Designation: E2975 – 23

Standard Test Method for Calibration or Calibration Verification of Concentric Cylinder Rotational Viscometers[1]

This standard is issued under the fixed designation E2975; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

Rotational viscometers have been commonly used for viscosity measurements since the first decade of the twentieth century. After more than one hundred years, there have been many ease-of-use, instrumentation, and data analysis improvements in these instruments. The initial constant torque apparatus gave way to the more popular constant speed apparatus. Spindles became available supplied with calibration constants. Computerization led to factory calibration and automatic viscosity calculation. Even with these improvements, however, apparatus of the very earliest design is still commonly used throughout the world. This standard seeks to provide users with the ability to calibrate or verify calibration of rotational viscosity apparatus in their own laboratory.

1. Scope*

1.1 This test method describes the calibration or calibration verification of rotational viscometers in which the rotational element is immersed in a Newtonian reference material under ambient temperature conditions. The method is applicable to rotational-type viscometers where a constant rotational speed results in a measured torque generated by the test specimen, and to Stormer viscometers where a constant applied torque results in a measured rotational speed. It is not intended for cone-and-plate or parallel plate viscometers.

1.2 Calibration shall be performed with Newtonian reference materials using experimental conditions such as temperature, viscosity range, and shear rate (rotational speed), as close as practical to those to be used for measurement of test specimens.

1.3 *Units*—The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions that are provided for information only and are not considered standard.

1.3.1 Common viscosity units of Poise (P) are related to the SI units by the equivalency 1 cP = 1 mPa·s.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E473 Terminology Relating to Thermal Analysis and Rheology

E1142 Terminology Relating to Thermophysical Properties

E1970 Practice for Statistical Treatment of Thermoanalytical Data

3. Terminology

3.1 *Definitions*—Specific technical terms used in this test method are described in Terminologies E473 and E1142 including *Newtonian, non-Newtonian, stress, strain, viscometer, viscometry*, and *viscosity*.

[1] This test method is under the jurisdiction of ASTM Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.08 on Rheology.

Current edition approved Jan. 1, 2023. Published January 2023. Originally approved in 2014. Last previous edition approved in 2016 as E2975 – 16^{ε1}. DOI: 10.1520/E2975-23.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

*A Summary of Changes section appears at the end of this standard

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *viscometer, Stormer, n*—a rotational viscometer where a constant torque is applied to a spindle and a resultant rotational speed is measured.

4. Summary of Test Method

4.1 A cylindrical spindle is rotated in a Newtonian reference fluid contained in a mating cylindrical container at a known (or measured) speed at a defined temperature. The viscous drag experienced by the immersed element is measured (or known) as torque. Viscosity is proportional to the torque and inversely proportional to the shear rate (see Eq 1). A number of proxies exist for torque and shear rate. For torque, proxies include, but are not limited to, mass (accelerated by gravity operating through a moment arm), and the percent extension of a spring-provided force. For shear rate, proxies include rotational speed in a variety of units including r/min and rad/s, time (for a constant number of revolutions), or number of revolutions (per constant time). A proportionality constant provides for the dimensions of the spindle and unit conversion (such as r/min to rad/s) factors (see Eq 2).

$$\eta = \tau / \sigma \quad (1)$$

$$\eta = E\tau / \acute{\omega} \quad (2)$$

where:

η = viscosity (Pa·s),
$\acute{\omega}$ = rotational speed (r/min),
E = calibration coefficient,
τ = torque (N·m), and
σ = shear rate, S^{-1}.

NOTE 1—1 Pa = 1 N/m^2; 1 cP = 1 mPa·s; 1 r/min = 0.1047 rad/s.

4.2 The dimensions of the calibration constant depend upon the units in which torque (or its proxy) and rotational speed (or its proxy) are observed.

4.3 Modern apparatus with onboard computers often produce the desired measured viscosity directly. In this case, only calibration verification is needed to ensure a properly operating apparatus.

4.4 Calibration or calibration verification of a viscometer and its associated spindle is achieved by comparing the viscosity indicated by the apparatus with that of the known viscosity of a calibration fluid as their quotient using Eq 3, under experimental conditions used in measuring an unknown fluid:

$$C = \eta_t / \eta_o \quad (3)$$

where:

η_t = the viscosity of the calibration fluid (Pa·s),
η_o = the viscosity indicated by the apparatus (Pa·s), and
C = calibration verification factor (dimensionless).

5. Significance and Use

5.1 This test method may be used to calibrate or verify calibration of a rotational viscometer with coaxial spindle geometries.

6. Apparatus

6.1 *Viscometer, Concentric Cylinder Rotational*—The essential instrumentation required providing the minimum rotational viscometer analytical capabilities for this test method include:

6.1.1 A *drive motor*, to apply a rotational displacement to the specimen at a rate from 0.5 r/min to 60 r/min constant to ±0.2 % of full scale or alternatively a torque to the specimen at a rate from 100 r/min to 200 r/min constant to ±0.2 % of full scale.

6.1.2 A *coupling shaft*, or other means to transmit the rotational displacement from the motor to the specimen.

NOTE 2—It is convenient to have a mark on the shaft to indicate the fluid level of the test specimen appropriate for the measurement.

6.1.3 A *cylindrical rotational element, spindle, bob, or tool*, composed of material inert to the material being tested, to fix the specimen between the drive shaft and a stationary position.

NOTE 3—Each spindle typically covers about two decades of viscosity. The spindle is selected so that the measured viscosity is between 10 % and 100 % of the torque range for that spindle.

NOTE 4—This test method is intended for spindles that are immersed in Newtonian viscosity reference fluids contained in a mating cylindrical container. It is not intended for cone-and-plate or parallel plate viscometers.

6.1.4 A *sensor* to measure the torque within ±1 % of full scale developed by the specimen or alternatively to measure rotational speed within ±1 % of full scale.

NOTE 5—For Stormer viscometers, this sensor is sometimes a rotational turns-counter and a timer.

6.1.5 A *temperature sensor* to provide an indication of the specimen temperature of the range of 19 °C to 26 °C to within ±0.1 °C.

6.1.6 A *temperature bath* to provide a controlled isothermal temperature environment for the specimen within the applicable temperature range of this test method.

6.1.7 A *temperature controller*, capable of maintaining the bath at a temperature constant to ±0.1 °C over the range of 19 °C to 26 °C.

6.1.8 A *data collection device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for rotational viscosity are a signal proportional to torque, a signal proportional to shear rate such as rotational speed, temperature, and time.

NOTE 6—Manual recording of measured variables is permitted.

6.1.9 A *stand*, to support, level, lower and raise the drive motor, shaft and spindle.

6.1.10 A specimen *container*, cylindrical in shape suitable for the spindle (6.1.3), to contain the test specimen during testing.

NOTE 7—The specific container may depend upon the spindle being used (see vendor's recommendation). In the absence of other information, a low-form Griffin beaker of 600 mL capacity shall be used.

6.1.11 Auxiliary instrumentation considered necessary or useful in conducting this test method includes: