Designation: D5338 – 15 (Reapproved 2021)

Standard Test Method for Determining Aerobic Biodegradation of Plastic Materials Under Controlled Composting Conditions, Incorporating Thermophilic Temperatures[1]

This standard is issued under the fixed designation D5338; 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 determines the degree and rate of aerobic biodegradation of plastic materials on exposure to a controlled-composting environment under laboratory conditions, at thermophilic temperatures. This test method is designed to yield reproducible and repeatable test results under controlled conditions that resemble composting conditions, where thermophilic temperatures are achieved. The test substances are exposed to an inoculum that is derived from compost from municipal solid waste. The aerobic composting takes place in an environment where temperature, aeration and humidity are closely monitored and controlled.

NOTE 1—During composting, thermophilic temperatures are most readily achieved in large-scale, professionally-managed facilities. However, these temperatures may also be reached in smaller residential composting units, frequently referred to as "backyard" or "home" composting.

1.2 This test method is designed to yield a percentage of conversion of carbon in the sample to carbon dioxide. The rate of biodegradation is monitored as well.

1.3 This test method is designed to be applicable to all plastic materials, which are intended to be composted in facilities that achieve thermophilic temperatures.

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

1.5 *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 hazard statements are given in Section 8.

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1.7 This test method is equivalent to ISO 14855.

1.8 *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:*[2]

D618 Practice for Conditioning Plastics for Testing

D883 Terminology Relating to Plastics

D1293 Test Methods for pH of 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:*

2540 D Total Suspended Solids Dried at 103 to 105°C[3]

2540 E Fixed and Volatile Solids Ignited at 550°C[3]

2.3 *ISO Standard:*

ISO 14855 Plastics—Evaluation of the Ultimate Aerobic Biodegradability and Disintegration Under Controlled Composting Conditions—Method by Analysis of Released Carbon Dioxide[4]

[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.

Current edition approved Jan. 15, 2021. Published January 2021. Originally approved in 1992. Last previous edition approved in 2015 as D5338 - 15. DOI: 10.1520/D5338-15R21.

[2] 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.

[3] *Standard Methods for the Examination of Water and Wastewater,* 17th Edition, 1989, American Public Health Association, 1740 Broadway, New York, NY 19919.

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

3. Terminology

3.1 *Definitions*—Definitions of terms applying to this test method appear in Terminology D883.

4. Summary of Test Method

4.1 This test method consists of the following:

4.1.1 Selection of plastic material for the determination of the aerobic biodegradability in a controlled-composting system,

4.1.2 Obtaining an inoculum from composted municipal solid waste,

4.1.3 Exposing the test substances to a controlled aerobic composting process in conjunction with the inoculum,

4.1.4 Measuring carbon dioxide evolved as a function of time, and

4.1.5 Assessing the degree of biodegradability.

4.2 The percentage of biodegradability is obtained by determining the percentage of carbon in the test substance that is converted to CO_2 during the duration of the test. This percentage of biodegradability will not include the amount of carbon converted from the test substance that is converted to cell biomass and that is not, in turn, metabolized to CO_2 during the course of the test.

4.3 The disintegration of a compact test material is visually determined at the end of the test. Additionally, the weight loss of the test material may be determined.

5. Significance and Use

5.1 Biodegradation of a plastic within a composting unit is an important phenomenon because it may affect the decomposition of other materials enclosed by the plastic and the resulting quality and appearance of the composted material. Biodegradation of plastics will also allow the safe disposal of these plastics through large, professionally-managed composting plants and well-run residential units, where thermophilic temperatures are achieved. This procedure has been developed to permit the determination of the rate and degree of aerobic biodegradability of plastic products when placed in a controlled composting process.

5.2 *Limitations*—Because there is a wide variation in the construction and operation of composting facilities and because regulatory requirements for composting systems vary, this procedure is not intended to simulate the environment of any particular composting system. However, it is expected to resemble the environment of a composting process operated under optimum conditions where thermophilic temperatures are achieved. More specifically, the procedure is intended to create a standard laboratory environment that will permit a rapid and reproducible determination of the aerobic biodegradability under controlled composting conditions.

6. Apparatus

6.1 *Composting Apparatus (see Fig. 1):*

6.1.1 A series of at least twelve composting vessels (one test substance, one blank, one positive and one negative control, all in three replicates) of 2 to 5 L of volume. For screening purposes, depending upon the test material, a smaller volume also may be used.

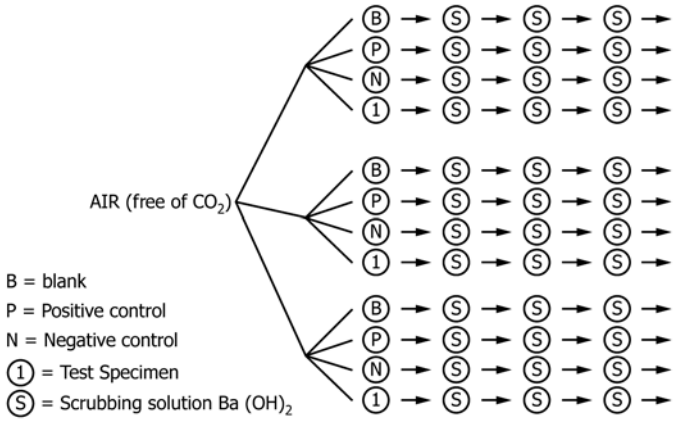

FIG. 1 Set-Up Using Carbon Dioxide-Trapping Apparatus

6.1.2 *Water Baths,* or other temperature controlling means capable of maintaining the temperature of the composting vessels at 58°C (±2°C).

6.1.3 *Pressurized-Air System,* that provides CO_2-free, H_2O-saturated air to each of the composting vessels at accurate aeration rates. If using a direct measurement of CO_2 (see 6.4), then normal air may be used.

6.1.4 Suitable devices for measuring oxygen and CO_2 concentrations in the exhaust air of the composting vessels, such as specific sensors or appropriate gas chromatographs.

6.2 *Carbon Dioxide-Trapping Apparatus for Each Composting Vessel:*

6.2.1 At least three 5000-mL bottles fitted with gas sparging and containing $Ba(OH)_2$ carbon-dioxide scrubbing solution.

6.2.2 *Flexible Tubing,* nonpermeable to carbon dioxide.

6.2.3 *Stoppers,* equipped with gas-sampling parts.

6.3 *Miscellaneous:*

6.3.1 *Analytical Balance,* (±1 mg) to weigh test specimen.

6.3.2 *100-mL Burette.*

6.3.3 *0.05 N HCl.*

6.3.4 *pH Meter.*

6.3.5 Suitable devices and analytical equipment for measuring dry solids (at 105°C), volatile solids (at 550°C), volatile fatty acids by aqueous-injection chromatography, total Kjeldahl nitrogen and carbon concentrations.

6.4 *Optional*—The carbon dioxide-trapping apparatus and titration equipment can be replaced by a gas flow meter plus a gas-chromatograph, or other apparatus equipped with suitable detector and column(s), for measuring CO_2 and O_2 concentrations in the exhaust air of each vessel. Take care to analyze CO_2 concentration on a sufficiently frequent basis in order to produce a reliable cumulative CO_2 production over the course of the test (for example, every 3 to 6 h). A standard gas should be injected to internally standardize the gas-chromatograph on a continuous basis over the course of the test. Operate the gas chromatograph in conformance with Practices E260 and E355 (see Fig. 2).

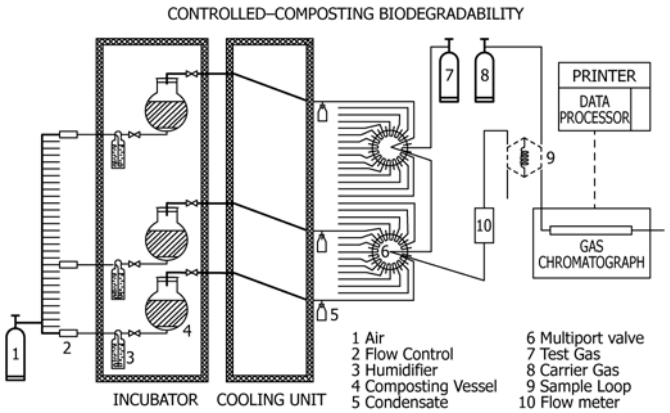

FIG. 2 Optional Set-Up Using a Gas Chromatograph

6.5 Ensure that all glassware is cleaned thoroughly and free from organic matter.

7. Reagents and Materials

7.1 *Barium Hydroxide Solution,* approximately 0.024 N and then standardized, prepared by dissolving 4.0 g $Ba(OH)_2$ per litre of distilled water. Filter through filter paper and store sealed as a clear solution to prevent absorption of CO_2 from the air.

7.2 *Analytical-Grade Cellulose,* for thin-layer chromatography with a particle size of less than 20 µm as positive control.[5]

7.3 *Polyethylene,* as a negative control. It should be in the same form as the form in which the sample is tested (polyethylene film for film samples, polyethylene pellets in case sample is in the form of pellets, etc.).

8. Hazards

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

8.2 The compost inoculum may contain sharp objects. Take care when handling it.

8.3 The composting vessels are not designed to withstand high pressures. The system should be operated at close to ambient pressure.

9. Compost Inoculum

9.1 The compost inoculum should be two to four months old well-aerated compost coming from the organic fraction of municipal solid waste and sieved on a screen of <10 mm. If such a compost is not available, compost from plants, treating green, or yard waste, or mixtures of green waste and municipal solid waste may be used. It is recommended that the compost inoculum produces between 50 and 150 mg of CO_2 per gram of volatile solids over the first ten days of the test, and has an ash content of less than 70 % and a pH between 7 and 8.2. Total dry solids should be between 50 and 55 %.

9.2 The compost inoculum should be as free from larger inert materials (glass, stones, metals, etc.) as possible. These items should be removed manually as much as possible to produce a homogeneous compost inoculum.

9.3 It is recommended to use compost of sufficient porosity to enable conditions to be as aerobic as possible. Addition of structural material, such as small wood particles, or persistent or poorly biodegradable inert material may prevent the compost from sticking together and clogging during the test.

10. Test Specimen

10.1 The test specimen should have sufficient carbon to yield carbon dioxide that can be adequately measured by the trapping apparatus or CO_2 measurements.

10.2 All basic composting parameters, such as C/N, oxygen in the composting vessel, porosity, and moisture content should be optimized so as to make a good composting process possible. The C/N ratio should preferably be between 10 and 40 for both the inoculum and test substance combined. Oxygen levels in the composting vessel should be at least 6 % at all times and no free-standing water nor clumps of material should be present.

10.3 Test specimens may be in the form of films, formed articles, dog bones, granules, powder, or other, and conform to Practice D618.

11. Procedure

11.1 *Preparation of the Samples:*

11.1.1 Obtain an inoculum from a properly operating aerobic composting plant treating municipal solid waste, or the organic fraction thereof. If required, further stabilize the inoculum at the laboratory in order to obtain a low CO_2 production (see 9.1.).

11.1.1.1 Screen the inoculum to less than 10 mm and manually remove and discard any large inert items (pieces of glass, stone, wood, etc.). Determine volatile solids, dry solids and nitrogen content according to Test Methods D3590, D1888, and APHA Test Methods 2540 D and 2540 E.

11.1.2 Determine volatile solids, dry solids and carbon content of all the test substances according to APHA Test Methods 2540 D and 2540 E and Test Method D4129.

11.1.3 Weigh out roughly 600 g of dry solids of inoculum and mix with about 100 g of dry solids coming from the sample. Adjust the dry solids content of the mixture in the vessel to approximately 50 % with distilled water. Add ammonium chloride if the C/N ratio is more than 40. Weigh vessels with all of the contents immediately before initiation of the composting process.

11.1.4 The blank consists of the inoculum only, containing about 600 g of dry solids. As references, use thin-layer chromatography cellulose as a positive control and polyethylene as a negative control.

11.1.5 The test material may be in the form of films, formed articles such as dog bones, granules, or powder. The maximum surface area of a compact test material used should be about 2 by 2 cm. In case the original test material is larger, reduce it in particle size.

[5] For development of this test method, Avicel, available from EM Chemicals, Inc., Hawthorne, New York, was used.

11.1.6 No more than about ¾ of the volume of the test vessel should be filled with test mixture. Sufficient headspace is required in order to provide enough space for manual shaking of the test mixture.

11.2 *Start-Up Procedure*—Initiate aeration of the composting vessels with air-flow rates that are sufficiently high to ensure that oxygen levels do not drop below 6 % in the exhaust air. Oxygen levels should be closely controlled during the first week and measured at least twice daily. Adjust air-flow rates as needed.

11.3 *Operating Procedure:*

11.3.1 The composting vessels are incubated in the dark or in diffuse light for a period of 45 days in an enclosure that is free from vapors toxic to microorganisms. The temperature is maintained at 58°C (±2°C). In special cases, for example, when the melting point of the test material is low, another temperature may be chosen. This temperature should be constant during the test and kept in a range of ±2°C. The change of temperature should be justified and clearly indicated in the test report.

11.3.2 Check CO_2 and O_2 concentrations in the outgoing air at least daily with a minimum time interval of 6 h after the first week for the remainder of the test.

11.3.3 Check air flow daily before the composting vessels and at the outlets, ensuring that no leaks are present in the complete system. Adjust air flow to maintain a CO_2 concentration of at least 2 % volume over volume to allow accurate determination of CO_2 level in the exhaust air.

11.3.4 Ensure proper composting conditions. Shake the composting vessels weekly to prevent extensive channelling, provide uniform attack on the test specimen and provide an even distribution of moisture. In case excessive moisture levels are observed, such as free-standing water in the vessels or clumping due to high moisture content, remove excess liquid by injecting dry air, or by drainage via air inlet. If excessively dry conditions are observed, that will severely slow down the breakdown process, add moisture. During the whole course of the test, make adjustments to ensure proper composting conditions. If adjustments are made, then CO_2 and O_2 concentrations must be monitored closely during the following 72 h and measured at least twice daily with a time interval of more than 6 h.

11.3.5 At the weekly shaking and at the end of the test, record visual observations with regard to compost structure, moisture content and color, fungal development, smell of the exhaust air, and sample disintegration.

11.3.6 The incubation time of 45 days may be extended if significant biodegradation of the test substance is still being observed.

11.4 *End of the Test:*

11.4.1 At the end of the test, weigh the vessels with the contents and determine the dry solids concentration remaining in the composted material. Volatile solids may be determined if weight loss is to be calculated.

11.4.2 Measure the pH in conformance with Test Methods D1293. If the pH is less than 7, measure the volatile fatty acids spectrum to indicate souring of the contents in the composting vessel in accordance with Practice D2908. Measure the pH by diluting the sample on a 5:1 w/w ratio of distilled water to compost inoculum or residue, mix by shaking manually and measure immediately.

11.4.3 If more than 2 g of volatile fatty acids per kilogram of dry matter in the composting vessel is formed, the test must be regarded as invalid.

12. Calculation

12.1 Determine the total carbon content of the test material by elemental analysis or by calculation if the chemical composition is well established. This allows the theoretical quantity of carbon dioxide evolution to be calculated as follows:

$$\text{material} = w\ \%\ \text{carbon}$$
$$w/100 \times \text{g of material charged} = Y\ \text{g carbon charged to compost vessel} = C_i$$
$$C + O_2 \rightarrow CO_2$$
$$12\ \text{g}\ C\ \text{yields}\ 44\ \text{g}\ CO_2$$
$$Y\ \text{g}\ C\ \text{yields}\ \frac{44 \times Y}{12}\ \text{g}\ CO_2$$

12.2 Determine the cumulative CO_2 production (in grams) from the test substances.

12.2.1 Determine the amount of CO_2 produced by the difference, in millilitres of titrant, between the test substance and blank $Ba(OH)_2$ traps. Perform the titration with 0.05 N HCl.

12.2.1.1 When CO_2 enters the absorber bottles, it reacts in the following manner:

$$Ba(OH)_2 + CO_2 \rightarrow BaCO_3 + H_2O$$

12.2.1.2 The $BaCO_3$ formed is insoluble and precipitates. Determine the amount of $Ba(OH)_2$ remaining in solution by end-point titration with HCl using phenolpthalein as an indicator according to the following equation:

$$Ba(OH)_2 + 2\,HCl \rightarrow BaCl_2 + 2\,H_2O$$

12.2.1.3 From the above two equations, it can be seen that the number of mmol of CO_2 produced is:

$$\text{mmoles of}\ CO_2 = \text{mmoles of}\ Ba(OH)_2\ \text{at start} - \frac{\text{mmoles HCl}}{2}$$

12.2.2 For the option with gas chromatography, the cumulative CO_2 production (in grams) is determined from the measurements of flow rate and gas composition and after recalculation to STP (standard temperature and pressure) conditions.

12.2.3 Calculate the amount of cumulative gaseous-carbon produced by each reactor.

12.2.4 Determine the mean (of the three replicates) net gaseous-carbon production by controlled composting 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 compound by the original average amount of carbon in the test compound and multiplying by 100: