

MECHANICAL MEASUREMENTS

The history of the development of the capillary method for measuring kinematic viscosity: from the Lomonosov viscometer to the information-measuring system

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Published online: 1 February 2024

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Abstract

The article describes the history of the development of capillary method for measuring viscosity measurements and considers the tasks of automation and improvement of this method. A comparative analysis of the currently-relevant vibrational, rotational, and capillary methods of measuring the viscosity of a liquid and the corresponding measuring instruments is also presented. Furthermore, the State verification schedule for the viscosity-measuring instruments, capillary method of measuring viscosity, and glass capillary viscometers used in this scheme are briefly described. The reasons why the capillary method is used in many countries as the predominant high-precision method for measuring the viscosity of liquids are also discussed. A detailed derivation of the equation for calculating the kinematic viscosity of the liquid under study is presented. Herein, an information-measuring system is proposed for measuring the kinematic viscosity of the liquid under study for further development of the capillary method. The operating principle of this system is described, and the implementation strategies are formulated. The accuracy of viscosity measurements of liquid media via the capillary method can be increased by automating measurements and installing certain types of detectors. The results of the analysis are relevant to ensuring the uniformity of viscosity measurements in the Russian Federation.

Keywords Kinematic viscosity · Glass capillary viscometer · Measurement methods · Information and measurement system · Standard

Introduction

Determining the viscosity of various liquid media is an important technical process in many industries, such as medical, petroleum, chemical, and food. Viscosity is defined as the property of a liquid to resist shearing force. The challenge of measuring viscosity has intrigued many outstanding scientists since ancient times and continues to be relevant.

Translated from *Izmeritel'naya Tekhnika*, Vol. 66, No. 8, pp. 53–59, August, 2023. Russian <https://doi.org/10.32446/0368-1025it.2023-8-53-59>

Original article submitted December 28, 2022. Original article reviewed April 24, 2023. Original article accepted July 10, 2023



The objective of the article is to describe the primary methods for measuring viscosity and analyze the history of the development of the capillary method, which is typically implemented in national standards for the unit of kinematic viscosity in different countries. This analysis will provide opportunities to examine the system of concepts, establish the reason for the emergence of the capillary method as the primary technique, and formulate a plan for further improvement of the method of measuring kinematic viscosity using capillary viscometers, considering the experience of previous generations.

Brief historical overview

Leonardo da Vinci (1452–1519) coined the term *surface tension* and used the capillary effect to analyze the movement of water on the earth. The article [1] included a quote from Leonardo da Vinci: “The water in the sandy bank of the river rises on its own, against the nature of gravity, and wets it only because the dryness attracts it.” A scientist attempting to explain the dynamics of the water flow could spend countless hours observing the water [2].

However, the English scientist Isaac Newton (1643–1727) initiated the study of liquid viscosity and introduced the terms *internal friction* and *viscosity*. He observed that when a liquid-filled cylinder rotates continuously, this rotation is gradually transmitted to the entire mass of the liquid. Based on the results of his observations, Newton formulated the law of internal friction [3]:

$$T = \eta D, \quad (1)$$

where T is the tangential stress of internal friction; η is the dynamic viscosity of the liquid; and D is the shear rate of the liquid mass (change in the speed of the liquid in space).

All fluids can be divided into two groups, namely Newtonian (also known as Navier-Stokes fluids) and non-Newtonian. Group 1 includes water, salt or sugar solutions, water-alcohol solutions, silicone oils, and other liquids that obey Newton’s law of viscous friction following Eq. 1, i.e., their viscosity η depends on temperature, pressure, and other influencing factors, which do not include shear rate in laminar flow mode. Group 2 comprises solutions such as starch solutions, polymer solutions, paints, egg whites, toothpaste, mayonnaise, tomato ketchup, petroleum, printing ink, and blood. The viscosity of these fluids depends on the shear stress.

Rotational, vibration, capillary, and other methods are used to measure the viscosity of a Newtonian fluid.

Rotational method. This method of measuring viscosity was developed based on ideas about the relative speeds of movement of concentric layers [4]. According to the Federal Information Fund for Ensuring the Uniformity of Measurements¹, rotational viscometers are the most common measuring instruments (MI) of viscosity, and can be divided according to the operating principle into single-cylinder and coaxial rotational viscometers. Furthermore, there are rotational viscometers with rotors of other (noncylindrical) geometric configurations, such as cone-plane and plane-plane.

Single-cylinder rotational viscometers are represented by a cylinder placed in a specific volume of the test sample, and two vertically aligned coaxial cylinders represent coaxial viscometers. The operating principle of such viscometers [5] is based on the dependence of torque moment M on viscosity:

$$\eta = kM/\omega,$$

¹ Federal Information Fund for Ensuring the Uniformity of Measurements [website]. URL: <https://fgis.gost.ru/fundmetrology/registry/> (access date 07/13/2023).

where k is a coefficient depending on the type (surface shape and geometric size) of the rotor, and ω is the angular speed of the rotor rotation.

For single-cylinder rotational viscometers, the angle of twist of the spring is fixed when the cylinder rotates uniformly, i.e., when the torque generated by viscosity and twist of the spring is in equilibrium. Such a device was developed for the first time by the Russian scientist Fyodor Nikiforovich Shvedov (1840–1905) [6].

When coaxial rotational viscometers operate, the torque is fixed on the rotor axis (moving cylinder), which is placed in an external fixed cylinder filled with the liquid under study. Consideration is given to the torque that develops during uniform movement of the cylinder. M. P. Volarovich² (1900–1987), an eminent Soviet scientist, proposed the design of this device.

The rotational method is considered a precision measurement technique; however, several rotors are required to achieve high precision and expand the range of measured values. However, this method exhibits some disadvantages such as constant increase in the temperature of the test sample due to the rotation of the rotor, affecting the accuracy of the viscosity measurement.

Vibration method. This method of measuring viscosity is based on the law of vibration of a flat plate placed in a small vessel. Oscillations are created by the application of an actuating force (electric current) f that varies according to the law [7]:

$$f = f_0 e^{vt},$$

where f_0 and v are the amplitude and frequency of force oscillations, respectively.

The viscometer plates, placed in the liquid under study, oscillate using a controlled oscillatory circuit. The vibration frequency is constant, but the vibration amplitude varies depending on the frictional force between the plate and the liquid, which is determined by viscosity. The quality factor of the oscillating circuit is converted into an accurate viscosity value using meter calibration factors based on an equation that depends on the plate geometry. Fork viscometer³ is an example of such a device.

Capillary method. This method of measuring viscosity is implemented in capillary viscometers that are part of the standard viscometer installations in many countries.

In the 17th century, the energy of moving water started being utilized widely by industries; consequently, the interest in fluid mechanics increased. In the 18th century, the effects of fluid movement and the laws governing them were established. In this context, Daniel Bernoulli (1700–1782), “Hydrodynamics, or Notes on the forces and movements of fluids” [8] and Leonhard Euler (1707–1783), “Principles of controlling the movement of fluids” [9] were published. These works present the foundations of the hydrodynamics of a perfect fluid and the equations of fluid motion as they are presently applied to the design of pipelines.

At approximately the same time, Russian scientist M.V. Lomonosov (1711–1765) invented the first relative viscometer [10], where he designed a funnel with a certain nozzle diameter in which liquids were compared based on their flow time [11]. However, Lomonosov could not explain the capabilities and nature of this viscometer operation; therefore, the purposes of use and operating principle of this device can only be determined based on the studies by other researchers. In this context, B.N. Menshutkin mentioned, “... devices of this type, certainly, improved, are used for this purpose even now” and suggested that “by the number of drops in a certain period

² Description and instructions for the rotational viscometer RV-8 of the system by prof. M. P. Volarovich (Department of Physics, Moscow Institute of Technology), Ministry of Higher and Secondary Vocational Education, Moscow Peat Institute, Training and Production Workshops, Laboratory of cartography, graphics and copying works of the Scientific Research Institute of Economic Development of the USSR, Moscow (1965).

³ Micro Motion FVM fork viscometer [website]. URL: <https://www.emerson.com/ru-ru/catalog/automation-solutions-ru-ru/measurementinstrumentation-ru-ru/micro-motion-density-viscosity-meter/micro-motion-sku-fvm-ru-ru> (access date 07/13/2023).

of time one can determine the liquid viscosity” [12]. V. Ya. Bilyk, an expert in foundry technology, concluded that Lomonosov’s device “implements the correct idea, namely to characterize the forces of mutual adhesion of liquid particles by drop formation” [11].

All of this served as a solid foundation for one of the most important works of Poiseuille [13], from which an equation was derived that forms the basis of hydrodynamics, where laminar fluid flow through pipelines is considered.

Up to this point, the dynamic viscosity of a liquid medium has been described, which increases with the increase in pressure and temperature of the liquid under study. The dimension of dynamic viscosity according to the International System of Units⁴ and Eq. 1 consists of the dimensions of tangential stress, namely force per unit area or pressure unit [Pa], and shear rate, which is the result of dividing the speed [m/s] by unit length [m], i.e. [s⁻¹], leading to the dimension [Pa·s].

While implementing the capillary method, kinematic viscosity is determined as the ratio of the dynamic viscosity [Pa·s] or [kg/(m·s)] of the liquid to its density [kg/m³], i.e. [m²/s].

Jean Louis Marie Poiseuille, a professor in Physics at the Paris Medical School from 1835–1842, found the relationship between the liquid flow rate Q , the pressure difference p at the ends of the capillary, the length L and the radius R of the capillary [14]:

$$Q = \pi p R^4 / (8L\eta). \tag{2}$$

Equation 2 determines the work spent on moving a certain volume of liquid through a capillary, i.e., overcoming frictional forces and acquiring kinetic energy. Thus, the pressure difference is the difference between the total work and the work expended to obtain kinetic energy:

$$p = P - \frac{\rho Q^2}{\pi^2 R^4} = \frac{8L\eta Q}{\pi R^4},$$

where P is the effective pressure; ρ is the density of the liquid flowing down the capillary.

Then, the value of dynamic viscosity can be expressed as follows:

$$\eta = \frac{\pi P R^4}{8QL} - \frac{\rho Q}{8\pi L}. \tag{3}$$

Equation 3 is valid for the middle part of the capillary; therefore, when determining the dynamic viscosity, it is necessary to consider the following corrections [15]:

- *Correction for the loss of kinetic energy of the liquid*, introduced based on the assumption that all the kinetic energy of the liquid is transferred into thermal energy when leaving the capillary. However, this is only feasible if the capillary ends are cut at right angles to the axis. Further, if the capillary ends are rounded, m is used as a correction factor. The exact value of this coefficient is unknown, but based on several studies, researchers speculated that it is in the range of 0.4–1.12;
- The Couette correction is introduced due to a change in the speed of the liquid stream as it exits the capillary and enters a wider container. The essence of the correction consists of adding a certain value n , proportional to the capillary radius to the capillary length.

⁴ The International System of Units (SI) [website]. URL: <https://www.bipm.org/en/measurement-units> (access date 07/13/2023).

Considering all corrections, Eq. 3 takes the following form:

$$\eta = \frac{\pi PR^4}{8Q(L+nR)} - \frac{m\rho Q}{8\pi(L+nR)}. \quad (4)$$

Using the capillary method, the dynamic viscosity of the test liquid is determined by the time τ of the flow of a constant volume V of this liquid through the capillary of the viscometer [16], i.e., the flow rate can be expressed as:

$$Q = V/\tau.$$

Considering atmospheric pressure, we presented an equation for the effective pressure in the following form:

$$P = gH(\rho - e),$$

where g is the acceleration of gravity; H is the height of the liquid column in the viscometer; e is the air density.

Hence, Eq. 4 takes the following form:

$$\eta = \frac{\pi R^4 \tau g H (\rho - e)}{8V(L+nR)} - \frac{m\rho V}{8\pi\tau(L+nR)}. \quad (5)$$

According to the definition of kinematic viscosity, i.e., dividing the left and right sides of Eq. 5 by the density of the liquid ρ , we obtained the following equation to calculate the kinematic viscosity ν of the medium:

$$\nu = \frac{\pi R^4 \tau g H}{8V(L+nR)} \left(1 - \frac{e}{\rho}\right) - \frac{mV}{8\pi\tau(L+nR)}. \quad (6)$$

In the literature, Eq. 6 is presented as follows for simplicity of understanding [17]:

$$\nu = C\tau(1 - e/\rho) - B/\tau, \quad (7)$$

where C is the viscometer calibration constant; B is the viscometer constant, depending on the loss of kinetic energy by the liquid.

Thus, the capillary method is considered the most developed and theoretically justified technique. Using this method, one can obtain the most accurate value of the viscosity of distilled water at 20 °C and atmospheric pressure 101,325 Pa, which is equal to $1,0034 \cdot 10^{-6} \text{ m}^2/\text{s}$ (ISO/TR 3666:1998⁵). This is a reference value for viscometers in many countries, allowing the capillary method to become the primary method for measuring the viscosity of standards in countries worldwide.

⁵ ISO/TR 3666:1998. Viscosity of water.

Implementation of the capillary method in viscosity standards

In many countries, a similar list is used for technical representation of the primary viscosity standards, such as a set of viscometers with a “hanging level,” a thermostat for establishing and maintaining the temperature of the liquid in viscometers, stopwatches, thermometers, and auxiliary equipment.

The State Primary Standard of Units of Dynamic and Kinematic Liquid Viscosity GET 17-2018 [18, 19] includes standard glass capillary viscometers with capillaries of various diameters as the primary MIs. These viscometers are designed to reproduce, store, and transmit a unit of kinematic viscosity of a liquid following the State verification schedule for MI of liquid viscosity⁶.

GET 17-2018 consists of the following reference complexes (EC)⁷:

- EK GET 17/1-KVI is followed for reproduction, storage, and transmission of kinematic viscosity units in the temperature range of 20 °C–40 °C;
- EK GET 17/2-KVN is followed for reproduction, storage, and transmission of kinematic viscosity units in the temperature ranges of –40 °C to +20 °C and 40 °C–150 °C;
- EK GET 17/3-DVP is followed for reproducing, storing, and transmitting a unit of dynamic viscosity of a liquid in a liquid flow for the temperature range of 20 °C–40 °C and pressure range of 0.5–4.0 MPa;
- EK GET 17/4-DVD is followed for reproduction, storage, and transmission of a unit of dynamic viscosity of a liquid in the temperature range of 20 °C–40 °C and pressure range of 0.1–4.0 MPa.

According to the State verification schedule for MI of viscosity of liquids, the unit of kinematic viscosity is transferred from GET 17-2018 to working standards of category 1. The working standards of category 1 are EK, which consist of the following instruments and devices [19]:

- standard glass capillary viscometers Ubbelohde with a “hanging level”
- thermostatic bath (or thermostat) to establish and maintain the measurement temperature
- MI of liquid flow time, temperature
- auxiliary equipment.

The unit of kinematic viscosity is transferred from the primary standard to the EK to compare with various stable and homogeneous liquids, which are calibration liquids. Calibration liquids with known nominal viscosity values are used for comparison (comparators). From the working standards of category 1, the units of dynamic and kinematic viscosity of the liquid are transferred to the working standards of category 2 to compare with a calibration fluid (comparator) and density unit standards. Working standards of category 2 include standard samples of liquid viscosity (calibration liquids) in the range of permissible certified values at temperatures of –40 °C to +150 °C (kinematic viscosity 4×10^{-7} – $1 \cdot 10^{-1}$ m²/s; dynamic viscosity 4×10^{-7} – 1×10^2 Pa·s) and Stabinger viscometers in the range of permissible certified values at temperatures of –40 °C to +100 °C (kinematic viscosity 2×10^{-7} – 4×10^{-2} m²/s; dynamic viscosity 2×10^{-4} –40 Pa·s) [19].

Using working standards of category 2, the MI of liquid viscosity is verified and calibrated by comparison methods using a calibration fluid (comparator) and direct measurements. The kinematic viscosity of capillary glass viscometers is calculated using Eq. 7, substituting the time interval for a certain volume of liquid through the capillary. Counting the flow time commences when the meniscus of the test liquid overpasses the upper mark applied to the wall of the viscometer capillary and stops when it reaches the lower mark.

In contrast to the rotational and vibrational methods, the capillary method requires the operator to perform all stages of studying a liquid sample, such as determining the moments when the test medium crosses the

⁶ Order of the Federal Agency for Technical Regulation and Metrology (Rosstandart) dated November 5, 2019 No. 2622 “On approval of the State verification schedule for instruments measuring the viscosity of liquids.”

⁷ Research laboratory of state standards in the field of measurements of liquid density and viscosity [website]. URL: <https://www.vniim.ru/nil-2302-gpe.html#get17> (access date 07/13/2023).

marks and starting or halting the timer. In this case, the human factor is one of the sources of uncertainty in determining kinematic viscosity. As proof of this statement, researchers [20] showed a comparative analysis of the results of measurements of the kinematic viscosity of liquid samples in the laboratories of nine different organizations that are the owners of working standards for the unit of kinematic viscosity of a liquid belonging to category 1. The measurement results of one participating laboratory had a significant deviation from the reference value. The authors [20] suggested that this was owing to insufficient qualifications of the operator who performed the measurements.

Thus, the automation of kinematic viscosity measurement requires immediate attention.

Problems of automation of kinematic viscosity measurements and the search for their solutions

In other countries, fully automated viscometers for measuring kinematic viscosity have already been developed. For example, the automatic viscosity measurement system PVS LAUDA (registration number in the state register⁸ 33885-07) or automatic viscometers for measuring kinematic viscosity AKV (registration number in the state register 37803-08⁹), which use an optical detector operating in near-infrared radiation to detect the meniscus of the liquid; and in the HVM 472 viscosity analyzers (registration number in the state register 30086-05¹⁰), an NTC sensor is used. In Russia, a laboratory capillary viscometer with photoelectric indication of the meniscus of the test medium (not received widespread practical use)¹¹ and an automated device for measuring the viscosity of a liquid using the Poiseuille method [21] have been developed. However, these systems are paired with a standard set of capillary viscometers, which does not correspond to research and measurement laboratory tasks because frequent changes of capillaries consume time, and complex calibration is also necessary.

The issues with automated measuring kinematic viscosity are studied at the All-Russian D. I. Mendeleyev Institute for Metrology. Without a ready-made solution for automating the measuring kinematic viscosity, the D. I. Mendeleyev Institute for Metrology specialists proposed partial automation of this process when detecting the liquid meniscus and starting/stopping the timer. This task can be solved using an information-measuring system for measuring kinematic viscosity. For this purpose, the following methods are required: a method for fixing the liquid meniscus; a method for attaching sensors at the required level behind the walls of the thermostat, where the outputs from sensors are combined with stopwatches into a common system; and a program for processing the measured data. Currently, the basic requirements for the developed information-measuring system have been formulated, where the measurement error of time intervals is no more than $+(0.01-0.04)$ s for time intervals of 200–2000 s in the temperature range of -40°C to $+150^{\circ}\text{C}$. Research is also being conducted to determine a method for indicating the meniscus of the liquid under study.

In addition, developing an information-measuring system for measuring the kinematic viscosity of a liquid will reduce the error in time measurement and increase the precision of measuring the viscosity value of the sample under investigation. This innovation will enable to record automatically the moment the liquid meniscus overpasses the marks on the wall of the capillary viscometer, thereby automating the process of starting and stopping the timer (within the digitalization and automation trends). Furthermore, the developed system will be applicable when working with toxic materials that are hazardous to humans and with increased requirements

⁸ Federal Information Fund for Ensuring the Uniformity of Measurements [website]. URL: <https://fgis.gost.ru/fundmetrology/registry/4/items/342616> (access date 07/29/2023).

⁹ Federal Information Fund for Ensuring the Uniformity of Measurements [website]. URL: <https://fgis.gost.ru/fundmetrology/registry/4/items/347427> (access date 07/29/2023).

¹⁰ Federal Information Fund for Ensuring the Uniformity of Measurements [website]. URL: <https://fgis.gost.ru/fundmetrology/registry/4/items/337666> (access date 07/29/2023).

¹¹ Laboratory capillary viscometer [website]. URL: https://yandex.ru/patents/doc/SU221389A1_19681211 (access date 07/29/2023).

for testing conditions (temperature control), as it will permit remote monitoring, processing, and storage of measurement results.

Conclusion

This study aims to develop and investigate an information-measuring system for measuring the viscosity of liquids and clarify the relationship between viscosity and temperature. Automating the process of detecting the meniscus of the liquid under study will avoid the operator's influence on the final measurement result and, thus, increase the accuracy of the measurements. The proposed system will be of great practical importance for improving the uniformity of viscosity measurements in the Russian Federation.

Conflict of interest The author declares no conflict of interest.

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