Designation: E 1640 – 99

An American National Standard

Standard Test Method for Assignment of the Glass Transition Temperature By Dynamic Mechanical Analysis[1]

This standard is issued under the fixed designation E 1640; 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 assignment of a glass transition temperature (Tg) of materials using dynamic mechanical analyzers.

1.2 This test method is applicable to thermoplastic polymers, thermoset polymers, and partially crystalline materials which are thermally stable in the glass transition region.

1.3 The applicable range of temperatures for this test method is dependent upon the instrumentation used, but, in order to encompass all materials, the minimum temperature should be about −150°C.

1.4 This test method is intended for materials having an elastic modulus in the range of 0.5 MPa to 100 GPa.

1.5 Electronic instrumentation or automated data analysis and data reduction systems or treatments equivalent to this test method may also be used.

NOTE 1—The user bears the responsibility for determining the precision, accuracy, and validity of the techniques and measurements made using dynamic mechanical analyzers in accordance with this standard. If disputes arise, only the manual procedures described in this standard are to be considered valid.

1.6 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 4065 Practice for Determining and Reporting Dynamic Mechanical Properties of Plastics[2]

D 4092 Terminology Relating to Dynamic Mechanical Measurements in Plastics[2]

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

E 1142 Terminology Relating to Thermophysical Properties[3]

E 1356 Test Method for Glass Transition Temperatures by Differential Scanning Calorimetry or Differential Thermal Analysis[3]

E 1363 Test Method for Temperature Calibration of Thermomechanical Analyzers[3]

E 1545 Test Method for the Determination of Glass Transition Temperatures by Thermomechanical Analysis[3]

2.2 *Other Standard:*

SRM 18R-94 Recommended Method for Glass Transition Temperature (Tg) Determination by DMA of Oriented Fiber-Resin Composites [4]

3. Terminology

3.1 *Definition:*

3.1.1 Specific technical terms used in this document are defined in Terminology D 4092 and E 1142.

3.1.2 *dynamic mechanical analyzer*—any of various commercial or experimental devices used to study the viscoelastic response of a specimen under a forced or free resonant oscillatory load. The force may be applied in torsion, flexure, or a combination of tension and compression.

4. Summary of Test Method

4.1 A specimen of known geometry is placed in mechanical oscillation at either fixed or resonant frequency and changes in the viscoelastic response of the material are monitored as a function of temperature. Under ideal conditions, the glass transition region is marked by a rapid decrease in the storage modulus and a rapid increase in the loss modulus. The glass transition of the test specimen is indicated by the extrapolated onset of the decrease in storage modulus which marks the transition from a glassy to a rubbery solid.

[1] This test method is under the jurisdiction of ASTM Committee E-37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.01 on Test Methods and Recommended Practices.

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[2] *Annual Book of ASTM Standards*, Vol 08.02.

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

[4] Available from Cuppliers of Advanced Composite Materials Association, Arlington, VA.

5. Significance and Use

5.1 This test method can be used to locate the glass transition region and assign a glass transition temperature of amorphous and semi-crystalline materials.

5.2 Dynamic mechanical analyzers monitor changes in the viscoelastic properties of a material as a function of temperature and frequency, providing a means to quantify these changes. In ideal cases, the temperature of the onset of the decrease in storage modulus marks the glass transition.

5.3 A glass transition temperature (T_g) is useful in characterizing many important physical attributes of thermoplastic, thermosets (see SRM 18R-94), and semi-crystalline materials including their thermal history, processing conditions, physical stability, progress of chemical reactions, degree of cure, and both mechanical and electrical behavior. T_g may be determined by a variety of techniques and may vary in accordance with the technique.

5.4 This test method is useful for quality control, specification acceptance, and research.

6. Interferences

6.1 Because the specimen size will usually be small, it is essential that each specimen be homogeneous and/or representative of the material as a whole.

6.2 An increase or decrease in heating rates from those specified may alter results.

6.3 A transition temperature is a function of the experimental frequency, therefore the frequency of test must always be specified. (The transition temperature increases with increasing frequency.) Extrapolation to a common frequency may be accomplished using a predetermined frequency shift factor or assuming the frequency shift factor of about 8°C per decade of frequency.[5]

7. Apparatus

7.1 The function of the apparatus is to hold a specimen of uniform dimension so that the sample acts as the elastic and dissipative element in a mechanically oscillated system. Dynamic mechanical analyzers typically operate in one of several modes. See Table 1.

TABLE 1 Modes for Dynamic Mechanical Analyzers

Mode	Mechanical Response			
	Tension	Flexural	Torsional	Compression
Free/dec	...	...	X	...
Forced/res/CA	...	X	X	...
Forced/fix/CA	X	X	X	X
Forced/fix/CS	X	X	...	X

Free = free oscillation; dec = decaying amplitude; forced = forced oscillation; CA = constant amplitude; res = resonant frequency; fix = fixed frequency; CS = controlled stress.

7.2 The apparatus shall consist of the following:

7.2.1 *Clamps*, a clamping arrangement that permits gripping of the specimen. Samples may be mounted by clamping at both ends (most systems), one end (for example, torsional pendulum), or neither end (free bending between knife edges).

7.2.2 *Oscillatory Stress (Strain)*, for applying an oscillatory deformation (strain) or oscillatory stress to the specimen. The deformation may be applied and then released, as in freely vibrating devices, or continuously applied, as in forced vibration devices.

7.2.3 *Detector*, for determining the dependent and independent experimental parameters, such as force (or stress), displacement (or strain), frequency, and temperature. Temperatures should be measurable with an accuracy of ±0.5°C, force to ±1 %, and frequency to ±0.1 Hz.

7.2.4 *Temperature Controller and Oven*, for controlling the specimen temperature, either by heating, cooling (in steps or ramps), or by maintaining a constant experimental environment. The temperature programmer shall be sufficiently stable to permit measurement of specimen temperature to ±0.5°C. The precision of the required temperature measurement is ±1.0°C.

7.2.5 *Output Device*, capable of displaying the storage modulus (either linearly or logarithmically) on the *Y* axis increasing in the upward direction and temperature on the *X* axis increasing to the right.

NOTE 2—Some instruments suitable for this test may display only linear or logarithm storage modulus while others may display either linear and/or logarithm storage modulus. Care must be taken to use the same modulus scale when comparing unknown specimens, and in the comparison of results from one instrument to another.

7.3 *Nitrogen, Helium* or other gas supplied for purging purposes.

7.4 *Calipers* or other length measuring device capable of measuring dimensions (or length within)± 0.01 mm.

8. Precautions

8.1 Toxic and corrosive, or both, effluents may be released when heating some materials and could be harmful to personnel and to apparatus.

8.2 *Multiple Transitions*—Under some experimental conditions it is possible to have transitions secondary to the primary glass transition. Secondary transitions may be related to the glass transition of a second polymeric phase, melt processes, crystallization, chemical reactions, the motion of groups pendent to the main backbone or the crankshaft motion of the polymer backbone.

9. Samples

9.1 Samples may be any uniform size or shape, but are ordinarily analyzed in rectangular form. If some heat treatment is applied to the specimen to obtain this preferred analytical form, such treatment should be noted in the report.

9.2 Due to the numerous types of dynamic mechanical analyzers, sample size is not fixed by this method. In many cases, specimens measuring between $1 \times 5 \times 20$ mm and $1 \times 10 \times 50$ mm are suitable.

NOTE 3—It is important to select a specimen size appropriate for both the material and the testing apparatus. For example, thick samples may be

[5] Ferry, D. "Viscoelastic Properties of Polymers," John Wiley & Sons, 1980.