Designation: ~~D7444 – 18~~ D7444 – 18a

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

This standard is issued under the fixed designation D7444; 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 indicates how to test the oxidative degradation characteristics of plastics that degrade in the environment under atmospheric pressure and thermal and humidity simulations, only, in the absence of any selected disposal environment such as soil, landfill, or compost. This practice does not by any extension or extrapolation of data or results generated indicate that such plastics are suitable for or will degrade on disposal in these said environments. It is particularly noted that in real world environments such as soil, compost and landfill oxidations, if they occur, will predominantly be under conditions where other interfering ingredients are present and, in the case of landfill, at sub-atmospheric oxygen concentrations. This practice, therefore, can only result in a relative ordering of the potential for oxidation of plastic materials under the conditions tested, which are not always reflective of their behavior in a particular real world disposal systems. Prediction of the oxidation of a plastic under real world disposal conditions is an essential further testing in appropriate methodologies, such as Test Method D5338 for composting. No claims can be made for real world behavior based on this practice.

1.2 This practice is only intended to define the exposure conditions of plastics at various temperatures in air at atmospheric pressure under controlled humidity levels for extended periods of time. The humidity levels and temperature ranges are selected to be within the variable recorded seasonal ranges (upper and lower levels) generally observed in disposal sites where such plastics are discarded. For example: soil (15 to 40 percent moisture); landfill (35 to 60 percent moisture), and compost (45 to 70 percent moisture). It is optional to expose the plastic at zero humidity, if comparison with specified humidity ranges is of interest. Only the procedures for heat and humidity exposures are specified, not the test method or specimen, necessary for the evaluation of the heat and humidity exposure effects. The effect of heat and humidity on any particular property is 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.

1.3 This practice is used to compare the effects of heat and humidity at any selected temperature, such as those found in the mentioned disposal environments, on the degradation of a particular plastic by selection of an appropriate test method and specimen.

1.4 This practice is to be used in order to apply selected exposure conditions when comparing the thermal-aging characteristics at controlled humidity levels of plastic materials as measured by the change in some property of interest (that is, embrittlement by means of loss of elongation, molecular weight, disintegration, etc.). 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

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

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

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

1.8 This practice recommends procedures for comparing the thermal and humidity aging characteristics of materials at a single temperature under dry or selected humidity conditions. Recommended procedures for determining the thermal aging characteristics

[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 and Biobased Products.

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*A Summary of Changes section appears at the end of this standard of a material at a series of temperatures and humidity conditions 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.

1.9 The values stated in SI units are to be regarded as the standard.

1.10 *The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.*

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

1.11 *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
D3045 Practice for Heat Aging of Plastics Without Load
~~D3593 Test Method for Molecular Weight Averages/ Distribution of Certain Polymers by Liquid Size-Exclusion Chromatography (Gel Permeation Chromatography GPC) Using Universal Calibration (Withdrawn 1993)[3]~~
D3826 Practice for Determining Degradation End Point in Degradable Polyethylene and Polypropylene Using a Tensile Test
D5032 Practice for Maintaining Constant Relative Humidity by Means of Aqueous Glycerin Solutions
D5338 Test Method for Determining Aerobic Biodegradation of Plastic Materials Under Controlled Composting Conditions, Incorporating Thermophilic Temperatures
D6954 Guide for Exposing and Testing Plastics that Degrade in the Environment by a Combination of Oxidation and Biodegradation
E104 Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions
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 Since the correlation between the conditions specified in this practice and actual disposal environments (for example, composting, soil or landfill) has not been determined or established, the test results are to be used only for comparative and ranking purposes in the laboratory. No extrapolation to real world disposal expectations or predictions are to be made from results obtained by this procedure. Real world evaluations and correlations are needed for such claims.

4.2 Degradable plastics exposed to heat and humidity are subject to many types of physical and chemical changes. The severity of the exposures in both time, temperature and humidity level, determines the extent and type of change that occurs. For example, 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, sometimes 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. The effects of humidity are less well understood and are more difficult to predict and depend on the degradable plastics characteristics such as hydrophilicity, polarity and composition.

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; increase crystallinity; and cause some change in color of the plastic or coloring agent, or both. Normally, additional shrinkage is expected with a loss of volatiles or advance in polymerization.

4.4 Some plastic materials such as PVC 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 is used to characterize any molecular-weight changes that are suspected to have occurred during thermal exposure.~~

[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.

4.6 The degree of change observed will depend on the property measured. Different properties do 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 can 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 level of the oven, air velocity over the specimen, and exposure period which are evaluated by this practice. Errors in exposure are cumulative with time; for example certain materials have the potential to be degraded due to the influence of humidity rather than oxidation in long-term tests and thus give misleading results. Materials susceptible to hydrolysis (that is, hydrolytically degradable plastics) undergo degradation when subjected to long-term thermal tests due to the presence of moisture rather than oxidation.

4.8 Do not infer 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-type and composition, 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 (time to reach failure is dependent on the exposure temperature and humidity). 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 plastic 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 plastic material.

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

4.11 There can be very large errors when Arrhenius plots or equations based on data from experiments at a series of temperatures and humidity 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 Type:*

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

5.2.2 *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, it is desirable to use a tubular-oven procedure, such as described in Specification E145, in accordance with 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 *Relative Humidity Control:*

5.4.1 The chamber shall be able to control relative humidity to within ±5 % when the set point temperature has an operational tolerance of ±1°C.

5.4.2 Relative humidity at different temperatures can be controlled within a narrow range by using saturated aqueous solutions in accordance with Practice E104 or Practice D5032.

6. Test Specimen

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 must be met at each time and temperature and humidity 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 and humidity selected.

6.2 The specimen thickness is to be comparable to but no greater than the maximum thickness of the intended article in its application, and its method of fabrication must be the same as that for the intended application.

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