



Designation: D 3364 – 99

Standard Test Method for Flow Rates for Poly(Vinyl Chloride) with Molecular Structural Implications¹

This standard is issued under the fixed designation D 3364; 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 This test method is an extension of Test Method D 1238 specific to the measurement of flow rates of poly(vinyl chloride) (PVC) compounds while detecting and controlling various polymer instabilities associated with the flow rate.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—There is no similar or equivalent ISO standard.

2. Referenced Documents

2.1 ASTM Standards:

- D 883 Terminology Relating to Plastics²
- D 1238 Test Method for Flow Rates of Thermoplastics by Extrusion Plastometer²
- D 3835 Test Method for Rheological Properties of Thermoplastics with a Capillary Rheometer³
- E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods⁴
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁴

3. Terminology

3.1 *Definitions*—For definitions related to plastics, see Terminology D 883.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 See Test Method D 3835, Sections 5.1, 5.2, and 5.3.

3.2.2 *Flow* is the reciprocal of the viscosity; therefore, the flow is defined as the volumetric displacement through a controlled orifice and is expressed as shear rate over shear stress.

NOTE 2—Since PVC obeys the power law function, the above relation-

ship can be expressed as follows:

(Viscosity) (Shear Rate)^{1-N} = (shear stress) in which the shear rate is expressed as $4Q/\pi R^3$ and depends on the power law exponent N .

Since Q is the volumetric flow rate in terms of cubic millimetres/second and R is the radius of the die, it follows that the flow rate varies much faster than the viscosity as a result of N . This means that the flow is much more sensitive to change than the viscosity. For PVC, N varies from 0.1 to 0.33.

3.2.3 Flow rate by this test method is the rate in milligrams/minute at which polymer flows through a specific die (see Fig. 1) with a total load on the ram of 20 kg at a temperature of 175°C.

4. Summary of Test Method

4.1 Conditions:

4.1.1 In order to test a wide variation of flow rates covering semirigid as well as nonrigid PVC compounds, the following standard conditions are used:

Temperature	175°C (347°F)
Total load on piston	20 000 g
Approximate pressure	2758 kPa (400 psi)
Charge	2.15 ± 0.05 g
Plugged orifice with 120° entrance angle	

4.2 Basis Principles:

4.2.1 The lower temperature (relative to 190°C) is chosen to minimize thermal decomposition, maximize sensitivity of the flow rate to structural changes in the PVC compound, and to allow a wide latitude of useful conditions associated with the load on the piston.

4.2.2 Modern extrusion plastometers have been redesigned to accommodate much higher loads. Current research for multi-weight testing has reached levels of 50 kg and these are particularly good for rigid PVC. Previously, flow rates for rigid PVC were limited to 190°C. Recent research has shown that rigid PVC can be run at 150°C and 50 kg loads. This weight is effective for all semirigid PVC and even some nonrigid compounds. For flow rates in excess of 10 g/10 min (1000 mg/min), lower weights are used, for example, color concentrates used in PVC extrusion may have flow rates in excess of 100 g/10 min (10 000 mg/min) and will require a 5-kg load on the piston.

4.2.3 When the flow rates become very small as in the case of the stiffest semirigid containing 25 parts of plasticizer (flow rates below 15 mg/min), it may be desirable to use the die (flat

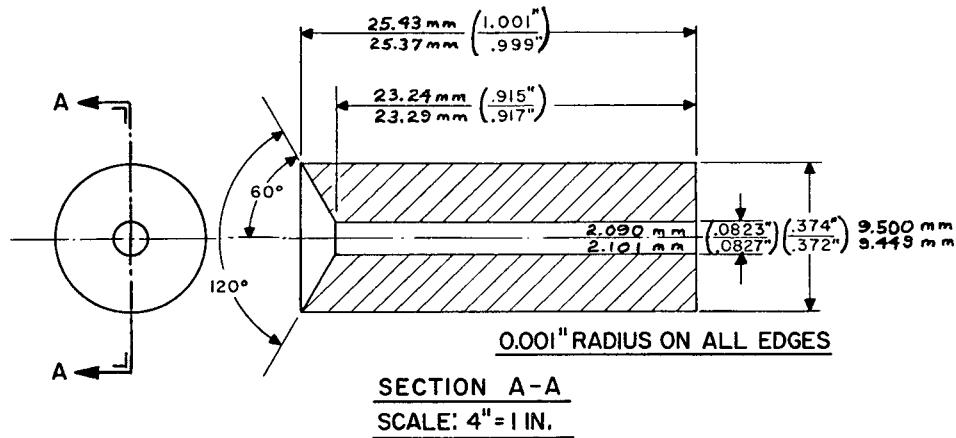
¹ This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.30 on Thermal Properties (Section D20.30.08).

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² *Annual Book of ASTM Standards*, Vol 08.01.

³ *Annual Book of ASTM Standards*, Vol 08.02.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.



NOTE—Specify material when ordering.

FIG. 1 Die Used for Test Method D 3364

entry 8 mm (0.315 in.) long) in Test Method D 1238. The flow is so slow that little rheological instability exists. By changing the die, approximately a tenfold increase in flow is achieved.

4.2.4 The charge size is important. Many PVC compounds are elastic in nature, causing a severe loss in pressure from the bottom of the piston through the material to the orifice of the die. Evidence indicates that the force to extrude may be reduced by as much as 67 % using a 9-g charge and 4.14 MPa (600 psi) on the piston.

4.2.5 The plugged orifice should be used in all PVC compound work since the amount of charge is limited and since the plugging for various times has been observed to give significant variations in the flow rate.

5. Significance and Use

5.1 This test method is useful for quality-control tests on PVC compounds having a wide range of melt viscosities. Measurements are made at shear rates close to 1 s^{-1} .

5.2 In addition to the properties mentioned in Test Method D 1238, this technique is sensitive to plasticizer content, polymer molecular weight, polymer stability (both thermally and rheologically), shear instability, and general composition. The sensitivity of the material to temperature necessitates slightly tighter controls than those stated in Test Method D 1238.

5.3 The sensitivity of this test method makes it useful for correlating with processing conditions and as an aid in predicting changes in processing. However, as a one-point measure of flow relative to shear rate, its one drawback is that the same PVC melt flow values can be obtained for materials having different processibility; the chance of this happening is minimized, however, if the compounds are similar in composition.

5.4 Correlations with a wide range of processing conditions have supported the conclusions that little or no change in composition occurs during the test. Thus, this test is able to detect and follow profound changes which occur during extrusion, injection molding, milling, or mixing. These changes are due to three types of measured instability in polymers:

5.4.1 Thermal instability due to temperature effect.

5.4.2 Shear instability due to breaking of polymer bonds.

5.4.3 Rheological instability due to nonuniform distributions of widely different viscosity or molecular weight elements.

5.4.4 Thus, implications with respect to PVC molecular structural changes can be detected and predicted.

6. Apparatus

6.1 *Plastometer*—The apparatus is identical to that used in Test Method D 1238 except for the die. An alternative thermoregulator is suggested for improved temperature control.

6.2 Die:

6.2.1 The die is approximately three times as long as the one used in Test Method D 1238, a major factor in controlling any rheological instability in the polymer. If instability still occurs, particularly at high flow rates, dies 2 in. or longer can be used to improve the L/D ratio. The dimensions of the regular die are shown in Fig. 1.

6.3 *Thermoregulator*—Although a thermoregulator identical to that used in Test Method D 1238 may be used (provided it regulates to $175 \pm 0.1^\circ\text{C}$), a unit⁵ coupled with a 90° angle thermometer, divided into 0.1°C is suggested for improved reproducibility. Most regulators designed for Test Method D 1238 cannot control better than $\pm 0.2^\circ\text{C}$. The sensitivity of this test method is illustrated by the fact that a 19 % change in flow rate is observed on a typical PVC compound with a 1°C change in temperatures.

6.4 *Thermometer*, measuring the standard temperature, should rest on the top of the orifice to conform with conditions in accordance with Test Method D 1238 ($1/2$ in. above the shorter orifice).

7. Procedure

7.1 Check that the temperature is controlled to $\pm 0.1^\circ\text{C}$ in the range from 174.9 to 175.1°C .

⁵ The "Thermo Watch" unit, manufactured by Instruments for Research and Industry, 103 Franklin Ave., Cheltenham, PA 19012, has been found satisfactory for this purpose.