Designation: D5099 – 08 (Reapproved 2013)

Standard Test Methods for Rubber—Measurement of Processing Properties Using Capillary Rheometry[1]

This standard is issued under the fixed designation D5099; 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 These test methods describe how capillary rheometry may be used to measure the rheological characteristics of rubber (raw or compounded). Two methods are addressed: Method A—using a piston type capillary rheometer, and Method B—using a screw extrusion type capillary rheometer. The two methods have important differences, as outlined in 7 – 10 and 11 – 14, respectively.

1.2 These test methods cover the use of a capillary rheometer for the measurement of the flow properties of thermoplastic elastomers, unvulcanized rubber, and rubber compounds. These material properties are related to factory processing.

1.3 Since piston type capillary rheometers impart only a small amount of shearing energy to the sample, these measurements directly relate to the state of the compound at the time of sampling. Piston type capillary rheometer measurements will usually differ from measurements with a screw extrusion type rheometer, which imparts shearing energy just before the rheological measurement.

1.4 Capillary rheometer measurements for plastics are described in Test Method D3835.

1.5 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.6 *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:*[2]

D1349 Practice for Rubber—Standard Conditions for Testing

D1418 Practice for Rubber and Rubber Latices—Nomenclature

D1485 Practice for Rubber from Natural Sources—Sampling and Sample Preparation

D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

D3835 Test Method for Determination of Properties of Polymeric Materials by Means of a Capillary Rheometer

D3896 Practice for Rubber From Synthetic Sources—Sampling

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 The following terms appear in logical order for the sake of clarity:

3.1.2 *capillary rheometer*—an instrument in which rubber 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.

3.1.3 *die entrance pressure (P)*—the pressure in the reservoir at the die entrance, in Pa.

3.1.4 *volumetric flow rate (Q)*—the flow rate through the capillary die, in mm^3/s.

3.1.5 *apparent (uncorrected) shear rate* ($\dot{\gamma}_a$)—shear strain rate (or velocity gradient) of the rubber extrudate as it passes through the capillary die (Eq 1), in s^{-1}.

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[1] These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and are the direct responsibility of Subcommittee D11.12 on Processability Tests.

Current edition approved Nov. 1, 2013. Published January 2014. Originally approved in 1993. Last previous edition approved in 2008 as D5099 – 08. DOI: 10.1520/D5099-08R13.

[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.

3.1.5.1 *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, at the wall of the capillary. By selecting a die diameter and controlling the volumetric flow rate (Q) through the die, a specific level of apparent shear rate may be achieved. Alternately, the shear stress (die entrance pressure, P) may be controlled, and the apparent shear rate measured.

Mathematically, the apparent shear rate for a Newtonian fluid at the wall is given as follows:

$$\dot{\gamma}_a = \frac{32\,Q}{\pi\,D^3} \tag{1}$$

where:

$\dot{\gamma}_a$ = apparent shear rate, s^{-1},
Q = volumetric flow rate, mm^3/s,
π = the constant pi, approximately 3.142, and
D = diameter of the capillary die, mm.

3.1.6 *apparent shear stress* (τ_a)— the measured resistance to flow through a capillary die (Eq 2).

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

where:

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

3.1.7 *apparent viscosity* (η_a)— ratio of apparent shear stress to apparent shear rate, in Pa-s.

3.1.7.1 *Discussion*—For a capillary rheometer, the apparent viscosity 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.

3.1.8 *Newtonian fluid*—a fluid for which viscosity does not vary with changing shear rate. Simple liquids such as rubber extender oils are Newtonian; most polymeric materials are not.

3.1.9 *power law fluid*—a fluid material for which the viscosity varies with the shear rate in accordance with the relationship:

$$\tau = K(\dot{\gamma})^N \tag{3}$$

where:

K = constant, often called consistency index, and
N = a material parameter generally called the power law index. It is equal to 1.0 for Newtonian fluids and generally between 0.18 and 0.33 for compounded rubbers or elastomers, or both, with some exceptions.

Most non-Newtonian fluids follow the relationship in Eq 3 for at least short ranges of the shear rate variable. Eq 3 is generally used in its logarithmic form, as:

$$\log(\tau) = \log(K) + N\log(\dot{\gamma}) \tag{4}$$

3.1.10 *corrected shear stress* (τ_w)— the shear stress at the wall of the capillary die; it is calculated from the apparent shear stress by applying the Bagley correction E in Eq. 5 for energy losses at the entrance and exit of the die.

3.1.10.1 *Discussion*—The Bagley correction, often termed "end effect," is normally applied as though it were an additional length of capillary, in terms of an added *L/D* ratio. The Capillary entrance angle and geometry have great influence on the magnitude of this correction.

3.1.10.2 *Discussion*—Since the magnitude of the Bagley correction is a function of shear rate, data for this correction are obtained by using two or more dies of different lengths but preferably of the same diameter and volumetric flow rate (and thus the same apparent shear rate). 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, of the following form:

$$P = c\left[\frac{L}{D} + E\right] \tag{5}$$

where:

c = slope of the line, and
E = Bagley correction, expressed as the negative capillary length to diameter (*L/D*) ratio resulting from extrapolating the pressure value to zero when plotted against *L/D*.

Both c and E values are functions of the rubber compound, the shear rate and the capillary entrance angle.

Corrected shear stress (τ_w) is therefore:

$$\tau_w = \frac{P}{4\left[\frac{L}{D} + E\right]} \tag{6}$$

or:

$$= \frac{P_L - P_s}{4\left[\frac{L_L}{D_L} - \frac{L_s}{D_s}\right]} \tag{7}$$

where:

P_L = pressure drop for long die, Pa,
P_S = pressure drop for short die, Pa,
L_L = length of the long die, mm,
L_S = length of the short die, mm,
D_L = diameter of the long die, mm, and
D_S = diameter of the short die, mm.

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

3.1.11.1 *Discussion*—The Rabinowitsch correction mathematically adjusts shear rate values for the fact that the fluid is non-Newtonian, using the power law fluid model (Eq 3). To obtain the corrected shear rate, at least two measurements of apparent shear stress and apparent shear rate are made, generally by increasing the volumetric flow rate (Q) with the same measuring capillary. The Bagley correction is made to the shear stress values; either by algebraic means if only two measurements are made, or by a regression equation for a greater number of points. Eq 3 may be solved for N, where N is designated as N', using *corrected* shear stress (τ_w) values and the corresponding *apparent* shear rate $\dot{\gamma}_a$ values. Although in theory, N calculated from Eq 3 using apparent shear stress (τ_a) and apparent shear rate $\dot{\gamma}_a$ values and N' calculated from Eq 3 using corrected shear stress (τ_w) and apparent shear rate $\dot{\gamma}_a$ values should be identical, their values may vary as the Bagley correction (E) varies, hence the designation of N' in (Eq 8).

The corrected shear rate $\dot{\gamma}_w$ is:

$$\dot{\gamma}_w = \dot{\gamma}_a \left[\frac{3N' + 1}{4N'} \right] \qquad (8)$$

For most rubbers or elastomers the correction factor for shear rate is typically between 1.5 and 2.1, with some exceptions.

3.1.12 *corrected viscosity* (η_w)— the ratio of corrected shear stress to corrected shear rate.

3.1.12.1 *Discussion*—Since the corrections used, as well as the material properties, are functions of shear rate, it is very important to state the particular value of shear rate at which the measurement was made.

3.1.13 *critical shear stress*—that value of shear stress at which there is a discontinuity in the slope of the log shear stress versus log shear rate plot; manifested by a sudden change in surface roughness of the extrudate (sometimes referred to as melt fracture).

4. Significance and Use

4.1 These test methods are useful for characterization of raw, or compounded, unvulcanized rubber in terms of viscosity, or resistance to flow.

4.2 The data produced by these test methods have been found useful for both quality control tests and compound development. However, direct correlation with factory conditions is not implied.

4.3 Flow performance data permits quality control of incoming raw rubbers because the flow parameters are sensitive to molecular weight and to molecular weight distribution. Therefore, these test methods may distinguish differences between lots.

4.4 The shear viscosity or flow viscosity of compounded rubber batches in the raw (unvulcanized) state will not only be sensitive to the raw polymer molecular properties, but will also be affected by type and amount of filler, plasticizer or softener levels, amount and type of copolymer blend, and other compounding materials. These test methods can serve as a quality control tool for either incoming custom mixed compounds or for in-house quality assurance checks on production mixing. These test methods are useful for research and development of new products by measuring the rheological effect on a rubber compound of new polymers, resins, softeners, etc.

5. Interferences

5.1 Since flow properties of these non-Newtonian fluids are not linear, capillary rheometers should be operated at conditions of flow (temperature, pressure, and rate) similar to that of selected commercial processes. These processes include mixing, calendering, extrusion, and molding of rubber compounds.

5.2 Piston type capillary rheometers impart only very small amounts of shear or mixing energy before the measurement is made. Consequently, the measurement relates to the state of the polymer or compound at the time the sample was taken. If it is desirable to relate directly to a down-stream process involving significant amounts of mixing energy, it is sometimes desirable to shear the polymer on a roll mill before the rheological measurement is made.

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

6. Sampling and Conditioning of Samples

6.1 Condition the sample obtained in accordance with Practice D1485 or D3896 until it has reached room temperature (23 ± 3°C (73 ± 5°F)) throughout.

6.2 *Massed Specimen*—Prepare a massed specimen, as in 6.2.1, only if indicated in Table 1. Massing is used to combine the rubber crumbs, homogenize the specimen, and extract trapped air.

6.2.1 Pass 250 ± 5 g of the sample between the rolls of the standard laboratory mill (described in Practice D3182) having a roll temperature of 50 ± 5°C (122 ± 9°F) and having a distance between the rolls of 1.4 ± 0.1 mm (0.055 ± 0.005 in.) as determined by a lead slug. Immediately fold the specimen in half and insert the folded end into the mill for a second pass. Repeat this procedure until a total of nine passes have been completed. Open the mill rolls to 3 ± 0.1 mm (0.125 ± 0.005 in.), fold the specimen in half, and pass it between the rolls once. Do not allow the specimen to rest between passes or to band on the mill rolls at any time.

TABLE 1 Sample Preparation

Type Rubber[A]	Sample Preparation, Reference Section	Test Temperature, °C
NBS 388	6.1 only	100 ± 0.5 or 125± 0.5
NR	6.1 only	100 ± 0.5
BR	6.1 only	100 ± 0.5
CR		
IR		
NBR		
SBR		
BIIR	6.1 only	100 ± 0.5 or 125 ± 0.5
CIIR		
IIR		
EPDM	6.1 only	125 ± 0.5
EPM		
Synthetic rubber black masterbatch	6.1 and 6.2.1	100 ± 0.5
Compounded stock	6.1 only reclaimed material	100 ± 0.5
Miscellaneous	If similar to any group above, test accordingly. If not, establish a procedure.	

[A] See Practice D1418.

6.3 Conditioning must be carefully controlled. Piston type rheometers impart very little shear energy; therefore, any structure that is formed on resting of the sample is still present when that sample reaches the die. Although screw-type rheometers do impart shear work during processing, it is important to standardize the amount of mill mastication prior to feeding to the extruder. Some compounds, especially silica filled ones, may reform bonds with the rubber matrix if more than four hours have passed since their initial mill processing. If so, they should be warmed up by giving them five passes through a tight mill. Do not let them band on the mill, in order to minimize polymer break down during this operation.

TEST METHOD A—PISTON TYPE CAPILLARY RHEOMETER

7. Summary of Test Method

7.1 Raw or compounded unvulcanized rubber is placed in a temperature controlled cylinder fitted at one end with a transition section of conical cross section and a precisely measured length of metal capillary tubing (the die). The other end of the cylinder contains a close fitting piston with provisions for driving this piston through the cylinder either at constant rate or with constant force. The sample is driven through the die while measuring or controlling the rate of capillary extrusion and the pressure on the sample at the entrance of the die.

7.2 The capillary extrusion is performed at two different rates through a standard die of 1.5 mm diameter and 15 mm (nominal) length (10:1 *L/D*) and at both of these rates through a die of 1.5 mm diameter and 22.5 mm length (15:1 *L/D*).

7.3 The data produced by this test method have been found useful for both quality control tests and compound development. However, direct correlation with factory conditions is not implied.

7.4 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.

8. Apparatus

8.1 A schematic diagram of a piston type 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. A piston force measurement is not required if extrusion pressure at the die entrance is measured.

8.2 The barrel, or cylinder, of the rheometer is a metallic tube with an inside diameter between 9 mm and 22 mm, and a length of 40 to 450 mm. The inside diameter shall be known to 0.1 mm. The barrel is equipped with heaters and heater controllers capable of maintaining the desired test temperature of the inside wall of the tube. This temperature shall be maintained stable within ±0.5°C for the region of the barrel 50 mm (2 in.) above the die opening to the die opening.

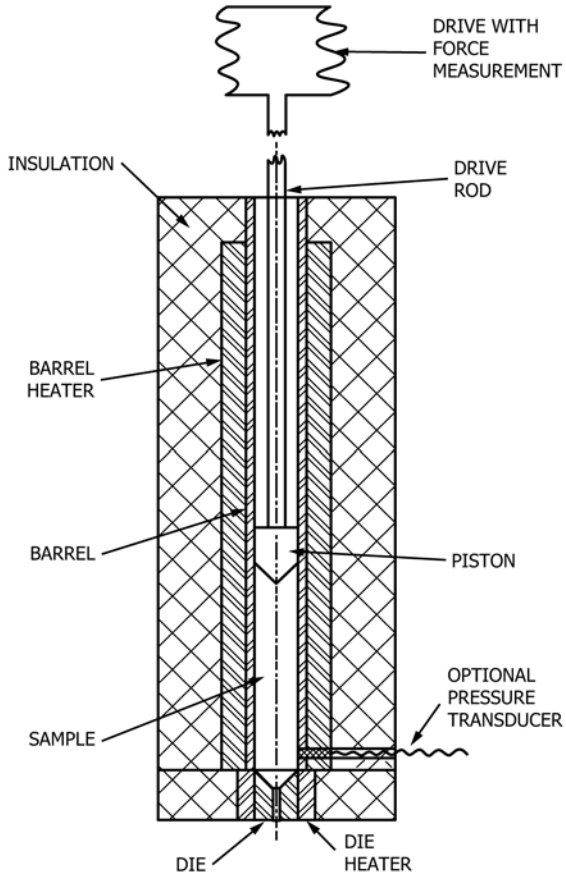

FIG. 1 Schematic of Piston Type Capillary Rheometer Cross Section

8.3 The dies are firmly secured to the bottom of the barrel. Two dies are used. A schematic of the dies is shown in Fig. 2. The dimensions are given in Table 2.

8.3.1 Dies must be made of wear resistant materials such as hardened steel, Stellite, hardened stainless steel, or tungsten carbide. Long and short die diameter should be within ±0.005 mm of each other.

8.3.2 For the purpose of the calculations, the length of the capillary shall be measured to ±0.1 mm, and the diameter to ±0.008 mm. The actual measured dimensions shall be used for these calculations.

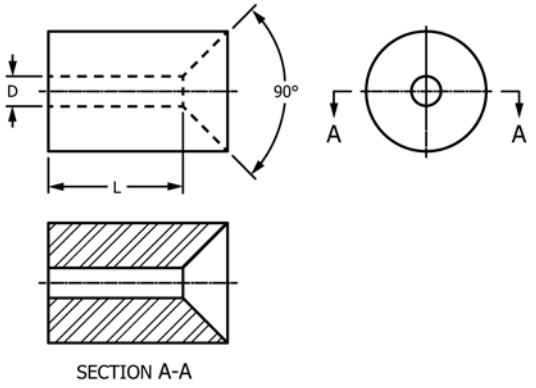

FIG. 2 Rheometer Die

TABLE 2 Dimensions of Capillary Dies

	Die A	Die B
Capillary length (*L*), mm	15 ± 1	22.5 ± 1
Capillary diameter (*D*), mm	1.5± 0.1	1.5 ± 0.1
Total included entrance angle (α), degrees	90 ± 2	90 ± 2
Capillary length to diameter ratio (*L/D*)	10 ± 2	15 ± 2

8.3.3 The die temperature shall be stabilized prior to the start of the test at the test temperature ±0.5°C. Separate die heaters are often used for this purpose.

8.3.4 The piston must fit sufficiently tight to avoid backflow of sample between the piston and barrel, but not so tightly as to add significant force due to friction to the measured value. Polytetrafluoroethylene (PTFE) seal rings may be used on the circumference of the piston to aid sealing if necessary. Blank runs (with no sample present) at the temperature to be used for testing may be used to estimate the force contributed by the frictional drag.

NOTE 1—On piston type capillary rheometers that do not have a pressure transducer directly measuring the extrusion pressure at the die entrance, piston friction is part of the measured pressure. This error must be considered as part of the 0.5 % force tolerance. See also 9.6.2.

8.4 The drive system may be of either a constant speed or a constant force type.

8.4.1 Constant speed drives are of a mechanical or servohydraulic type. The rate of motion of the piston shall be known within ±0.5 %, and shall vary by less than 0.5 % throughout the duration of the test. In many constant speed drive instruments, the force is measured at the drive head or crossbar by means of a force transducer or by means of a hydraulic pressure gage. This force must be measured to± 0.5 % of applied force. See also Note 1.

8.4.2 Constant force drives employ a mass acting under gravity, or a pressurized gas or liquid above the piston. The rate of piston movement also should be known to 0.5 %. The force on these instruments may be measured by the fluid pressure above the piston or the value of the dead weight and any lever employed. See also Note 1.

NOTE 2—These tolerances are 0.5 % of set rate and not 0.5 % of range.

8.4.3 The pressure on the rubber sample being tested may be more directly measured using a pressure transducer whose measuring element is placed directly above the die entrance.

8.5 Calibrate apparatus in accordance with the manufacturer's recommendations.

8.5.1 Mechanical calibration is accomplished by use of known masses applied vertically to force cells and pressure gages, stopwatch measurements of rates of travel, and micrometric measurement of internal rheometer parts.

8.5.2 While new dies are quite adequately measured to the tolerances of this test method, this measurement is not easy on dies after use. In many cases, it is advisable to use a reference material and reference die to calibrate the system, using the calibration methods given in this test method to determine the equivalent dimensions for the die. Low density polyethylene at a test temperature of 190°C (374°F) has been recommended for this purpose. This material is stable, and can be stored for up to two years.

9. Procedure

9.1 Assemble the rheometer using Die A ($L/D = 10$).

9.2 Preheat the rheometer to the test temperature. This temperature should model that of the next forming operation, if known. For material properties, test at temperatures indicated in Table 1. For alternate test temperatures modeling process conditions, refer to Practice D1349.

9.3 Cut the test specimen into pieces approximately 5 by 5 by 10 mm (¼ by ¼ by ½ in.) with scissors or knife. Hand pack these pieces into the rheometer with minimum air entrapment by using layers of about 25 mm each, and using a stainless steel, brass, or aluminum rod for packing.

NOTE 3—Air can be eliminated from some compounds by forcing the rheometer piston down on the loaded specimen, then releasing the force.

9.4 Heat the specimen to test temperature. The size of the reservoir will affect the preheat time required. For a 9-mm barrel, temperature recovery requires 1 min. For a 12-mm barrel, temperature recovery requires 2 min. For a 19-mm barrel allow 4 min for temperature recovery at rubber processing temperatures (less than 200°C).

9.5 If the material being tested is heat stable, doubling the equilibration time is advisable. If the material being tested is a rubber compound with curatives, use the times given in 9.4.

NOTE 4—These times are approximate for carbon black filled materials. Independent tests to verify the time required to achieve uniform temperature and stable pressure may be required.

9.6 *Capillary Extrusion Procedure*—Start the drive system to force the piston through the barrel, at 330 ± 2 mm^3/s flow rate (apparent shear rate of about 1000 s^{-1}). This requires a nominal piston speed of 5.2 mm/s in a 9-mm diameter barrel, 2.9 mm/s in a 12-mm barrel, or 1.2 mm/s in a 19-mm barrel. With some instruments, piston speed control limitations may produce slight deviations from the nominal apparent shear rate test conditions. Choose the piston speed necessary to reach the shear rate closest to the nominal test condition.

NOTE 5—For rheometers with a barrel diameter, D_{barrel} , other than those noted above, piston speed in mm/s may be calculated with the following formula: if fitted with a 1.5-mm die, speed = $25312/D_{barrel}{}^2$.

9.6.1 If the rheometer is equipped with a pressure transducer in the die entrance area, extrude the specimen until the pressure trace is stable.

9.6.2 If the rheometer measures the force on the piston, note the position of the piston at the beginning of flow exiting the die, extrude for at least 2 min, then note the position of the piston again. Due to energy losses in the barrel with some rubber compounds, the recorded force for the extrusion is the force at zero barrel length (that is, piston touching die), which is calculated by extrapolation.

9.7 Repeat test steps in 9.3 – 9.6.2 at 100 ± 2 mm^3/s flow rate (apparent shear rate of approximately 300 s^{-1}). Steps in 9.6 and 9.7 can be combined into one capillary extrusion test if the equipment allows it.

9.8 Change to Die B ($L/D = 15$).

NOTE 6—If several compounds are to be tested, it is more convenient to run all tests with Die A before changing dies. Be careful to clean barrel when changing compound to be tested.

9.9 Repeat test steps in 9.3 – 9.6.2 with Die B at 330 ± 2 mm^3/s flow rate (apparent shear rate of about 1000 s^{-1}).

9.10 Repeat test steps in 9.3 – 9.6.2 with Die B, at 100 ± 2 mm^3/s flow rate (apparent shear rate of about 300 s^{-1}). Steps 9.9 and 9.10 can be combined into one capillary extrusion test if the equipment allows it.

9.11 Remove the capillary die and clean the barrel between specimens by forcing a wad of dry cheese cloth or other cotton material through the barrel. Clean excess material from the surface of the dies. The material in the capillary is displaced by the following sample.

10. Calculation

10.1 For all calculations, use the measured values for die dimensions and barrel dimensions, rather than the nominal dimensions.

10.2 Calculate the apparent shear rate for the test described in 9.6 as follows:

$$\dot{\gamma}_{a,A1000} = 8(D_{barrel})^2(V_{A1000}/D_A{}^3) \tag{9}$$

where:

$\dot{\gamma}_{a,A1000}$ = the apparent shear rate (s^{-1}), for Die A at a nominal shear rate of 1000 s^{-1} ,
D_{barrel} = the diameter of the barrel, mm,
V_{A1000} = the speed of the piston, mm/s for Die A at a nominal shear rate of 1000 s^{-1}, and
D_A = the capillary diameter for Die A, mm.

10.2.1 The apparent shear rates for 9.7 ($\dot{\gamma}_{a,A300}$), 9.9 ($\dot{\gamma}_{a,B1000}$), and 9.10 ($\dot{\gamma}_{a,B300}$) are calculated similarly.

10.3 Calculate the apparent shear stress, $\tau_{a,A1000}$, for Die A at the nominal apparent shear rate of 1000 s^{-1} for the test in 9.6 as follows:

$$\tau_{a,A1000} = \frac{P_{A1000}}{4(L_A/D_A)} \tag{10}$$

where:

P_{A1000} = pressure from transducer at die entrance, Pa, using Die A and the nominal apparent shear rate of 1000 s^{-1}.

or:

$$P_{A1000} = 4F_P/[\pi(D_{barrel})^2] \tag{11}$$

where:

F_P = force on the piston extrapolated to zero barrel length (9.6.2), *N*.

10.3.1 The apparent shear stress for 9.7 ($\tau_{a,A300}$) is calculated similarly.

10.4 Calculate the apparent shear stresses for the longer Die B used in 9.9 ($\tau_{a,B1000}$) and 9.10 ($\tau_{a,B300}$) in a similar manner. For example, using the Die B, length L_B and diameter D_B at the nominal apparent shear rate of 1000 s^{-1}:

$$\tau_{a,B1000} = \frac{P_{B1000}}{4(L_B/D_B)} \tag{12}$$

10.5 If desired, calculate the apparent viscosity, $\eta_{a,A1000}$, for Die A at 1000 s^{-1} nominal apparent shear rate as follows:

$$\eta_{a,A1000} = \frac{\tau_{a,A1000}}{\dot{\gamma}_{a,A1000}} \tag{13}$$

10.5.1 The apparent viscosities for Die A at 300 s^{-1}, and Die B at 1000 s^{-1} and 300 s^{-1} are calculated similarly ($\eta_{a,A300}$, $\eta_{a,B1000}$, and $\eta_{a,B300}$, respectively).

10.6 Calculate the entrance/exit effects (Bagley correction) at 1000 s^{-1} nominal apparent shear rate as follows as follows:

$$E_{1000} = \frac{P_{B1000}(L_A/D_A) - P_{A1000}(L_B/D_B)}{P_{A1000} - P_{B1000}} \tag{14}$$

where:

E_{1000} = Bagley correction at a nominal apparent shear rate of 1000 s^{-1}. Also calculate an E_{300} value for the 300 s^{-1} nominal apparent shear rate.

10.7 Calculate the corrected shear stress, $\tau_{w,1000}$, as follows:

$$\tau_{w,1000} = \frac{P_{A1000}}{4[(L_A/D_A)+E_{1000}]} \tag{15}$$

or:

$$\tau_{w,1000} = \frac{P_{A1000} - P_{B1000}}{4\left[\frac{L_A}{D_A} - \frac{L_B}{D_B}\right]} \tag{16}$$

where:

$\tau_{w,1000}$ = corrected shear stress at an apparent shear rate of about 1000 s^{-1} .

10.7.1 Calculate the $\tau_{w,300}$ corrected shear stress at the 300 s^{-1} nominal apparent shear rate.

10.8 Calculate shear sensitivity, N'_A, for Die A test results as follows:

$$N'_A = \frac{\log\tau_{w,A1000} - \log\tau_{w,A300}}{\log\dot{\gamma}_{a,A1000} - \log\dot{\gamma}_{a,A300}} \tag{17}$$

10.8.1 The shear sensitivity, N'_B, for Die B test results is calculated similarly. Average the two to determine N' for (Eq 18).

10.9 Calculate corrected shear rate, $\dot{\gamma}_w$, at each nominal apparent shear rate as follows:

$$\dot{\gamma}_{w,1000} = \dot{\gamma}_{a,A1000}\left[\frac{3N'+1}{4N'}\right] \tag{18}$$

10.9.1 Use the same correction to convert $\dot{\gamma}_{a,A300}$ to $\dot{\gamma}_{w,300}$.

10.10 Calculate corrected viscosity, η_w , for each desired shear rate as follows:

$$\eta_w = \frac{\tau_w}{\dot{\gamma}_w} \tag{19}$$

10.10.1 This value is only valid at the shear rate at which it is calculated, and must be given in annotated form, for example, $\eta_{w,1000}$.

10.11 *Determination of Corrected Values of Shear Stress and Viscosity at Corrected Shear Rates:*

10.11.1 *Graphical Method*—Plot the values of corrected shear stress determined in 10.7 on log/log graph paper as a function of corrected shear rate determined in 10.9 for the nominal apparent shear rates of 300 and 1000 s^{-1}. Draw a straight line through the points taken at the nominal apparent shear rates of 300 s^{-1} and 1000 s^{-1}. Determine a corrected shear stress at each corrected shear rate by determining where this line crosses the point on the corrected shear rate axis.

10.11.2 *Mathematical Method*—Calculate the consistency index, K, using Eq 20, and the corrected shear stress, τ_w, at each corrected shear rate using Eq 21:

$$K = \left[\frac{\tau_{w,1000}}{(\dot{\gamma}_{w,1000})^{N'}} \right] \tag{20}$$

where:

$\tau_{w,1000}$ = corrected shear stress, at corrected shear rate corresponding to a nominal apparent shear rate of 1000 s^{-1}, Pa,
$\dot{\gamma}_{w,1000}$ = corrected shear rate corresponding to a nominal apparent shear rate of 1000 s^{-1}, and
N' = power law index, calculated as in 10.8.

and:

$$\tau_w = K(\dot{\gamma}_w)^{N'} \tag{21}$$

10.11.3 Calculate a corrected viscosity (η_w) at each corrected shear rate by dividing the corrected shear stress by the corrected shear rate.

TEST METHOD B—SCREW EXTRUSION TYPE CAPILLARY RHEOMETER

11. Summary of Test Method

11.1 Raw rubber or unvulcanized elastomeric compound is formed into sheets on a two-roll mill. Strips cut from these sheets are fed to a laboratory extruder whose barrel is equipped with temperature control. The output end of the extruder is equipped with a transition section of conical cross section and a precisely measured length of metal capillary tubing (the die). A suitable pressure transducer and temperature measuring device, such as a thermocouple, are placed in the chamber before the die.

11.2 The rate of extrusion is calculated from the amount of extrudate collected over a timed interval. The rate of extrusion is controlled by adjustment of the drive speed.

11.3 The extrusion is performed at two different rates through a standard die of 1.5 mm diameter and 15 mm (nominal) length, then again at both of these rates through a die of 1.5 mm diameter and 22.5 mm length.

11.4 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.

12. Apparatus

12.1 A schematic diagram of a screw extrusion capillary rheometer is shown in Fig. 3. 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.

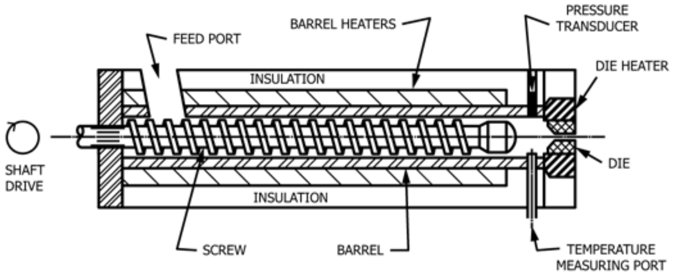

FIG. 3 Schematic of Screw Extrusion Type Capillary Rheometer Cross Section

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

12.2.1 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 *L/D* ratio of the barrel should be not less than 10:1 nor more than 20:1.

12.2.2 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 in the feed section. The difference in the volume is referred to as compression ratio. The compression ratio of the screw should be not more than 2.0:1 for rubbery materials; 1.0:1 or 1.5:1 is preferred.

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

12.2.4 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 the inside surface of the capillary die assembly. The monitoring devices shall have a sensitivity of ±1.0°C.

12.3 The dies are firmly secured to the end of the barrel. Two dies are used. A schematic of the die is shown in Fig. 2. The dimensions are given in Table 2.

12.3.1 Dies must be made of wear resistant materials such as hardened steel, Stellite, or hardened stainless steel. Calibration of pressure transducers generally requires removal of the transducer from its mounting, followed by calibration in an appropriate pressure testing apparatus, and then reattachment to the extruder. Calibrate thermocouples according to manufacturer's recommendations.

12.3.2 For the purpose of the calculations, the length shall be measured to ±0.1 mm, and the diameter to ±0.008 mm. The actual measured dimensions shall be used for these calculations. Calibrate apparatus in accordance with the manufacturer's recommendations.

13. Extrusion Procedure

13.1 Determine the melt density of the compound or raw rubber being tested. This is necessary because the throughput is measured in mass units but the calculations are based on volumetric flow.

13.2 Prepare the stock for feed to the screw extruder.

13.2.1 To obtain equilibrium plastication and flow of rubber or rubber compounds through a screw extruder, it is necessary to feed the material at a constant rate to the feed section of the screw. It should be fed as pre-cut strips from the mill sheet with a thickness no greater than the depth of the screw flight channels, and a width no greater than the distance between flights.

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

13.3 Equip the screw extruder with Die A (12.3).

13.4 Preheat the rheometer die and die holder to the test temperature. This temperature should model that of the next forming operation, if known; for material properties, test at temperature indicated in Table 1. Barrel temperature should be 10 to 15°C below the die temperature at the start of the equilibration period. For alternate test temperatures modeling process conditions, refer to Practice D1349.

13.5 Establish equilibrium extrusion conditions.

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

13.5.2 Check the rate of extrusion by cutting the extruded strand with a sharp knife, collecting the extrudate for a precisely timed period of 2 min, then cutting the strand again. Weigh the extrudate collected. Adjust the speed of the extrusion to approximately 330 mm^3/s (19.8 cm^3/min) (apparent shear rate of approximately 1000 s^{-1}) by adjusting the variable speed drive.

13.5.3 Monitor the barrel temperatures and the die stock temperature for at least 5 min continuous running. During this line-out period, the pressures in the head and particularly in the capillary die assembly must be in a state of equilibrium before readings for viscosity measurements can be taken. Barrel temperatures should be 5 to 10°C cooler than stock temperatures or die temperature.

13.6 Collect the extrudate for 2 min, again using a sharp knife to cut the strand before and after the timed period. Note the pressure on the transducer and the stock temperature during the sample collection. Weigh the sample to the nearest milligram, then convert the weight to volume by use of the density.

13.7 Repeat the extrusion of steps 13.5 and 13.6 at a rate of approximately 100 mm^3/s (6.0 cm^3/min) (apparent shear rate of approximately 300 s^{-1}).

13.8 Change the die to Die B (15 *L/D*).

TABLE 3 Typical Screw Flight Dimensions

Screw Diameter		Flight Channel Width		Flight Channel Depth	
mm	(in.)	mm	(in.)	mm	(in.)
19.0	(0.759)	19.05	(0.75)	3.86	(0.150)
31.7	(1.25)	31.75	(1.25)	6.35	(0.250)

13.9 Repeat extrusion steps in 13.5 and 13.6 with Die B at a rate of approximately 330 mm^3/s (19.8 cm^3/min) (apparent shear rate of approximately 1000 s^{-1}).

NOTE 7—If several compounds are to be tested, it is more convenient to run all extrusions through Die A before changing dies. Ensure that sufficient throughput of new specimens is run off to guarantee removal of all the previous sample.

13.10 Repeat extrusion steps in 13.5 and 13.6 with Die B at a rate of approximately 100 mm^3/s (6.0 cm^3/min) (apparent shear rate of approximately 300 s^{-1}).

14. Calculation

14.1 For all calculations it is advisable to use actual measured values for die dimensions instead of the nominal values shown in 12.3.

14.2 Calculate the apparent shear rate, $\dot{\gamma}_{a,die,SR}$, of 13.6, 13.7, 13.9, and 13.10 for each die and apparent shear rate as follows:

$$\dot{\gamma}_{a,die,SR} = [(32Q_{SR})/(\pi D_{die}{}^3)] \tag{22}$$

where:

Q_{SR} = volumetric flow rate, mm^3/s for nominal apparent shear rate, SR, and

D_{die} = diameter of die, mm.

14.3 Calculate the apparent shear stress, $\tau_{a,A,1000}$, for Die A, and the apparent shear rate of 1000 s^{-1} as follows:

$$\tau_{a,A,1000} = \frac{P_{A,1000}}{4(L_A/D_A)} \tag{23}$$

14.3.1 The apparent shear stress for 13.7 is calculated similarly ($\tau_{a,A,300}$). The apparent shear stress for Die B used in 13.9 and 13.10 is calculated in a similar manner, using the dimensions of Die B, length L_B, and diameter D_B.

14.4 Calculate corrected shear rate, shear stress, and viscosity using corrections detailed in Section 10 (Test Method A).

15. Report

15.1 Report the following information:

15.1.1 Type of capillary rheometer used,

15.1.2 Identity of sample,

15.1.3 Pretreatment of sample, if any,

15.1.4 Temperature of test,

15.1.5 Corrected shear stress at 300 s^{-1},

15.1.6 Corrected shear stress at 1000 s^{-1},

15.1.7 Corrected viscosity at 300 s^{-1},

15.1.8 Corrected viscosity at 1000 s^{-1},

15.1.9 Shear sensitivity, *N*, and

15.1.10 Entrance effect, *E*.

16. Precision and Bias

16.1 Precision and bias studies for these test methods are currently being planned using Practice D4483.

17. Keywords

17.1 capillary rheometer; flow properties; piston; processing properties; screw extrusion; shear rate; shear stress; viscosity

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