Designation: D5526 – 18

Standard Test Method for Determining Anaerobic Biodegradation of Plastic Materials Under Accelerated Landfill Conditions[1]

This standard is issued under the fixed designation D5526; 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 covers determination of the degree and rate of anaerobic biodegradation of plastic materials in an accelerated-landfill test environment. This test method is also designed to produce mixtures of household waste and plastic materials after different degrees of decomposition under conditions that resemble landfill conditions. The test materials are mixed with pretreated household waste and exposed to a methanogenic inoculum derived from anaerobic digesters operating only on pretreated household waste. The anaerobic decomposition occurs under dry (more than 30 % total solids) and static nonmixed conditions. The mixtures obtained after this test method can be used to assess the environmental and health risks of plastic materials that are degraded in a landfill.

1.2 This test method is designed to yield a percentage of conversion of carbon in the sample to carbon in the gaseous form under conditions that resemble landfill conditions. It is possible that this test method will not simulate all conditions found in landfills, especially biologically inactive landfills. This test method more closely resembles those types of landfills in which the gas generated is recovered or even actively promoted, or both, for example, by inoculation (codeposition 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 recirculated leachate) (**1-7**).[2]

1.3 This test method is designed to produce partially degraded mixtures of municipal solid waste and plastics that can be used to assess the ecotoxicological risks associated with the anaerobic degradation of plastics after various stages of anaerobic biodegradation in a landfill.

1.4 Claims of performance shall be limited to the numerical result obtained in the test and not be used for unqualified "biodegradable" claims. Reports shall clearly state the percentage of net gaseous carbon generation for both the test and reference samples at the completion of the test. Furthermore, results shall not be extrapolated past the actual duration of the test.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific hazards statements are given in Section 8.

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

1.7 *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:*[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

D4129 Test Method for Total and Organic Carbon in Water by High Temperature Oxidation and by Coulometric Detection

E260 Practice for Packed Column Gas Chromatography

[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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[2] The boldface numbers in parentheses refer to the 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.

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.

4. Summary of Test Method

4.1 This test method described consists of the following: (*1*) selecting and analyzing material for testing; (*2*) obtaining a pretreated municipal-solid-waste fraction and a concentrated anaerobic inoculum from an anaerobic digester; (*3*) exposing the material to an anaerobic static batch fermentation at more than 30 % solids; (*4*) measuring total carbon in the gas (CO_2 and CH_4) evolved as a function of time; (*5*) removing the specimens for cleaning (optional), conditioning, testing, and reporting; (*6*) assessing the degree of biodegradability; and (*7*) assessing the degree of biodegradability under less than optimum conditions.

4.2 The percentage of biodegradability is obtained by determining the percent of conversion of carbon from the test material to carbon in the gaseous phase (CH_4 and CO_2). This percentage of biodegradability will not include the amount of carbon from the test substance that is converted to cell biomass and that is not, in turn, metabolized to CO_2 and CH_4.

5. Significance and Use

5.1 Decomposition of a plastic within a landfill involves biological processes that will affect the decomposition of other materials enclosed by, or in close proximity to, the plastic. Rapid degradation of the plastic has the ability to 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 biodegradation during the active life of the landfill. This procedure has been developed to permit determination of the anaerobic biodegradability of plastic products when placed in biologically active environments simulating landfill conditions.

5.2 As degradation occurs inevitably 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 have the potential to occur in a landfill. The mixtures remaining after completion of the test method, containing fully or partially degraded plastic materials or extracts, can be submitted subsequently to ecotoxicity testing in order to assess the environmental hazards posed by the breakdown of plastics to varying degrees in landfills. This test method has been designed to assess biodegradation under optimum and less-than-optimum conditions.

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 environment 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 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.

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 2 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 (Fig. 1).

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

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.

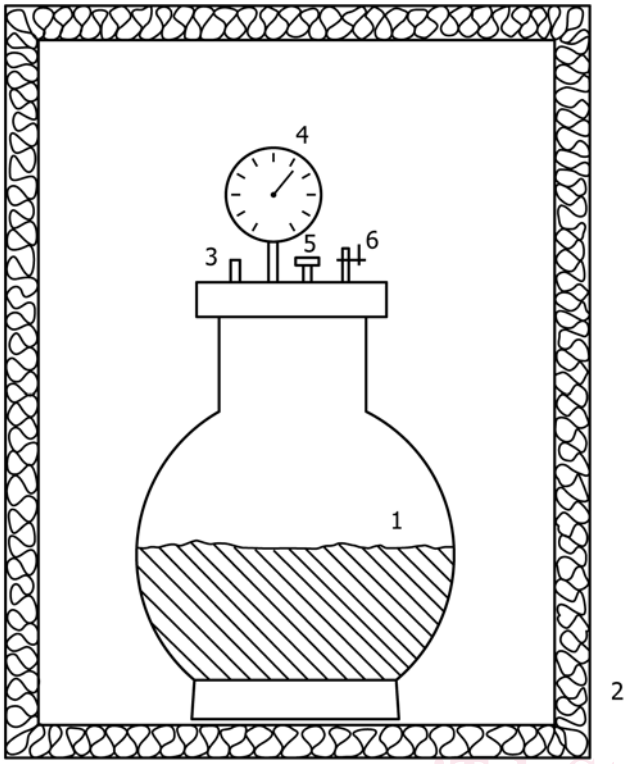
6.6 *Suitable Devices,* for determining volatile fatty acids by aqueous-injection chromatography, total Kjeldahl nitrogen, ammonia nitrogen, dry solids (105°C), and volatile solids (550°C) concentrations.

7. Reagents and Materials

7.1 *Pretreated-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, and aerobically stabilized over a period of 2 to 4 weeks by blowing air into the material and maintaining a dry-matter content of 50 ± 5 % and a temperature of 55 ± 10°C. (Optional: the pretreated household waste can be replaced by a similarly pretreated simulated solid waste.)

7.2 *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.

[5] *Standard Methods for the Examination of Water and Wastewater*, 20th ed., 1999, available from American Public Health Association, 800 I Street, NW, Washington, D.C. 20001-3710, or http://www.standardmethods.org.



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

FIG. 1 Setup of Accelerated Landfill

7.3 *Cellulose, Analytical-Grade,* for thin-layer chromatography as a positive control.[6]

7.4 *Polyethylene* (optional), as a negative control. It needs to 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.

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 It is possible that the solid waste mixture will 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

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 7 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 needs to undergo a short mesophilic post-fermentation of approximately 7 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 shall be as follows:

9.3.1 *pH*—Between 7.5 and 8.5 (in accordance with Test Methods 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 needs to 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 It is acceptable for the test specimen to be in the form of films, powder, pellets, or formed articles, or in the form of a dog bone and in accordance with Practice D618.

11. Procedure

11.1 *Preparation of the Mixtures:*

11.1.1 Determine the volatile solids, dry solids, and nitrogen content of the pretreated household waste and the inoculum in accordance with Test Methods D1888, D3590, and APHA 2540D and 2540E.

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