Designation: E2254 – 23

Standard Test Method for Storage Modulus Calibration of Dynamic Mechanical Analyzers[1]

This standard is issued under the fixed designation E2254; 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 calibration or performance confirmation for the storage modulus scale of a commercial or custom built dynamic mechanical analyzer (DMA) over the temperature range of –100 °C to 300 °C using reference materials in the range of 1 GPa to 200 GPa.

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]

E473 Terminology Relating to Thermal Analysis and Rheology
E698 Test Method for Kinetic Parameters for Thermally Unstable Materials Using Differential Scanning Calorimetry and the Flynn/Wall/Ozawa Method
E1142 Terminology Relating to Thermophysical Properties
E2425 Test Method for Loss Modulus Conformance of Dynamic Mechanical Analyzers
D638 Test Method for Tensile Properties of Plastics
E3142 Test Method for Thermal Lag of Thermal Analysis Apparatus

3. Terminology

3.1 *Definitions:*

3.1.1 Specific technical terms used in this test method are defined in Terminologies E473 and E1142 including *Celsius, dynamic mechanical analysis*, and *storage modulus*.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *accepted reference value (ARV), n*—a value that serves as an agreed upon reference for comparison and which is derived as either a theoretical or established value, based on scientific principles, or as assigned value, based on experimental work.

4. Summary of Test Method

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

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

5. Significance and Use

5.1 This test method calibrates or demonstrates conformity of a dynamic mechanical analyzer at an isothermal temperature within the range of –100 °C to 300 °C.

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

5.3 A calibration factor may be required to obtain corrected storage modulus values.

5.4 This 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:

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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**

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

6.1.6 *Temperature Sensor,* to provide an indication of the specimen temperature to ±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.

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

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

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

6.3 *Micrometer,* calipers or other length measuring device capable of measuring length of 1.0 mm to 100 mm with a precision of ±0.01 mm.

7. Reagents and Materials

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

NOTE 1—The storage modulus of the calibration materials used in this standard is often similar to that of the construction materials of the test apparatus. Thus the examination of high modulus materials may result in instrument compliance during testing. The test apparatus calibration procedure (see 9.1) should include a compliance correction. The user of this standard shall verify whether or not such compliance corrections are included prior to its use.

8. Sampling

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

NOTE 2—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 storage modulus of such test specimens reads low due to imperfect sample geometry.

9. Calibration and Standardization

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

9.2 Perform a temperature calibration at isothermal temperature conditions using Test Method E3142 (see Appendix X1).

NOTE 3—Quality initiatives call for apparatus calibration at least annually.

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 specimens. Unless otherwise indicated, the temperature condition shall be isothermal between 20 °C and 23 °C.

10.2 Ensure that the storage modulus signal is less than 1 MPa with no test specimen loaded and at an oscillation test frequency of 1 Hz.

NOTE 4—Alternatively, a thin specimen of a low modulus material (for example, a thin piece of paper) may be used. The dimensions of the test specimen (see 10.3) shall be used rather than the true dimensions of the thin low modulus material.

10.3 Measure and record the dimension of the test specimen to a precision of ±0.01 mm.

TABLE 1 Accepted Reference Values for Material Modulus[A,B,C]

	Storage Modulus, GPa					
Temperature, °C	Carbon Steel[D]	Monel[E]	Copper[F]	Aluminum	UHMWPE	Nimonic 75 (BCR-661)[G,H]
–198	207	185	121	77.9	. . .	. . .
–101	201	182	116	75.8	. . .	. . .
–46	198	180	114	74.5	. . .	. . .
21	192	179	114	73.1	1.26	210.0 ± 1.5
93	191	179	112	71.7	. . .	. . .
149	189	178	112	70.3	. . .	. . .
204	186	177	110	65.5	. . .	. . .
260	182	175	. . .	. . .	. . .	. . .
316	177	170	. . .	. . .	. . .	. . .

[A] American Society of Mechanical Engineers, *Refrigeration Piping*, B31.5a, New York, NY, 1992, p. 45.
[B] *Perry's Chemical Engineers' Handbook*, R.H. Perry, D.W. Green, J.O. Maloney (eds.), 6th Edition, McGraw-Hill, New York, NY, 1984, pp. 6–92.
[C] *Ultra High Molecular Weight Polyethylene Standard Reference Material SRM 8456*, National Institute of Standards and Technology, Gaithersburg, MD 20899, 2000.
[D] 3.5 % Ni, <0.30 C.
[E] 67 % Ni, 30 % Cu.
[F] 99.90 % Cu, Alloy C12000, C12200.
[G] Available from Institute of Reference Materials and Measurements, Retieseweg 111, B-2440 Geel, Belgium.
[H] Lord, J. D., and Morrell, R., "Elastic Modulus Measurement," *Measurement Good Practice Guide No. 98*, National Physical Laboratory, Teddington, Middlesex, UK, 2006.

10.4 Open the apparatus, place the reference material into the specimen holder, and reassemble the apparatus. Equilibrate the reference material at the test conditions. Unless otherwise indicated, the test frequency shall be 1 Hz 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 regions of the sample typically less than 1 % strain.

NOTE 5—Typical specimen size is 50 mm × 9 mm × 1 mm. The specimen should have a length-to-thickness ratio greater than 10-to-1.

10.5 Record the storage modulus observed by the apparatus as *Eo*.

10.6 Record the storage modulus of the reference material from its certificate or from Table 1 as *Es*.

10.7 Calculate and report the value of the slope (*S*) and percent conformity (*C*) of the measurement using Eq 2 and 3.

11. Calculation

11.1 For the purpose of this test method, it is assumed that the relationship between observed storage modulus (*Eo*) and the reference storage modulus (*Es*) is linear and governed by the slope (*S*) of Eq 1.

$$Es = Eo \times S \tag{1}$$

11.2 By using the storage modulus values taken from 10.5 and 10.6 calculate and report *S* using Eq 2 to four decimal places.

$$S = Es/Eo \tag{2}$$

11.3 The percent conformity (*C*) (that is, the percent difference between the experimental slope and unity) of the instrument storage 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 conformity is better than 10 %.

11.4 Report the value of *S* and the percent conformity, (*C*).

11.5 Using the slope (*S*) from Eq 2, the observed storage modulus (*Eo*) can provide a corrected storage modulus (*E*) using Eq 4.

$$E = Eo \times S \tag{4}$$

12. Report

12.1 The report shall include the following information:

12.1.1 Details and description, including the manufacturer and instrument model number, where applicable, of the dynamic mechanical analyzer. Also report the test mode, strain amplitude, and applied static load.

12.1.1.1 Whether or not the instrument calibration includes compliance correction.

12.1.2 The value of *S* determined in 11.2, reported to at least four decimal places.

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

12.1.4 The specific dated version of this method used.

13. Precision and Bias

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

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 0.041 GPa resulting in a repeatability relative standard deviation of 4.8 % with 48 degrees of experimental freedom. The repeatability value r thus 0.11 GPa.

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 limit. That is, results obtained from 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 was 0.16 GPa resulting in a reproducibility relative standard deviation of 14 %. The reproducibility value R thus is 0.45 GPa.

13.3 *Bias:*

13.3.1 Bias is the difference between the mean value obtained and an acceptable reference value for the same material.

13.3.2 The Young's modulus calculated from the storage modulus (*E*') value determined by this standard and the loss modulus (*E*") value determined by Test Method E2425 may be compared to the Young's modulus (*E*) obtained by Test Method E698 by the equation:

$$E' = [E^2 - E''^2]^{1/2}$$

13.3.3 The value for Young's modulus determined for this material by Test Method D638 is 1.256 GPa.

13.3.4 The mean value for storage modulus (*E*') determined by this standard is 1.184 GPa.

13.3.5 The mean value for loss modulus (*E*") for this material determined by Test Method E2425 was 62.0 MPa.

13.3.6 Based upon the Student's "t" test, these values for Young's modulus are equivalent. That is, no bias is detected.

[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.