



Designation: **D3835—08 D3835 – 16**

## Standard Test Method for Determination of Properties of Polymeric Materials by Means of a Capillary Rheometer<sup>1</sup>

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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope\*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 and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D618 Practice for Conditioning Plastics for Testing

D883 Terminology Relating to Plastics

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

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method <https://standards.iteh.ai/> [5/astm-d3835-16](https://standards.iteh.ai/astm-d3835-16)

2.2 *ANSI Standard:*

B46.1 Surface Texture<sup>3</sup>

### 3. Terminology

3.1 For definitions of general terms, see Terminology D883.

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).

<sup>1</sup> 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. Current edition approved Dec. 1, 2008; May 1, 2016. Published December 2008; May 2016. Originally approved in 1979. Last previous edition approved in 2002 as D3835–02; D3835–08. DOI: 10.1520/D3835-08.10.1520/D3835-16.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard’s Document Summary page on the ASTM website.

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

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

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

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<sup>-1</sup> (s<sup>-1</sup>).

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*,  $\eta_0$ —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 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:

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

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(B) For a constant-rate rheometer:

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

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

where:

$E^*$  = energy of activation,

$R$  = gas constant (8.3 J/K·mol), and

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$T$  = 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, it is possible that  $b$  may not be constant. The effect of pressure variation on output rate, the other variables remaining constant, is given by:

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

Thus a 0.5 % error in pressure measurement implies an error of  $b/2$  % in output rate. As the value of  $b$  ~~can range~~ 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  $b$  ~~can range~~ normally ranges from 1 to 3, the resultant error in  $Q$  due to a variation in  $r$  of  $\pm 0.5$  % can be 2 to 3 %, and the resultant error in  $Q$  due to variation in  $L$  of  $\pm 0.5$  % 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~~ 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 ~~can be~~ 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 1**~~2~~) 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. ~~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~~The capillary (**Note 3**) shall have a smooth straight bore that is held to within  $\pm 0.00762$  mm ( $\pm 0.0003$  in.) in diameter and shall be held to within  $\pm 0.025$  mm ( $\pm 0.001$  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 ( $180^\circ$ ) 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~~ It is possible to use other inlet angles, but comparisons must 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**).<sup>4</sup>

6.3.1 The precision with which capillary dimensions ~~can be~~ are 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  $0.0254 \pm 0.007$  mm ( $0.0010 \pm 0.0003$  in.) smaller in diameter than the barrel and at least  $6.35 \pm 0.13$  mm ( $0.250 \pm 0.005$  in.) in length. Alternative piston-barrel-sealing methods (O-rings, split seals, multi-lands, etc.) outside these tolerances are acceptable, provided there is less than 0.1 g of material going past the sealing device (see 12.4). 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  $\pm 0.2^\circ\text{C}$  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~~ holding this degree of control during an actual test. ~~test might not be possible~~. 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.

<sup>4</sup> The boldface numbers in parentheses refer to the list of references at the end of this test method.

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~~ shall be adhered to, as required, for sufficient accuracy. ~~Charging—It is acceptable to omit 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~~ must 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~~ must 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.

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~~ shall be in any form that can be introduced into the bore of the cylinder of any form such as powder, beads, pellets, strips of film, or molded slugs. ~~slugs, which when introduced into the bore of the cylinder will do so without undue force. In some cases it may be desirable to preform or pelletize a powder. preforming or pelletizing a powder is desirable.~~ 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 possibly~~ otherwise affect reproducibility of flow-rate measurements. If conditioning is necessary, see the applicable material specification and Practice D618.

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~~ Using shear stress and shear rate conditions ~~applied should also that~~ closely approximate those observed in the actual processing. ~~actual processing generally will provide the most useful data.~~

	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 10.10 (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~~ shall go effortlessly all the way into the die from both ends. The no-go (red) pin ~~should~~ shall not enter more than 1 mm in either end of the die. All errors in pin production ~~should~~ shall 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 piston and, if necessary to lay it down, place it 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~~must 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~~shall be reported with the data.

10.7 If the specific material being tested has previously been demonstrated thermally stable at the current test temperature, it is acceptable to use any combination of shear rates or shear stress ~~may be applied, stress,~~ 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~~are 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~~If a 0.5 % change or greater ~~be~~is observed in the viscosity per minute, the rate data ~~should~~shall be considered confounded with the time dependence and so noted. ~~The user may then wish to revert—~~Reverting back to the previous method to explore the nature of the thermal ~~instability; instability is an option.~~

10.9 If the percent extrudate swell is desired, measure the extrudate diameter using any National Metrology Institute (for example, NIST) traceable device capable of measuring diameters to within  $\pm 0.5$  %. 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~~shall have the measurement being made 25 mm away from the die exit. ~~At~~Use at least 8 independent samplings ~~should be used to and~~ report an average extrudate diameter. The associated real time shear viscosity data ~~should~~shall 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~~shall 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—~~It is acceptable to clean the capillary by dissolving the residue in a solvent. In certain cases where materials of a given class having similar flow characteristics are being tested consecutively, interim capillary cleaning is not required. In such cases, however, the effect of cleaning upon viscosity determinations must be shown to be negligible.

NOTE 9—Depending on material type and test conditions, it may be desirable to clean the capillary 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  $550 \pm 10^\circ\text{C}$  and clean with a small nitrogen purge through the die. The use of purge materials has been found useful to extend the time required between full cleanings of the measurement system.

## 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  $0.040 \pm 0.030$  mL/s (0.07 to 0.01 mL/s). The die diameter and length ~~should~~must 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:

$$\rho = \frac{m}{tQ} \quad (5)$$

where:

- $\rho$  = 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<sup>2</sup>.

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

NOTE 10—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 (3-8))

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  $\pm 0.00762$  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, subtract the frictional force ~~should be subtracted~~ from the force reading.

12.4 *Polymer Back Flow*—The clearance between the plunger and the barrel ~~may~~ might 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~~ sometimes necessary. This is evidenced by material exuding past the top of the land on the piston. ~~This material should be scraped~~ Scrape this material from the plunger, ~~weighed, and compared~~ weigh it, and compare it 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~~ is easily ~~be~~ made; for example, using a published equation of state for polystyrene (9), a compressibility correction chart can be made for this material.