METHOD FOR CALIBRATION OF CAPILLARY GLASS VISCOMETERS AND ASSESSMENT OF MEASUREMENT UNCERTAINTY

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Abstract*. The article discusses current issues of creating conditions for improving the quality and competitiveness of domestic products through the development and improvement of calibration systems for measuring instruments, the development of mechanisms for mutually beneficial cooperation in the field of metrology with international and regional metrological organizations.*
Keywords: *regional metrological organizations, metrology, viscometer.*

Testing and calibration laboratories ensuring metrological traceability to the International System of Units (SI) is one of the main technical requirements of the international standard ISO/IEC 17025:2017 “General requirements for the competence of testing and calibration laboratories”, which is achieved by promoting the integration of the Republic of Uzbekistan into the international economy and international systems to ensure uniformity of measurements as an equal partner. Today, according to the State Unitary Enterprise "Accreditation Center", there are more than 600 laboratories in the state register, the technical competence of which has been officially confirmed.

Main part

Brief description of the calibration performed:

Calibration is carried out by measuring the flow time using two reference viscometers of the same class, selected depending on the viscometer whose capillary viscometer constant is to be determined.

Area of application/object of calibration:

Capillary viscometer constant

Range:

0,001 - 100 mm^2/s^2

Calibration process

Calibration setup

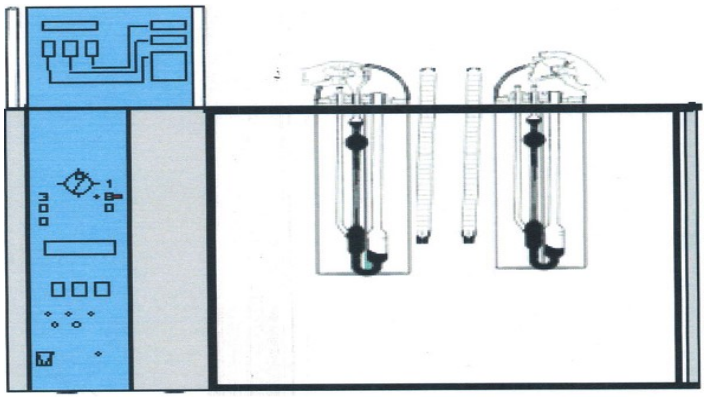


Figure 1. Capillary Viscometer Calibration Setup

EQUIPMENT USED

Name of device	Brand	Type	Serial number	Explanation (period and place of calibiration)
Ubbelohde Viscometer	Psl-rheotek	0	75525	UKAS Calibration Certificate (ISO 17025) 02.12.2021
			73942	
		0C	88545	
			75477	
		0B	94534	
			94540	
		1	95031	
			95032	
		1C	92315	
			93086	
		1B	92979	
			92960	
		2	85473	
			91824	
		2C	95628	
			95627	
		2B	90629	
			92297	
		3	95520	
			95522	
		3C	94444	
			94443	
		3B	72258	
			93743	
		4	94428	
			94433	
		4C	74071	
			74067	

		4B	50875	
			70449	
		5	88534	
			88535	

Device name	Brand	type	Serial number	Explanation (period and place of calibration)
Viscometric bath	Tamson	TV7000 LT	19T024	1 year “UzNIM”
Glass thermometer	TOT IMM AMA	SR5/5OC	84785	TÜBİTAK UME
Glass thermometer	TOT IMM AMA	GP/105C	83741	TÜBİTAK UME
Millikelvin thermometer	Anton Paar	MKT 50	82779658	1 year “UzNIM”
Digital contact thermometer	Tamson	E20	41B108	1 year “UzNIM”
Stopwatch	STOPWATCH	ZSD-808	6079	TÜBİTAK UME
Stopwatch	Tamson	Timer 8-channel	18TT41	TÜBİTAK UME

Description of the calibration process

Determination of flow time

Set the temperature-controlled bath that will be used for calibration to the temperature at which the measurement will be made, then to better observe the temperature deviation, place a glass thermometer or digital contact thermometer in the temperature-controlled bath. Setting the temperature in the thermostat bath with instability in maintaining the set value during calibration ± 0.02 °C.

You can select Newtonian fluids in the table below according to your test viscometer

Name of group	Liquid standard	Flow time
0	0	between 350 - 450
	1	between 600 - 800
	2	between 1000 - 1500
between OC-1A	0	between 180 - 250
	1	between 400 - 600
	2	between 800 - 1200
between II-V	1	between 300 - 500
	2	between 700 - 1200

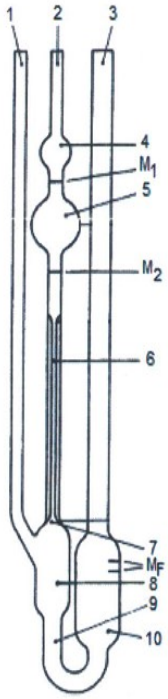
It is possible to select the ubbelohde reference viscometers to match the test viscometers. You must use two viscometers that are the same size. Once the viscometer class has been determined, the flow time should be determined using the liquids taken from the table above using a test and reference viscometers.

1. Place the viscometers on the appropriate holders, which may be provided by the manufacturers.

2. The test liquid should be poured into viscometers to the point between the scaled lines. (MF).

3. Viscometers filled with test liquids should be placed in a bath as shown in Figure 1. Before placing it, check whether the surface is smooth using a water bath (alcohol level). After installation, make sure that the height of the water in the bath is approximately 20 mm above the top liquid of the viscometer.

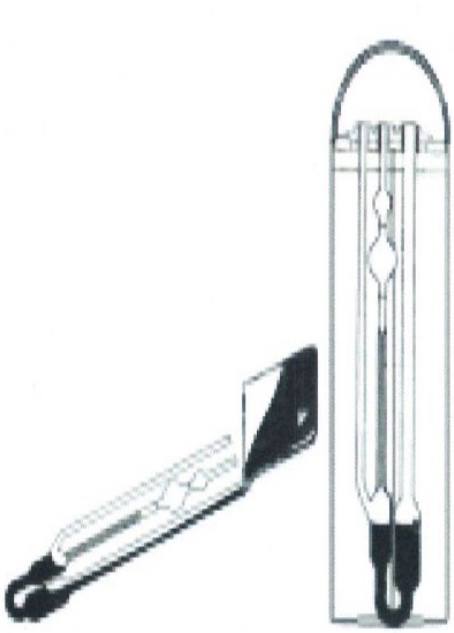
4. Make sure that the viscometers placed in the bath do not touch each other during the measurement. For this purpose also ensure verticality.



1. Ventilation pipe
2. Capillary tube
3. Filling tube
4. Upper reservoir
5. Synchronization lamp
6. Capillary
7. Pendant level lamp
8. Suspended level
9. Compensation tube
10. Bottom reservoir

Ring marks M1 and M2
MF fill out the marks

Figure 2. Viscometer



Ubbelohde

5. Close the viscometer air duct using the device (plug) indicated
In picture 1above.

6. Vacuum the liquid in the capillary tube (labeled 2 in Figure 2) of the viscometer from the top, either with an absorber or with a device, and lift it to approximately the top level of the upper liquid pan (labeled 4). Perform this operation very slowly to avoid the formation of air bubbles in the liquid. When the vacuum operation is finished, close the pipe from above using a device (plug).

7. Open the viscometer air duct (top) and wait until the liquid reaches the bottom of the immersion level marked 8 in Figure 2 (~5 minutes).

8. Open the capillary tube (labeled 2 in Figure 2) of the viscometer again and observe the downward movement of the liquid in the capillary tube and start the stopwatch when it reaches the line marked M1. Stop the stopwatch when the liquid reaches the line marked M2.

9. Save the stopwatch value in the log and repeat the above processes for two viscometers (6, 7, 8, 9) and save 5 flow values. If the flow time is greater than 400s, you can take three measurements. You can omit the first measured flow time and take another measurement.

Formulas and calculations necessary to determine the kinematic viscosity and constant of the Ubbelohde viscometer.

Follow the following processes to calculate kinematic viscosity.

1. Determine the average flow time for each viscometer.

$$t = \frac{1}{n}\sum t_1$$

2. Find the relative difference between the longest and shortest flow times of each viscometer.

$$\varepsilon_t = \frac{t_{\max} - t_{\min}}{t}$$

3. ε_t check the t equation for both two viscometers. If there is no equality, repeat measurement.

$$\varepsilon_v \leq \begin{cases} для 1.10^{-3} v \leq 1000 mm^2/s \\ для 2.10^{-3} v \triangleright 1000 mm^2/s \end{cases}$$

4. The kinematic viscosity values of standard liquids can be determined using standard viscometers. The K0, K1, K2 values are calculated using the formula below using the average flow time (for each liquid we measured).

$$K_0 = \frac{v_0}{t_0}; \quad K_1 = \frac{v_1}{t_1}; \quad K_2 = \frac{v_2}{t_2};$$

5. The given viscometer constants can be used in the expression below and check whether the equation is equal, otherwise the measurement should be repeated using liquids for K_1 and K_2.

$$\frac{|K_1 - K_2|}{t_0} \leq U'_v$$

if the relative uncertainty of the viscosity U'_v values is different, then the average of these values should be taken U'_v and written instead.

6. If the calibrated viscometer is in the range of 0 to 1a, we should see if the requirements below are met.

$$\frac{|K_0 - K_2|}{K_2} \geq 1.5 \cdot \frac{|K_1 - K_2|}{K_2}; \qquad \frac{|K_0 - K_1|}{K_1} \geq 1.5 \cdot \frac{|K_1 - K_2|}{K_2}$$

7. If the requirements are met, the kinetic energy correction must be calculated using the formula below

$$H = K_2 \cdot t_0^3 - v_0 \cdot t_0^2;$$

*If the value of H is positive, we have to find the adjusted flow time using the formula below and the value of K1 is again calculated using the adjusted flow time.

$$t_{1korr} = t_1 \cdot \Delta t_H = t_1 - \frac{H}{v_1 \cdot t_1}$$

As a result, the test viscometer constant is calculated using the formula below.

$$K = \frac{|K_1 + K_2|}{2}$$

Model function

$$S_k = S_{KN} \cdot (\delta S_{Ti} + \delta S_t + \delta S_{incl} + \delta S_{\vartheta\upsilon}$$

S_v	Kinematic viscosity value of calibration fluid
S_k	Constant value of the reference viscometer
δS_{Timer}	Influence of the chronometer used in the measurement
δS_t	Effect of expiration time
δS_{incl}	Tilt effect
$\delta S_{\vartheta\upsilon}$	Effect of temperature changes on measurement

Components of Uncertainty

a) Viscometer constant S'_{KN^2}

Explanations for the uncertainty associated with the reference viscometer

$$(S'_{KN})^2 = \frac{1}{4}(U'_{KN})^2$$

The calibration U'_{KN} uncertainty of the Ubbelohde reference viscometer was calibrated by the UKAS Viscosity Laboratory viscometer, where the coverage factor (volume) is k=2 and the confidence level is 95%.

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