Designation: D7475 – 11

Standard Test Method for Determining the Aerobic Degradation and Anaerobic Biodegradation of Plastic Materials under Accelerated Bioreactor Landfill Conditions[1]

This standard is issued under the fixed designation D7475; 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 modification of Test Method D5526, which only considered anaerobic degradation, is used to determine the degree and rate of aerobic degradation (as indicated by loss of tensile strength, molecular weight, possibly resulting in disintegration and fragmentation) and anaerobic biodegradation of plastic materials in an accelerated bioreactor landfill test environment. It simulates the change from aerobic to anaerobic environments over time as landfill depth increases. Plastic materials found in landfills include discarded plastic products such as bags and wrappers and also deliberately applied plastic covers as inter-layer sealers between daily refuse fills to prevent windblown scatter of garbage overnight or at other down times. This modification is a two-tiered test method in which the two tiers, which address aerobic degradation and anaerobic biodegradation, are most preferably run sequentially to more closely resemble the real world condition of a biologically active landfill, or a bioreactor landfill, but are functional independently and separately depending on the plastic under evaluation and the information sought: either aerobic degradation or anaerobic biodegradation or both. The tiered system approach is shown schematically in Fig. 1. In Tier 1, the test plastic material is mixed with household waste, then pretreated and stabilized aerobically in the presence of air, in a sealed vessel in a temperature range that is consistent with the average temperature range of those recorded for landfills for a time period of four weeks. The tier is an accelerated simulation of degradation with concomitant oxygen consumption and depletion with time as if oxidative degradation proceeds. In Tier 2 samples of the plastic materials pretreated aerobically as described in Tier 1, are exposed to a methanogenic inoculum derived from anaerobic digesters operating only on pretreated household waste. The anaerobic decomposition and biodegradation occur under dry (more than 30 % total solids) and static non-mixed conditions. If it is desired to only assess anaerobic biodegradation of a plastic material, Tier 2 is run using preconditioned household waste, as described in Tier 1 but without the added plastic. The mixtures obtained from Tier 1 and Tier 2 in this test method are sampled and used to assess the environmental and health risks of plastic materials that are degraded in a landfill under aerobic and anaerobic conditions.

1.2 This test method generates comparative data for several materials and must not be used to make claims regarding benefits of placing degradable or biodegradable plastics in landfills. Claims must be limited to and dependent on the results obtained from each tier.

1.2.1 If only Tier 1 is run, then the claims must state: Will modify the performance/physical properties (for example, mechanical properties will degrade), up to a measured percent, X%, in a given time period, Y days using Test Methods D3593 (Molecular weight change) and Test Method D3826 (tensile strength change) in a biologically active "bioreactor" landfill. Report measured percent property changes and standards used to measure the test results which are, for example, changes in tensile strength, mass and molecular weight, as well as residual particle size ranges in Section 14 to support the extent of such claims.

1.2.2 If both Tier 1 and Tier 2 are run, then claims shall state: Will biodegrade in a biologically active "bioreactor" landfill to a degree, X%, in Y days established by the test results based on the extent to which the plastic sample is converted to gaseous carbon in the form of carbon dioxide and methane and this shall be made available according to Section 14 to support the extent of such claims. It should be noted that biodegradation testing is very dependent on conditions chosen in this laboratory test and may well vary widely when the test is run with different inoculum, The results reported pertain only to the test conditions run and do not rule out potential biodegradation under other conditions and real world environments.

1.3 Tier 1 of this test method is designed to estimate the aerobic degradation of plastics, that is disintegration and fragmentation, only, by measuring the loss of physical and chemical properties of said plastics. The test environment is then changed to that of Tier 2, an anaerobic condition, and

[1] This test method 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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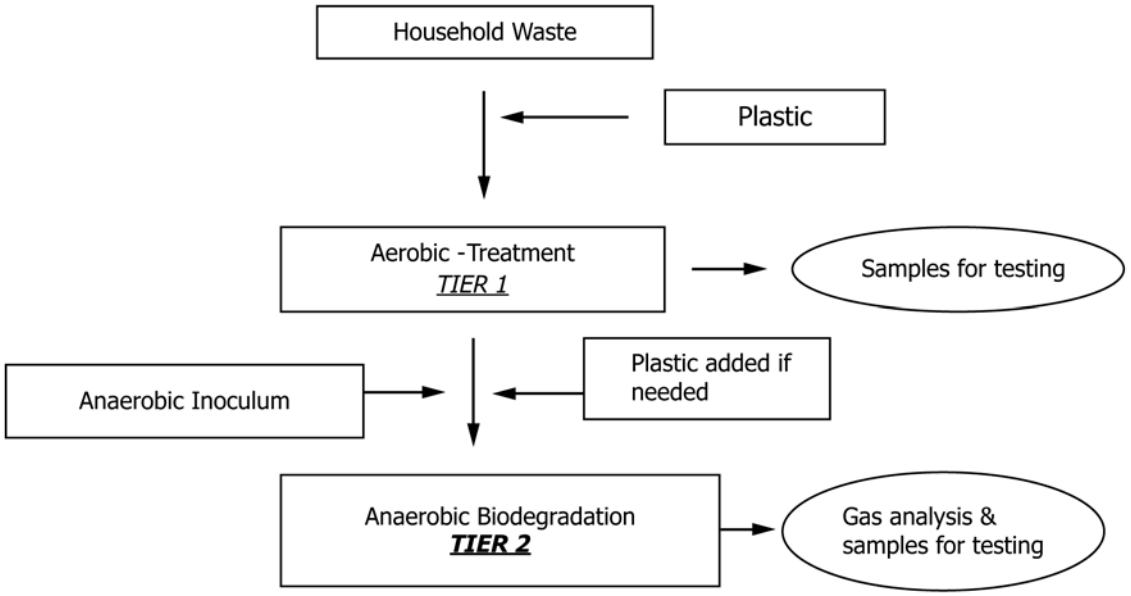


NOTE 1—The original D5526 schematic is represented by eliminating the household waste and plastic portion of that shown above.

FIG. 1 Schematic for testing Tiers 1 and 2 for Aerobic Degradation and Anaerobic Biodegradation

biodegradation is measured by a combination of evolved carbon dioxide and methane gases as a percentage of the conversion of carbon in the plastic sample to carbon in the gaseous form under conditions that resemble landfill conditions. This test method does not simulate all conditions found in landfills, especially those found in biologically inactive landfills. This test method more closely resembles those types of bioreactor landfills in which the gas generated is recovered or even actively promoted, or both, for example, by inoculation (co-deposition of anaerobic sewage sludge and anaerobic leachate recirculation), moisture control in the landfill (leachate recirculation), and temperature control (short-term injection of oxygen and heating of re-circulated leachate) (**1-7**).[2]

1.4 This test method produces partially degraded mixtures of municipal solid waste and plastics that, where required, are used to assess the ecotoxicological risks associated with the degradation of plastics after various stages of aerobic degradation and anaerobic biodegradation in a landfill.

1.5 The intended use of this method is for a comparison and ranking of aerobic degradation and anaerobic biodegradation of plastics after disposal in a bioreactor landfill. It is not designed or intended to be used to support claims recommending the value of plastic degradation in full-scale landfills. This simulation of an active landfill allows measurement of the percentage of aerobic degradation and anaerobic biodegradation (biogas evolution) in specified time periods, only.

1.6 Though the test method is in two tiers, they are meant to simulate a real world cycle of degradation in a landfill and are most preferably run consecutively and not independently or separately.

1.7 It is cautioned that the results of any laboratory landfill simulation cannot be directly extrapolated to actual disposal environments: confirmation to real world exposure is ultimately required as with all ASTM Standards. This confirmation is essential for landfill as the types of landfills vary widely, some are even heavily lined, tombs, and these will limit degradation severely.

1.8 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.9 *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 known ISO equivalent to this standard.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D618 Practice for Conditioning Plastics for Testing
D883 Terminology Relating to Plastics
D1293 Test Methods for pH of Water
D1888 Methods Of Test for Particulate and Dissolved Matter in Water (Withdrawn 1989)[4]
D2908 Practice for Measuring Volatile Organic Matter in Water by Aqueous-Injection Gas Chromatography
D3590 Test Methods for Total Kjeldahl Nitrogen in Water

[2] The boldface numbers in parentheses refer to a list of references at the end of this standard.

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

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)[4]
D3826 Practice for Determining Degradation End Point in Degradable Polyethylene and Polypropylene Using a Tensile Test
D4129 Test Method for Total and Organic Carbon in Water by High Temperature Oxidation and by Coulometric Detection
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)[4]
D6954 Guide for Exposing and Testing Plastics that Degrade in the Environment by a Combination of Oxidation and Biodegradation
E260 Practice for Packed Column Gas Chromatography
E355 Practice for Gas Chromatography Terms and Relationships

2.2 *APHA-AWWA-WPCF Standards:*[5]
2540D Total Suspended Solids Dried at 103°–105°C
2540E Fixed and Volatile Solids Ignited at 550°C
212 Nitrogen Ammonia

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method see Terminology D883.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *methanogenic inoculum*——anaerobically digested organic waste containing a high concentration of anaerobic methane-producing microorganisms.

3.2.2 *aerobic degradation of a plastic*—degradation of properties promoted by oxidation and is synonymous with defined oxidative degradation of plastics.

4. Summary of Test Method

4.1 *Combination Aerobic Degradation and Anaerobic Biodegradation*—This two-tiered test method described herein consists of the following: Tier 1 (aerobic degradation): (*1*) selecting and analyzing material for testing; (*2*) exposing the test plastic material for degradation in a sealed aerobic environment together with municipal solid waste during pretreatment and stabilizing (*3*) measuring oxidative degradations occurring in the plastic material by property changes over time. Tier 2 (anaerobic biodegradation): (*1*) either utilizing the degraded stabilized pretreated mixture of municipal solid waste and the plastic aerobically degraded (products from Tier 1) or combining a previously pretreated and stabilized solid municipal waste (in the absence of test plastic material) and new added plastic material with a concentrated anaerobic inoculum from an anaerobic digester; (*2*) exposing the mix to an anaerobic static batch fermentation at more than 30 % solids; (*3*) measuring total carbon in the gas (CO_2 and CH_4) evolved as a function of time; (*4*) removing the specimens for cleaning (optional), conditioning, testing, and reporting; (*5*) assessing the degree of degradability and/or biodegradability under less than optimum conditions.

4.2 The rate of aerobic degradation in Tier 1 is obtained by determining chemical and physical property changes, such as tensile strength, friability, molecular weight, or other selected characteristic with time, relative to the initial material.

4.3 The rate of environment conversion from aerobic to anaerobic is followed by the head space gas sampling and analysis of the reactor over time. Any increase in carbon dioxide or methane production indicates some biodegradation is occurring.

NOTE 2—Test Methods D3593 and D3826 are key standards that must be used for molecular weight and tensile strength measurements, though additional measurements are acceptable where considered appropriate. In all cases results must be recorded.

4.4 The percent and rate of conversion of carbon from the test material introduced in Tier 2 to carbon in the gaseous phase (methane and carbon dioxide) indicates the degree of anaerobic biodegradation.

4.5 If anaerobic biodegradation is the major focus and degradation under aerobic conditions is not of interest, the plastic material for evaluation is introduced only into Tier 2 using the pretreated solid municipal waste as in Tier 1.

4.6 It is recognized that the two Tiers are laboratory contrivances to allow the degradation stages, aerobic and anaerobic, to be studied independently where normally in the real world these are concurrent and or consecutive processes.

5. Significance and Use

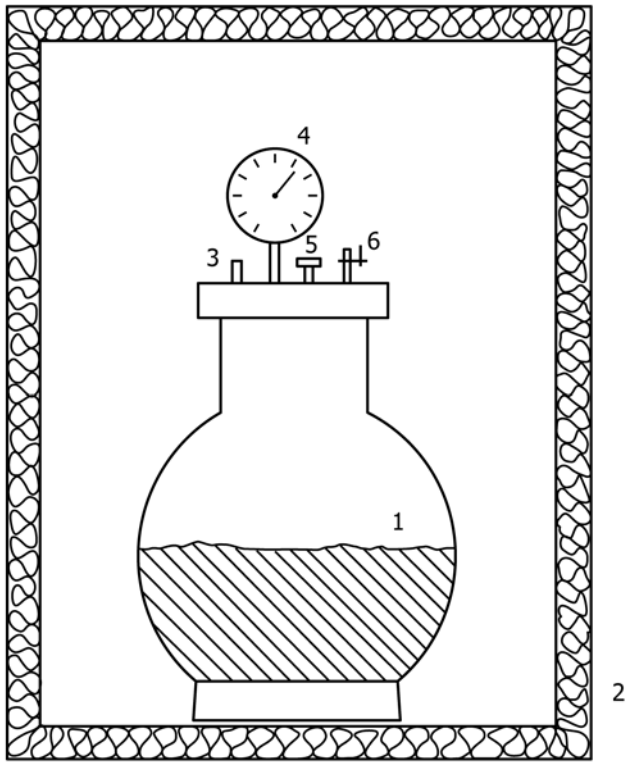
5.1 Decomposition of a plastic within a landfill involves processes in aerobic and anaerobic environmental conditions that can affect the decomposition of other materials enclosed by or in close proximity to the plastic. The rate of change from aerobic to anaerobic conditions is probably a characteristic of the particular landfill site, its garbage and the filling technique and is therefore difficult to assess with any degree of accuracy. Different sources indicate days to months (Refs **(8)** and **(9)**) for this change with the spread dependent on the perspective of what is aerobic or anaerobic and how fast the environment changes, 30 days is chosen in this method as a compromise time period. (Note, even very low levels of oxygen, far below normal atmospheric concentration can promote oxidative degradation). Obviously, there will be pockets of protected (in bags, cans, etc.) aerobic activity enclosed in any landfill. There is currently no evidence or data to support claims that rapid degradation of the plastic (when compared to conventional non-degradable plastic) can increase the economic feasibility of landfill-gas recovery, minimize the duration of after-care of the landfill, and make possible the recovery of the volume reduction of the waste due to degradation and biodegradation during the active life of the landfill. Additionally, it is possible that the rapid degradation and biodegradation of plastics can

[5] Standard Methods for the Examination of Water and Wastewater, 17th ed., 1989, available from American Public Health Association, 1740 Broadway, New York, NY 10018.

create hazardous conditions in landfills, such as the shifting of cells and overall stability. This standard method has been developed to permit determination of the aerobic degradation and anaerobic biodegradation of plastic products when placed in biologically active environments simulating some landfill conditions.

5.2 The decomposition of plastic materials in a landfill is of importance, as most landfills are biologically active and are an increasingly significant source of renewable energy. As degradation occurs in a landfill, it is of immediate concern that the plastic materials do not produce toxic metabolites or end products under the various conditions that occur in a landfill. The mixtures remaining after completion of the test method, containing fully or partially degraded plastic materials or extracts can be, when appropriate, submitted subsequently to ecotoxicity testing, see Practice D5951 and Guide D6954 for details, in order to assess the environmental hazards posed by the breakdown of plastics to varying degrees in landfills, especially if leaching occurs. This test method has been designed to assess aerobic degradation and anaerobic biodegradation under optimum and less-than-optimum conditions and toxicity.

5.3 *Limitations*—Because a wide variation exists in the construction and operation of landfills, and because regulatory requirements for landfills vary greatly, this procedure is not intended to simulate the environments of all landfills. However, it is expected to closely resemble the environment of a biologically active landfill. More specifically, the procedure is intended to create a standard laboratory environment that permits rapid and reproducible determination of the aerobic degradability and anaerobic biodegradability under accelerated landfill conditions, while at the same time producing reproducible mixtures of fully and partially decomposed household waste with plastic materials for ecotoxicological assessment.



1 = Digester.
2 = Incubation chamber.
3 = Overpressure valve.
4 = Manometer.
5 = Septum.
6 = Valve.

FIG. 2 Setup of Accelerated Landfill

6. Apparatus

6.1 *Pressure-Resistant Glass Vessels*—Twenty-seven, each with a volume of 4 to 6 L, which can be closed airtight and capable of withstanding an overpressure of two atmospheres. The lids of the reactors are equipped with an overpressure valve (to prevent the overpressure from becoming higher than two bars), a manometer that provides a rough indication of the overpressure, a septum that allows one to take gas samples and measure the exact overpressure, and, finally, a valve to release the overpressure (see Fig. 2).

6.2 *Incubators,* sufficient to store the vessels in the dark at 35 ± 2°C for the duration of the anaerobic testing in Tier 2.

6.3 *Pressure Transducer,* connected to a syringe needle to measure the headspace pressure in the test vessel.

6.4 *Gas Chromatograph,* or other apparatus, equipped with a suitable detector and column(s) for measuring methane and carbon dioxide concentrations in the evolved gases.

6.5 *pH Meter,* precision balance (±0.1 g), analytical balance (±0.1 mg), thermometer, and barometer.

6.6 *Suitable Devices,* for determining volatile fatty acids by aqueous-injection chromatography, total Kjeldahl nitrogen, ammonia nitrogen, dry solids (105°C), moisture content and volatile solids (550°C) concentrations.

7. Reagents and Materials

7.1 *Household Waste:* Derived from mixed municipal solid waste or the organic fraction thereof, after homogenizing, screening over a screen with holes of a diameter of 40 to 80 mm.

7.2 *Pretreated Household Waste:* Household waste aerobically stabilized over a period of 4 ± 2 weeks in an air flow and maintaining a dry-matter content of 50 ± 5 % and a temperature of 35 ± 2°C. (*Optional:* the pretreated household waste can be replaced by a similarly pretreated simulated solid waste.)

7.3 *Anaerobic Inoculum,* derived from a properly operating anaerobic digester with pretreated household waste as a sole substrate or a digester that treats predominantly household waste.

7.4 *Cellulose,* analytical-grade, or other suitable standards such as Kraft paper, thin-layer chromatography paper, etc. as a positive control[6] in the anaerobic Tier 2 testing.

[6] Avicel, available from EM Chemicals, Inc., Hawthorne, NY, was used for development of this test method.

7.5 *Polyethylene as a negative control for aerobic degradation in Tier 1.* The polyethylene must be in the same form as that in which the sample is tested: film polyethylene for film samples, pellets of polyethylene in case the sample is in the form of pellets, etc.

7.6 *Plastics* and other test materials, are included to ascertain aerobic degradation and anaerobic biodegradation under these accelerated test conditions.

7.7 *Fabricated polyethylene bags* with perforations for encapsulating test samples in whatever form they are to be tested, allowing for easy retrieval of samples.

8. Hazards

8.1 This procedure involves the use of inoculum and municipal solid waste containing biologically and possibly chemically active materials known to produce a variety of diseases. Avoid contact with these materials by wearing gloves and other appropriate protective equipment. Use good personal hygiene to minimize exposure.

8.2 The solid-waste mixture can sometimes contain sharp objects. Take extreme care when handling this mixture to avoid injury.

8.3 This test method includes the use of hazardous chemicals. Avoid contact with the chemicals and follow the manufacturer's instructions and material safety data sheets.

8.4 The methane produced during the procedure is explosive and flammable. Upon release of the biogas from the gas-collection system, take care in venting the biogas to the outside or to a hood.

9. Inoculum for Tier 2 Testing

9.1 The inoculum can be derived either from a laboratory-scale or full-scale continuous digester or batch digester, operating at 35°C and functioning with an organic fraction of household waste as the predominant substrate. In case the inoculum is derived from a continuous laboratory-scale or full-scale digester, the digester must be operating for a period of at least one month on the organic fraction of household waste, with a maximum retention time of 30 days under mesophilic conditions (35 ± 2°C). Gas production yields must be at least 15 mL at standard temperature and pressure of biogas/gram of dry solids in the digester and per day for at least seven days. In case the inoculum is derived from a batch digester, the gas production rate must have exceeded 1 L/kg waste/day, and the methane concentration of the biogas being produced must be above 60 %.

9.2 The prepared inoculum is allowed to undergo a short mesophilic post-fermentation of approximately seven days at the same dry-matter content as the digester from which it was derived. This means that the inoculum is not fed but is allowed to post-ferment anaerobically by itself. This is to ensure that large, easily biodegradable particles are degraded during this period and also to reduce the background level of degradation of the inoculum itself.

9.3 The biochemical characteristics of the inoculum are as follows:

9.3.1 *pH*—Between 7.5 and 8.5 (in accordance with Test Method D1293).

9.3.2 *Volatile Fatty Acids (VFA)*—Below 1 g/kg wet weight (in accordance with Practice D2908); and

9.3.3 NH_4^+-*N*—Between 0.5 and 2 g/kg (in accordance with APHA Test 212 and Test Method D3590).

9.4 Analyses are performed after dilution of the inoculum with distilled water on a ratio of distilled water to inoculum of 5 to 1 on a weight-over-weight basis.

10. Test Specimen

10.1 The test specimen must be of sufficient carbon content, analyzed in accordance with Test Method D4129, to yield carbon dioxide and methane volumes that can be measured accurately by the trapping devices described. Add more test specimen when low biodegradability is expected, up to 100 g of dry matter of the test specimen.

10.2 The test specimen must be in the form that the product will be used such as film or formed article, or in the form of a dog bone and in accordance with Practice D618. The test setup is capable of handling articles that are 100 by 50 by 4 mm thick.

11. Procedures

11.1 *Tier 1:* Aerobic degradation during pretreatment of household waste

11.2 Specimens and controls (polyethylene or other suitable plastics) are characterized (for molecular weight by Test Method D3593 for tensile strength by Test Method D3826), other properties measured and methods used to accomplish them, such as range of particle sizes obtained through disintegration, must be included in the test report.

11.2.1 Place the specimens and controls in perforated polyethylene bags (these do not degrade under the test conditions).

11.2.2 Mix the bags containing the test specimens, with the household waste at a specimen weight ratio of 1:10. The same operation is done with the control specimen, in parallel.

11.2.3 Place this mixture in the pressure-resistant glass vessel and pass air through the mixture for at least 30 minutes, and then seal the vessel to prevent further oxygen entry to the reactor. Heat the reactor to 30°C and maintain at 30 ± 10°C for at least four weeks in the dark, and continue until gas volume change in the reactor ceases.

NOTE 3—The intent here in this modification is to accelerate oxidation and degradation and the higher temperature is consistent with published information on landfill temperature cycles (Refs. (**8**) and (**9**)).

11.3 When appropriate or required, remove sufficient residual specimen material and control material from the perforated bag in the vessel and also a sample of the pretreated household waste (to ensure no toxic substance migration from the bags) during and on completion of the procedure and submit to ecotoxicity testing, in accordance with appropriate standard test methods and practices.

11.4 Head space gas samples are taken at suitable intervals to estimate the rate of anaerobic development. Report changes in oxygen levels, carbon dioxide and any methane formation.

11.5 Premature opening of the reactor is not allowed due to the danger of atmosphere changes. If intermediate results are desirable, it is recommended to make multiple runs and to terminate them at selected intervals to obtain samples for analysis. Measure molecular weight in accordance with Test Method D3593 and tensile properties in accordance with Test Method D3826. If other properties are measured determine them by the methods used for the initial measurements. Determine property changes in accordance with Section 12 and report the changes in accordance with Section 12 and report the changes in accordance with Section 14 for Tier 1 testing.

11.6 *Tier 2:* Anaerobic biodegradation

11.7 *Preparation of the Mixtures:*

11.7.1 Determine the volatile solids, dry solids, and nitrogen content of the pretreated household waste (and plastic material when Tier 1 product is used) and the inoculum in accordance with Test Methods D1888, D3590, and APHA 2540D and 2540E.

11.7.2 Determine the volatile solids, dry solids, and carbon content of all plastic test substances in accordance with APHA 2540D and 2540E and Test Method D4129.

11.7.3 Weigh and combine the components and adjust the dry matter content of the final mixtures with water to reach the desired dry-matter content for each vessel. Roughly weigh out 600 g on a dry-weight basis of pretreated household waste, and mix it with 100 g on a dry-weight basis of mesophilic anaerobic inoculum from a continuously operating digester or 150 g on a dry-weight basis of anaerobic inoculum from a batch digester. Add 60 to 100 g of dry matter of the plastic test substance. Add water until the appropriate final dry matter content is reached. (In order to reach 60 % dry matter content in the mixture, when necessary remove water prior to combining the different components of the mixture. This can be accomplished by drying the pretreated household waste or centrifuging the anaerobic inoculum.) Mix the required amounts of pretreated household waste, inoculum, and test substance in a small container for 2 to 3 minutes. Introduce the mixture in the vessel, weigh the vessel with all of the contents, and close it airtight. Prepare the pressure vessels in the triplicate at each of the following dry matter contents: 35, 45, and 60 %, so nine vessels are necessary for each plastic sample.

11.7.4 The blanks consist of 600-g dry matter of pretreated household waste and anaerobic inoculum at the respective total dry-matter contents. As references, thin-layer chromatography cellulose must be used as a positive control. The blank and reference are performed in triplicate at the three different dry-matter contents.

11.8 *Start-Up Procedure*—After all reactors are filled and closed, place them in incubators at 35 ± 2°C. Acclimate the reactors for approximately one hour and release the pressure, which originates from the temperature increase, to the atmosphere. Incubate the reactors in the dark for a period of four months.

11.9 *Operating Procedure:*

11.9.1 Check the gas production (measured as a pressure increase) at least weekly. When the overpressure reaches more than 700 mbar, measure the pressure exactly with the pressure transducer and release to atmospheric pressure. Take care that the temperature decrease, due to the opening of the incubator or incubation room, is not more than 1°C during measurement of the overpressure.

11.9.2 Analyze the gas composition periodically, biweekly or longer as the composition stabilizes. Determine the methane and carbon dioxide concentration by using analytical devices suitable for the detection and quantification of these gases, such as a gas chromatograph with an appropriate detector, conforming to Practices E260 and E355. Pay special attention to the occurrence of leaks through the septum.

11.10 *End of the Test:*

11.10.1 The incubation time is extended, depending on the activity of the inoculum, until no significant gas production in excess of the blank has been recorded during one week. The positive control reference must reach 70 % conversion to gaseous carbon for the test to be valid and the time for this must be recorded. At this point in time, the extent of gas evolution from the sample should be recorded. as an indication of relative biodegradation rates.

11.10.2 At the end of the test, analyze the dry matter, volatile fatty acids, and pH for each of the reactor mixtures, in accordance with APHA 2540E, Practice D2908, and Test Methods D1293

11.10.3 Remove sufficient residual material from the vessel and submit to ecotoxicity testing, in accordance with appropriate standard test methods and practices (optional).

12. Calculation

12.1 *Tier 1: Aerobic Degradation*

12.1.1 Calculate the changes in physical and chemical properties before and after exposure that correlate with degradation.

12.1.2 Changes in tensile strength, melt flow index, molecular weight, friability, particle size, spectroscopic carbonyl index in infra red spectra measured by usual standard procedures are all indicators that are used. Molecular weight, Test Method D3593, and tensile strength, Test Method D3826, changes must be recorded, others changes are discretionary,

12.2 *Tier 2: Anaerobic Biodegradation*

12.2.1 By using the total carbon in the test specimen, calculate the maximum theoretical gas production (carbon dioxide plus methane) originating from the anaerobic biodegradation of the test specimen, based on the following biochemical transformations:

$$C + 2H_2 \rightarrow CH_4 \quad (1)$$

$$C + O_2 \rightarrow CO_2$$

Each millimole (12 mg) of organic carbon from the test sample can be converted into 1 mmole of gaseous CH_4 or CO_2, or a mixture of the two. One millimole of gas produced occupies 22.4 mL at standard temperature and pressure (STP).

12.3 *Temperature and Pressure*—Measure the percentages of CH_4 and CO_2, and transform the gas volumes to STP. Also correct for vapor pressure and atmospheric pressure variation during the test. Calculate the amount of gaseous carbon.

Determine the mean (of the three replicates) net gaseous carbon production by anaerobic biodegradation of the test substances by subtracting the mean gaseous carbon production of the control (three replicates) containing only the inoculum.

12.4 Calculate the percent of biodegradation for each dry-matter concentration by dividing the average net gaseous-carbon production of the test material by the original average amount of total carbon of the test compound and multiplying by 100.

$$\%\ biodegradation = \frac{mean\ C_g\ (test) - mean\ C_g\ (blank)}{C_i} \times 100 \quad (2)$$

where:

C_g = amount of gaseous carbon produced, g, and
C_i = amount of carbon in test compound added, g.

Calculate the standard error, s_e, of the percentage of biodegradation as follows:

$$s_e = SQRT\left(\left(s_{test}^2/n1\right) + \left(SQRT\left(s_{blank}^2/n2\right)\right) \times 100 / C_i \right) \quad (3)$$

where:

$n1$ *and* $n2$ = number of replicate test and blank digesters, respectively, and
s = standard deviation of the total gaseous carbon produced.

Calculate the 95 % confidence limits as follows:

$$95\ \%\ CL = \%\ biodegradation \pm \left(t \times s_e\right) \quad (4)$$

where:

t = t-distribution value for 95 % probability with (n1+n2–2) degrees of freedom; thus n=3+3–2=4.

13. Interpretation

13.1 Information on the rate and degree of aerobic degradation and anaerobic biodegradation of plastics in landfill disposal will permit comparisons of degradation potential of different plastic materials. While this test method generates comparative data for several materials, it does not support any overall benefits to degradation, or biodegradation, or both, under conditions found in landfills.

13.2 Information on toxicity is also to be established, where appropriate.

14. Report

14.1 *Tier 1:* Report the following:

14.1.1 Temperature range of the test plotted over the time period of the test.

14.1.2 Duration of the test.

14.1.3 Indicators of degradation reported at zero time and at test completion. Intermediate values also reported where available. Report any indication of crosslinking of the test material.

14.1.4 Information on the household waste samples used in the pretreatment stage where aerobic degradation is performed.

14.1.5 Resin grade plus the commercial name of the formulation and additives or the level of catalyst used.

NOTE 4—Identification of test samples needs to be sufficient to inform the readers of the commercial identification of the formulation and the additives and their availability in the market place.

14.1.6 Complete mass balances to be recorded.

14.1.7 Report gas composition changes in head space, if measured, and rate and time to reach anaerobic conditions.

14.1.8 Changes in sample molecular weight, weight, tensile, or other used characteristic. relative to control are key requirements to be reported.

14.1.9 Report the form of the samples tested.

14.1.10 Toxicity testing results, if generated.

14.2 *Tier 2:* Report the following:

14.2.1 Information on the inoculum, including source, pH, volatile fatty acids (in milligram per kilogram wet weight), NH_4^+-N (in gram per kilogram wet weight), percent dry solids, percent volatile solids, date of collection and use, storage time and conditions, handling, and potential acclimation to the test material.

14.2.2 Information on the pretreated household waste used to produce the inoculum and used as a substance. In case a simulated solid waste is used, report the composition of the mix. For both pretreated household waste and simulated solid waste, report the source, pH, type of pretreatment, NH_4^+-N (in gram per kilogram), percent dry solids, percent volatile solids, date of collection, storage time and conditions, handling, and transportation.

14.2.3 Carbon content of the plastic material and the positive control and maximum theoretical gas production (carbon dioxide and methane) for each.

14.2.4 Record and display graphically the cumulative gas evolution over time.

14.2.5 Analysis of gas as percent methane and percent carbon dioxide for each reading at the end of the test, or each time the gas is released to the atmosphere during the course of the test. Concomitantly, report the barometric pressure and temperature in the incubator and in the gas-collection device.

14.2.6 Record the percent of carbon conversion, along with the form of plastic material, that is, sheet, powder, pellets, etc. Record specific information on the size, shape, volume, and thickness of the plastic materials and control substances tested.

14.2.7 Percent of biodegradation and time to attain relative to cellulose.

14.2.8 Report extent of biodegradation of the cellulose or other standard to support inoculum activity.

14.2.9 Standard deviation and 95 % confidence interval for the percentage of biodegradation for each triplicate set.

14.2.10 In case biogas production has not reached a plateau for the vessels at 45 and 60 % dry matter, report total biogas production as percentage of total biogas production at 35 % dry matter.

14.2.11 Wet-weight loss (optional).

14.2.12 Toxicity results, if tested.

14.3 Combined test results for aerobic degradation and anaerobic biodegradation when applicable.

NOTE 5—The reporting of these results, above, shall not convey to the reader that the degradation and biodegradation of the plastic materials provides an overall benefit to the landfill, its contents, the conditions found in the landfill, the amount of gas recovered or recovery rate. Unqualified claims of biodegradability (for example, completely biodegradable) cannot be made. Biodegradation and degradation rates and final levels can be measured and claimed only to extent of X% in Y days as measured in the specific test and inoculum used, results my be completely different with other conditions. Extrapolations must not be made. This is the reason

multiple tests are recommended before assurance of biodegradation or non-biodegradation can be made. Additionally, it is important that the report should limit its results only to landfills that are actively managed to promote and recover landfill gas.

15. Precision and Bias

15.1 The precision and bias of the procedure presented in this test method for measuring both the aerobic degradation and the anaerobic biodegradation of plastic materials under accelerated landfill conditions is being determined.

15.2 Preliminary results for anaerobic biodegradation of household waste at 40 % dry-matter content and 35°C are presented in Fig. 3. The curves in Fig. 2 represent the biogas production in litres from 1 kg of pretreated household waste plus 10 % mesophilic inoculum without sample (blank) and with 60 g of cellulose (plus positive control).

16. Keywords

16.1 accelerated landfill; anaerobic biodegradation; biodegradation; aerobic degradation; dry digestion; ecotoxicity; landfill; metabolites; plastics; test method

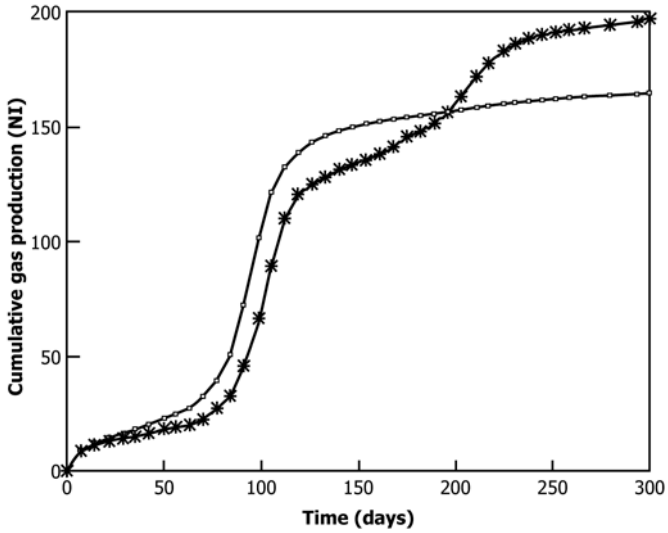


FIG. 3 Cumulative Biogas Production Over a Period of 300 Days for 1 kg of Pretreated Household Waste Plus 100 g of Mesophilic Anaerobic Inoculum without Sample (Blank) and with 60 g of Cellulose (Plus Positive Control)

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