

Designation: D2538 - 18

Standard Practice for Fusion of Poly(Vinyl Chloride) (PVC) Compounds Using a Torque Rheometer¹

This standard is issued under the fixed designation D2538; 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 practice covers the relative fusion characteristics of poly(vinyl chloride) compounds.
 - 1.2 The test procedures appear in the following order:

Section

Fusion Test	9
Thermal Stability Test	10
Color-Hold Stability Test	11
Shear Stability Test	12

- 1.3 The values stated in SI units are to be regarded as the standard.
- 1.4 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use. Specific hazards statements are given in Section 8.

Note 1—There are no ISO standards covering the primary subject matter of this ASTM standard.

1.5 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 ASTM Standards:²

D883 Terminology Relating to Plastics

D1600 Terminology for Abbreviated Terms Relating to Plastics

E691 Practice for Conducting an Interlaboratory Study to

Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions*—Definitions are in accordance with Terminologies D883 and D1600 unless otherwise indicated.

4. Summary of Practice

- 4.1 A sample of powder-mix compound is added to the heated roller mixer chamber and is transformed into a fused mass.
- 4.2 The resulting torque curve can be used to determine the relative fusion time and fusion characteristics.

5. Significance and Use

- 5.1 When PVC compounds are mixed under appropriate conditions of heat and shear, a fused mass is produced. This mass has certain melt characteristics which can be defined with a torque rheometer operated under fixed conditions of shear and temperature. The fusion characteristics of a PVC compound are manifest as fusion time, fusion torque, melt torque, melt viscosity, and heat and color stability.
- 5.2 A control lot is to be used as a standard against which other test results are to be compared. Test data are to be evaluated relative to the control lot.

6. Apparatus

6.1 *Microprocessor Torque Rheometer*, ³ equipped with a high-shear mixer with roller-style blades, bowl-jacket thermocouple, stock thermocouple, and temperature recorder.

Note 2—A torque rheometer without microprocessor capability can be used to perform the fusion, thermal stability, and color hold tests.

6.1.1 For flexible and rigid compounds, use a Type 6 roller head with a rotor ratio of 3 Drive: 2 Driven.

Note 3—A Type 5 roller head can also be used, but the data generated cannot be compared with the Type 6 data.

6.2 Quick-Loading Powder Chute or equivalent.

¹ This practice is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials.

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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.

³ Suitable equipment may be obtained from C. W. Brabender, 50 E. Wesley St., South Hackensack, NJ 07606, and Haake Buchler Instruments, 244 Saddle River Rd., Saddle Brook, NJ 07662.

- 6.3 Brass Knife.
- 6.4 Brass Wool or Brush.
- 6.5 Insulated Gloves.
- 6.6 *Balance*, 500-g minimum capacity, with a 0.1-g sensitivity.
 - 6.7 Beaker, stainless steel, 400 mL.
 - 6.8 Oven.
 - 6.9 Aluminum Foil.
 - 6.10 Timer.
 - 6.11 Long-Nose Pliers.
 - 6.12 Hand-Press Mold.

7. Materials

- 7.1 Poly(Vinyl Chloride) Resin.
- 7.2 Filter.
- 7.3 Lubricants.
- 7.4 Plasticizer.
- 7.5 Process-Aid.
- 7.6 Impact Modifier.
- 7.7 Stabilizer.
- 7.8 Pigments.

8. Hazards

- 8.1 Do not exceed the power capacity of the instrument, as damage to the mixer or to the torque rheometer may result.
- 8.2 Do not attempt to clean or poke objects into the mixer while it is running.
- 8.3 Gloves with sufficient insulation to enable the operator to handle the hot equipment should be worn when conducting these tests.

9. Fusion Test Method

- 9.1 Compound Preparation:
- 9.1.1 The compound may be beaker-mixed, blended in an intensive mixer or a ribbon blender, or blended and pelletized. If the compound is beaker-mixed, the total weight of the compound should equal the amount charged to the roller-head bowl.
 - 9.2 Equipment Preparation:
 - 9.2.1 Mount the roller head on the torque rheometer.
- 9.2.2 Select a temperature/rotor speed combination that will permit the test to be completed within a reasonable time constraint. Suggested combinations for several types of PVC compound are found in Appendix X2.
- 9.2.3 With the mixer empty and running, zero the recording pen on the chart.
 - 9.3 Procedure:
- 9.3.1 Determine the sample size to be added to the mixer, using the following formula:

sample size =
$$[(V - D) \times 65\%] \times \text{specific gravity}$$
 (1)

where:

V = volume of mixer bowl without rotors, and

D = volume displacement or rotors.

Note 4—The correct sample size for the mixer is when the fusion curve will duplicate itself. As the mixer wears, it will be necessary to increase the sample size to reproduce a fusion curve equivalent to previous curves.

- 9.3.2 Weigh a sample of the test compound in accordance with 9.3.1. With the mixer running, position the quick-loading chute on the roller-head mixer and pour in the sample compound. Place the ram into the chute and add the weight. When the torque curve indicates maximum torque has been reached, remove the loading chute and weight.
- 9.3.3 Continue mixing until the melt torque achieves a steady state.
- 9.3.4 Stop the mixer and open the bowl. Clean the compound from the bowl and blades using the brass knife or wool, or both.
- 9.3.5 Reassemble the mixing bowl and repeat 9.3.2 9.3.4 for additional tests. Since some cooling takes place when cleaning the bowl, allow sufficient time to confirm that the mixing bowl has reached equilibrium at the test temperature before using again.

Note 5—The quick-loading chute should be at the same temperature at the start of each test. Heat or cool as required.

- 9.4 Interpretation of Torque Rheometer Fusion Test Curve (Fig. 1):
- 9.4.1 *A—Loading Peak*—The point of maximum torque of the unfused, cold raw material.
- 9.4.2 *B—Minimum*—The time from the point of loading to the point of minimum torque.
- 9.4.3 *FS—Inflection Point*—The change in the slope of the curve after the minimum. It will help to characterize the speed of the particle rearrangement (see fusion speed).
- 9.4.4 *X—Maximum—Fusion Torque*—The point where the fused material has its maximum torque.
- 9.4.5 *E—End Point*—User-defined endpoint where the torque curve is stable and steady.
- 9.4.6 *Melt Torque*—The fusion where the torque curve is relatively flat (not displayed in the graph).
- 9.4.7 A...X—Fusion Time—Time between the loading peak A and the Maximum (fusion Torque) X.
- 9.4.8 FS ± 20 %—Fusion Speed—The speed of the progressing fusion of the particles.
 - 9.5 *Report*—Report the following information:
 - 9.5.1 The fusion torque to the nearest 100 m·g.
- 9.5.1.1 Report to the nearest 25 m·g when using a 0 to 1000 scale.
 - 9.5.2 The fusion time to the nearest $\frac{1}{2}$ min.
 - 9.5.3 The melt torque to the nearest 100 m·g.
- 9.5.3.1 Report to the nearest 25 m⋅g when using a 0 to 1000 scale.

Note 6—If the melt torque is not steady, approximate the value and note whether the torque is increasing or decreasing.

9.5.4 Temperature of test, rotor revolutions per minute, and sample size used.

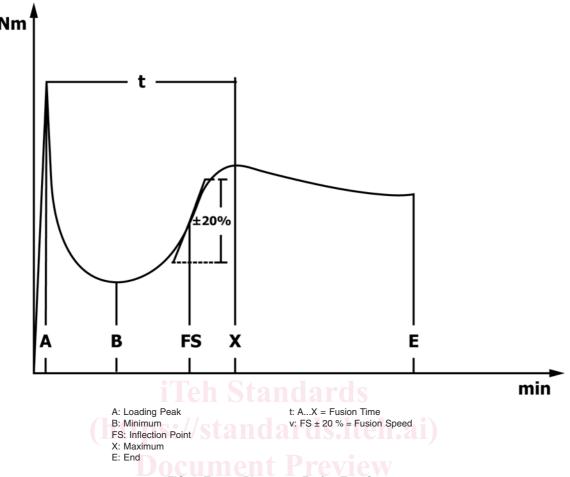


FIG. 1 Torque Rheometer Fusion Test Curve

10. Thermal Stability Test Method

- 10.1 Prepare the test compound in accordance with 9.1.
- 10.2 Prepare the test equipment in accordance with 9.2.
- 10.3 Procedure:
- 10.3.1 Weigh a sample of the test compound in accordance with 9.3.1. With the mixer running, position the quick-loading chute on the roller-head mixer and pour in the sample compound. Place the ram into the chute and add the weight. When the torque curve indicates fusion, remove the loading chute and weight.
- 10.3.2 Continue running until there is a sudden rise in the torque curve, indicating decomposition of the PVC compound.
- 10.3.3 Stop the mixer and open the bowl. Clean the compound from the bowl and blades using the brass knife or wool, or both.
- 10.3.4 Reassemble the mixing bowl and repeat 10.3.1 10.3.3 for additional tests. Confirm that the bowl has reached the test temperature before starting.
 - 10.4 Interpretation of Torque Rheometer Curve (Fig. 1):
- 10.4.1 *Compound Heat Stability*—The time from the point of maximum torque (fusion torque) to the point of sudden torque increase.
 - 10.5 Report—Report the following information:

- 10.5.1 The compound heat stability to the nearest ½ min, and
- 10.5.2 The temperature of test, rotor revolutions per minute, and the sample size used.

11. Color-Hold Stability Test Method

- 11.1 Prepare the test compound in accordance with 9.1.
- 11.2 Prepare the test equipment in accordance with 9.2.
- 11.3 Procedure:
- 11.3.1 Weigh a sample of the test compound in accordance with 9.3.1. With the mixer running, position the quick-loading chute on the roller-head mixer and pour in the sample compound. Place the ram into the chute and add the weight. When the torque curve indicates fusion, remove the loading chute and the weight.
- 11.3.2 Using the point of maximum torque (fusion torque) as zero time, remove a sample of the compound at regular time intervals of 2, 3, or 5 min. Stop the rotors and use a pair of long-nose pliers to remove a sample from the bowl. Restart the rotors. Place the compound into a hand press to shape the sample. Trim the sample and return the excess to the mixing bowl
 - 11.3.3 Mount the samples in a sequential time order.