# Designation: E2425 − 16

# Standard Test Method for Loss Modulus Conformance of Dynamic Mechanical Analyzers[1]

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 (ε) 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 There is no ISO standard equivalent to this test method.

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 to determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

2.1 *ASTM Standards:*[2]

E473 Terminology Relating to Thermal Analysis and Rheology

E1142 Terminology Relating to Thermophysical Properties

E1867 Test Methods for Temperature Calibration of Dynamic Mechanical Analyzers

## 3. Terminology

3.1 *Definitions*—Specific technical terms used in this test method are defined in Terminologies 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 ±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 to +300°C.

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

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[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.

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

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**TABLE 1 Reference Material Loss Modulus[A]**

| Temperature (°C) | Material | Frequency Hz | Loss Modulus (MPa) |
|---|---|---|---|
| 21 | Ultra-High Molecular Weight Polyethylene[B] | 1 | 62.0 |

[A] 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.
[B] SRM 8456 available from the National Institute for Standards and Technology (NIST), Gaithersburg, MD, USA.

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 ± 1°C (that is, between 20 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 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.

$$E_s = E_o \times S \quad (1)$$

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.

$$S = E_s / E_o \quad (2)$$

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.

$$C = (S - 1.0000) \times 100\,\% \quad (3)$$

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 0.990 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 0.9000 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 number, where applicable. Also report the test mode, strain amplitude, frequency and applied static load.

12.1.1.1 Whether or not the instrument calibration included compliance corrections.

12.1.2 The value of slope ($S$) determined in 11.2, reported to at least four decimal places.

12.1.3 The conformity ($C$), as determined in 11.3.

12.1.4 The specific dated version of this test method used.

## 13. Precision and Bias

13.1 An interlaboratory study was conducted in 2010 that included participation by 15 laboratories using 7 instrument models from 4 manufacturers.[3] A single sample of ultra-high molecular weight polyethylene[4] was characterized in quintuplicate.

13.2 *Precision:*

13.2.1 Within laboratory variability may be described using the repeatability value (r) obtained by multiplying the repeatability standard deviation by 2.8. The repeatability value estimates the 95 % confidence limit. That is, two results from the same laboratory should be considered suspect (at the 95 % confidence level) if they differ by more than the repeatability value.

13.2.2 The within laboratory repeatability standard deviation was 2.3 MPa resulting in a repeatability relative standard deviation of 3.8 % with 48 degrees of experimental freedom The repeatability value r is 6.7 MPa.

[3] 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.

[4] SRM 8456 available from the National Institute for Standards and Technology (NIST), Gaithersburg, MD, USA.

13.2.3 The between laboratory variability may be described using the reproducibility value (R) obtained by multiplying the reproducibility standard deviation by 2.8. The reproducibility value estimates the 95 % confidence level. That is, results obtained by two different laboratories, operators or apparatus should be considered suspect (at the 95 % confidence level) if they differ by more than the reproducibility value.

13.2.4 The between laboratory reproducibility standard deviation 8.6 MPa resulting in a reproducibility relative standard deviation of 14 %. The reproducibility value R is 24 MPa.

13.3 *Bias:*

13.3.1 Bias is the difference between the mean value obtained and an acceptable reference value for the same material. To the knowledge of the committee, no acceptable loss modulus reference material is available. Therefore, bias is unable to be determined.

13.3.2 The mean loss modulus value for the ultra-high molecular weight polyethylene was 62.0 MPa.

## 14. Keywords

14.1 calibration; conformity; dynamic mechanical analysis; loss modulus

## SUMMARY OF CHANGES

Committee E37 has identified the location of selected changes to this standard since the last issue (E2425 – 11) that may impact the use of this standard. (Approved June 1, 2016.)

*(1)* Technical change to 10.3.

*(2)* Editorial changes to 3.1, Table 1, and 13.1.

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