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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}.

[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.

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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.

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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

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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:

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

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.

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