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Standard Test Method for Linear Thermal Expansion of Electrode Carbons¹

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1. Scope

1.1 This test method covers the determination of the coefficient of linear thermal expansion (CTE) for carbon anodes and cathodes used in the aluminum industry, in baked form, by use of a vitreous silica dilatometer.

1.2 The applicable temperature range for this test method for research purposes is ambient to 1000°C. The recommended maximum use temperature for product evaluation is 500°C.

1.3 This test method and procedure is based on Test Method E228, which is a generic all-encompassing method. Specifics dictated by the nature of electrode carbons and the purposes for which they are used are addressed by this procedure.

1.4 Electrode carbons in the baked form will only exhibit primarily reversible dimensional changes when heated.

1.5 The values stated in SI units are to be regarded as standard.

1.6 *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
E228 Test Method for Linear Thermal Expansion of Solid Materials With a Push-Rod Dilatometer

3. Terminology

3.1 Definitions:

3.1.1 *linear thermal expansion*—the change in length per unit length resulting from a temperature change. Linear thermal expansion is symbolically represented by $\Delta L/L_0$, where ΔL is the length change of the specimen ($L_1 - L_0$), L_0 and L_1 are the

specimens lengths at reference temperature T_0 and test temperature T_1 , respectively. Linear thermal expansion is often expressed as a percentage or in parts per million (such as $\mu\text{m/m}$).

3.1.2 *mean coefficient of linear thermal expansion (CTE)*—The linear thermal expansion per change in temperature; the mean coefficient of linear thermal expansion is represented by:

$$\bar{\alpha}_{T_1} = \frac{\Delta L/L_0}{\Delta T} = \frac{1}{L_0} \cdot \frac{\Delta L}{\Delta T} = \frac{1}{L_0} \frac{L_1 - L_0}{T_1 - T_0} \quad (1)$$

3.1.2.1 This has to be accompanied by the values of the two temperatures to be meaningful; the reference temperature (T_0) is 20°C, and the notation may then only contain a single number, such as $\bar{\alpha}_{200}$, meaning the mean coefficient of linear thermal expansion between 20 and 200°C.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *reference specimen*—a particularly identified or pedigreed material sample, with well-characterized behavior and independently documented performance.

3.2.2 *specimen*—a representative piece of a larger body (anode, cathode, and so forth) that is considered to be fairly typical of a portion or of the entire piece.

3.2.3 *vitreous silica dilatometer*—a device used to determine linear thermal expansion, by measuring the difference in linear thermal expansion between a test specimen and the vitreous silica parts of the dilatometer.

4. Summary of Test Method

4.1 A representative specimen is placed into a vitreous silica dilatometer and heated, while its linear expansion is continuously recorded. The change of the specimen length is recorded as a function of temperature. The coefficient of linear thermal expansion is then calculated from these recorded data.

5. Significance and Use

5.1 Coefficients of linear thermal expansion are used for design and quality control purposes and to determine dimensional changes of parts and components (such as carbon anodes, cathodes, and so forth) when subjected to varying temperatures.

6. Apparatus

6.1 *Dilatometer*—The dilatometer consists of the following:

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.05 on Properties of Fuels, Petroleum Coke and Carbon Material.

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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.

6.1.1 *Specimen Holder and Push-rod*, both made of vitreous silica. The design of the device shall ensure that the push-rod load on the specimen by itself is not causing deformation. The use of pressure distribution quartz plates on top of the specimen is permissible.

NOTE 1—Dilatometers are usually constructed in horizontal or vertical configurations (1)³ Vertical devices are preferred for very large samples and when extensive shrinkage is expected. Horizontal configurations usually afford better temperature uniformity over the specimen, but are subject to drooping when large specimens are employed. Horizontal devices, when used with very large specimens, require special provisions to reduce friction between the specimen and the dilatometer tube to minimize push-rod pressure required to keep the specimen in contact with the end plate. For this application, either configuration is acceptable.

NOTE 2—For basic construction details and fused silica annealing schedule, consult Test Method E228.

NOTE 3—Multiple rods supporting a platform in place of large diameter tubes have been also used successfully in the vertical configuration.

6.1.2 *Transducer or Indicator*, for measuring the difference in length between the specimen and the dilatometer with an accuracy within $\pm 2 \mu\text{m}$. The transducer shall translate these movements into an electrical signal suitable for displaying or recording. The non-linearity of this conversion must be less than 0.25 % of the full scale value of the output. The transducer shall be protected or mounted so that the maximum temperature change observed in the transducer during a test will affect the transducer readings by less than $1 \mu\text{m}$.

6.1.3 *Temperature Sensors*, for determining the mean temperature of the specimen with an accuracy within $\pm 0.5^\circ\text{C}$. When a thermocouple is used, it shall be referenced (cold junction compensated) to the ice point with an ice-water bath or an equivalent system.

6.1.3.1 Due to the large size of the specimen, a minimum of one thermocouple per 40 mm specimen length must be employed. It is permissible to read the output of each thermocouple independently and average the readings or to connect them in series and divide the single reading by the number of thermocouples to obtain the average. In the latter case, interconnections must be made at or beyond the point of cold junction compensation.

6.1.3.2 The temperature sensors shall be in close proximity to the specimen, preferably in between the quartz dilatometer tube and the specimen. The temperature sensors shall not be directly exposed to the furnace walls.

6.2 *Readout or Recording of Data:*

6.2.1 Manual recording of expansion and temperature values indicated at selected temperature points may be made if the transducer is equipped with or connected to a suitable display and the thermocouples outputs are determined with a potentiometer or millivolt meter.

6.2.2 Chart or data logger recording of the expansion and temperature signals may be accomplished using a device whose resolution is at least 1000 times higher than the expected maximum output signal. All calculations and corrections (see Section 10) must be done externally based on the recorded values.

6.2.3 Computerized recording may be used with similar restriction to 6.2.2. Calculations and corrections may be done using suitable software.

6.3 *Furnace*—The furnace is used for uniformly heating the specimen over the temperature range of interest, but not above 1000°C . Temperature uniformity shall be at least $\pm 0.5^\circ\text{C}$ per 50 mm of sample length. The temperatures shall be controlled as a function of time. The furnace may have a muffle (quartz, mullite, alumina, inconel, monel, or stainless steel are most common) or other provisions to provide a protective atmosphere for the specimen. The furnace shall have provisions for continuous purging with an inert gas at a sufficient rate, and exclude air from the specimen while a purge is maintained.

6.4 *Caliper*—The caliper (micrometer or Vernier type) is for measuring the initial length of the specimen, L_0 , with an accuracy within $\pm 25 \mu\text{m}$, and a capacity to open to the length of the specimen plus 1 mm.

7. Test Specimen

7.1 Specimens shall be cylindrical, preferably with a 50 ± 2 mm diameter. Slightly smaller or larger diameters can also be accommodated without degradation of data. The length of the specimens shall be between 50 and 130 mm in length and have flat and parallel ends to within $\pm 25 \mu\text{m}$.

7.2 It is permissible to stack up to five disks of smaller lengths to obtain a proper length specimen. The interfaces, however, must be flat and parallel within $\pm 25 \mu\text{m}$ to prevent rocking.

7.3 The dimensions of the specimens should be ordinarily measured as received.

7.4 If water was used in conjunction with their preparation, each specimen must be kept in an oven at $110 \pm 5^\circ\text{C}$ for at least 6 h and allowed to cool down thereafter, prior to testing. If any heat or mechanical treatment is applied to the specimen prior to testing, this treatment should be noted in the report.

8. Calibration

8.1 The transducer should be calibrated by imposing a series of known displacements with a precision screw micrometer, gage blocks, or equally accurate device. For absolute transducers (such as digital encoders, and so forth), this procedure is omitted and periodic verification is sufficient.

8.2 Verification of the calibration of the temperature sensors separately from the dilatometer is to be performed periodically or when contamination of the junction is suspected.

8.3 Regardless of independent calibrations of the transducer and the thermocouples, the dilatometer, as a total system, shall be calibrated by determining the thermal expansion of at least one reference material of known thermal expansion. Recommended reference materials are listed in Annex A1.

8.3.1 The calibration should be done using approximately the same thermal cycle as that used for testing (see 9.7 and 9.8).

8.3.2 The calibration constant may be derived as follows:

$$A = \left(\frac{\Delta L}{L_0} \right)_t - \left(\frac{\Delta L}{L_0} \right)_m \quad (2)$$

where:

³ Hidnert, P. and Krider, H.S., "Thermal Expansion Measurements," *Journal of Research*, National Bureau of Standards, Vol 48, 1952, p. 209.

m = measured expansion of the reference material, and
 t = true or certified expansion of the reference material.

8.4 If the calibration specimen is considerably smaller in the cross section than the specimen, it is necessary to provide a thermal jacket around it to prevent errors caused by convective currents. A thermal jacket may be produced by drilling a hole in the axis of a carbon specimen and loosely fitting the calibration specimen into it. The carbon jacket must be about 1 mm shorter than the calibration specimen.

8.5 The use of published values of thermal expansion for quartz may not be used to compute a correction factor. Accounting for the expansion of the dilatometer parts through calculations in place of a calibration procedure described above is not permitted.

8.6 Materials usable for calibration or verification of operation fall into four categories.

8.6.1 *Standard Reference Materials*—These are actual specimens supplied with a certificate by NIST⁴, or a similar national standards organization of another country.^{5,6}

NOTE 4—These materials are very few and NIST supplies have been exhausted in some cases. To determine the absolute accuracy of a device, materials in this category are the most preferred.

8.6.2 *Traceable Reference Materials*—These are extensively investigated materials, substantially described in published literature and considered stable. Often they are generically identical to Standard Reference Materials. Specific lots when tested in a systematic fashion using a dilatometer calibrated with a certified Standard Reference Material can be referred to as Traceable Reference Materials. They may also serve well in comparing equipment or test procedures at different laboratories and to arbitrate disputes and differences.

8.6.3 *Reference Materials*—These are widely investigated, well-characterized materials that were found to be stable with time and temperature exposure and performance data are readily available in the literature; for example, platinum. These materials are well suited for round-robin, day-to-day verification (working reference) of equipment performance and periodic verification programs. Purity and physical parameters (density, electrical resistivity, and so forth) must be reasonably matched to use literature data.

8.6.4 *Characterized Private Stock Materials*—These are substances that are mainly used for in-house verifications. Even though they may be thought of as being well characterized, the data is primarily self consistent. If such a material is found to be very stable by independent tests, it may be used in round-robin tests, but caution should be exercised when the data is intended to arbitrate disputes or differences between facilities. Typical use should be limited to that of an in-house working reference. Primary reason for use is having thermal characteristics closely resembling those of actual test specimens.

⁴ National Institute of Standards and Technology (formerly the National Bureau of Standards, Gaithersburg, MD 20899-0001.

⁵ Standard Reference Materials SRM731 and SRM720, National Institute of Standards and Technology, (certificate), Washington, DC 20234.

⁶ Standard Reference Material SRM739, National Institute of Standards and Technology, (certificate), Gaithersburg, MD 20899.

9. Procedure

9.1 Measure the initial (room temperature) length of the specimen, and record it as L_0 .

9.2 Place the specimen into the dilatometer after making certain that all contacting surfaces are free of foreign material. It is important to have good seating of the specimen in a stable position. (**Warning**—Alkali contamination will adversely affect fused silica parts. Avoid touching them with hands.)

9.3 Ensure that the temperature sensors shall not restrict movement of the specimen in the dilatometer. Do not allow an exposed junction to contact any carbonaceous materials.

9.4 Make certain that the push-rod is in stable contact with the specimen. A pressure distribution plate made of fused silica may be used between the push-rod and the specimen.

9.5 Insert the loaded dilatometer into the furnace (at ambient temperature) and allow the temperature of the specimen to come to equilibrium.

9.6 Record the initial readings of the temperature sensors, T_0 , and the transducer, X_0 .

9.7 Heat the furnace to 300 or $500 \pm 10^\circ\text{C}$ at a rate not exceeding $10^\circ\text{C}/\text{min}$. Allow the furnace and specimen to stabilize at that temperature for 60 min. Record readings of the temperature sensors, T_1 , and the transducer, X_1 . If data obtained during ramping is to be used, heating rates above $1^\circ\text{C}/\text{min}$ are not permitted.

9.8 Alternate to 9.7. Heat the furnace at rates up to $10^\circ\text{C}/\text{min}$. Hold the furnace and specimen at a single or series of constant temperatures until the transducer reading reaches a constant value (variation $< \pm 2.6 \mu\text{m}$). At that point, the indicated temperature of the specimen shall not vary by more than $\pm 2^\circ\text{C}$ and the temperature gradient in the specimen shall not exceed 2 % of its actual temperature. The dwell time is a function of the thermal mass of the dilatometer and the specimen. It will vary with temperature and heating rate. The length of the dwell shall be sufficient as to limit consecutive length change readings taken in 5 min intervals to less than 4 μm . Readings of temperature, T_i , and specimen length changes, X_i , need to be recorded at each constant temperature. When this alternate procedure is used, it is important that the calibration cycle be performed in a nearly identical manner. (**Warning**—Heating vitreous silica above 800°C will cause viscous flow and a non-reversible, time-dependent change in its thermal expansion. The magnitude of these effects will depend on the particular type of vitreous silica and the mechanical loads applied to a piece. Slow devitrification will occur above 900°C , and, therefore, regular use above 800°C should be minimized.)

10. Calculation

10.1 *Manual Data Analysis*—Calculate the linear thermal expansion of the test specimen.

10.1.1 At temperature T_1 :

$$\Delta L = X_1 - X_0 \quad (3)$$

10.1.2 Or more generally at any temperature T_1 :

$$(\Delta L)_i = X_i - X_0 \quad (4)$$

$$\left(\frac{\Delta L}{L_0}\right)_c = \left(\frac{\Delta L}{L_0}\right)_m + A \quad (5)$$