



Designation: C 372 – 94 (Reapproved 2001)<sup>ε1</sup>

# Standard Test Method for Linear Thermal Expansion of Porcelain Enamel and Glaze Frits and Fired Ceramic Whiteware Products by the Dilatometer Method<sup>1</sup>

This standard is issued under the fixed designation C 372; 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 approval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

<sup>ε1</sup> NOTE—7.2.4.1 was editorially corrected in February 2007.

## 1. Scope

1.1 This test method covers the determination of the linear thermal expansion of premelted frit (porcelain enamel and glaze) and ceramic whiteware products by the thermal dilatometer method. This test method is applicable to apparatus meeting the reproducibility and accuracy requirements of this test method, which are to produce percent linear expansion accuracy of  $\pm 3\%$  or better and coefficient of linear expansion accuracy of  $\pm 5\%$  or better.

1.2 *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:

- E 220 Method for Calibration of Thermocouples by Comparison Techniques<sup>2</sup>
- E 228 Test Method for Linear Thermal Expansion of Solid Materials with a Vitreous Silica Dilatometer<sup>3</sup>
- E 230 Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples<sup>2</sup>
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>3</sup>

## 3. Terminology

### 3.1 Definitions:

3.1.1 *mean coefficient of linear thermal expansion*—from temperature  $T_1$  to temperature  $T_2$  ( $T_1 < T_2$ ):

$$\alpha, \text{ mm/mm}\cdot^\circ\text{C or in./in.}\cdot^\circ\text{C} = \frac{0.01 P}{T_2 - T_1}$$

where:

$\alpha$  = mean coefficient of linear thermal expansion from temperature  $T_1$  to  $T_2$ , and

$P$  = percent linear thermal expansion as defined in 3.1.2.

3.1.2 *percent linear thermal expansion*—from temperature  $T_1$  to temperature  $T_2$  ( $T_1 < T_2$ ):

$$P = \frac{L_2 - L_1}{L_0} \times 100 + A$$

$$= \frac{\Delta L}{L_0} \times 100 + A$$

where:

$P$  = percent linear thermal expansion from temperature  $T_1$  to  $T_2$ ,

$L_0$  = sample length at  $T_0$  ( $T_0$  between 20 and 30°C),

$L_1$  = sample length at  $T_1$ ,

$L_2$  = sample length at  $T_2$ , and

$A$  = instrument correction.

## 4. Significance and Use

4.1 Measurement of thermal expansion is useful for predicting stress within joined materials or single materials under conditions of changing or nonuniform temperature. It can also serve as an indicator of phase composition or changes in structure.

## 5. Apparatus

### 5.1 Thermal Dilatometer:

5.1.1 *General Description*—A thermal dilatometer is an apparatus that provides means for varying the temperature of a test specimen in a controlled manner, measuring the specimen length, and measuring the temperature of the specimen for each reading of specimen length. There are several different types as follows:

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 14.03.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 14.02.

5.1.1.1 *Manual*—A manual dilatometer is one in which any or all of the above are done by manual means and the corrected percent linear thermal expansion curve is plotted by hand.

5.1.1.2 *Recording*—A recording dilatometer is an apparatus by which the above are recorded by instrumental means but the final corrected percent linear thermal expansion curve is plotted by hand.

5.1.1.3 *Automatic Recording*—An automatic recording dilatometer is a recording dilatometer with provision for automatically plotting the corrected percent linear thermal expansion curve.

5.1.2 Any generally accepted apparatus that is capable of measuring the length changes produced by thermal expansion may be used in this method. The accuracy of the expansion-measuring apparatus including transducer, electronic, mechanical or optical amplification and readout device must be  $\pm 0.0001$  in. ( $\pm 0.003$  mm) and should be reproducible to  $\pm 0.00005$  in. ( $\pm 0.0013$  mm). A dilatometer may use a direct method of sighting on either of the two ends of the test specimen or suitable markings at the ends, by means of two telescopes mounted on a measuring bank. Another method transmits the change in length of the specimen to a sensitive dial gage or transducer by means of members that are chemically inert and free of phase transformations, having ground and polished surfaces at points of contact with the test specimens.

5.2 *Scale or Caliper*, capable of measuring the length of the specimen with an accuracy of  $\pm 0.0005$  in. ( $\pm 0.010$  mm) must be used.

5.3 *Furnace* that is electrically heated and designed so that the thermal gradient over the length of the test specimen shall be less than  $3^{\circ}\text{C}$ . This may be accomplished by electrical shuntings, individually controlled zones, or other methods.

5.4 *Temperature-Measuring Device*—Temperature measurements shall be made by means of a thermocouple placed in contact with the test specimen approximately at its mid-length. The thermocouple shall have the accuracy specified in Table 15 of Standard **E 230**. Type S or Type K thermocouples are recommended for this method.

5.5 *Temperature-Indicating Device*—The temperature-indicating device may be a millivolt potentiometer, a calibrated meter or recorder, or other apparatus with a precision of  $\pm 5^{\circ}\text{C}$  and an accuracy specification equivalent to the precision.

## 6. Test Specimens

6.1 For frit or dried slip samples, specimens shall be prepared as follows:

6.1.1 Frit should be crushed and screened through a 10-mesh sieve to remove large lumps. Then, a refractory boat crucible shall be filled with the sample material. If it is desired to reuse the crucible, it should be first lined with powdered alumina as a parting agent. The crucible can be of any suitable refractory material such as porcelain or alumina, but shall be unglazed. For frits that will be fired at less than  $800^{\circ}\text{C}$ , a metal mold may be used, if desired.

6.1.2 The test specimen shall be subjected to the same firing cycle used commercially in order to give a smooth surface on a bulk sample.

NOTE 1—The sample must be cooled slowly over several hours to preserve structural integrity.

6.2 For all samples, test specimens may be of any convenient length, provided the uniformity of the furnace has been determined over that length. The minimum thickness of the specimen shall be 0.2 in. (5.1 mm) and the maximum cross-sectional area shall be  $0.45$  in.<sup>2</sup> ( $2.9$  cm<sup>2</sup>). The ends of the specimen shall be ground flat and perpendicular to the axis of the specimen.

6.3 Test specimens shall be conditioned in accordance with the history of the specimen. Conditioning shall include drying, annealing, or protection against moisture expansion, as may be necessary.

6.4 The length of the specimen shall be measured to within an accuracy of 0.1 %.

## 7. Calibration

7.1 Periodic calibration of the thermal dilatometer is recommended to assure the accuracy required by this method. Procedures for calibrating the component parts of the dilatometer are given below. A less time-consuming method for standardizing a complete apparatus, especially the recording type, is also given. A calibration check of the components of the apparatus should be done on an annual basis and calibration of the complete instrument using a standard sample should be done within 90 days preceding a report prepared under this method. The date of last calibration by either method should be included on the report.

### 7.2 Calibration Procedures:

#### 7.2.1 Dilatometer:

7.2.1.1 If a direct sighting method is used, the dilatometer can be calibrated with a standard sample with a known length that has been measured by a micrometer with an accuracy of  $\pm 0.0001$  in. (0.003 mm). The reference sample should be made from a material that has a very low thermal expansion, such as fused silica or invar. The dilatometer system can be calibrated by measuring the length of the sample using a movable telescope and comparing it with the known value.

7.2.1.2 If a dial gage transducer system is used, the dilatometer can be calibrated with a micrometer or thickness gage. Fix the dial gage transducer and micrometer in position on the instrument itself or in a special fixture during calibration. The system can be calibrated by displacing the probe of the transducer a known amount with the micrometer or thickness gage and adjusting the instrument to give that value. Whichever technique is used, the micrometer or thickness gage shall be accurate to  $\pm 0.0001$  in. (0.003 mm).

7.2.2 *Furnace*—The thermal gradient that occurs over the sample length within the furnace should be determined by simultaneously measuring the temperature at the center, and at the ends of an alumina sample  $\frac{3}{8}$  to  $\frac{1}{2}$  in. (10 to 13 mm) in diameter and equal in length to the standard size sample for which the apparatus is intended. The thermocouples shall be Type S or Type K. Thermocouple wire of 0.010-in. (0.25-mm) diameter or less should be used. The thermocouple beads should be in contact with the test sample surface. Bring thermocouple wires out of the furnace for termination. A common negative wire may be used for all three thermocouples to reduce the number of leads brought from the furnace.