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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~~ Scope*

1.1 This test method describes the calibration ~~(or performance validation)~~or calibration verification of rotational viscometers in which the rotational element is immersed in ~~the test fluid~~ 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,~~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*.

[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.

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *apparent viscosity (η), viscometer, Stormer, n*—viscosity determined by this test method.a rotational viscometer where a constant torque is applied to a spindle and a resultant rotational speed is measured.

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 A cylindrical spindle is rotated in a Newtonian fluid reference fluid contained in a mating cylindrical container at a known (or measured) speed. speed at a defined temperature. 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 torque. Viscosity is proportional to the torque and inversely proportional to the shear rate (see Eq 1rotational speed) and). 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 rotational element by equations suchspindle and unit conversion (such as Eq 1 and 2.r/min to rad/s) factors (see Eq 2).

$$\eta = 9.55\, E\, \tau\, S / \acute{\omega} \quad (1)$$

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

$$S = [(r_c^2 - r_g^2)/(4 \pi r_c^2 r_g^2 L)] \quad (2)$$

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

where:

η = viscosity (Pa·s),
$\acute{\omega}$ = rotational speed (r/min),
E = calibration coefficient (dimensionless),
E = calibration coefficient,
τ = torque (N·m), and
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).
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 rotational element may be spindle is achieved by comparing the viscosity indicated by the apparatus with that of the known viscosity of a calibration fluid as their productquotient using Eq 3, under experimental conditions used in measuring an unknown fluid:

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

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

where:

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

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 and its associated rotational element.with coaxial spindle geometries.

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

~~$$\eta = E\,\eta_o \quad (4)$$~~

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~~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 ~~rotational element~~ spindle typically covers about two decades of viscosity. The ~~rotational element~~ spindle is selected so that the measured viscosity is between ~~15~~10 % and ~~95 %~~ 100 % of the torque range for that ~~element.~~spindle.

NOTE 4—This test method is intended for ~~rotational elements~~ spindles that are immersed in ~~the test specimen.~~ 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 ~~torque,~~ 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 ~~rotational element.~~spindle.

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

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

NOTE 8—Viscometers and their ~~rotational elements~~ 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 ~~rotational element~~ 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 ~~rotational elements~~ spindles and assemblies. Protect them from dust, corrosive deposits, and mechanical abuse. Avoid touching the calibrated section of the ~~rotational elements~~ spindles with the hands. Clean the ~~element~~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 ~~rotational element~~ 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 minutes.~~ 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.