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Standard Test Method for Linear Thermal Expansion of Solid Materials With a Push-Rod Dilatometer[1]

This standard is issued under the fixed designation E228; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the determination of the linear thermal expansion of rigid solid materials using push-rod dilatometers. This method is applicable over any practical temperature range where a device can be constructed to satisfy the performance requirements set forth in this standard.

NOTE 1—Initially, this method was developed for vitreous silica dilatometers operating over a temperature range of –180 to 900°C. The concepts and principles have been amply documented in the literature to be equally applicable for operating at higher temperatures. The precision and bias of these systems is believed to be of the same order as that for silica systems up to 900°C. However, their precision and bias have not yet been established over the relevant total range of temperature due to the lack of well-characterized reference materials and the need for interlaboratory comparisons.

1.2 For this purpose, a rigid solid is defined as a material that, at test temperature and under the stresses imposed by instrumentation, has a negligible creep or elastic strain rate, or both, thus insignificantly affecting the precision of thermal-length change measurements. This includes, as examples, metals, ceramics, refractories, glasses, rocks and minerals, graphites, plastics, cements, cured mortars, woods, and a variety of composites.

1.3 The precision of this comparative test method is higher than that of other push-rod dilatometry techniques (for example, Test Method D696) and thermomechanical analysis (for example, Test Method E831) but is significantly lower than that of absolute methods such as interferometry (for example, Test Method E289). It is generally applicable to materials having absolute linear expansion coefficients exceeding 0.5 μm/(m·°C) for a 1000°C range, and under special circumstances can be used for lower expansion materials when special precautions are used to ensure that the produced expansion of the specimen falls within the capabilities of the measuring system. In such cases, a sufficiently long specimen was found to meet the specification.

1.4 Computer- or electronic-based instrumentation, techniques, and data analysis systems may be used in conjunction with this test method, as long as it is established that such a system strictly adheres to the principles and computational schemes set forth in this method. Users of the test method are expressly advised that all such instruments or techniques may not be equivalent and may omit or deviate from the methodology described hereunder. It is the responsibility of the user to determine the necessary equivalency prior to use.

~~1.5SI units are the standard.~~

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 There is no ISO method equivalent to this standard.

1.7 *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]

D696 Test Method for Coefficient of Linear Thermal Expansion of Plastics Between 30C and 30C with a Vitreous Silica Dilatometer

E220 Test Method for Calibration of Thermocouples By Comparison Techniques

E289 Test Method for Linear Thermal Expansion of Rigid Solids with Interferometry

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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 E37.05 on Thermophysical 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.

E473 Terminology Relating to Thermal Analysis and Rheology
E644 Test Methods for Testing Industrial Resistance Thermometers
E831 Test Method for Linear Thermal Expansion of Solid Materials by Thermomechanical Analysis
E1142 Terminology Relating to Thermophysical Properties

3. Terminology

3.1 *Definitions*—The following terms are applicable to this test method and are listed in Terminologies E473 and E1142: *coefficient of linear thermal expansion*, *thermodilatometry*, and *thermomechanical analysis*.

3.2 *Symbols*:

α_m ~~=mean coefficient of linear thermal expansion, µm/(m·°C) or °C~~ = mean or average coefficient of linear thermal expansion over a temperature range, µm/(m·°C), K^{-1}, or $°C^{-1}$

α_T ~~=expansivity at temperature~~ = *expansivity or instantaneous coefficient of linear thermal expansion at temperature T*, ~~µm/(m·°C) or °C~~ µm/(m·°C), K^{-1}, or $°C^{-1}$

L_0 = original length of specimen at temperature T_0, mm
L_1 = length of specimen at temperature T_1, mm
L_2 = length of specimen at temperature T_2, mm
L_i = length of specimen at a particular temperature T_i, mm
ΔL = change in length of specimen between any two temperatures T_1 and T_2, T_0 and T_1, etc., µm
$(\Delta L/L_0)$ = expansion
T_0 = temperature at which initial length is L_0, °C
T_1, T_2 = two temperatures at which measurements are made, °C
T_i = temperature at which length is L_i, °C
ΔT = temperature difference between any two temperatures T_2 and T_1, T_1 and T_0, etc., °C
m = measured expansion of the reference material,
t = true or certified expansion of the reference material,
s = assumed or known expansion of the parts of the dilatometer,
A = numerical calibration constant

3.3 *Definitions of Terms Specific to This Standard:*

3.3.1 *linear thermal expansion,* $\Delta L/L_0$—the change in length relative to the initial length of the specimen accompanying a change in temperature, between temperatures T_0 and T_1, expressed as:

$$\frac{\Delta L}{L_0} = \frac{L_1 - L_0}{L_0} \tag{1}$$

3.3.1.1 *Discussion*—It is a dimensionless quantity, but for practical reasons the units most often used are µm/m, $(m/m)\cdot 10^{-6}$, $(in./in.)\cdot 10^{-6}$, ppm or percent (%).

3.3.2 *mean (average) coefficient of linear thermal expansion,* α_m—the ratio between the expansion and the temperature difference that is causing it. It is referred to as the average coefficient of thermal expansion for the temperature range between T_0 and T_1.

$$\alpha_m = \frac{1}{L_0}\frac{\Delta L}{\Delta T} \tag{2}$$

3.3.2.1 *Discussion*—Most commonly, it is expressed in µm/(m °C) or $°C^{-1}$, and it is determined for a sequence of temperature ranges, starting with 20°C by convention, being presented as a function of temperature. In case the reference temperature differs from 20°C, the specific temperature used for reference has to be indicated in the report.

3.3.3 *thermal expansivity (instantaneous coefficient of thermal expansion),* α_T—identical to the above, except that the derivative replaces the finite differences of Eq 2. The thermal expansivity is related to the length change for an infinitesimally narrow temperature range, at any temperature T (essentially a "tangent" point), and is defined as follows:

$$\alpha_T = \frac{1}{L_0}\left(\frac{dL}{dT}\right)_T \tag{3}$$

3.3.3.1 *Discussion*—It is expressed in the same units as the average coefficient of thermal expansion. In terms of physical meaning, the instantaneous coefficient of thermal expansion is the derivative of the expansion curve when plotted versus temperature, at the temperature T. It has a rather limited utility for engineering applications, and therefore it is more common to use the average coefficient of thermal expansion, than the instantaneous one.

3.3.4 *dilatometer*—a device that measures the difference in linear thermal expansion between a test specimen and its own parts adjacent to the sample.

3.3.4.1 *Discussion*—Thermomechanical analyzers (TMA), instruments used in thermal analysis, are often also characterized as dilatometers, due to their ability to determine linear thermal expansion characteristics. Typically, they employ specimens much smaller than dilatometers; however, TMA systems with sufficiently large specimen size capability have been shown to measure thermal expansion accurately. When using the small TMA specimen size, this utilization of TMA equipment should be limited to

testing only very high expansion materials, such as polymers, otherwise the data obtained may be substantially in error. Conversely, some dilatometers can perform some of the TMA functions, but the two devices should not be considered equivalent or interchangeable in all applications.

4. Summary of Test Method

4.1 This test method uses a single push-rod tube type dilatometer to determine the change in length of a solid material relative to that of the holder as a function of temperature. A special variation of the basic configuration known as a differential dilatometer employs dual push rods, where a reference specimen is kept in the second placement at all times and expansion of the unknown is determined relative to the reference material rather than to the specimen holder.

4.2 The temperature is controlled either over a series of steps or at a slow constant heating or cooling rate over the entire range.

4.3 The linear thermal expansion and the coefficients of linear thermal expansion are calculated from the recorded data.

5. Significance and Use

5.1 Coefficients of linear thermal expansion are required for design purposes and are used, for example, to determine dimensional behavior of structures subject to temperature changes, or thermal stresses that can occur and cause failure of a solid artifact composed of different materials when it is subjected to a temperature excursion.

5.2 This test method is a reliable method of determining the linear thermal expansion of solid materials.

5.3 For accurate determinations of thermal expansion, it is absolutely necessary that the dilatometer be calibrated by using a reference material that has a known and reproducible thermal expansion. The appendix contains information relating to reference materials in current general use.

5.4 The measurement of thermal expansion involves two parameters: change of length and change of temperature, both of them equally important. Neglecting proper and accurate temperature measurement will inevitably result in increased uncertainties in the final data.

5.5 The test method can be used for research, development, specification acceptance, quality control (QC) and quality assurance (QA).

6. Interferences

6.1 *Materials Considerations*:

6.1.1 The materials of construction may have substantial impact on the performance of the dilatometer. It is imperative that regardless of the materials used, steps be taken to ascertain that the expansion behavior is stabilized, so that repeated thermal cycling (within the operating range of the device) causes no measurable change.

6.2 *General Considerations*:

6.2.1 Inelastic creep of a specimen at elevated temperatures can often be prevented by making its cross section sufficiently large.

6.2.2 Avoid moisture in the dilatometer, especially when used at cryogenic temperatures.

6.2.3 Means to separate the bath from the specimen are required when the dilatometer is immersed in a liquid bath.

6.2.4 Support or hold the specimen in a position so that it is stable during the test without unduly restricting its free movement.

6.2.5 The specimen holder and push-rod shall be made from the same material. The user must not practice uncontrolled substitutions (such as when replacing broken parts), as serious increase of the uncertainties in the measured expansion may result.

6.2.6 A general verification of a dilatometer is a test run using a specimen cut from the same material as the push rod and specimen holder. The resultant mean coefficient of linear thermal expansion should be smaller than ±0.3 µm/(m·°C) for a properly constructed system (after applying the system's correction).

6.2.7 Conditioning of specimens is often necessary before reproducible expansion data can be obtained. For example, heat treatments are frequently necessary to eliminate certain effects (stress caused by machining, moisture, etc.) that may introduce irreversible length changes that are not associated with thermal expansion.

7. Apparatus

7.1 *Push-Rod Dilatometer System*, consisting of the following:

7.1.1 *Specimen Holder*—A structure of thermally stable material constructed in a fashion such that when a specimen of the same material is placed into it for a test, the qualifications given in 6.2.7 are satisfied. In any push rod dilatometer, ~~to satisfy this standard,~~ both the sample holder and the push-rod(s) shall be made of the same material, ~~and have~~having been proven to exhibit thermal expansion characteristics within ±1 % of each other. Illustrations of typical tube and rod-type configurations are given in Fig. 1. It is often practiced to configure specimen holders that are not shaped as a tube, but serve the same structural purpose. This is an acceptable practice, as long as the shape is mechanically stable and is not prone to reversible configurational changes (such as twisting, etc.) upon heating and cooling.

NOTE 2—The tube and the push-rod beyond the specimen, while parallel to each other, are expected to have identical thermal gradients along them, thereby identical thermal expansion. This is a critical factor, as differences in net expansion between the tube and the push-rod will appear very much like expansion produced by the specimen. To a limited extent, calibration (see Section 9) can be used to account for these differences in the thermal expansion of the two parts, however, it is noted that this is one of the most fundamental of all practical limitations for dilatometers. To minimize this effect, the tube and the push-rod shall be in close proximity of each other and heated slowly enough to prevent substantial thermal gradients that occur radially.

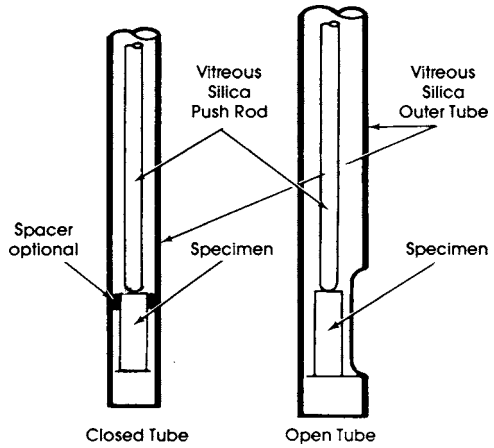

FIG. 1 Common Forms Specimen Holders

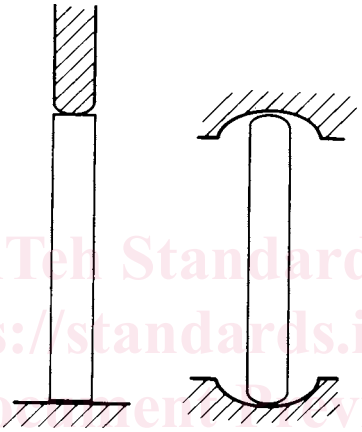

FIG. 2 Suggested Shapes of Specimen's and Push-Rod Ends

7.1.2 *Test Chamber*, composed of:

7.1.2.1 *Furnace, Cryostat, or Bath*, used for heating or cooling the specimen uniformly at a controlled rate over the temperature range of interest, and able to maintain the temperature uniform along the sample during its heating, cooling, or just equilibrating.

NOTE 3—Extreme care must be exercised in using furnaces for high temperatures, to prevent interaction with the dilatometer's parts or with the specimen. In many instances, it is necessary to protect the specimen and the dilatometer from oxidation and in some cases this may be accomplished with the use of a muffle tube. If it is necessary, the furnace, in such cases, shall contain provisions to provide inert atmosphere or vacuum environment, as well as provisions to protect against air back-streaming on cooling.

NOTE 4—Unless it is absolutely necessary to have the specimen tested in vacuum, measurements of thermal expansion in vacuum are not recommended due to extreme thermal gradients, thermal lags, etc. between various components of the dilatometer and the specimen, that are caused by the very poor heat transfer that occurs in the absence of a gas.

7.1.2.2 *Temperature Controller* (or circuitry with equivalent function) capable of executing a specific temperature program by operating the furnace(s) between selected temperature limits at rates programmed, and supported by the furnace(s)'s thermal performance. Temperature control of a constant rate shall be monotonous within ±2°C ~~monotonicity,~~ (exclusive of the approximately 5 % of instrument's maximum temperature of its operating range). The control of equilibrium temperatures shall be within ±1°C or ±0.05 % of maximum temperature of the dilatometer's operating range, whichever is larger.

7.1.2.3 Means to cool furnace(s) or cryostat for operation below ambient.

7.1.2.4 Means to seal the space occupied by the dilatometer, contain pressurized gas, or evacuated, without collapse or gradual deformation with time and temperature, or without bursting when pressure is applied within specified limits.

7.1.3 *Transducer*—A device to convert and magnify the minute mechanical translation conveyed by the push-rod(s) resulting from the expansion of the specimen, into visually discernable or electrically measurable signals, with a constant and defined functionality.

7.1.3.1 Visually readable devices, such as dial gauges, optical levers, rulers, etc.

7.1.3.2 Electromechanical devices with a defined electrical output corresponding to a defined mechanical displacement input. Examples of these are: linear variable differential transformers, digital absolute or incremental encoders, capacitive sensors, optical displacement sensors, etc.

7.1.3.3 The transducer must be selected such as to cover the expected displacement within its linear range.

7.1.3.4 Transducers employed in dilatometry must have a resolution (visible or sensible) of not less than 0.1 % of ~~its~~their linear range, with an attendant proven linearity of at least ±0.1 % of ~~its~~their range.

7.1.3.5 The linearity band of a transducer limits the maximum resolution that can be assigned to the dilatometer. (Since the band bound by linearity is considered unresolvable, even with special calibrations, resolution cannot be ensured much beyond the transducer's range of linearity.) Adding empty amplification (extra digits to the readout), may give the impression of more sensitivity, but in reality it does not. Nonlinearity errors cannot be effectively accounted for with calibration.

7.1.4 *Temperature Measurement System*, consisting of:

7.1.4.1 *Calibrated Sensor or Sensors*, to provide indication of the specimen temperature ±0.5°C, or ±1 % of the overall temperature range, whichever is larger. Temperature sensors may be, but are not limited to, thermocouples, pyrometers, resistance thermometers, thermistors, mercury thermometers, etc.

7.1.4.2 *Manual, Electronic, or Equivalent Readout*, such that the indicated temperature can be determined without additional degradation to the sensor's performance.

NOTE 5—In all cases in which thermocouples are used, they shall be referenced to 0°C by means of an ice water bath or equivalent electronic reference.

NOTE 6—Special attention must be paid to prevent contamination of the thermocouple by the specimen or even by the dilatometer tube itself (for example, type C thermocouple in a graphitic environment). Interaction between atmosphere and the thermocouple (for example, type S thermocouple in hydrogen atmosphere) can also be extremely detrimental.

NOTE 7—Placement of thermocouples is important, and the user is frequently given a choice. It is often a practice to bring the bead of a thermocouple in contact with the specimen, or even embed it in a hole, as opposed to having it laying on top of it or in its proximity. While the former seems better, it is actually sometimes the cause of mechanical interference with the specimen, source of contamination, and while it registers a more true specimen temperature, it neglects the temperature of the specimen holder around it. A good practice is to have the bead of the thermocouple reside equidistant between the specimen holder and the specimen itself, ensuring that it is shielded from direct view of the heater or muffle tube (if one is employed).

NOTE 8—The thermocouple positioning during the test should be the same as was used in the instrument's calibration. Frequent verification of thermocouple performance is highly recommended.

7.2 *Measuring Tool*, such as a vernier micrometer or calipers capable of reading to at least ±25 μm in order to determine the initial and final lengths of the test specimen.

8. Test Specimens

8.1 The specimen length shall be such that the accuracy of determining $\Delta L/L_0$ is at least ±20 μm/m. Where possible, the specimen should be between 25 and 60 mm long and between 5 and 10 mm in diameter (or equivalent, if not cylindrical), however, there is no fundamental limitation on either dimension, as long as the dilatometer can accommodate the specimen with a maximum thermal gradient of 2°C, either determined in 50 mm intervals over the entire specimen, or maintained uniformly within ±2°C/50 mm.

8.2 The cross sectional shape of the specimen, the cross sectional uniformity along its length, or the condition of the surface along its length, have no bearing on the test. The ends, however, must be smooth and parallel. The cross section must be robust enough to prevent buckling or creep.

8.2.1 In the instances when limitations of the source material precludes the forming of a cylinder or slab, irregular shaped samples may be tested. However, care must be exercised not to have the sample form a point contact with the dilatometer, as it may lead to deformations during the test.

8.2.2 Thin sheets of materials can be formed into a specimen by ~~rolling them~~being rolled into a ~~tube,~~tube or ~~bending~~bent into a "V" shaped piece for increased stiffness.

8.2.3 Smaller pieces may be stacked to obtain a longer specimen, often without any cementing, as long as all interfaces are kept flat and parallel, and no wobble is observable when the push-rod is raised and lowered to contact it.

8.2.4 Hollow (tube) or irregular shaped specimens often require the use of a cover plate between them and the push-rod for well defined contact. Such plate is preferred to be made out of the same material as the dilatometer, to neglect its contribution in the calculations. Alternately, a very thin sheet of a stiff material, other than the dilatometer's, may be used, but with proper accounting for its contribution.

8.2.5 Specimens of semi-rigid materials that would deform when in contact with the tip of the push-rod can be successfully tested according to 8.2.4, aimed at reducing the pressure on the specimen (counterweights, etc.), provided the material is able to maintain its shape otherwise. This is an extension of the method and care must be exercised when practiced.

9. Calibration

9.1 Calibrate the transducer by imposing a series of known displacements with a precision screw micrometer, gauge blocks, or any other device that is more accurate than the transducer that is being calibrated. This step may be omitted for absolute transducers.

NOTE 9—Any transducer system, however, including digital encoders, shall be calibrated or verified prior to use for thermal expansion measurements, to ensure proper performance. The frequency of calibration or verification necessary would need to be established by the user of the dilatometer.

9.2 Calibrate the temperature sensor according to Test Methods E220 and E644, the procedure recommended by the National

Institute of Standards and Technology (NIST) **(1)**[3] or the appropriate procedure applicable by the maker of the device, if other procedures are not applicable.

NOTE 10—Periodic cross-checking of optical pyrometers with temporarily inserted thermocouples of the proper type and protection is recommended whenever possible. Alternative means, such as melting or eutectic point sensing, is also acceptable for elevated temperatures.

9.3 As a total system, calibrate the dilatometer by measuring one reference material of known thermal expansion. Select a reference material with expansion close to that of the specimen material.

9.4 Calibrate the dilatometer using the same test conditions and procedures as those used for the test specimen, for example the same specimen length, temperature program, and gaseous environment (including flow rate).

NOTE 11—As closely identical thermal cycles as possible should be used for the test and the calibration. Deviation from identical thermal cycles will inevitably generate some error. The magnitude of this error can be estimated by performing a test on the same reference material used for the instrument's calibration.

9.4.1 General guidelines that shall be followed for calibration:

9.4.1.1 Use only a calibration function generated with a constant temperature ramp for tests to be performed at the same constant ramp.

9.4.1.2 Use only a calibration function generated using stepwise heating (equilibrium points) for tests composed of equilibrium steps.

9.4.1.3 Vacuum and purge gas flow and composition may have substantial effect on thermal lags, therefore, always use the same atmosphere conditions for both test and calibration.

9.4.1.4 Observe the direction of progress as to heating or cooling, as they cause different thermal lags. Using a calibration function obtained on heating for a test involving cooling will inevitably bring in differences.

9.4.1.5 Reliance on statistically averaged multiple (replicate) calibration runs is recommended for each heating schedule.

9.4.2 Because a dilatometer always indicates the difference in expansion between the specimen and the portion of the tube that is positioned parallel to it, a calibration constant, A_{Ti} (defined in Eq 4), has to be determined at each temperature interval between T_i and T_0, with $T_0 < T_i < T$. The calibration constants determined through Eq 4 can be used as either point-by-point corrections at discrete temperatures, or can be converted into a function and used to correct data expressed as expansion versus temperature.

$$A_{Ti} = \left[\left(\frac{\Delta L}{L_0}\right)_t - \left(\frac{\Delta L}{L_0}\right)_m\right]_{Ti} \tag{4}$$

10. Procedure

10.1 Measure the initial length of the specimen at room temperature, as L_0.

10.2 Place the specimen in the dilatometer after making certain that all contacting surfaces are free of foreign material. Ensure good seating of the specimen in a stable position.

10.3 Ensure that the push-rod is in stable contact with the specimen.

10.4 Insert the assembled dilatometer into the furnace, cryostat, or bath, and allow the temperature of the specimen to reach equilibrium with the environment, at room temperature.

10.5 Record the initial reading of the temperature sensor, T_0, and zero the transducer.

10.6 Select the heating schedule that best suits the application for the material being tested.

10.6.1 The most precise measurement is achieved by heating (or cooling) the specimen successively to a number of incremental constant temperatures and allowing the system to equilibrate until the transducer reading attains a constant value (variation less than ±2 µm). At that point, the indicated temperature of the specimen shall not vary by more than ±2°C, and the temperature gradient in the specimen shall not exceed 0.5°C/cm. The hold time is a function of the total thermal mass of the dilatometer and specimen, and will vary for different temperatures and different instruments. Readings of temperature T_i and changed specimen length L_i are recorded at each constant temperature T_i after full equilibration.

10.6.2 Alternatively, heat or cool at a constant rate equal to or less than 5°C/min. When using this procedure, the mean temperature of the specimen will probably differ from the measured temperature (lower on heating and higher on cooling), but the measured expansion of the test specimen will be accurate if the system is calibrated correctly with a reference material. Readings of temperature and change of length should be recorded continuously or in frequent time intervals.

NOTE 12—Large specimens or materials with low thermal diffusivity will take much longer to reach a near-uniform internal temperature distribution. The extent to which radial gradients exist is determined by the characteristics of the specimen. Users must select heating rates and/or equilibration (soak) times prudently, to ensure that no lag occurs and that data truly represents that for the whole specimen at a uniform temperature. A good practice is to test large samples in stepwise fashion and monitor the approach to a constant value of the expansion as an indication of thermal equilibrium.

10.7 A retest should be considered if the length of the specimen at the end of the test differs from that at the beginning by more than 20 µm/m. Alternately, this permanent deformation should be taken into account when reporting the expansion values. This retest, however, is not relevant for tests involving sintering, softening point determinations, etc., where permanent deformation is part of the expected behavior.

[3] The boldface numbers in parentheses refer to the list of references at the end of this standard.