Designation: E 2347 – 04

Standard Test Method for Indentation Softening Temperature by Thermomechanical Analysis[1]

This standard is issued under the fixed designation E 2347; 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 is applicable to materials that soften upon heating to a modulus less than 6.0 MPa. This test method describes the determination of the temperature at which the specific modulus of either 6.65 (Method A) or 33.3 MPa (Method B) (equivalent to Test Method D 1525) of a test specimen is realized by indentation measurement using a thermomechanical analyzer as the test specimen is heated. This temperature is identified as the indentation softening temperature. The test may be performed over the temperature range of ambient to 300°C.

NOTE 1—This test method is intended to provide results similar to those of Test Method D 1525 but is performed on a thermomechanical analyzer using a smaller diameter indenting probe. Equivalence of results to those obtained by Test Method D 1525 has been demonstrated on a limited number of materials. Until the user demonstrates equivalence, the results of this Test Method shall be considered to be independent and unrelated to those of Test Method D 1525.

1.2 This test method is not recommended for ethyl cellulose, poly (vinyl chloride), poly (vinylidene chloride) and other materials having a large measurement imprecision (see Test Method D 1525 and sections 5.3 and 14.1.2).

1.3 Electronic instrumentation or automated data analysis and reduction systems or treatments equivalent to this test method may be used.

NOTE 2—Since all electronic data treatments are not equivalent, the user shall verify equivalency to this test method.

1.4 SI values are the standard.

1.5 There is no ISO standard equivalent to this test method.

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]

D 1525 Test Method for Vicat Softening Temperature of Plastics

E 473 Terminology Relating to Thermal Analysis

E 1142 Terminology Relating to Thermophysical Properties

E 1363 Test Method for Temperature Calibration of Thermomechanical Analyzers

E 2113 Test Method for Length Change Calibration of Thermomechanical Analyzers

E 2206 Test Method for Force Calibration of Thermomechanical Analyzers

3. Terminology

3.1 *Definitions*—Specific technical terms used in this standard are defined in Terminologies E 473 and E 1142.

3.2 *penetration softening temperature,* [°C], *n*—the temperature at which a test specimen has a modulus of either 6.65 or 33.3 MPa as measured in penetration.

4. Summary of Test Method

4.1 The modulus of a material may be determined by the indentation (penetration) of a circular, flat tipped probe. The relationship between modulus of a material (stress divided by strain) and penetration depth is given by:

$$E = 3\,F / (4\,D\,d) \quad (1)$$

where:

E = modulus, MPa,
F = force, N,
D = diameter of a circular, flat tipped probe, mm, and
d = penetration depth, mm.

NOTE 3—Note the identity Pa = N / m^2

[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 Thermal Analysis and Rheology Methods.

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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.

4.2 Some materials soften upon heating. For such materials, the modulus may be determined by penetration as the sample is heated. This test method identifies the temperature at which the modulus of the specimen is determined to be 6.65 MPa (Method A) or 33.3 MPa (Method B).

4.3 Specifically, a test specimen is tested in penetration using a circular, flat tipped probe. A known stress is applied to the center of a test specimen as it is heated at a constant rate from ambient temperature to the upper temperature limit for the material. The penetration (that is, deflection) of the test specimen is recorded as a function of temperature. The temperature at which the modulus of the specimen is determined to be 6.65 MPa (Method A) or 33.3 MPa (Method B) is determined to be the penetration softening temperature.

5. Significance and Use

5.1 Data obtained by this test method shall not be used to predict the behavior of materials at elevated temperatures except in applications in which the conditions of time, temperature, method of loading, and stress are similar to those specified in the test.

5.2 This standard is particularly suited for quality control and development work. The data are not intended for use in design or predicting endurance at elevated temperatures.

5.3 Ruggedness testing indicates that some materials, such as poly (vinyl chloride) exhibit substantially greater imprecision than that described in Section 14 for "well behaved" materials.

6. Apparatus

6.1 A thermomechanical analyzer consisting of:

6.1.1 *Rigid Specimen Holder*, of inert, low expansivity material (< 1 μm m^{-1} $°C^{-1}$) to center the specimen in the furnace and to fix the specimen to mechanical ground.

6.1.2 *Rigid Penetration Probe*, of inert, low expansivity material (< 1 μm m^{-1} $°C^{-1}$) that contacts the specimen with an applied compression force (see Fig. 1). The tip shall be 0.1 to 1.0 mm in diameter, free of burrs and be perpendicular to the axis of the probe. The tip shall protrude at least 0.1 mm from the end of the probe.

6.1.3 *Deflection Sensing Element*, having a linear output over a minimum range of 5 mm to measure the displacement of the rigid penetration probe (see 6.1.2) to within ± 0.1 μm.

6.1.4 *Programmable Force Transducer*, to generate a constant force (± 2.5 %) between 0.05 and 1.0 N that is applied to the specimen through the rigid penetration probe (see 6.1.2).

NOTE 4—Other forces may be used but shall be reported.

6.1.5 *Temperature Sensor*, that can be positioned reproducibly in close proximity to the specimen to measure its temperature over the range of 25 to 300°C to ± 0.1°C.

6.1.6 *Temperature Programmer and Furnace*, capable of temperature programming the test specimen from ambient to 300°C at a linear rate of at least 2.0 ± 0.2°C/min.

6.1.7 *Means of Providing a Specimen Environment*, of inert gas at a purge rate of 50 mL/min ± 5 %.

NOTE 5—Typically, inert purge gas that inhibits specimen oxidation are 99.9+ % pure nitrogen, helium or argon. Dry gases are recommended for all experiments unless the effect of moisture is part of the study.

6.1.8 *Recording Device*, to record and display the experimental parameters of penetration on the *Y*-axis (ordinate) to a sensitivity of ± 0.1 μm and of temperature on the *X*-axis (abscissa) to a sensitivity of ± 0.1°C.

6.2 *Calipers, Micrometer*, or other length measuring device capable of a length measurement of up to 2 mm with a precision of ± 1 μm.

7. Hazards

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

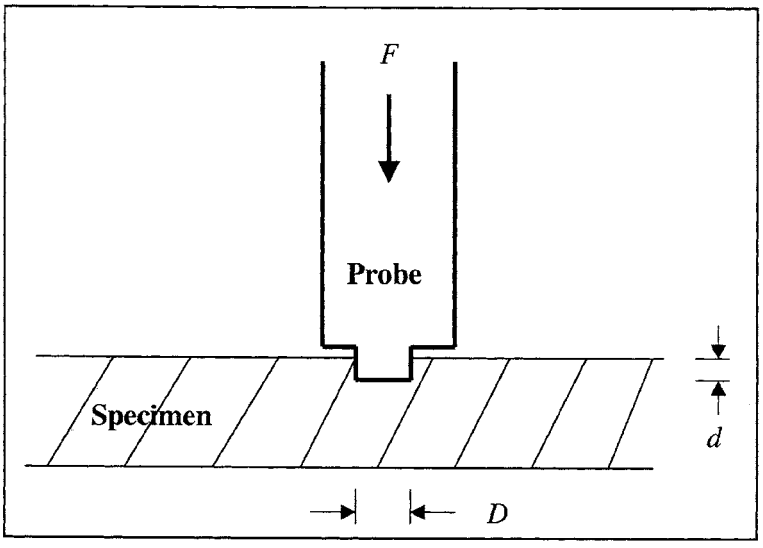

FIG. 1 Penetration Probe