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Standard Test Method for Determining Anaerobic Biodegradation of Plastic Materials Under High-Solids Anaerobic-Digestion Conditions[1]

This standard is issued under the fixed designation D5511; 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 the determination of the degree and rate of anaerobic biodegradation of plastic materials in high-solids anaerobic conditions. The test materials are exposed to a methanogenic inoculum derived from anaerobic digesters operating only on pretreated household waste. The anaerobic decomposition takes place under high-solids (more than 30 % total solids) and static non-mixed conditions.

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 found in high-solids anaerobic digesters, treating municipal solid waste **(1, 2, 3, 4)**.[2] This test method may also ~~resembles other anaerobic~~ resemble some conditions in biologically active landfills where the gas generated is recovered and biogas production is actively promoted by inoculation (for example, codeposition of anaerobic sewage sludge, anaerobic leachate recirculation), moisture control (for example, leachate recirculation), and temperature control (for example, short-term injection of oxygen, heating of recirculated leachate) **(5, 6, 7)**.

1.3 This test method is designed to be applicable to all plastic materials that are not inhibitory to the microorganisms present in anaerobic digesters operating on household waste.

1.4 Claims of performance shall be limited to the numerical result obtained in the test<usb> 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 given 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.* Specific hazards are given in Section 8.

NOTE 1—This test method is equivalent to ISO 15985.

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 ~~Test Methods for Particulate and Dissolved Matter, Solids, or Residue in Water~~ Methods Of Test for Particulate and Dissolved Matter in Water

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

E355 Practice for Gas Chromatography Terms and Relationships

2.2 *APHA-AWWA-WPCF Standards:*

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[1] This test method is under the jurisdiction of ASTM Committee of 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 is parentheses refer to a list of references at the end of the text.

[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.

2540 D Total Suspended Solids Dried at 103°–105°C[4]
2540 E Fixed and Volatile Solids Ignited at 550°C[4]
212 Nitrogen Ammonia[4]
2.3 *ISO Standard:*[5]
ISO 13641-1 Water quality—Determination of inhibition of gas production of anaerobic bacteria—Part 1: General test
ISO 15985 Plastics—Determination of the ultimate anaerobic biodegradability and disintegration under high-solids anaerobic-digestion conditions—Method by analysis of released biogas

3. Terminology

3.1 *Definitions*—Definitions of terms applying to this test method appear in 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 consists of selection and analysis of material for testing, obtaining a concentrated anaerobic inoculum from an anaerobic laboratory-scale digester, exposing the material to an anaerobic-static-batch fermentation at more than 20 % solids, measuring total carbon in the gas (CO_2 and CH_4) evolved as a function of time, and assessing the degree of biodegradability.
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 Biodegradation of a plastic within a high-solids anaerobic digestion unit is an important phenomenon because it will affect the decomposition of other waste materials enclosed by the plastic and the resulting quality and appearance of the ~~compost~~digestate after an anaerobic digestion process. Biodegradation of plastics could allow for the safe disposal of these plastics through aerobic and anaerobic solid-waste-treatment plants. This procedure has been developed to permit the determination of the rate and degree of anaerobic biodegradability of plastic products when placed in a high-solids anaerobic digester for the production of ~~compost~~digestate from municipal solid waste.
5.2 *Limitations*—Because there is a wide variation in the construction and operation of anaerobic-digestion systems and because regulatory requirements for composting systems vary, this procedure is not intended to simulate the environment of any particular high-solids anaerobic-digestion system. However, it is expected to resemble the environment of a high-solids anaerobic-digestion process operated under optimum conditions. More specifically, the procedure is intended to create a standard laboratory environment that will permit a rapid and reproducible determination of the anaerobic biodegradability under high-solids digestion conditions.

6. Apparatus

6.1 *Inverted Graduated Cylinder or Plastic Column*, in water or other suitable device for measuring gas volume. The water in contact with the gas must be at a pH of less than two during the whole period of the test to avoid CO_2 loss through dissolution in the water. The gas-volume-measuring device, as well as the gas tubing, shall be of sufficient quality to prevent gas migration and diffusion between the system and the surrounding air (see Fig. 1).
6.2 *Gas Chromatograph*, (optional) or other apparatus, equipped with a suitable detector and column(s) for measuring methane and carbon dioxide concentration in the evolved gases.
6.3 *Incubator*, or hot-water bath capable of maintaining the test bottles at 37°C (±2°C) or 52°C (±2°C) for the duration of the test.
6.4 *Erlenmeyer Flasks*, with sufficient capacity for the experiment and openings of at least 7-cm diameter, set up so that no loss of gas occurs.
6.5 *pH Meter*, precision balance (±0.1 g), analytical balance (±0.1 mg), thermometer, and barometer.
6.6 *Devices*, suitable 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 *Anaerboic Inoculum*, derived from a properly operating anaerobic digester with pretreated household waste as a sole substrate.
7.2 *Analytical-Grade Cellulose* , for thin-layer chromatography as a positive control.

[4] *Standard Methods for the Examination of Water and Wastewater*, 17th Edition, 1989, American Public Health Association, 1740 Broadway, New York, NY 10018.
[5] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

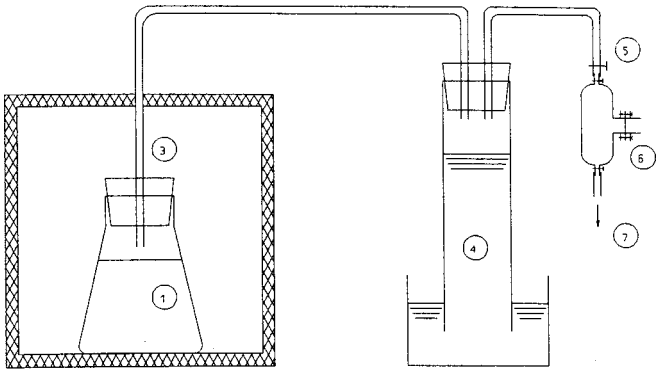

FIG. 1 Test Setup

7.3 *Polyethylene*, as a negative control (optional). It is optimal if it is in the same form as the form in which the sample is tested (for example, film polyethylene for film samples, pellets of polyethylene if the sample is in the form of pellets, etc.).

8. Hazards

8.1 The procedure given in this test method involves the use of an inoculum composed of 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 garments. Use good personal hygiene to minimize exposure.

8.2 It is possible that the solid-waste mixture contains sharp objects. Take extreme care when handling this mixture to avoid injury.

8.3 The biological reactor is not designed to withstand high pressures; operate it at close to ambient pressure.

8.4 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.

8.5 The methane produced during this 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 must be derived from a properly operating anaerobic digester functioning with a pretreated household waste as a sole substrate. The pretreated household waste shall come from an existing waste treatment facility treating municipal solid waste, where through sorting, shredding, sieving, or other means, a fairly homogeneous organic fraction is produced of less than 60 mm. The digester shall be operating for a period of at least four months on the organic fraction, with a retention time of a maximum of 30 days under thermophilic conditions (52 ± 2°C). Gas-production yields shall be at least 15 mL at standard temperature and pressure of biogas per gram of dry solids in the digester and per day on the average for at least 30 days.

9.1.1 It is preferable to derive the inoculum from a digester operating under dry (>20 % total solids) conditions, but it is acceptable to derive it from a wet fermentation whereby the anaerobically digested sludge is dewatered through centrifugation, with a press or through drying at a maximum temperature of 55°C to a dry-solids content of at least 20 %.

9.2 The prepared inoculum shall undergo a short post-fermentation of approximately seven days at the same operating temperature from which it was derived. This means that the inoculum is not fed but 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.

9.2.1 The most important biochemical characteristics of the inoculum shall be as follows:

9.2.1.1 *pH*—Between 7.5 and 8.5 (in accordance with Test Methods D1293),

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

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

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

10.2 It is acceptable if the test specimen is in the form of films, powder, pellets, formed articles, or in the form of a dog bone and conforming to Practice D618. The test set-up shall be able to handle articles that are 100 mm by 50 mm by 4 mm thick.

11. Procedure

11.1 *Inoculum Medium*:

11.1.1 Remove enough inoculum (approximately 15 kg) from the post-fermentation vessel and mix carefully and consistently by hand in order to obtain a homogeneous medium.

11.1.2 Test three replicates each of a blank (inoculum only), positive control (thin-layer chromatography cellulose), negative control (polyethylene), and the test substance being evaluated.

11.1.2.1 Manually mix 1000 g wet weight (at least 20 % dry solids) of inoculum in a small container for a period of 2 to 3 min with 15 to 100 g of volatile solids of the test substance or the controls for each replicate. (Determine dry solids and volatile solids in accordance with APHA Standards , , and Test Method D1888).

11.1.2.2 For the three blanks containing inoculum only, manually mix 1000 g of the same inoculum in a small container for a period of 2 to 3 min with the same intensity as was done for the other vessels containing test substance or controls.

11.1.2.3 Determine the weight of the inoculum and test substance added to each individual Erlenmeyer flask accurately.

11.1.2.4 If formed plastic articles are added, it is possible that a specific number of articles be added and retrieved at the end of the digestion period.

11.1.3 Add the mixtures to a 2-L wide-mouth Erlenmeyer flask and gently spread and compact the material evenly in the flask to a uniform density.

11.1.4 After placing the Erlenmeyer flask in a water bath or incubator, connect it with the gas-measurement or gas-collection device.

11.1.5 Record room temperature and atmospheric pressure prior to turning on the heating system of the incubator or water bath.

11.2 *Incubation*:

11.2.1 Incubate the Erlenmeyer flasks in the dark or in diffused light at 52°C (±2°C) for thermophilic conditions, or 37°C (±2°C) for mesophilic conditions for a period of normally 15-30 days.

11.2.1.1 For the test to be considered valid, the positive control must achieve 70 % biodegradation within 30 days.

11.2.1.2 The incubation time shall be run until no net gas production is noted for at least five days from both the positive control and test substance reactors.

11.2.1.3 The test substance and the positive control shall be run for the same duration.

11.2.2 Control the pH of the water used to measure biogas production to less than two by adding HCl.

11.3 *Analytical Measurements*:

11.3.1 Make at least five measurements of gas volume per week in order to establish the gas production as a function of time.

11.3.2 Determine 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, in accordance with Practices E260 and E355.

11.3.3 Verify the quality of the inoculum by analyses for pH, volatile fatty acids, and total Kjeldahl nitrogen (in accordance with Test Methods D1293 and D3590 and Practice D2908).

11.4 At the end of the digestion period, allow the setup to cool to room temperature for 8 h and determine the following parameters:

11.4.1 Total gas-volume production produced during the test,

11.4.2 Gas composition at the end of the test,

11.4.3 Weight loss of each vessel, and

11.4.4 Room temperature and atmospheric pressure at the end of the test.

12. Calculation

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

$$C + 2\,H_2 \rightarrow CH_4$$
$$C + O_2 \rightarrow CO_2 \quad (1)$$

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

12.2 *Temperature and Pressure*—Measure the percentages of CH_4 and CO_2 and transform the gas volumes to STP. Correct also for water 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.

12.3 Calculate the percent of biodegradation 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:

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