Designation: E2975 – 15

Standard Test Method for Calibration 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.

1. Scope

1.1 This test method describes the calibration (or performance validation) of rotational viscometers in which the rotational element is immersed in the test fluid under ambient temperature conditions. 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 to inch-pound units 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.*

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*.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *apparent viscosity (η), n*—viscosity determined by this test method.

3.2.1.1 *Discussion*—Because the velocity gradient in this test method may not be the same at all points of the rotational element for non-Newtonian fluids, the result determined may not be the true viscosity. Therefore, the viscosity determined by this test method is called the "apparent viscosity."

4. Summary of Test Method

4.1 An element is rotated in a Newtonian fluid at a known (or measured) speed. The viscous drag experienced by the immersed element is measured (or known) as a torque. Viscosity may then be determined from these properties (torque and rotational speed) and the dimensions of the rotational element by equations such as Eq 1 and 2.

$$\eta = 9.55\, E\, \tau\, S / \dot{\omega} \tag{1}$$

$$S = \left[\left(r_c^2 - r_g^2\right) / \left(4\ \pi\ r_c^2\ r_g^2\ L\right)\right] \tag{2}$$

where:

η = viscosity (Pa·s),
$\dot{\omega}$ = rotational speed (r/min),
E = calibration coefficient (dimensionless),
τ = torque (N·m),
S = rotational element factor (mm^{-3}) supplied by the apparatus vendor,
L = length of the cylindrically shaped rotational element (mm),
r_g = radius of the cylindrically shaped rotational element (m), and
r_c = radius of the cylindrically shaped container (m).

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

4.2 Calibration of a viscometer and its associated rotational element may be achieved by comparing the viscosity indicated by the apparatus with the known viscosity of a calibration fluid as their product using Eq 3, under experimental conditions used in measuring an unknown fluid:

$$E = \eta_t / \eta_o \tag{3}$$

where:

η_t = the viscosity of the calibration fluid (Pa·s), and

[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.

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[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.

η_o = the viscosity indicated by the apparatus (Pa·s).

5. Significance and Use

5.1 This test method may be used to calibrate a rotational viscometer and its associated rotational element.

5.2 The apparent viscosity (η) of a test specimen may then be obtained using Eq 4:

$$\eta = E\,\eta_o \tag{4}$$

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 to 60 r/min constant to ±0.2 % of full scale or alternatively a torque to the specimen 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 rotational element typically covers about two decades of viscosity. The rotational element is selected so that the measured viscosity is between 15 and 95 % of the range that element.

NOTE 4—This test method is intended for rotational elements that are immersed in the test specimen. 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.

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 torque, rotational speed, temperature, and time.

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

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

NOTE 5—The specific container may depend upon the rotational element being used (see vendors 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 rotational element.

NOTE 6—Viscometers and their rotational elements are precision equipment and shall be kept from undue shock and mishandling. Physical damage to the instrument may reveal itself as erratic torque indication when the instrument, with or without a rotational element 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 7—Care shall be taken in the storage and handling of rotational elements and assemblies. Protect them from dust, corrosive deposits, and mechanical abuse. Avoid touching the calibrated section of the rotational elements with the hands. Clean the element 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 8—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 rotational element 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 minutes. Measure the temperature bath and ensure that its temperature is 23 ± 0.2°C.

NOTE 9—Other temperatures may be used but shall be reported.

9. Procedure

9.1 *Selection of the Rotational Element:*

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

NOTE 10—Where more than one rotational element is available for the range selected, choose an element that produces results nearest the midpoint of the measurable viscosity range. Viscometer scale reading shall be within the range of 15 to 95 % of full scale.

9.2 *Preparation of the Viscosity Reference Material:*

9.2.1 Place the required amount of the reference material measured to the nearest 0.5 mL in the sample container.

NOTE 11—Pour the reference material slowly down the side of the sample container taking care to prevent incorporation of air into the material.

NOTE 12—The amount of viscosity reference material varies with each rotational element and container combination. See the manufacturer's instruction manual for the correct amount of liquid for each element/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 minutes (see Note 9).