



# Standard Test Method for Temperature Calibration of Dielectric Analyzers<sup>1</sup>

This standard is issued under the fixed designation E 2038; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method describes the temperature calibration of dielectric analyzers over the temperature range from  $-100$  to  $300$  °C and is applicable to commercial and custom-built apparatus. The calibration is performed by observing the melting transition of standard reference materials having known transition temperatures within the temperature range of use.

1.2 Electronic instrumentation or automated data analysis and data reductions systems or treatment equivalent to this test method may be used.

1.3 The values stated in SI units are to be reported as the standard.

1.4 *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 to determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

E 473 Terminology Relating to Thermal Analysis

E 1142 Terminology Relating to Thermophysical Properties

## 3. Terminology

3.1 *Definitions*—Specific technical terms used in this test method are defined in accordance with Terminologies E 473 and E 1142.

## 4. Summary of Test Method

4.1 A test specimen of known solid-solid or solid-liquid (melting) transition temperature is characterized for its dielectric properties in a dielectric analyzer of interest as a function of temperature. At the transition, a sharp change in the dielectric properties is observed. The temperature observed for the transition by the apparatus is recorded. Using a linear

equation, for one or more transitions, the temperature scale or readout of the apparatus is calibrated based upon the known and observed transition temperatures.

## 5. Significance and Use

5.1 This test method permits interlaboratory comparison and intralaboratory correlation of instrumental temperature scale data.

5.2 Dielectric analyzers are used to characterize a broad range of materials that possess dielectric moments. One of the desired values to be assigned by the measurement is the temperature at which significant changes occur in the properties of the test specimen. In order to obtain consistent results from one period of time to another and from one laboratory to another, the temperature signal from the apparatus must be calibrated accurately over the temperature range of interest.

## 6. Interferences

6.1 Because the specimen size usually is small, care must be taken to ensure that each specimen is homogeneous and representative of the sample as a whole.

6.2 This test method measures dielectric properties under specific experimental conditions. Should those experimental conditions change, there may be an effect on the calibration of the apparatus.

6.3 Contact or adhesion of the specimen to the sensor should not be lost during the course of the measurement, otherwise erroneous values will be recorded.

## 7. Apparatus

7.1 *Dielectric Analyzer*, consisting of the following items:

7.1.1 *Sensors*, electrodes for imparting the alternating electric field and measuring the induced current and phasing in the specimen. These usually are solid platforms that also serve to hold the specimen. These may be either two parallel plate electrodes or a single plate containing a series of interdigitated electrodes.

NOTE 1—When using parallel plate electrodes, provision shall be made to prevent the electrodes from contacting each other upon the melting of the test specimen.

7.1.2 *Temperature Sensor*, for measuring the specimen temperature to within  $\pm 0.1$  °C.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.01 on Test Methods and Recommended Practices.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

7.1.3 *Specimen Container*, for containing the test specimen in liquid form (after it melts).

7.1.4 *Temperature Programmer and Furnace*, capable of temperature programming the test specimen from –100 to 300 °C at a rate of at least 1°C/min, or performing isothermal temperature operation at temperatures over the range of interest to ± 1 °C.

7.1.5 *Cooling Device and Supplies*, to provide operating temperatures below ambient temperatures. This may be a mechanical refrigerator or other coolant such as liquid nitrogen.

7.1.6 *Specimen Atmosphere Control System*, capable of supplying inert gas, usually nitrogen, argon, or helium, with an operator selectable flow rate of 50 to 100 mL/min to within ± 5 mL/min.

7.1.7 *Recording Device*, either digital or analog, to record and display the dielectric thermal curve consisting of permittivity on the *Y* axis (ordinate) and temperature on the *X* axis (abscissa).

7.1.8 While not required, it is convenient to have a data analysis device that will perform and display the calculations of this test method.

## 8. Reagents and Materials

8.1 *Inert Gas*, purified, dry nitrogen, argon, or helium.

NOTE 2—If calibration is to be done at low temperatures, that is, below the dew point, it is essential to have a dry environment as condensed moisture can affect the results.

8.2 *Calibration Materials*, two materials possessing dielectric properties that undergo a solid-liquid (melting) or solid-solid transition within the temperature range of interest. Several suitable materials are listed in Table 1.

## 9. Hazards

9.1 Toxic or corrosive effluents, or both, may be released when heating some materials and could be harmful to personnel and to apparatus.

9.2 High voltages may exist on the exposed electrodes during the procedure. Care should be taken to avoid contact with the electrodes.

9.3 Some components of the test circuit, including the sample itself, may retain electrical charge even after the test is completed and the voltage source is disconnected. Ensure that all charges are eliminated from these components before touching the instrument.

**TABLE 1 Calibration Materials<sup>A</sup>**

Calibration Materials	Transition Temperature (°C)
1,2-dichloroethane	–35.7 (solid - liquid)
benzil	94.9 (solid - liquid)
acetanalide	114.4 (solid - liquid)
benzoic acid	122.4
diphenylacetic acid	147.3 (solid - liquid)
anisic acid	183.3
carbazole	245.6 (solid - liquid)

<sup>A</sup>Available from the Laboratory of the Government Chemist, Queens Road, Teddington, Middlesex, United Kingdom TW11 0LW.

## 10. Sampling

10.1 Samples usually are analyzed on an “as received” basis. If some thermal or mechanical treatment, such as grinding or sieving, is applied to the sample prior to analysis, it shall be indicated in the report.

10.2 Since small test specimens are used, they must be homogeneous and representative of the sample. The mixing or stirring of samples prior to analysis is recommended.

10.3 The test specimen must cover the entire surface of parallel plate electrodes. The test specimen thickness depends on the dielectric properties of the specimen and the sensor size; however, a minimum thickness of 0.10 mm is recommended.

10.4 For interdigitated electrodes the test specimen should cover the electrode array completely. The thickness of the test specimen should be at least 1.5 times the electrode spacing.

## 11. Calibration and Standardization

11.1 Calibrate the permittivity and temperature sensors of the apparatus using the procedure described by the manufacturer in the operator’s manual.

11.2 Calibration materials with a dielectric moment and with a solid-solid or solid-liquid (melting) transition of known value may be used. The 99.9+ % pure materials listed in Table 1 may be used for calibration.

## 12. Procedure

12.1 Select two calibration materials (see Table 1) that have transitions near the extremes of the temperature range of interest.

12.2 Load the calibration material with the lower transition temperature into the apparatus as a test specimen.

12.3 Set the initial temperature of the apparatus to a value about 30 °C below the estimated transition temperature of the calibration material.

12.4 Initiate the measurement of permittivity at a test frequency of 1000 Hz. Initiate a temperature program of constant heating rate of 1 to 3 °C/min to a temperature 20 °C above the estimated transition temperature of the calibration materials. Record permittivity, on a linear scale, as a function of temperature.

NOTE 3—Other test frequencies may be used but shall be indicated in the report.

NOTE 4—Other heating rates may be used but shall be indicated in the report.

12.5 Remove residue reference material and clean the electrodes at the end of the experiment.

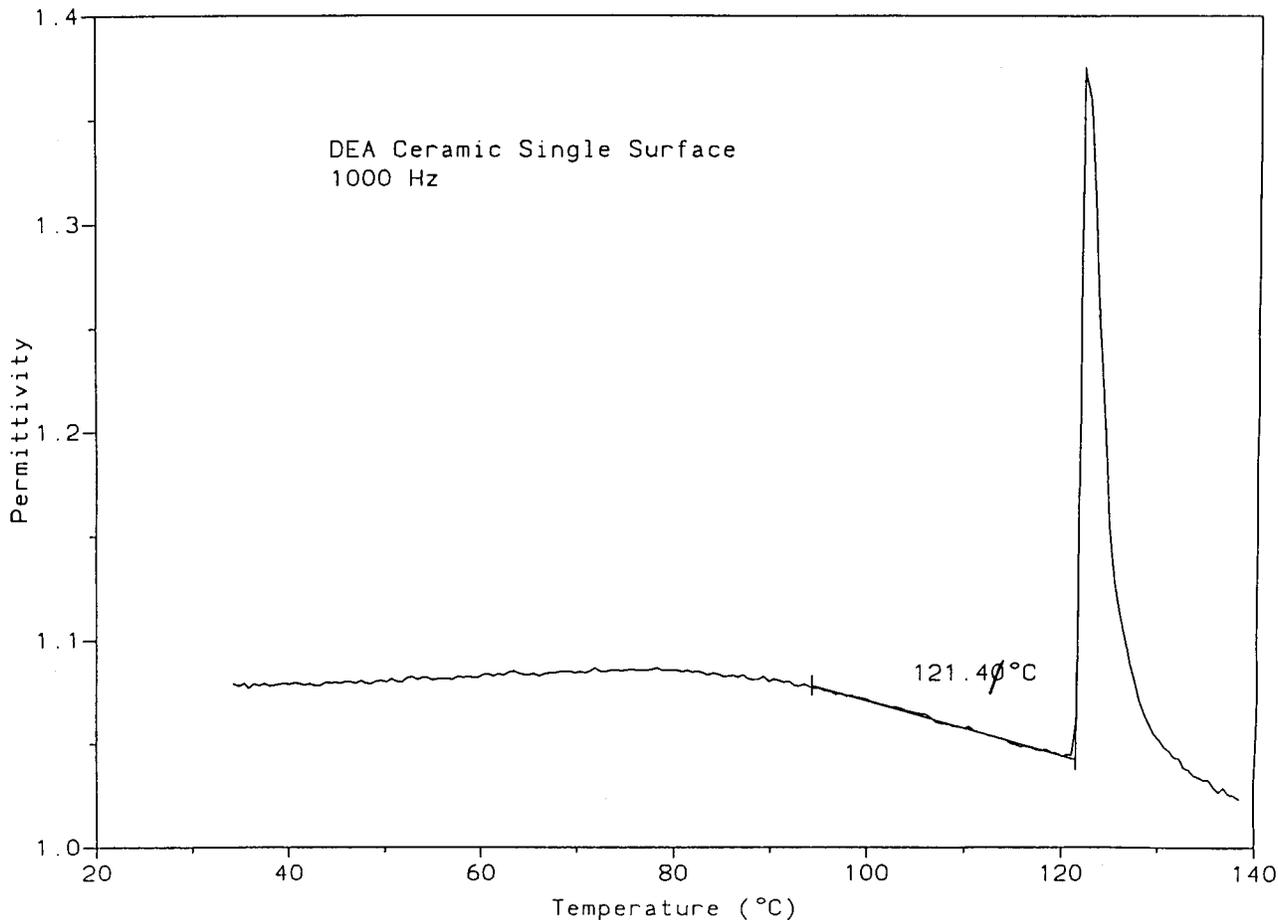
NOTE 5—Cleaning of the electrodes may be accomplished by washing with a suitable dissolving solvent, then drying. Alternatively, the electrodes may be heated to a temperature sufficiently high to evaporate any organic materials remaining provided this temperature does not exceed the temperature limit of the electrode.

12.6 Determine the observed temperature as the extrapolated onset of the increase in permittivity occurring at the transition (see Fig. 1).

12.6.1 On a linear display of permittivity versus temperature, construct a tangent to the baseline prior to the transition.

12.6.2 Construct a tangent to the permittivity curve at the steepest portion of the curve following the transition.

# DEA



Sample: Benzoic acid evaporate and rerun  
 Size: 0.8000 mm  
 Method: 3 °C/min, cool, reheat  
 Comment: N2 purge = 500 mL/min. Look for signal with minimum sample.

FIG. 1

12.6.3 The temperature value at the intersections of the tangents from 12.6.1 and 12.6.2 is taken as the observed temperature ( $TO_1$ ).

12.7 Repeat 12.2-12.6 for the calibration material with the higher transition temperature. The observed value is taken to be  $TO_2$ .

### 13. Calculation

13.1 For the purposes of this test method, it is assumed that the relationship between observed temperature ( $TO$ ) and the actual specimen temperature ( $T$ ) is a linear one governed by Eq 1.

$$T = (TO \times S) + I \quad (1)$$

where:

$S$  and  $I$  = the slope and intercept, respectively.

NOTE 6—For some instruments, the assumption of a linear relations between observed and actual specimen temperature may not hold. Under

such conditions, calibration temperatures sufficiently close together shall be used so that the instrument calibration is achieved with a series of linear relations.

#### 13.2 Two-Point Calibration:

13.2.1 Using the standard temperature ( $TS_1$  and  $TS_2$ ) values taken from Table 1 and the corresponding observed temperatures ( $TO_1$  and  $TO_2$ ), taken from the experimental section above, calculate the slope ( $S$ ) and intercept ( $I$ ) using Eq 2 and 3.

$$S = (TS_1 - TS_2) / (TO_1 - TO_2) \quad (2)$$

$$I = [(TO_1 \times TS_2) - (TS_1 \times TO_2)] / (TO_1 - TO_2) \quad (3)$$

where:

$S$  = Slope (nominal value = 1.0000),

$I$  = Intercept,

- $TS_1$  = Reference transition temperature for Standard 1 from Table 1,  
 $TS_2$  = Reference transition temperature for Standard 2 from Table 1,  
 $TO_1$  = Observed transition temperature for Standard 1 determined in 12.6, and  
 $TO_2$  = Observed transition temperature for Standard 2 determined in 12.7.

NOTE 7— $I$  has the same units (that is °C or K) as  $TS_1$ ,  $TS_2$ ,  $TO_1$  and  $TO_2$ , which are consistent with each other. The value for  $I$  will be different, depending on the units used.  $S$  is a dimensionless number whose value is independent of the units of  $I$  or  $T$ .

13.2.2 When performing these calculations, retain all available decimal places in the measured values and in intermediate values of the calculation, such as the values for  $S$  and  $I$ . The final calculated or corrected temperature should be rounded to the decimal place equivalent to two significant places in the standard deviation. If this temperature is to be used in subsequent calculations, however, all available decimal places should be retained.

13.3 *One-Point Calibration*—If the slope value determined in 13.2.1 is sufficiently close to 1.0000, only the intercept need be determined through a subsequent one-point calibration procedure.

$$I = TS_1 - TO_1 \quad (4)$$

13.4 Using the determined values of  $S$  and  $I$ , Eq 1 may be used to calculate the actual specimen transition temperature ( $T$ ) from an observed transition temperature ( $TO$ ).

## 14. Report

14.1 Report the following information:

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14.1.1 Complete identification and description of the standard reference materials used for calibration, including source and purity.

14.1.2 Model number and description of the instrument used for calibration, including location of the temperature sensor.

14.1.3 Details of the procedure used for calibration, including a description of the type of sensors, sample container, and any departures from the described procedure.

14.1.4 Identification of gas composition, flow rate, and purity of the specimen atmosphere.

14.1.5 Heating rate and excitation frequency used.

14.1.6 A copy of relevant original records.

14.1.7 The values of  $S$  and  $I$ .

## 15. Precision and Bias

15.1 The precision and bias of this test method have not yet been determined. An interlaboratory test program is planned to provide such information. Anyone wishing to participate in such a test program should contact the E37.01 Subcommittee Chairman in care of ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

15.2 A preliminary study<sup>3</sup> indicates that the pooled standard deviation of triplicate determinations of  $TO$  on seven materials is  $\pm 0.70^\circ\text{C}$  with an average bias ( $TS - TO$ ) of  $2.4^\circ\text{C}$ .

## 16. Keywords

16.1 calibration; dielectric analyzers (DEA); melting; temperature; thermal analysis

<sup>3</sup> Foreman, J.A., Lundgren, C.J., and Blaine, R.L., "Temperature Calibration of Dielectric Analyzers," *Proceedings of the 23rd North American Thermal Analysis Society Conference*, 1994, pp. 444-448.