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Standard Test Method for Performance Validation of Thermomechanical Analyzers¹

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

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.

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

E3142 Test Method for Thermal Lag of Thermal Analysis Apparatus

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.

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

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.

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 ($< 0.6 \mu\text{m/m } ^\circ\text{C}$) 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 ($< 0.6 \mu\text{m/m K}$) 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 $\pm 50 \text{ nm}$ 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) $\pm 2.5 \%$ 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 $2 ^\circ\text{C/min}$ and $10 ^\circ\text{C/min}$ (or greater) to within $\pm 0.1 ^\circ\text{C/min}$ or at an isothermal temperature constant to $\pm 0.1 ^\circ\text{C}$.

6.1.7 A *temperature sensor* to provide an indication of the specimen/furnace temperature over the range from $20 ^\circ\text{C}$ to $300 ^\circ\text{C}$ (or greater) readable to $\pm 0.1 ^\circ\text{C}$.

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 $50 \text{ mL/min} \pm 5 \text{ mL/min}$.

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 $\pm 25 \mu\text{m}$.

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 $\pm 25 \mu\text{m}$.

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 $\pm 25 \mu\text{m}$.

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 $\pm 25 \mu\text{m}$.