of the pressure drop data as a function of flow rate gives the slope (see Figure 2), which allows the calculation of the inner diameter of the micro tube with the knowledge of the length of the micro tube and the dynamic viscosity of pure water (see Equation (2)).

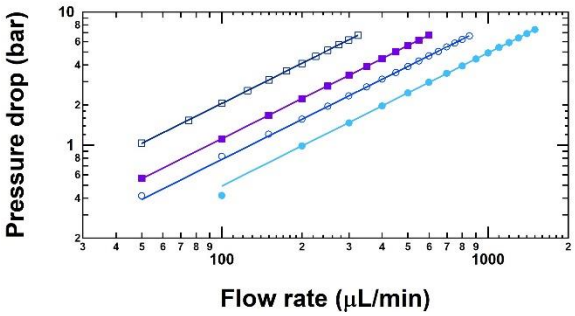

Figure 2: Pressure drop versus flow rate for the laminar flow through the glass micro tube of water (light blue solid circles), reference liquid 2AW (blue open circles), reference liquid 2BW (violet solid squares) and reference liquid 5AW (dark blue open squares) with the corresponding linear fit with forced zero intercept.

The following values of the dynamic viscosity and density of pure water listed in Table 1 were taken from the NIST database [1], which is based on the IAPWS R12-08 [5].

Table 1: Density and dynamic viscosity of water according to the NIST database [1] at a temperature of 21.3 °C and several values of absolute pressure.

Reference liquid	Absolute pressure (bar)	Dynamic viscosity (mPa·s)	Density (kg/m³)
H_2O	1.0	0.97052	997.93
H_2O	2.0	0.97048	997.97
H_2O	4.0	0.97041	998.07
H_2O	8.0	0.97026	998.25

Even that these values of the density and the dynamic viscosity show a slight dependence on pressure, the values at 1 bar absolute pressure are used. 1 bar absolute pressure corresponds roughly to the ambient pressure of the laboratory conditions. The change rate of the dynamic viscosity as a function of the pressure is $-4 \cdot 10^{-5}$ mPa*s/bar. As the measurements are performed for pressure drops in the range from 0.5 bar to 8.0 bar the maximum average pressure in the liquid over the full capillary is half of the maximum pressure drop being 4.0 bar. This implies a maximum change in the dynamic viscosity of $1.6 \cdot 10^{-4}$ mPa·s, which corresponds to 0.016 % of the dynamic viscosity at ambient conditions. This contribution is negligible as we assume the uncertainty contribution from the calculation of the dynamic viscosity of pure water with the measured temperature as 0.5 % (see Table 2).

3.2 μ-CT method

The μ-CT method has been used for the calibration of the inner diameter of the soda glass micro tube in order to compare it to the flow calibration method and validate the latter method.

μ-CT measurements were performed on the high-accuracy METAS-CT system [4,6]. Grey value volumes were analysed in VG Studio MAX (Volume Graphics) as follows: Gradient based surface determination and subsequent least-squares fitting of cylindrical primitives to determine the mean diameter for the soda glass micro tube. Measurement uncertainties were estimated using a hybrid approach, which combines reference measurements and a CT simulation of the actual workpiece [4].

Figure 3 shows the cross section of the soda glass micro tube with a nominal inner diameter of 0.130 mm determined by means of the μ-CT method.

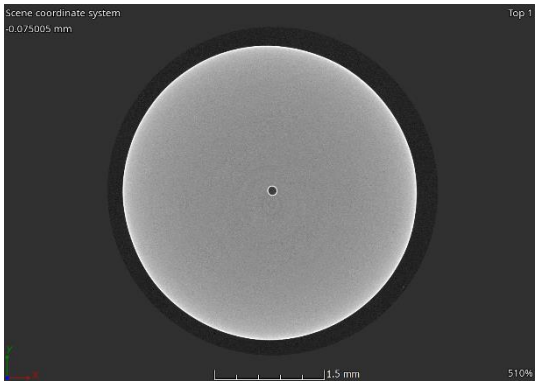

Figure 3: Cross section of the glass micro tube determined by means of the μ-CT.

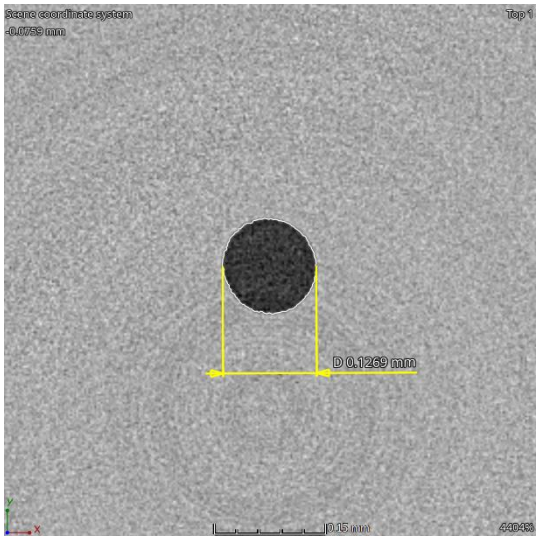

Figure 4: Zoom into the cross section of the micro tube shown in Figure 3 for the determination of the inner diameter of the μ-CT method.

The surface of the cylindrical hole is identified to determine the inner diameter of the micro tube as can be seen in Figure 4. The average inner diameter of the soda glass micro tube measured at 9 different positions is (0.1269 ± 0.0010) mm. The inner diameter of this micro tube has also been calibrated with the flow calibration method and the result is (0.1267 ± 0.0003) mm, which is in perfect agreement with the result of the μ-CT technique.

4. Measurement uncertainty analysis

Several contributions to the measurement uncertainty have already been mentioned in the text above. In this paragraph, an overview of the contributions to the uncertainty is given for the flow calibration method to determine the inner diameter of the micro tube in the pipe viscometer with a liquid of known dynamic viscosity. In the second part, the uncertainty contributions for the pipe viscometer are described and the main contributions are highlighted.

4.1 Flow calibration method

The contributions to the uncertainty of the determination of the inner diameter of the micro tube in the pipe viscometer by means of the flow calibration method are listed in Table 2. The main contribution is related to the calculation of the dynamic viscosity of water by measuring the temperature and pressure of the water [1]. The expanded uncertainty of the temperature measurement is 0.2 °C leading to an extended uncertainty contribution of the dynamic viscosity of 0.5 %. Additionally, the uncertainty of the formula is stated being 0.5 % [7], which lead to an extended uncertainty contribution of 0.71 % for the dynamic viscosity of water. The other important contribution is the pressure measurements upstream and downstream of the micro tube leading to an extended uncertainty of 0.28 % for the pressure drop measurement. Therefore, the extended uncertainty of the determination of the inner diameter is 0.20 %.

Table 2: Uncertainty contributions for the determination of the inner diameter of the micro tube with the flow calibration method.

Contribution	Uncertainty (k=2)	Coefficient
Piston prover for the generation of flow	0.10 %	0.25
Length measurement of the micro tube	0.01 %	0.25
Pressure drop measured by 2 sensors of maximum pressure at 10 bar.	0.28 %	0.25
Calculation of dynamic viscosity of water according to [1] (details in text) - contribution 0.50 %. Uncertainty from the formula is 0.50 % [5,7].	0.71 %	0.25
Total uncertainty of the inner diameter (k=2).	0.20 %	

4.2 METAS pipe viscometer

With the uncertainty contribution of the inner diameter of the micro tube the list of contributions to the uncertainty of the METAS pipe viscometer is complete (see Table 3). Three main contributions are identified. The largest contribution is the measurement of the pressure drop at the micro tube with an uncertainty contribution of 0.28 %. Other important contributions are the uncertainty of the inner diameter of the micro tube being 0.20 % and the uncertainty of the single point analysis for the determination of the dynamic viscosity being 0.20 %. This value has been determined empirically by analyzing several sets of data, where the linear fit method has been compared to the method, where the dynamic viscosity is calculated from a single dataset of flow rate and pressure drop (data not shown). Therefore, the expanded uncertainty is set to 0.90 % for the pipe viscometer build with the glass micro tube.

Table 3: Uncertainty contributions of the pipe viscometer for the determination of the dynamic viscosity.

Contribution	Uncertainty (k=2)	Coefficient
Piston prover for the generation of flow	0.10 %	1.0
Length measurement of the micro tube	0.01 %	1.0
Pressure drop measured by 2 sensors of maximum pressure at 10 bar.	0.28 %	1.0
Inner diameter of micro tubes determined with the pipe viscometer and the reference liquid water.	0.20 %	4.0
Single point analysis at each flow rate for a given pressure drop vs Linear Regression over the flow rate range.	0.20 %	1.0
Total uncertainty of the pipe viscometer (k=2).	0.90 % (0.88 %)	

5. Validation of the METAS pipe viscometer

The uncertainty of the METAS pipe viscometer has been validated by determining the dynamic viscosity of reference liquids. These reference liquids are traceable to SI-units and their calibration certificates state the density and dynamic viscosity as a function of temperature. Three reference liquids with dynamic viscosities in the range from 1.5 mPa·s to 4.0 mPa·s have been calibrated with the METAS pipe viscometer at 21.3 °C, which corresponds to the liquid temperature in the tubing being measured by several temperature sensors. The reference values of the density and the dynamic viscosity of the liquids are listed in Table 4.

These values have been determined by a first order linear fit applied to the data of the density and a second order linear fit applied to the date of the dynamic viscosity as a function of temperature at 20 °C, 23 °C, 25 °C, 30 °C and 40 °C.

Table 4: Reference liquids [8] with traceable density and dynamic viscosity at a temperature of 21.3 °C with the stated expanded (k=2) uncertainty of 0.20 % for the dynamic viscosity and 0.1 kg/m^3 for the density.

Reference liquid name	Dynamic viscosity (mPa·s)	Density (kg/m^3)
2AW	1.5319 ± 0.0031	751.2 ± 0.1
2BW	2.1913 ± 0.0044	764.7 ± 0.1
5AW	4.0393 ± 0.0081	785.3 ± 0.1

The three reference liquids "2AW", "2BW" and "5AW" [8] have been calibrated with the pipe viscometer with the soda glass micro tube (see Figure 2) and the results are listed in Table 5.

The calibrations results are consistent with the reference values (Table 4) within the expanded uncertainty of this pipe viscometer being 0.90 %. The calibration results of the three reference liquids lead to a deviation in respect to the reference values between +0.42 % and +0.43 %, being smaller than the stated measurement uncertainty of 0.90 %.

Table 5: The determination of the dynamic viscosities of reference liquids 2AW, 2BW, 5AW [25] by means of the METAS pipe viscometer in the laminar regime.

	2AW	2BW	5AW
Dynamic viscosity (mPa·s)	1.538 ± 0.014	2.201 ± 0.020	4.056 ± 0.037
Deviation (%)	+0.43 ± 0.90	+0.42 ± 0.90	+0.42 ± 0.90

6. Conclusions

The METAS pipe viscometer for the traceable in-line measurement of the dynamic viscosity for low flow rates from 1 µL/min to 150 mL/min and pressure drops up to 10 bar has been presented. The extended measurement uncertainty of the pipe viscometer is set to 0.90 % and has been validated with measurements of the dynamic viscosity of three traceable reference liquids. The measurements showed consistent results with the reference values within the stated uncertainties. Additionally, the glass micro tube has been characterized by means of the µ-CT at METAS, which is traceable to the SI-units. These measurements showed also consistent results of the inner diameter compared to the flow calibration method.

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