Designation: D5422 – 09

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

This standard is issued under the fixed designation D5422; 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.

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 D3835).

1.3 Capillary rheometer measurements for thermoplastics and thermoplastic compounds are described in Test Method D3835.

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 no known ISO equivalent to this standard.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D618 Practice for Conditioning Plastics for Testing
D883 Terminology Relating to Plastics
D1238 Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer
D3835 Test Method for Determination of Properties of Polymeric Materials by Means of a Capillary Rheometer
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:* (See Terminology D883):

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 is achieved. Alternately, the shear stress (entrance pressure) is 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:

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

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:

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

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

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

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

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:

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

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:

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

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:

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

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:

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

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:

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

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:

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

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. The extrusion rate is 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 will 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.

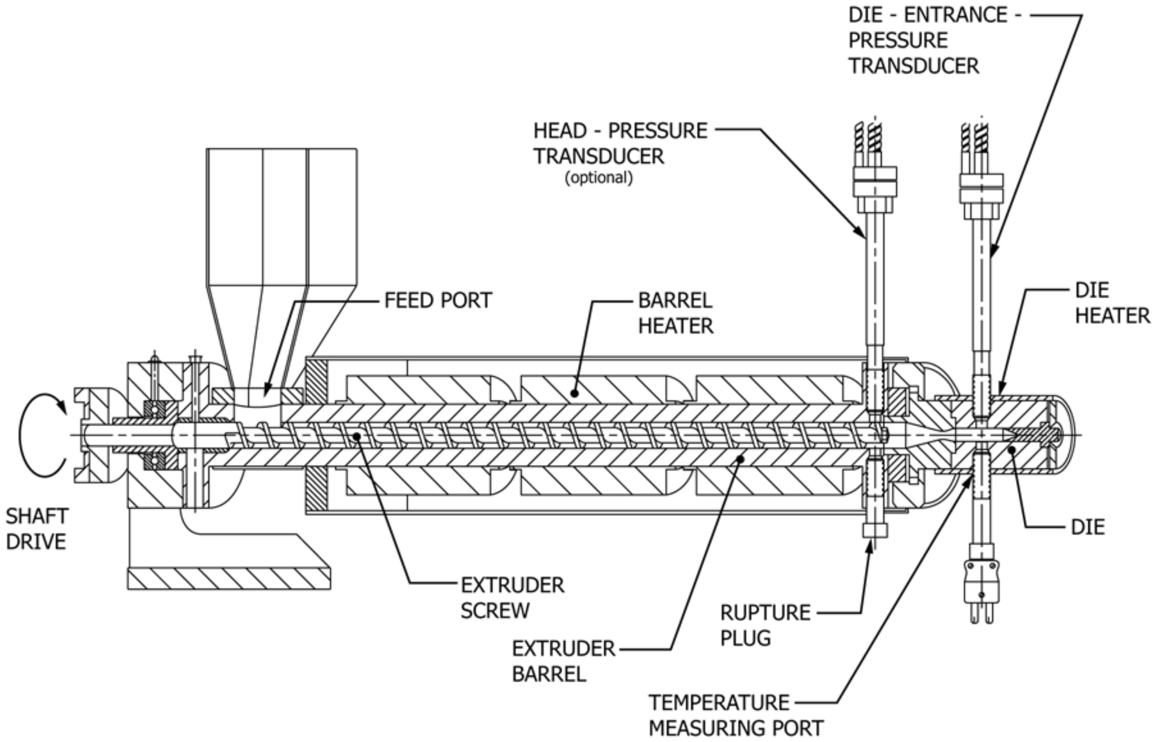

FIG. 1 Schematic of Screw-Extrusion-Type Rheometer Cross-Section

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 compression ratio is 3.0:1.0.

NOTE 2—Refer to material specifications or to recommendations of the manufacturer when selecting the proper compression ratio for the screw.

7.2.3 Both the barrel and the screw should be constructed of hardened stainless steel with suitable surface treatments to render them resistant to wear and chemical attack.

7.2.4 The extruder shall be equipped with instrumentation capable of monitoring the wall temperature of each portion of the barrel. The stock temperatures should also be measured at the extruder head and at any inside surface of the capillary-die assembly. The monitoring devices should have an accuracy of ±1.0°C, and a precision of ±0.2°C.

7.3 The dies are firmly secured to the end of the barrel. A minimum of two die inserts are needed. A schematic of the die is shown in Fig. 2. The dimensions are given in Tables 1 and 2.

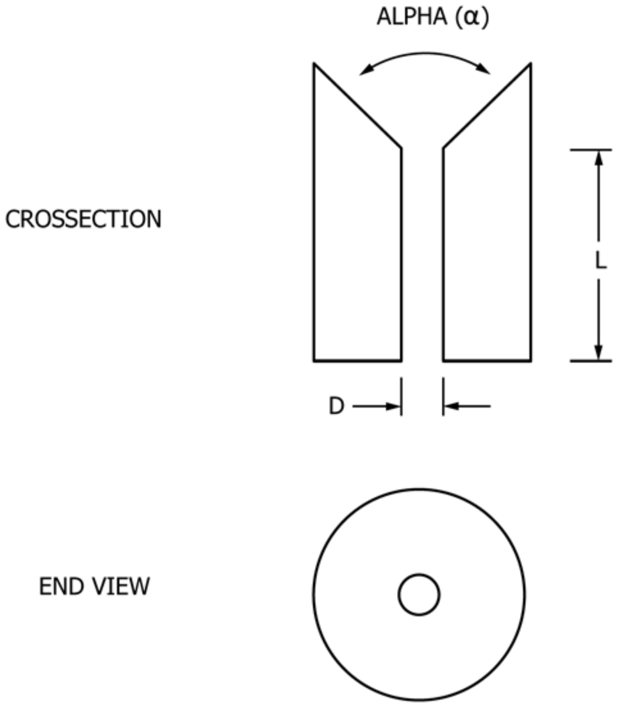

FIG. 2 Rheometer Die

NOTE 3—Refer to recommendations of the manufacturer when selecting the proper diameter for the die nozzle. The 2.0 mm insert series is recommended for rigid PVC, glass-reinforced resins, and materials with heat/shear sensitive properties.

7.4 For the purpose of calculation, the length of each insert shall be measured to ±0.14 mm, and the diameter to ±0.014

TABLE 1 1.0 mm Die Nozzle Dimensions

Item	Die A	Die B	Die C
Capillary length, (L), mm	15.0 ± 0.13	20.0 ± 0.13	30.0 ± 0.13
Capillary diameter, (D), mm	1.0 ± 0.013	1.0± 0.013	1.0± 0.013
Total included entrance angle, (α),°	40° ± 0°30'	40°± 0°30'	40°± 0°30'
Capillary length to diameter ratio, (L/D)	15.0 ± 0.13	20.0 ± 0.13	30.0 ± 0.13

TABLE 2 2.0 mm Die Nozzle Dimensions

Item	Die D	Die E	Die F
Capillary length, (L), mm	20.0 ± 0.13	30.0 ± 0.13	40.0 ± 0.13
Capillary diameter, (D), mm	2.0 ± 0.013	2.0 ± 0.013	2.0 ± 0.013
Total included entrance angle, (α),°	40° ± 0°30'	40° ± 0°30'	40° ± 0°30'
Capillary length to diameter ratio, (L/D)	10.0 ± 0.13	15.0 ± 0.13	20.0 ± 0.13

mm. The actual measured dimensions shall be used for these calculations. Calibration of the apparatus shall be carried out in accordance with the manufacturer's recommendations.

7.4.1 Dies must be made of wear-resistant materials such as hardened steel, hardened stainless steel, or carbide. Calibration of the pressure transducer generally requires removal of the transducer from its mounting, followed by the calibration in an appropriate testing apparatus, and then reattachment to the extruder. Thermocouples are calibrated according to the recommendations of the manufacturer.

8. Sampling and Conditioning of Samples

8.1 Many thermoplastics and thermoplastic compounds 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 Test Method D618.

9. Procedure

9.1 Determine the melt density of the thermoplastic or thermoplastic compound being tested (see Test Method D1238). This is necessary since the throughput is measured as a mass, but the calculations are based on volumetric flow.

9.2 Prepare the sample for introduction into the extruder.

9.2.1 To obtain equilibrium plastication and flow of thermoplastics or thermoplastic compounds, it is necessary to feed the material at a constant rate into the feed section of the screw. Material should be supplied in either pellet or powder form.

9.2.2 Typical screw-flight dimensions for the feed section of the laboratory scaled extruders are shown in Table 3.

9.3 Equip the extruder with Die A if using the 1.0 mm insert series, or Die D if using the 2.0 mm insert series (See 7.3).

9.4 Preheat the rheometer die and die holder to the test temperature. This temperature should model that of the processing operation. For material-processing temperatures, refer to the supplier recommendations. The die and die holder shall be preheated until thermal equilibrium of each heating zone has been achieved, and then maintained at the test temperature for at least 5 min.

TABLE 3 Typical Screw-Flight Dimensions

Compression Ratio	Flight Channel Width, mm	Flight Channel Depth, mm
19.05 mm screw diameter	15.88 ± 0.14	1.91 ± 0.03
2:1	15.88 ± 0.14	1.27 ± 0.03
3:1	15.88 ± 0.14	0.95 ± 0.03
4:1	15.88 ± 0.14	0.76 ± 0.03
5:1		
31.75 mm screw diameter	27.00 ± 0.14	3.18 ± 0.03
2:1	27.00 ± 0.14	2.12 ± 0.03
3:1	27.00 ± 0.14	1.59 ± 0.03
4:1	27.00 ± 0.14	1.27 ± 0.03
5:1		

9.5 Establish equilibrium extrusion conditions.

9.5.1 To ensure that equilibrium-flow conditions prevail before any viscosity measurements are taken, screw-extruder-type capillary rheometers require an additional running period, generally referred to as " line-out." Sufficient sample must be fed to the turning screw to fill and maintain the volume in the screw, head, and die under equilibrium conditions.

9.5.2 Check the rate of extrusion by cutting the extruded strand with a sharp knife, collecting the extrudate for precisely 2 min, and then cutting the strand. Weigh the collected extrudate.

NOTE 4—The apparent shear rate will increase or decrease by adjusting the extruder variable-speed drive.

9.5.3 Monitor the barrel temperatures and the die-stock temperature for at least 5 min of continuous running. During this period, the pressures in the head and particularly in the capillary-die assembly shall be in equilibrium before taking readings for viscosity measurements.

9.6 Collect the extrudate for 2 min, again using a sharp knife to cut the strand before and after the time period. Note the pressure and stock temperature in the die during extrudate collection. Weigh the extrudate to the nearest milligram, then convert the mass to volume, using the melt density of the material.

9.7 Repeat the extrusion procedure of 9.5 and 9.6 at least twice at different drive speeds to obtain an apparent shear-rate range. It may be necessary to adjust the die-set temperature in order to compensate for the shear heating of the material (see 6.3).

9.8 Change to Die B after using Die A when using the 1.0 mm insert series. Change to Die E after using Die D when using the 2.0 mm insert series.

9.9 Repeat the procedure in 9.5 – 9.7 using the same drive speeds.

NOTE 5—If a third die insert is necessary, follow the procedures in 9.8 and 9.9, substituting Die C for Die B, or substituting Die F for Die E. Use of three or more die nozzles of different lengths may better ensure the linearity of data required to calculate the Bagley Correction Factor.

10. Calculation

NOTE 6—See Section 3 for terminology used in the following calculations. For all calculations, it is advisable to use the actual measured values

TABLE 4 Repeatability Data

Corrected Shear Stress (KPa) at Fixed Corrected Shear Rate (s-1)

Mean Level in Ascending Order			Within Laboratory		
Material	Shear Rate	Avg. Stress (KPa)	S_r	r	(r)
LDPE	1000	120.3	2617	7328	6.1 %
	2500	157.3	1574	4409	2.8 %
	5000	192.6	216	606	0.3 %
	7500	216.9	884	2475	1.1 %
	10 000	236.0	1831	5128	2.2 %
	12 500	251.9	2676	7494	3.0 %
Pooled Values			1633	4573	2.5 %

Corrected Viscosity at Fixed Corrected Shear Rate (s-1) and Shear Stress (KPa)

Mean Level in Ascending Order			Within Laboratory		
Material	Shear Rate	Viscosity	S_r	r	(r)
LDPE	1000	120.3	2.6	7.3	6.1 %
	2500	63.0	0.6	1.8	2.8 %
	5000	38.6	0.1	0.2	0.5 %
	7500	28.9	0.1	0.4	1.4 %
	10 000	23.6	0.1	0.4	1.7 %
	12 500	20.2	0.2	0.6	2.9 %
Pooled Values			0.6	1.8	2.5 %

of the die dimensions instead of the nominal values discussed in 7.3.

10.1 *Apparent Shear Rate* ($\dot{\gamma}_{aA1}$):

10.1.1 Calculate the apparent shear rate of 9.5.2, 9.7, and 9.9 as follows:

$$\dot{\gamma}_{aA1} = \left[(32\cdot Q_{A1})/(\pi\cdot D_A{}^3)\right] \quad (9)$$

10.2 *Apparent Shear Stress* (τ_{aA1}):

10.2.1 The apparent shear stress is calculated as follows:

$$\tau_{aA1} = 4\cdot(P_{A1}/L_A/D_A) \quad (10)$$

10.3 *Apparent Viscosity* (η_a):

10.3.1 The apparent viscosity is calculated as follows:

$$\eta_a = \frac{\tau_{aA1}}{\dot{\gamma}_{aA1}} \quad (11)$$

10.4 *Entrance/Exit Effects* (*Bagley*):

10.4.1 The Bagley Correction Factor (E) is calculated as follows:

$$E = \frac{[P_{B1}\cdot(L_A/D_A)] - [P_{A1}\cdot(L_B/D_B)]}{[P_{A1} - P_{B1}]} \quad (12)$$

10.5 *Corrected Shear Stress* (τ_w):

10.5.1 The corrected shear stress is calculated as follows:

$$\tau_w = \frac{P_{A1}}{4\cdot[(L_A/D_A)+E]} \quad (13)$$

10.6 *Shear Sensitivity* (*n*):

10.6.1 Shear sensitivity is calculated as follows:

$$n = \frac{d\log(\tau_w)}{d\log(\dot{\gamma}_a)} \quad (14)$$

10.7 *Corrected Shear Rate* ($\dot{\gamma}_w$):

10.7.1 The corrected shear rate is calculated as follows:

$$\dot{\gamma}_w = \left[\frac{3n+1}{4n}\right]\cdot\dot{\gamma}_a \quad (15)$$

NOTE 7—Corrected shear stress (τ_W) is calculated first, and then used in the determination of *n*.

10.8 *Corrected Viscosity* (η):

10.8.1 The corrected viscosity is calculated as follows:

$$\eta = \frac{\tau_w}{\dot{\gamma}_w} \quad (16)$$

11. Report

11.1 Report the following information:

11.1.1 Type of capillary rheometer used,

11.1.2 Dimensions of capillary-die inserts, mm,

11.1.3 Entrance angle of capillary-die inserts, rad,

11.1.4 Position of pressure transducer (distance from capillary entrance), mm,

11.1.5 Identity of sample,

11.1.6 Pretreatment of sample, if any,

11.1.7 Temperature of test, °C or °K,

11.1.8 Shear sensitivity (*n*), dimensionless,

11.1.9 End effect (*E*), mm,

11.1.10 Corrected shear stress values, Pa,

11.1.11 Corrected shear rate values, s^{-1}, and

11.1.12 Corrected viscosity values, Pa·s.

12. Precision and Bias

12.1 Table 4 is based on a single laboratory repeatability study conducted in 2002 involving one LDPE homopolymer resin. The laboratory used a computerized torque rheometer and a three zone electrically heated 19.0 mm single screw extruder, 3:1 metering extruder screw, electrically heated one zone gear pump (1.16 cc/rev), and an electrically heated two zone capillary rheology die to process the sample. The sample was tested once on two separate days yielding two data points per value. (**Warning—**The explanation of r (12.2.1 and 12.2.2) is only intended to present a meaningful way of considering the approximate precision of this test method. The data in Table 4 should not be applied to acceptance or rejection of materials, as these data apply only to the materials tested in the round robin and are unlikely to be rigorously representative of other lots, formulations, conditions, materials, or laboratories. Users of this method should apply the principles outlined in Practice E691 to generate data specific to their materials and laboratory (or between specific laboratories). The principles of 12.2.1 and 12.2.2 would then be valid for such data.)

NOTE 8—SYMBOLS ARE DEFINED AS FOLLOWS:

S_r = within laboratory standard deviation

r = within laboratory repeatability, measurement units
(r) = within laboratory repeatability, %

12.2 *Concept of r and R in* Table 4—If S_r has been calculated from a large enough body of data, and for test results that were averages from testing one point for each test result, then:

12.2.1 *Repeatability (r)*—Two results obtained within one laboratory shall be judged not equivalent if they differ by more than the r-value, where $r = 2.8S_r$. r is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

12.2.2 *Reproducibility (R)*—The number of laboratories, one, participating in the round robin does not meet the minimum requirement for inclusion of information on between laboratory variability. Because this method does not contain a recognized round robin precision and bias statement, it shall not be used as a referee test method in case of dispute. Anyone wishing to participate in the development of precision and bias data should contact the Chairman, Subcommittee D20.30 (Section D20.30.08) ASTM 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959.

12.3 Any judgment in accordance with 12.2.1 or 12.2.2 would have an approximate 95 % (0.95) probability of being correct.

12.4 There are no recognized standards by which to estimate bias of this method.

13. Keywords

13.1 capillary rheometer; flow properties; material properties; molecular properties; rheological properties; rheology; screw extrusion

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D5422 - 03) that may impact the use of this standard. (April 1, 2009)

(1) Five year review.
(2) Revised Note 1 to current ISO equivalency statement.
(3) Added Section D20.30.08 as responsible for this test method in Footnote 1.