



# Standard Test Method for Storage Modulus Calibration of Dynamic Mechanical Analyzers<sup>1</sup>

This standard is issued under the fixed designation E 2254; 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 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 to 300 °C using reference materials in the range of 1 to 200 GPa.

1.2 SI units are the 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 and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

E 473 Terminology Relating to Thermal Analysis and Rheology

E 1142 Terminology Relating to Thermophysical Properties

## 3. Terminology

3.1 *Definitions*—Specific technical terms used in this test method are defined in Terminologies E 473 and E 1142.

## 4. Summary of Test Method

4.1 The storage modulus signal measured 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 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:

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 developed by the specimen.

6.1.6 *Temperature Sensor*, to provide an indication of the specimen temperature to  $\pm 1$  °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 to +300 °C.

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

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.01 on Thermal Test Methods and Practices.

Current edition approved Feb. 1, 2008. Published March 2008. Original approved in 2003. Last previous edition approved in 2003 as E 2254-03.

<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.

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 to 100 mm with a precision of  $\pm 0.01$  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.

TABLE 1 Reference Material Modulus<sup>A,B,C</sup>

| Storage Modulus, GPa |                           |                    |                     |          |        |
|----------------------|---------------------------|--------------------|---------------------|----------|--------|
| Temperature, °C      | Carbon Steel <sup>D</sup> | Monel <sup>E</sup> | Copper <sup>F</sup> | Aluminum | UHMWPE |
| -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   |
| 93                   | 191                       | 179                | 112                 | 71.7     | ...    |
| 149                  | 189                       | 178                | 112                 | 70.3     | ...    |
| 204                  | 186                       | 177                | 110                 | 65.5     | ...    |
| 260                  | 182                       | 175                | ...                 | ...      | ...    |
| 316                  | 177                       | 170                | ...                 | ...      | ...    |

<sup>A</sup> American Society of Mechanical Engineers, New York, NY, B31.5a, *Refrigeration Piping*, page 45 (1992).

<sup>B</sup> *Perry's Chemical Engineers' Handbook*, R.H. Perry, D.W. Green, J.O. Maloney (eds.), 6th Edition, McGraw-Hill, New York, NY, page 6-92 (1984).

<sup>C</sup> *Ultra High Molecular Weight Polyethylene Standard Reference Material SRM 8456*, National Institute of Standards and Technology, Gaithersburg, MD 20899 (2000).

<sup>D</sup> 3.5 % Ni, <0.30 C.

<sup>E</sup> 67 % Ni, 30 % Cu.

<sup>F</sup> 99.90 % Cu, Alloy C12000, C12200.

## 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 and 22 °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 3—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  $\pm 0.01$  mm. 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 2 and 23 °C. Ensure that the applied strain (or stress) amplitude is within the linear viscoelastic regions of the sample.

NOTE 4—Typical specimen size is 50 x 9 x 1 mm.

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

10.5 Record the storage 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 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 ( $E_o$ ) and the reference storage 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 storage 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 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](#).

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

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 0.9990 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 ( $E_o$ ) can provide a corrected storage modulus ( $E$ ) using Eq 4.

$$E = E_o \times S \quad (4)$$

## 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 is planned for 2009–2010 to generate precision and bias information for this method.

Anyone wishing to participate in this study may contact the E37 Staff Manager at ASTM Headquarters.

### 13.2 Precision:

13.2.1 The intralaboratory repeatability standard deviation for  $S$  for a single instrument using carbon steel and was found to be 0.67 %.

### 13.3 Bias:

13.3.1 The measurement of conformity in this test method is a comparison of the calibration constant  $S$  with the theoretical value of 1.0000 and provides an indication of bias.

13.3.2 The intralaboratory conformity for  $C$  for a single instrument using ultra high molecular weight polyethylene following calibration with carbon steel was 4.0 %.

## 14. Keywords

14.1 calibration; conformity; dynamic mechanical analysis; storage modulus

*ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.*

*This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.*

*This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).*