Designation: E2918 – 18

Standard Test Method for Performance Validation of Thermomechanical Analyzers[1]

This standard is issued under the fixed designation E2918; 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 provides procedures for validating temperature and length change measurements of thermomechanical analyzers (TMA) and analytical methods based upon the measurement of temperature and length change. Performance parameters include temperature repeatability, linearity and bias; and dimension change repeatability, detection limit, quantitation limit, linearity and bias.

1.2 Validation of apparatus performance and analytical methods is a necessary requirement for quality initiatives. Results may also be used for legal purposes.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *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

E1142 Terminology Relating to Thermophysical Properties

E1363 Test Method for Temperature Calibration of Thermomechanical Analyzers

E1970 Practice for Statistical Treatment of Thermoanalytical Data

E2113 Test Method for Length Change Calibration of Thermomechanical Analyzers

E2161 Terminology Relating to Performance Validation in Thermal Analysis and Rheology

3. Terminology

3.1 Technical terms used in this test method are defined in Terminologies E473, E1142, and E2161, including terms *analyte, bow, Celsius, coefficient of linear thermal expansion, detection limit, linearity, quantification limit, relative standard deviation, repeatability, standard deviation, thermodilatometry, thermomechanical analysis,* and *validation.*

4. Summary of Test Method

4.1 Temperature and time are the primary independent parameters and length change is the primary dependent experimental parameters provided by thermomechanical analysis.

4.2 Temperature, a measured value, is validated by performing a measurement of the penetration in sharply melting materials at three (or more) different known melting temperatures.

4.3 Length change, a measured value, is validated by performing a measurement of the linear thermal expansion for three (or more) test materials.

4.4 Validation of a thermomechanical test method based upon length change may be performed using the test specimen as the analyte.

4.5 The length change of three (or more) specimens (nominally representing the maximum, midpoint and minimum of the range of the test method) are measured in triplicate (or more). A fourth blank specimen, containing no analyte, is measured in triplicate (or more).

NOTE 1—Repeatability is determined by performing a sufficient number of determinations to calculate valid estimates of the standard deviation or relative standard deviation of the measurement.

4.5.1 Temperature and length change measurement linearity and bias are determined from the linear regression correlation of the results from measurements of the three (or more) specimens.

[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.

4.5.2 Length change detection limit and quantitation limit are determined from the standard deviation of the blank determination with no analyte present.

4.5.3 Temperature and length change repeatability are determined from the repeatability measurements of three (or more) specimens.

4.5.4 Length change validation is independent of the temperature validation. The respective validations need not involve consistent ranges.

5. Significance and Use

5.1 This test method may be used to determine and validate the performance of a particular thermomechanical analyzer apparatus.

5.2 This test method may be used to determine and validate the performance of a particular method based upon thermomechanical analyzer temperature or length change measurements.

5.3 This test method may be used to determine the repeatability of a particular apparatus, operator, or laboratory.

5.4 This test method may be used for specification and regulatory compliance purposes.

6. Apparatus

6.1 *Thermomechanical Analyzer (TMA)*—The essential instrumentation required to provide the minimum thermomechanical analytical or thermodilatometric capability for this test method include:

6.1.1 A rigid *specimen holder* of an inert, low expansivity material (< 0.6 µm/m °C) to center the specimen in a furnace and to fix the specimen to mechanical ground.

NOTE 2—Apparatus capable of higher temperature operation may be constructed of materials with greater expansivity. Additionally, a correction for expansion of the material of construction is included in dimensional change measurements.

6.1.2 A rigid *expansion probe* of inert low expansivity material (<0.6 µm/m K) that contacts the specimen with an applied compressive force (see Note 2). The circular area in contact with the test specimen shall have a diameter between 0.5 mm and 1.1 mm.

NOTE 3—Expansion probes of other diameters may be used but shall be reported.

6.1.3 A *sensing element*, linear over a minimum range of 2 mm, to measure the displacement of the rigid expansion probe with a minimum resolution of ±50 nm due to resultant changes in length of the specimen.

6.1.4 A *force transducer or weight* to generate a constant force of 1.0 mN to 100 mN (0.1 g to 10 g) ± 2.5 % that is applied through the rigid expansion probe to the specimen.

6.1.5 A *furnace* to provide uniform and controlled heating or cooling of a specimen to a constant temperature or at a constant rate within the applicable temperature range of this method.

6.1.6 A *temperature controller* capable of executing a specific temperature program by operating the furnace between selected temperature limits at a constant rate of temperature change between 2 °C/min and 10 °C/min (or greater) to within ±0.1 °C/min or at an isothermal temperature constant to ±0.1 °C.

6.1.7 A *temperature sensor* to provide an indication of the specimen/furnace temperature over the range from 20 °C to 300 °C (or greater) readable to ±0.1 °C.

NOTE 4—This temperature range is the minimum required to perform this validation. Many thermomechanical analyzers are applicable to a broader temperature range.

6.1.8 A means of sustaining an environment around the specimen of a dry, inert gas at a purge rate of 10 mL/min to 50 mL/min ± 5 mL/min.

NOTE 5—Typically 99+ % pure nitrogen, argon or helium is employed when oxidation in air is a concern. Unless effects of moisture are to be studied, use of dry purge gas is recommended and is essential for operation at subambient temperatures.

6.1.9 A *data collection device*, to provide a mean of acquiring, storing and displaying measured or calculated signals or both. The minimum output signals required for thermomechanical analysis are change in linear dimension (length), temperature, and time.

NOTE 6—A data acquisition rate of equal to or greater than 1 data point per second is required to achieve the desired measurement precision.

6.1.10 Auxiliary instrumentation considered useful (but not essential) in conducting this method includes:

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

6.1.10.2 Specimen *containers*, stable and inert to the temperature of interest to protect the specimen holder from the test specimen melt. Such containers are typically constructed of the same material as the specimen holder and expansion probe.

6.2 A *micrometer* or other length measuring device to determine specimen dimension of up to 10 mm with an accuracy of ±25 µm.

7. Reagents and Materials

7.1 *Indium (In),* 99.99+ % purity, preferably a certified reference material for which the melting temperature is known.

7.2 *Bismuth (Bi),* 99.99+ % purity, preferably a certified reference material for which the melting temperature is known.

7.3 *Zinc (Zn),* 99.99+ % purity, preferably a certified reference material for which the melting temperature is known.

7.4 *Tungsten (W),* 99.9+ % pure, a right circular cylinder, 6.0 mm to 6.5 mm in diameter, 7 mm to 9 mm in length having flat and parallel ends to within ±25 µm.

7.5 *Lead (Pb),* 99.9 + pure, a right circular cylinder, 6.0 mm to 6.5 mm in diameter and 7 mm to 9 mm in length having flat and parallel ends to within ±25 µm.

7.6 *Copper (Cu),* 99.9+ % pure, a right circular cylinder, 6.0 mm to 6.5 mm in diameter and 7 mm to 9 mm in length having flat and parallel ends to within ±25 µm.

8. Calibration and Standardization

8.1 Turn on the power and allow the instrument to equilibrate for at least one hour prior to any measurements.

8.2 Perform any cleaning and calibration procedures described by the manufacturer in the apparatus operator's manual.

8.3 Perform temperature and length change calibration according to Test Methods E1363 and E2113, respectively, using the same purge gas, purge gas flow rate, and heating rate (here 5 °C/min) to be used for validation experiments.

NOTE 7—The position of the temperature sensor is critical and shall not be changed during the course of this procedure.

9. Procedure for Determining Temperature Repeatability, Linearity, and Bias

9.1 This process involves characterizing, three (or more) test specimens taken to represent the high, medium and low portions of the temperature range over which performance is to be validated (see Table 1).

NOTE 8—The details of this procedure are written using zinc, bismuth and indium as analytes with their nominal melting temperatures at 420 °C (high), 271 °C (medium), and 157 °C (low). Other materials, such as those indicated in Table 1, with melting temperatures approximately equidistant on the temperature scale may be used but shall be reported.

9.2 Prepare three (or more) high melting (zinc), minimum melting (bismuth), and low melting (indium) test specimens weighing between 10 mg and 15 mg.

NOTE 9—The specimen should have a smooth surface on both top and bottom. Avoid the use of specimens with sharp ridges and irregular surfaces. These may lead to false values for the onset temperature.

9.3 Place the largest zinc specimen on the specimen holder.

NOTE 10—The test specimen may be placed in a specimen container on the specimen holder to protect the specimen holder from the melted test specimen.

9.4 Move the furnace to enclose the specimen holder so that the specimen is centered in the uniform temperature zone.

9.5 Place the expansion probe in contact with the test specimen and apply a load of 50 mN (5 g) ± 2.5 %.

9.6 Purge the sample chamber with inert purge gas at a rate of 10 mL/min to 50 mL/min constant to within ±5 mL/min.

TABLE 1 Recommended Melting Temperature Metals Used in Thermoanalytical Methods[A]

Material	Melting Temperature (°C)
Gallium[B]	29.7666
Indium[B]	156.5936
Tin[C]	231.928
Bismuth[B]	271.402
Lead[B]	327.462
Zinc[C]	419.527
Aluminum[C]	660.323
Silver[C]	961.78
Gold[C]	1064.18

[A] The values in Table 1 were determined using special very high purity materials, and highly accurate steady state conditions that are not attainable or applicable to thermal analysis techniques.
[B] Bedford, R.E., Bonnier, G., Maas, H., and Pavese, F., "Recommended Values of Temperature on the International Temperature Scale of 1990 for a Selected Set of Secondary Reference Points," *Metrologia*, Vol 33, 1996, pp. 133–154.
[C] Mangum, B. W., "Special Report on the International Temperature Scale of 1990," *Journal of Research of the National Institute of Standards and Technology*, Vol 95, 1990, pp. 69–77.

NOTE 11—Use the same temperature sensor position, purge gas, and purge gas flow rate throughout all calibration and specimen testing experiments.

9.7 Heat (or cool) the test specimen to a temperature about 50 °C below the calibration melting temperature of the test specimen (see Table 1) and allow the apparatus to equilibrate for at least 1 min.

9.8 Heat the specimen at 5.0 °C/min through the melting transition until the probe reaches a point of maximum penetration after the transition. Record the thermal curve (see Fig. 1).

NOTE 12—Other heating rates may be used but shall be reported. Analytical performance may be affected by heating rate, purge gas and purge gas flow rate. Slower heating rates increase precision.

NOTE 13—Validation is limited to the heating rate, purge gas, purge gas flow rate, temperature range, and length change examined.

9.9 Cool the test specimen to ambient temperature. The thermal curve need not be recorded.

9.10 Prepare a thermal curve with dimension on the Y-axis and temperature on the X-axis (see Fig. 1). Determine the extrapolated onset temperature and report as T(Zn)1.

9.10.1 Extrapolate the baseline before the transition into the transition region.

9.10.2 Construct a tangent to the curve at the steepest slope of the penetration region.

9.10.3 Determine the temperature corresponding to the intersection of the lines constructed in steps 9.10.1 and 9.10.2.

NOTE 14—Retain all available digits.

9.11 Repeat steps 9.3 – 9.10 for the largest medium melting temperature (bismuth) specimen. Record the temperature as T(Bi)1.

NOTE 15—Loading and unloading of the specimen is required to determine analytical repeatability.

9.12 Repeat steps 9.3 – 9.10 for the largest low melting temperature (indium) specimen. Record the temperature as T(In)1.

9.13 Repeat steps 9.3 – 9.10 for each of the two remaining high melting temperature (zinc) specimens (see Note 10 and Note 15). Record these values as T(Zn)2 and T(Zn)3.

9.14 Repeat steps 9.3 – 9.10 for each of the two remaining medium melting temperature (bismuth) specimens (see Note 10 and Note 15). Record these values as T(Bi)2 and T(Bi)3.

9.15 Repeat steps 9.3 – 9.10 for each of the remaining low melting temperature specimens (see Note 10 and Note 15). Record these values as T(In)2 and T(In)3.

9.16 Using the three (or more) values from steps 9.10 and 9.13, calculate the mean high melting temperature (T(Zn)) and standard deviation (s(Zn)) for the highest melting temperature measurements.

NOTE 16—See Practice E1970 for the determination of mean and standard deviation.

9.17 Using the three (or more) values from steps 9.11 and 9.14, calculate the mean melting temperature (T(Bi)), and standard deviation (s(Bi)) for the medium melting temperature measurements.