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Standard Test Method for
Measurement of Properties of Thermoplastic Materials by
Screw-Extrusion Capillary Rheometer[1]

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INTRODUCTION

This test method uses capillary rheometry to measure the rheological properties of thermoplastics and thermoplastic compounds. This test method utilizes a screw-extrusion-type capillary rheometer.

1. Scope *

1.1 This test method covers the use of a screw-extrusion-type capillary rheometer for the measurement of flow properties of thermoplastics and thermoplastic compounds. The measured flow properties, which are obtained through laboratory investigation, may help to describe the material behavior that occurs in factory processing.

1.2 Since a screw-type capillary rheometer imparts shear energy to the material during testing, the measurements will usually differ from those obtained with a piston-type capillary rheometer (see Test Method D 3835).

1.3 Capillary rheometer measurements for thermoplastics and thermoplastic compounds are described in Test Method D 3835.

1.4 The values stated in SI units are to be regarded as standard.

1.5 *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.*

Note 1—There is currently no equivalent ISO standard.

2. Referenced Documents

2.1 *ASTM Standards:*
D 618 Practice for Conditioning Plastics for Testing[2]
D 883 Terminology Relating to Plastics[2]
D 1238 Test Method for Flow Rates of Thermoplastics by Extrusion Plastometer[2]
D 3835 Test Method for Determination of Properties of

Polymeric Materials by Means of a Capillary Rheometer[3]

3. Terminology

3.1 *Definitions* (*See Terminology D 883*) :

3.1.1 *apparent shear rate* ($\dot{\gamma}_a$)—shear strain rate (or velocity gradient) of the thermoplastic or thermoplastic compound extrudate as it passes through the capillary die.

3.1.1.1 *Discussion*—This velocity gradient is not uniform through the cross-section of the capillary die. The shear rate is calculated for the region of highest shear, which is at the wall of the capillary. By selecting a die diameter and controlling the volume flow per unit time through the die, a specific level of apparent shear rate may be achieved. Alternately, the shear stress (entrance pressure) may be controlled, and the apparent shear rate measured.

3.1.1.2 *Discussion*—Mathematically, the apparent shear rate at the wall of the capillary for a Newtonian fluid at the capillary wall is given by the following:

$$\dot{\gamma}_a = \frac{32 \cdot Q}{\pi \cdot D^3} \qquad (1)$$

where:
$\dot{\gamma}_a$ = apparent shear rate, s^{-1},
Q = quantity of fluid extruded per time, mm^3 /s,
π = 3.142, and
D = diameter of the measuring capillary, mm.

3.1.2 *apparent shear stress* (τ_a)—the measured resistance to the flow through a capillary die. It may be determined by measuring the die entrance pressure for a specific die, then applying appropriate geometric factors.

3.1.2.1 *Discussion*—Mathematically, apparent shear stress is given by the following:

$$\tau_a = \frac{P}{4 \cdot (L/D)} \qquad (2)$$

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

where:
τ_a = apparent shear stress, Pa,
P = pressure at the entrance of the measuring capillary, Pa,
L = length of the measuring capillary, mm, and
D = diameter of the measuring capillary, mm.

3.1.3 *apparent viscosity* (η_a)—ratio of apparent shear stress to apparent shear rate, Pa·s.

3.1.3.1 *Discussion*—For an extrusion capillary rheometer, the ratio is usually calculated at a given shear rate. At constant temperature, the apparent viscosity of most polymers is not constant, but varies with shear rate. The viscosity is generally annotated with the shear rate at which the measurement was made.

3.1.4 *capillary rheometer*—an instrument in which thermoplastics or thermoplastic compounds can be forced from a reservoir through a capillary die. The temperature, pressure entering the die, and flow rate through the die can be controlled and accurately measured.

3.1.5 *corrected shear rate* ($\dot{\gamma}_w$)—the actual shear rate at the wall of the capillary die determined by applying the Rabinowitsch correction for non-Newtonian materials, s^{-1}.

3.1.5.1 *Discussion*—The Rabinowitsch correction mathematically adjusts the shear-rate values to compensate for non-Newtonian behavior of the polymer. To obtain corrected shear rate, at least two measurements of apparent shear stress and apparent shear rate are made. This is generally accomplished by increasing the rate of extrusion (Q) while using the same measuring capillary.

3.1.5.2 *Discussion*—As a first step, the Bagley correction (as stated in 3.1.6) is made to the shear-stress values. Then, either by algebraic means (if only two measurements are made), or by a regression method (for a greater number of points), the equation in 3.1.11 is solved for n, using the corrected shear stress (τ_w).

3.1.5.3 *Discussion*—The corrected shear rate ($\dot{\gamma}_w$) is determined by the following:

$$\dot{\gamma}_w = \left[\frac{3_n + 1}{4_n}\right] \cdot \dot{\gamma}_a \qquad (3)$$

For most thermoplastics and thermoplastic compounds, the magnitude of shear sensitivity (n) will vary, depending on material composition.

3.1.6 *corrected shear stress* (τ_W)—the actual shear stress at the wall of the capillary die, Pa. The corrected shear stress is obtained by applying the Bagley Correction Factor (E) to the apparent shear stress (see 3.1.7.1 and 3.1.7.2). The Bagley correction compensates for energy losses at the entrance and exit of the die.

3.1.6.1 *Discussion*—This correction is often applied as though it were an additional length of capillary. The correction is often termed "end effect." Capillary entrance angle and geometry have great influence on the magnitude of this correction.

3.1.6.2 *Discussion*—The Bagley correction will also remove the influence of any static pressure in the system that does not vary with die length.

3.1.6.3 *Discussion*—Since the magnitude of correction is a function of shear rate, data for this correction are obtained by

using two or more dies of different length, but of the same diameter (and thus the same apparent shear rate, as calculated in 3.1.4.2). If the data from these additional dies are compared, either graphically or mathematically, a linear relationship of extrusion pressure with die geometry is usually obtained in the following form:

$$P = c \cdot \left[\frac{L}{D} + E\right] \qquad (4)$$

where:
E = the Bagley Correction Factor. (This term is often called "end effect." It is expressed as the equivalent length of capillary necessary to extrapolate the pressure-line value on the P versus L/D plot to zero, mm.)
c = slope of the line.

The Bagley Correction Factor (E) and the slope (c) are functions of the thermoplastic compound and the shear rate. Corrected shear stress is therefore as follows:

$$\tau_w = \frac{P}{4 \cdot [(L/D) + E]} \qquad (5)$$

3.1.6.4 *Discussion*—Each value of corrected shear stress must be annotated with the shear rate with which it is measured.

3.1.7 *corrected viscosity* (η)—the ratio of corrected shear stress to corrected shear rate, Pa·s.

3.1.7.1 *Discussion*—Since both the material properties and the correction equations are functions of shear rate, it is very important to state the particular value of shear rate at which each measurement is made.

3.1.7.2 *Discussion*—Other corrections to measured values are often made in rheological research studies to compensate for the effects of pressure, viscous heating, compressibility, time effects, etc. The terms "true shear stress," "true shear rate," and "true viscosity" are often used for the results of such exhaustive calculations. This test method addresses only the two most important corrections, Bagley and Rabinowitsch.

3.1.8 *die entrance pressure (P)*—the pressure in the reservoir at the die entrance.

3.1.9 *newtonian fluid*—a material for which the measurement of viscosity is not changed by changing the shear rate. Simple liquids, such as water, are considered Newtonian whereas most polymeric materials are not.

3.1.10 *power-law fluid*—a material for which the viscosity varies with the shear rate in accordance with the following known relationship:

$$\tau_a = K \cdot (\dot{\gamma}_a)^n \qquad (6)$$

where:
τ_a = apparent shear stress, Pa,
$\dot{\gamma}_a$ = apparent shear rate, s^{-1},
K = a material constant, often called "consistency index," and
n = shear sensitivity, dimensionless.

Most non-Newtonian fluids follow this relationship for at least short ranges of the shear rate variable. The power-law equation is generally used in its logarithmic form as follows:

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$$\log (\tau_a) = \log (K) + n (\log (\dot{\gamma}_a)) \qquad (7)$$

3.1.11 *shear sensitivity (n)*—a dimensionless material parameter, also called the "power-law index," that represents the magnitude of the shear sensitivity of a polymer. It is equal to 1.00 for Newtonian fluids, and generally less than 0.8 for non-Newtonian fluids.

3.1.11.1 *Discussion*—Mathematically, the shear sensitivity is given by the following:

$$n = \frac{d \, \log(\tau_w)}{d \log (\dot{\gamma}_a)} \qquad (8)$$

where:
$d \log (\tau_w)$ = the change in log corrected shear stress over varying extrusion rates, and
$d \log (\dot{\gamma}_a)$ = the change in log apparent shear rate over varying extrusion rates.

4. Summary of Test Method

4.1 The thermoplastic material is fed into a laboratory extruder, the barrel of which is equipped with a temperature control. The output end of the extruder is equipped with a capillary die containing an insert of specified dimensions. Temperatures of the extruder barrel and capillary die are normally kept constant. (It may be necessary to alter the die-set temperature only to compensate for shear heating of the material at different extrusion rates.)

4.2 A suitable pressure transducer and temperature-measuring device, such as a thermocouple, are positioned in the die just before the entrance to the insert.

4.3 The rate of material extrusion, or mass throughput (*Q*) is determined by collecting extrudate over a timed interval and then weighing it. Extrusion rate may be controlled by adjusting the drive speed.

4.4 In order to calculate the flow properties of the material, extrusion is performed at a minimum of two different drive speeds through an insert of specified dimensions (Die A). Then, extrusion is performed again, at the same drive speeds, through at least one additional die insert of different specified dimensions (Die B or Die C).

4.5 This procedure allows for the determination of apparent shear rate, apparent shear stress, apparent viscosity, corrected shear stress, corrected shear rate, corrected viscosity, shear sensitivity, and entrance/exit effects.

5. Significance and Use

5.1 This test method is useful for the characterization of thermoplastics and thermoplastic compounds, in terms of viscosity, or resistance to flow.

5.2 The data produced by this test method has been found useful in both quality-control testing and compound development. However, direct correlation with factory conditions is not implied.

5.3 Flow-performance data permits quality control of incoming thermoplastics and thermoplastic compounds because the flow parameters are sensitive to molecular weight and molecular-weight distribution. Therefore, this test method may distinguish differences between lots.

5.4 The shear viscosity or flow viscosity of thermoplastics and thermoplastic compounds will not only be sensitive to the raw-polymer molecular properties, but will also be affected by the type and amount of filler, additive, plasticizer, or stabilizer, by the type of copolymer blend, and by the addition of other compounding materials. This test method can serve as a quality-control tool for either incoming materials or for in-house quality-assurance checks on production mixing. This test method is useful to the research and development of new products in that the rheological behavior of a yet uncharacterized thermoplastic or thermoplastic compound can be measured and considered for comparative analysis.

6. Interferences and Precautions

6.1 Since the flow properties of non-Newtonian materials are not linear, capillary rheometers should be operated at conditions of flow (temperature, pressure, and rate) similar to those of selected commercial processes. These processes include mixing, calendering, molding, and extrusion of thermoplastics and thermoplastic compounds.

6.2 Screw-extrusion-type rheometers impart significant amounts of energy to the thermoplastic or thermoplastic compound before the measurement is made. Interpretation of the data for factory operations such as production extrusion, calendering, or injection molding is therefore more straightforward than for compression-molding operations, where factory-work input is quite small.

6.3 Increasing the rate of extrusion will induce shear heating, and therefore may alter the temperature of the material flowing through the capillary die. It is essential to maintain a constant melt temperature in the die in order to perform accurate viscosity measurements. It may be necessary to compensate for shear heating by manually adjusting the die-heater set temperature.

6.4 Extruder residence time and shearing actions at a particular melt temperature may often affect a material's viscosity. It is recommended that consideration be given to the temperature and shear-stability characteristics of each thermoplastic or thermoplastic compound before using this test method.

7. Apparatus

7.1 A schematic diagram of a screw-extrusion capillary rheometer is shown in Fig. 1. Only those parts essential to the measurement are depicted. Suitable supports, drive components, and fixtures, such as devices for securing the die to the barrel are essential, but are not shown.

7.2 The screw-extrusion system controls both the rate of extrusion and the temperature of the stock at the die entrance.

7.2.1 A single-screw-type laboratory extruder having a barrel diameter of not greater than 31.7 mm nor less than 19 mm is recommended. The length to diameter (*L/D*) ratio of the barrel should not be less than 20:1 nor more than 30:1.

7.2.2 Compression of the stock is accomplished by transport action of the rotating screw. In some extruders, the volume between the screw and the wall, occupied by the polymeric compounds, is less at the end of the barrel than at the feed section. The ratio of the volume in the feed section to the volume at the end of the screw is referred to as the "compression ratio." The compression ratio of the screw should not be less than 2.0:1.0 nor greater than 5.0:1.0. The suggested