Designation: D3835 – 24

Standard Test Method for Determination of Properties of Polymeric Materials by Means of a Capillary Rheometer [1]

This standard is issued under the fixed designation D3835; 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 covers measurement of the rheological properties of polymeric materials at various temperatures and shear rates common to processing equipment. It covers measurement of melt viscosity, sensitivity, or stability of melt viscosity with respect to temperature and polymer dwell time in the rheometer, die swell ratio (polymer memory), and shear sensitivity when extruding under constant rate or stress. The techniques described permit the characterization of materials that exhibit both stable and unstable melt viscosity properties.

1.2 This test method has been found useful for quality control tests on both reinforced and unreinforced thermoplastics, cure cycles of thermosetting materials, and other polymeric materials having a wide range of melt viscosities.

1.3 The values stated in SI units are to be regarded as standard. The inch-pound units given in parentheses are for information only.

NOTE 1—Although this test method and ISO 11443–1995, "Plastic—Determination of the Fluidity of Plastics Using Capillary and Slit-Die Rheometers" differ in approach or detail, the data obtained using ISO 11443, Method A is technically equivalent to this test method

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]

D618 Practice for Conditioning Plastics for Testing

D883 Terminology Relating to Plastics

D1238 Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer

E456 Terminology Relating to Quality and Statistics

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E2935 Practice for Evaluating Equivalence of Two Testing Processes

2.2 *ANSI Standard:*

B46.1 Surface Texture[3]

3. Terminology

3.1 Terms used in this standard are defined in accordance with Terminology D883, unless otherwise specified. For terms relating to precision and bias and associated issues, the terms used in this standard are defined in accordance with Terminology E456.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *apparent values*—viscosity, shear rate, and shear stress values calculated assuming Newtonian behavior and that all pressure drops occur within the capillary.

3.2.2 *critical shear rate*—the shear rate corresponding to the critical shear stress (1/s).

3.2.3 *critical shear stress*—the value of the shear stress at which there is a discontinuity in the slope of log shear stress versus log shear rate plot or periodic roughness of the polymer strand occurs as it exits the rheometer die (MPa).

3.2.4 *delay time*—the time delay between piston stop and start when multiple data points are acquired from a single charge(s).

3.2.5 *melt density*—the density of the material in the molten form expressed in g/mL.

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.30 on Thermal Properties.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at www.astm.org/contact. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

3.2.6 *melt time*—the time interval between the completion of polymer charge and beginning of piston travel(s).

3.2.7 *percent extrudate swell*—the percentage change in the extrudate diameter relative to the die diameter.

3.2.8 *shear rate*—rate of shear strain or velocity gradient in the melt, usually expressed as reciprocal time such as second^{-1} (s^{-1}).

3.2.9 *shear stress*—force per area, usually expressed in pascals (Pa).

3.2.10 *swell ratio*—the ratio of the diameter of the extruded strand to the diameter of the capillary (die).

3.2.11 *viscosity*—ratio of shear stress to shear rate at a given shear rate or shear stress. It is usually expressed in pascal seconds (Pa·s).

3.2.11.1 Viscosity determined on molten polymers is sometimes referred to as melt viscosity.

3.2.11.2 Viscosity determined on materials exhibiting non-Newtonian flow behavior is referred to as apparent viscosity unless corrections are made as specified in Section 11.

3.2.12 *zero shear viscosity, η_0*—the limiting viscosity as the shear rate falls to zero.

4. Significance and Use

4.1 This test method is sensitive to polymer molecular weight and molecular weight distribution, polymer stability—both thermal and rheological, shear instability, and additives such as plasticizers, lubricants, moisture reinforcements, or inert fillers, or combination thereof.

4.2 The sensitivity of this test method makes the data useful for correlating with processing conditions and aids in predicting necessary changes in processing conditions. Unlike Test Method D1238, which makes a one-point measure at a shear rate typically below processing conditions, this test method determines the shear sensitivity and flow characteristics at processing shear rates, and therefore is used to compare materials of different compositions.

5. Interferences

5.1 Relatively minor changes in the design and arrangement of the component parts have not been shown to cause differences in results between laboratories. However, it is important for the best interlaboratory agreement that the design adhere closely to the description herein; otherwise, it must be determined that modifications do not influence the results.

5.1.1 *Temperature*—The effect of temperature variation on output rate, Q, or resultant pressure, P, the other variables remaining constant, is given approximately by:

(A) For a constant-stress rheometer:

$$\% \text{ error in } Q = \frac{dQ}{Q} \times 100 = \frac{E^*}{RT^2}\, dT \times 100 \quad (1)$$

(B) For a constant-rate rheometer:

$$\% \text{ error in } P = \frac{dP}{P} \times 100 = \frac{E^*}{RT^2}\, dT \times 100 \quad (2)$$

where:

E^* = energy of activation,
R = gas constant (8.3 J/K·mol), and
T = absolute temperature, K.

For some thermoplastics $dT = 0.2$ K will produce up to 5 % error in Q or P. Therefore, the temperature control shall meet the requirements specified in 6.1.5.

5.1.2 *Force and Output Rate*—The output rate varies approximately as the pressure, P, raised to some power, b, greater than unity. It is possible that b is not constant over a range of output rates. The effect of pressure variation on output rate, the other variables remaining constant, is given by:

$$\% \text{ error in } Q = \frac{dQ}{Q} \times 100 = b\, \frac{dP}{P} \times 100 \quad (3)$$

Thus a 0.5 % error in pressure measurement implies an error of $b/2$ % in output rate. As the value of b normally ranges from 1 to 3, a corresponding error in Q of 0.5 to 1.5 % could result from this 0.5 % error in P. It is therefore necessary that the precision of the force and output rate measurements be within 1.0 % of the absolute values.

5.1.3 *Capillary Dimensions*—The output rate and force vary with $r^3 + bL - b$, where b is as defined in 5.1.2, r is the capillary radius, and L the length of land. The error that arises in Q due to variations only in r and L is given by:

$$\% \text{ error in } Q = \frac{dQ}{Q} \times 100 = b\, \frac{dP}{P} \times 100 = (3+b)\frac{dr}{r} \times 100 - b\, \frac{dL}{L} \times 100 \quad (4)$$

As the value of b normally ranges from 1 to 3, the resultant error in Q due to a variation in r of ±0.5 % can be 2 to 3 %, and the resultant error in Q due to variation in L of ±0.5 % can be 0.5 to 1.5 %. If Q is being held constant, similar variations in r and L can result in an error of 1.0 to 2.0 % and 0.5 %, respectively, in P.

6. Apparatus

6.1 *Rheometer*—Any capillary rheometer is satisfactory in which it is possible to force molten thermoplastic from a reservoir through a capillary die and in which temperature, applied force, output rate, and barrel and die dimensions are controlled and measured accurately as described as follows. Equipment that operates under constant stress or constant rate has been shown to be equally useful.

6.2 *Barrel*—The barrel (Note 2) shall have a smooth, straight bore. The barrel bore shall be finished by techniques known to produce approximately 12 rms or better in accordance with ANSI B46.1. Care must be taken to ensure that the preheat time is adequate for the barrel diameter to reach temperature homogeneity and that such time is not adversely affecting material degradation.

NOTE 2—Cylinders with Rockwell hardness, C scale, greater than 50 have shown good service life when used at temperatures below 300°C.

6.3 The capillary (Note 3) shall have a smooth straight bore that is held to within ±0.00762 mm (±0.0003 in.) in diameter and shall be held to within ±0.025 mm (±0.001 in.) in length. The bore and its finish are critical. It shall have no visible drill or other tool marks and no detectable eccentricity. The capillary bore shall be finished by techniques known to produce