

Designation: D1238 - 10

Standard Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer¹

This standard is issued under the fixed designation D1238; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

- 1.1This test method covers measurement of the rate of extrusion of molten resins through a die of a specified length and diameter under prescribed conditions of temperature, load, and piston position in the barrel as the timed measurement is being made.
- 1.2Procedure A is a manual cutoff operation based on time used for materials having flow rates that fall generally between 0.15 and 50 g/10 min. Procedure B is an automatically timed flow rate measurement used for materials having flows from 0.50 to 900 g/10 min. By both procedures, the piston travel is generally the same during the timed measurement; the piston foot is about 46 and 20.6 mm above the die. Comparable flow rates have been obtained by these procedures in interlaboratory round-robin measurements of several materials described in 14.1. Provision is made for calculation of melt volume-flow rate as well as melt mass-flow rate.
- 1.1 This test method covers the determination of the rate of extrusion of molten thermoplastic resins using an extrusion plastometer. After a specified preheating time, resin is extruded through a die with a specified length and orifice diameter under prescribed conditions of temperature, load, and piston position in the barrel. Four procedures are described. Comparable results have been obtained by these procedures in interlaboratory round-robin measurements of several materials and are described in Section 15.
- 1.2 Procedure A is used to determine the melt flow rate (MFR) of a thermoplastic material. The units of measure are grams of material/10 minutes (g/10 min). It is based on the measurement of the mass of material that extrudes from the die over a given period of time. It is generally used for materials having melt flow rates that fall between 0.15 and 50 g/10 min (see Note 1).
- 1.3 Procedure B is an automatically timed measurement used to determine the melt flow rate (MFR) as well as the melt volume rate (MVR) of thermoplastic materials. MFR measurements made with Procedure B are reported in g/10 minutes. MVR measurements are reported in cubic centimetres/ten minutes (cm³/10 min). Procedure B measurements are based on the determination of the volume of material extruded from the die over a given period of time. The volume is converted to a mass measurement by multiplying the result by the melt density value for the material (see Note 2). Procedure B is generally used with materials having melt flow rates from 0.50 to 1500 g/10 min.
- 1.4 Procedure C is an automatically timed measurement used to determine the melt flow rate (MFR) of polyolefin materials. It is generally used as an alternative to Procedure B on samples having melt flow rates greater than 75 g/10 min. Procedure C involves the use of a modified die, commonly referred to as a "half-die," which has half the height and half the internal diameter of the standard die specified for use in Procedures A and B thus maintaining the same length to diameter ratio. The test procedure is similar to Procedure B, but the results obtained with Procedure C shall not be assumed to be half of those results produced with Procedure B.
- 1.5 Procedure D is a multi-weight test commonly referred to as a "Flow Rate Ratio" (FRR) test. Procedure D is designed to allow MFR determinations to be made using two or three different test loads (either increasing or decreasing the load during the test) on one charge of material. The FRR is a dimensionless number derived by dividing the MFR at the higher test load by the MFR at the lower test load. Results generated from multi-weight tests shall not be directly compared with results derived from Procedure A or Procedure B.

Note1—Round-robin testing indicates this test method may be suitable at flow rates up to 1500 g/10 min if the timing clock resolves the clapsed time to the nearest 0.01 s. 1—Polymers having melt flow rates less than 0.15 or greater than 900 g/10 min may be tested by the procedures in this test method; however, precision data have not been developed.

Note2—This test method and ISO 1133-1991 are technically equivalent.

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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1.3This standard does not purport to address 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. Specific precautionary statements are given in 5.7, 10.2.12, and 15.1.2. 2—Melt density is the density of the material in it molten state. It is not to be confused with the standard density value of the material. See Table 1.

Note 3—This test method and ISO 1133 address the same subject matter, but differ in technical content.

1.6 This standard does not purport to address 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

D618 Practice for Conditioning Plastics for Testing

D883 Terminology Relating to Plastics

D3364 Test Method for Flow Rates for Poly(Vinyl Chloride) with Molecular Structural Implications

D4000 Classification System for Specifying Plastic Materials

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

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ASTM D1238-10

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TABLE-2 $\underline{\mathbf{1}}$ Standard Test Conditions, Sample Mass, A and Testing Time B

Flow Range, g/10 min	Suggested Mass of Sample in Cylinder, g	Time Interval, min	Factor for Obtaining Flow Rate in g/10 min
0.15 to 1.0	2.5 to 3.0	6.00	1.67
>1.0 to 3.5	3.0 to 5.0	3.00	3.33
>3.5 to 10	4.0 to 8.0	1.00	10.00
>10 to 25	4.0 to 8.0	0.50	20.00
>25	4.0 to 8.0	0.25	40.00

^A This is a suggested mass for materials with melt densities of about 0.7 g/cm³. Correspondingly, greater quantities are suggested for materials of greater melt densities. Density of the molten resin (without filler) may be obtained using the procedure described by Terry, B. W., and Yang, K., "A New Method for Determining Melt Density as a Function of Pressure and Temperature," *SPE Journal*, SPEJA, Vol. 20, No. 6, June 1964, p. 540 or the procedure described by Zoller, Paul, "The Pressure-Volume-Temperature Properties of Polyolefins," *Journal of Applied Polymer Science*, Vol 23, 1979, p. 1051. It may also be obtained from the weight of an extruded known volume of resin at the desired temperature. For example, 25.4 mm (1 in.) of piston movement extrudes 1.804 cm³ of resin. An estimate of the density of the material can be calculated from the following equation:

resin density at test temperature = M/1.804

where:

M = mass of extruded resin.

^B See 9.143.

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.



2.2. ANSI Standard:

B46.1 on Surface Texture³

2.3 ISO Standard:

ISO 1133-1991<u>ISO 1133</u> Determination of the Melt-Mass Flow Rate (MFR) and the Melt Volume-Flow Rate (MVR) of Thermoplastics³

3. Terminology

- 3.1General:
- 3.1.1For definition of some of the technical terms used in this test method refer to Terminology
- 3.1 General:
- 3.1.1 Definitions are in accordance with Terminology D883 -unless otherwise specified.

4. Significance and Use

- 4.1 This test method is particularly useful for quality control tests on thermoplastics. Note3—Polymers having flow rates less than 0.15 or greater than 900 g/10 min may be tested by the procedures in this test method; however, precision data have not been developed.
- 4.2This test method serves to indicate the uniformity of the flow rate of the polymer as made by an individual process and, in this ease, may be indicative of uniformity of other properties. However, uniformity of flow rate among various polymers as made by various processes does not, in the absence of other tests, indicate uniformity of other properties.
- 4.3The flow rate obtained with the extrusion plastometer is not a fundamental polymer property. It is an empirically defined parameter critically influenced by the physical properties and molecular structure of the polymer and the conditions of measurement. The rheological characteristics of polymer melts depend on a number of variables. Since the values of these variables occurring in this test may differ substantially from those in large-scale processes, test results may not correlate directly with processing behavior.
 - 4.4The flow rate of a material may be measured under any of the conditions listed for it in
- 4.2 The data produced by this test method serves to indicate the uniformity of the flow rate of the polymer as made by an individual process. It is not to be used as an indication of uniformity of other properties without valid correlation with data from other tests.
- 4.3 The flow rate obtained with the extrusion plastometer is not a fundamental polymer property. It is an empirically defined parameter critically influenced by the physical properties and molecular structure of the polymer and the conditions of measurement. The rheological characteristics of polymer melts depend on a number of variables. It is possible that the values of these variables occurring in this test will differ substantially from those in large-scale processes, which would result in data that does not correlate directly with processing behavior.
- 4.4 Measure the flow rate of a material using any of the conditions listed for the material in 8.2. Additional characterization of a material can be obtained if more than one condition is used. In case two conditions are employed, a Flow Rate Ratio (FRR) may be obtained by dividing the flow rate at one condition by the flow rate at the other condition. For many materials, there are specifications that require the use of this test method, but with some procedural modifications that take precedence when adhering to the specification. Therefore, it is advisable to refer to that material specification before using this test method. Table 1 in Classification D4000 lists the ASTM materials standards that currently exist. An alternative test method for poly (vinyl chloride) (PVC) compounds is found in D3364.
- 4.5 Additional characterization of a material can be obtained if more than one condition is used. In the case that two or more conditions are employed, a Flow Rate Ratio (FRR) is obtained by dividing the flow rate at one condition by the flow rate at another condition. Procedure D provides one method to measure more than one condition in a single charge.
- 4.6 Frequently, variations in test technique, apparatus geometry, or test conditions, which defy all but the most careful scrutiny, exist, causing discrepancies in flow rate determinations. A troubleshooting guide is found in Appendix X2 and it is a resource to be used to identify sources of test error.

5. Apparatus

- 5.1 *Plastometer* Extrusion Plastometer (Alternative Names—Melt Indexer, Melt Flow Indexer):
- 5.1.1The apparatus shall be a dead-weight piston plastometer consisting of a thermostatically controlled heated steel cylinder with a die at the lower end and a weighted piston operating within the cylinder. The essential features of the plastometer, illustrated in Figs. 1 and
- Note 4—Older plastometers that were manufactured in accordance with "design specifications" detailed in previous revisions of this test method (pre D1238 04c) are deemed to be acceptable, as long as they meet the dimensional and performance specifications stated in this section.
- Note 5—Relatively minor changes in the design and arrangement of the component parts have been shown to cause differences in results among laboratories. For the best interlaboratory agreement, it is important that the design adhere closely to the description herein; otherwise, it should be determined that modifications do not influence the results. Refer to Fig. 1.

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

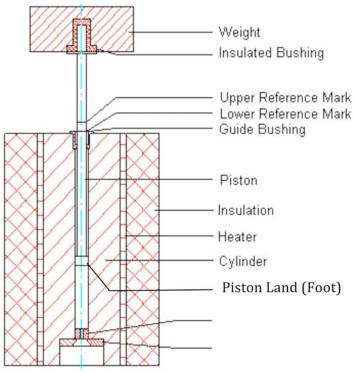


FIG. 1 General Arrangement of Extrusion Plastometer (See Section 5.)

- 5.1.1 The apparatus shall be a dead-weight piston plastometer consisting of a thermostatically controlled heated steel cylinder with a bore that contains a die at the lower end, and a weighted piston operating within the cylinder. The essential features of the plastometer, illustrated in Figs. 1 and 2, are described in 5.2-5.8. All dimensional measurements shall be made when the article being measured is at 23±5°C.
- 5.1.2Relatively minor changes in the design and arrangement of the component parts have been shown to cause differences in results among laboratories. It is important, therefore, 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. 5.2-5.12. The bore of the extrusion plastometer shall be properly aligned in the vertical direction (see Appendix X1). All dimensional measurements shall be made when the article being measured is at 23 ± 5 °C.
- 5.2 Cylinder—The steel cylinder shall be 50.8 mm in diameter, 162 mm in length with a smooth, straight hole 9.5504±0.0076 mm in diameter, displaced 4.8 mm from the cylinder axis. Wells for a thermal sensor (thermoregulator, thermistor, etc.) and thermometer shall be provided as shown in Fig. 1. A 3.2-mm plate shall be attached to the bottom of the cylinder to retain the die. A hole in this plate, centered under the die and countersunk from below, allows free passage of the extrudate. The cylinder may be supported by at least two 6.4-mm high-strength screws at the top (radially positioned at right angles to the applied load) or by

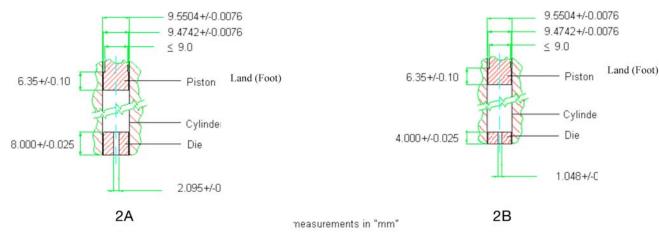


FIG. 2 Details of Extrusion Plastometer



at least two 10-mm diameter rods screwed into the side of the cylinder for attaching to a vertical support. The essential dimensions of a satisfactory cylinder of this type are shown in Fig. 1 (Note 4). The cylinder bore should be finished by techniques known to produce approximately 12 rms or better in accordance with ANSI B46.1.

Note4—Cylinders made of SAE 52100 or other equivalent steel heat-hardened to 60–65 Rockwell Hardness Scale C give good service when used at temperatures below 200°C. Cylinder liners of cobalt-chromium-tungsten alloy are also satisfactory to 300°C.—The cylinder shall be 50 mm ± 10 mm in diameter, 115 to 180 mm in length with a smooth, straight bore 9.5504 ± 0.0076 mm in diameter. The cylinder bore shall be manufactured in a way that produces a finish approximately 12 rms or better in accordance with ANSI B46.1. Means shall be provided to monitor the temperature inside the bore.

- 5.3 *Die*—The outside of the steel die shall be such diameter that it will fall freely to the bottom of the 9.5504±0.0076 mm diameter hole in the cylinder (Note 5). The die shall have a smooth straight bore 2.0955±0.0051 mm in diameter and shall be 8.000±0.025 mm in length. The bore and its finish are critical. It shall have no visible drill or other tool marks and no detectable eccentricity. The die bore shall be finished by techniques known to produce approximately 12 rms or better in accordance with ANSI B46.1. Die (Orifice):
- 5.3.1 Standard Die—The outside diameter of the die shall be such that it will fall freely to the bottom of the hole in the cylinder. The orifice of the die shall have a smooth straight bore 2.095 ± 0.005 mm in diameter and shall be 8.000 ± 0.025 mm in length (see Fig. 2). The bore of the orifice and its finish are critical. It shall have no visible drill or other tool marks and no detectable eccentricity. The bore of the orifice shall be manufactured by techniques known to produce finishes approximately 12 rms or better in accordance with ANSI B46.1.
- 5.3.2 "Half" Die—Used for Procedure C. When testing polyolefins with a MFR of 75 or greater (using the standard die), an alternate die has shown to improve the reproducibility of results by reducing the flow rate of these materials. The outside diameter of the die shall be such that it will fall freely to the bottom of the hole in the cylinder. The orifice shall have a smooth straight bore 1.048 ± 0.005 mm in diameter and shall be 4.000 ± 0.025 mm in length (see Fig. 2A). The bore of the orifice and its finish are critical. It shall have no visible drill or other tool marks and no detectable eccentricity. The bore of the orifice shall be manufactured by techniques known to produce finishes approximately 12 rms or better in accordance with ANSI B46.1 (Note Note 6). No spacer shall be used with this die.

Note5—Recommended die material is tungsten carbide. Also satisfactory are steel, synthetic sapphire, and cobalt-chromium-tungsten alloy. 6—Recommended die material is tungsten carbide. Also satisfactory are steel, synthetic sapphire, and cobalt-chromium-tungsten alloy. When softer materials are used, it will be necessary to conduct critical dimensional checks and visual inspections on the die more often.

5.4 Piston:

5.4.1The piston shall be made of steel with an insulating bushing at the top as a barrier to heat transfer from the piston to the weight. The land of the piston shall be 9.4742±0.0076 mm in diameter and 6.35±0.13 mm in length. The piston design may incorporate means for land replacement, for example, having threads and flats immediately above the land. Above the land, the piston shall be no larger than 8.915 mm in diameter (Note 6). The finish of the piston foot shall be 12 rms in accordance with ANSI B46.1. If wear or corrosion is a problem, the piston should be of stainless steel and equipped with a detachable foot for ease of replacement.

Note6—To improve standardization it is preferable that the piston be guided with a loose-fitting metal sleeve at the top of the cylinder.

Note7—Pistons of SAE 52100 steel with the bottom 25 mm, including the foot, hardened to a Rockwell hardness, C scale, of 55 to 59 have been found to give good service when used at temperatures below 200°C.

- 5.4.2The piston shall be scribed with two reference marks 4 mm apart in such fashion that when the lower mark coincides with the top of the cylinder or other suitable reference point, the bottom of the piston is 48 mm above the top of the die (see
- 5.4.1 The piston shall be made of steel. There shall be insulation at the top as a barrier to heat transfer from the piston to the weight. The piston shall be prevented from rubbing on the bore. Most commercially available instruments use a loose fitting metal guide sleeve, but other methods are acceptable. The weight of the sleeve shall not be considered as part of the test load. The land (foot) of the piston shall be 9.4742 ± 0.0076 mm in diameter and 6.35 ± 0.10 mm in length. Above the land, the piston shall be relieved to ≤ 9.0 mm in diameter (see Fig. 2). The piston land shall be manufactured by techniques known to produce finishes approximately 12 rms in accordance with ANSI B46.1. If corrosion is a problem, the piston or piston land, if removable, shall be made of corrosion resistant material.
- 5.4.2 For procedure A, the piston shall be scribed with two reference marks 4 mm apart in such fashion that when the lower mark coincides with the top of the cylinder, guide sleeve or other suitable reference point, the bottom of the piston is 48 mm above the top of the die (see Fig. 1) and the timed test run shall start within these two reference marks. The targeted starting point shall be 46 ± 2 mm above the upper face of the die. (see Fig. 1).
 - 5.4.3 The combined weight of piston and load shall be within a tolerance of ± 0.5 % of the selected load.
 - 5.5 *Heater*Temperature Control System:
- 5.5.1The equipment must have a heater capable of heating the apparatus so that the temperature at 10 mm above the die can be maintained within ± 0.2 °C of the desired temperature during the test. The temperature of the barrel, from 10 mm to 75 mm above the top of the die, must be maintained within $\pm 1\%$ of the set temperature (°C).

Notes —At temperatures higher than 200°C this degree of temperature control may be more difficult to obtain.

5.5.2Calibrate the temperature-indicating device by means of a light-gage probe-type thermocouple or a platinum-resistance temperature sensor having a short sensing length. The thermocouple should be encased in a metallic sheath having a diameter of approximately 1.6 mm with its hot junction grounded to the end of the sheath. Insert the temperature sensor into the melt from the top of the cylinder so that it is 10±1 mm above the upper face of the die. The temperature sensors shall be used with a potentiometer having a sensitivity of at least 0.005 mV, or a temperature readout having a sensitivity of at least 0.1°C. Calibration should also be verified at 75 mm above the upper face of the die. An alternate technique for calibration is to use a sheathed thermocouple or platinum-resistance temperature sensor with tip diameter of 9.4±0.1 mm for insertion in the bore without material present. An example of this is shown in Fig. 3. Calibration of the temperature-indicating device shall be verified at each run temperature.

Note9—The response of the temperature sensing device may be affected by immersion level. Take care to ensure adequate insulation of the device sensor and stabilization of the barrel temperature.

- 5.5.3Heat shall be supplied by electric band heater(s) covering the entire length of the cylinder. The heater(s) may be single-or multi-element, depending upon the manufacturer's control means. The heater(s) plus control system must be capable of maintaining the temperature within the required ±0.2°C of the set point. The temperature sensor and readout equipment must be calibrated to a traceable national standard (that is, NIST) at least once per year. The cylinder with the heater(s) shall be lagged with 38 mm of foamed-glass insulation. An insulating plate 3.2 mm in thickness shall be attached to the bottom of the cylinder to minimize heat loss at this point.
- 5.5.1 The equipment shall have the capability of heating and maintaining the temperature inside the bore of the cylinder in accordance with the requirements specified in Table 2 throughout the duration of the test.
- 5.5.2 The preferred method for calibrating the temperature is to use a temperature sensor assembly having a sensor with at least an accuracy of ± 0.08 °C at 200°C and a 20 ± 0.5 -mm long brass tip press fit on the end of the sensor. The diameter of the brass tip shall closely match the diameter of the die and the length of the active measuring length of the temperature sensor (see Appendix X3).
- 5.5.3 Temperatures shall be verified with the bottom of the temperature sensor at 10 and 75 ± 1 mm above the upper face of the die and at each test temperature, without touching the die. Allow at least four minutes for equilibrium of temperature to be reached for each position. Temperature variation shall be determined over a minimum of 15 minutes. When using the "half" die, the temperature indicating device shall be calibrated as stated in Table 2 except temperatures are measured at 79 ± 1 mm and 14 ± 1 mm above the upper surface of the die.
- 5.5.4 An alternative method is to insert the temperature sensor without a brass tip into the melt from the top of the cylinder so that it is 10 and 75 ± 1 mm above the upper face of the die.
- 5.5.5 The temperature sensor and readout equipment used for calibration of the extrusion plastometer shall be traceable to a national standard (for example, NIST).
- 5.6 Temperature Controller—The type of controller and sensor must be capable of meeting the required control tolerance specified in 5.5.1 Timing Device/System—For Procedure A, a timing device with an accuracy of 0.1 s shall be used. For Procedures B, C, and D, an automatic timing system shall measure and time piston movement within the specified travel range. The requirements of the automatic timing system shall be as follows:
 - 5.6.1 Sense and indicate the piston travel time within ± 0.01 s.
 - 5.6.2 Measure piston travel within $\pm 0.4\%$ of the nominal selected value (see 10.7) for use in the flow rate calculations.
- 5.6.3 Operate within a fixed portion of the cylinder. This is defined as the portion of the cylinder between 48 mm and 18.35 mm above the top of the die.
- 5.6.4 Any effects on the applied load caused by the Timing Device/System must be included in the allowable tolerance given in 5.4.3.
- 5.7Thermometer— Thermometers having a range of 4°C graduated in 0.2°C divisions may be used to indicate temperature. The temperature at this point may not necessarily be the temperature of the material 10 mm above the die. The thermometer may be used to monitor indirectly the temperature of the material 10 mm above the die and may be calibrated by reference to a thermocouple or platinum resistance temperature sensor inserted in the material 10 mm above the die. See 5.5.2 for a description of a method for measuring temperature. (Warning—Caution should be observed with the use of a mercury-filled thermometer. Mercury vaporization occurs if the thermometer is broken. Mercury thermometers are not to be used at or above the boiling point of mercury, which is 357°C.)

5.8

- 5.6.5 The equipment used to calibrate the Timing Device/System shall be traceable to a national standard (for example, NIST). 5.7 *Operating Tools*:
- <u>5.7.1</u> Level—Provision shall be made for—Used to verify the vertical alignment of the bore of the extrusion plastometer. This is necessary to minimize subtractive loads resulting from rubbing or friction between the piston tip and sidewall. Means of alignment are discussed in Appendix X1.
 - 5.7.2 Calibrated Go/No-Go Gauge:
- 5.7.2.1 For the standard die, a go/no-go gauge suitable to inspect the inner diameter of the hole in the die. The go member of the gauge shall be no smaller than 2.090 mm. The no-go member shall be no larger than 2.100 mm.



TABLE 1 2 StMandximum Alloward Tblest Cond Variations, in Temperature, with Distand Loce and Time Throughout the Test

Condition	Temperature , °C	To	Approximate Pres	sure	
Condition	l'emperature, o	tal Load including Piston, kg	Test temperature , °C	Temperature tolerance, °C	
Standard Designation	kP a	psi	rest temperature, o	et point	
<u>T °C</u>	At 75 ± 1 mm	At 10 ± 1 mm			
	above the die	above the die			
	surface (°C) ^A	surface (°C) ^A			
80/2.16	80	2.16			
$125 \le T < 250$	<u>±2.0</u>	<u>±0.</u> 16		2	
125/0. 325	125	0.325	44.8	6 .5	
$250 \le T < 300$	<u>±2.5</u>	<u>±0</u> .325	44.8	6 .5	
125/2.16	125	2.16	298.2	43.25	
<u>300 ≤ T</u>	<u>±3.0</u>	<u>±1</u> 6	298.2	43.25	
150/2.16	150	2.16	298.2	43.25	
190/0.325	190	0.325	44.8	6.5	
190/2.16	190	2.16	298.2	43.25	
190/21.60	190	21.60	2982.2	432.5	
200/5.0	200	5.0	689.5	100.0	
230/1.2	230	1.2	165.4	24.0	
230/3.8	230	3.8	524.0	76.0	
265/12.5	265	12.5	1723.7	250.0	
275/0.325	275	0.325	44.8	6.5	
230/2.16	230	2.16	298.2	43.25	
190/1.05	190	1.05	144.7	21.0	
190/10.0	190	10.0	1379.0	200.0	
300/1.2	300	1.2	165.4	24.0	
190/5.0	190	5.0	689.5	100.0	
235/1.0	235	1.0	138.2	20.05	
235/2.16	235	2.16	298.2	43.25	
235/5.0	235	5.0	689.5	100.0	
250/2.16	250	2.16	298.2	43.25	
310/12.5	310	12.5	1723.7	250.0	
210/2.16	/ 210	2.16	298.2	43.25	
285/2.16	285	2.16	298.2	43.25	
315/5.0	315	5.0	689.5	100.0	
372/2.16	372	2.16	298.2	43.25	
372/5.0	372	5.0 V AV	689.5	100	
297/5.0	297	5.0	689.5	100	
230/21.6	230	21.6	2982.2	432.5	
230/5.0	230	5.0	689.5	100	
265/21.6	265	11 2 2 2 1 21.6	2982.2	432.5	
265/31.6	265	31.6	4361.2	632.5	
271.5/2.16	/sist 271.5 cf	44-775@ 2.16 d2a-a52	1_30 298.2 6h6a	43.25	
220/10	220	10.0	1379.0	200.0	
250/1.2	250	1.2	165.4	24.0	
265/3.8	265	3.8	524.0	76.0	
265/5	265	5.0	689.5	100 .0	

 $^A\!W\!$ hen using the "half" die, the temperature indicating device shall be calibrated as stated in this table except temperatures are measured at nominal 79 \pm 1 mm and 14 mm \pm 1mm above the upper surface of the die.

- 5.7.2.2 For the "half" die, a go/no-go gauge suitable to inspect the hole in the die. The go member of the gauge shall be no smaller than 1.043 mm. The no-go member shall be no larger than 1.053 mm.
 - 5.7.3 *Funnel*—For charging samples to the cylinder
 - 5.7.4 Packing Tool—For charging samples to the cylinder
 - 5.7.5 Spatula—Or similar device used to cut extrudate
 - 5.7.6 Balance—Capable of weighing to 0.001 g
 - 5.8 Cleaning Equipment:
 - 5.8.1 Cylinder bore cleaning tool
 - 5.8.2 Die cleaning tool
 - 5.8.3 *Cotton patches*
- 5.9 Accessory Equipment—Necessary accessories include equipment for charging samples to the cylinder, a funnel, a die plug, a tool for cutting off the extruded sample, a timer or stop watch, cleaning equipment, go/no-go gages, a balance accurate to ±0.001 g, and, when required, a weight or weight-piston support. Weight Support—Used with high Melt Flow Rate material to prevent material from flowing out during the preheat period.
- 5.10 *Die Plug*—Used with high melt flow rate material to plug the die when weight support measures are not enough to prevent material from flowing out during the preheat period.
 - 5.11 Automatic Weight Lowering and Lifting Device—Optional for Procedures A, B, and C, but required for Procedure D.



Device for automatically applying test loads to the piston. This device is often useful as a weight support.

5.12 Multi-Weight (Flow Rate Ratio) Accessory—For testing in accordance with Procedure D, it is necessary to have an accessory that permits Melt Flow Rate determinations to be made using two or three different test loads on one charge of material by loading or unloading test loads, or both, at pre-set heights.

Note10—Satisfactory operation of the apparatus for polyethylenes can be ascertained by making measurements on NIST Standard Reference Materials (SRMs) certified for melt flow rate. The four SRMs certified under condition 190/2.16 are SRM 1473 with a flow rate of 1.29 g/min, SRM 1474 with a flow rate of 5.03 g/10 min, SRM 1496 with a flow rate of 0.26 g/10 min, and SRM 1497 with a flow rate of 0.19 g/10 min. SRM 1475a is certified under condition 190/3.25 with a flow rate of 2.20 g/10 min. _7—Different manufacturers of equipment may offer options that help to automate the test and/or data collection. These are acceptable for use provided they operate in a manner that does not conflict with descriptions in Section 5 and the procedures listed in Sections 9, 10, 11, and 12.

6. Test Specimen

6.1The test specimen may be in any form that can be introduced into the bore of the cylinder, for example, powder, granules, strips of film, or molded slugs. It may be desirable to preform or pelletize a powder.

6.1 The test specimen is permitted to be in any form that allows it to be introduced into the bore of the cylinder, for example, powder, granules, strips of film, or molded slugs.

Note 8—It may be desirable to pre-form or pelletize a powder. Trapped air causes the piston to fall faster, hence measurements are affected.

7. Conditioning

7.1 Many thermoplastic materials do not require conditioning prior to testing. Materials which contain volatile components, are chemically reactive, or have other special characteristics most probably require appropriate conditioning procedures. Moisture not only affects reproducibility of flow rate measurement but, in some types of materials, degradation is accelerated by moisture at the high temperatures used in testing. Check the applicable material specification for any conditioning requirements before using this test. See Practice D618 for appropriate conditioning practices.

8. Procedural Conditions

8.1 Standard conditions of test are given in <u>Table 1 Table 2</u>. Test conditions shall be shown as: Condition _ _ _ / _ _ _, where the temperature in degrees Celsius is shown first, followed by the weight in kilograms. For example: Condition 190/2.16.

8.2The following conditions have been found satisfactory for the material listed:

Material D	Condition	
Acetals (copolymer and homopolymer)	190/2.16	190/1.05
Acrylics	230/1.2	230/3.8
Acrylonitrile-butadiene-styrene	200/5.0	230/3.8
	220/10	
Acrylonitrile/butadiene/styrene/polycarbonate ASTM D1238-	230/3.8	250/1.2
blanda	265/3.8	265/5.0
Gellulose esters teh.ai/catalog/standards/sist/32acf044-775	190/0.325 ab Z 1 - 50 e9 le 6 b 6 a 6 5 / a	190/2.16
	190/21.60	210/2.16
Ethylene-chlorotrifluoroethylene-copolymer	271.5/2.16	
Ethylene-tetrafluoroethylene-copolymer	297/5.0	
Nylon	275/0.325	235/1.0
•	235/2.16	235/5.0
	275/5.0	
Perfluoro(ethylene-propylene) copolymer	372/2.16	
Perfluoroalkoxyalkane	372/5.0	
Polycaprolactone	125/2.16	80/2.16
Polychlorotrifluorethylene	265/12.5	
Polyether sulfone (PES)	380/2.16	360/10
	343/2.16	
Polyethylene	125/0.325	125/2.16
	250/1.2	
	190/0.325	190/2.16
	190/21.60	190/10
	310/12.5	
Polycarbonate	300/1.2	
Polymonochlorotrifluoroethylene	265/21.6	
	265/31.6	
Polypropylene	230/2.16	
Polyphenyl sulfone (PPSU)	365/5.0	380/2.16
Polystyrene	200/5.0	230/1.2
	230/3.8	190/5.0
Polysulfone (PSU)	343/2.16	360/10
Polyterephthalate	250/2.16	210/2.16
	285/2.16	
Poly(vinyl acetal)	150/21.6	
Poly(vinylidene fluoride)	230/21.6	
	230/5.0	
Poly(phenylene sulfide)	315/5.0	



Styrene acrylonitrile	220/10	230/10
	230/3.8	
Styrenic Thermoplastic Elastomer	190/2.16	200/5.0
Thermoplastic Elastomer-Ether-Ester	190/2.16	220/2.16
·	230/2.16	240/2.16
		250/2.16
Thermoplastic elastomers (TEO)	230/2.16	
Vinylidene fluoride copolymers	230/21.6	
	230/5.0	
		for T _m = 100° use 120/5.0 or 21.6

8.2 Table 3 shows conditions that have been found satisfactory for the material listed.

Note11—Some materials may require special materials of construction or handling for performing this test. Please refer to the material specification for appropriate recommendations.

8.3If more than one condition is used and the material is polyethylene, the determination of Flow Rate Ratio (FRR) has been

TABLE 3 Test Conditions for Select Materials

TABLE 3 Test Conditions for Select Materials				
Material	Temperatures	Weights		
Acetals (copolymer and homopolymer)	190	1.05 / 2.16		
Acrylics	230	1.2 / 3.8		
Acrylonitrile-butadienestyrene	200	5.0		
	220	10		
	230	3.8		
Acrylonitrile/butadiene/styrene/polycarbonate blends	230	3.8		
	250	1.2		
	265	3.8 / 5.0		
Cellulose esters	190	0.325 / 2.16 / 21.6		
	<u>210</u>	<u>2.16</u>		
Ethylenechlorotrifluoroethylene copolymer	271.5	<u>2.16 / 5.0</u>		
Ethylene-tetrafluoroethylene copolymer	297	<u>5.0</u>		
Ethylene-tetrafluoroethylene copolymer Nylon	297 235 275 372 372 80	1.0 / 2.16 / 5.0		
	<u>275</u>	0.325 / 5.0		
Perfluoro(ethylenepropylene) copolymer	372	<u>2.16</u>		
Perfluoro(ethylenepropylene) copolymer Perfluoro(ethylenepropylene) copolymer Perfluoroalkane	$1021(\frac{372}{2})$, 1021.2	<u>5.0</u>		
Polycaprolactone	<u>80</u>	<u>2.16</u>		
	nt P (265 view)	2.16		
Polychlorotrifluorethylene		12.5		
Polyetheretherketone (PEEK)	360	10		
	380	2.16		
D (PEO)	400	2.16		
Polyether sulfone (PES) ASTIV	4 D1238-1 343	2.16		
Polyethylene Polyethylene	125	0.325 / 2.16		
	of044-775d <u>190</u> 12a-a521-30e	9 fe 6 b <u>0.325 / 2.16 / 5 / 10 / 21.6</u>] ()		
	310	1.2 12.5		
Polycarbonate	300	1.2		
Polymonochlorotrifluoroethylene	<u>265</u>	21.6 / 31.6		
Polypropylene	230	2.16		
Polyphenyl sulfone (PPSU)	<u>265</u> 365	5.0		
<u>. s.yp.iony. canone (i 1 55)</u>	380	2.16		
Polystyrene	190	5.0		
	200	5.0		
	230	1.2 / 3.8		
Polysulfone (PSU)	343	2.16		
	360	10		
Polyterephthalate	210	2.16		
	250	2.16		
	285	2.16		
Poly(vinyl acetal)	150	21.6		
Polyvinyl chloride (PVC), rigid compound	190	<u>21.6</u>		
Poly(vinylidene fluoride)	230	<u>5.0 / 21.6</u>		
Poly(phenylene sulfide)	315	<u>5.0</u>		
Styrene acrylonitrile	220	10		
	230	3.8 / 10		
Styrenic Thermoplastic Elastomer	190	<u>2.16</u>		
They manifestic Floateman Filter Filter	200	5.0		
Thermoplastic Elastomer-Ether-Ester	190	2.16 2.16		
	220 230	2.16 2.16		
	230 240			
	240 250	<u>2.16</u> 2.16		
Thermoplastic elastomers (TEO)	250 230	2.16 2.16		
Vinylidene fluoride copolymers	120*	5.0 / 21.6*		
*For Tm = 100°C	230	2.16 / 5.0		
<u> 1 01 1111 – 100 0</u>	200	<u> </u>		



found to be useful. The FRR is a dimensionless number derived by dividing the flow rate at Condition 190/10 by the flow rate at Condition 190/2.16.

Note12—When determining such a ratio of flow rates for a material at the same temperature under different loads, it has been found that precision is maximized when one operator uses one Procedure (A or B), the same plastometer, and the same die for both measurements (the die need not be removed from the plastometer between the two determinations).

9. 9—Some materials may require special materials of construction or handling for performing this test. Please refer to the material specification for appropriate recommendations.

9. Procedure A—Manual Operation

- 9.1Select conditions of temperature and load from Table 1 in accordance with material specifications such that flow rates will fall between 0.15 to 50 g/10 min.
 - 9.2Ensure that the bore of the extrusion plastometer is properly aligned in the vertical direction. (See Appendix X1.)
 - 9.3Inspect the apparatus and die for cleanliness. If it is not clean, see 9.11.
- 9.1 Select conditions of temperature and load from 8.2 or in accordance with material specifications. Where multiple test conditions exist, test conditions shall be agreed upon by the cooperating laboratories. If test conditions are not known, select conditions that result in flow rates between 0.15 to 50 g/10 min.
- 9.2 Inspect the extrusion plastometer for cleanliness (see Note 10). All surfaces of the cylinder bore, die and piston shall be free of any residue from previous tests.

Note13—The degree of cleanliness can significantly influence the flow rate results, therefore a thorough method of cleaning must be established. It has been found that three swabs of the barrel is satisfactory for most materials and that the die, barrel, and piston are more easily cleaned while hot-

- 9.4Cheek the die bore diameter with appropriately sized no-go/go gages prior to testing. Make frequent cheeks to determine whether the die diameter (tested with die at 23±5°C) is within the tolerances given in 5.3.—10—The degree of cleanliness can significantly influence the flow rate results, therefore a thorough method of cleaning should be established. It has been found that swabbing the barrel with a clean cotton patch several times is satisfactory for most materials and that the die, barrel, and piston are more easily cleaned while hot. For materials that are difficult to clean from the metal surfaces, use of a brass brush has been found to be satisfactory.
- 9.3 Check the die bore diameter at frequent intervals with appropriately sized go/no-go gauges (checked with die at $23 \pm 5^{\circ}$ C) to verify that the die is within the tolerances given in 5.3.1. Visually examine the die bore to verify that it is not scratched or damaged. Also visually inspect the land of the piston foot to verify that it is not scratched or damaged and use a calibrated micrometer to verify that the dimensions are within the tolerances given in 5.4.1 (see Note 11).

Note14—Cleaning and usage can result in a die diameter that is out of specifications. Data has shown that erroneous results will be obtained if the die diameter is not within the appropriate tolerances.

- 9.5 Verify that the temperature is stable and within ±0.2 °C of the appropriate test temperature as specified in 5.5.1.
- 9.6Insert the die and the piston. The temperature of the cylinder with the piston and die in place must be stable at the appropriate test temperature 15 min before testing is begun. When equipment is used repetitiously, it should not be necessary to heat the piston and die for 15 min. 11—Cleaning and usage will eventually cause damage or wear to the, bore, die and the land of the piston. Data has shown that erroneous results will be obtained if these components are not within the appropriate tolerances.
 - 9.4 Set the temperature in accordance with the manufacturer's instructions.
- 9.5 Insert the die and the piston into the bore. Allow the temperature of the cylinder, with the piston and die in place, to stabilize within $\pm 0.2^{\circ}$ C of the selected test temperature for at least 15 min before starting a test. When equipment is used continuously, it is not necessary to heat the piston and die for 15 minutes when runs of the same or similar material at the same test temperature are being measured over a continuous time frame, provided the piston and die are cleaned and re-inserted into the bore within five minutes after removal from the extrusion plastometer at the end of each test. If the piston, or die, or both, are removed from the bore for longer than five minutes, they shall be considered "cold" and the full 15 minutes heating stabilization time shall be required.
- 9.6 Remove the piston from the bore (see Note 12). Within 60 seconds, charge the cylinder with a weighed portion of the sample in accordance with the expected flow rate (as given in Table 1), reinsert the piston and add the appropriate weight. The charging weights given in Table 1 are merely suggestions, and the actual charging weight for a specific sample, if not known, will need to be determined by trial and error. Adjust the charge weight so that the piston is in the proper position at the end of the pre-heat period. If necessary, it is acceptable to purge excess material from the cylinder bore so that the piston is in the proper position at the end of the pre-heat period. Purging of material done at conditions with greater force than testing conditions shall be completed at least 2 min prior to making the initial cut-off (see Note 13).

Note15—The reduction in heating time when the unit is being used repetitiously is only allowed when runs of the same or similar material are being measured over a continuous time frame. If the piston and die are removed and cleaned, they should be considered "cold" and the full 15 minutes heating stabilization time required.

9.7Remove the piston and place it on an insulated surface. Charge the cylinder within 1 min with a weighed portion of the sample in accordance with the expected flow rate, as given in Table 2. Reinsert the piston and add the appropriate weight. 12—Placing