Designation: D 6003 – 96

Standard Test Method for Determining Weight Loss From Plastic Materials Exposed to Simulated Municipal Solid-Waste (MSW) Aerobic Compost Environment [1]

This standard is issued under the fixed designation D 6003; 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 test method is used to determine the degree and rate of aerobic biodegradation of plastic materials exposed to a controlled composting environment. Aerobic composting takes place in an environment where temperature, aeration, and humidity are closely monitored and controlled.

1.2 The test is designed to determine the biodegradability of plastic materials, relative to that of a standard material, in an aerobic environment. Aeration of the test reactors is maintained at a constant rate throughout the test and reactor vessels of a size no greater than 4-L volume are used to ensure that the temperature of the vessels is approximately the same as that of the controlled environment chamber.

1.3 Biodegradability of the plastic is assessed by determining the amount of weight loss from samples exposed to a biologically active compost relative to the weight loss from samples exposed to a "poisoned" control.

1.4 The test is designed to be applicable to all plastic materials that are not inhibitory to the bacteria and fungi present in the simulated Municipal Solid Waste (MSW).

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

1.6 *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—There is no similar or equivalent ISO method.

2. Referenced Documents

2.1 *ASTM Standards:*

D 618 Practice for Conditioning of Plastics and Electrical Insulating Materials for Testing[2]

D 883 Terminology Relating to Plastics[2]

D 1193 Specification for Reagent Water[3]

D 1898 Practice for Sampling of Plastics[2]

D 2973 Test Method for Total Nitrogen in Peat Materials[4]

D 2974 Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils[4]

D 2976 Test Method for pH of Peat Materials[4]

D 2980 Test Method for Volume Weights, Water-Holding Capacity, and Air Capacity of Water-Saturated Peat Materials[4]

D 3593 Molecular Weight Averages and Molecular Weight Distribution of Certain Polymers by Liquid Size Exclusion Chromatography (Gel Permeation Chromatography)[5]

D 4129 Test Method for Total and Organic Carbon in Water by High-Temperature Oxidation and Coulometric Detection[6]

D 5338 Test Method for Determining Aerobic Biodegradation of Plastic Materials under Controlled Composting Conditions[7]

D 5509 Practice for Exposing Plastics to a Simulated Compost Environment[7]

D 5512 Practice for Exposing Plastics to a Simulated Compost Environment Using an Externally Heated Reactor[7]

2.2 *APHA-AWWA-WPCF Standards:*

2540 G Total, Fixed, and Volatile Solids in Solid and Semi-Solid Samples

3. Terminology

3.1 *Definitions*—Definitions of terms applying to this test method appear in Terminology D 883 and Practice D 5509.

4. Summary of Test Method

4.1 The test method consists of the following:

4.1.1 Selecting plastic materials for exposure in a controlled aerobic composting environment;

4.1.2 Preparing and characterizing a simulated compost with the proper C:N ratio, pH, water holding capacity, porosity, and inoculum to establish and maintain a high biological activity;

[1] This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.96 on Environmentally Degradable Plastics.

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

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 04.08.

[5] *Discontinued, 1993*. Replaced by Test Method D 5296.

[6] *Annual Book of ASTM Standards*, Vol 11.02.

[7] *Annual Book of ASTM Standards*, Vol 08.03.

4.1.3 Exposing the test materials to the compost under controlled, aerobic conditions;

4.1.4 Removing the test specimens for cleaning; and

4.1.5 Assessing the degradability of the plastics by measuring weight loss from the test specimens.

5. Significance and Use

5.1 Aerobic composting represents an attractive alternative to the disposal of solid wastes in landfills. Composting by biologically mediated oxidative decomposition produces highly stable organic matter that may be used for land applications or horticulture. However, the degradation of plastics within a compost can affect the decomposition of materials enclosed by the plastic, other non-plastic materials in the compost, and the resulting composition and appearance of the composted material. This test is intended to help assess the environmental degradability of plastics under standard composting conditions. Characterization of the ability of a plastic to degrade under controlled, environmentally relevant conditions is essential when developing products with a programmed lifetime.

5.2 Considering the diversity of materials that may be introduced into a particular compost, as well as the variety of designs of composting facilities, it is important to recognize that no single test can adequately simulate all the conditions which may occur during composting. Consequently, this test is intended to provide a uniform, standardized environment simulating a representative MSW compost operating at near-optimum conditions.

5.3 Because a specimen degrades to the point where it can not be distinguished from the other materials within the compost does not mean that it has become fully mineralized. Determination of a plastic's degradation products and their potential toxicity requires further testing. Mineralization of the plastic material (that is, conversion of polymer-C to CO_2) should be investigated using Test Method D 5338.

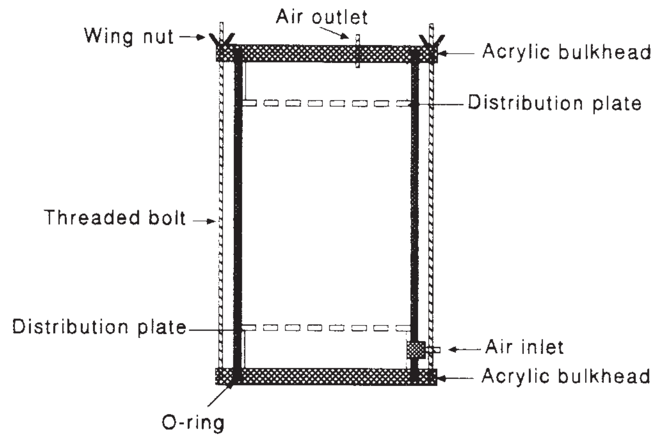
5.4 Predicting long-term environmental fate and effects of a plastic from the results of short-term exposure to a simulated waste disposal environment is difficult. Thus, caution should be exercised when extrapolating the results obtained from this or any other controlled-environment test to disposal in the natural environment.

6. Apparatus

6.1 *Composting Apparatus*:

6.1.1 A suitable bioreactor vessel (see Fig. 1) consists of a 127-mm (i.d.) by 300-mm long acrylic cylinder; two acrylic bulkhead plates (150 mm × 150 mm); two acrylic distribution plates, positioned 25 mm from the bulkhead plates; and four all-thread bolts with wing nuts. Air enters the bioreactor through an inlet (6.4-mm i.d.) positioned about 25 mm above the bottom bulkhead, and exits the bioreactor through an outlet (6.4-mm i.d.) in the top bulkhead.

NOTE 2—The size of the reactor may be changed as long as there is sufficient volume to allow for the even distribution of the MSW and test materials. However, the internal volume of the reaction vessel should not exceed 4 L; this will allow adequate control of the internal temperature of the compost via the exchange of heat between the contents of the reaction vessel and the environment chamber.



NOTE 1—Bioreactor = acrylic cylinder; 127-mm (ID) × 300 mm long.
Air exit port = 6.4 mm diameter.
Air inlet port = 6.4 mm diameter; 25 mm above the acrylic bulkhead.
Perforated distribution plate: positioned 50 mm above the acrylic bulkhead.

FIG. 1 Schematic Drawing of the Aerobic Bioreactor

6.1.2 The bioreactors are connected to a filtered air supply capable of providing water-saturated air at a rate of 100 to 200 mL min^{-1}. The air supply to each bioreactor is humidified by passing through a fritted-glass air dispersion tube immersed in distilled water (300 mL in a 500-mL Erlenmeyer flask) and regulated via a flow meter (see Fig. 2). Water in the humidifier flask must be maintained at the temperature of the environment chamber.

6.1.3 A controlled-environment chamber capable of maintaining a temperature of 30 (±2) to 70 (±2) °C.

6.2 *Analytical Equipment*:

6.2.1 *Analytical Balance*, to weigh test materials, (±0.1 mg).

6.2.2 *Top Loading Balance*, to weigh MSW samples for determining water content, (±0.01 g).

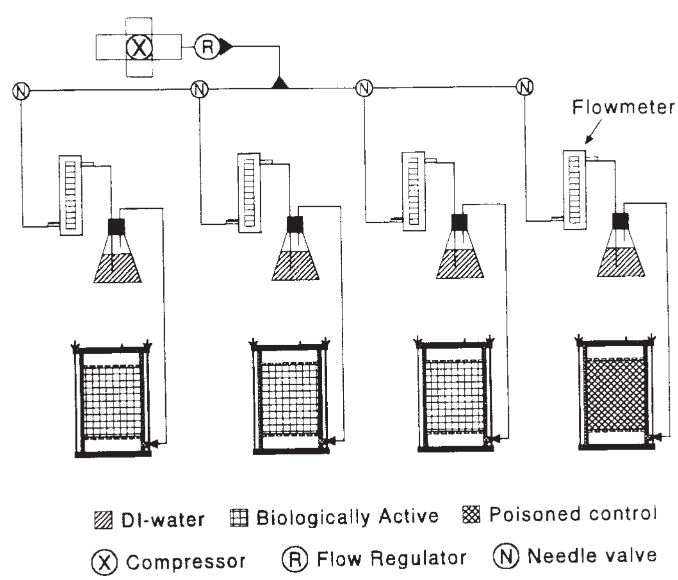


FIG. 2 Schematic of the Film Weight-Loss Bioreactor System

6.2.3 *Oven*, for determining the water content of the simulated MSW compost, set at 103 to 105°C.

6.2.4 *Muffle Furnace*, for determining the volatile solids and ash content of the simulated MSW compost, set to 550 (±50) °C.

6.2.5 *pH Meter*.

7. Reagents and Materials

7.1 All chemicals shall be of American Chemical Society (ACS) reagent-grade quality.

7.2 Distilled water should be prepared in accordance with Specification D 1193.

7.3 *Simulated Municipal Solid Waste (MSW) Compost*—Simulated MSW composts range from the relatively simple to the complex; the mixes listed in Tables 1 and 2 provide suitable environments for this test.

7.3.1 *Mix 2* **(1)**[8]—940 g shredded leaves (1:1, w/w, mix of oak and maple); 340 g shredded paper (1:1, w/w, mix of newspaper and computer paper); 140 g mixed, frozen vegetables; 120 g meat waste (added as a 1:1, w/w, mix of dried dog food and dried cat food); 360 g dehydrated cow manure; 40 g sawdust; 40 g urea; and 20 g commercial compost seed. Enough water is added to bring the mix to 60 % water holding capacity (Test Method D 2980). The C:N ratio of the starting mix is 14:1.

7.3.2 *Mix 4* **(3)**—3500 g dehydrated alfalfa meal; 1300 g cottonseed meal; 1400 g Poplar sawdust; 1000 g fresh cow manure; 1500 g black garden soil; 2500 g shredded newspaper; 480 g $CaCO_3$; 40 g $NaHCO_3$; and 13 L of water. The mix is blended in a Hobart Mixer until the average particle size is 3 to 4 mm. The C:N ratio of the starting mix is 30:1.

7.3.3 An alternative MSW compost, designed to simulate a particular waste stream, may be used.

7.4 A simulated yard-waste mix composed of dried grass and leaves (67 %, w/w) and twigs (33 %, w/w) may be used in place of the MSW. The ratio of grass (typically rich in nitrogen) to leaves (typically low in nitrogen) should be adjusted to provide a C:N ratio of about 25:1.

8. Hazards

8.1 This test method requires the use of hazardous chemicals. Avoid contact with chemicals and follow manufacturer's instructions and Material Safety Data Sheets.

[8] The boldface numbers in parentheses refer to a list of references at the end of this test method.

TABLE 1 Mix 1[A]

Category	Wet Weight (%)	Wet Weight of Specific Component (%)
Food waste	16.6	Tomatoes (3.3), Lettuce (3.3), Meat (3.3), Cottage Cheese (3.3), Bread (3.4)
Garden waste	13.9	Leaves (6.9), Grass (7.0)
Paper	58.5	Bleached (19.5), Brown (19.5), Cardboard (19.5)
Plastics	7.7	Test material plus HDPE from shredded plastic milk bottles
Textiles	0.8	Cotton
Wood	2.5	Twigs

[A] See Practice D 5509.

TABLE 2 Mix 3 (2)

Component	Weight (g)	Carbon (g)	Nitrogen (g)	Water (g)
Water	214.1	...	...	214.1
Sand	107.0	...	...	...
Rabbit chow (Purina HF 5326)	72.1	30.9	1.7	7.2
Newspaper[A]	53.6	24.9	0.1	4.8
Manure composted cow	5.4	2.2	0.1	...
Total	452.2	58.0	1.9	226.1

[A] Shredded; ca. 2 mm × 25 mm.

8.2 The MSW may contain sharp objects; thus, to avoid injury, extreme care should be taken when handling such mixtures.

8.3 The bioreactor vessel is not designed to withstand high pressures; it should be operated at close to ambient pressure.

9. Inoculum

9.1 *Suitable inocula include*:

9.1.1 Commercial compost seed (for example, Recycle Compost Maker®, Ringer Corp., Minneapolis, MN).

9.1.2 Material from a commercial composting process, or previous composting exposure.

9.1.3 Composted potting soil from a garden supply store or a horizon material from a native soil.

10. Test Specimen

10.1 Test specimens should conform to Practice D 618.

10.1.1 Plastic materials may be tested in the form of films (25 mm × 75 mm) prepared by casting from solution or by melt forming (compression molding or extrusion).

10.1.2 Fabricated parts, or sections cut from fabricated parts, may also be used as test specimen.

NOTE 3—Test and control specimens should have essentially the same dimensions. An important factor in the choice of specimen configuration is the ratio of surface area to internal volume. Specimens of the same type of material but with different dimensions may produce percent weight loss values (not normalized to the surface area) that differ significantly. It may be necessary to test a range of forms and sizes to determine how this affects the extent or rate of biodegradation.

10.2 The test should include both degradable and nondegradable reference materials to follow the activity of the compost and standardize between-run testing.

10.2.1 Suitable positive reference materials include: uncoated cellophane and cellulose acetate with an average degree of substitution of less than or equal to two acetate esters per glucose monomer.

10.2.2 High-density polyethylene that has not been exposed to toxic materials (for example, the HDPE used to make plastic milk bottles) is suitable as the negative reference material.

NOTE 4—In devising a test program to determine quantitative changes occurring during and after composting, it is essential that the number of replicate specimen be sufficient to establish a reliable value for the property in question. For a homogeneous material, three test specimen per sampling point are usually adequate for assessing the visual effects of exposure or for determining weight-loss; however, five replicate specimens are usually required for assessing changes in tensile-properties. Always sample and test the same number of specimens for each exposure interval. It is to be expected that the physical properties of the specimens will vary as a function of exposure in the compost environment; hence