Designation: D 3835 – 96

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 D 3835; 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—There is no similar or equivalent ISO 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing[2]

D 1238 Test Method for Flow Rates of Thermoplastics by Extrusion Plastometer[2]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

2.2 *ANSI Standard:*

B46.1 Surface Texture[4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

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

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

3.1.3 *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).

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

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

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

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

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

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

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

3.1.11 *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).

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

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

3.1.12 *zero shear viscosity*, η_0—the limiting viscosity as the

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[1] This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.30 on Thermal Properties (Section D20.30.08).

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[2] *Annual Book of ASTM Standards*, Vol 08.01.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

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 D 1238, 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 can be 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 should 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:

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

(*B*) For a constant-rate rheometer:

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

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 should 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. Over a range of output rates, b may not be constant. The effect of pressure variation on output rate, the other variables remaining constant, is given by:

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

Thus a 0.5 % error in pressure measurement implies an error of $b/2$ % in output rate. As the value of b can range 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:

$$
\begin{aligned}
\%\ \text{error in } Q &= \frac{\mathrm{d}Q}{Q} \times 100 \\
&= b\frac{\mathrm{d}P}{P} \times 100 \\
&= (3 + b)\frac{\mathrm{d}r}{r} \times 100 - b\frac{\mathrm{d}L}{L} \times 100 \qquad (4)
\end{aligned}
$$

As the value of b can range 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 molten thermoplastic can be forced from a reservoir through a capillary die and in which temperature, applied force, output rate, and barrel and die dimensions can be 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 1) shall have a smooth, straight bore between 6.35 and 19 mm in diameter. Well(s) for temperature sensor(s) shall be provided as close to the barrel inside wall as possible. The barrel bore should be finished by techniques known to produce approximately 12 rms or better in accordance with ANSI B46.1.

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 about 12 rms or better when measured in accordance with ANSI B46.1. Dies having a flat (180°) inlet angle and die length to diameter ratios greater than or equal to 20 are recommended. Other inlet angles may be used, but comparisons should be made using only dies with identical inlet cones. The inlet cone shall expand from the capillary at fixed angle to a diameter no less than 50 % of the barrel diameter.

NOTE 3—Hardened steel, tungsten carbide, Stellite, and Hastelloy are the most generally used capillary materials. The capillary shall have a diameter such that the ratio of barrel diameter, D, to capillary diameter, d, is normally between 3 and 15. The length-to-diameter ratio of the capillary shall normally be between 15 and 40. Smaller ratios of L/D may be used in selected situations, but are more likely to result in the necessity of applying large corrections to the data **(1, 2)**.[5]

6.3.1 The precision with which capillary dimensions can be measured is dependent upon both the capillary radius and length. With capillaries of diameter smaller than 1.25 mm (0.050 in.) the specified precision is difficult. Due to the extreme sensitivity of flow data to capillary dimensions, it is most important that both the capillary dimensions and the precision with which the dimensions are measured are known and reported.

6.4 *Piston*—The piston shall be made of metal of a hardness of Rockwell hardness, C scale, of greater than 45. The land of the piston shall be 0.0254 ± 0.007 mm (0.0010 ± 0.0003 in.) smaller in diameter than the barrel and at least 6.35 ± 0.13 mm (0.250 ± 0.005 in.) in length. Alternative piston-barrel-sealing methods (O-rings, split seals, multi-lands, etc.) outside these tolerances may be used, provided there is less than 0.1 g of material going past the sealing device. Machines that measure plunger force must demonstrate that piston-tip frictional effects are less than 1 % over the range of force measurement, or correct for this effect. Demonstration of low frictional force is not required for pressure-measurement devices; however, adequate seals are still needed for proper flow-rate calculations. Above the land, the piston shall be relieved at least 0.25 mm (0.010 in.) less than the barrel diameter. The finish of the piston foot shall be 12 rms when measured in accordance with ANSI B46.1.

6.5 Make provisions for heating and temperature control systems such that the apparatus maintains the temperature of a fluid, at rest, in the barrel to within ±0.2°C of the set temperature (see Note 4). Due to shear heating and chemical or physical changes in the material, it may not be possible to hold this degree of control during an actual test. In such a case, the temperature shall be reported with each data point collected. The temperature specified shall be the temperature of the material 6 min after a full charging of the barrel measured in the center of the barrel 12.7 mm above the top of the die.

NOTE 4—A high melt-flow-rate polypropylene >20 (g/10 min) has been found useful for calibrations of control probes.

6.6 The temperature sensing device in the apparatus shall be calibrated by the following method. A traceable temperature sensor shall be inserted into the rheometer barrel containing a typical charge of material (see Note 5). The combined accuracy of the sensor and display unit shall be 0.1°C or better. The reference unit shall display temperature to 0.1°C or better. The sensor shall be positioned such that it acquires the average temperature centered vertically at 12.7 mm above the top of the die and centered radially within the barrel. For large sensor (for example, large bulb thermometers) elements provisions shall be made to avoid direct contact of the sensing element with the die or barrel wall. Proper insulation or immersion levels, or both, should be adhered to, as required, for sufficient accuracy. Charging the barrel with typical material can be omitted if it has been demonstrated that for the sensor in question the steady-state temperature in air results are statistically equivalent (95 % confidence limits) to the standard charge temperature results. The controlling point temperature device should be calibrated to within ±0.1°C of the reference temperature sensor after steady-state temperature has been achieved. Subsequent temperature checks of the controlling temperature probe should not exceed ±0.2°C of the reference probe temperature. Calibration of the temperature-indicating device shall be verified at a temperature that is within ±25°C of each run temperature.

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[5] The boldface numbers in parentheses refer to the list of references at the end of this test method.

NOTE 5—Any type of temperature sensor (thermometer, RTD, optic probe, etc.) is allowed under 6.1.6 provided it is traceable and falls within the element size restriction and positioning requirements.

7. Test Specimen

7.1 The test specimen may be in any form that can be introduced into the bore of the cylinder such as powder, beads, pellets, strips of film, or molded slugs. In some cases it may be desirable to preform or pelletize a powder. In the case of preformed plugs, any application of heat to the sample must be kept to a minimum and shall be held constant for all specimens thus formed.

8. Conditioning

8.1 Many thermoplastic materials do not require conditioning prior to testing. Materials that contain volatile components, are chemically reactive, or have other unique characteristics are most likely to require special conditioning procedures. In many cases, moisture accelerates degradation or may otherwise affect reproducibility of flow-rate measurements. If conditioning is necessary, see the applicable material specification and Practice D 618.

9. Procedural Conditions

9.1 Typical test temperature conditions of several materials are given as follows. These are listed for information only. The most useful data are generally obtained at temperatures consistent with processing experience. The shear stress and shear rate conditions applied should also closely approximate those observed in the actual processing.

	Typical Test Temperature, °C
Acetals	190
Acrylics	230
Acrylonitrile-butadiene-styrene	200
Cellulose esters	190
Nylon	235 to 275
Polychlorotrifluoroethylene	265
Polyethylene	190
Polycarbonate	300
Polypropylene	230
Polystyrene	190 to 230
Poly(vinyl chloride)	170 to 205
Poly(butylene terephthalate)	250
Thermoplastic Elastomer (TES) Unsaturated	150 to 210
Thermoplastic Elastomer (TES) Saturated	180 to 260

10. Procedure

10.1 Select test temperature shear rates and shear stress in accordance with materials specifications (see the ASTM document for the specific material) and within the limitations of the testing equipment.

10.2 Before beginning determinations, inspect the rheometer and clean it if necessary, as described in 10.11 (see Note 6). Ensure that cleaning procedures or previous use have not changed the dimensions. Make frequent checks to determine the die diameter and to ensure that it is within the tolerances given in 6.1.3. A go/no-go pin with the smallest pin (green) being the low end of the specification (for example, 0.99238 mm for a nominal 1-mm diameter die) and the largest pin (red) being the largest end of the specification (for example, 1.00762 mm for a nominal 1.0-mm diameter die) is effective for checking die diameter. The go (green) pin should go effortlessly all the way into the die from both ends. The no-go (red) pin should not enter more than 1 mm in either end of the die. All errors in pin production should be in the direction of making the specification tighter.

NOTE 6—Experience has shown that an initial purge of the rheometer with the test material is often good practice after periods of equipment inactivity and when changing material types. Purging is also effective at reducing the variability of unstable materials (PVC); it is important, however, that both the barrel and die be cleaned after the purge prior to running the sample.

10.3 Replace the die and piston in the barrel and allow the assembled apparatus to reach thermal equilibrium.

10.4 Remove the piston, place on an insulated surface, and charge the barrel with the sample until the barrel is filled to within approximately 12.5 mm (0.5 in.) of the top. Manually tamp the charge several times during the loading to minimize air pockets. Charging should be accomplished in not more than 2 min.

10.5 Place the piston in the barrel, start the melt time timer, and immediately apply a load that imparts a constant stress on the polymer, or start the piston moving at a constant rate. Extrude, at least, a small portion of the barrel charge. Stop the piston movement until the full melt time has expired.

NOTE 7—There may be cases where 6 min of preheat time may not be sufficient or desirable. Longer preheat periods are permissible and often useful, as are shorter preheat times when proved to be sufficient or necessary due to thermal degradation.

NOTE 8—Running first rates that correspond to forces that exceed the nominal packing force used to charge the sample often results in lower operator-to-operator variability on subsequent rates that correspond to forces lower than the packing force. Additionally, running from higher to lower rates (or stress) tends to reduce the time necessary to achieve steady-state.

10.6 Reactivate the piston to start extrusion. After the system has reached steady-state operation, record the force on the piston and the data necessary to calculate the output rate, Q. The criterion used for steady-state determination should be reported with the data.

10.7 If the specific material being tested has previously been demonstrated thermally stable at the current test temperature, any combination of shear rates or shear stress may be applied, provided data is taken under steady-state conditions.

10.8 If the rheological thermal stability of the material has not been determined, perform either of the following:

10.8.1 Run a constant rate test (or a constant shear stress test in the Newtonian region) with sufficient delay time to cover the expected time for the subsequent multi-point shear rate or shear stress run and collect a minimum of four data points. If the viscosity of the material changes by more than 0.5 % (higher or lower) per minute at any point along the viscosity-time curve, the material is considered thermally unstable rheologically from that point on. Subsequent tests must be performed before this time is reached. If tests must be performed at times exceeding the thermal stability time limit, they must be made at constant time. This requires a new sample to be charged for each rate or stress point collected.

10.8.2 Run a multiple rate or multiple stress level test, or both, in a manner that both rate effects and time effects can be estimated within the same run. The minimum requirements for such a test would be that, at least, one condition (rate or stress) must be repeated and the time difference between them be equal to, at least, half the total test time. Should a 0.5 % change or greater be observed in the viscosity per minute, the rate data should be considered confounded with the time dependence and so noted. The user may then wish to revert back to the previous method to explore the nature of the thermal instability.

10.9 If the percent extrudate swell is desired, measure the extrudate diameter using any NIST traceable device capable of measuring diameters to within ±0.5 %. If measured after cutting a piece of extrudate away from the die, measure the diameter 6.25 mm away from the die exit.

10.9.1 Scanning devices measuring extrudate diameter during a test that are operating at ambient temperature should have the measurement being made 25 mm away from the die exit. At least 8 independent samplings should be used to report an average extrudate diameter. The associated real time shear viscosity data should be collected within 2 s of the real time extrudate measurement. At extrudate exit speeds of less than approximately 200 mm/min, the extrudate should be cut such that its total length is approximately 50 mm at the time of measurement.

10.10 Discharge the remainder of the specimen and remove the capillary from the barrel. Clean the piston and capillary thoroughly and swab out the barrel with cotton cloth patches or a brush softer than the barrel, in the manner of cleaning a pistol barrel. The capillary may be cleaned by dissolving the residue in a solvent. The method of pyrolytic decomposition of the residue in a nitrogen atmosphere is useful only on capillaries made from materials that will not themselves be softened or oxidized by the pyrolysis operation. Place the die in a tubular combustion furnace or other device for heating to 550 ± 10°C and clean with a small nitrogen purge through the die. In certain cases where materials of a given class having similar flow characteristics are being tested consecutively, interim capillary cleaning may not be required. In such cases, however, the effect of cleaning upon viscosity determinations must be shown to be negligible.

11. Procedure for Determination of Melt Density for Thermally Stable Materials

11.1 Set the machine to run under controlled rate to achieve a volumetric flow rate of 0.040 ± 0.030 mL/s (0.07 to 0.01 mL/s). The die diameter and length should be selected to keep drooling from the die at a minimum and to keep average barrel extrusion pressures below 15 MPa.

11.2 Start the test in accordance with 10.1-10.5.

11.3 Let the material flow from the die until the extrudate is bubble free and the force reading is stable.

11.4 Hold a cutting device against the die or fixed member.

11.5 Simultaneously cut the extrudate and start a timing device.

11.6 Carefully collect the extrudate onto a clean surface for a minimum of 20 s.

11.7 End the sample collection by repositioning the cutting device to the same position as in 11.4 then simultaneously cut the extrudate and stop the timing device.

11.8 Report the actual collection time (time between cuts) to 0.01 s with a precision of 0.01 s or better.

11.9 Report the mass of material collected to 0.01 g with a precision of 0.01 g or better. If the total sample mass is less than 1.0 g, increase the collection time to achieve an extrudate weight greater than 1 g.

11.10 Repeat 11.3-11.8 until three bubble-free extrudates are collected.

11.11 Calculate the melt density from the following equation:

$$\rho = \frac{m}{tQ} \tag{5}$$

where:

ρ = melt density, g/mL,
m = mass of the extrudate collected, g,
t = extrudate collection time, s, and
Q = volumetric flow rate, mL/s.

The volumetric flow rate, Q, shall be calculated from the product of ram speed in cm/s and barrel cross sectional-area in cm^2.

11.12 Calculate an average melt density and extrusion pressure from the three samplings.

NOTE 9—The results from this test method should be used with caution for PVC, anomalous results have been observed with regards to temperature dependence.

12. Errors and Corrections (See Refs (4-9)

12.1 In some cases it is necessary to have more exacting rheological data from capillary rheometry measurements. In this event, data may be reported in different terms than given in Section 3. For example, true shear rates, corrected for non-Newtonian flow behavior and true shear stresses, corrected for end effects or kinetic energy losses, may be calculated. In such cases, the exact details of the mode of correction must be reported. The application of these corrections is discussed in the references at the end of this test method.

12.2 *Capillary Calibration*—No completely satisfactory method for determining capillary inside diameter has yet been developed. Since apparent viscosity varies with the fourth power of r, it is desirable to know this value within ±0.00762 mm (0.0003 in.).

12.3 *Piston Friction*—This is caused by contact of the piston with the barrel. Normally the frictional force is negligible compared to the pressure drop through the capillary. When significant, the frictional force should be subtracted from the force reading.

12.4 *Polymer Back Flow*—The clearance between the plunger and the barrel may permit a small amount of melt to flow back along the piston instead of through the capillary. This causes the real shear rate to be lower than that calculated from the piston velocity. Usually this error is negligible. However, in some cases, particularly when slow piston speeds are run at high loads, a back-flow correction may be necessary. This is evidenced by material exuding past the top of the land on the piston. This material should be scraped from the plunger, weighed, and compared to the weight of the capillary extrudate for the same time period to determine the percent back-flow error. A second method for determining the magnitude of this error consists in measuring the rate of capillary extrudate and comparing this with the actual piston displacement rate, taking into account the change in fluid density.

12.5 *Melt Compressibility*—Some fluids are compressible to a significant degree. As shear rate at the capillary wall is calculated from the piston displacement rate, an error is introduced by the drop in hydrostatic pressure (and in fluid density) along the capillary. As the hydrostatic pressure diminishes along the capillary, the fluid density decreases and the flow rate increases. This results in an increase in shear rate down the capillary. If the compressibility or the equation of state for the material under study is known, this correction can easily be made; for example, using a published equation of state for polystyrene **(3)**, a compressibility correction chart can be made for this material.

12.6 *Barrel Pressure Drop*—It is assumed in most work that the pressure drop in the rheometer barrel is negligible compared to the pressure drop through the capillary. This is not true for short capillaries of large diameter. Under isothermal conditions, the pressure drop of Newtonian materials varies as

$$\frac{\Delta P_1}{\Delta P_2} = \left(\frac{L_B}{L_C}\right)\left(\frac{R}{r}\right)^4 \tag{6}$$

where L_B refers to the rheometer barrel length and L_C to the capillary length. When the pressure drop in the barrel is significant, it should be subtracted from the overall pressure drop of the system in order to calculate shear stress.

12.7 *Determining True Shear Stress*—The correction method according to Bagley will be used to calculate true stress. To obtain the true shear stress, perform the following procedure: Using a minimum of two dies (although preferably three or more) having the same entrance angle and same diameter (*D*) yet of differing capillary lengths (*L*), collect steady-state flow data on shear rate and test pressure (or plunger force). At least one *L/D* ratio should be less than 10, and at least one should be greater than 16. Prepare a plot of pressure (or plunger force) versus the length to diameter (*L/D*) ratio of the dies used. For points at constant apparent shear rate, draw the best straight line through the data and determine the intercept with the pressure axis (P_c) or force axis (F_c). Obtain true shear stress using the following equation:

$$\tau = \frac{(P - P_c)D}{4L} = \frac{(F - F_c)D}{4L\,A_B} \tag{7}$$

where:

τ = true shear stress,
P = melt pressure,
P_c = intercept obtained for a given shear rate from the above described plot (see Fig. 1),
D = die diameter, and
L = die length.

For plunger force measuring devices, *F* is the force on the plunger, F_c is the intercept force on the Bagley plot described above, and A_B is the cross-sectional area of the barrel. Devices that measure plunger force must acquire data for a given shear rate (a given line on the graph) at the same position in the barrel for the various dies used. In this way barrel pressure drop effects will be removed along with the other stationary pressures in the system when the Bagley correction is performed.

NOTE 10—When using very long dies, there may be nonlinear changes in the pressure versus *L/D* plots due to the effects of pressure on viscosity or viscous heating. In such cases use only the data from shorter capillaries which do not exhibit the effect.

NOTE 11—The Bagley correction may be performed using computer programs. If it is performed in such a manner, inherent in the computer program will be code assessing the validity of the assumption of having straight lines in the Bagley plot. Users will be warned that the Bagley correction is not valid under such circumstances where the straight line conditions are not met.

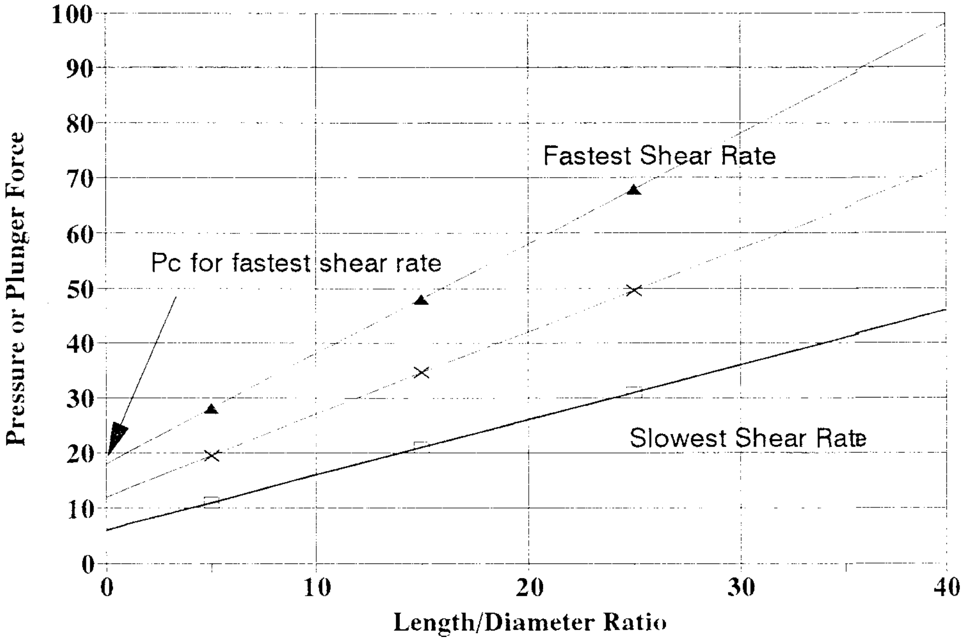

FIG. 1 Bagley Correction

12.8 *Determining True Shear Rate*—The Weissenberg Rabinowitsch shear rate correction accounts for the fact that the true shear rate is often larger than the apparent shear rate for non-Newtonian materials. The true shear rate can be calculated using the following equation:

$$\dot{\gamma} = \frac{(3n + 1)}{4n} \dot{\gamma}_a \tag{8}$$

where:

n = tangent slope of the log true shear stress versus log apparent shear rate curve at the apparent shear rate being corrected,
$\dot{\gamma}$ = true shear rate, and
$\dot{\gamma}_a$ = apparent shear rate described in 13.1 (see Fig. 2).

The stationary pressure correction (Bagley entrance correction) should always be performed prior to the Rabinowitsch correction.

13. Calculation

13.1 Perform calculations using the following equations:

$$\text{Shear stress, Pa} = \frac{Pr}{2L} = \frac{Fr}{2\pi R^2 L} \tag{9}$$

$$\text{Shear rate, s}^{-1} = \frac{4Q}{\pi r^3} = \frac{4V}{\pi r^3 t} \tag{10}$$

$$\text{Viscosity, Pa·s} = \frac{p\pi r^4}{8LQ} = \frac{Fr^4 t}{8R^2 LV} \tag{11}$$

where:

P = pressure by ram, Pa,
F = force on ram, N,
r = radius of capillary, m,
R = radius of barrel, m,
L = length of capillary, m,
Q = flow rate, m^3/s,
V = volume extruded, m^3, and
t = extrusion time, s.

13.1.1 The equations given in 13.1 yield true shear rate and true viscosity for Newtonian fluids only; for non-Newtonian fluids, the apparent shear rate and viscosity are obtained. (See Section 12.)

13.2 Calculate swell ratio and percent memory as follows:

$$\text{swell ratio} = \frac{\text{strand diameter}}{\text{capillary diameter}}$$

$$\text{\% extrudate swell} = \frac{\text{strand diameter} - \text{capillary diameter}}{\text{capillary diameter}} \times 100$$

14. Report

14.1 Report the following information:

14.1.1 *Information Other Than Flow Data*:

14.1.1.1 Description of the material being tested,

14.1.1.2 Description of the rheometer used,

14.1.1.3 Temperature at which the data were obtained and the precision of the temperature measurement (°C),

14.1.1.4 Diameter, *d*, and the length to diameter ratio, *L*/*d*, of the straight section, and the precision of these measurements (mm),

14.1.1.5 Die-entry-cone maximum diameter and angle,

14.1.1.6 Statement as to any preconditioning which the sample has undergone, and

14.1.1.7 Melt time and dwell times (s).

14.2 *Flow Data*—These data should be reported in tabular or graphical form, stating either “apparent values,”“ Rabinowitsch corrected,” “Bagley corrected,” or “Rabinowitsch and

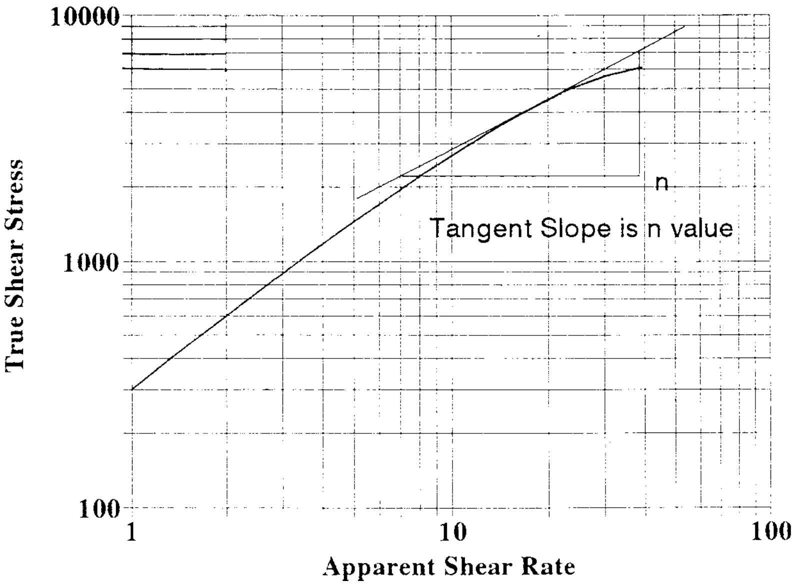

FIG. 2 Weissenberg Rabinowitsch Correction for True Shear Rate

Bagley corrected." If no die-wall slippage was assumed in the Rabinowitsch correction, it should be noted. Corrections of other types should be noted if greater than 1 %.

14.2.1 Log shear stress versus log shear rate,

14.2.2 Log viscosity versus log shear stress or log shear rate,

14.2.3 Log viscosity versus the reciprocal of the absolute temperature at a constant shear stress or shear rate,

14.2.4 Log viscosity versus the temperature in degrees Celsius at a constant shear stress or shear rate,

14.2.5 Log critical shear stress or log critical shear rate versus the reciprocal of the absolute temperature, and

14.2.6 Log critical shear stress or log critical shear rate versus the temperature in degrees Celsius.

14.3 Individual data obtained at a single set of test conditions should include the following information:

14.3.1 Shear stress, τ, Pa,

14.3.2 Shear rate, $\dot{\gamma}$, s^{-1},

14.3.3 Intrinsic melt viscosity (see Appendix X1), η_a, Pa·s,

14.3.4 Melt viscosity stability, δ (%/min),

14.3.5 Percent extrudate swell or swell ratio.

14.4 *Visual Observation*—In cases where observation is possible, gloss character or melt fracture and distortion of the monofilament may be noted at or above a certain shear stress. These values may correspond to a critical shear stress. The data shall be reported separately as "visual" critical shear stress. In addition, the general color of the extrudate at the conditions of test or the dwell time at which a distinct color change occurs, or both, can be noted.

14.5 *Melt Density Results (if performed)*:

14.5.1 The average melt density, g/mL,

14.5.2 The barrel extrusion pressure, MPa,

14.5.3 If the standard flow rate of 0.04 mL/s or minimum cut time of 20 s are not followed, report the flow rate, mL/s, and nominal cut time, s, and

14.5.4 All items in 14.1.1-14.1.1.7 must be reported with melt density results.

15. Precision and Bias[6]

15.1 *Precision*:

15.1.1 Fig. 3 and Table 1 are based on a round robin conducted in 1992 in accordance with Practice E 691, involving materials tested by 13 laboratories. Three materials were used in the round robin: polypropylene copolymer, polystyrene, and low-density polyethylene. Each material was prepared by a single source and underwent no additional conditioning (drying, etc.) prior to testing. The number of measurements made by a given laboratory is noted in the tables. Typically each laboratory ran three tests per material. It should be noted that the full-scale capacity of the pressure transducer or load cell and the proper die selection can significantly affect the ability to measure at low rates (low stresses).

NOTE 12—The following explanations of r and R are intended to present a meaningful way of considering the approximate precision of this test method. The data in Table 1 should not be rigorously applied to the acceptance or rejection of material, as those data are specific to the round robin and may not be representative of other lots, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their laboratory and materials or between specific laboratories.

15.1.2 *Concept of r and R*—If S_r and S_R have been calculated from a large enough body of data, and the test result of interest is that obtained from a single viscosity versus rate sweep, then the following applies:

15.1.2.1 *Repeatability (r)*—Comparing two results for the same material, obtained by the same operator using the same equipment on the same day, the two test results should be judged not equivalent if they differ by more than the r value, where $r = 2.8\ S_r$.

15.1.2.2 *Reproducibility (R)*—Comparing two results for

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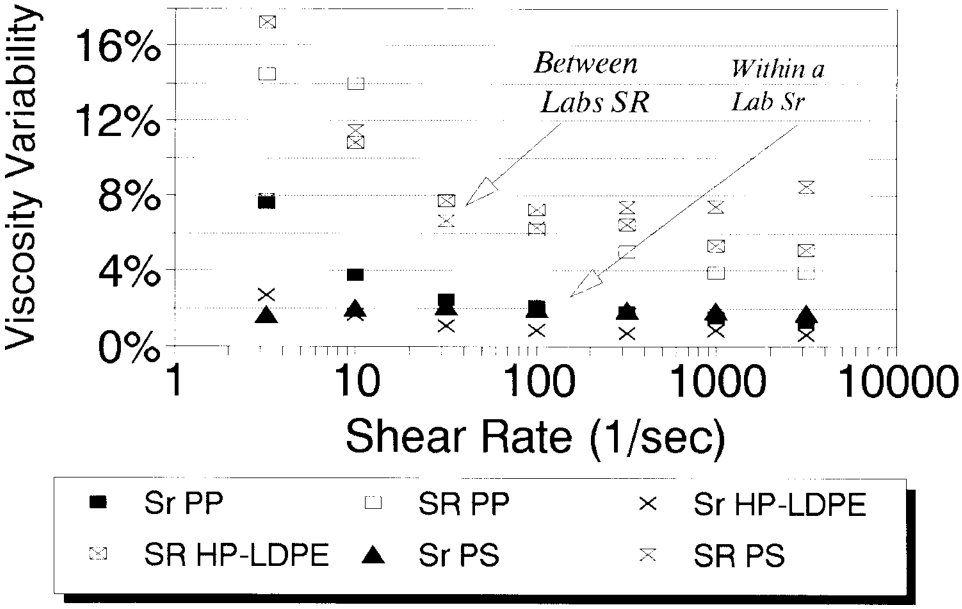
[6] Supporting data giving results of the interlaboratory tests have been filed at ASTM Headquarters. Request RR: D-20-1076.

TABLE 1 Summary of Round Robin for Test Method D 3835 Conducted in 1992

			Points Used		Points Used		Points Used		Points Used		Points Used		Points Used		Points Used		Points Used
PP	Set Rate, 1/s	3162	...	1000	...	316	...	100	...	32	...	10	...	3.2	...	100	...
	Stress, kPa	150	...	103	...	70	...	44	...	24	...	12	...	5	...	43	...
	Viscosity, Pa·s	47.3	...	103.2	...	222.3	...	440.5	...	734.5	...	1249.2	...	1544.9	...	434.9	...
	S_R, Pa·s[A]	1.8	13	4.0	13	11.2	12	27.3	13	56.5	13	174.3	12	223.1	7	30.7	10
	S_r, Pa·s[B]	0.6	36	1.6	36	4.0	33	9.4	36	18.3	36	47.3	33	117.0	18	8.5	30
	S_r/average,%	1.28	...	1.52	...	1.81	...	2.13	...	2.49	...	3.79	...	7.57	...	1.95	...
	S_R/average,%	3.89	...	3.91	...	5.05	...	6.20	...	7.69	...	13.95	...	14.44	...	7.05	...
HP-LDPE	Set Rate, 1/s	3162	...	1000	...	316	...	100	...	32	...	10	...	3.2	...	100	...
	Stress, kPa	320	...	214	...	139	...	83	...	48	...	28	...	15	...	82	...
	Viscosity, Pa·s	101.2	...	213.7	...	439.3	...	834.0	...	1509.1	...	2837.3	...	4651.6	...	817.9	...
	S_R, Pa·s[A]	5.2	13	11.4	13	28.3	12	60.3	13	116.3	13	307.7	12	802.5	8	46.4	10
	S_r, Pa·s[B]	0.7	36	1.8	36	2.9	33	7.2	36	16.5	36	48.8	33	125.7	21	5.1	30
	S_r/average,%	0.65	...	0.84	...	0.66	...	0.86	...	1.09	...	1.72	...	2.70	...	0.63	...
	S_R/average,%	5.10	...	5.32	...	6.45	...	7.23	...	7.71	...	10.85	...	17.25	...	5.67	...
PS	Set Rate, 1/s	3162	...	1000	...	316	...	100	...	32	...	10	...	3.2	...	100	...
	Stress, kPa	299	...	220	...	163	...	121	...	84	...	59	...	37	...	118	...
	Viscosity, Pa·s	94.5	...	220.3	13	516.9	...	1207.6	...	2638.0	...	5928.4	...	11 696.5	...	1179.0	...
	S_R, Pa·s[A]	8.0	13	16.4	35	38.0	12	76.0	13	175.7	13	679.7	12	907.3	8	76.1	10
	S_r, Pa·s[B]	1.6	35	4.2	...	9.9	32	23.9	35	55.6	35	122.7	32	197.6	21	18.4	29
	S_r/average,%	1.74	...	1.90	...	1.92	...	1.98	...	2.11	...	2.07	...	1.69	...	1.56	...
	S_R/average,%	8.51	...	7.43	...	7.35	...	6.30	...	6.66	...	11.47	...	7.76	...	6.45	...

[A] S_R = between laboratory standard deviation.
[B] S_r = within-laboratory standard deviation (pooled estimate).



NOTE 1—By material, within-laboratory and laboratory-to-laboratory.

FIG. 3 Variability versus Shear Rate

the same material, obtained by different operators using different equipment on different days, the two test results should be judged not equivalent if they differ by more than the *R* value, where $R = 2.8\ S_R$.

15.2 Any judgment pertaining to the repeatability or reproducibility would have an approximate 95 % (0.95) probability of being correct.

15.3 *Bias*—There are no recognized standards by which to estimate the bias of this test method.

16. Keywords

16.1 capillary; plastics; polymers; rheology; thermal flow; viscosity

APPENDIXES

(Nonmandatory Information)

X1. PROCEDURE FOR DETERMINATION OF INTRINSIC MELT VISCOSITY AND MELT FLOW STABILITY

X1.1 Measure the melt viscosity at constant conditions after at least four dwell times in the barrel.

X1.2 Plot the four or more melt viscosity values on semilogarithmic paper with viscosity plotted on the log scale and dwell time on the linear scale (see Fig. X1.1 and Fig. X1.2). In most cases these data will fall on a straight line. A single data point that does not fall on the line drawn through the other data points can be attributed to polymer heterogeneity or test techniques and can be discarded.

X1.3 Draw a straight line through the data and extrapolate to the *y* axis (corresponding to dwell time = v 0). The melt viscosity value thus defined by the intercept of the data line should be recorded as *intrinsic melt viscosity.* This parameter has been found to correlate with polymer molecular weight average, as defined by solution techniques for linear polymers.

X1.4 Calculate the slope of the best fit line to obtain the rate of change of the viscosity as a function of time at a specified temperature. This rate shall be called the *melt viscosity stability*, *S*, of the material at the conditions of test (Note X1.1 and Note X1.2).

NOTE X1.1—The total dwell times for viscosity measurement should be selected according to the stability of the material. A highly unstable material can be accurately characterized for its stability factor in relatively short times (for example, 10 min). A material exhibiting small changes in viscosity may require 20 to 30-min dwell times to accurately define the rate of viscosity change.

NOTE X1.2—In the case of materials such as PVC, the material often exhibits stable flow for an initial period of time until the stabilizer becomes ineffective and unstable flow commences. In cases such as this, the dwell time at which unstable flow initiates can be determined and the effectiveness of the stabilizer can thus be defined.

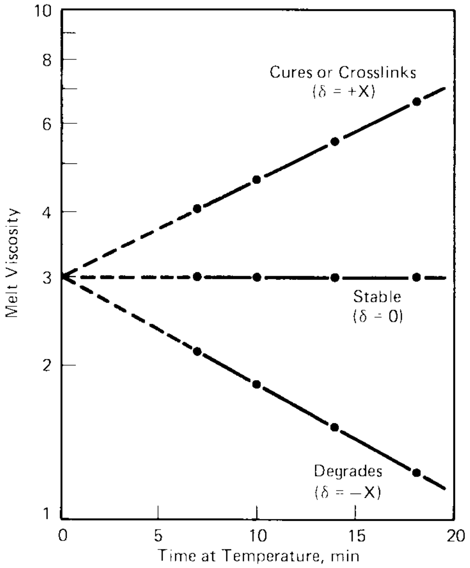


FIG. X1.1 Determination of Intrinsic Melt Viscosity and Stability Factor, δ

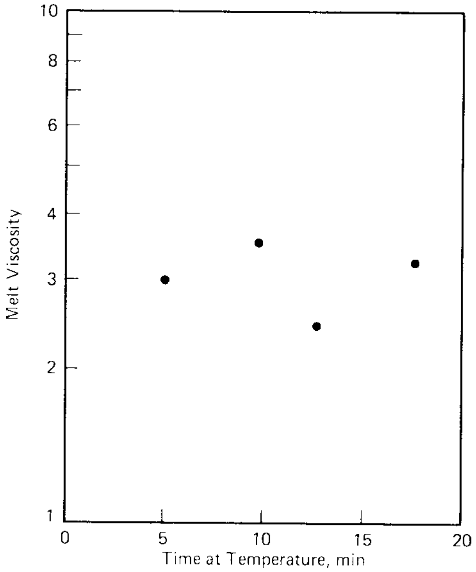


FIG. X1.2 Example of Flow Data Obtained on Heterogeneous Material

X2. STEADY-STATE ALGORITHM PROCEDURE

X2.1 The following is a description of a suggested and nonmandatory algorithmic process to determine steady-state flow conditions during capillary rheometry.

X2.2 *Terminology:*

X2.2.1 *acquisition rate*—the time between acquired plunger force or barrel pressure values in hertz (1/s).

X2.2.2 *acquisition volume*—the volume of material displaced by the plunger, assuming a perfect seal (that is, based on barrel diameter and plunger speed), during the acquisition of the data currently under consideration.

X2.2.3 *minimum volume element (MVE)*—the smallest amount of volumetric flow on which a valid moving average can be generated. A value of 0.02 mL is suggested.

X2.2.4 *pairwise force difference (PFD)*—the difference between two consecutive plunger force or barrel pressure values (for example, where i is the current point and F_i is the current plunger force, then the pairwise force differences are $\{F_i - F_{i-1}\}$, $\{F_{i-2} - F_{i-3}\}$ etc.).

X2.2.5 *average pairwise force difference (APFD)*—the sum of the pairwise force differences divided by the number of differences performed.

X2.2.6 *average force (AF)*—the average plunger force or barrel pressure value over a specific acquisition volume.

X2.2.7 *NPTS*—the number of acquired data points considered in the current acquisition volume.

X2.3 *Criterion Necessary for Analysis and Acquisition:*

X2.3.1 Delete or ignore all data acquired which is less than 0.5 % of the plunger force or barrel pressure measurement device's full-scale capacity (for example, 5 lb for a 1000-lb plunger force load cell).

X2.3.2 The analog to digital conversion resolution must be less than or equal to 0.05 % of the plunger force or barrel pressure measurement device's full-scale capacity (for example, 12 bit is adequate, ±0.5 lb on 2000-lb plunger force load cell).

X2.3.3 The acquisition rate shall not exceed 1/{3* rise time of the plunger force or barrel pressure measurement device and electronics system}.

NOTE X2.1—The rise time of the plunger force or barrel pressure measurement device and electronics system is available from the capillary rheometer equipment manufacturer.

NOTE X2.2—To keep the acquired values independent of one another, the acquisition rates should be no faster than about 2000 Hz for a load cell. A typical capillary Hg-filled pressure transducer's data should be acquired no greater than 25 Hz. Piezo pressure sensors can be used to acquire data very quickly but may lack long-term stability at low barrel pressures.

X2.3.4 A minimum of six (6) data points (3 PFD values) must be collected within the current acquisition volume. The acquisition volume considered must be no smaller than the MVE.

X2.4 *Procedure:*

X2.4.1 Assume flow is not at steady state.

X2.4.2 Calculate the pairwise force or pressure difference for the incoming data. Repeat this step until the pairwise force difference becomes less than 0.5 % of full scale. (Allow for user manual override, and allow a reasonable upper time limit depending on other test factors.)

NOTE X2.3—Use the pairwise force difference and not the difference of each value against the last. If the difference of each value against the last is used, errors will occur because the average difference for this case is always the difference of the first and last point divided by the number of points.

NOTE X2.4—If sufficient computing power is available all the independent differences can be used in the analysis, rather than just the contiguous pairwise difference described in X2.2.4.

X2.4.3 Generate the APFD and the standard deviation of the PFD collected over the acquisition volume.

NOTE X2.5—The standard deviation of the PFD can be estimated by summing the absolute value of adjacent points (abs($F_i - F_{i-1}$) + abs ($F_{i-1} - F_{i-2}$) ... etc.) divided by NPTS yielding Rbar. Rbar/1.13 = Standard Deviation Estimate.

NOTE X2.6—The number of PFD values is half the number of acquired data points (NPTS).

X2.4.4 Generate the AF over the same acquisition volume used in X2.4.3.

X2.4.5 Steady-state flow conditions for a specific acquisition volume have been achieved when the following conditions have both been satisfied:

X2.4.5.1 The absolute value of the APFD/AF (average pairwise force difference divided by the average force) is less than 0.25 %.

X2.4.5.2 The {standard deviation of the PFD}/AF (standard deviation of the pairwise force differences divided by the average force) is less than 2 %.

NOTE X2.7—While written assuming controlled plunger rate, this procedure can easily be adapted for constant stress control if the time between a fixed volumetric displacement (that is, plunger displacement assuming perfect plunger tip seal) is used as an input instead of force or pressure. The minimum volumetric displacement per time acquisition would be MVE/6 and could be triggered based on encoder pulses etc. related to plunger movement and hence volumetric displacement. Delta times should always exceed 50 times the internal clock resolution otherwise the volumetric element should be increased.

NOTE X2.8—The minimum volume element dictates a minimum sample time which depends on flow rate or ram speed. A plunger rate of 600 mm/min (24 in./min) on a 9-mm barrel diameter dictates a minimum acquisition rate of about 6 Hz if the MVE is used. Larger volume elements would allow for slower acquisition rates at the expense of using up more of the sample. Plunger speeds of 0.05 mm/min require a minimum of 6 points over 335 s. Larger barrels have higher flow rates for given plunger movement and would require only slightly higher acquisition rates.

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