Designation: D6954 − 24

Standard Guide for Exposing and Testing Plastics that Degrade in the Environment by a Combination of Oxidation and Biodegradation[1]

This standard is issued under the fixed designation D6954; 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 guide provides a framework or road map to compare and rank the controlled laboratory rates of degradation and degree of physical property losses of polymers by thermal and photooxidation processes as well as the biodegradation and ecological impacts in defined applications and disposal environments after degradation. Disposal environments range from exposure in soil, landfill, and municipal or industrial compost in which thermal oxidation may occur and land cover and agricultural use in which photooxidation may also occur.

1.2 In this guide, established ASTM International standards are used in three tiers for accelerating and measuring the loss in properties and molecular weight by both thermal and photooxidation processes and other abiotic processes (Tier 1), measuring biodegradation (Tier 2), and assessing ecological impact of the products from these processes (Tier 3).

1.3 The Tier 1 conditions selected for thermal oxidation and photooxidation accelerate the degradation likely to occur in a chosen application and disposal environment. The conditions should include a range of humidity or water concentrations based on the application and disposal environment in mind. The measured rate of degradation at typical oxidation temperatures is required to compare and rank the polymers being evaluated in that chosen application to reach a molecular weight that constitutes a demonstrable biodegradable residue (using ASTM International biometer tests for CO_2 evolution appropriate to the chosen environment). By way of example, accelerated oxidation data must be obtained at temperatures and humidity ranges typical in that chosen application and disposal environment, for example, in soil (20 to 30°C), landfill (20 to 35°C), and municipal or industrial composting facilities (30 to 65°C). For applications in soils, local temperatures and humidity ranges must be considered as they vary widely with geography. At least one temperature must be reasonably close to the end use or disposal temperature, but under no circumstances should this be more than 20°C away from the removed that temperature. It must also be established that the polymer does not undergo a phase change, such as glass transition temperature (*Tg*) within the temperature range of testing.

1.4 The residues resulting from the oxidations are then exposed to appropriate disposal or use environments in standard biometric test methods to measure the rate and degree of biodegradation (Tier 2).

1.5 The data generated under Tier 1 evaluation and the determined time for the biodegradation in the chosen environment (Tier 2) allow ranking relative to other polymers evaluated under similar environmental conditions with this guide. The degree and time for biodegradation should be consistent with ASTM International methods, and any residues from the intermediate oxidation stage and from biodegradation must be shown to be environmentally benign and not persistent (Tier 3).

NOTE 1—The intended use of this guide is for comparison and ranking of data to aid in the design and development and the reduction of environmental impacts of polymers that require no more than 24 months to oxidize and biodegrade in the intended use and disposal options and create no harmful or persistent residues under the appropriate disposal conditions (for example, two seasons of crop-growing conditions in soil).

1.6 It is cautioned that the results of any laboratory exposure in this guide cannot be directly extrapolated to actual disposal environments; confirmation to real world exposure is ultimately required as with all ASTM International standards.

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

NOTE 2—There is no ISO standard that is the equivalent of this standard guide.

1.8 *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.*

1.9 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the*

---

[1] This guide 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.

Current edition approved Jan. 1, 2024. Published January 2024. Originally approved in 2004. Last previous edition approved in 2018 as D6954 - 18. DOI: 10.1520/D6954-24.

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

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]

D883 Terminology Relating to Plastics

D3826 Practice for Determining Degradation End Point in Degradable Polyethylene and Polypropylene Using a Tensile Test

D3987 Practice for Shake Extraction of Solid Waste with Water

D5071 Practice for Exposure of Photodegradable Plastics in a Xenon Arc Apparatus

D5208 Practice for Fluorescent Ultraviolet (UV) Exposure of Photodegradable Plastics

D5272 Practice for Outdoor Exposure Testing of Photodegradable Plastics

D5338 Test Method for Determining Aerobic Biodegradation of Plastic Materials Under Controlled Composting Conditions, Incorporating Thermophilic Temperatures

D5510 Practice for Heat Aging of Oxidatively Degradable Plastics (Withdrawn 2010)[3]

D5526 Test Method for Determining Anaerobic Biodegradation of Plastic Materials Under Accelerated Landfill Conditions

D5951 Practice for Preparing Residual Solids Obtained After Biodegradability Standard Methods for Plastics in Solid Waste for Toxicity and Compost Quality Testing (Withdrawn 2011)[3]

D5988 Test Method for Determining Aerobic Biodegradation of Plastic Materials in Soil

D6002 Guide for Assessing the Compostability of Environmentally Degradable Plastics (Withdrawn 2011)[3]

D6400 Specification for Labeling of Plastics Designed to be Aerobically Composted in Municipal or Industrial Facilities

E1440 Guide for Acute Toxicity Test with the Rotifer Brachionus

2.2 *Other Standards:*

EPA TITLE 40 CFA 40CFR62, 40CFR50-189, 40CFR260-299, 40CFR300-399, 700-799, and 49CFR100-180[4]

OECD Guideline 207 Earthworm, Acute Toxicity Tests[5]

OECD Guideline 208 Terrestrial Plants, Growth Test[5]

3. Terminology

3.1 *Definitions:*

3.1.1 Definitions of most terms applicable to this guide appear in Terminology D883 and Guide D6002.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *environmental degradation of a plastic, n*—abiotic or biotic degradation process or both that occurs in a given environment and includes photodegradation, oxidation, hydrolysis, and biodegradation. Living organisms effect biotic degradation processes and abiotic degradation processes are nonbiological in nature.

3.2.1.1 *Discussion*—Term not defined in Terminology D883.

3.2.2 *gels, n*—cross-linked polymer structures insoluble in solvents that do not break the primary or cross-linking bonds in the polymer. Cross-links created during oxobiodegradadation of polymers are chemical bonds created by the degradation process, mostly carbon-carbon bonds, and thus extremely resistant to solvent degradation.[6]

3.2.3 *oxidation, n*—process promoted thermally or by irradiation in the presence of oxygen.

4. Summary of Guide

4.1 This guide may be used to compare and rank the rate and degree of thermal oxidative degradation of a plastic material relatively to a molecular weight range that can be established as biodegradable in a chosen environment. Subsequently, the biodegradation of these degraded polymers in diverse environments such as soil, municipal or industrial compost, landfill, and water may be compared and ranked using standard biometric test methods and measuring carbon dioxide evolution.

NOTE 3—If municipal or industrial composting is the designated disposal route, Specification D6400 is the only ultimate and definitive applicable specification for measuring biodegradation in a municipal or industrial compostability process. Oxidation followed by biodegradation under the conditions found in this guide does not confer the designation "municipally or industrially compostable" or any connotation that the applications are acceptable for composting in a commercial or municipal composting facility.

4.2 This guide uses a tiered criteria-based approach to assess the consecutive oxidation and biodegradability of plastic products and ecological impacts in defined applications. This is shown schematically in Section 6. Each tier in this guide includes objectives and a summary that presents test methods, method principles, test duration, and interpretation of results.

4.3 The tiered approach is chosen in the laboratory for convenient separation of oxidative degradation, biodegradation, and ecological impact stages even though in the real world all three are likely to be concurrent rather than consecutive.

4.4 *Tiered Methodology:*

4.4.1 Tier 1 measures the rate and extent of molecular weight loss resulting from oxidation that is indicative of losses in physical properties from oxidation. Tier 1 requires either accelerated testing or long-term testing over a range of relative humidity or amount of moisture. Accelerated testing must be performed under conditions and temperatures that are acceptably typical of the specific application and disposal environments under consideration. Practices D5208, D5510, and

---

[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] The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from United States Environmental Protection Association (EPA), Ariel Rios Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460.

[5] Available from OECD, 2 rue Andre Pascal, F-75775 Paris Cedex 16, France.

[6] Vollmert, B., *Polymer Chemistry*, Springer-Verlag, New York, 1973, p. 27, also pp. 543- 561.

D5071 may be used to specify the oxidative conditions and Practice D3826 may be used to define the point of embrittlement.

NOTE 4—For measuring the extent of disintegration/fragmentation, a sieve test is required. In this tier, the fragments are subjected to molecular weight analysis and a total mass balance is obtained in the process. Exposure temperatures may range from 20 to 70°C in the presence of air and specified moisture or water levels for selected periods of time. At least one temperature must be reasonably close to the end use or disposal temperature, but under no circumstances should this be more than 20°C away from that temperature. It must also be established that the polymer does not undergo a phase change, such as glass transition temperature (*Tg*) within the temperature range of testing. As an alternate degradation process, the test samples may be exposed to photooxidation in air as per Practices D5208 or D5071 and the mass change of the plastic recorded after exposure.

4.4.2 Tier 1 accelerated oxidation tests are not indicators of biodegradability and should not be used for the purpose of meeting the specifications as described in Specification D6400 and claiming municipal or industrial compostability or biodegradation during municipal or industrial composting. (If oxidation is thought to be sufficiently rapid in Tier 1, suggesting that municipal or industrial composting may be a disposal environment, then Specification D6400 must be done and all the specifications in Section 6 (Detailed Requirements) must be met.

4.5 *Gel Formation and Consequences During Oxidation Phase—Discussion:*

4.5.1 Gel formation is a frequent side reaction of the oxidative degradation of polymers, especially polyolefins. Gels are cross-linked structures arising from the free radical nature of oxidative degradation. They are insoluble in nonreactive solvents, that is, solvents that do not break additional bonds. Normally, gels are not available to biodegradation. Some gels dissolve on further oxidative degradation and become available for ultimate biodegradation. However, the prooxidant (catalyst) may be excluded from the gel structure because of solubility changes in gel phase. In this case, the gel would become a nondegradable or very slowly degradable new fraction within the polymer. It is important to establish the extent of gel and its nature or permanence in the polymer residue and report these findings.

4.5.2 The purpose of Tier 2 is to estimate the biodegradability of all the fragmented products from Tier 1 under laboratory scale conditions appropriate to the application using current Test Methods D5988 and D5338. The entire material from the Tier 1 exposure is subjected to biodegradation testing. The percent biodegradation shall be calculated and reported as stated in the above referenced standards. The results from Tier 1 and Tier 2 shall be combined and used for comparison and ranking purposes between polymers of interest.

4.5.3 Tier 3 involves considerations of the ecological impacts in the final disposal medium such as soil, as in all biodegradation testing methods, which is basically a comparison of the test medium before and following oxidation and biodegradation.

5. Significance and Use

5.1 This guide is a sequential assembly of extant but unconnected standard tests and practices for the oxidation and biodegradation of plastics, which will permit the comparison and ranking of the overall rate of environmental degradation of plastics that require thermal or photooxidation to initiate degradation. Each degradation stage is independently evaluated to allow a combined evaluation of a polymer's environmental performance under a controlled laboratory setting. This enables a laboratory assessment of its disposal performance in, soil, municipal or industrial compost, landfill, and water and for use in agricultural products such as mulch film without detriment to that particular environment.

NOTE 5—For determining biodegradation rates under municipal or industrial composting conditions, Specification D6400 is to be used, including test methods and conditions as specified.

5.2 The correlation of results from this guide to actual disposal environments (for example, agricultural mulch films, municipal or industrial composting, or landfill applications) has not been determined, and as such, the results should be used only for comparative and ranking purposes.

5.3 The results of laboratory exposure cannot be directly extrapolated to estimate absolute rate of deterioration by the environment because the acceleration factor is material dependent and can be significantly different for each material and for different formulations of the same material. However, exposure of a similar material of known outdoor performance, a control, at the same time as the test specimens allows comparison of the durability relative to that of the control under the test conditions.

6. Procedures

6.1 Test sample selected to be in the thickness of the application form.

6.2 The tier testing procedure is outlined schematically in Fig. 1.

6.3 In Tier 1, the test sample is exposed to several discrete temperatures (one being within 20°C of the end use temperature) within the range of 20 to 70°C in the presence of specified levels of air and specified moisture or water levels for defined periods of time using the procedure apparatus and following the calculations in Practice D5510. The temperature ranges specified in Practice D5510, paragraphs 8.3.1 and 8.3.4 are not germane to this guide and need not be followed. For thin films, the exposure period would be the time required for the film to reach 5 % or less elongation to break (Practice D3826) and the fragmented film to reach a recorded average weight-average molecular weight (*MW*) of 5000 or less. The time at three different temperatures required (with the mentioned caveats) for molecular weight reduction should be established.

6.3.1 A sample from Tier 1 residue should be dissolved in an appropriate nonreactive solvent and the gel phase, if any, separated by filtration, the gel dried, and the amount of gel reported as weight fraction of total sample. This should be regarded as nondegradable fraction of polymer.

6.3.2 The gel may be subjected to further oxidative degradation and extent of subsequent reversion to soluble and degradable material reported.

6.3.3 The acceptable amount of gel will depend on the particular application and the maximum acceptable wt % gel written into specifications for the particular class of end uses.