Designation: D5510 – 94 (Reapproved 2001)

Standard Practice for Heat Aging of Oxidatively Degradable Plastics[1]

This standard is issued under the fixed designation D5510; 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 practice is intended to define the exposure conditions of plastics at various temperatures when exposed solely to hot air for extended periods of time. Only the procedures for heat exposure are specified, not the test method or specimen. The effect of heat on any particular property may be determined by selection of the appropriate test method and specimen; however, it is recommended that Practice D3826 be used to determine the embrittlement endpoint, which is defined as that point in the history of a material when 75 % of the specimens tested have a tensile elongation at break of 5 % or less at an initial strain rate of 0.1 mm/mm min.

1.2 This practice should be used as a guide for comparing the thermal-aging characteristics of materials as measured by the change in some property of interest (that is, embrittlement by means of loss of elongation). It is very similar to Practice D3045 but is intended for use in evaluating plastics designed to be oxidized easily after use. The exposure times used for this practice will be significantly shorter than those used for Practice D3045.

1.3 The type of oven used can affect the results obtained from this practice. The user can use one of two methods for oven exposure. The results based on one method should not be mixed with those based on the other.

1.3.1 *Procedure A: Gravity-Convection Oven*—Recommended for film specimens having a nominal thickness not greater than 0.25 mm (0.010 in.).

1.3.2 *Procedure B: Forced-Ventilation Oven*—Recommended for specimens having a nominal thickness greater than 0.25 mm (0.010 in.).

1.4 This practice recommends procedures for comparing the thermal aging characteristics of materials at a single temperature. Recommended procedures for determining the thermal aging characteristics of a material at a series of temperatures for the purpose of estimating time to a defined property change at some lower temperature are also described. This practice does not predict thermal aging characteristics where interactions between stress, environment, temperature, and time control failure.

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

NOTE 1—There is no ISO standard that is equivalent to this standard.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D618 Practice for Conditioning Plastics for Testing
D883 Terminology Relating to Plastics
D1870 Practice for Elevated Temperature Aging Using a Tubular Oven
D2436 NO TITLE
D3045 Practice for Heat Aging of Plastics Without Load
D3593 NO TITLE
D3826 Practice for Determining Degradation End Point in Degradable Polyethylene and Polypropylene Using a Tensile Test
E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

3. Terminology

3.1 *Definitions*—The definitions used in this practice are in accordance with Terminology D883.

4. Significance and Use

4.1 The correlation of results obtained from this practice to actual disposal environments (for example, composting) has not been determined, and, as such, the results should be used only for comparative and ranking purposes.

4.2 Degradable plastics exposed to heat may be subject to many types of physical and chemical changes. The severity of the exposures in both time and temperature determines the extent and type of change that occurs. Short exposure times at elevated temperatures generally serve to shorten the induction period of oxidatively degradable plastics during which the

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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.

depletion of antioxidants and stabilizers occurs. Physical properties, such as tensile and impact strength and elongation and modulus, may change during this induction period; however, these changes are generally not due to molecular-weight degradation, but are merely a temperature-dependent response, such as increased crystallinity or loss of volatile material, or both.

4.3 Generally, short exposures at elevated temperatures may drive out volatiles such as moisture, solvents, or plasticizers; relieve molding stresses; advance the cure of thermosets; increase crystallinity; and cause some change in color of the plastic or coloring agent, or both. Normally, additional shrinkage should be expected with a loss of volatiles or advance in polymerization.

4.4 Some plastic materials such as PVC may become brittle due to loss of plasticizers or to molecular breakdown of the polymer. Polypropylene and its copolymers tend to become very brittle as molecular degradation occurs, whereas polyethylene tends to become soft and weak before it embrittles with resultant loss in tensile strength and elongation.

4.5 Embrittlement of a material is not necessarily commensurate with a decrease in molecular weight. Test Method D3593 should be used to characterize any molecular-weight changes that may have occurred during thermal exposure.

4.6 The degree of change observed will depend on the property measured. Different properties may not change at the same rate. In most cases, ultimate properties, such as break strength or break elongation, are more sensitive to degradation than bulk properties such as modulus.

4.7 Effects of exposure may be quite variable, especially when samples are exposed for long intervals of time. Factors that affect the reproducibility of data are the degree of temperature control of the enclosure, humidity of the oven, air velocity over the specimen, and exposure period. Errors in exposure are cumulative with time. Certain materials are susceptible to degradation due to the influence of humidity in long-term tests. Materials susceptible to hydrolysis (that is, hydrolytically degradable plastics) may undergo degradation when subjected to long-term thermal tests due to moisture.

4.8 It should not be inferred that comparative material ranking is undesirable or unworkable. On the contrary, this practice is designed to provide information that can be used for such comparative purposes after appropriate physical property tests are performed following exposure. However, since it does not account for the influence of stress or environment that is involved in most real life applications, the information obtained from this practice must be used cautiously by the designer, who must inevitably make material choices using additional information, such as moisture, soil, and mechanical-action effects that are consistent with the requirements of the particular application.

4.9 It is possible for many temperature indices to exist, in fact, one for each failure criterion. Therefore, for any application of the temperature index to be valid, the thermal-aging program must duplicate the intended exposure conditions of the end product. If the material is exposed in the end use in a manner not evaluated in the aging program, the temperature index thus derived is not applicable to the use of the material.

4.10 In some situations, a material may be exposed to one temperature for a particular period of time, followed by exposure to another temperature for a particular period of time. This practice can be used for such applications. The heat-aging curve of the first temperature should be derived, followed by derivation of the heat-aging curve for the second temperature after exposure of samples to the first temperature.

4.11 There can be very large errors when Arrhenius plots or equations based on data from experiments at a series of temperatures are used to estimate time to produce a defined property change at some lower temperature. This estimate of time to produce the property change or failure must always be accompanied by a 95 % confidence interval for the range of times possible based on the calculation or estimate.

5. Apparatus

5.1 *Provisions for Conditioning*, at specified standard conditions.

5.2 *Oven*.

5.2.1 *Procedure A: Gravity-Convection Oven*—Recommended for film specimens having a nominal thickness not greater than 0.25 mm (0.010 in.).

5.2.2 *Procedure B: Forced-Ventilation Oven*—Recommended for specimens having a nominal thickness greater than 0.25 mm (0.010 in.). When it is necessary to avoid contamination among specimens or materials, the tubular-oven procedure, such as specified in Practice D1870, may be desirable. Oven apparatus shall be in accordance with Specifications D2436 and E145, Type 1A and Type IIB, with 50 ± 10 air changes/h and the requirements for uniformity extended to include the range of test temperatures. Recording instrumentation to monitor the temperature and humidity of exposure is recommended.

5.3 *Specimen Rack*—A specimen rack or frame of suitable design to allow ready air circulation around the specimens.

5.4 *Test Equipment*, in accordance with appropriate ASTM procedures to determine the selected property or properties.

6. Test Specimens

6.1 The number and type of test specimens required shall be in accordance with the ASTM test method for the specific property to be determined; this requirement should be met at each time and temperature selected. Unless otherwise specified or agreed upon by all interested parties, expose a minimum of three replicates of each material at each time and temperature selected.

6.2 The specimen thickness should be comparable to but no greater than the minimum thickness of the intended application.

6.3 The method of specimen fabrication should be the same as that for the intended application.

6.4 All test specimens for a series of temperatures should be of the same age, preferably from the same manufacturing run and date.

7. Conditioning

7.1 Conduct initial tests in the standard laboratory atmosphere as specified in Practice D618, and with the specimens