



Designation: E2425 – 21

# Standard Test Method for Loss Modulus Conformance of Dynamic Mechanical Analyzers<sup>1</sup>

This standard is issued under the fixed designation E2425; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method describes the performance confirmation or measurement of conformance for the loss modulus scale of a commercial or custom-built dynamic mechanical analyzer (DMA) at 21 °C using ultra-high molecular weight polyethylene as a reference material.

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:*<sup>2</sup>

[D4092 Terminology for Plastics: Dynamic Mechanical Properties](#)

[E473 Terminology Relating to Thermal Analysis and Rheology](#)

[E1142 Terminology Relating to Thermophysical Properties](#)

[E1867 Test Methods for Temperature Calibration of Dynamic Mechanical Analyzers](#)

<sup>1</sup> 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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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

## 3. Terminology

3.1 *Definitions*—Specific technical terms used in this test method are defined in Terminologies [D4092](#), [E473](#), and [E1142](#), including *Celsius*, *dynamic mechanical analysis*, and *loss modulus*.

## 4. Summary of Test Method

4.1 The loss modulus signal measured by a dynamic mechanical analyzer for an elastic material is compared to the reported loss modulus for that reference material. A linear relationship is used to correlate the experimental loss modulus signal with the reported value of the reference material.

4.2 The mode of deformation (for example, tensile, flexure, compression, shear, etc.) shall be reported.

## 5. Significance and Use

5.1 This test method demonstrates conformity of a dynamic mechanical analyzer at an isothermal temperature of 21 °C.

5.2 Dynamic mechanical analysis experiments often use linear temperature change. This test method does not address the effect of that change in temperature on the loss modulus.

5.3 This test method may be used in research and development, specification acceptance, and quality control or assurance.

## 6. Apparatus

6.1 The essential instrumentation required to provide the minimum dynamic mechanical capability for this test method includes:

6.1.1 *Drive Actuator*, to apply force (or displacement) to the specimen in a periodic manner. This actuator may also be capable of providing static force or displacement to the specimen.

6.1.2 *Coupling Shaft*, or other means to transmit the force from the motor to the specimen.

6.1.3 *Clamping System(s)*, to fix the specimen between the drive shaft and the stationary clamp(s).

6.1.4 *Position Sensor*, to measure the change in position of the specimen during dynamic motion, or

6.1.5 *Force Sensor*, to measure the force developed by the specimen.

6.1.6 *Temperature Sensor*, to provide an indication of the specimen temperature to within  $\pm 1$  °C.

6.1.7 *Furnace*, to provide controlled heating or cooling of a specimen at a constant temperature or at a constant rate within the applicable temperature range of  $-100$  °C to  $+300$  °C.

6.1.8 *Temperature Controller*, capable of executing a specific temperature program by operating the furnace between  $-100$  °C and  $+300$  °C and at a constant temperature within that range.

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

6.2 Auxiliary instrumentation considered necessary in conducting this method near or below ambient temperature:

6.2.1 *Cooling* capability to sustain a constant temperature at or below ambient temperature or to provide controlled cooling.

6.2.2 *Data Analysis* capability to provide loss modulus information derived from measured signals.

**7. Reagents and Materials**

7.1 A reference material of known loss modulus, formed to the shape suitable for characterization by the particular dynamic mechanical analyzer (see Table 1).

**8. Sampling and Test Specimens**

8.1 Test specimens are typically prepared in the form of a rectangular test bars or film strips.

NOTE 1—It is common practice to bevel or “break” edges of machined parts. This practice shall not be followed in the preparation of test specimens for this method. The measured loss modulus of such specimens reads low due to imperfect sample geometry.

**9. Calibration and Standardization**

9.1 Perform any loss modulus signal calibration procedure recommended by the manufacturer of the dynamic mechanical analyzer as described in the operations manual.

9.2 If not already done so, calibrate the temperature scale of the dynamic mechanical analyzer as near as is practical to the test temperature (see Table 1) using Test Methods E1867.

**10. Procedure**

10.1 Prepare the dynamic mechanical analyzer for operation under the test conditions (for example, specimen clamps, purge gas, etc.) to be used for the characterization of the test

specimen(s). Unless otherwise indicated the temperature condition shall be isothermal at  $21$  °C  $\pm 1$  °C (that is, between  $20$  °C and  $22$  °C).

10.2 Ensure that the loss modulus signal is less than  $1$  MPa with no test specimen loaded and at an oscillation test frequency to be used in this test (see Table 1).

NOTE 2—Alternatively, a thin specimen of a low modulus material (for example, a thin piece of paper) may be used. The dimension of the test specimen shall be used rather than the true dimensions of the thin, low modulus material.

10.3 Open the apparatus, place the reference material into the specimen holder, and reassemble the apparatus. Equilibrate the reference material at the test conditions for  $30$  min. Unless otherwise indicated, the test frequency shall be that indicated in Table 1 and the temperature shall be isothermal between  $20$  °C and  $22$  °C. Ensure that the applied strain (or stress) amplitude is within the linear viscoelastic region of the sample.

10.4 Record the loss modulus observed by the apparatus as  $E_o$ .

10.5 Record the loss modulus of the reference material from its certificate or from Table 1 as  $E_s$ .

10.6 Calculate and report the value of the slope ( $S$ ) and conformity ( $C$ ) of the measurement using Eq 2 and Eq 3.

**11. Calculation**

11.1 For the purpose of this test method, it is assumed that the relationship between the observed loss modulus ( $E_o$ ) and the reference loss modulus ( $E_s$ ) is linear and governed by the slope ( $S$ ) of Eq 1:

$$E_s = E_o \times S \tag{1}$$

11.2 By using the loss modulus values taken from 10.4 and 10.5, calculate and report  $S$  using Eq 2 to four decimal places.

$$S = E_s/E_o \tag{2}$$

11.3 The conformity ( $C$ ) (that is, the percent difference between the experimental slope and unity) of the instrument loss modulus scale is calculated using the value of  $S$  from 11.2 and Eq 3.

$$C = (S - 1.0000) \times 100 \% \tag{3}$$

11.3.1 Conformity may be estimated to one significant figure using the following criteria:

11.3.1.1 If the value of  $S$  is between  $0.9990$  and  $0.9999$  or between  $1.0001$  and  $1.0010$ , then the conformity is better than  $0.1$  %.

11.3.1.2 If the value of  $S$  is between  $0.9900$  and  $0.9990$  or between  $1.0010$  and  $1.0100$ , then conformity is better than  $1$  %.

11.3.1.3 If the value of  $S$  is between  $0.9000$  and  $0.9900$  or between  $1.0100$  and  $1.1000$ , then the conformity is better than  $10$  %.

11.4 Report the value of slope ( $S$ ) and the conformity ( $C$ ).

**12. Report**

12.1 The report shall include the following information:

12.1.1 Details and description of the dynamic mechanical analyzer, including the manufacturer and instrument model

**TABLE 1 Reference Material Loss Modulus<sup>A</sup>**

Temperature (°C)	Material	Frequency Hz	Loss Modulus (MPa)
21	Ultra-High Molecular Weight Polyethylene <sup>B</sup>	1	62.0

<sup>A</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E37-1041. Contact ASTM Customer Service at service@astm.org.

<sup>B</sup> SRM 8456 available from the National Institute for Standards and Technology (NIST), Gaithersburg, MD, USA.