Designation: E 228 – 95

Standard Test Method for Linear Thermal Expansion of Solid Materials With a Vitreous Silica Dilatometer[1]

This standard is issued under the fixed designation E 228; 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 covers the determination of the linear thermal expansion of rigid solid materials over the temperature range of −180 to 900°C using vitreous silica push-rod or tube dilatometers.

NOTE 1—The temperature range for push-rod dilatometers can be extended to 1600°C by using high-purity alumina push-rod systems and up to over 2500°C using isotropic graphite systems. 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, regarding significantly affecting the precision of thermal-length change measurements. This includes metals, ceramics, refractories, glasses, rocks and minerals, graphites, plastics, cements, mortars, woods, and fiber, and other reinforced matrix composites.

1.3 Many materials and certain material applications require that detailed preconditioning and specific thermal test schedules be followed for the correct evaluation of thermal expansion. Since a general test method cannot cover all specific requirements, details of this nature should be contained in the relevant material specification.

1.4 The precision of this comparative test method is greater than that of other push-rod dilatometery (for example, Test Method D 696) and thermomechanical analysis (for example, Test Method E 831) techniques but is significantly lower than that of absolute methods such as interferometry (for example, Test Method E 289). It is generally applicable to materials having linear expansion coefficients above 5 μm/m·K and can be used for lower expansion coefficient materials for which a sufficient length of specimen is available.

1.5 Computer- or electronic-based instrumentation, techniques, and data analysis systems equivalent to this test method can be used. Users of the test method are expressly advised that all such instruments or techniques may not be equivalent. It is the responsibility of the user to determine the necessary equivalency prior to use. In the case of dispute only, the manual procedures described herein are to be considered valid.

1.6 The values stated in SI units are to be regarded as the 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:*

D 696 Test Method for Coefficient of Linear Thermal Expansion of Plastics[2]

E 220 Test Method for Calibration of Thermocouples by Comparison Techniques[3]

E 289 Test Method for Linear Thermal Expansion of Rigid Solids with Interferometry[4]

E 473 Terminology Relating to Thermal Analysis[4]

E 644 Test Methods for Industrial Resistance Thermometers[3]

E 831 Test Method for Linear Thermal Expansion of Solid Materials by Thermomechanical Analysis[4]

E 1142 Terminology Relating to Thermophysical Properties[4]

3. Terminology

3.1 *Definitions*—The following terms are applicable to this test method and are listed in Terminologies E 473 and E 1142: *coefficient of linear thermal expansion, thermodilatometry, and thermomechanical analysis*.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *mean coefficient of linear thermal expansion,* α_m—the average change in length relative to the length of the specimen accompanying a change in temperature, between temperatures T_1 and T_2 , expressed as follows:

[1] This test method is under the jurisdiction of ASTM Committee E-37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.05 on Thermophysical Properties.

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[2] *Annual Book of ASTM Standards*, Vol 08.01.

[3] *Annual Book of ASTM Standards*, Vol 14.03.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

$$\alpha_m = [(L_2 - L_1)/L_0\,(T_2 - T_1)] = (\Delta L/L_0)/\Delta T \quad (1)$$

α_m is, therefore, obtained by dividing the linear thermal expansion ($\Delta L/L_0$) by the change of temperature (ΔT). It is normally expressed as µm/m·K.

3.2.2 *thermal expansivity,* α_T—at temperature T, this is calculated as follows:

$$\alpha_T = \frac{1}{L_i}\,\underset{T_2 \to T}{limit}\,\frac{(L_2 - L_1)}{(T_2 - T_1)} = (dL/d_T)/L_i\,(T_1 < T_1 < T_2) \quad (2)$$

and expressed as µm/m·K.

NOTE 2—Thermal expansivity is sometimes referred to as the instantaneous coefficient of linear expansion.

3.2.3 *vitreous silica dilatometer*—a device that measures the difference in linear thermal expansion between a test specimen and the vitreous silica parts of the dilatometer.

3.3 *Symbols:Symbols:*

α_m	=	mean coefficient of linear thermal expansion, (see 3.2.1), µm/m·K
α_T	=	expansivity at temperature T (see 3.2.2), µm/m·K
L_0	=	original length of specimen at temperature T_0, mm
L_1	=	length at temperature T_1, mm
L_2	=	length at temperature T_2, mm
L_i	=	length at a particular temperature T_i, mm
ΔL	=	change in length of specimen between temperature T_1 and T_2, µm
$(\Delta L/L_0)_a$	=	expansion indicated by the measurement transducer, µm
T_0	=	temperature at which initial length is L_0, °K
T_1, T_2	=	two temperatures at which measurements are made, °K
T_i	=	temperature at which length is T_i, °K
ΔT	=	temperature difference between T_2 and T_1, °K
m	=	measured expansion of the reference material, µm
t	=	true or certified expansion of the reference material, µm
s	=	assumed or known expansion of the vitreous silica parts of the dilatometer, µm
A,B	=	numerical constants (see 9.3.1).

4. Summary of Test Method

4.1 This test method uses a vitreous silica dilatometer of the single push-rod or tube type 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 is sometimes used. This form is described briefly in Appendix X1.

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 expansion are required for design purposes and are used, for example, to determine thermal stresses that can occur and cause failure of a solid artifact composed of different materials when it is subjected to a temperature excursion(s).

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 vitreous silica dilatometer be calibrated by using a reference material that has a known reproducible thermal expansion. Table 1 and Table 2 contain information on the current thermal expansion standard reference materials available for such purposes. Table 3 contains information relating to other reference materials in current general use.

5.4 The measurement of thermal expansion involves two parameters: change of length and change of temperature. Since measurements of the first parameter can be made by this test method with good precision, it is essential that great attention also be paid to the second. In order to ensure the necessary uniformity in temperature of the specimen, it is essential that the uniform temperature zone of the surrounding furnace or environmental chamber be made significantly longer than the length of the specimen. Temperature gradients depend on the length/diameter ratio and insulation quality of the furnace. They can be reduced by adjusting the furnace heater windings such that they are closer at the ends than at the center. In addition, for high-temperature operations, it is recommended that a heavy metal liner and radiation shields be used. Temperature control is best attained when the sensing element of the controller is either the same as that for measuring the specimen temperature (radiant heating furnace) or very close to the heating element (resistive heated furnace).

5.5 This test method contains essential details of the design principles, specimen configurations, and procedures for providing precise values of thermal expansion. It is not practical in a test method of this type to try to establish specific details of design, construction, and procedures to cover all contingencies that might present difficulties to an individual not having the technical knowledge relating to the thermal measurements and general testing practice. Standardization of the test method is not intended to restrict further development of improved methodology in any way.

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

6. Interferences

6.1 Heating unannealed vitreous silica above 800°C may cause viscous flow and a time-dependent change in its thermal expansion. The magnitude of these effects will depend on the particular type of vitreous silica.

6.1.1 *Recommended Annealing Procedure*—Annealing the vitreous silica specimen and dilatometer parts in the following steps may reduce any excessive lot-to-lot differences in linear thermal expansion:

6.1.1.1 Heat at 100°C/h to 1200°C,

6.1.1.2 Hold at 1200°C for 2 h,

6.1.1.3 Cool at 60°C/h to 1000°C,

6.1.1.4 Cool at 120°C/h to 600°C, and

6.1.1.5 Cool at 200°C/h to room temperature.

TABLE 1 Thermal Expansion of Various Standard Reference Materials

Temperature,° K	SRM 731 (Borosilicate Glass)[A]		SRM 738 (Stainless Steel)[A]		SRM 739 (Fused Silica)[A]	
	Expansion, 10^6 $\Delta L/L_{293}$	10^6 Expansivity	Expansion, 10^6 $\Delta L/L_{292}$	10^6 Expansivity	Expansion, 10^6 $\Delta L/L_{293}$	10^6 Expansivity
80	–819	...	...	...	–1	–0.07
100	–771	2.64	...	...	–13	–0.53
120	–714	3.07	...	...	–22.5	–0.38
140	–649	3.43	...	...	–28.5	–0.24
160	–578	3.72	...	...	–32	–0.10
180	–501	3.97	...	...	–32.5	0.02
200	–419	4.17	...	...	–31	0.13
220	–334	4.34	...	...	–27.5	0.23
240	–246	4.48	...	...	–22	0.32
260	–155	4.60	...	...	–14	0.39
280	–62	4.71	...	...	–6	0.45
293	0	4.78	0	9.76	0	0.48
300	34	4.82	69	9.81	...	...
320	131	4.91	...	...	13.5	0.53
340	230	4.99	466	10.04	24.5	0.56
380	432	5.11	872	10.28	47.5	0.60
420	638	5.19	1288	10.52	72	0.62
460	847	5.23	1714	10.76	97	0.63
500	1057	5.26	2149	11.00	122	0.63
540	1267	5.27	2593	11.23	...	...
560	1372	5.27	...	...	159	0.61
580	1478	5.27	3048	11.47	...	...
600	1583	5.27	...	...	183	0.59
620	1689	5.28	3511	11.71	...	...
640	1794	5.29	...	...	206	0.56
660	1900	...	3984	11.95	...	...
680	2007	...	...	...	228	0.54
700	...	...	4467	12.19	...	...
720	...	...	...	...	249	0.51
740	...	...	4959	12.42	...	...
760	...	...	...	...	269	0.49
780	...	...	5461	12.66	...	...
800	...	...	...	...	228	0.47
840	...	...	...	...	307	0.44
880	...	...	...	...	324	0.42
920	...	...	...	...	340	0.40
960	...	...	...	...	356	0.38
1000	...	...	...	...	371	0.37

[A] Available from NIST, Gaithersburg, MD. Values in table from relevant certificate for the reference material.

6.2 Heating vitreous silica that is contaminated with alkali compounds above 500°C may cause it to crystallize. To prevent this, clean the component by immersion in an aqueous solution containing 10 % hydrofluoric acid for 1 min followed by a thorough rinse with distilled water. To prevent contamination with alkali compounds do not touch the vitreous silica with the hands after cleaning.

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

6.4 Care is necessary to control the temperature gradient in long specimens.

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

6.6 A closed specimen holder is required when the dilatometer is immersed in a liquid bath.

6.7 Support or hold the specimen in a position so that it is stable during the test.

6.8 The specimen holder and push-rod shall consist of the same type of vitreous silica.

6.8.1 When applying the system calibration outlined in Section 9, a test run with a vitreous silica specimen should not result in an apparent mean coefficient of linear thermal expansion greater than ± 0.3 µm/m·K.

6.9 Since the precision and accuracy of the length measurements are fixed for a specific apparatus, to obtain the same percent uncertainty in the coefficient, a larger temperature range must be used for low-expansion materials than that for high expansion materials. Conversely, the percent uncertainty of the coefficient will be much larger for the low-expansion material if the same temperature range is used.

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

7. Apparatus

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

TABLE 2 Thermal Expansion of Standard Reference Material Kieselglas K001[A]

Temperature, °C	Expansion, $10^6\Delta L/L_0$		Expansivity, $10^6\alpha$ (*t*)		$10^6\alpha$, K^{-1}	
−170	−2.16	±0.67	−0.565	±0.019	0.013	±0.004
−160	−7.35	±0.55	−0.475	±0.016	0.046	±0.003
−140	−15.15	±0.44	−0.300	±0.011	0.108	±0.003
−120	−19.80	±0.43	−0.160	±0.007	0.165	±0.004
−100	−21.67	±0.43	−0.030	±0.005	0.217	±0.004
−80	−21.09	±0.43	0.085	±0.004	0.264	±0.006
−60	−18.36	±0.41	0.185	±0.004	0.306	±0.007
−40	−13.76	±0.38	0.272	±0.004	0.344	±0.010
−20	−7.57	±0.36	0.346	±0.005	0.378	±0.019
−10	−3.94	±0.34	0.379	±0.005	0.394	±0.038
0	0.00	±0.34	0.409	±0.005	...	...
10	4.23	±0.33	0.436	±0.005	0.423	±0.038
20	8.71	±0.33	0.46	±0.004	0.436	±0.019
40	18.38	±0.35	0.504	±0.004	0.459	±0.010
60	28.81	±0.37	0.538	±0.004	0.480	±0.007
80	39.86	±0.39	0.565	±0.003	0.498	±0.006
100	51.37	±0.41	0.585	±0.003	0.514	±0.005
120	63.22	±0.43	0.599	±0.003	0.527	±0.004
140	75.31	±0.44	0.608	±0.003	0.538	±0.004
160	87.52	±0.44	0.612	±0.003	0.547	±0.003
180	99.78	±0.45	0.613	±0.003	0.554	±0.003
200	112.01	±0.45	0.610	±0.004	0.560	±0.003
220	124.16	±0.45	0.605	±0.004	0.564	±0.002
240	136.18	±0.46	0.567	±0.004	0.567	±0.002
260	148.03	±0.47	0.588	±0.004	0.569	±0.002
280	159.68	±0.48	0.577	±0.004	0.570	±0.002
300	171.10	0.50	0.565	0.004	0.570	±0.002

[A] Fused silica available from Physikalisch Technische Bundesanstalt, Braunschweig, Germany. Values in table from relevant certificate for the reference material.

TABLE 3 Linear Thermal Expansion

Temperature, °C	Platinum,[A] $10^6\Delta L/L_0$	Aluminum,[B] $10^6\Delta L/L_0$
−195	−1756.66	...
−150	−1420.6	−3430
−100	−1024.09	−2550
−50	−607.96	−1550
0	−176.2	−460
20	0	0
50	268.06	710
100	722.38	1900
200	1654.6	4450
300	2612.01	7130
400	3692.18	10 050
500	4596.55	13 230
600	5628.65	16 760
700	6692.81	...
800	7793.27	...

[A] See Refs **(1-3)**.
[B] See Refs **(4-8)**.

7.1.1 *Specimen Holder and Push-Rod or Tube*, both made of annealed vitreous silica. Illustrations of typical tube and rod-type configurations are given in Fig. 1. Paragraph 6.1.1 contains a recommended annealing procedure for vitreous silica.

7.1.2 *Furnace, Cryostat, and Bath*, used for heating or cooling the specimen uniformly at a controlled rate over the temperature range of interest but not below −180°C or above 900°C.

7.1.3 *Transducer*, for example, a digital encoder, differential or dial-gage transformer for measuring the difference in length between the specimen and specimen holder or probe with a precision of at least ± 1.3 µm. The transducer shall be protected or mounted so that the maximum temperature change

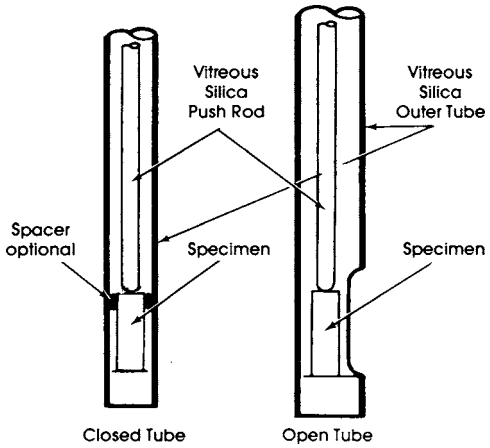

FIG. 1 Common Forms of Tube Types

observed in the transducer during a test will affect the transducer readings by not more than 1.0 µm.

7.1.4 *Temperature Measurement System*, consisting of a calibrated sensor or sensors, together with manual, electronic, or equivalent readout such that the indicated temperature can be determined to better than ±0.5°C.

7.1.4.1 Since this test method is used over a broad temperature range, different types of sensors may have to cover the complete range. The common sensors are fine gage (32 AWG or smaller wire) or thin foil thermocouples calibrated in accordance with Test Method E 220 or fine-gage resistance thermometers calibrated according to Test Methods E 644.

7.1.4.2 Type E and T are recommended for the temperature range of 190 to 350°C, and Types K, S, and N are recommended for temperatures of 0 to 900°C. If Type K is used continuously, regular checking of the calibration should be undertaken to ensure that contamination or phase change phenomena due to alloy component migration from the junction has not occurred during testing.

7.1.4.3 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 system insulated from the effects of temperature variations in the immediate surrounding ambient environment.

7.1.4.4 Additional information relating to the precise determination of temperature in dilatometry is contained in Appendix X2.

7.2 *Measurement Instrument*, such as an index micrometer or calipers capable of reading to at least± 25 µm in order to determine the initial and final lengths of the test specimen (and other relevant components, where required).

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 at least 25± 0.1-mm long and between 5 and 10 mm in diameter.

NOTE 3—For example, if the transducer is accurate to ±2 µm, the length must be at least 0.1 m.

8.2 The end surfaces of the specimen (as well as the contacting surface of the specimen holder or rods, or both)