



Designation: E1640 – 23

Standard Test Method for Assignment of the Glass Transition Temperature By Dynamic Mechanical Analysis¹

This standard is issued under the fixed designation E1640; 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 (T_g) 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 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *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

¹ This test method is under the jurisdiction of ASTM Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.10 on Fundamental, Statistical and Mechanical Properties.

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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.

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

E1142 Terminology Relating to Thermophysical Properties

E1363 Test Method for Temperature Calibration of Thermomechanical Analyzers

E1545 Test Method for Assignment of the Glass Transition Temperature by Thermomechanical Analysis

E1867 Test Methods for Temperature Calibration of Dynamic Mechanical Analyzers

E2254 Test Method for Storage Modulus Calibration of Dynamic Mechanical Analyzers

E2425 Test Method for Loss Modulus Conformance of Dynamic Mechanical Analyzers

E3142 Test Method for Thermal Lag of Thermal Analysis Apparatus

3. Terminology

3.1 Definitions:

3.1.1 Specific technical terms used in this document are defined in Terminologies **D4092** and **E1142** including *Celsius*, *dynamic mechanical analyzer*, *glass transition*, *glass transition temperature*, *loss modulus*, *storage modulus*, *tangent delta*, and *viscoelasticity*.

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, during heating, the glass transition region is marked by a rapid decrease in the storage modulus and a rapid increase in the loss modulus and tangent delta. 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.

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

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

changes. In ideal cases, the temperature of the onset of the decrease in storage modulus marks the glass transition.

5.3 The glass transition takes place over a temperature range. This method assigns a single temperature (T_g) to represent that temperature range as measured by dynamic mechanical analysis. T_g may be determined by a variety of techniques and may vary according to that technique.

5.4 A glass transition temperature (T_g) is useful in characterizing many important physical attributes of thermoplastic, thermosets, 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.

5.5 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 or representative of the material as a whole, or both.

6.2 An increase or decrease in heating rates from those specified may alter results (see [Appendix X2](#)).

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.³ Such extrapolation shall be reported.

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](#).

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 shall be readable to within ± 0.1 °C, force to ± 1 %, and frequency to ± 0.1 Hz.

NOTE 1—The temperature sensor shall be placed as close as is practical, but not touching, the test specimen

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 *Data Collection Device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals require for dynamic mechanical analysis are storage modulus, loss modulus, tangent delta, temperature and time.

NOTE 2—Some instruments suitable for this test may display only linear or logarithm storage modulus while others may display either linear or logarithm storage modulus, or both. 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.

NOTE 3—The same purge gas shall be used for calibration and measurement of the test specimen.

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 pendant 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 reported.

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

³ Ferry, D., *Viscoelastic Properties of Polymers*, John Wiley & Sons, 1980.

TABLE 1 Modes for Dynamic Mechanical Analyzers

NOTE 1—Free = free oscillation; dec = decaying amplitude; forced = forced oscillation; CA = constant amplitude; res = resonant frequency; fix = fixed frequency; CS = controlled stress.

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