Bilateral comparison of viscosity measurement standard system between KRISS and PTB

S. H. Lee[1], J. Rauch[2], B.-R. Yoon[1]

[1]Korea Research Institute of Standards and Science, 267 Gajeong-ro, Yuseong-Gu, Daejeon 34113, Korea
[2]Physikalisch-Technische Bundesanstalt, Bundesallee 100, 38116 Braunschweig, Germany
E-mail (corresponding author): seokhwan.lee@seokhwan.lee

Abstract

This study was conducted to compare the viscosity measurement standard systems of the KRISS and PTB, as well as to confirm the international equivalence of the standard viscosity measurement system built in the KRISS. The KRISS constructed a viscosity measurement standard system using an Ubbelohde-type capillary viscometer. In the KRISS, the viscometer was calibrated based on the water viscosity standard ISO TR 3666, and 16 viscometer coefficients were obtained using the step-up method. The measured viscosity was corrected by evaluating the surface tension, buoyancy, and kinetic energy. The uncertainty of the measurement system, including the temperature and measurement time, was evaluated. The measurement range of the viscosity measurement standard system was 0.3 to 100000 mm^2/s, with 0.13%–0.5 % uncertainty (U, k = 2). A bilateral comparison of the viscosity measurement standard system between KRISS and PTB was conducted using three different viscosity standard liquids (5A, 2000A, and 50000A) synthesized by the PTB. The viscosity of the standard liquid was measured at three different temperatures (15 °C, 20 °C, and 40 °C), and comparisons were performed under all six experimental conditions (5A/15 °C, 5A/20 °C, 2000A/20 °C, 2000A/40 °C, 50000A/20 °C, and 50000A/40 °C). By considering the uncertainty, the calculated En was less than 1 (0.17–0.72) for all experimental cases. Therefore, it was confirmed that the recently constructed viscosity standard system of the KRISS exhibits mutual equivalence with the viscosity measurement standard system of the PTB. In the future, KRISS will register the viscosity measurement standard system in a CMC based on the results of this bilateral comparison.

1. Introduction

Viscosity measurements are important both in many fields of industry and in research. Viscosity is a technological quantity which concerns the flow of matter. It is a physical quantity related to the transport properties. Many (National Metrology Institutes (NMIs) have established viscosity measurement standards to provide viscosity measurement services [1,2]. And recently, the results of the viscosity CCM key comparison have been reported [3]. This report describes the third CCM key comparison in capillary viscometry at twelve National Metrology Institutes (NMIs), which was carried out between October 2012 and February 2013. In CCM key comparison, the degrees of equivalences were evaluated by difference from the KCRV and, with a few exceptions, these differences were almost equal to or less than expanded uncertainties, showing a good equivalencies of capabilities at the participating NMIs for the viscosity measurements in wide range of viscosities covered from 5 mm^2/s to 160000 mm^2/s.

The national standard of viscosity provided by KRISS consists of a set of Ubbelohde viscometers covering the measuring range of kinematic viscosities from about 0.3 mm^2/s to 100000 mm^2/s. At the low viscosity end of the scale, long-capillary viscometers are used as primary standards which are directly calibrated with water. KRISS has a maximum measurement uncertainty of 0.5% (k=2) in the above viscosity measurement range.

Recently, a bilateral comparison of the viscosity measurement standard system between KRISS and PTB was conducted using three different viscosity standard liquids (5A, 2000A, and 50000A) synthesized by the PTB. The viscosity of the standard liquid was measured at three different temperatures (15 °C, 20 °C, and 40 °C), and comparisons were performed under all six experimental conditions (5A/15 °C, 5A/20 °C, 2000A/20 °C, 2000A/40 °C, 50000A/20 °C, and 50000A/40 °C). The calculated En was less than 1 (0.16–0.45) for all experimental cases shown in Table 1. Therefore, it was confirmed that the recently constructed viscosity standard system of the KRISS exhibits mutual equivalence with the viscosity measurement standard system of the PTB.

2. KRISS viscosity standard system

2.1 The viscosity scale of KRISS

The KRISS viscosity standard is an independent standard based on viscosity of water at 20 °C, with overlapping measuring ranges covering the range of kinematic viscosities from about 0.3 mm²/s to 100,000 mm²/s. It consists of sixteen master viscometers of the Ubbelohde type having 400 mm long capillaries. Each viscometer has a different capillary diameter and a corresponding viscosity measurement range as shown in table 1. The figure 1 (a) shows the picture of 16 viscometers of KRISS. We used viscosity liquids (Fig. 1(b), Cannon Instrument Company) to obtain the viscometer coefficient. This viscosity liquid was used to independently construct the viscosity coefficient using the step-up procedure.

Table 1: Specifications of the Ubbelohde type capillary viscometers of KRISS

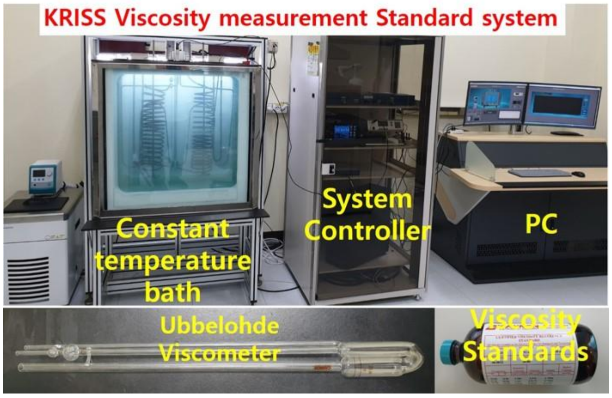
No.	Capillary radius(R) *mm*	Capillary Volume (V)*mL*	Capillary length (L) *mm*	Viscosity measurement range mm²/s
0	0.24	1	400	0.3 – 1
0C	0.36	2	400	0.6 – 3
0B	0.46	3	400	1 – 5
1	0.58	4	400	2 – 10
1C	0.77	4	400	6 – 30
1B	0.88	4	400	10 – 50
2	1.03	4	400	20 – 100
2C	1.36	4	400	60 – 300
2B	1.55	4	400	100 – 500
3	1.83	4	400	200 – 1000
3C	2.43	4	400	600 – 3000
3B	2.75	4	400	1000 – 5000
4	3.27	4	400	2000 – 10000
4C	4.21	4	400	6000 – 30000
4B	5.2	5	400	10000 – 50000
5	6.25	5	400	20000 - 100000

(a)

(b)

Figure 1: Picture of (a) 16 viscometers set, (b) viscosity liquid

2.2 Experimental facilities



(a)

(b)

Figure 2: Picture of (a) viscosity measurement standard system at KRISS and (b) operating software of viscosity measurement standard system at KRISS.

The thermostat is manufactured directly by KRISS and consists of a heater and an external cooling unit. Its performance had previously been assessed in the range between 20 C and 40 C. Temperature gradient was found to be less than 10 mK in the whole measurement zone both as thermal stability and as uniformity of the bath. Temperature measurements are usually performed by Pt25 platinum resistance thermometers and a thermometer (WIKAI CTR3000). The uncertainty due to the temperature measuring apparatus is 10 mK.

The flow time when the fluid meniscus passed the two lines of the viscometer was measured by stopwatch (SEIKO, WO73-4000). The stopwatch is displayed up to 0.01 s and has an accuracy of about 0.001%.

2.3 Measurement principle

Viscometers which are directly calibrated with water are called primary viscometers. The measurement principle is based on the Hagen-Poiseuille law.

$$\nu = \frac{\pi \cdot R^4 \cdot g \cdot h_m}{8 \cdot V \cdot L} \cdot t \qquad (1)$$

Where V is the liquid volume which during the flow time t flows through the capillary of length L and radius R under the influence of acceleration due to gravity g. The height of the average pressure head in the viscometer during the measurement is denoted by hm. All the constants can be combined in the viscometer coefficient C.

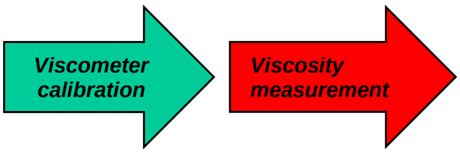
$$\nu = C \cdot t \qquad (2)$$

At this time, if a Ubbelohde type viscometer is used in a capillary viscometer, the effect of thermal expansion can be ignored, and if the measurement time is 300 seconds or more, the correction of kinetic energy can be ignored. Therefore, it may be expressed as a correction equation in consideration of only the surface tension C_s and the air buoyancy C_b as follows (ASTM D2162).

$$\nu = C \cdot t \cdot (1 + c_b + c_S) \qquad (3)$$

2.3 Step up procedure

Table 2: Specifications of the Ubbelohde type capillary viscometers of KRISS



Step No	Viscosity liquids for calibration of viscometer	Viscometer	Obtained viscosity from viscometer
1	Water	0	N1,0, N2.0
2	Water, N1.0	0C	N2.0
3	N1.0, N2.0	0B	S3.0
4	S3.0	1	S6.0
5	S6.0	1C	N7.5, N10
6	N7.5, N10	1B	N14, S20
7	N14, S20	2	N35
8	N35	2C	N44, S60
9	N44, S60	2B	N75, N100
10	N75, N100	3	N250, N350
11	N250, N350	3C	S600, N750
12	S600, N750	3B	N1000
13	N1000	4	S2000, N2500
14	S2000, N2500	4C	N4000, N500
15	N4000, N5100	4B	S8000
16	N5100, S8000	5	S8000

Table 2 shows the viscometers and viscosity liquids used for the step-up procedure. The step-up procedure starting with freshly distilled water at 20 °C (ν = 1.0034 mm²/s) in the first step to calibrate viscometer No. 0. in the first step. Then, the viscosity values of the viscosity liquids N1.0 and N2.0 are measured using the calibrated viscometer 0. In the second step, the viscometer 0C is calibrated using water and N1.0 viscosity liquid. At this time, the viscometer coefficient obtained by two liquids is averaged to obtain the exact coefficient of viscometer 0C. As the step continues in this way, the viscometer is calibrated up to the viscometer 5 and the viscosity values of all the viscosity liquids are measured. Figure 2 shows the measurement range of 16 viscometers and the viscosity liquid to be matched with those used in step up procedure. The viscometer was calibrated using the viscosity liquid that was machined respectively, and through this, the KRISS viscosity measurement system could ensure the measurement range of viscosity from 0.3 mm²/s to 10000 mm²/s.

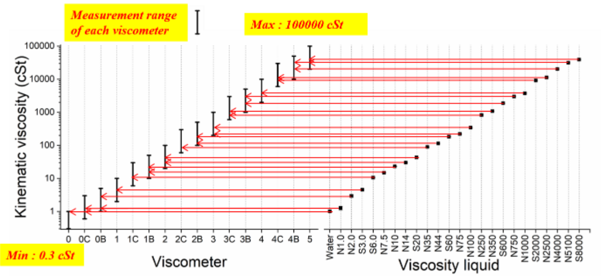


Figure 2: The range of each viscometer in step up procedure and the viscosity value of the viscosity liquid matched to each viscometer

2.4 Uncertainty

The relative standard uncertainty of the kinematic viscosity measurement can be expressed as follows.

$$\left(\frac{u(\nu_n)}{\nu_n}\right)^2 \cong \left(c(C_n)\frac{u(C_n)}{C_n}\right)^2 + \left(c(t)\frac{u(t)}{t}\right)^2 + \left(c(c_s)u(c_s)\right)^2 + \left(c(c_b)u(c_b)\right)^2 + \left(c(T)\frac{u(T)}{T}\right)^2 \qquad (4)$$

The uncertainty factors of viscosity are the uncertainty of the viscometer coefficient, the uncertainty of time measurement, the uncertainty of surface tension correction, the uncertainty of buoyancy correction, and the uncertainty of temperature measurement.

2.3.1 Uncertainty for viscometer coefficients $u_r(C_n)$

Use the uncertainty shown in the viscometer's report to determine the uncertainty of the viscometer coefficient. The uncertainty indicated in the viscometer report is the extended uncertainty

(U), and in order to express the relative standard uncertainty ($u_r(Cn)$) of the viscometer, the inclusion factor (k) indicated in the report is divided as follows. At this time, the uncertainty of the viscometer constant obtained by the gradual method increases as the viscosity increases, and the uncertainty accumulates and increases.

$$u_r(C_n) = \frac{U}{k} \quad (5)$$

2.3.2 Uncertainty for time measurement $u_r(t)$

The uncertainty by time measurement can be expressed as the following equation.

$$u_r(t) = \left\{ \left(u_r(t_{res})\right)^2 + \left(u_r(t_{rea})\right)^2 + \left(u_r(t_{rep})\right)^2 \right\}^{\frac{1}{2}} \quad (6)$$

$u_r(t_{res})$ is the relative standard uncertainty by the resolution of the timer measuring the outflow time of the viscometer. The resolution of the timer is 0.01 seconds, and the time resolution can be obtained by dividing it by the outflow time of the viscometer. The probability density function is assumed to be a rectangular probability distribution, so the effective degree of freedom is ∞.

$$u_r(t_{res}) = \frac{\left\{\frac{\text{timer resolution}(0.01\text{ s})}{\text{measuring time}} \times 100\right\}}{\sqrt{3}} \quad (7)$$

$u_r(t_{rea})$ determined that 0.1 sec of uncertainty occurs as an uncertainty that occurs when an operator reads the time during time measurement. The probability density function is a rectangular probability distribution and the effective degree of freedom is ∞.

$u_r(t_{rep})$ represents the uncertainty of repeated measurements of the outflow time. The repeated measurement uncertainty that occurs when the outflow time is repeatedly measured can be expressed as follows.

$$u_r\left(t_{rep}\right) = \left\{ \frac{\sum_{j=1}^{n} \left(t_j - \bar{t}\right)^2}{n(n-1)} \right\}^{\frac{1}{2}} \quad (8)$$

Here, t_j is the measured outflow time and $\bar{t}$ is the average value of the outflow time. n represents the number of times the experiment was conducted.

2.3.3 Uncertainty for temperature measurement $u_r(T)$

Uncertainty by temperature measurement can be expressed as the following equation.

$$u_r(T) = \left\{ \left(u_r(T_s)\right)^2 + \left(u_r(T_B)\right)^2 + \left(u_r(T_A)\right)^2 + \left(u_r(T_r)\right)^2 \right\}^{\frac{1}{2}} \quad (9)$$

$u_r(T_S)$: Standard uncertainty of the reference thermometer
$u_r(T_B)$: Standard uncertainty according to temperature stability and distribution of thermostat
$u_r(T_A)$: Standard uncertainty according to long-term stability of the reference thermometer
$u_r(T_r)$: Standard uncertainty of resolution of the reference thermometer

Table 3 shows the uncertainty budget for viscosity measurements for N75 viscosity liquids. The largest uncertainty factor is the uncertainty of the viscometer coefficient and the second is the uncertainty of the temperature measurement.

Table 3: Example of uncertainty budget for viscosity measurement of N75 viscosity liquid

Sources		Std u(%)	Sensitivity	Contri bution
Viscometer coefficient (2B)	$u(C_n)$	0.09	1.00	0.09
Time*	u(t)	0.01	1.00	0.01
Buoyancy	$u(C_b)$	0.01	0.00	0.00
Temperature	u(T)	0.06	1.00	0.06
Surface tension	$u(C_S)$	0.01	0.00	0.00
Combined uncertainty(%)				0.11
k				2
Expended uncertainty(%)				0.21

2.5 Combined uncertainty for viscosity determination

Table 4 shows the viscometer coefficient of KRISS viscosity measurement system obtained using step up procedure starting from water and its uncertainty. As the viscosity measurement range of the viscometer increases, the uncertainty of the previous viscometer accumulates. The uncertainty of viscometer 5, which measures the highest viscosity, is 0.48%. Table 5 summarises the typical limits of uncertainty at 95% to be associated with the viscosity values in the kinematic viscosities range from about 0.3 mm^2/s to 100000 mm^2/s by the KRISS primary viscometers. The viscosity liquid S8000 measured by viscometer 5, which measures the highest viscosity range, has a maximum uncertainty of 0.5%.

Table 4: Viscosity Coefficients and Uncertainty of KRISS Viscosity Measurement System

No.	Viscometer coefficient	U (*k*=2) %
0	0.0010	0.12
0C	0.0030	0.17
0B	0.0054	0.17
1	0.0101	0.17
1C	0.0378	0.17
1B	0.0614	0.17
2	0.1054	0.17
2C	0.3437	0.17
2B	0.5678	0.17
3	1.279	0.17
3C	3.404	0.17
3B	5.423	0.17
4	10.87	0.18
4C	34.67	0.20
4B	54.41	0.26
5	116.8	0.48

Table 5: Viscosity value and uncertainty of viscosity liquid measured by KRISS viscosity measurement system

Viscosity liquids	Viscosity (mm^2/s)	U (*k*=2) (%)
Water	1.00	0
N1.0	1.26	0.17
N2.0	2.89	0.17
S3.0	4.46	0.21
S6.0	10.36	0.21
N7.5	14.45	0.21
N10	22.38	0.21
N14	29.14	0.21
S20	41.82	0.21
N35	86.01	0.21
N44	109.74	0.21
S60	174.18	0.21
N75	210.47	0.21
N100	328.93	0.21
N250	778.19	0.21
N350	1024.10	0.21
S600	1765.02	0.21
N750	2804.28	0.21
N1000	3506.49	0.21
S2000	8497.36	0.22
N2500	10480.71	0.22
N4000	18698.74	0.23
N5100	28874.63	0.24
S8000	36062.48	0.28
S8000	36054.56	0.50

3. Bilateral comparison of viscosity measurement standard system between KRISS and PTB

2.1 The viscosity scale of KRISS

A bilateral comparison of viscosity standard measurement system of KRISS and PTB was performed. To compare the viscosity measurement system of KRISS and PTB, three samples were selected and two temperatures per sample were set (Table 6). Figure 3 shows three samples used for bilateral comparison of KRISS-PTB.

Table 6: Experimental samples and temperatures for KRISS-PTB comparison

Cases	Samples No.	Temperature (℃)
1	5 A	15
2	5 A	20
3	2000 A	20
4	2000 A	40
5	50000 A	20
6	50000 A	40

Figure 3: Viscosity liquids samples for KRISS-PTB comparison

Table 6 shows the bilateral comparison results of the viscosity measurement standard systems of the KRISS and PTB. Measurements were performed from September to October 2021 at both the KRISS and PTB. When the temperature of the 5A sample was 20 °C, the viscosities of the KRISS and PTB liquids were 6.31 and 6.32 mm^2/s, respectively; when the temperature of the 50000 A sample was 20 °C, the viscosity of the KRISS and PTB liquids were 53423 and 53470 mm^2/s, respectively. By considering the uncertainty, the calculated E_n was less than 1 (0.17–0.72) for all experimental cases shown in Table 6. Therefore, it was confirmed that the recently constructed viscosity standard system of the KRISS exhibits mutual equivalence with the viscosity measurement standard system of the PTB. In the future, KRISS will register the viscosity measurement standard system in a CMC based on the results of this bilateral comparison.

Table 6: Results of bilateral comparison of viscosity measurement standard system between KRISS and PTB

No.	Tem.	KRISS		PTB		E_n
	T/℃	v/mm^2/s	U/mm^2/s	v/mm^2/s	U/mm^2/s	
5 A	15	7.340	0.01	7.350	0.010	0.72
5 A	20	6.310	0.01	6.320	0.010	0.59
2000 A	20	2023.1	4.34	2020.7	3.1	0.44
2000 A	40	592.48	1.27	591.89	0.92	0.38
50000 A	20	53423	264.90	53470	110	0.17
50000 A	40	15464	44.05	15444	32	0.37

7. Conclusion

In this paper, a bilateral comparison of viscosity measurement systems of KRISS and PTB was conducted. The KRISS constructed a viscosity measurement standard system using an Ubbelohde-type capillary viscometer. In the KRISS, the viscometer was calibrated based on the water viscosity standard ISO TR 3666, and 16 viscometer coefficients were obtained using the step-up method. The measurement range of the viscosity measurement standard system was 0.3 to 100000 mm^2/s, with 0.12%–0.5 % uncertainty (U, k = 2). A bilateral comparison of the viscosity measurement standard system between KRISS and PTB was conducted using three different viscosity standard liquids (5A, 2000A, and 50000A) synthesized by the PTB. The viscosity of the standard liquid was measured at three different temperatures (15 °C, 20 °C, and 40 °C), and comparisons were performed under all six experimental conditions (5A/15 °C, 5A/20 °C, 2000A/20 °C, 2000A/40 °C, 50000A/20 °C, and 50000A/40 °C). By considering the uncertainty, the calculated En was less than 1 (0.17–0.72) for all experimental cases. Therefore, it was confirmed that the recently constructed viscosity standard system of the KRISS exhibits mutual equivalence with the viscosity measurement standard system of the PTB. In the future, KRISS will register the viscosity measurement standard system in a CMC based on the results of this bilateral comparison.

References

[1] Lorefice S and Soba F, "The Italian primary kinematic viscosity standard: The viscosity scale", *Measurement*, **Vol 112**, pp 1-8, 2017.

[2] Fujita Y, Kurano Y and Fujii K, "Evaluation of uncertainty in viscosity measurements by capillary master viscometers", *Metrologia*, **Vol 46**, pp 237-248, 2009.

[3] Final report CCM:V-K3: CCM key comparison of viscosity, https://www.bipm.org/documents/20126/48150889/CCM.V-K3.A/6d021a2c-fe56-54ee-2f63-6eecc85f2b9b