



Standard Test Method for Determining and Reporting Dynamic Dielectric Properties¹

This standard is issued under the fixed designation E 2039; 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.

1. Scope

1.1 This test method describes the gathering and reporting of dynamic dielectric data. It incorporates laboratory test method for determining dynamic dielectric properties of specimens subjected to an oscillatory electric field using a variety of dielectric sensor/cell configurations on a variety of instruments called dielectric, microdielectric, DETA (DiElectric Thermal Analysis), or DEA (DiElectric Analysis) analyzers.

1.2 This test method determines permittivity, loss factor, ionic conductivity (or resistivity), dipole relaxation times, and transition temperatures, and is intended for materials that have a relative permittivity in the range of 1 to 10^5 ; loss factors in the range of 0 to 10^8 ; and, conductivities in the range 10^{16} to 10^{10} S/cm.

1.3 The test method is primarily useful when conducted over a range of temperatures for nonreactive systems (-160°C to degradation) and over time (and temperature) for reactive systems and is valid for frequencies ranging from 1 mHz to 100 kHz.

1.4 Apparent discrepancies may arise in results obtained under differing experimental conditions. Without changing the observed data, completely reporting the conditions (as described in this test method) under which the data were obtained, in full, will enable apparent differences observed in another study to be reconciled.

1.5 SI units are the 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.* Specific precautionary statements are given in Section 10.

2. Referenced Documents

2.1 ASTM Standards:²

D 150 Test Method for A-C Characteristics and Permittivity (Dielectric Constant) of Solid Electrical Insulating Materials

E 473 Terminology Relating to Thermal Analysis

E 1142 Terminology Relating to Thermophysical Properties

E 2038 Test Method for Temperature Calibration of Dielectric Analyzers

3. Terminology

3.1 Definitions:

3.1.1 The following technical terms are applicable to this document and are defined in Terminologies E 473 and E 1142: dielectric thermal analysis, angular frequency, capacitance, conductivity, dielectric constant, dielectric dissipation factor, dielectric loss angle, dipole relaxation time, dissipation factor, frequency, loss factor, permittivity, phase angle, and tangent delta.

3.1.2 *Relative permittivity* and *loss factor* are dimensionless quantities and are relative to the permittivity of free space ($\epsilon_0 = 8.854 \text{ pF/m}$). Relative permittivity also is known as the dielectric constant.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *dielectric (or microdielectric) sensor, n*—a set of at least two (perhaps three) contacting electrodes for measuring the dielectric response of materials.

3.2.1.1 *Discussion*—The sensor generally consists of parallel, circular, conducting (metallic) plates or discs, between which the sample is placed. The sensor also may be a set of interdigitated conductors on an insulating substrate. In some cases, the sensor may incorporate amplifying electronics or a temperature sensing device (see Fig. 1), or both.

3.2.2 *interdigitated electrode, n*—an electrode configuration consisting of two nonconnected, interpenetrating conductors firmly attached to an insulating substrate and exposed to the specimen on top.

3.2.2.1 *Discussion*—Interdigitated electrodes of different geometry are available, such as, interpenetrating “fingers” or “combs,” interpenetrating circular spirals, or interpenetrating square spirals (see Fig. 1).

Whereas parallel plate electrodes contact a specimen on a “top” and “bottom” surface, the interdigitated electrodes make contact on only one side (single-sided contact) of the specimen.

¹ 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.

Current edition approved Feb. 1, 2004. Published March 2004. Originally approved in 1999. Last previous edition approved in 1999 as E 2039-99.

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.

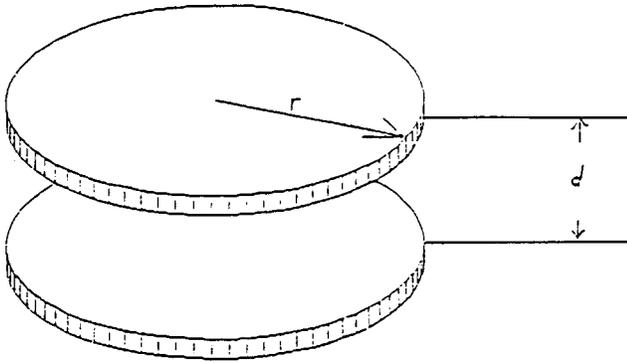


FIG. 1 Parallel Plate Electrodes

- A = plate area (calculated as $2 \times \pi \times r^2$) (see Fig. 1)
- C_p = parallel capacitance (see Test Method D 150)
- R_p = parallel resistance (see Test Method D 150)
- r = radius of circular plate (see Fig. 1).

4. Summary of Practice

4.1 An oscillatory electric potential (voltage) is applied to a test specimen by means of an electrode of known geometry. An electric current is measured at a sensing electrode separated from the transmitting electrode by the specimen under test. From the amplitude and phase shift of the measured current relative to the applied voltage and from known geometrical constants, such as electrode spacing and electrode arrangement, desired dielectric properties of the specimen under test may be obtained. Such properties include conductivity, dielectric constant, dielectric dissipation factor, dielectric loss angle, dipole relaxation time, dissipation factor, relative permittivity, loss factor, and tangent delta. The desired dielectric properties may be obtained as a function of frequency, temperature, or time by varying and measuring these independent parameters during the course of the experiment.

NOTE 1—The particular method for measurement of amplitude and phase shift depends upon the operating principle of the instrument used.

5. Significance and Use

5.1 Dielectric measurement and testing provide a method for determining the permittivity and loss factors as a function of temperature, frequency, time, or a combination of these variables. Plots of the dielectric properties against these variables yield important information and characteristics about the specimen under test.

5.2 This procedure can be used to do the following:

5.2.1 Locate transition temperatures of polymers and other organic materials, that is, changes in molecular motion (or

3.2.3 *electrode spacing* (E_S), n —for interdigitated electrodes, the width of the insulator strip between adjacent electrodes in the electrode array (see Fig. 1).

3.2.4 *electrode width* (E_W), n —for interdigitated electrodes, the width of a single electrode in the electrode array (see Fig. 1).

3.2.5 *electrode height* (E_h), n —for interdigitated electrodes, the thickness of an electrode normal to the surface of the substrate upon which it is situated (see Fig. 1).

3.2.6 *meander length* (M_L), n —for interdigitated electrodes, the total length of the zig-zag path between the two sets of conductors in the electrode array.

3.2.7 *substrate capacitance* (C_{sub}), n —for interdigitated electrodes, the capacitance of the sensor due to the insulating substrate.

3.2.7.1 *Discussion*—This value depends only on the geometry of the sensor and the material of the substrate, and not on the specimen under test on top of the interdigitated electrodes.

3.3 Abbreviations: Abbreviations:

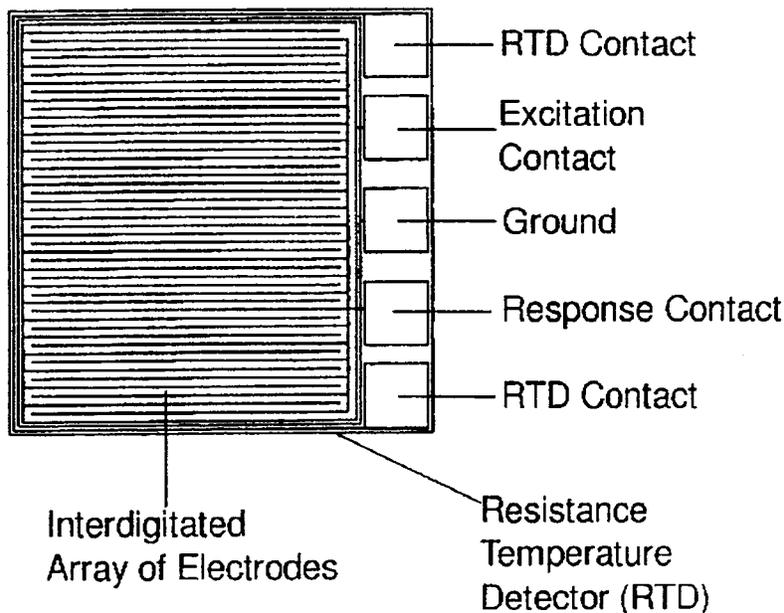


FIG. 2 Interdigitated Electrodes

atomic motion in the case of ions) of the material. In temperature regions where significant changes occur, permittivity increases with increasing temperature (at a given frequency) or with decreasing frequency (at constant temperature). A maximum is observed for the loss factor in cases where dipole motions dominate over ionic movement.³

5.2.2 Track the reaction in polymerization and curing reactions. This may be done under either isothermal or nonisothermal conditions. Increasing molecular weight or degree of crosslinking normally leads to decreases in conductivity.⁴

5.2.3 Determine diffusion coefficients of polar gases or liquids into polymer films on dielectric sensors. The observed change in permittivity typically is linear with diffusant concentration, as long as the total concentration is relatively low.⁵

5.3 This procedure can be used, for example, to evaluate by comparison to known reference materials:

5.3.1 The mix ratio of two different organic materials. This may be determined either through use of permittivity or loss factor values. In early studies, permittivity has been found to be linear with concentration.⁶

5.3.2 The degree of phase separation in multicomponent systems.

5.3.3 The filler type, amount, pretreatment, and dispersion.

5.4 This test method can be used for observing annealing and the submelting point crystallization process.

5.5 This test method can be used for quality control, specification acceptance, and process control.

6. Interferences

6.1 Since small quantities of specimen are used for these measurements, it is necessary that the specimens be both homogeneous and representative.

6.2 Specimens that contain conductive inhomogeneities, such as graphite fibers or carbon black, may short the dielectric sensing electrodes causing erroneous readings. Conductive particles should be removed or filtered from the sensing area using a porous medium, such as glass cloth.

6.3 Dielectric measurements require the specimen to be in intimate contact with the sensor electrodes and complete coverage of the specimen over the electrodes is essential. For solid specimens, extreme care shall be taken to ensure that electrodes are in contact with the specimen over the entire surface of the electrodes.

6.4 Dielectric measurements are sensitive to moisture in the specimen environment. Close control of humidity in the specimen chamber and elimination of condensed water vapor (liquid or ice) below the dew point is required for reproducible measurements.

³ Hedvig, P., "Dielectric Spectroscopy of Polymers," New York, Wiley and Sons (1977).

⁴ Senturia and Sheppard, "Dielectric Analysis of Thermoset Cure," *Advances in Polymer Science* (1985).

⁵ Day, D. R., "Moisture Monitoring at the Polyimide-S102 Interface Using Microdielectric Sensors," *Polyimides: Materials, Chemistry, and Characterization*, ed. C. Feger, Elsevier, Amsterdam (1989).

⁶ Day, D.R., and Lee, H.L., "Analysis and Control of Polyester Part to Part Variations," *Proceedings of SPI* (1991).

7. Apparatus

7.1 *Specimen Chamber*, capable of holding the solid or viscous liquid specimen in contact with the sensing electrodes. Some compressive force on the parallel plates may be necessary to ensure constant, uniform contact between the plates and solid samples. Similarly, some force may be needed to ensure proper contact between a solid specimen and the single-sided, interdigitated electrodes.

7.1.1 Some instruments provide a remote sensing capability. The remote sensor, generally an interdigitated electrode pattern on an insulating, inert substrate, can be completely immersed in the liquid specimen and is attached to the measuring instrument by an electrically insulating cable.

7.2 *Oscillatory (AC) voltage source*, capable of applying a time varying, sinusoidal voltage through the electrodes to the specimen under test. A source of fixed frequency and amplitude may be used but the ability to scan a range of frequencies and vary the excitation amplitude is recommended.

7.3 *Detector(s)*, capable of measuring independent experimental parameters, such as specimen temperature, and dependent parameters, such as response amplitude and phase angle. Temperature should be measurable with an accuracy of $\pm 1^\circ\text{C}$; response amplitude and phase angle to $\pm 5\%$.

NOTE 2—Different instruments measure different dependent parameters. Most measure response amplitude and phase angle (or phase shift) and from these, along with the known geometry of the electrodes, all of the dielectric properties of the specimen can be calculated. Some of the methods used for measurement and some of the mathematical algorithms involve complex and proprietary functions well beyond the scope of this test method.

7.4 *Temperature Controller, Oven, or Heated Press*, capable of heating or cooling the specimen and sensor electrodes at a constant rate or maintaining an isothermal condition.

7.5 A supply of dry nitrogen, vacuum, or other protective gas may be used with certain specimens.

NOTE 3—If measurements are 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. Test Specimen

8.1 Samples usually are analyzed on an "as-received" basis. Should some thermal or mechanical treatment, such as grinding or sieving, be applied to the sample prior to analysis, it shall be indicated in the report.

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

8.3 The test specimen must cover the entire surface of parallel plate electrodes and shall be of uniform thickness. For viscous liquids or solids, which may melt during the test, the distance between the parallel plates shall be fixed and not allowed to change because of changes in the viscosity of the specimen. Changes due to thermal expansion or contraction normally are neglected (provided the specimen does not pull away from the electrodes). For parallel plate cells, the optimum test specimen thickness actually depends on the dielectric properties of the specimen, sensor size, and choice of independent variables (temperature, frequency); however, a minimum thickness of 0.1 mm is recommended.

8.4 For interdigitated electrodes, the thickness of the test specimen, whether liquid or solid, should be at least 1.5 times the electrode spacing and fully cover the interdigitated electrode array. Any thickness change during the experiment may be neglected providing the preceding minimum thickness is maintained.

9. Calibration

9.1 Temperature calibration may be obtained by observing the position of the loss peak of a reference material. This calibration should be performed at either end of the temperature range of interest, in a temperature scanning mode at the same heating (or cooling) rate that will be used for the actual test. All other conditions of the calibration and test should be either identical or as close as practically possible. Possible reference materials and a standard calibration method may be found in Test Method E 2038.

10. Precautions

10.1 Toxic or corrosive effluents, or both, may be released when heating the specimen near its decomposition point and can be harmful to personnel or to the apparatus.

10.2 Care should be taken so that contact or adhesion of the specimen to the sensor is not lost during the course of the measurement.

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

10.4 Some components of the testing 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.

11. Procedure

11.1 Where temperature is to be the independent variable, do the following:

11.1.1 Set the test frequency to a value from 1 mHz to 100 kHz. Frequencies outside of this range may be used but the procedures covered in this test method may no longer be valid. It is up to the user to ensure the validity of the resulting data.

11.1.2 Vary the temperature of the test specimen from the lowest to the highest temperature of interest while measuring its permittivity and loss factor.

NOTE 4—Preferably, tests conducted over a temperature range should be performed in incremental steps or at a rate slow enough to allow temperature equilibrium throughout the entire specimen. The time to reach equilibrium will depend upon the mass of the particular specimen and the sensor arrangement. Temperature program rates of 1 to 3 °C/min or 2 to 5 % step intervals held for 3 to 5 min have been found suitable. The effect of heating rate may be observed by running specimens at two or more rates and comparing the permittivity and loss factors obtained.

NOTE 5—The accuracy required of the temperature measurement will depend upon the rate of change of dielectric properties with temperature of the material being investigated. In transition regions, experience has indicated that the specimen temperature should be read to the nearest °C.

11.2 Where frequency is to be the independent variable, do the following:

11.2.1 Fix the temperature at the desired value.

11.2.2 Vary the frequency applied to the test specimen as desired while measuring the permittivity and loss factor.

11.2.3 Examine duplicate specimens and report the mean results.

12. Calculation

12.1 Most dielectric instrumentation have internal, complex, proprietary software that determines the dielectric properties. From the amplitude and phase shift of the measured current relative to the applied voltage, the parallel capacitance (C_p) and parallel resistance (R_p) is determined (see Test Method D 150). The conversion from parallel capacitance and resistance to permittivity and loss factor is geometry and sensor dependent. Simple calculation methods for permittivity (ϵ') and loss factor (ϵ'') are as follows for a parallel plate geometry and for a generic interdigitated electrode.

Parallel plate:

$$\epsilon' = \frac{C_p \times d}{\epsilon_0 \times A} \quad \epsilon'' = \frac{d}{R_p \times \epsilon_0 \times A \times \omega} \quad (1)$$

Interdigitated electrode:

$$\epsilon' = \frac{(C_p - C_{sub}) \times d}{\epsilon_0 \times A} \quad \epsilon'' = \frac{d}{R_p \times \epsilon_0 \times A \times \omega} \quad (2)$$

NOTE 6—The equivalent area (A) of an interdigitated electrode may be estimated as ($E_s \cdot M_L$). The equivalent plate separation (d) may be estimated as E_s ; therefore, d/A can be estimated as $1/M_L$.

NOTE 7—The interdigitated electrode equation is only an approximation and at that is best used when the substrate is an excellent insulator and the substrate thickness, T_s , is at least 1.5 times the electrode separation ($T_s > 1.5 \cdot E_s$). The most accurate permittivity sensing interdigitated electrode sensors use conductive ground planes located underneath the substrate at distances much less than T_s . Proprietary but much more accurate conversion functions are utilized in some commercial instruments.

NOTE 8—The substrate capacitance (C_{sub}) is not the capacitance measured in air. The capacitance measured in air is the sum of C_{sub} and the capacitance due to coupling through the air. C_{sub} is best determined by making both an air measurement and a measurement in a material of known dielectric constant. C_{sub} may then be determined by the following equation:

$$C_{sub} = \frac{\epsilon'_{cal} \cdot C_{air} - C_{cal}}{\epsilon'_{cal} - 1} \cdot \frac{d}{A} = \frac{\epsilon'_{cal} \cdot \epsilon_0}{(C_{cal} - C_{sub})} = \frac{\epsilon_0}{(C_{air} - C_{sub})} \quad (3)$$

where:

ϵ'_{cal} = the permittivity of the calibration material,

C_{cal} = the measured capacitance of the calibration material, and

C_{air} = the capacitance measured in air.

The ratio of d/A may then be estimated by the second equation shown above using the determined C_{sub} value.

12.2 *Calculating Conductivity*—Under certain conditions the ionic conductivity may be calculated from the loss factor. The measured loss factor generally is the sum of a dipole loss contribution and an ionic conductivity contribution. If, and only if, the dipole contribution is negligible, then:

$$\text{Ionic conductivity} = \epsilon' \cdot \epsilon_0 \cdot \omega \quad (\text{see Terminology E})$$

12.2.1 For typical organic materials and typical electrode geometries, this equation is valid if $\epsilon' > 5$ and $\epsilon'' < 100$. This results from the fact that dipole contributions in typical materials rarely exceed an ϵ'' value of 3 usually are much less. If ϵ' exceeds 100, then most likely, electrode polarization is influencing both the permittivity and loss factor measurements. These assumptions are not true when electrode separation is

less than 0.0005 cm, artificial insulating layers are placed between the electrodes and the sample, and the material has a dielectric constant greater than water (80). The following are two methods for determining ionic conductivity levels.

12.2.2 *Single Frequency Data*—Using the above assumption, conductivity may be calculated from single frequency data as follows:

If $\epsilon' < 5$ AND $\epsilon'' < 100$, then:

Ionic Conductivity = $(\epsilon'' \cdot \epsilon_0 \cdot \omega)$

If $\epsilon' < 5$ OR $\epsilon'' > 100$, then:

Conductivity may not be determined at that frequency; try another frequency.

12.2.3 *Multifrequency Data*—If multifrequency data is available, preferably over many decades of frequency, then plot the quantity as follows:

$(\epsilon' \cdot \epsilon'' \cdot \omega)$ versus time or temperature for all frequencies.

Regions where frequencies superimpose represent true ionic conductivity values. Regions of non-superposition represent either dipole dominance or electrode polarization. Regions of non-superposition should not be labeled as ionic conductivity.

13. Report

13.1 Report the following information:

13.1.1 Complete identification and description of the material tested including name, stock or code number, data made, form source, and so forth.

13.1.2 Date of test.

13.1.3 Description of the instrument used for test.

13.1.4 Description of all calibration procedures.

13.1.5 Identification of specimen environment.

13.1.6 Details of the preconditioning.

13.1.7 The temperature program including linear ramp rates, hold temperatures and times.

13.1.8 Frequencies used and excitation amplitude.

13.1.9 Table of data and results, including number of samples used.

13.1.10 Any equations used to calculate values.

13.1.11 A plot of the permittivity, loss factor, conductivity, or resistivity versus temperature, frequency, or time.

13.1.11.1 Permittivity, loss factor, ionic conductivity, and resistivity normally are plotted on the ordinate (except for Cole-Cole plots, 13.1.11.3) with upward deflections indicating increases in those values. Permittivity normally should be plotted on a linear scale while loss factor, conductivity, and resistivity are plotted on a \log_{10} scale. The ordinate should be labeled clearly with title and unit of measurement.

13.1.11.2 Temperature, frequency, and time should be plotted on the abscissa, increasing from left to right. The abscissa should be labeled clearly with title and units of measurement.

13.1.11.3 Cole-Cole plots consist of loss factor on the ordinate and permittivity on the abscissa, both plotted on a linear scale. Both axes should be labeled clearly with title and units of measurement.

13.1.11.4 Transition temperatures are taken, when possible, from the peak values of dipole-response-dominated loss factors. Care should be taken not to assign transitions to loss factor peaks caused by electrode polarization. In cases where the ϵ'' peak is obscured by ionic conductivity influence, the transition point may be estimated by the midpoint in ϵ' between the low frequency (relaxed) permittivity (ϵ_0') and the high frequency (unrelaxed) permittivity (ϵ_∞). (see Terminology E 1142).

13.1.11.5 Wherever possible, each thermal or frequency effect should be identified and supplementary supporting evidence reported.

13.1.11.6 The specific dated version of this test method used.

14. Precision and Bias

14.1 An interlaboratory test is planned for 2005–2006 to provide precision and bias information. Anyone wishing to participate in this interlaboratory test should contact the E37 Staff Manager at ASTM International headquarters.

15. Keywords

15.1 conductivity; dielectric constant; dielectric measurement; dielectric properties; dissipation; loss factor; permittivity

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