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.

Designation: E228 – 22

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 U.S. 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 °C 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 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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.*

1.6 *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]

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

E220 Test Method for Calibration of Thermocouples By Comparison Techniques

E230/E230M Specification for Temperature-Electromotive Force (emf) Tables for Standardized Thermocouples

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

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:

[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.

Current edition approved Dec. 1, 2022. Published January 2023. Originally approved in 1963. Last previous edition approved in 2017 as E228 – 17. DOI: 10.1520/E0228-22.

[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.

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coefficient of linear thermal expansion, *thermodilatometry*, and *thermomechanical analysis*.

3.2 *Definitions of Terms Specific to This Standard:*

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

3.2.1.1 *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.

3.2.2 *linear thermal expansion, $\Delta L/L_0$, n*—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:

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

3.2.2.1 *Discussion*—It is a dimensionless quantity, but for practical reasons the units most often used are µm/m.

3.2.3 *mean (average) coefficient of linear thermal expansion, α_m, n*—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.

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

3.2.3.1 *Discussion*—Most commonly, it is expressed in µm/(m °C), 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.

3.2.4 *thermal expansivity (instantaneous coefficient of thermal expansion), α_T, n*—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:

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

3.2.4.1 *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 *Symbols:*

α_m = mean or average coefficient of linear thermal expansion over a temperature range, µm/(m·°C)
α_T = expansivity or instantaneous coefficient of linear thermal expansion at temperature T, µm/(m·°C)
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

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, both the sample holder and the push-rod(s) shall be made of the same material, 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

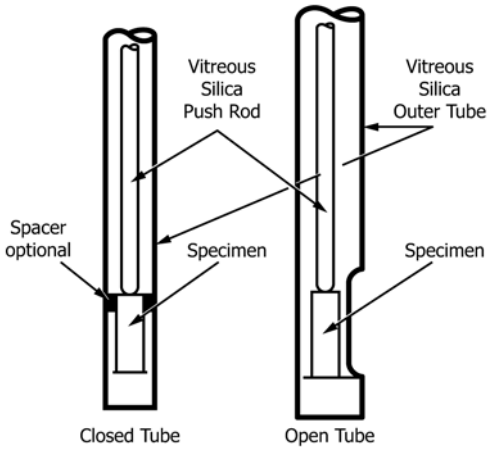

FIG. 1 Common Forms Specimen Holders

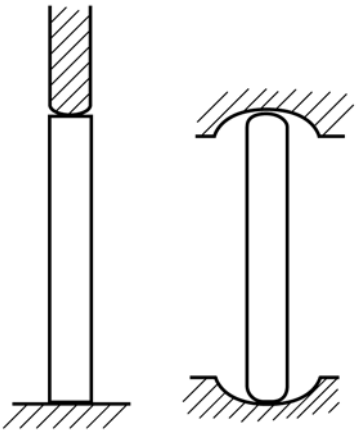

FIG. 2 Suggested Shapes of Specimen's and Push-Rod Ends

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.

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 backstreaming on cooling.