

Designation: E3070 – 22

Standard Test Method for Shear Thinning Index of Non-Newtonian Liquids Using a Rotational Viscometer¹

This standard is issued under the fixed designation E3070; 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 determination of the shear thinning index of a shear-rate dependent (non-Newtonian) fluid using a rotational viscometer. A value of the shear thinning index of unity indicates that the material is Newtonian in behavior. A value greater than unity indicates shear thinning behavior.

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

1.3 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.

1.4 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 E2975 Test Method for Calibration or Calibration Verification of Concentric Cylinder Rotational Viscometers

3. Terminology

3.1 *Definitions*—Specific technical terms used in this standard method are described in Terminologies E473 and E1142 including Celsius, non-Newtonian, rheometry, viscosity, and viscometer.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *shear thinning*, *n*—a decrease in viscosity with increasing shear rate.

3.2.2 *shear thinning index, n*—the ratio of apparent viscosity at two rotational speeds or shear rates.

4. Summary of Test Method

4.1 For Newtonian fluids, viscosity is independent of shear rate. Non-Newtonian fluids are those for which the viscosity changes as a function of shear rate. Many materials of interest are non-Newtonian in behavior.

4.2 The viscosity of a non-Newtonian fluid is measured at different shear rates and the values compared as their ratio. This is known as the shear thinning index. For Newtonian fluids the shear thinning ratio is unity. For non-Newtonian fluids the shear thinning ratio increases with increasing non-Newtonian nature.

4.3 The shear thinning index of non-Newtonian fluids is determined by the ratio of viscosity measurements made at two rotational speeds, preferably a decade apart.

Note 1—The shear thinning index depends upon experimental conditions including geometry and rotational speed. These values shall be reported.

5. Significance and Use

5.1 The flow behavior of many fluids of interest is non-Newtonian in nature. Non-Newtonian behavior is best studied using rheometry apparatus. Nonetheless, estimations on non-Newtonian behavior may be made by recording viscosity at differing rotational speeds (or shear rates) using rotational viscometers.

5.2 The shear thinning index provides a tool for the estimation of the amount of non-Newtonian behavior.

5.3 The shear thinning index may be used in quality assessment, trouble shooting, specification acceptance, and research.

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.

6. Apparatus

6.1 *Rotational Viscometer*—The essential instrumentation required providing the minimum rotational viscometer analytical capabilities include:

6.1.1 A *drive motor*, to apply a unidirectional rotational displacement to the specimen at 5 or more rates of between 0.2 r/min and 200 r/min constant to within ± 1 %.

6.1.2 A *force sensor* to measure the torque developed by the specimen to with ± 1 %.

6.1.3 A *coupling shaft* or other means to transmit the rotational displacement from the motor to the rotational element.

6.1.4 A *spindle, geometry, tool, or rotational element* to fix the specimen between the coupling shaft and a stationary position.

6.1.5 A specimen *container* with the capacity to contain the test specimen during testing.

Note 2—The size of the container used is determined by the size and design of the rotational element used. The container used may be specified by mutual agreement among the parties involved. In the absence of other information, a low form Griffin beaker of 600 mL capacity shall be used.

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

Note 3-Other temperature ranges may be used but shall be reported.

6.1.7 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.

Note 4-Manual acquiring, storing, and displaying of measured or calculated signals is permitted.

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

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

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

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

6.1.9.3 A *guard* to protect the spindle from mechanical damage.

6.2 A *temperature bath* to provide a controlled isothermal temperature environment for the specimen readable to within ± 0.1 °C within the temperature range of 19 °C to 26 °C.

NOTE 5-Other temperatures may be used but shall be reported.

7. Preparation and Calibration of Apparatus

7.1 Perform any viscometer preparation or calibration procedures described in the operations manual.

7.2 If not previously performed, verify the calibration of the viscosity signal using Test Method E2975.

8. Procedure – Determination of Non-Newtonian Behavior and Selection of Experimental Conditions

8.1 Select a rotational viscometer and spindle suited to the viscosity range of the test material such that the spindle

operating at its maximum rotational speed results in a torque reading near full scale torque.

8.2 Install the viscometer as described in the operation's manual.

8.3 Pour a sufficient quantity of the test specimen into the container to cover the immersed spindle to the level indicated by the operations manual.

8.4 Slowly immerse the spindle into the test specimen to the depth recommended by the operations manual.

8.5 Equilibrate the test specimen and container until its temperature rate of change is less than 0.1 °C in 10 min.

8.6 Start the viscometer motor at the 0.2 r/min. Increase the speed until the torque reading is a minimum but greater than 10 % full scale. Maintain this speed for at least 5 spindle rotations. Record the rotational speed and the torque according to the operations manual.

8.7 Without stopping the motor, increase the rotational speed until the torque is approximately 50 % of full scale and maintain this speed for at least 5 rotations. Record the rotational speed, viscosity, and torque.

8.8 Without stopping the motor, increase the rotational speed to the maximum readable torque (up to 100 % full scale) is achieved. Maintain this speed for at least 5 rotations. Record the rotational speed, viscosity, and torque

8.9 Using the rotational speed, viscosity and torque data from steps 8.6 to 8.8, prepare a display of rotational speed on the X-axis versus viscosity on the Y-axis. A non-linear display indicates the shear-rate dependency of the materials.

9. Determination of Shear Thinning Index

9.1 Using the data from Section 8 or by mutual agreement, select a spindle, and a low and a high rotation speeds a factor of 10 apart but within the torque range of 10 % to 100 % full scale.

9.2 Install the viscometer as described in the operation's manual.

9.3 Pour a sufficient quantity of the test specimen into the container to cover the immersed spindle to the level indicated by the operations manual.

9.4 Slowly immerse the spindle into the test specimen to the depth recommended by the operations manual. Equilibrate the test specimen and container until the temperature rate-of-change is less than 0.1 $^{\circ}$ C in 10 min.

9.5 Start the viscometer motor at the low spindle rotational speed from 9.1. After at least 5 spindle rotations, record the rotational speed (*N*1), torque (τ 1), and viscosity (η 1).

9.6 Without stopping the motor, increase the rotational speed to the high speed from 9.1. After a least 5 spindle rotations, record the rotational speed (N2), torque (τ 2), and viscosity (η 2).

9.7 Determine the shear thinning index from the torque values at the two rotational speeds using Eq 1. Report this value and the rotational speeds.

Note 6—Rotational speeds that differ by an order of magnitude are commonly selected (for example, 2 r/min and 20 r/min). The rotational speed for the measurement of shear thinning index shall be selected by agreement among the parties involved.

10. Calculation

10.1 Shear thinning index (STI) is given by Eq 1:

$$STI = \eta 1 / \eta 2 \text{ at } N1 \text{ and } N2 \tag{1}$$

where:

STI = shear thinning index (dimensionless),

 $\eta 1$ = apparent viscosity at the lower rotational speed,

 η^2 = apparent viscosity at the higher rotational speed,

N1 = lower rotational speed, and

N2 = higher rotational speed.

11. Report

11.1 Report the following information:

11.1.1 Complete identification of the test specimen including any conditioning procedure employed in preparation of the test specimen for testing.

11.1.2 Complete identification of the rotational viscometer to including manufacturer, model, and spindle identification.

11.1.3 The shear thinning index at two different rotation speeds and the test temperature. For example:

 $STI(2 \text{ and } 20 \text{ r} / \text{min}) = 80 \text{ mPa-s}/20 \text{ mPa-s} = 4 \text{ at } 23 \text{ }^{\circ}\text{C}$ (2)

11.1.4 The dated version of this test method used. \square

12. Precision and Bias

12.1 An interlaboratory study was conducted in 2021 on a single test specimen in which 9 laboratories participated using 8 instrument models from 5 manufacturers.

12.2 Precision:

deviation by 2.8. The repeatability value estimates the 95 % confidence limit. That is, two values obtained in the same laboratory, using the same apparatus and the same user, on the same specimen, closely spaced in time have a 95 % probability of being within the repeatability value of each other.

12.2.2 Within laboratory relative standard deviation was observed to be 2.2 %.

12.2.3 The repeatability value (r) is 6.0 %.

12.2.4 Between laboratory variability may be described using the reproducibility value (R) obtained by multiplying the reproducibility standard deviation by 2.8. The reproducibility value estimates the 95 % confidence limit. That is, two values obtained in different laboratories, or using different apparatus, or obtained by different users, or distant in time have a 95 % probability of being within the repeatability value of each other.

12.2.5 Between laboratory relative standard deviation was observed to be 14.7 %.

12.2.6 The reproducibility value (R) is 41 %.

Note 7—No attempt was made in this study to specify a common spindle type, rotational speed, or shear rate. Better reproducibility is anticipated if users mutual agree to a common spindle configuration and rotational speed.

12.3 Bias:

12.3.1 Bias is the difference between the determined value and that of the known value for a materials.

12.3.2 There are no reference materials of established shear thinning index known to the committee at this time. Bias of this test method is therefore unknown.

13. Keywords

12.2.1 Within laboratory variability may be described using 13.1 apparent viscosity; rotational viscometer; rotational viscosity; shear thinning index; viscosity 13.1 apparent viscosity; rotational viscometer; rotational viscosity; shear thinning index; viscosity

SUMMARY OF CHANGES

Committee E37 has identified the location of selected changes to this standard since the last issue (E3070 - 18) that may impact the use of this standard. (Approved Jan. 1, 2022.)

(1) Addition of Sections 8 and 9.

(2) Addition in Section 12 of precision and bias information from an interlaboratory study.