



# Standard Test Method for Thermal Flow, Cure, and Behavior Properties of Pourable Thermosetting Materials by Torque Rheometer<sup>1</sup>

This standard is issued under the fixed designation D 3795; 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 covers the apparatus, and a specific test method, including the evaluation of results required for the determination of the thermal flow and cure behavior properties of pourable thermosetting materials.

1.2 This test method can be used:

1.2.1 As a control for the development and production of pourable thermosetting materials and to measure the different properties (for example, melting behavior, cure behavior, etc.) as well as the influence of various additives and fillers in any given formulations, and

1.2.2 Verify the uniformity of different production batches of the same formulation.

1.3 The values are stated in SI units.

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 precautions are given in Section 7.

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

## 2. Referenced Documents

### 2.1 ASTM Standards:

D 792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement<sup>2</sup>

D 883 Terminology Relating to Plastics<sup>2</sup>

D 1898 Practice for Sampling of Plastics<sup>3</sup>

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>4</sup>

<sup>1</sup> 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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<sup>2</sup> Annual Book of ASTM Standards, Vol 08.01.

<sup>3</sup> Discontinued; see 1997 Annual Book of ASTM Standards, Vol 08.02.

<sup>4</sup> Annual Book of ASTM Standards, Vol 14.02

## 3. Terminology

3.1 *Definitions of Terms Specific to This Standard*—For the purpose of this test method, the following definitions apply, (see Fig. 1):

3.1.1 *flow and cure behavior*—the flow behavior is represented by the recorded torque curve from the loading peak (Point  $t_1$ ), to the torque minimum (Point  $t_5$ ). The cure behavior is represented by the recorded torque curve from the torque minimum (Point  $t_5$ ) to the torque maximum (Point  $t_4$ ). The rate of curing is represented by the slope of the torque curve.

3.1.2 *time*—the residence time at torque  $t_5 \times X$ , where  $X$  is a factor (preferably 1.3) is  $t_v$  (s). To determine  $t_v$ , draw a line at  $t_5 \times X$  parallel with the time axis. The intersection of this line with the left branch of the curve is  $t_2$ . The intersection of this line with the right branch of the curve is  $t_3$ :

$$t_v = t_3 - t_2 \text{ units are seconds (s)} \quad (1)$$

3.1.2.1 *Discussion*—Depending on the manufacturer of the equipment, the software analysis program for the designated values in this test method may differ ( $t_1$ ,  $t_2$ , etc....).

3.1.2.2 *Discussion*—Upon agreement between interested parties, the value of  $X$  may be changed and be listed in any report.

3.1.3 *residence time or duration of plastic life* ( $t_2 - t_3$ )—the residence time is represented by a section of the recorded torque curve in which the molten material causes the lowest torque, s.

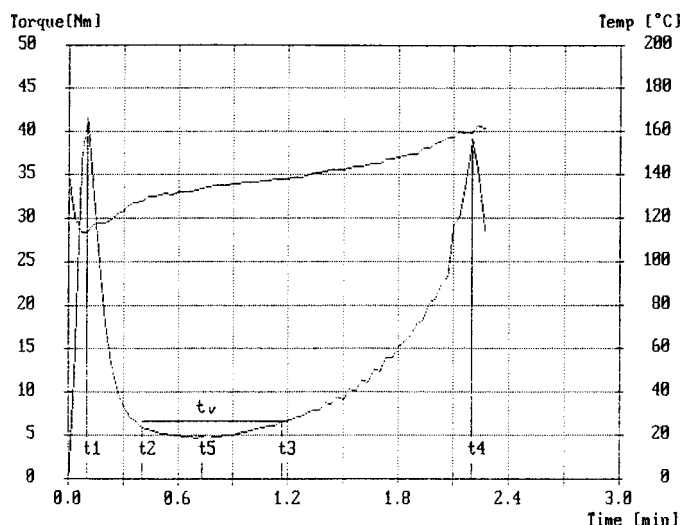
3.1.4 *total cure time* ( $t_4 - t_0$ )—time from when the material is loaded into the mixer chamber up to complete cure, s.

3.1.5 *torque*:

3.1.5.1 *initial torque* ( $t_1$ )—the initial high torque peak once material is loaded into the mixer chamber. Sometimes referred to as the loading peak, Nm (Newton-meters).

3.1.5.2 *minimum torque* ( $t_5$ )—the lowest point on the torque curve representing maximum fluxing of material, Nm.

3.1.5.3 *final torque or cure peak* ( $t_4$ )—the final maximum torque value representing the final cure of material, Nm.



NOTE 1—Top curve associated with temperature axis; bottom curve associated with torque axis.

FIG. 1 Torque Rheometer Curve

#### 4. Summary of Test Method

4.1 A sample of thermosetting material is charged into the temperature controlled mixer/measuring head in which the material is compacted, melted, cross-linked, hardened, and crushed under constant shear.

#### 5. Significance and Use

5.1 The continuous recording of torque and temperature while going through these various stages can be used to predict the behavior of the material during processing.

5.2 The torque rheometer test has two important functions. First, it is a means to predict flow/viscosity and cure characteristics of pourable thermosetting compounds. For example, the test provides useful data to predict the processability of a material in a particular molding method. This information is also useful to optimize process conditions for a particular material such as the minimum pressure to fill a mold and the time to cure a part. A second capability of the test is to provide a graphic record of the batch-to-batch uniformity of the molding compound.

#### 6. Apparatus

6.1 *Torque Rheometer*, with a mixing bowl.

6.1.1 The torque rheometer shall be equipped with a drive motor with a load-independent speed stability of  $\pm 0.5\%$  of the top rotor speed.

6.1.2 The recording device selected shall be capable of recording the measurable variables of torque, stock temperature, and rotation per minute (RPM) as a function of time. The rheometer should also be equipped with a real time RPM indicator.

6.1.3 For the measurement, a surface hardened laboratory internal mixer is used, specified by a bowl volume of 25, 30 or 60 cm<sup>3</sup>, that can be attached to the above mentioned torque rheometer. Either a set of triangular or roller blades shall be used counter-rotating with a speed ratio of 3:2 (left to right). (The mixer bowl may be heated with a circulated liquid

temperature controlled by a thermostat or electrically with at least two heating zones (see Note 2).

NOTE 2—Only the results obtained with identical measuring systems can be compared with one another. In this context, the mixer type, type of heating/cooling and loading weight used are of decisive importance.

6.1.4 Liquid heated mixers shall be equipped with a circulation pump that has a capacity of at least 24 L/min at a back pressure of approximately 500 mbars. The heat transfer medium shall be stabilized silicone oil, with a maximum viscosity of 20 mm<sup>2</sup>/s at 25°C. The oil temperature shall be monitored by a device which has a resolution of 0.2°C or better.

6.1.5 Electrically heated mixer bowls shall have a minimum of one independent electric controller and a maximum of two independent electric controllers that utilize modern control techniques and algorithms. These controllers shall provide both heating and cooling cycles. The temperature control ensembles that include the sensor, controller and actuators shall be accurate to within 2.0°C throughout their working range. Reported values shall have a precision of 0.2°C or better.

6.1.6 The torque recording ensemble shall be accurate to 0.25 % of the reading.

6.1.7 For feeding flowable or granular sample materials a loading device shall be used. For feeding other coarse materials, a pressure ram actuated manually or pneumatically shall be used. The loading chute has to be mounted onto the mixer, with a ram and either a 5 or 2 kg weight or with an adjustable pneumatic cylinder (see Note 2).

6.1.8 For recording of the stock temperature during the measuring process, the temperature measuring device is mounted from below into the bottom of the measuring mixer in such a way that it penetrates 1.5 mm into the mixer bowl. The stock temperature versus time, is recorded simultaneously together with the torque curve.

6.1.9 *Soft Brass Spatula or Stiff Brass Bristle Cleaning Tool.*

#### 7. Hazards

7.1 Do not exceed the rated power of the instrument as damage to the mixer or to the torque rheometer may result.

7.2 Do not attempt to clean or insert objects into the mixer while it is running.

7.3 Use adequate exhausts and safety devices necessary to meet applicable safety codes.

7.4 Use insulated gloves to protect operator from hot mixer surface.

7.5 Refer to manufacturers' operating instructions.

#### 8. Sampling

8.1 A batch of compound shall be considered as a unit of manufacture as prepared for shipment and may consist of a manufacturer's blend of one or more production runs of material.

8.2 Suitable methods of sampling shall follow Practice D 1898. A 400-g sample will be sufficient for tests required.

8.3 Crush any compound in a preform state to a particle size that would pass through the loading chute.

## 9. Sample Selection, Handling and Use for Rheometer Standardization

9.1 *Selection*—The selection of the sample should be determined by the use for which it is intended. If it is to be an intralaboratory standardization sample, (for example in a material compounder's laboratory), the sample should be chosen to closely approximate the materials expected to be tested. (For compounders having a wide range of product plasticities it is recommended that one sample for each maximum torque range be available.) For interlaboratory standardization, the sample should be of the product type being molded.

### 9.1.1 *Handling*:

9.1.1.1 Once the selection of the sample has been agreed upon among the interested parties it shall be gathered in sufficient quantity that the supply of sample for each laboratory can be expected to outlast the need to change the mixing head or blades by about 50 % of the life of the head or blades. This is to avoid running out of standardization standard at the same time as an equipment change takes place on the rheometer (see Note 3).

NOTE 3—The determination for wear of the measuring mixer and blades shall be measured volumetrically. The equipment manufacturer shall provide the procedure and values for the individual measuring mixers.

9.1.1.2 It is recommended that the sample be broken up into preweighed charges, the charge weight being based on the specific gravity of the sample and the size of the mixing head in use on the respective rheometers. These should be heat-sealed in individual polyethylene pouches. If the samples are of any compound which has a defined shelf-life, they should be stored at, or near freezing in order to protect their plasticity properties from changing. If the samples have indefinite shelf-life, they should be stored below the temperature at which volatile material could be driven off due to excessive vapor pressure.

9.1.1.3 Samples stored in this manner should be allowed a full 24-h to reach equilibrium temperature with standard laboratory conditions of  $23 \pm 2^\circ\text{C}$  before removing from storage pouches for standardization testing.

9.1.2 Samples should be used to test the standardization of the respective rheometers at agreed upon intervals of operation. It is recommended that the standardization be tested at least every 120 h of operation. Operating time is defined not as the total testing time; but rather as the total elapsed time that the rheometer is powered up. This would be once per week for a full time, five day per week laboratory operation. The samples should be used two at a time, where the first sample tested will be used to condition the instrument mixing head and blades, and the second sample will be used as actual standardization data.

9.1.2.1 It is recommended that sample pouches be drawn from storage five at a time for conditioning to ambient conditions. The first will be used for rheometer conditioning, the second for standardization testing, and the remaining be used for additional standardization testing in the event that the instrument needs to be adjusted, or restandardized. If the extra

three samples are not needed they may be safely returned to storage if they have not been opened.

9.2 *Calibration*—To ensure reliability of this test method, it is essential that the torque rheometer be calibrated periodically using a reference material. A control chart indicating the results of these calibrations should be kept. Entire equipment calibrations shall be done when data from a reference material calibration versus the control chart, causes the instrumentation to be suspect.

9.2.1 *Equipment Calibration*—The torque, temperature control, and RPM systems shall be calibrated using national or international regulatory body traceable standards and procedures.

9.2.2 Record all “as found” measured values versus accepted values before making any attempts at corrective action. All “as left” measured values should be recorded upon completing any adjustments.

9.2.3 Upon completion of the calibration, provide a Certificate of Calibration. This document shall include the data values, traceability of each standard used, and a statistical estimation of the uncertainties associated with each procedure and standard versus national or international regulatory body standards.

## 10. Procedure

10.1 Select a mixer temperature that corresponds to the mean processing temperature of the material to be tested:

Material Type (granular)	Suggested Temperature, °C
Alkyd	150
Crosslineable polyethylene	145, 175
DAP	150
Epoxy	150, 175
Phenolic	125, 150
Polyester	150
Silicone	140
Silicone-epoxy	140

10.2 Adjust the required mixer temperature at the bath and the circulation thermostat or at the temperature controller for the electrically heated mixer. Condition the measuring mixer at this temperature until the mixer has reached equilibrium.

10.3 Start the torque rheometer before starting the first test and obtain a uniform temperature in the system. Make sure that the mixing blades are rotating during this time.

10.4 Weigh the test charge with a precision of  $\pm 0.1$  g and to an accuracy of 0.5 % of the total sample mass for each measurement. Depending on the density of the material, the optimum charge for the measuring mixer may vary. Eq 2 is a reference for determining a good load charge:

$$G = 0.7 \times V \times P \quad (2)$$

where:

$G$  = the sample mass, in kilograms,  
 $V$  = the free mixer volume, in liters,  
 $P$  = the density of sample material, in kilograms per liter, and (density in accordance with Test Method D 792).

The density of the sample material shall be given with an accuracy of  $\pm 0.03$  kg/L.

10.5 Start the drive of the torque rheometer and adjust the rotor speed to 40 RPM. Charge the running mixer with the

sample quickly and as uniformly as possible using the loading chute and the pressure ram, respectively. Loading shall be completed within 20 s. As soon as all material has been loaded into the mixer, place the 5 or 2 kg weight onto the ram of the loading chute in order to close the mixer. If a pneumatic loading device is used, the ram shall be kept under pressure.

10.6 Immediately after the measured torque curve has passed through the initial torque  $t_1$ , (see Fig. 1), remove the loading chute or pressure ram, or lift the pneumatic piston with the ram and then immediately close weighted ram pressure arm.

10.7 At the end of the test, clean the rotors and inside of the mixing chamber. Exercise care not to scratch the blades or bowl. Reassemble the chamber and preheat as required for the next test. The loading chute should be cooled before each test to prevent the sample from sticking in the chute.

## 11. Report

11.1 Include the following in the test report:

11.1.1 Type or designation of the test material, or both,

11.1.2 Date of test,

11.1.3 Sample mass, kilograms,

11.1.4 Mixer type (including type of heating used),

11.1.5 Mixer blade type,

11.1.6 Load chute type and size of weight used,

11.1.7 Selected mixer temperature, °C,

11.1.8 Rotor speed (RPM), and

11.1.9 Stock temperature at start of test, °C.

11.2 Also include the following data values in the test report:

11.2.1 Minimum torque  $t_5$ , Nm,

11.2.2 Time to load the mixer, s,

11.2.3 Initial torque ( $t_1$ ), Nm,

11.2.4 Residence time ( $t_1 = t_3 - t_2$ ), s,

11.2.5 Final torque or cure peak  $t_4$ , Nm, and

11.2.6 Total time ( $t_1 - t_0$ ), s.

## 12. Precision and Bias

12.1 *Precision*—Table 1 is based on a round robin conducted in 1999 in accordance with Practice E 691, involving 5 thermoset materials tested by 4 laboratories. All laboratories used a computerized torque rheometer and an electrically heated half-size mixer/measuring head 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. Materials A, C, and D were standard phenolic compounds. Material B was a two-stage cure phenolic compound. Material E was a polyester compound. Each sample was tested 3 times on 2 separate days yielding 6 points per data value.

NOTE 4—**Caution:** The explanation of  $r$  and  $R$  (12.2-12.2.3) 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 test method should apply the principles in accordance with Practice E 691 to generate data specific to their materials

**TABLE 1 Repeatability Results for Thermoset Samples**

Data Value—Initial Torque, Point A, Nm, See 11.2.3				
Mean Level in Ascending Order		Within Laboratories		
Material	Mean Level	Sr	r	(r)
Material E	15.9	1.83	5.13	32.2 %
Material A	17.8	1.33	3.73	21.0 %
Material D	19.1	1.25	3.51	18.3 %
Material C	25.6	1.10	3.09	12.0 %
Material B	50.4	2.84	7.95	15.8 %
Pooled values	25.8	1.67	4.68	19.9 %
Data Value—Time to Initial Torque, 0-Point A, s, See 11.2.2				
Mean Level in Ascending Order		Within Laboratories		
Material	Mean Level	Sr	r	(r)
Material C	5.7	0.93	2.61	46.0 %
Material D	6.3	0.93	2.61	41.2 %
Material A	6.5	1.00	2.80	43.1 %
Material E	11.8	2.14	5.98	50.9 %
Material B	17.1	2.09	5.85	34.3 %
Pooled values	9.5	1.42	3.97	43.1 %
Data Value—Minimum Torque, Point B, Nm, See 11.2.1				
Mean Level in Ascending Order		Within Laboratories		
Material	Mean Level	Sr	r	(r)
Material E	3.4	0.05	0.15	4.4 %
Material D	3.5	0.07	0.19	5.3 %
Material A	4.7	0.11	0.30	6.3 %
Material C	7.0	0.10	0.29	4.1 %
Material B	11.3	0.43	1.19	10.6 %
Pooled values	6.0	0.15	0.42	6.1 %
Data Value—Total Time, $t_v$ , s, See 11.2.4				
Mean Level in Ascending Order		Within Laboratories		
Material	Mean Level	Sr	r	(r)
Material E	18.7	2.32	6.49	34.7 %
Material C	23.2	0.85	2.37	10.2 %
Material B	26.7	3.96	11.08	41.5 %
Material A	31.5	1.83	5.12	16.3 %
Material D	33.0	1.83	5.13	15.5 %
Pooled values	26.6	2.16	6.04	23.7 %
Data Value—Final Torque, Point X, Nm, See 11.2.5				
Mean Level in Ascending Order		Within Laboratories		
Material	Mean Level	Sr	r	(r)
Material E	13.2	0.80	2.25	17.1 %
Material A	13.7	0.69	1.94	14.2 %
Material C	18.1	0.79	2.20	12.1 %
Material D	19.3	0.75	2.10	10.9 %
Material B	24.6	1.06	2.96	12.0 %
Pooled values	17.8	0.82	2.29	13.3 %
Data Value—Time to Final Torque, Point A-Point X, s, See 11.2.6				
Mean Level in Ascending Order		Within Laboratories		
Material	Mean Level	Sr	r	(r)
Material E	52.7	5.07	14.20	27.0 %
Material C	71.5	3.69	10.33	14.4 %
Material D	84.1	4.51	12.62	15.0 %
Material A	94.2	6.06	16.97	18.0 %
Material B	146.1	11.10	31.09	21.3 %
Pooled values	89.7	6.09	17.04	19.1 %

and laboratory (or between specific laboratories). The principles of 12.2-12.2.3 would then be valid for such data.

NOTE 5—Symbols are defined as follows:

Sr = within laboratory standard deviation,

r = within laboratory repeatability, measurement units, and



(r) = within laboratory repeatability, %.

12.2 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:

12.2.1 *Repeatability (r)*—Two results obtained within one laboratory shall be judged not equivalent if they differ by more than the  $r$  value for that material. The concept of  $r$  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.

12.2.2 *Reproducibility (R)*—The number of laboratories, four, participating in the round robin does not meet the minimum requirement for inclusion of information on between laboratory variability. Because this test method does not

contain a valid round-robin-based numerical precision and bias statement, it shall not be used as a referee test method in case of dispute. Anyone wishing to participate in the development of precision and bias data should contact the Chairman, Subcommittee D20.30 (Section 20.30.08), ASTM, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428–2959.

12.2.3 Any judgment in accordance with 12.2.1 or 12.2.2 would have an approximate 95 % (0.95) probability of being correct.

12.3 *Bias*—There are no recognized standards by which to estimate bias of this test method.

### 13. Keywords

13.1 flow/cure properties; pourable; thermosets; torque rheometer

## SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of this test method. This section may include descriptions of the changes or the reasons for the changes, or both.

*D 3795 – 97:*

(1) This test method has been significantly expanded in this revision. Title, scope, terminology and apparatus sections have been modified. In addition, significant sections have been added on standardization and calibration of equipment. A new round robin study will be required. Most of these changes have

been prepared and recommended by a subgroup in SPI.

*D 3795 – 00:*

(1) Clarifications to 1.1, 3.1.5.1–3.1.5.3, 6.1.3 and 6.1.5.

*D 3795 – 00a:*

(1) Revised the Precision and Bias section.

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