



Designation: D 5152 – 91

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Practice for Water Extraction of Residual Solids From Degraded Plastics for Toxicity Testing¹

This standard is issued under the fixed designation D 5152; 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 provides a procedure for leaching residual solids from degraded plastics, yielding an aqueous solution which can be used to determine the aquatic toxicity of the materials leached under the specified test conditions, and determine the materials extractable under nonspecified testing conditions. This practice may be used in conjunction with Test Methods D 4229, E 1192, E 1295 or other currently accepted toxicity test methods (see OECD Guideline 203 or 40FR 797).

1.2 This practice provides for the shaking of a known weight of photodegraded plastic with water of specified composition (see Terminology D 1129) and the separation of the aqueous phase (extract) for analysis. It describes the collection of samples from biodegradation and chemodegradation tests to be used for further analyses. In the event that a particular sample does not pass the toxicity test, chemical characterization of the degradation products can be performed on the aqueous extract to determine the source of the toxicity. The description of the performance of these analyses is beyond the scope of this practice.

1.3 *This standard does not purport to address all of the safety problems, 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.*

2. Referenced Documents

2.1 ASTM Standards:

D 1129 Terminology Relating to Water²

D 1293 Test Methods for pH of Water²

D 4229 Practice for Conducting Static Acute Toxicity Tests on Wastewater with Daphnia³

E 729 Guide for Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates and Amphibians³

E 1192 Guide for Conducting Acute Toxicity Tests on Aqueous Effluents with Fishes, Macroinvertebrates and Amphibians³

E 1295 Guide for Conducting Three-Brood Renewal Toxic-

ity Tests with Ceriodaphnia dubia³

2.2 Other Standard:

OECD Guideline 203, Fish, Acute Toxicity Test 40FR797A (USEPA), A Variety of Toxicity Testing Procedures⁴

3. Terminology

3.1 For definitions and terms used in this practice, see Terminology D 1129 and the terminology sections in Practice D 4229 and Guide E 729.

4. Significance and Use

4.1 This practice is intended as a rapid means of obtaining an extract from the residual solids of degraded plastics. The extract may be used to estimate the release of certain constituents of degradable plastics under the laboratory conditions described in this procedure and the toxicity, or both, of these releases to aquatic organisms.

4.2 Some photodegradable plastic degradation test methods involve accelerated light exposure of plastic in an apparatus which may include cyclic applications of water spray, mist or condensation. However, for the purpose of this practice, the exposure of the plastic should be performed dry, with the moisture cycle turned off. This prevents premature elution of soluble degradation products from the plastic, and limits extraneous sources of contamination.

4.3 In plastic degradation test methods that are water based, the water utilized in the test method should be collected, preserved and stored in accordance with acceptable procedures for the specific chemical characterizations or toxicity assays to be carried out. Specific procedures for collection, preservation, and storage of these liquids are beyond the scope of this practice.

4.4 This practice is not intended to simulate any or all of the site specific leaching conditions to which degraded plastics may be subjected. It has not been scientifically demonstrated to represent actual disposal-site leaching conditions such as oceans, beaches, soils, compost heaps, or landfills.

4.5 This practice is end-point specific as written, in that it is focused on a post-degradation analysis. Water collection and storage as recommended here allows for further qualitative and quantitative toxicological testing procedures which are beyond

¹ Precision and Bias This practice is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.96 on Environmentally Degradable Plastics.

Current edition approved July 15, 1991. Published September 1991.

² Annual Book of ASTM Standards, Vol 11.01.

³ Annual Book of ASTM Standards, Vol 11.04.

⁴ Available from Superintendent of Documents, U.S. Government Printing Office, Washington D.C. 20402.

the scope of this practice. These procedures may be useful in the characterization of degradation products derived from biodegradable and chemically degradable plastics.

4.6 Two scenarios have been focused on to recommend water to plastic extraction ratios. For litter, it has been estimated that 2 g of plastic per litre of extraction water is reasonable. For landfills, a reasonable estimate is 100 g/L of extraction water. The former was based on beach litter found in a 1988 survey of 25 states,⁵ annual rainfall amounts exceed by >95 % of the continental U.S.,⁶ multiplied by a safety factor of 10. The landfill estimate was based on average amounts of refuse, surface area, and rainfall projected over 20 years of operation at 18 currently operating landfills across the continental U.S.⁷

5. Apparatus

5.1 *Agitation equipment*, of any type that rotates about a central axis at a rate of 30 (RPM).⁸

5.2 *Membrane filter assembly*, or borosilicate glass or stainless steel with a flat fritted base and filter holder.

5.3 *Plastic grinder*, suitable for grinding plastic samples to a particle size between 2 mm and 10 mm.

5.4 *Containers*, wide-mouthed, of glass or other composition suitable to the nature of degradable plastics, which will not allow sorption of constituents of interest or otherwise contaminate the test materials. Containers of 1- or 2-L volume may be used with the appropriate 2-g or 4-g quantities of test material. Utilize 100 g/L if it is wished to perform analyses consistent with a simulated landfill environment. Containers should be cleaned in a manner consistent with the analyses to be performed.

6. Reagents

6.1 *Purity of Reagents*:

6.1.1 Reagent-grade chemicals shall be used in all tests.

6.2 *Purity of Water*:

6.2.1 Prepare as specified in the toxicity test to be used.

7. Sampling

7.1 Obtain a representative sample of degraded plastic from a chemical degradation, photodegradation, thermal degradation or biodegradation test. The degraded plastic will have less than 5 % absolute tensile elongation (polyolefins), or otherwise conform to the definitions of *degraded* (as described by appropriate ASTM standards for a particular plastic material). Record sample weight change during degradation.

7.2 In the process of degrading the sample, if an accelerating light exposure apparatus is used, any spray or moisture cycle should be deactivated.

7.3 Samples prepared by biodegradation should be gently removed from bioreactors, chemical reactors or biodegradation

environments and carefully cleaned to remove extraneous material while minimizing removal of plastic degradation products. Collect and store all waters used in biodegradation or chemical-degradation tests. Quantitative acute and chronic toxicologic testing procedures required for analyses of these plastics are beyond the scope of this practice.

7.4 In all of the preceding test situations, utilize appropriate controls consisting of a non-degradable plastic of the same basic resin type, subjected to the same procedure, cleaning and collection of aqueous phase.

8. Sample Preparation

8.1 Grind the sample to pass through a 10-mm sieve and be returned through a 2-mm sieve.

8.2 Divide the sample into equal portions of 2 g each or 100 g each for replicate testing.

9. Procedure

9.1 Record the sample size after cleaning and grinding so far as it is known.

9.2 Determine the air-dry weight of the sample by allowing one portion to dry in a desiccator at room temperature (20 to 25°C). Weigh on successive days until a constant weight is attained.

9.3 Add 2 g (litter test) or 100 g (landfill test) of sample to triplicate shake containers.

9.4 Add 1 L of water, prepared as needed for the toxicity test chosen.

9.5 Measure and record the initial pH of the sample/water mixture (see Test Methods D 1293).

9.6 Agitate continuously for 24 h at 20 to 25°C. Record the agitation time and temperature.

9.7 Open the container. Observe and record any physical changes in the sample and leaching solution.

9.8 The extract should be used for toxicity testing with the plastic present if possible. However, tests with some organisms may require screening or filtering to reduce the amount of particulate. If so, filter the extract with a pre-washed 0.45 µm glass filter or equivalent.

10. Report

10.1 Report the following information:

10.1.1 The source, type of sample, and the sample date,

10.1.2 The sample's degradation characteristics,

10.1.3 The sample-cleaning technique used, if any,

10.1.4 Physical characteristics of the sample (sieve size, solids content),

10.1.5 Weight of sample in each of the triplicate shake containers,

10.1.6 Volume of water used in shaker containers,

10.1.7 Initial pH of extract,

10.1.8 Exact type of filter used to remove particulates, if any,

10.1.9 Volume of filtrate recovered,

10.1.10 Final pH of extract before and after filtering,

10.1.11 Observations of changes in test material during extraction,

10.1.12 The extract preservation technique, and

10.1.13 The initial and post degradation weight of the sample.

⁵ "Study Finds Most Litter on Beach is Plastic," *Plastics News*, July 17, 1989.

⁶ *The Water Encyclopedia*, Lewis Publishers, Chelsea, MI, 1990.

⁷ Pohland, F. G., and Harper, S. R., "Biogas Developments in North America," in *Proceedings 4th Intl. Symposium on Anaerobic Digestion*, Guangzhou, China, November 1937.

⁸ Orbital shakers such as B. Braun Autoshake R have been found suitable for this purpose.