First comparison of inline measurements of dynamic viscosity

H. Bissig[1], O. Büker[2], K. Stolt[2], E. Graham[3], L. Wales[3], A. Furtado[4], S. Moura[4], Z. Metaxiotou[5], S.H. Lee[6], S. Kartmann[7], J. Groenesteijn[8], J.C. Lötters[8]

[1]Federal Institute of Metrology METAS, 3003 Bern-Wabern, Switzerland
[2]RISE Research Institutes of Sweden, Borås, Sweden
[3]TÜV SÜD National Engineering Laboratory NEL, Glasgow, Scotland
[4]Instituto Português da Qualidade IPQ, Caparica, Portugal
[5]Hellenic Institue of Metrology NQIS/EIM, Thessaloniki, Greece
[6]Korea Research Institute of Standards and Science KRISS, Daejeon, South Korea
[7]Hahn-Schickard, Freiburg, Germany
[8]Bronkhorst High-Tech B.V., Ruurlo, Netherlands
E-mail (corresponding author): hugo.bissig@metas.ch

Abstract

Microfluidic devices are gaining importance in various fields of pharmacy, flow chemistry and healthcare. In the embedded microchannel, the flow rates, the dynamic viscosity of the transported fluids and the fluid dynamic properties play an important role. Various auxiliary functional components of microfluidic devices such as flow restrictors, valves and flow meters need to be characterised with liquids used in several microfluidic applications. However, calibration with water does not always reflect the behaviour of the fluids used in the different applications. Therefore, several National Metrology Institutes (NMI) have developed micro pipe viscometers for traceable in-line measurement of the dynamic viscosity of liquids used in flow applications as part of the EMPIR 18HLT08 MeDDII project. These micro pipe viscometers allow the calibration of any flow device at different flow rates and the calibration of the dynamic viscosity of the liquid or liquid mixture used under actual flow conditions. The traceability of the micro pipe viscometer, the validation of the stated measurement uncertainty with eight liquids as well as dynamic viscosity measurements with in-line sensors are presented in this paper.

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1. Introduction

Microfluidic devices are gaining importance in various fields of pharmacy, flow chemistry and healthcare. In the embedded microchannel, the flow rates, the dynamic viscosity of the transported fluids and the fluid dynamic properties play an important role. However, calibration with water does not always reflect the behaviour of the fluids used in the different applications. Therefore, several National Metrology Institutes (NMI) have developed micro pipe viscometers for traceable in-line measurement of the dynamic viscosity of liquids used in flow applications as part of the EMPIR 18HLT08 MeDDII project [1]. These micro pipe viscometers allow the calibration of any flow device at different flow rates and the calibration of the dynamic viscosity of the liquid or liquid mixture used under actual flow conditions.

2. Pipe viscometers at METAS, RISE and NEL

The micro pipe viscometer consists of a flow generator (piston prover) connected to a tube. Appropriate temperature and pressure sensors are installed upstream and downstream of the micro tube to determine the pressure drop as a function of flow rate for the calculation of the dynamic viscosity of the liquid.

2.1 Pipe viscometers at METAS

The flow facility at METAS [2] has currently been modified to include a section with a micro pipe viscometer as can be seen in Figure 1 [3,4]. The pipe viscometer consists of a piston prover to generate the flow and a micro tube with temperature and pressure sensors up- and downstream. The tubing connected to the pressure sensor and the connectors to the micro tube have much larger diameters (more than 2 mm) than the nominal inner diameter of the micro tubes being 0.13 mm in order to guarantee that the recorded pressure drop between the two pressure sensors is essentially due to the pressure drop over the micro tube. Flow rates from 1 μL/min to 150 mL/min can be generated for the in-line measurement of the dynamic viscosity with a pressure drop up to 10 bar. Glass micro tubes with larger inner diameter are also available, but the measurement results presented in this paper have been obtained with the glass micro tube with the inner diameter of 0.13 mm. The expanded uncertainty $U(k=2)$ of the dynamic viscosity measurement is 0.90 %.

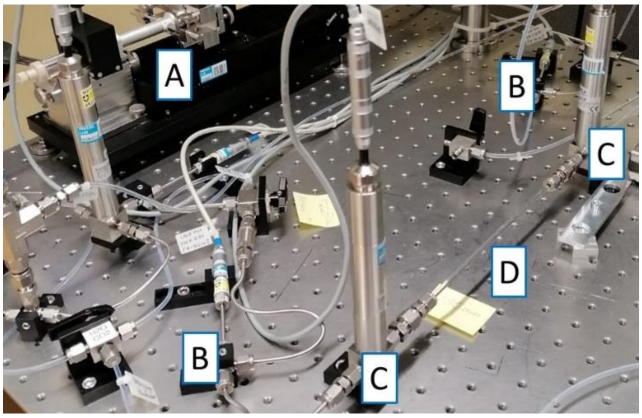

Figure 1: The micro pipe viscometer at METAS: (A) piston prover, (B) temperature sensors, (C) pressure sensors and (D) the glass micro tube with an inner diameter of 0.13 mm and a length of 200 mm.

2.2 Pipe viscometers at RISE

The micro pipe viscometer at RISE consists of a micro tube holder and an associated stainless steel micro tube with a nominal inner diameter of 0.18 mm, an outer diameter of 1/16" and a length of 300 mm [4]. The pressure drop is measured by pressure sensors up- and downstream the micro tube at pressures up to 5 bar (Figure 2). The temperature of the test liquid is measured indirectly with two type K thermocouples attached to the inlet and outlet of the capillary holder. The desired flow rate is generated using a syringe pump and calibrated syringes. To account for the pressure loss in all connections, additional measurements have been performed using a micro tube with a shorter length of 50 mm. The expanded uncertainty U*(k=2)* of the dynamic viscosity measurement is 2.0%.

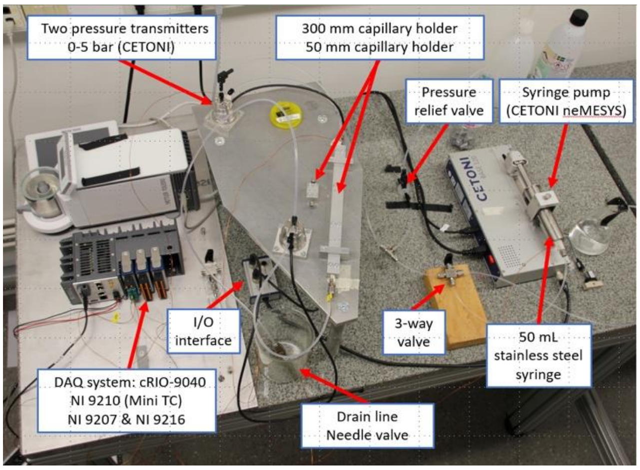

Figure 2: The micro pipe viscometer at RISE with capillaries of length of 50 mm and 300 mm.

2.3 Pipe viscometers at NEL

The micro pipe viscometer at NEL is shown in Figure 3 [4]. The calibrated syringe pump generates traceable flow rates and the Coriolis meter provides secondary flow rate indication, fluid density information as well as the identification of any bubbles or flow disturbances. Two temperature sensors (upstream and downstream sensors) are attached to the outside of the stainless-steel capillaries and used to determine the average fluid temperature. Two calibrated Elveflow pressure sensors (1 bar and 340 mbar) are directly connected to the capillaries to measure the pressure drop. The expanded uncertainty U(*k=2)* of the dynamic viscosity measurement is 1.0 %.

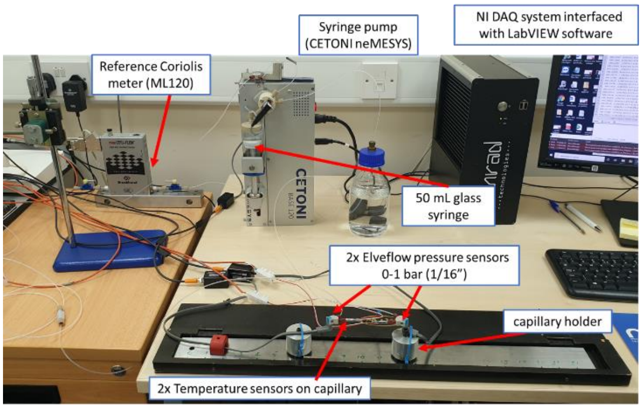

Figure 3: NEL pipe viscometer with a stainless-steel micro-tube with outer diameter of 1/16 inch (1.6 mm), length of 100 mm and nominal inner diameter of 180 µm (manufacturer's specification).

2.4 Calibration of the inner diameter of the micro tube

An important geometrical dimension is the inner diameter of the micro tube, which has to be calibrated to get the micro pipe viscometer traceable. The calibration of the inner diameter of the micro tube is performed by measuring the pressure drop as a function of flow rate at a given temperature with water or a reference liquid, where the temperature-dependent dynamic viscosity is known. Applying the law of Hagen-Poiseuille in the laminar flow regime and calculating the dynamic viscosity of water with the measured temperature allows the determination of the inner diameter of the micro tube. With the calibration of all the geometrical dimensions of the micro pipe viscometer the dynamic viscosity of other liquids can be determined according to the law of Hagen-Poiseuille.

3. Liquids and facilities/instruments

The validation of the micro pipe viscometers has been performed with eight different liquids typically administered in hospitals, as listed in Table 1.

Table 1: Eight liquids to be characterized for the density and the dynamic viscosity.

Liquid	Description
A	Saline solution of 0.9 %wt NaCl
B	Glucose solution 10 %wt
C	Glucose solution 20 %wt
D	Solution of NaCl 0.22 %wt and Glucose 2.75 %wt
E	Solution of NaCl 0.22 %wt and Glucose 5.55 %wt
F	Solution of NaCl 0.45 %wt and Glucose 5.54 %wt
G	Solution of Glycerol 52.0 %wt
H	Solution of Glycerol 58.8 %wt

The kinematic viscosity of these liquids are also calibrated with conventional capillary viscometers (instruments with sampling) and converted to dynamic viscosity with the measured density. The results show the consistency between these conventional capillary viscometers and the micro pipe viscometers. The different facilities/instruments used by each laboratory are listed in Table 2.

Table 2: Facility/instrument used for the measurement of the kinematic or dynamic viscosity of the liquids.

Institute	Facility/instrument	Liquid property
METAS	Pipe viscometer	Dynamic viscosity
RISE	Pipe viscometer	Dynamic viscosity
NEL	Pipe viscometer	Dynamic viscosity
NQIS/EIM	TAMSON TV2000 AKV	Kinematic viscosity
IPQ	Capillary glass viscosity meters DMA 5000, Anton Paar	Kinematic viscosity Density
Hahn-Schickard	Rheometer Physica MCR 101, Anton Paar	Dynamic viscosity
KRISS	Capillary glass viscosity meters DMA 5000M, Anton Paar	Kinematic viscosity Density

4. Validation of the pipe viscometers

The laboratories determined either the dynamic viscosity and/or the kinematic viscosity of the liquids and two laboratories determined the density of the liquids. As the measurements with the micro pipe viscometers determine the dynamic viscosity, the results of the laboratories which determined the kinematic viscosity were converted into the dynamic viscosity using the density values of the liquids determined by IPQ at different temperatures (20 °C – 27 °C) [5]. The density values at 22 °C are listed in Table 3. NQIS/EIM and IPQ carried out measurements for the kinematic viscosity at different temperatures namely 20 ° C, 21 °C, 22 °C, 23 °C, 24 °C, and 27 °C [5]. The calculated dynamic viscosities at different temperatures were used to determine the interpolation factors for temperatures between 22 °C and 24 °C, which cover the temperatures of the measurements of the other laboratories and allow the interpolation of the individual results to 22 °C. The results of the dynamic viscosity and the corresponding uncertainties are listed in Table 4. KRISS measured the kinematic viscosity and the density and calculated the dynamic viscosity with their own values.

Table 3: Density measured by the laboratories IPQ and KRISS at a temperature of 22 °C in the unit kg/m^3. The results presented are the measured values including the expanded measurement uncertainty U(*k=2*).

Liquid	IPQ	KRISS
A	1004.172 ± 0.033	Not measured
B	1038.171 ± 0.033	1038.163 ± 0.058
C	1079.829 ± 0.033	1079.799 ± 0.058
D	1010.362 ± 0.033	Not measured
E	1020.217 ± 0.033	1020.330 ± 0.058
F	1021.882 ± 0.033	1021.902 ± 0.058
G	1131.303 ± 0.033	1131.274 ± 0.058
H	1150.192 ± 0.033	Not measured

The validation of the results of the pipe viscometers is carried out according to the rules of an intercomparison of laboratories, which not only describes the calculation of the reference value, but also defines the consistency check by means of the χ^2-test defining the laboratories contributing to the calculation of the reference value [6]. The results contributing to the calculation of the reference value are highlighted in green in Table 4.
The deviations of each laboratory with respect to the reference value are listed in Table 5 and shown in Figure 4.

Table 4: Dynamic viscosities measured by the different laboratories and methods at a temperature of 22 °C. The results presented are the measured values including the expanded measurement uncertainty U(*k=2*) in the unit mPa·s. The reference value has been determined with the results of the laboratories highlighted in green.

Liquid	METAS	RISE	NEL	NQIS/EIM
A	0.9706 ± 0.0088	0.959 ± 0.020	0.9742 ± 0.0098	0.9722 ± 0.0098
B	1.276 ± 0.012	1.279 ± 0.026	1.263 ± 0.013	1.280 ± 0.013
C	1.810 ± 0.017	1.800 ± 0.037	1.795 ± 0.018	1.823 ± 0.019
D	1.037 ± 0.010	1.056 ± 0.022	1.046 ± 0.011	1.048 ± 0.011
E	1.110 ± 0.010	1.112 ± 0.023	1.123 ± 0.012	1.119 ± 0.012
F	1.111 ± 0.011	1.113 ± 0.023	1.158 ± 0.012	1.119 ± 0.012
G	6.290 ± 0.057	6.33 ± 0.13	6.482 ± 0.064	6.329 ± 0.064
H	9.418 ± 0.085	9.35 ± 0.19	9.57 ± 0.10	9.405 ± 0.095
Liquid	**IPQ**	**KRISS**	**Hahn-Schickard**	**Reference value**
A	1.018 ± 0.047	Not measured	0.898 ± 0.145	0.972 ± 0.005
B	1.327 ± 0.059	1.277 ± 0.026	1.166 ± 0.042	1.274 ± 0.007
C	1.862 ± 0.078	1.803 ± 0.036	1.596 ± 0.021	1.809 ± 0.009
D	1.069 ± 0.051	Not measured	0.902 ± 0.031	1.045 ± 0.006
E	1.144 ± 0.081	1.107 ± 0.023	0.972 ± 0.042	1.116 ± 0.006
F	1.160 ± 0.053	1.110 ± 0.023	1.044 ± 0.042	1.115 ± 0.007
G	6.45 ± 0.26	6.28 ± 0.13	5.014 ± 0.084	6.310 ± 0.038
H	10.11 ± 0.76	Not measured	7.749 ± 0.085	9.453 ± 0.051

Table 5: Deviations and uncertainties of each laboratory compared to the reference value in the unit % after the χ^2-test evaluation.

Liquid	METAS	RISE	NEL	NQIS/EIM
A	-0.13 ± 0.90	-1.29 ± 2.00	0.24 ± 1.00	0.03 ± 1.00
B	0.14 ± 0.90	0.36 ± 2.00	-0.91 ± 1.00	0.43 ± 1.00
C	0.07 ± 0.90	-0.50 ± 2.00	-0.77 ± 1.00	0.75 ± 1.00
D	-0.69 ± 0.90	1.13 ± 2.00	0.15 ± 1.00	0.35 ± 1.00
E	-0.53 ± 0.90	-0.34 ± 2.00	0.64 ± 1.00	0.28 ± 1.00
F	-0.32 ± 0.90	-0.17 ± 2.00	3.81 ± 1.00	0.37 ± 1.00
G	-0.32 ± 0.90	0.26 ± 2.00	2.72 ± 1.00	0.31 ± 1.00
H	-0.37 ± 0.90	-1.12 ± 2.00	1.19 ± 1.00	-0.51 ± 1.00
Liquid	**IPQ**	**KRISS**	**Hahn-Schickard**	**Reference value**
A	4.70 ± 4.55	Not measured	-7.66 ± 16.1	0 ± 0.54
B	4.14 ± 4.41	0.18 ± 2.00	-8.52 ± 3.52	0 ± 0.52
C	2.95 ± 4.17	-0.34 ± 2.00	-11.79 ± 1.30	0 ± 0.51
D	2.38 ± 4.77	Not measured	-13.69 ± 3.43	0 ± 0.54
E	2.49 ± 7.02	-0.84 ± 2.00	-12.96 ± 4.25	0 ± 0.52
F	4.04 ± 4.52	-0.48 ± 2.00	-6.37 ± 3.93	0 ± 0.60
G	2.23 ± 3.93	-0.45 ± 2.00	-20.54 ± 1.67	0 ± 0.60
H	6.95 ± 7.52	Not measured	-18.02 ± 1.08	0 ± 0.53

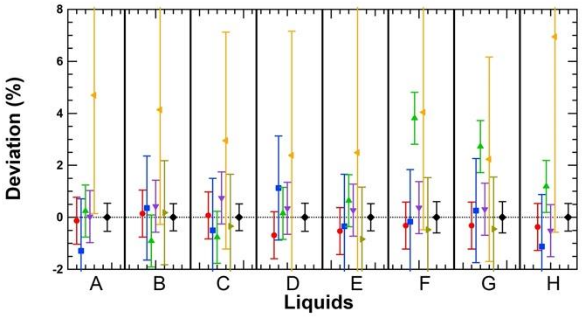

Figure 4: Results for the deviations of the dynamic viscosity measurements performed by the different laboratories with respect to the reference value after applying the χ^2-test evaluation. METAS (red circle), RISE (blue square), NEL (green triangle up), EIM (violet triangle down), IPQ (orange triangle left), KRISS (dark yellow triangle right), Reference value (black diamond). The deviations of Hahn-Schickard are not shown in the Figure as the deviations are larger than the selected range.

4.1 Normalised equivalence value (En) analysis

The equivalence of the results of each laboratory with respect to the reference value (REF) can be quantified in the form of the normalised equivalence value *En* value (*En*) [6-9]. For the laboratory, where the result is contributing to the reference value REF, the *En* value is calculated according to Equation (1):

$$En = \frac{|LAB - REF|}{\sqrt{(U_{95}LAB)^2 - (U_{95}REF)^2}} \qquad (1)$$

For the laboratory, where the result is not contributing to the reference value REF, the *En* value is calculated according to Equation (2):

$$En = \frac{|LAB - REF|}{\sqrt{(U_{95}LAB)^2 + (U_{95}REF)^2}} \qquad (2)$$

The *En* values for each laboratory and each liquid are listed in Table 6.

The interpretation of the absolute value of the *En* value is as follows:

- $En < 1.0$: the result of the laboratory is consistent with the reference value (green).
- $1.0 < En \leq 1.2$: the result of the laboratory is not clearly consistent with the reference value (orange). A warning is issued. Measurement procedure and uncertainty has to be checked.
- $1.2 < En$: the result of the laboratory is inconsistent with the reference value and the reason has to be identified (red).

Table 6: En-Values for the laboratories performed the measurements of the dynamic viscosity.

Liquid	METAS	RISE	NEL	NQIS/EIM
A	0.19	0.67	0.28	0.03
B	0.19	0.18	1.07	0.50
C	0.09	0.26	0.90	0.88
D	0.95	0.58	0.17	0.42
E	0.71	0.18	0.74	0.33
F	0.48	0.09	3.29	0.46
G	0.48	0.13	2.36	0.39
H	0.51	0.58	1.41	0.61
Liquid	**IPQ**	**KRISS**	**Hahn-Schickard**	
A	1.04	n/a	0.47	
B	0.95	0.09	2.39	
C	0.71	0.18	8.42	
D	0.50	n/a	3.95	
E	0.36	0.43	3.03	
F	0.90	0.25	1.60	
G	0.58	0.23	11.59	
H	0.93	n/a	14.91	

The measurement results of the pipe viscometers show in most cases consistent results with the reference values. NEL is inconsistent with the reference value for the liquids F, G and H. It is worth to mention here that NEL has performed the measurements with the liquids A – E in a first stage and that the liquids F – G were measured in a later stage.

Overall, the performed validation of the pipe viscometers shows a large consistency with well-established measurement methods for the measurement of the kinematic viscosity or the dynamic viscosity. The laboratories with inconsistent results of the *En* value will have to investigate the reasons for this and to check the uncertainty value as well as the measurement stability of the pipe viscometer, where temperature and pressure stability plays an important role.

5. In-line sensors for dynamic viscosity

Measurement results of a commercially available instrument (VLO-M1 from TrueDyne Sensors AG [10]) and a technology demonstrator (multi-parameter chip based on the Coriolis measuring principle from Bronkhorst High-Tech B.V. [11]) for the inline measurement of dynamic viscosity and density are presented in this chapter.

5.1 Commercial sensor VLO-M1 from TrueDyne Sensors AG

This sensor measures the temperature of the tubing, the density and the dynamic viscosity of the liquid. Measurements with all the liquids A – H have been performed. The temperature of the tubing has been taken to determine the interpolation of the density and the dynamic viscosity measurements at 22 °C in order to compare the results to the reference value.

The results of the density and the dynamic viscosity are listed in Table 7 and Table 8, respectively. Note that these measurements have been performed 6 months after preparation of the solution and the density and kinematic viscosity measurements at IPQ. The other laboratories have performed their viscosity measurements within this period of 6 month.

The deviations are well within the stated accuracy of the sensor and these results confirm that the measurement procedure by pushing one liquid after the other through the sensor works well and no contamination is expected.

Table 7: Reference value of the density measured by the laboratory IPQ and the results of the VLO-M1 sensor at a temperature of 22 °C. The results presented are the measured values including the expanded measurement uncertainty U*(k=2)*. The deviation (Dev) of the VLO-M1 with respect to the reference value and its measurement uncertainty is also represented in this table.

Liquid	Reference value (kg/m^3)	VLO-M1 (kg/m^3)	VLO-M1 Dev (%)
A	1004.172 ± 0.033	1004.29 ± 0.20	0.011 ± 0.020
B	1038.171 ± 0.033	1038.30 ± 0.20	0.012 ± 0.020
C	1079.829 ± 0.033	1079.76 ± 0.20	-0.006 ± 0.019
D	1010.362 ± 0.033	1010.47 ± 0.20	0.010 ± 0.020
E	1020.217 ± 0.033	1020.32 ± 0.20	0.010 ± 0.020
F	1021.882 ± 0.033	1021.97 ± 0.20	0.009 ± 0.020
G	1131.303 ± 0.033	1131.42 ± 0.20	0.011 ± 0.018
H	1150.192 ± 0.033	1150.41 ± 0.20	0.019 ± 0.018

Table 8: The reference values and the results of the VLO-M1 sensor for the measurement of the dynamic viscosity of the eight liquids at a temperature of 22 °C in the unit mPa·s. The deviation of the VLO-M1 with respect to the reference value and its measurement uncertainty is also represented in this table with the unit %.

Liquid	Reference value (mPa·s)	VLO-M1 (mPa·s)	VLO-M1 Dev (%)
A	0.972 ± 0.005	0.945 ± 0.047	-2.8 ± 5.0
B	1.274 ± 0.007	1.239 ± 0.062	-2.8 ± 5.0
C	1.809 ± 0.009	1.740 ± 0.087	-3.8 ± 5.0
D	1.045 ± 0.006	1.013 ± 0.050	-3.0 ± 5.0
E	1.116 ± 0.006	1.077 ± 0.054	-3.5 ± 5.0
F	1.126 ± 0.006	1.080 ± 0.054	-4.0 ± 5.0
G	6.310 ± 0.038	6.15 ± 0.31	-2.6 ± 5.0
H	9.453 ± 0.051	9.44 ± 0.47	-0.2 ± 5.0

5.2 Technology demonstrator multi-parameter measuring system (MMS)

This sensor measures the mass flow rate, the temperature of the tubing, the pressure upstream and downstream of the tubing, the density and the dynamic viscosity. The multi-parameter chip containing the micro Coriolis flow sensor, two pressure sensors and temperature sensors is shown in Figure 5 [11].

Figure 5: Multi-parameter chip of Bronkhorst High Tech B.V. containing the micro Coriolis flow sensor and two pressure sensors used for measurements of the density and the dynamic viscosity.

The densities of water and the liquids A, B, E and F were measured and are listed in Table 9. Note that these measurements have been performed 15 months after preparation of the solution and the density measurements at IPQ. No further density calibrations have been performed since then by IPQ or any other lab. The other liquids could not be measured due to time restrictions.

The various results of the density measurement with water listed in Table 9 have been performed at different flow rates.

Table 9: Results of the measured density by the MMS at different flow rates (average values of at least two measurements). The reference values for density of water are determined by the NIST database [12]. The reference values for density of the other liquids are measured by IPQ.

Liquid	**Tube T (°C)**	**Reference value (kg/m^3)**	**MMS (kg/m^3)**	**MMS Dev (%)**
Water	29.6	995.75 ± 0.10	996.4 ± 5.0	0.07 ± 0.50
Water	29.6	995.75 ± 0.10	994.7 ± 5.0	-0.11 ± 0.50
Water	32.9	994.81 ± 0.10	994.5 ± 5.0	-0.03 ± 0.50
A	32.9	1001.06 ± 0.10	996.9 ± 5.0	-0.42 ± 0.50
B	33.0	1034.67 ± 0.10	1025.8 ± 5.0	-0.85 ± 0.50
E	32.9	1016.94 ± 0.10	1012.2 ± 5.0	-0.46 ± 0.50
F	32.9	1018.57 ± 0.10	1012.9 ± 5.0	-0.55 ± 0.50

Further investigations with freshly prepared liquid solutions, where the density is calibrated before the measurements with the sensor, are needed to study the accuracy of the sensor in more detail.

The stated measurement uncertainty is the target accuracy for this technology demonstrator. An exact analysis of the measurement uncertainty is under investigation. Since the system for the measurement of dynamic viscosity is still under development, the measurement results of the dynamic viscosity are not presented in this paper.

6. Conclusion

These measurements of the dynamic viscosity of the liquids A – H reported here have shown that the pipe viscometers are a valuable primary standard for the in-line determination of the dynamic viscosity of liquids. The measurement results of the pipe viscometers show in most cases consistent results with the reference values, which are determined by the results of the pipe viscometers and well-established measurement methods for the kinematic viscosity with glass capillary viscometers. In-line sensors for dynamic viscosity are either commercially available or under development. The traceable pipe viscometers allow the calibration of these in-line sensors under flow conditions.

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