INTERNATIONAL

Designation: D3045 – 18

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

This standard is issued under the fixed designation D3045; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 This practice is intended to define the exposure conditions for evaluating the thermal endurance of plastics when exposed solely to hot air for extended periods of time. Only the procedure for heat exposure is specified. The effect of elevated temperature on any particular property is determined by selection of the appropriate test method and test specimens for that property.

1.2 This practice can be used as a guide to compare thermal aging characteristics of materials as measured by the change in some property of interest. The property of interest is measured at room temperature.

1.3 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 using a series of elevated temperatures for the purpose of estimating endurance time to a defined property change at a lower temperature are also described; the applicability of the Arrhenius relation for making predictions to other temperatures, is assumed in this case.

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

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—This standard and ISO-2578 address the same subject matter but differ in technical content.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D618 Practice for Conditioning Plastics for Testing

D883 Terminology Relating to Plastics

D5374 Test Methods for Forced-Convection Laboratory Ovens for Evaluation of Electrical Insulation

D5423 Specification for Forced-Convection Laboratory Ovens for Evaluation of Electrical Insulation

E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

E456 Terminology Relating to Quality and Statistics

2.2 *ISO Standards:*[3]

ISO 2578 (1993) Determination of Time-Temperature Limits After Exposure to Prolonged Action of Heat

ISO 9080 (2012) Plastic Piping and Ducting Systems—Determination of the Long-Term Hydrostatic Strength of Thermoplastic Materials in Pipe Form by Extrapolation

3. Terminology

3.1 *General*—The terminology given in Terminology D883 and Terminology E456 is applicable to this practice. Terminology not in place is defined in 3.2.

3.2 *Definitions:*

3.2.1 *continuous use temperature (CUT)*—the temperature in degrees Celsius corresponding to a given thermal endurance time for a given failure criterion (typically 50 % reduction in property), derived from the Arrhenius relation of endurance time and temperature, determined by heat aging at several elevated temperatures. Several CUT values can exist, one for each property, endurance time and endurance criterion.

3.2.1.1 *Discussion*—In practice, the continuous use temperature for a plastic, involves other environmental considerations as discussed elsewhere in this standard, than thermal

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

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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.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

***A Summary of Changes section appears at the end of this standard**

endurance alone. The term, CUT, used here, is intended as an index for thermal endurance alone. The use of this term is found in automotive applications of plastics. The endurance time used for the CUT value reported should be specified as CUT (endurance time).

3.2.2 *temperature index (TI), also referred to as thermal index (TI)*—the temperature in degrees Celsius corresponding to an endurance time of 20,000 hours for a given failure criterion (typically 50% reduction in property), derived from the Arrhenius relation of endurance time and temperature, usually determined by heat aging at several elevated temperatures.

3.2.2.1 *Discussion*—The TI can thus be seen as a special case of CUT where the endurance time is fixed at 20,000 hours. A given plastic material and property and its retention criterion may be characterized by several CUT times, for example, CUT (1000 hours), CUT (5000 hours), etc. as needed per requirements of different applications. Further, several TI values can exist, one for each property and endurance criterion.

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 are 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. However, extended periods of exposure of plastics to elevated temperatures will generally cause some degradation, with progressive changes in physical properties. Specific properties and failure (or lifetime) criteria for these properties are typically chosen for the evaluation of thermal endurance.

4.3 Generally, short exposures at elevated temperatures 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 become brittle due to loss of plasticizers after exposure at elevated temperatures. Other types of plastics 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 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 The material studied can change inherent behavior with change in temperature as for example when crossing α, β, and γ transitions. These transitions should be avoided both in the range of aging temperatures used, as well as in extrapolation of the lifeline. Arrhenius principles may only be used to accelerate a chemical mechanism if there are no fundamental changes in the material properties. With semi-crystalline and highly crystalline polymers, elevated temperatures may cause significant changes to the morphology of the material, invalidating or compromising that assumption.

NOTE 2—Caution should be exercised in using the Arrhenius relation and knowledge of physical changes in the material at elevated temperatures is important. Guidance given in ISO 9080 for characterizing lifetime of plastic materials in pipe form by extrapolation suggests that the highest oven aging temperature should be at least 15°C lower than the Vicat softening temperature for glassy amorphous polymers, and at least 15°C lower than the melting point for semi-crystalline polymers.

4.7 Effects of exposure can 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 the influence of humidity.

4.8 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 the specific application.

4.9 It is possible for many CUT and TI values to exist. Therefore, for any application of the CUT or the TI (temperature index) to be valid, either the thermal aging program must duplicate the intended thermal exposure conditions of the end product, or the Arrhenius relation must apply.

4.10 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;" however, using this term should be avoided as this implies the tester has information on specific failure criteria in end-use, while numerous factors are not under the scope of this test. It is preferable to use terms such as "end point," "thermal endurance time," and such. Because of the errors associated with these calculations, this endurance 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. To harmonize with ISO 2578 and IEC standards, it is preferable to use ovens that comply with the set temperature, temperature variation and air change requirements of Specification D5423 as evaluated by the test methods of Test Methods D5374. Alternatively, Specification E145 may be used, with Specification E145, Type IIB ovens for aging temperatures up to 70°C, and Specification E145, Type IIA ovens for higher temperatures. Indicate the specific oven standard used in the test report.

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

6. Sampling

6.1 Use the number and type of test specimens required by the applicable test method each time the specific property is determined.

7. Test Specimens

7.1 Use the number and type of test specimens required by the applicable test method each time the specific property is determined.

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

7.3 If possible, fabricate test specimens by the same method as used in the intended application.

8. Conditioning

8.1 Conduct all tests in the standard laboratory atmosphere as specified in Practice D618, Method A, and with specimens conditioned in accordance with the requirements of the test method for determining the specific property or properties required.

8.2 Condition specimens following exposure at elevated temperature in the standard laboratory atmosphere as described in 8.1 prior to testing.

8.3 If possible, avoid simultaneous aging of mixed groups of different compounds which might cause cross contamination from off-gassing during heat aging.

9. Procedure

9.1 If possible, for each specific test and temperature, all materials must be exposed for the same time in the same oven (caution: see 8.3). In case of a single temperature study, use 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, covering a range adequate for extrapolation of the time-temperature relation. The following procedures are recommended for selecting four exposure temperatures:

9.2.1 The lowest temperature should produce the desired level of property change or product failure after at least 5000 hours of exposure.

9.2.2 The highest temperature should produce the desired level of property change or product failure after at least 500 hours of exposure.

9.2.3 When possible, select the exposure temperatures from Table 1 (taken from the list of standard temperatures in Practice D618). 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) are recommended to 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

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 $1/T \times 10^{-3}$ /°K	Estimated TI (Temperature/Thermal Index)[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 TI (Temperature/Thermal Index)*—the best estimate of temperature/thermal index available prior to the testing program. This is based on prior knowledge of similar materials, and subsequently 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.

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 retention percentage 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 at each temperature. Use the regression equation to determine the exposure time necessary to produce a predetermined level of property change at each temperature; the level of 50 % retention is typically used although a minimum threshold value can also be used as the criterion. An acceptable regression equation must have an r^2 of at least 0.80. 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, showing logarithm of thermal endurance time versus reciprocal temperature. Use linear 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.

NOTE 3—Annex A of ISO 2578 provides an example of calculating the Arrhenius relation.

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. The equation can also be used to determine the TI (temperature/thermal index).

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

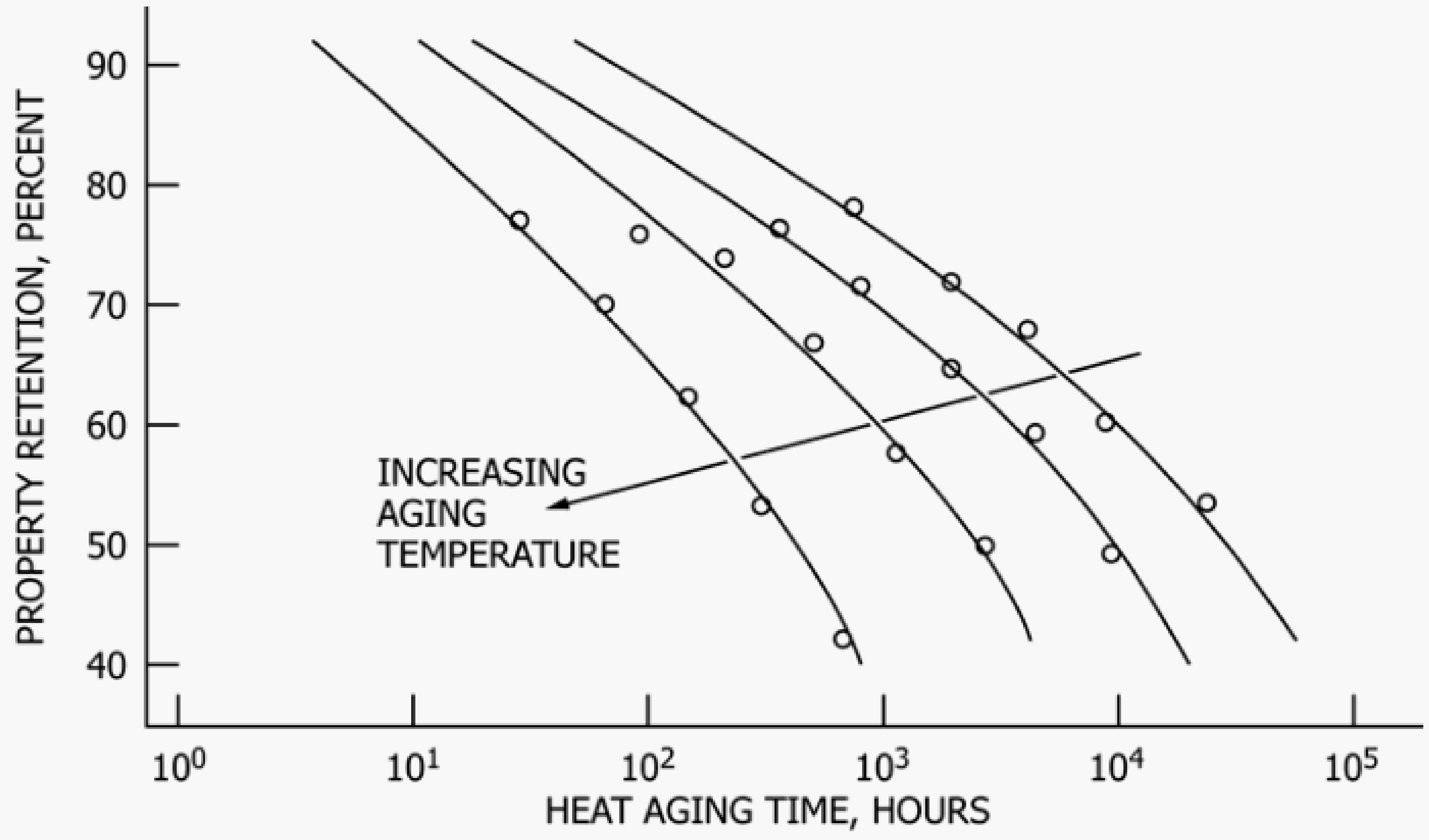

FIG. 1 Heat Aging Curves—Property Retention Versus Aging Time

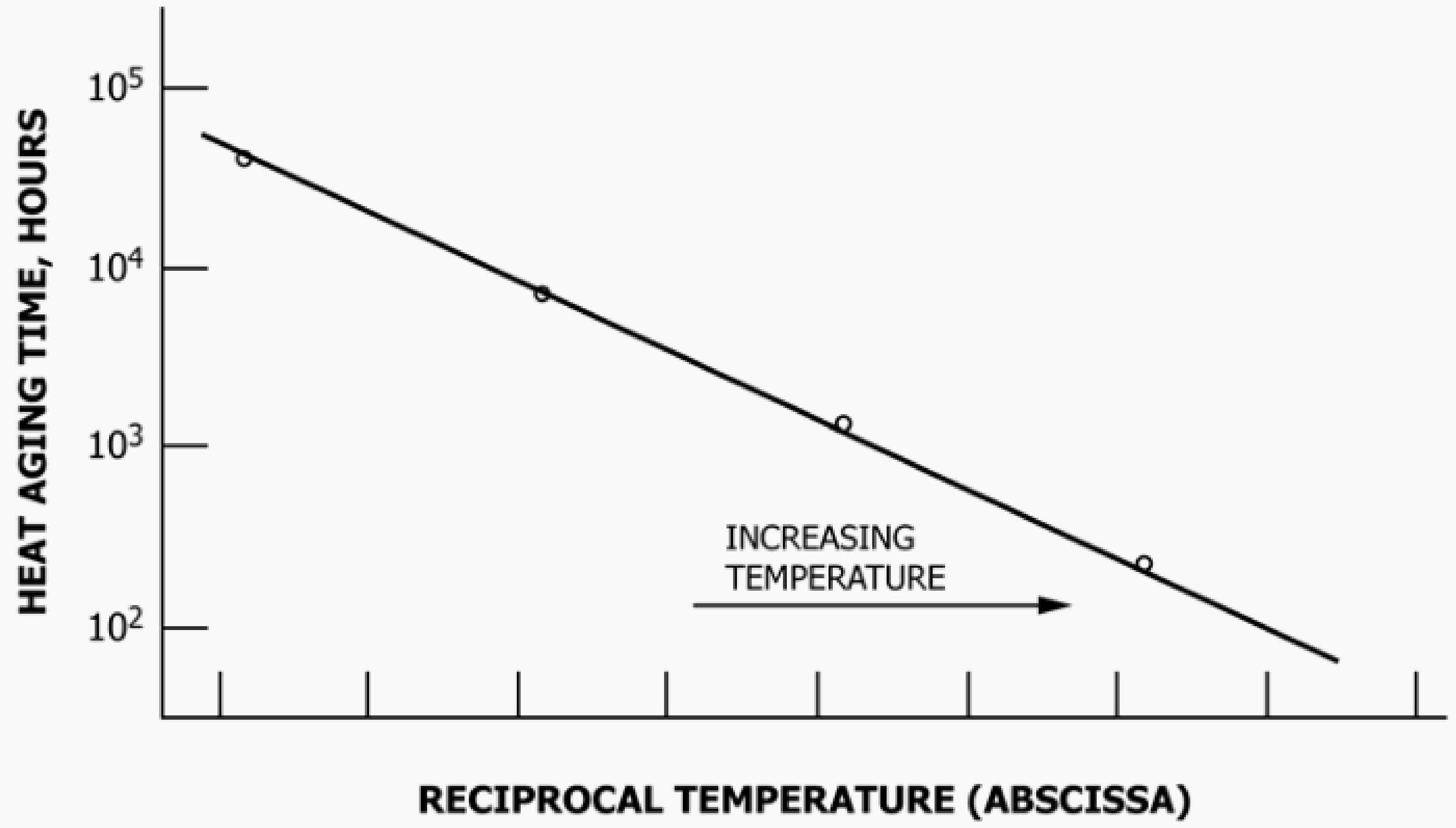

FIG. 2 Arrhenius Plot—Time of 50 % Property Retention (Life Time/Failure Time) Versus Reciprocal of Absolute Temperature

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, including the oven standard utilized,

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

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 report the following 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 The estimated TI (temperature/thermal index) and CUT values for each material, property and endurance criterion tested.

11.1.8.6 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.

13. Keywords

13.1 aging; continuous use temperature; exposure; heat; heat-aging; thermal-aging; oven; temperature index

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D3045–92(2010)) that may impact the use of this standard. (August 1, 2018)

(1) Deleted reference to use of tubular ovens, and added ASTM D5423 and ASTM D5374 as oven standards.
(2) Added definitions of continuous use temperature, CUT, and temperature/thermal index, TI.
(3) Reduced constraints on temperature selection, while adding caution on temperature selection and applicability of the Arrhenius relation.
(4) Clarified conditioning requirements.
(5) Various edits throughout the standard to improve readability and understanding.