Designation: E2975 – 16$^{\varepsilon 1}$

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.

ε^1 NOTE—Research report information was editorially added to 15.4 in May 2017.

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 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 and health 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 Dec. 1, 2016. Published January 2017. Originally approved in 2014. Last previous edition approved in 2015 as E2975 – 15. DOI: 10.1520/E2975-16E01.

[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:

6.1.11.1 *Data analysis capability* to provide viscosity, stress, or other useful quantities derived from measured signals.

6.1.11.2 A *level* to indicate the vertical plumb of the drive motor, shaft, and spindle.

NOTE 8—Viscometers and their spindles are precision equipment and shall be kept from undue shock and mishandling. Physical damage to the instrument may reveal itself as erratic torque or rotational speed indication when the instrument, with or without a spindle in place, is operated in air. When operating normally, the indicated signal will be stable and have a value of zero when operated in air.

NOTE 9—Care shall be taken in the storage and handling of spindles and assemblies. Protect them from dust, corrosive deposits, and mechanical abuse. Avoid touching the calibrated section of the spindles with the hands. Clean the spindle and sample container thoroughly after each use.

7. Reagents and Materials

7.1 One or more viscosity reference fluid (with its accompanying certification) in the range of that anticipated for the test specimen measurement.

NOTE 10—Viscosity reference materials are typically available from the viscometer supplier.

8. Preparation of Apparatus

8.1 Perform any viscometer preparation or calibration procedures described by the manufacturer in the operations manual.

8.2 Operate the viscometer in air with a connected spindle in place. The indicator shall be stable and indicate a zero value.

8.3 Set the temperature bath to 23°C and equilibrate for 30 min. Measure the temperature bath and ensure that its temperature is 23 ± 0.2°C.

NOTE 11—Other temperatures may be used but shall be reported.

9. Procedure Preparation

9.1 *Selection of the Spindle:*

9.1.1 From the estimated viscosity of the test specimen, select a spindle that will produce readings in the desired range.

NOTE 12—Where more than one spindle is available for the range selected, choose a spindle that produces results nearest the midpoint of the measurable viscosity (or torque or rotational speed) range.

9.2 *Preparation of the Viscosity Reference Material:*

9.2.1 Place the required amount of the reference material measured in the sample container.

NOTE 13—Pour the reference material slowly down the side of the sample container, taking care to prevent incorporation of air into the material.

NOTE 14—The amount of viscosity reference material varies with each spindle and container combination. See the manufacturer's instruction manual for the correct amount of liquid for each spindle/container pair.

9.2.2 Place the container with its reference material in the temperature bath at 23.0 ± 0.2°C and equilibrate for 30 min (see Note 11).

9.2.3 Record the viscosity of the calibration material from its certificate at the test temperature T as η_t.

9.3 *Assemble the Apparatus:*

9.3.1 Vertically align and level the viscometer on its supporting stand.

9.3.2 Connect the spindle selected in 9.1.1 to the coupling link.

9.3.3 Align the spindle (and apparatus) over the sample container.

9.4 *Spindle Insertion:*

9.4.1 Slowly lower the spindle into the reference material container until the fluid covers the spindle and reaches a level approximately 3 mm above the spindle active area.

NOTE 15—The shaft may have a mark to indicate the appropriate fluid level for measurement.

NOTE 16—Take care not to trap any air bubbles under the spindle.

9.4.2 Initiate the rotation of the spindle at the lowest speed available for 30 min.

9.4.3 Measure the temperature of the test specimen (T).

NOTE 17—If the temperature is not 23.0 ± 0.2°C, allow the test specimen to equilibrate for an addition 30 min or until the desired temperature range and stability are observed.

10. Method A Procedure – For Apparatus Reporting Viscosity at Constant Speed

10.1 Turn on the motor and rotate the spindle at its lowest speed.

10.2 Increase the rotational speed to that required to produce a reading nearest the midpoint of the torque scale.

10.3 Stop the spindle rotation and wait for 1 min.

10.4 Restart the spindle rotation at the same rotational speed and allow at least five revolutions of the spindle.

10.5 Measure the observed viscosity ($\eta_o(1)$) and rotational speed ($\acute{\omega}(1)$).

10.6 Repeat steps 10.3 – 10.5 two more times measuring the indicated viscosity as $\eta_o(2)$ and $\eta_o(3)$, respectively.

10.7 Calculate the mean viscosity value from steps 10.5 and 10.6 and report as η_o (see Practice E1970).

10.8 Using the values from 9.2.3 and 10.7, calculate the calibration verification factor (C) using Eq 4.

NOTE 18—Calibration verification factors outside of the range of 0.95 to 1.05 may indicate that the apparatus needs service.

11. Method B Procedure – For Apparatus Reporting Torque Proxy or Shear Rate Proxies

11.1 Turn on the motor and rotate the spindle at its lowest speed.

11.2 Increase the torque to that required to produce a rotational speed near mid-scale for the range of 100 to 200 r/min.

11.3 Stop the spindle and wait for 1 min.

11.4 Restart the spindle rotation at the same torque and allow at least five spindle rotations.

11.5 Measure and record the rotational speed proxy as $\Omega_o(1)$ and the corresponding torque proxy as $Ŧ_o(1)$.

11.6 Repeat steps 11.3 – 11.5 two more times measuring the rotational speed proxies $\Omega_o(2)$ and $\Omega_o(3)$ and torque proxies as $Ŧ_o(2)$ and $Ŧ_o(3)$.

11.7 Calculate the mean rotational speed proxy and torque proxies from steps 11.5 and 11.6 and report as Ω_o and $Ŧ_o$ (see Practice E1970).

11.8 Determine the calibration coefficient *E* (including the corresponding units) using Eq 5.

NOTE 19—The calibration coefficient will have units that depend upon the units of the proxy torque and proxy rotational speed.

12. Shut Down Procedure

12.1 Remove the spindle from the test fluid by elevating the measurement apparatus on its stand.

12.2 Disassemble and clean the spindle.

NOTE 20—The spindle and sample container may be cleaned with a solvent compatible with the test fluid and the element. Water, xylene, ethanol, or higher alcohols are commonly used.

NOTE 21—Care shall be taken to avoid scratching or deforming the spindle.

12.3 Safely store or dispose of the reference material.

13. Calculation or Interpretation of Results

13.1 For the determination of the calibration verification factor (*C*) from viscosity-reporting apparatus:

$$C = \eta_t / \eta_o \quad (4)$$

where:

E = calibration coefficient,
η_o = observed viscosity (Pa·S),
$Ŧ_o$ = observed torque proxy (such as N-m, torque %, or mass),
Ω_o = observed rotation speed proxy (such as reciprocal time for a fixed number of rotations), and
C = calibration verification factor (dimensionless).

13.2 For the determination of the calibration coefficient:

$$E = \eta_t \Omega_o / Ŧ_o \quad (5)$$

NOTE 22—The units for *E* in Eq 5 depend upon the units of the rotational speed and torque proxies.

13.3 For the determination of an unknown viscosity (η_x) from the observed rotation speed proxy and torque proxy:

$$\eta_x = E Ŧ_o / \Omega_o \quad (6)$$

14. Report

14.1 The report shall include the following:

14.1.1 A complete description of the rotational viscometer, its mode of operation (that is, constant rotational velocity or constant torque), and its spindle;

14.1.2 A complete description of the calibration fluid including its supplier, model number, and serial number (or date of manufacture);

14.1.3 A statement of measurement conditions including temperature and rotational speed (or rotational speed proxy and torque or torque proxy);

14.1.4 Calibration coefficient or calibration verification factor; and

14.1.5 The dated version of this test method used.

14.1.6 *For Example:* calibration constant *E* = (value) at 23°C with (supplier) disk spindle 12.6-mm diameter and 1.8-mm thickness at 60 r/min.

15. Precision and Bias

15.1 An interlaboratory test involving 20 laboratories was conducted in 2015 to establish the within laboratory repeatability of Method A of this standard.[3] Two specimens were examined; a calibration material and a Newtonian test specimen. Participants used nine instrument models from five manufacturers.

15.2 *Precision:*

15.2.1 Within laboratory variability may be described using the repeatability value (*r*) obtained by multiplying the relative standard deviation by 2.8. The repeatability value estimates the 95 % confidence limit. That is, two results obtained in the same laboratory, using the same apparatus, on the same specimen, closely spaced in time have a 95 % probability of being within the repeatability value of each other.

15.2.2 The relative repeatability value (*r*) for the determination of the calibration constant was 0.67 %.

15.2.3 The relative repeatability value (*r*) for a Newtonian fluid was 1.8 %.

15.2.4 No statistically significant difference (at the 95 % confidence level) was observed in within laboratory precision for apparatus from differing vendors.

15.3 *Bias:*

15.3.1 Bias is the difference between a mean determined value and an accepted reference value.

15.3.2 This test method is used to determine the bias in a calibration result. In a perfect system, the calibration verification factor value should be 1.000. Its difference from this unity is the indicator of bias.

15.4 A single laboratory study was conducted in 2016 to establish the within laboratory precision of Methods A and B,[4] in which a rotational viscometer and a Stormer viscometer were calibrated using a certified reference material of about 500 mPa·s.

15.4.1 For Method A, the repeatability relative standard deviation was found to be 0.040 %.

15.4.2 For Method B, using a Stormer viscometer reporting reciprocal time as the rotational speed proxy and mass as the torque proxy, the repeatability standard deviation was found to be 0.43 %.

15.4.3 For Method A, the calibration verification factor was 0.998 corresponding to a bias of –0.2 %. Since this value is less that the repeatability value of 15.2.3, no bias is indicated and no correction of data is needed.

16. Keywords

16.1 calibration; rotational viscometer; viscosity; viscometer; viscometry

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E37-1047. Contact ASTM Customer Service at service@astm.org.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E37-1049. Contact ASTM Customer Service at service@astm.org.

SUMMARY OF CHANGES

Committee E37 has identified the location of selected changes to this standard since the last issue (E2975 – 15) that may impact the use of the standard. (Approved Dec. 1, 2016.)

(1) Addition of an Introduction.
(2) Addition of Procedure for Method B (Section 11), with a corresponding renumbering of the following sections.
(3) Revision of Section 13 on calculations.
(4) Addition of single laboratory precision for Method B.