Designation: D3795 – 00a (Reapproved 2012)

Standard Test Method for Thermal Flow, Cure, and Behavior Properties of Pourable Thermosetting Materials by Torque Rheometer[1]

This standard is issued under the fixed designation D3795; 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:*[2]

D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement

D883 Terminology Relating to Plastics

D1898 Practice for Sampling of Plastics (Withdrawn 1998)[3]

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

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 For the purpose of this test method, the following definitions apply, (see Fig. 1):

3.1.2 *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.

3.1.3 *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 :

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

3.1.3.1 *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....).

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

3.1.4 *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.

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

3.1.6 *torque:*

3.1.6.1 *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).

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

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

4. Summary of Test Method

4.1 A sample of thermosetting material is charged into the temperature controlled mixer/measuring head in which the

[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.30.08).

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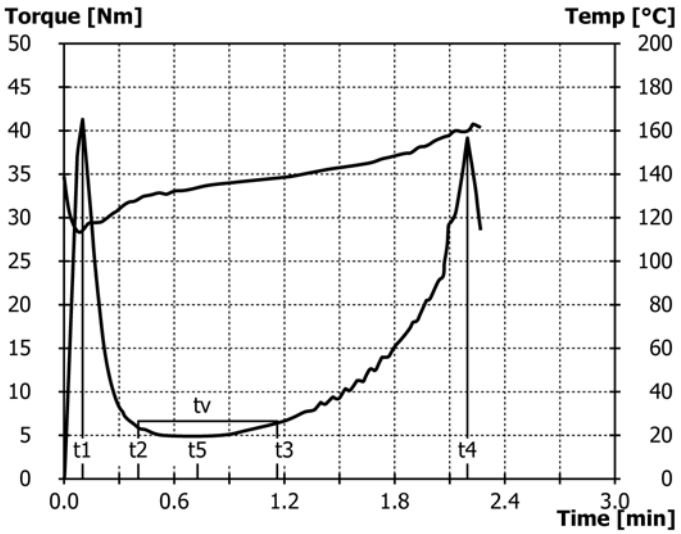
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

*A Summary of Changes section appears at the end of this standard



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

FIG. 1 Torque Rheometer Curve

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 processibility 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 ±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^3, 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^2 /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 D1898. 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