Designation: D 5510 – 94 (Reapproved 2001)

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

This standard is issued under the fixed designation D 5510; 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 D 3826 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 D 3045 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 D 3045.

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:*

D 618 Practice for Conditioning Plastics for Testing[2]

D 883 Terminology Relating to Plastics[2]

D 1870 Practice for Elevated Temperature Aging Using a Tubular Oven[3]

D 2436 Specification for Forced-Convection Laboratory Ovens for Electrical Insulation[4]

D 3045 Practice for Heat Aging of Plastics Without Load[5]

D 3593 Test Method for Molecular Weight Averages and Molecular Weight Distribution of Certain Polymers by Liquid Size-Exclusion Chromatography (Gel Permeation Chromatography GPC) Using Universal Calibration[5]

D 3826 Practice for Determining Degradation End Point in Degradable Polyolefins Using a Tensile Test[5]

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens[6]

3. Terminology

3.1 *Definitions*—The definitions used in this practice are in accordance with Terminology D 883.

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

---

[1] This practice is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.96 on Environmentally Degradable Plastics.

Current edition approved Feb. 15, 1994. Published April 1994.

[2] *Annual Book of ASTM Standards*, Vol 08.01.

[3] Discontinued; see *1997 Annual Book of ASTM Standards*, Vol 08.01.

[4] *Annual Book of ASTM Standards*, Vol 10.01.

[5] *Annual Book of ASTM Standards*, Vol 08.02.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

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 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 D 3593 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 D 1870, may be desirable. Oven apparatus shall be in accordance with Specifications D 2436 and E 145, 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 D 618, and with the specimens conditioned in accordance with the requirements of the ASTM test method for determining the specific property or properties required.

7.2 When required, the conditioning of specimens following exposure at elevated temperature and prior to testing, unless otherwise specified, shall be in accordance with Practice D 618.

8. Procedure

8.1 Select Procedure A or B, depending on the type of ovens available (5.2).

8.2 When tests at a single temperature are used, all materials must be exposed at the same time in the same device. Use a sufficient number of replicates of each material for each exposure time so that results of tests used to characterize the material property can be compared by analysis of variance or a similar statistical data analysis procedure.

8.3 Use a minimum of four exposure temperatures when testing at a series of temperatures in order to determine the relationship between a defined property change and temperature. The following procedures are recommended for selecting exposure temperatures:

8.3.1 The lowest temperature should produce the desired level of property change or product failure in approximately six months. The next higher temperature should produce the same level of property change or product failure at approximately one month.

8.3.2 The third and fourth temperatures should produce the desired level of property change or product failure in approximately 1 week and 1 day, respectively.

8.3.3 Select the exposure temperatures from Table 1 when possible. If the suggested heat aging times in 8.3.1 and 8.3.2 are followed, the exposure time Schedules A, B, C, D, and E may be used.

8.3.4 The purpose of Table 1, giving time schedules at specific temperatures, is to show a typical heat aging schedule for a particular property of some material. In practice, it is often difficult to estimate the effect of heat aging before obtaining the test data. It is therefore usually necessary to start only the short-term heat aging at one or two temperatures until data are obtained to be used as a basis for selecting the remainder of the heat aging temperatures. Since the temperature dependency of oxidatively degradable plastics can vary considerably, Table 1 should be used only as a starting guide. To obtain more accurate data, exposure times and temperatures intermediate to those given in Table 1 can be used.

NOTE 2—The activation energy of certain materials at higher temperatures may be different than the activation energy of the material at lower temperatures. Caution should be used when developing Table 1 relationships on the basis of data from only the highest aging temperature.

8.4 Test one set of nonexposed specimens for the selected property in accordance with the appropriate test method, including provisions for conditioning.

8.5 Mount the test specimens in specimen racks or frames, and place the frames in the oven such that both sides of the specimens are exposed to air flow. In order to minimize any effects from temperature variation within the oven, it is recommended that the frames or specimens be repositioned periodically.

8.6 Expose the remaining sets of specimens for the selected time intervals at the prescribed temperatures. Following exposure, condition these specimens in accordance with established procedure, and then test. If an effect of aging without heat is anticipated, condition and test a parallel set of aged unexposed specimens.

TABLE 1 Suggested Temperatures and Exposure Times for the Determination of Heat Aging of Oxidatively Degradable Plastics[A]

Suggested Exposure Temperature (°C)	Logarithm Temperature (°C)	Estimated Failure Time (h) at 90°C				
		1–10	11–24	25–48	49–96	97–192
30	1.477	A				
40	1.602	B	A			
50	1.699	C	B	A		
60	1.778	D	C	B	A	
70	1.845	E	D	C	B	A
80	1.903		E	D	C	B
90	1.954			E	D	C
100	2.000				E	D
110	2.041					E

[A] Suggested exposure times: A-2, 4, 8, 16, 24, 32 weeks; B-3, 6, 12, 24, 36, 48 days; C-1, 2, 4, 8, 12, 16 days; D-8, 16, 32, 64, 96, 128 h; and E-2, 4, 8, 16, 24, 32 h.

9. Calculation

9.1 When materials are compared at a single temperature, use analysis of variance to compare the mean of the measured property data for each material at each exposure time. Use the results from each replicate of each material being compared for the analysis of variance. It is recommended that the F statistic for 95 % confidence be used to determine significance for the results from the analysis of variance calculations.

9.2 When materials are being compared using a range of different temperatures, use the following procedure to analyze the data and to estimate the exposure time necessary to produce a predetermined level of property change at some temperature lower than the test temperature used. This time can be used for general ranking of materials for temperature stability or as an estimate of the maximum expected service life at the temperature selected.

9.2.1 Prepare plots of the measured property as a function of exposure time for all temperatures used. Plots should be prepared in accordance with Fig. 1 where the abscissa is a logarithmic time scale and the value of the measured property is the ordinate.

9.2.2 Use regression analysis to determine the relationship between the logarithm of exposure time and measured property. Use the regression equation to determine the exposure time necessary to produce a predetermined level of property change. An acceptable regression equation must have an r^2 of at least 80 %. A plot of the residuals (value of property retention predicted by regression equation minus actual value) versus aging time must show a random distribution. Use of

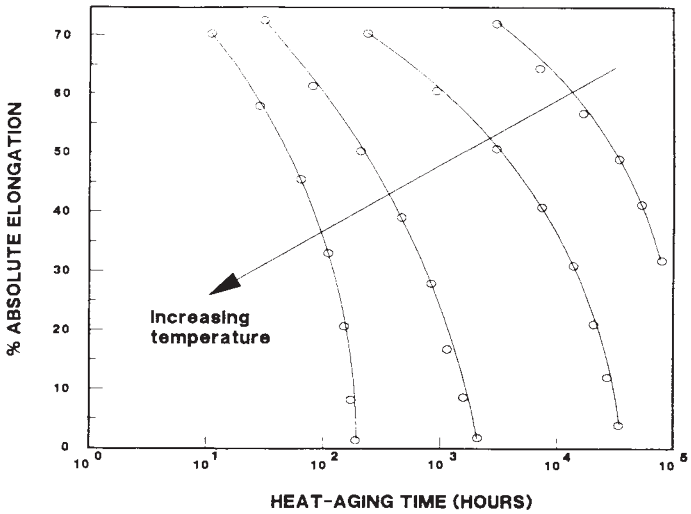

FIG. 1 Typical Heat-Aging Curves—% Absolute Elongation Versus Time (for Example only)

graphical interpretation to estimate the exposure time necessary to produce the predetermined level of property change is not recommended.

9.2.3 Plot the logarithm of the calculated times to produce the predetermined level of property change (determined by the acceptable regression equation) as a function of the reciprocal of the absolute temperature (1/T in °K) of each exposure used. A typical plot of this type (known as an Arrhenius plot) is shown in Fig. 2. Use regression analysis to determine the equation defining the log time/reciprocal temperature relationship. An acceptable regression equation must meet the requirements described in 9.2.2.

9.2.4 Use the equation for the log of the time to produce the defined property change as a function of the reciprocal absolute temperature to determine the time to produce this property change at a preselected temperature agreed upon by all interested parties.

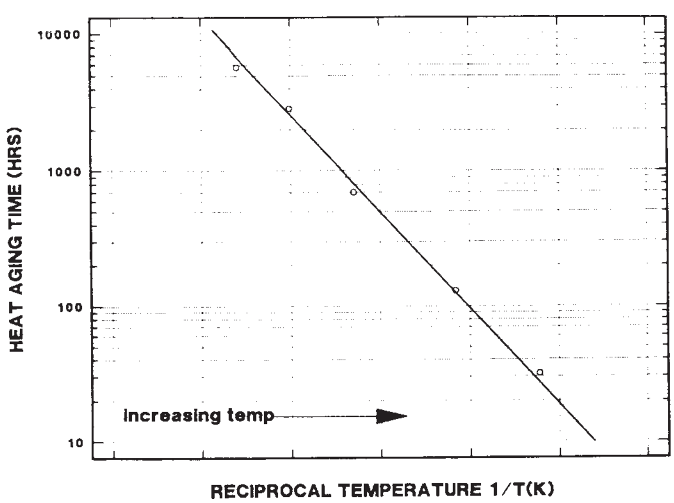

FIG. 2 Typical Arrhenius Plot—Heat Aging Time Versus Reciprocal Temperature (for Example only)

9.2.5 Calculate the 95 % confidence interval for the time to produce the defined property change using the "standard error" from the regression analysis for the estimated time for the selected temperature. This is readily available from most software packages that do regression analysis. This 95 % confidence interval can be determined by taking the calculated time $\pm$ (2 $\times$ standard error for estimated time).

10. Report

10.1 Include the following in the test report:

10.1.1 Material, type, and thickness of plastic subjected to exposure, along with specimen preparation procedure;

10.1.2 Pre-conditioning and post-conditioning procedures followed;

10.1.3 Test methods used for evaluation of each property;

10.1.4 Observations of any visible changes in the test specimens;

10.1.5 Procedure used, A or B;

10.1.6 Exposure temperatures used, and times of exposure at each temperature;

10.1.7 Humidity of oven during exposure;

10.1.8 Linear velocity of air flow within the oven(s);

10.1.9 Results from analysis of variance, comparing the results for each material for each exposure time when a single temperature is used; and

10.1.10 When a series of temperatures is used to expose materials, the following shall be reported for each material tested:

10.1.10.1 Graphs derived in accordance with 9.2.1 and 9.2.3,

10.1.10.2 Regression equations for property change as a function of exposure time for each temperature used,

10.1.10.3 Regression equation for time to produce a defined property change as a function of reciprocal absolute temperature,

10.1.10.4 Estimated time to produce the defined property change at the selected temperature for each material tested, and

10.1.10.5 The 95 % confidence interval for times to produce the defined property change at the selected temperature (calculated in accordance with 9.2.5) for each material tested.

11. Precision and Bias

11.1 No statement of precision and bias is applicable to this practice. However, the precision and bias associated with different test methods and analytical procedures used to study exposed samples must be taken into account in interpreting the data obtained from methods used in conjunction with this practice.

12. Keywords

12.1 age; degradable; embrittlement; oven; oxidation