Designation: E3277 – 21

Standard Test Method for Determining Whether a Material is a Liquid or a Solid by Rheometry[1]

This standard is issued under the fixed designation E3277; 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 Using rheometry, this test method determines, for regulatory purposes, whether a viscose viscous material is a liquid or a solid. Very small amounts of material (typical less than 3 g) may be used for this measurement.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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.*

1.4 *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]

D4092 Terminology for Plastics: Dynamic Mechanical Properties

D4473 Test Method for Plastics: Dynamic Mechanical Properties: Cure Behavior

E473 Terminology Relating to Thermal Analysis and Rheology

3. Terminology

3.1 *Definitions*—Technical terms used in this standard are defined in Terminologies D4092 and E473 including *dynamic mechanical analyzer, loss modulus, phase angle, rheometer, shear, storage modulus, strain, stress*, and *tan delta*.

3.1.1 *dissipative, adj—in dynamic mechanical analysis*, the irrecoverably loss of mechanical energy usually as heat.

3.1.2 *gel point, n*—the stage at which a liquid begins to exhibit pseudo-elastic properties.

4. Summary of Test Method

4.1 Viscoelastic materials exhibit both solid-like and liquid-like characteristics. The loss modulus of a material is a measure of its liquid-like characteristic while the storage modulus of a material indicates its solid-like characteristics. When the loss modulus is greater than the storage modulus, a material is said to be predominantly a liquid. When the storage modulus is greater than the loss modulus, the material is said to be predominantly a solid. The ratio of loss modulus to storage modulus is known as tangent angle delta (tan δ).

4.2 Loss modulus and storage modulus of a material is measured using a cone-and-plate or parallel plate rheometer at ambient temperature conditions and tan δ is then calculated. Materials with a tan δ greater than unity (tan δ ≥ 1.0) are identified as liquids. Those materials with a tan δ less than unity are identified as solids.

5. Significance and Use

5.1 Shipping regulations often require the identification of a material as either a liquid or a solid. This test method may be used to make that determination for regulatory purposes.

5.2 For liquid thermosetting resin, as cure progresses, the liquid resin becomes a solid. A thermosetting resin is more easily worked or shaped while in the liquid-like form and becomes more difficult to do so as the cure advances. The point at which the solid-like character becomes dominant is called the gel point and is considered to be the end of the period where the thermosetting resin is workable. Gel point is identified as that point where tan δ = 1 as determined in Test Method D4473.

NOTE 1—Gel point at ambient temperature is seldom a useful parameter. Use of this test method at the more useful elevated temperatures requires capabilities readily available but outside of 7.2.6, 7.2.7, and Section 10.

5.3 This test method may be used in research, development, and for regulatory compliance.

[1] This test method is under the jurisdiction of ASTM Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.08 on Rheology.

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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.

6. Interferences

6.1 Since small quantities of material are used, it is essential that the specimen be representative of the sample being tested.

7. Apparatus

7.1 A rheometer is a type of dynamic mechanical analyzer especially suited for testing liquid materials. The function of the rheometer apparatus is to hold a liquid test specimen of known volume and dimensions in which the material acts as the elastic and dissipative elements in a mechanically driven oscillatory shear system.

7.2 *Rheometer*—The essential instrumentation required providing the minimum rheological analytical capabilities for this test method include:

7.2.1 A *drive motor*, to apply force or displacement to the specimen in a periodic manner capable of frequencies of oscillation from 0.5 Hz to 2 Hz. This motor may also be capable of providing static force or displacement on the specimen.

7.2.2 A *coupling shaft*, or other means to transmit the force or displacement from the motor to the specimen.

7.2.3 A *fixture, geometry, or tool* to fix the specimen between the drive shaft and a stationary position.

7.2.3.1 Either polished cone-and-plate fixture of diameter D having a known cone angle α (see Fig. 1a) or,

7.2.3.2 Polished parallel plate fixture of known diameter D (see Fig. 1b).

NOTE 2—25-mm or 50-mm diameter cone-and-plate fixtures have been found suitable for this standard. Other diameters may be used.

7.2.4 Either a *force sensor* to measure the force developed by the specimen to within 1 %.

7.2.5 Or *position sensor* to measure the displacement of the test specimen to within 1 %.

7.2.6 A *temperature sensor* to provide an indicate of the specimen temperature over the range of 20 °C to 25 °C readable to within ±0.1 °C.

7.2.7 A *temperature controller* the maintain the temperature of the test specimen over the temperature range of 20 °C to 30 °C to within 0.5 °C.

7.2.8 A *stress or strain controller*, capable of executing a specific unidirectional or oscillatory stress or strain program between selected stress or strain limits to with 1 % or at an iso-stress or iso-strain constant to within 1 %.

7.2.9 A *data collection device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for this measurement are temperature, frequency, loss modulus and storage modulus.

7.2.10 Auxiliary instrumentation considered necessary or useful in conducting this test method includes:

7.2.10.1 *Cooling capability* to sustain an isothermal temperature in the range of 20 °C to 30 °C stable to with 0.5 °C.

7.2.10.2 *Data analysis* capability to provide storage modulus, loss modulus and tangent angle delta or other useful parameters derived from the measured signals of stress and strain.

8. Reagents and Materials

8.1 *Nitrogen* or other inert gas supply for purging purposes.

9. Calibration and Standardization

9.1 Calibrate the instrument using procedures recommended by the manufacturer as described in the operations manual.

10. Procedure

10.1 Apply the test specimen onto the bottom plate of the test fixture. Be certain that there is sufficient material to cover the bottom plate uniformly.

10.2 Lower the upper test fixture (cone or parallel plate) so that it is touching the test specimen. The gap distance between the two plates or the tip of the cone and its plate in approximately 0.5 mm (see H in Fig. 1a or Fig. 1b).

NOTE 3—The recommended minimum gap setting may be equipment dependent and reference shall be made to the manufacturer's operations manual for the correct gap setting. The gap setting may also depend on the homogeneity of the material. The gap shall be greater than 10 times the size of the largest filler particle.

10.3 Ensure that the test specimen temperature is between 21 °C and 25 °C. Report the test temperature.

10.4 Initiate a rotational oscillatory motion to the upper plate or cone of 1 Hz and measure the loss modulus and storage modulus of the test specimen.

NOTE 4—Maximum strain amplitude shall be used to ensure adequate torque signal. The maximum strain amplitude is that which produces a 5 % or less (≤5 %) storage modulus change from that at 0.01 % strain. The strain amplitude may vary from 1 % up to 50 % and still be within the linear viscoelastic region.

10.5 Determine and report the tan δ value from storage shear modulus (G') and loss shear modulus (G") using Eq 1. If

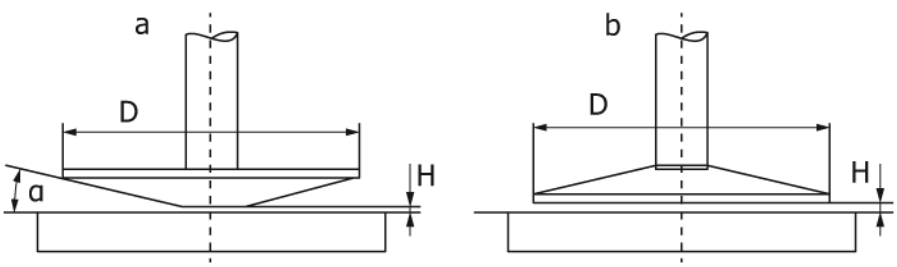

FIG. 1 Schematic Diagram of Cone-and-Plate (a) and Parallel Plate (b) Fixtures