Designation: D 2538 – 02

# Standard Practice for Fusion of Poly(Vinyl Chloride) (PVC) Compounds Using a Torque Rheometer[1]

This standard is issued under the fixed designation D 2538; 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 and health 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.

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 883 Terminology Relating to Plastics[2]

D 1600 Terminology for Abbreviated Terms Relating to Plastics[2]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

## 3. Terminology

3.1 *Definitions*—Definitions are in accordance with Terminologies D 883 and D 1600 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,*[4] 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.

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*.

[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] *Annual Book of ASTM Standards*, Vol 08.01.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] 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.

## 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:

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

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 Curve (Fig. 1)*:

9.4.1 *Fusion Torque*—The point of maximum torque.

9.4.2 *Fusion Time*—The time from the point of loading to the point of maximum torque.

9.4.3 *Melt Torque*—The fusion where the torque curve is relatively flat.

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 ½ 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.

## 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.

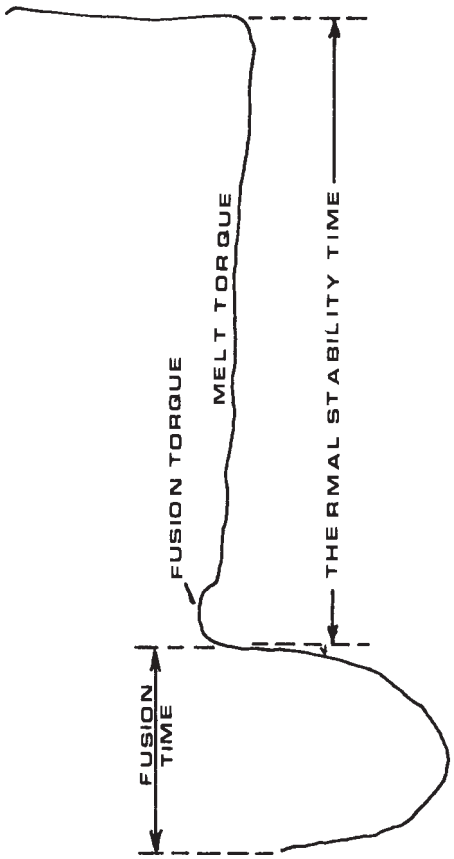



**FIG. 1 Torque Rheometer Curve**

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.

11.3.4 Continue sampling until the desired color change has been observed.

NOTE 7—Depleting the sample from the bowl can affect the amount of working the compound receives. Select a time sequence that does not remove more than half the sample.

11.3.5 Stop the mixer and open the bowl. Clean the compound from the bowl and blades, using the brass knife or wool, or both.

11.3.6 Reassemble the mixing bowl and repeat 11.3.1-11.3.5 for additional tests. Confirm that the bowl has reached the test temperature before starting.

11.4 *Report*—Report the following information:

11.4.1 The time to equivalent color change and

11.4.2 The temperature of test, rotor revolutions per minute, and the sample size used.

11.5 *Alternative Procedure*:

11.5.1 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.

11.5.2 Using the point of maximum torque (fusion torque) as zero time, run the mixer until the compound starts to discolor. Stop the mixer, remove the contents, and press into a plaque.

11.5.3 Clean the bowl and blades and reassemble the mixer.

11.5.4 Run additional tests, stopping at the same point in time as 11.5.2.

11.5.5 Measure the color of the plaque in a Hunter Colorimeter (or equivalent).

11.6 *Report*—Report the following information:

11.6.1 The color number measured on the pressed plaque and

11.6.2 The temperature of the test, rotor revolutions per minute, the sample size, and the time as determined in 11.5.2.

## 12. Shear Stability Test Method

12.1 Prepare the test compound in accordance with 9.1.

12.2 Prepare the test equipment in accordance with 9.2.

NOTE 8—Use the microprocessor torque rheometer for this test.

12.3 *Procedure*:

12.3.1 Set the microprocessor for the desired temperature and program the revolutions per minute as follows:

12.3.1.1 Run at preselected revolutions per minute for ten min.

12.3.1.2 Return to 0 r/min.

12.3.1.3 Program the speed control to run from 0 to 100 r/min in 1 min and stop.

12.3.2 When the bowl temperature has stabilized, weigh out a sample of the test compound. Position the quick-loading chute on the roller-head mixer, turn on the torque rheometer, and pour in the sample compound. Place the ram into the chute and add the weight. When the ram bottoms, remove the loading chute.

12.3.3 When the sample run is finished, use the microprocessor to plot the torque versus revolutions per minute. Use the plotted curve for analysis.

12.3.4 When the mixer has stopped, open the bowl. Clean the compound from the bowl and blades, using the brass knife or wool, or both.

12.3.5 Reassemble the mixing bowl and repeat 12.3.1-12.3.5 for additional tests.

## 13. Interpretation of Torque Rheometer Results

13.1 *Visual Analysis*:

13.1.1 The first 10 min of the graph represents a standard fusion curve which can be used to determine the fusion time, fusion torque and melt, or stabilized torque.

13.1.2 The torque versus revolutions per minute plot can be used as a relative comparison of shear sensitivity.

13.1.3 Bearing in mind that a straight inclined line would denote a Newtonian material, the shear sensitivity can be

estimated by the degree of arc. A large arc or loop denotes more shear-sensitive material than does a smaller arc or loop. A visual observation can be made to determine the relative shear sensitivity between sample materials.

13.2 *Technical Analysis*:

13.2.1 The torque versus revolutions per minute plotted graph can be used in conjunction with the microprocessor software to provide a shear-sensitivity index number. The software is programmed to give such a number. A higher index number denotes a more shear-sensitive material.

13.2.2 The calculation programmed into the software is shown in the Appendix X1.

## 14. Precision and Bias

14.1 Table 1 is based on a round robin[5] conducted in 2001 in accordance with Practice E 691, involving five rigid PVC dry blend compounds tested by ten laboratories. All laboratories used a computerized torque rheometer and a single zone electrically heated mixer/measuring head (3:2 gear ratio) to process the samples. For each material, all the samples were prepared at one source, but the individual specimens were prepared at the laboratories that tested them. Each sample was tested three times on two separate days yielding six points per data value.

NOTE 9—**Caution:** The explanation of *r* and *R* (14.2-14.2.3) is only intended to present a meaningful way of considering the approximate precision of this test method. The data in Table 1 should not be applied to acceptance or rejection of materials, as these data apply only to the materials tested in the round robin and are unlikely to be rigorously representative of other lots, formulations, conditions, materials, or laboratories. Users of this method should apply the principles outlined in Practice E 691 to generate data specific to their materials and laboratory (or between specific laboratories). The principles of 14.2-14.2.3 would then be valid for such data.

[5] Supporting data giving results of the round robin have been filed at ASTM International Headquarters. Request RR: D20–XXXX.

**TABLE 1 Round Robin Test Results**

Parameter 1– Fusion Torque, Point X (Newton-meters), Mean Level in Ascending Order

| | | Within Laboratories | | | Between Laboratories | | |
|---|---|---|---|---|---|---|---|
| Material | Mean Level | $S_r$ | r | (r) | $S_R$ | R | (R) |
| Material C | 29.5 | 0.58 | 1.62 | 5.5 % | 2.40 | 6.72 | 22.8 % |
| Material B | 30.3 | 0.78 | 2.18 | 7.2 % | 2.47 | 6.92 | 22.8 % |
| Material E | 31.4 | 0.39 | 1.08 | 3.4 % | 2.00 | 5.59 | 17.8 % |
| Material A | 35.2 | 1.15 | 3.21 | 9.1 % | 2.57 | 7.19 | 20.4 % |
| Material D | 39.1 | 1.05 | 2.94 | 7.5 % | 3.20 | 8.97 | 23.0 % |
| Pooled Values | 33.1 | 0.79 | 2.21 | 6.6 % | 2.53 | 7.08 | 21.4 % |

Parameter 2– Fusion Torque Temperature, (degrees Celsius), Mean Level in Ascending Order

| | | Within Laboratories | | | Between Laboratories | | |
|---|---|---|---|---|---|---|---|
| Material | Mean Level | $S_r$ | r | (r) | $S_R$ | R | (R) |
| Material B | 171.0 | 0.97 | 2.71 | 1.6 % | 1.52 | 4.25 | 2.5 % |
| Material D | 174.0 | 1.62 | 4.53 | 2.6 % | 3.72 | 10.41 | 6.0 % |
| Material E | 181.6 | 1.38 | 3.85 | 2.1 % | 4.97 | 13.92 | 7.7 % |
| Material A | 183.2 | 2.09 | 5.86 | 3.2 % | 4.73 | 13.24 | 7.2 % |
| Material C | 188.6 | 0.82 | 2.30 | 1.2 % | 2.23 | 6.25 | 3.3 % |
| Pooled Values | 179.7 | 1.38 | 3.85 | 2.1 % | 3.43 | 9.61 | 5.3 % |

Parameter 3– End Torque, Point E (Newton-meters), Mean Level in Ascending Order

| | | Within Laboratories | | | Between Laboratories | | |
|---|---|---|---|---|---|---|---|
| Material | Mean Level | $S_r$ | r | (r) | $S_R$ | R | (R) |
| Material B | 17.9 | 0.30 | 0.85 | 4.8 % | 0.79 | 2.21 | 12.3 % |
| Material E | 22.5 | 0.26 | 0.73 | 3.3 % | 0.90 | 2.53 | 11.3 % |
| Material C | 23.6 | 0.48 | 1.33 | 5.6 % | 0.91 | 2.55 | 10.8 % |
| Material A | 25.5 | 0.35 | 0.98 | 3.8 % | 0.95 | 2.65 | 10.4 % |
| Material D | 27.9 | 0.64 | 1.78 | 6.4 % | 1.06 | 2.98 | 10.7 % |
| Pooled Values | 23.5 | 0.41 | 1.14 | 4.8 % | 0.92 | 2.58 | 11.1 % |

Parameter 4– Fusion Time (s), Time at Fusion Minus Time at Load Torque, Mean Level in Ascending Order

| | | Within Laboratories | | | Between Laboratories | | |
|---|---|---|---|---|---|---|---|
| Material | Mean Level | $S_r$ | r | (r) | $S_R$ | R | (R) |
| Material D | 40.7 | 4.78 | 13.39 | 32.9 % | 7.45 | 20.86 | 51.2 % |
| Material B | 47.3 | 2.65 | 7.41 | 15.7 % | 9.58 | 26.82 | 56.7 % |
| Material E | 56.8 | 3.91 | 10.94 | 19.2 % | 13.21 | 36.99 | 65.1 % |
| Material A | 70.6 | 7.61 | 21.31 | 30.2 % | 13.36 | 37.40 | 52.9 % |
| Material C | 85.6 | 8.15 | 22.83 | 26.7 % | 20.38 | 57.05 | 66.6 % |
| Pooled Values | 60.2 | 5.42 | 15.17 | 24.9 % | 12.79 | 35.82 | 58.5 % |

NOTE 10—Symbols are defined as follows:
$S_r$= within laboratory standard deviation
r = within laboratory repeatability, measurement units
(r) = within laboratory repeatability, %
$S_R$= between laboratory standard deviation
R = between laboratory reproducibility, measurement units
(R) = between laboratory reproducibility, %

14.2 *Concept of r and R in Table 1*—If $S_r$ and $S_R$ have been calculated from a large enough body of data, and for test results that were averages from testing one point for each test result, then:

14.2.1 *Repeatability (r)*—Two results obtained within one laboratory shall be judged not equivalent if they differ by more than the r-value, where $r = 2.8S_r$. *r* is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

14.2.2 *Reproducibility (R)*—Two results obtained between two laboratories shall be judged not equivalent if they differ by more than the R-value, where $R = 2.8S_R$. *R* is the interval representing the critical difference between two test results for the same material, obtained by a different operators using different equipment on different days.

14.2.3 Any judgment in accordance with 14.2.1 or 14.2.2 would have an approximate 95 % (0.95) probability of being correct.

## 15. Keywords

15.1 color stability; fusion of PVC compound; heat stability; PVC compounds; rheometry

## APPENDIXES

**(Nonmandatory Information)**

### X1. CALCULATION OF SHEAR SENSITIVITY

X1.1 The torque versus revolutions per minute curve at test temperature can be obtained by using speed programming. Use the first half of the torque versus revolutions per minute curve (0 to 50 r/min) in order to avoid melt temperature rise due to frictional heat (Fig. X1.1).

X1.2 Draw a reference line from the origin to the intercept between the reference revolutions per minute and the torque curve at 50 r/min as shown in Fig. X1.2.

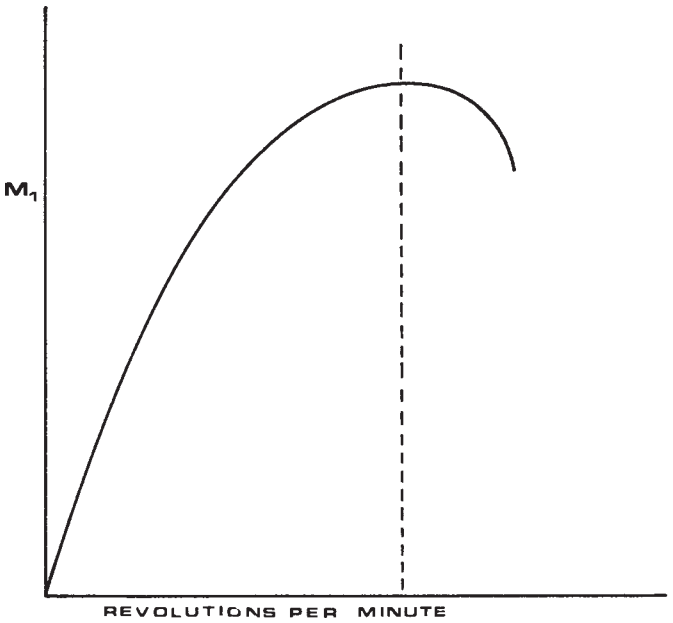



**FIG. X1.1 Torque Versus Revolutions Per Minute Curve**

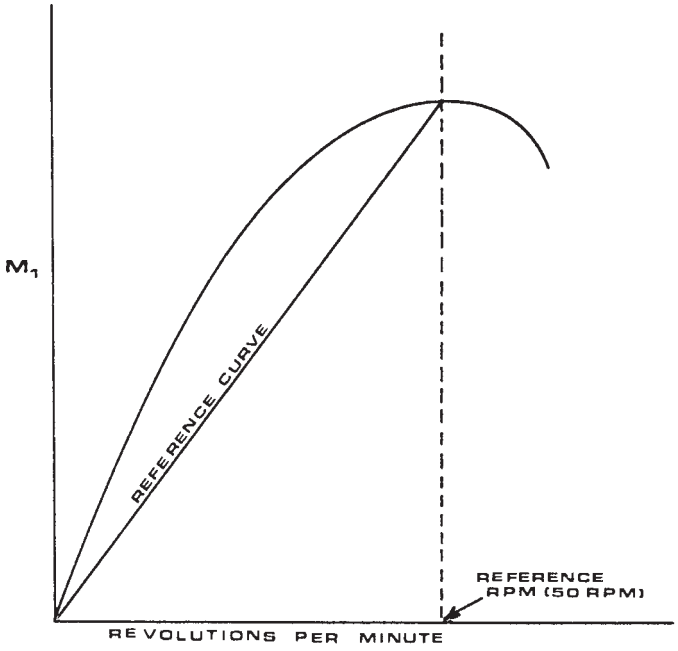


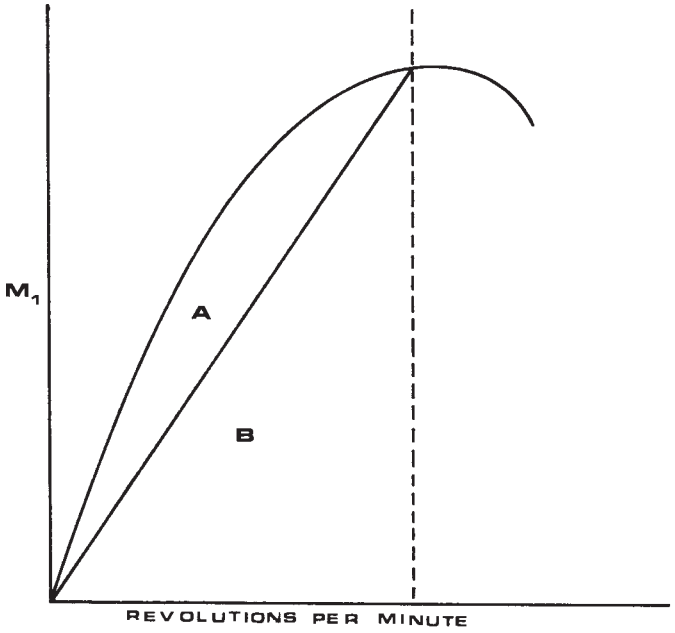

**FIG. X1.2 Reference Curve**

X1.3 *Calculation*—Calculate the shear sensitivity of pseudoplastics, *S*, as shown in Fig. X1.3.

X1.4 *Data Interpretation* (see Fig. X1.3):

X1.4.1 If the shear-sensitivity index approaches one (powerlaw index *n* approaches zero), it is more shear sensitive.

X1.4.2 If the shear-sensitivity approaches zero (*n* approaches one), it is less shear sensitive.




where:
*A* = area of arc,
*B* = area of triangle, and
*M* = torque (m·g).

**FIG. X1.3 Shear Sensitivity of Pseudoplastic Materials**

## X2. SUGGESTED TEMPERATURE/ROTOR SPEED COMBINATIONS FOR PVC COMPOUNDS

X2.1 Suggested temperature/rotor speed combinations for PVC compounds are as follows:

| Compound Type | Suggested Set Temperature/Rotor Speed |
|---|---|
| Flexible Compound | 140°C (284°F) @ 31 r/min |
| Semirigid Compound | 180°C (356°F) @ 50 r/min |
| Rigid Compound | 197°C (387°F) @ 60 r/min |