Designation: ~~E3116 – 18~~ E3116 – 23

Standard Test Method for Viscosity Measurement Validation of Rotational Viscometers[1]

This standard is issued under the fixed designation E3116; 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 provides procedures for validating viscosity measurements by rotational viscometers of Newtonian fluids. Performance parameters determined include viscosity repeatability (precision), detection limit, quantitation limit, linearity, and bias.

1.2 Validation of apparatus performance and analytical methods is requested or required for quality initiatives or where results may be used for legal purposes.

1.3 The values stated in SI units are ~~to be regarded as~~ the standard. No other units of measurement are included in this standard.

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]

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E473 Terminology Relating to Thermal Analysis and Rheology
E1142 Terminology Relating to Thermophysical Properties
E1970 Practice for Statistical Treatment of Thermoanalytical Data
E2161 Terminology Relating to Performance Validation in Thermal Analysis and Rheology
E2975 Test Method for Calibration or Calibration Verification of Concentric Cylinder Rotational Viscometers

3. Terminology

3.1 *Definitions:*

3.1.1 Technical terms used in this standard are defined in Practice E177 and in Terminologies E473, E1142, and E2161 including

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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.

analyte, calibration, Celsius, detection limit, linearity, Newtonian, non-Newtonian, precision, quantitation limit, reference materials, relative standard deviation, repeatability, shear, slope, standard deviation, validation, viscometer, and *viscosity*.

4. Summary of Test Method

4.1 Viscosity is the primary dependent parameter and either torque or rotational speed is the primary independent parameter measured by rotational viscometry. Viscosity is strongly dependent upon temperature and so temperature is usually held constant.

4.2 Viscosity is validated by the direct measurement using a concentric cylinder rotational viscometer at an isothermal temperature using reference materials of known viscosity as an analyte.

4.3 Alternatively, validation of a rotational viscometry method based upon viscosity measurement may be performed using a specific test specimen as the analyte.

4.4 The viscosity of three or more specimens, nominally representing the minimum, midpoint and maximum of the range of the test method, are measured at least in triplicate.

NOTE 1—Repeatability is determined by performing a sufficient number of determinations to calculate statistically valid estimates of the standard deviation or relative standard deviation of the measurements.

4.4.1 Viscosity linearity and bias are determined from the linear regression (best-fit) straight-line correlation of the results from measurements of the three or more specimens.

4.4.2 Viscosity repeatability, detection limits and quantitation limits are determined from the standard deviation of the measurement of three or more analyte-containing specimens.

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

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

$$S = (r_c^2 - r_s^2)/(4 \; \pi \; r_c^2 \; r_s^2 \; L) \tag{2}$$

where:

η = viscosity, Pa-s,
ω = rotational speed, r/min,
τ = torque, N-m,
r_c = radius of the cylindrically shaped container, m,
r_s = radius of the cylindrically shaped spindle, m,
L = length of the cylindrically shaped spindle, m, and
S = instrument or experimental calibration constant, m^{-3}.

NOTE 2—1 Pa = 1 N/m^2; 60 s = 1 min.

5. Significance and Use

5.1 This test method may be used to validate the performance of a specific rotational viscometer apparatus.

5.2 This test method may be used to validate the performance of a specific method based upon the measurement of viscosity using rotational viscometer apparatus.

5.3 This test method may be used to determine the repeatability of a specific apparatus, operator, or laboratory.

5.4 This test method may be used for specification or regulatory compliance purposes.

6. Interferences

6.1 Viscosity is highly dependent upon temperature. Materials may change by as much as 7 % per Celsius degree. For this reason, all measurements shall be made in the same temperature controlled environment.

6.2 This test method assumes that the analyte exhibits Newtonian behavior, that is viscosity is linearly dependent upon shear rate (rotational speed) and a linear applied force (torque). Many materials of interest, however, are non-Newtonian in nature where the measured viscosity varies with shear rate. Care shall be exercised in the selection of a reference material analyte for this test method that is Newtonian in nature.

7. Apparatus

7.1 *Viscometer, Rotational*—The essential instrumental required providing the minimum rotational viscometer analytical capabilities for this test method include:

7.1.1 A *drive motor*, to apply a rotational displacement to the specimen at a rate of 0.2 r/min to 200 r/min constant to ±0.2 % of full scale.

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

NOTE 3—It is convenient to have a mark on the shaft to indicate the fluid level of the test specimen appropriate for the measurement.

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

7.1.4 A sensor to measure the torque within ±1 % of full scale developed by the specimen.

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

7.1.6 A *data collection device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required are torque, rotational speed, temperature, and time.

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

7.2 A specimen *container*, cylindrical or disk in shape, to contain the test specimen during testing.

NOTE 4—The specific container may depend upon the rotational element being used (see vendor's recommendations). In the absence of other information, a low form Griffin beaker of 600-mL capacity shall be used.

7.3 A *temperature bath and controller* to provide a controlled isothermal temperature environment for the specimen within the range of ~~19 to 26°C~~19 °C to 26 °C constant to within ~~±0.1°C.~~±0.1 °C.

7.4 Auxiliary instrumentation considered useful in conducting this test method includes:

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

7.4.2 A *level* to indicate the vertical plumb of the drive motor, shaft and rotational element.

8. Reagents and Materials

8.1 Three or more viscosity reference fluids (with accompanying certificates) representing the minimum, midpoint and maximum viscosity range to be validated by the procedure.

NOTE 5—Viscosity reference materials are typically available from the viscometer supplier.

9. Preparation of Apparatus

9.1 Perform any viscometer preparation or calibration procedures described in the apparatus operations manual or according to Test Method E2975.

9.2 Operate the viscometer in air with a connected rotational element in place. The indicator shall be stable and indicate a zero value.

9.3 Set the temperature bath at ~~25°C~~25 °C and equilibrate for 30 min. Measure the temperature bath and ensure that it is ~~25 ± 0.2°C.~~25 °C ± 0.2 °C.

NOTE 6—Other temperatures may be used but shall be reported.

10. Procedure

10.1 Place the required amount of medium viscosity reference material in the specimen container. Record the reference viscosity value from its certificate as η_{med}REF.

NOTE 7—The amount of viscosity reference material needed varies with each rotational element and container combination. See the instrument operation's manual for the correct amount of liquid for each element-container pair.

NOTE 8—Pour the reference material slowly down the side of specimen container taking care to prevent incorporation of air bubbles into the material.

10.2 Place the container with its reference material in the temperature bath at ~~25 ± 0.2°C~~25 °C ± 0.2 °C and equilibrate for 30 minutes.

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

10.3 Vertically align and level the viscometer motor, shaft and rotational element on its supporting stand over the specimen container.

10.4 Slowly lower the rotational element into the test specimen until the fluid reaches the appropriate level indicated in the operation's manual.

NOTE 10—The shaft may have a mark to indicate the appropriate fluid level for the measurement.

NOTE 11—Ensure that no air bubbles are trapped under the rotational element.

10.5 Initiate the rotation of the rotational element at 0.5 r/min for 30 min.

10.6 Increase (or decrease) the rotational speed to that required to produce a reading nearest the midpoint of the scale.

10.7 Stop the rotation of the element and wait for 1 min.

10.8 Restart the rotational at the same rotational speed (or torque) and allow at least 5 revolutions of the element.

10.9 Measure and record the observed viscosity $\eta_{med}1$ (or torque $\tau_{med}1$), rotational speed $\omega_{med}1$ and temperature $T_{med}1$.

NOTE 12—If the temperature is not ~~25.0 ± 0.2°C,~~25.0 °C ± 0.2 °C, discard the measurement, allow the test specimen to equilibrate for an additional 30 min or until the desired temperature range and stability is observed, and remeasure.

10.10 Repeat steps 10.7 through 10.9 two more times, measuring the indicated viscosity as $\eta_{med}2$ and $\eta_{med}3$ (or torque $\tau_{med}2$ and $\tau_{med}3$), rotational speed $\omega_{med}2$ and $\omega_{med}3$, and temperature $T_{med}2$ and $T_{med}3$.

10.11 Using the three (n_{med} = 3) values for viscosity from steps 10.9 and 10.10, calculate the mean value for η_{med} and σ_{med} (see Practice E1970).

10.12 Repeat steps 10.1 through 10.11 for (n_{max} = 3) the maximum viscosity reference material recording η_{max}REF, $\eta_{max}1$, $\eta_{max}2$, $\eta_{max}3$, $\omega_{max}1$, $\omega_{max}2$, $\omega_{max}3$, $T_{max}1$, $T_{max}2$, and $T_{max}3$.

10.13 Determine the mean viscosity value for ηmax and its standard deviation σ_{max}.

10.14 Repeat steps 10.1 through 10.10 for (n_{min} = 3) the minimum viscosity reference material recording η_{min}REF, $\eta_{min}1$, $\eta_{min}2$, $\eta_{min}3$, $\omega_{min}1$, $\omega_{min}2$, $\omega_{min}3$, $\mathrm{T}_{min}1$, $\mathrm{T}_{min}2$, and $\mathrm{T}_{min}3$.

10.15 Calculate the mean viscosity value for η_{min} and its standard deviation σ_{min}.

10.16 Using the values from 10.11, 10.13, and 10.15 and Eq 3, calculate the viscosity relative standard deviation for RSD_{min}, RSD_{med}, and RSD_{max}.

10.17 Using the values from 10.11, 10.13, 10.15, and 10.16 and Eq 4, calculate the repeatability value *r*.

10.18 Calculate the detectability limit (*DL*) using Eq 5.

10.19 Calculate the quantitation limit (*QL*) using Eq 6.

10.20 Using the reference viscosity values (η_xREF) for the minimum (min), medium (med) and maximum (max) viscosity reference material from steps 10.1, 10.12, and 10.14 as the independent (*X*) values and the three (or more) determined mean viscosity values from steps 10.11, 10.13, and 10.15 as the dependent (*Y*) values, determine by linear regression the slope (*m*), and intercept (*b*) of a correlation best-fit line (see Practice E1970).

NOTE 13—The value of *m* will be close to unity. *b* has the units of viscosity (nominally Pa-s).

10.21 Determine the deviation values (δY_{min}, δY_{med}, and δY_{max}) for each of the mean minimum, medium, and maximum viscosity values using Eq 7 and the values of *m* and *b* from 10.20.

10.22 Determine which deviation value from step 10.21 has the largest absolute value and identify it as $\delta Y_{largest}$.

10.23 Determine linearity (*L*) using Eq 8.

10.24 Determine the bias using Eq 9.

11. Calculations

11.1 Relative standard deviation (*RSD*) is given by Eq 3 (see Practice E1970):

$$RSD = \sigma \times 100\,\% / \text{mean value} \tag{3}$$

11.2 The repeatability (*r*) value is given by Eq 4:

$$r = \left(\left[\{(n_{min} - 1)\,RSD^2_{min}\}+\{(n_{med} - 1)\,RSD^2_{med}\}+\{(n_{max} - 1)\,RSD^2_{max}\}\right]/\left[(n_{min} - 1)+(n_{med} - 1)+(n_{max} - 1)\right]\right)^{1/2} \tag{4}$$