Designation: E2509 – 14

Standard Test Method for
Temperature Calibration of Rheometers in Isothermal Mode[1]

This standard is issued under the fixed designation E2509; 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 temperature calibration or conformance of rheometers. The applicable temperature range is 0 to 80°C however other ranges may be selected for the purpose at hand.

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 are no ISO equivalents to this standard.

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 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

3. Terminology

3.1 *Definitions*—Specific technical terms found in this standard are defined in Terminologies E473 and E1142, including *rheometer* and *rheometry*.

4. Summary of Test Method

4.1 An electronic thermometer of known characteristics is placed in the center of a dummy test specimen in contact with the torque applying instrument plates of a rheometer at constant (isothermal) temperature. The difference between the rheometer set temperature and that indicated by the thermometer is used to calibrate the rheometer temperature signal.

5. Significance and Use

5.1 Rheological properties such as viscosity and storage and loss modulus change rapidly with temperature. High quality determinations of these properties depend upon a stable and well-known temperature of the measuring apparatus.

6. Interferences

6.1 In many rheological experiments, temperature is varied with time. The calibration in this test method is made under stable and isothermal temperature conditions. Thus the effects of changes in temperature with time are not addressed. This isothermal calibration does not provide any information about the specimen under temperature scanning conditions.

7. Apparatus

7.1 An electronic thermometer that includes:

7.1.1 *Temperature sensor,* (such as a thermocouple, platinum resistance thermometer, thermistor, etc.) with an accuracy (traceable to a known absolute standard) and resolution of ±0.1°C and a range of 0 to 80°C.

NOTE 1—Sensors with other temperature ranges may be used at the operator's convenience.

NOTE 2—Some sensors are available already affixed with dummy test specimens from section 7.2

7.1.2 *Temperature indicator,* to convert the signal presented by the temperature sensor into a digital electronic temperature display with the accuracy and precision indicated in section 7.1.1.

7.2 *Dummy test specimen,* two polymer sheets each 1 mm in thickness of such a diameter to fill the space (that is, gap) between the instrument plates.

NOTE 3—The dummy test specimen may be composed of the material to be tested or some other representative polymer material. Polydimethylsiloxane (PDMS) (for example, "Silly Putty"[3]) may be used for this purpose.

NOTE 4—Polydimethylsiloxane may leave a residue of silicone oil on the surfaces of the instrument plates. This oil should be removed prior to subsequent use.

7.3 *Rheometer,* the essential instrumentation required providing the minimum rheological analytical capabilities for this test method include:

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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 E37.08 on Rheology.

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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.

[3] The trademark Silly Putty is registered to Crayola Properties, inc., Easton, PA, 18042.

7.3.1 A *drive actuator*, to apply torque or displacement to the specimen in a periodic manner capable of frequencies of oscillation from 0.01 to 10 rad/s (0.0016 to 1.6 Hz). This actuator may also be capable of providing static force or transient step or displacement of the test specimen.

7.3.2 A *coupling shaft*, or other means to transmit the torque or displacement from the actuator to the specimen.

7.3.3 A *geometry, tools or plates*, to fix the specimen between the coupling shaft and a stationary position. For the purposes of this test, parallel plates are the preferred configuration

7.3.4 Either a *torque sensor*, to measure force developed by the specimen or a position sensor to measure the angular displacement , either one being capable of measuring within limits appropriate to the specimen and test being performed.

7.3.5 A *temperature sensor*, to provide an indication of the specimen temperature readable to within ±0.1°C.

7.3.6 A *furnace or heating/cooling element*, to provide controlled heating or cooling of a specimen to a constant temperature constant to within ±0.1°C over the temperature range of interest.

7.3.7 A *temperature controller*, capable of executing a specific temperature program by operating the furnace or heating/cooling element between selected temperature limits constant to within ±0.1°C.

7.3.8 A *stress or stain controller*, capable of executing a specific unidirectional or oscillatory stress or strain program between selected stress or strain limits capable of controlling within limits appropriate to the specimen and test being performed.

7.3.9 A *data collecting device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required include applied force, position or frequency or calculated signal (such as viscosity, storage modulus, loss modulus, or tangent delta) using a linear or logarithmic scale and the independent experimental parameters (such as temperature, time, stress, strain, or frequency of oscillation).

7.3.10 Auxiliary instrumentation considered necessary or useful in conducting this test method includes:

7.3.10.1 A *cooling capability* to hasten cool down from elevated temperatures, to provide constant cooling rates, or to sustain an isothermal subambient temperature.

7.3.10.2 *Data analysis capability*, to provide determined signals (such as viscosity, storage, or loss modulus) or other useful parameters derived from the measured signals.

8. Preparation of Apparatus

8.1 Turn on the rheometer and allow it to equilibrate for at least 30 minutes prior to temperature calibration.

8.2 Assemble the rheometer with the instrument plates to be used during subsequent tests.

9. Calibration and Standardization

9.1 Perform any temperature calibration procedures recommended by the rheometer manufacturer as described in the instruments operations manual.

10. Procedure

10.1 Insert the temperature sensor so that it is located at the vertical and radial center of the dummy test specimen.

NOTE 5—This may be accomplished by placing the sensor between two sheets of the dummy test specimen.

10.2 Mount the dummy test specimen between the instrument plates. Close the gap to the dimension to be used for the test specimen, keeping the temperature sensor centered vertically and radially.

NOTE 6—Other gaps and plate diameters may be used but shall be reported.

NOTE 7—It is not necessary to trim the dummy test specimen but a large excess of material beyond the edges of the plates should be avoided.

10.3 Heat (or cool) the plates to the desired calibration temperature and equilibrate until the indicated temperature changes by less than ±0.1°C in 5 min.

10.4 Measure and record the temperature indicated by the thermometer as T_o and that of set temperature of the rheometer as T_s.

10.5 Determine the temperature calibration value according to 11.

NOTE 8—Depending upon the needs of the user, a single-point temperature calibration may be adequate. In this case, a single offset calibration value is determined. Others may prefer a two-point temperature calibration where the temperature values of interest are selected to encompass all test temperatures. Here, a linear interpolation of results between the two temperature calibration points may be used. Some users may wish to calibrate the apparatus at temperature intervals over the full range of the temperature range. In this case, a working curve composed of offset values as a function of temperature should be created.

11. Calculation or Interpretation of Results

11.1 The temperature response of the apparatus is assumed to be linear and is described by the equation:

$$T_o = S\,T_s + b \tag{1}$$

where:

T_o = observed temperature in °C,
T_s = requested controller temperature in °C,
S = slope of the plot of T_o versus T_s, dimensionless, and
b = temperature offset or bias (intercept of the T_o versus T_s plot) in °C.

11.2 *Single-Point Temperature Calibration:*

11.2.1 In a single-point temperature calibration, it is assumed that the slope (S) for the instrument calibration is 1.00000 and that there is only an offset between the observed and requested temperature. This is a reasonable assumption where the temperature range to be used is narrow.

11.2.2 The offset or bias (b) is given by:

$$b = T_o - T_s \tag{2}$$

11.2.3 The value for b is determined by entering the values for T_o and T_s measured according to 10.4 into Eq 2.

11.2.4 The true value for an instrument requested temperature is then given by:

$$T = T_s + b \tag{3}$$

where:

T = true specimen temperature in °C.

11.3 *Two-Point Temperature Calibration:*

11.3.1 In a two-point temperature calibration, the response of the instrument is assumed to be linear and the slope and offset may be used to describe the relationship between the requested temperature and that achieved. This is a reasonable assumption over a broad temperature range for well-designed instruments.

11.3.2 The slope (S) of the calibration plot is given by:

$$S = [T_o(hi) - T_o(lo)]/[T_s(hi) - T_s(lo)] = \Delta T_o/\Delta T_s \qquad (4)$$

where:

$T_o(hi)$ = high observed temperature in °C,
$T_o(lo)$ = low observed temperature in °C,
$T_s(hi)$ = high set temperature in °C, and
$T_s(lo)$ = low set temperature in °C.

are taken from measurements according to 10.4.

11.3.3 The offset (b) is the intercept of the calibration plot and is given by:

$$b = \{[T_s(hi) \times T_o(lo)] - [T_s(lo) \times T_o(hi)]\}/[T_s(hi) - T_s(lo)] \qquad (5)$$

where:

b = calibration intercept in °C.

11.3.4 The true temperature for an observed temperature measurement is then given by:

$$T = S\,T_s + b \qquad (6)$$

11.4 *Multi-Point Temperature Calibration:*

11.4.1 In the multi-point temperature calibration, the response of the apparatus is considered to be linear over the short difference interval between observation points, but non-linear over the large temperature interval of the whole range of the apparatus.

11.4.2 Prepare a calibration working table with three columns labeled observed temperature (T_o), requested controller temperature (T_s) and temperature difference (b) where:

$$b = T_o - T_s \qquad (7)$$

where:

$T_o(hi)$ = high observed temperature in °C,
$T_o(lo)$ = low observed temperature in °C,
$T_s(hi)$ = high set temperature in °C, and
$T_s(lo)$ = low set temperature in °C.

are taken from measurements according to 10.4.

11.4.3 Fill in the table with observed values measured according to 10.4 and calculated offset values from 11.4.2.

11.4.4 The true temperature for a requested temperature is determined by interpolation of the adjacent temperature points in the calibration working table and Eq 3 where the value of b is the offset for the corresponding value of T_s.

NOTE 9—Alternatively, the results of the calibration working table may be plotted with T_o on the ordinate (Y-axis) and T_s on the abscissa (X-axis). Moreover, the data may be fitted by a polynomial, cubic spline, or other mathematical curve fitting technique to obtain a calibration working equation. This equation may be used to determine the true temperature from an observed temperature measurement.

12. Report

12.1 Report the following information:

12.1.1 Description of the instrument (manufacturer and model number) as well as the data-handling device used in the test.

12.1.2 Description of the dimension, geometry, and material of the dummy test specimen.

12.1.3 *Method of Calibration*—single-point, two-point, or multi-point temperature calibration.

12.1.4 For the single-point temperature calibration, the temperature of calibration and the value for the bias (b).

12.1.5 For the two-point temperature calibration, the high and low calibration temperatures (known as the calibration temperature range) , and the values of calibration slope (S) and intercept (I).

12.1.6 For the multi-point temperature calibration, the high and low calibration temperatures (known as the calibration temperature range) and the calibration working table.

12.1.7 The specific dated version of this test method used.

13. Precision and Bias

13.1 The precision and bias of this test method will be determined in an interlaboratory test program schedule for 2015–2020. Anyone wishing to participate in the interlaboratory test should contact the E37 Staff Manager at ASTM International Headquarters.

13.2 A limited interlaboratory test was conducted in 2007 involving two laboratories and six replicate determinations. The within-laboratory repeatability standard deviation was 0.16°C and the between-laboratory reproducibility standard deviation was 0.38°C. The mean bias was found to be 0.28°C.

14. Keywords

14.1 calibration; rheometer; temperature; thermal analysis

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