Designation: D 3045 – 92 (Reapproved 2003)

# Standard Practice for Heat Aging of Plastics Without Load[1]

This standard is issued under the fixed designation D 3045; 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 for testing the resistance of plastics to oxidation or other degradation when exposed solely to hot air for extended periods of time. Only the procedure for heat exposure is 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.

1.2 This practice should be used as a guide to compare thermal aging characteristics of materials as measured by the change in some property of interest. 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.

1.3 This practice does not predict thermal aging characteristics where interactions between stress, environment, temperature, and time control failure occurs.

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 determine the applicability of regulatory limitations prior to use.*

NOTE 1—ISO-2578 is considered to be technically equivalent to this practice.

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 573 Test Method for Rubber—Deterioration in an Air Oven[2]

D 618 Practice for Conditioning Plastics for Testing[3]

D 883 Terminology Relating to Plastics[3]

D 1870 Practice for Elevated Temperature Aging Using a Tubular Oven[4]

D 1898 Practice for Sampling of Plastics[4]

E 145 Specifications for Gravity-Convection and Forced-Ventilation Ovens[5]

E 456 Terminology Relating to Quality and Statistics[6]

2.2 *ISO Standard:*

ISO 2578 (1974) Determination of Time-Temperature Limits After Exposure to Prolonged Action of Heat[7]

## 3. Terminology

3.1 The terminology given in Terminology D 883 and Terminology E 456 is applicable to this practice.

## 4. Significance and Use

4.1 The use of this practice presupposes that the failure criteria selected to evaluate materials (that is, the property or properties being measured as a function of exposure time) and the duration of the exposure can be shown to relate to the intended use of the materials.

4.2 Plastic materials 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 takes place. A plastic material is not necessarily degraded by exposure to elevated temperatures, but may be unchanged or improved. However, extended periods of exposure of plastics to elevated temperatures will generally cause some degradation, with progressive change in physical properties.

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, and may cause some change in color of the plastic or coloring agent, or both. Normally, additional shrinkage should be expected with loss of volatiles or advance in polymerization.

4.4 Some plastic materials may become brittle due to loss of plasticizers after exposure at elevated temperatures. Other types of plastics may become soft and sticky, either due to sorption of volatilized plasticizer or due to breakdown of the polymer.

4.5 The degree of change observed will depend on the property measured. Different properties, mechanical or electrical, may not change at the same rate. For instance, the arc

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[1] This practice is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.50 on Permanence Properties.

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[2] *Annual Book of ASTM Standards*, Vol 09.01.

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

[4] Discontinued. See 1997 *Annual Book of ASTM Standards*, Vol 08.01.

[5] *Annual Book of ASTM Standards*, Vol 14.04.

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

[7] Available from American National Standards Institute, 25 W. 43rd St., 4th Floor, New York, NY 10036.

resistance of thermosetting compounds improves up to the carbonization point of the material. Mechanical properties, such as flexural properties, are sensitive to heat degradation and may change at a more rapid rate. Ultimate properties such as strength or elongation are more sensitive to degradation than bulk properties such as modulus, in most cases.

4.6 Effects of exposure may be quite variable, especially when specimens 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 period of exposure. Errors in exposure are cumulative with time. Certain materials are susceptible to degradation due to the influence of humidity in long-term heat resistance tests. Materials susceptible to hydrolysis may undergo degradation when subjected to long-term heat resistance tests.

4.7 It is not to be inferred that comparative material ranking is undesirable or unworkable. On the contrary, this practice is designed to provide data which can be used for such comparative purposes. However, the data obtained from this practice, since it does not account for the influence of stress or environment that is involved in most real life applications, must be used cautiously by the designer, who must inevitably make material choices using additional data such as creep and creep rupture that are consistent with the requirements of his specific application.

4.8 It is possible for many temperature indexes 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 stressed in the end product in a manner not evaluated in the aging program, the temperature index thus derived is not applicable to the use of the material in that product.

4.9 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" at the lower temperature is often called the "service life." Because of the errors associated with these calculations, this time should be considered as "maximum expected" rather than "typical."

## 5. Apparatus

5.1 Provisions for conditioning at specified standard conditions.

5.2 *Oven*—A controlled horizontal or vertical air flow oven, employing forced-draft circulation with substantial constant fresh air intake is recommended. When it is necessary to avoid contamination among specimens or materials, a tubular oven method such as Practice D 1870 may be desirable. Oven apparatus shall be in accordance with Specifications E 145, Type IIB for temperature up to 70°C. For higher temperature, Type IIA is required. Provision should be made for suspending specimens without touching each other or the side of the chamber. Recording instrumentation to monitor the temperature of exposure is recommended.

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

## 6. Sampling

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.

6.2 Sampling should also be in accordance with the pertinent considerations outlined in Practice D 1898.

## 7. Test Specimens

7.1 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.

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

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

## 8. Conditioning

8.1 Conduct initial tests in the standard laboratory atmosphere as specified in Practice D 618, and with specimens conditioned in accordance with the requirements of the ASTM test method for determining the specific property or properties required.

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

8.3 If possible, avoid simultaneous aging of mixed groups of different compounds which might cause cross contamination.

## 9. Procedure

9.1 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 similar statistical data analysis procedure.

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

9.2.1 The lowest temperature should produce the desired level of property change or product failure in approximately nine to twelve months. The next higher temperature should produce the same level of property change or product failure at approximately six months.

9.2.2 The third and fourth temperatures should produce the desired level of property change or product failure in approximately three months and one month, respectively.

9.2.3 When possible, select the exposure temperatures from Table 1 (taken from the list of standard temperatures in Practice

**TABLE 1 Suggested Temperatures and Exposure Times for the Determination of Heat Aging of Plastics**

| Suggested Exposure Temperatures *t*, °C | Reciprocal Temperature in Degrees Absolute 1/*T* × 10[3] | Estimated Limiting Temperatures[A] $t_L$,° C | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | 40 | 55 | 70 | 85 | 100 | 115 | 135 | 155 | 180 | 210 | 240 |
| 50 | 3.09 | A | | | | | | | | | | |
| 70 | 2.91 | B | A | | | | | | | | | |
| 90 | 2.75 | C | B | A | | | | | | | | |
| 105 | 2.64 | D | C | B | A | | | | | | | |
| 120 | 2.54 | | D | C | B | A | | | | | | |
| 130 | 2.48 | | | D | C | B | A | | | | | |
| 155 | 2.34 | | | | D | C | B | A | | | | |
| 180 | 2.21 | | | | | D | C | B | A | | | |
| 200 | 2.11 | | | | | | D | C | B | A | | |
| 225 | 2.01 | | | | | | | D | C | B | A | |
| 250 | 1.91 | | | | | | | | D | C | B | A |
| 275 | 1.82 | | | | | | | | | D | C | B |
| 300 | 1.74 | | | | | | | | | | D | C |
| 325 | 1.67 | | | | | | | | | | | D |

[A] *Estimated Limiting Temperature*—the best estimate of limiting temperature available prior to the testing program. This may be based on prior knowledge of similar materials, and may subsequently be amended on the basis of the described short term data, as in 9.1.

Suggested Exposure Times: A—3, 6, 12, 24, 48 weeks; B—1, 3, 6, 12, 24 weeks; C—6, 12, 24, 48, 96 days; D—2, 4, 8, 16, 32 days.

D 618). If the suggested heat aging times in 9.2.1 and 9.2.2 are followed, then the exposure times (Schedules A, B, C, and D) may be used.

9.2.4 The purpose of Table 1 showing 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 test data. Therefore, it is 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. Exercise care to avoid aging at known transition temperatures since aging rates of materials usually change significantly at their transition temperatures.

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

9.4 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, likewise condition and test a parallel set or aged unexposed specimens. If necessary, establish a procedure for cooling after oven exposure.

## 10. Calculation

10.1 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.

10.2 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 temperatures 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.

10.2.1 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.

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

10.2.3 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 10.2.2.

10.2.4 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.

10.2.5 Calculate the 95 % confidence interval for 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 %

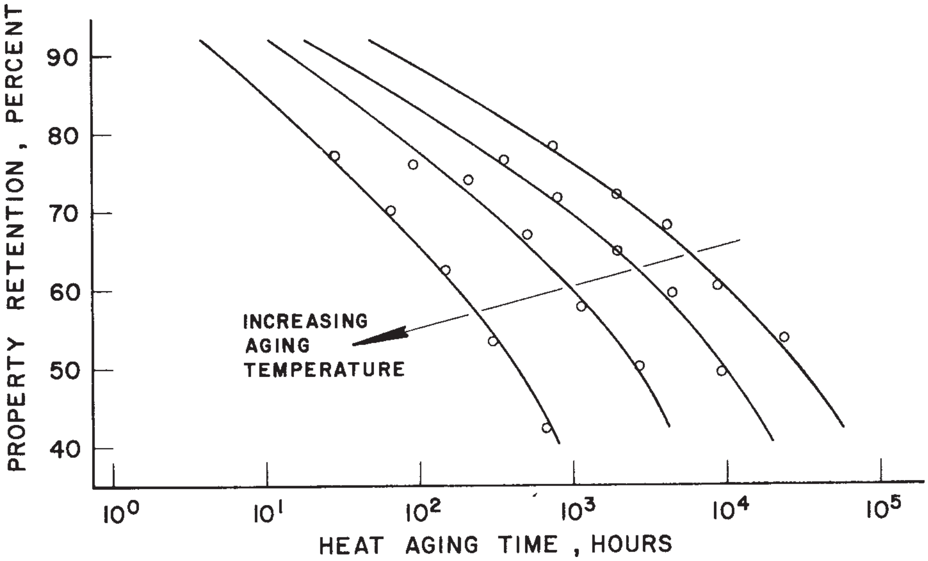


**FIG. 1 Heat Aging Curves—Property Retention Versus Aging Time**

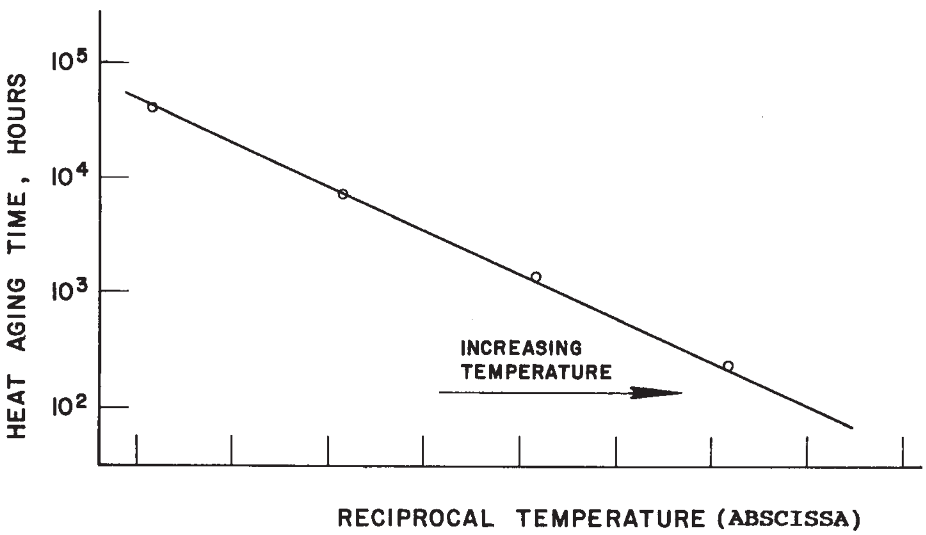


**FIG. 2 Arrhenius Plot—Time of 50 % Property Retention Versus Reciprocal of Absolute Temperature**

confidence interval can be determined by taking the calculated time ± (2 × *standard error for estimated time*).

## 11. Report

11.1 Report the following information:

11.1.1 Material and type of plastic subjected to exposure along with specimen preparation procedure,

11.1.2 Pre-conditioning and post-conditioning procedures followed,

11.1.3 Test methods utilized for evaluation of each property,

11.1.4 Observations of any visible changes in the test specimens,

11.1.5 Type of oven used,

11.1.6 Exposure temperatures utilized, and times of exposure at each temperature, and

11.1.7 Results from analysis of variance comparing the results for each material for each exposure time when a single temperature is used.

11.1.8 When a series of temperatures are used to expose materials the following shall be reported for each material tested:

11.1.8.1 Graphs derived in accordance with 10.2.1 and 10.2.3,

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

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

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

11.1.8.5 95 % confidence interval for times to produce the defined property change at the selected temperature (calculated in accordance with 10.2.5) for each material tested, and

11.1.9 The level of property change used as the basis for all calculations.

## 12. Precision and Bias

12.1 No statements of precision and bias are applicable to this practice; these are dependent upon the ASTM method for the specific properties to be determined.

## 13. Keywords

13.1 aging; exposure; heat; heat-aging; thermal-aging