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ASTM STANDARDS PERTAINING TO THE BIODEGRADABILITY AND COMPOSTABILITY OF PLASTICS

Sponsored by Subcommittee D20.96 on Environmentally Degradable Plastics

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FOREWORD

This book is a compilation of the key documents that were developed by Subcommittee D20.96 on Environmentally Degradable Plastics for the testing and identification of plastics that will biodegrade and compost satisfactorily. These standards are based on the work initiated by International Standards Research at the request of ASTM as well as leading scientists in the U.S. and other parts of the world. The key standard is Specification D 6400-99. The other standards are referenced in Specification D 6400-99 and are necessary to meet the criteria listed in Specification D 6400-99.

This compilation contains the following standards:

D 6400-99, Specification for Compostable Plastics, which (*1*) identifies plastics and products made from plastics designed to be composted in municipal and industrial aerobic composting facilities, and (*2*) determine if plastics and products made from plastics will compost satisfactorily, including biodegrading at a rate comparable to known compostable materials.

D 6002-96, Guide for Assessing the Compostability of Environmentally Degradable Plastics, which outlines the recommended criteria, procedures, and a general approach to establish the compostability of plastics.

D 5338-98, Test Method for Determining Aerobic Biodegradation of Plastic Materials Under Controlled Composting Conditions, which determines the degree and rate of aerobic biodegradation of plastic materials on exposure to a controlled-composting environment under laboratory conditions.

D 6340-98, Test Methods for Determining Aerobic Biodegradation of Radiolabeled Plastic Materials in an Aqueous or Compost Environment, which are applicable for determining the degree and rate of aerobic biodegradation of plastics whose biodegradation rate is slow and requires test periods for as long as 365 days. These test methods require radiolabeling the selected carbon of the plastic polymer materials and montitoring the conversion of the radiolabeled carbon of the polymer to CO_2.

Suggestions and comments regarding this compilation are welcome and should be addressed to Product Manager, Publications, ASTM, 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959.

ASTM

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Designation: D 6400 – 99

AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428

Standard Specification for Compostable Plastics[1]

This standard is issued under the fixed designation D 6400; 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 specification covers plastics and products made from plastics that are designed to be composted in municipal and industrial aerobic composting facilities.

1.2 This specification is intended to establish the requirements for labeling of materials and products, including packaging made from plastics, as "compostable in municipal and industrial composting facilities."

1.3 The properties in this specification are those required to determine if plastics and products made from plastics will compost satisfactorily, including biodegrading at a rate comparable to known compostable materials. Further, the properties in the specification are required to assure that the degradation of these materials will not diminish the value or utility of the compost resulting from the composting process.

1.4 The following safety hazards caveat pertains to the test methods portion of this standard: *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 health and safety practices and to determine the applicability of regulatory limitations prior to use.*

NOTE 1—No equivalent ISO specifications exist for this standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 883 Terminology Relating to Plastics[2]

D 5338 Test Method for Determining Aerobic Biodegradation of Plastic Materials Under Controlled Composting Conditions[3]

D 6002 Guide for Assessing the Compostability of Environmentally Degradable Plastics[3]

2.2 *Organization for Economic Development (OECD) Standard:*[4]

OECD Guideline 208 Terrestrial Plants, Growth Test

2.3 *Association Francaise de Normalisation (CEN) Standard:*[5]

CEN/TC 261/SC 4 N 99 Packaging—Requirements for Packaging Recoverable through Composting and Biodegradation—Test Scheme and Evaluation Criteria for the Final Acceptance of Packaging (WI 261 236)

2.4 *ISO Standard:*[5]

ISO 14855 Evaluation of the Ultimate Aerobic Biodegradability and Disintegration of Plastics under Controlled Composting Conditions—Method by Analysis of Evolved Carbon Dioxide

2.5 *Government Standard:*[6]

40 CFR Part 503.13 Standards for the Use or Disposal of Sewage Sludge

3. Terminology

3.1 *Definitions*—Definitions appearing in this specification are found in Terminology D 883, unless otherwise noted.

3.1.1 *biodegradable plastic*—a degradable plastic in which the degradation results from the action of naturally occurring microorganisms such as bacteria, fungi, and algae.

3.1.2 *compostable plastic*—a plastic that undergoes degradation by biological processes during composting to yield CO^2, water, inorganic compounds, and biomass at a rate consistent with other known compostable materials and leave no visible, distinguishable or toxic residue.

3.1.3 *composting*[7]—a managed process that controls the biological decomposition and transformation of biodegradable materials into a humus-like substance called compost: the aerobic mesophilic and thermophilic degradation of organic matter to make compost; the transformation of biologically decomposable material through a controlled process of biooxidation that proceed through mesophilic and thermophilic phases and results in the production of carbon dioxide, water, minerals, and stabilized organic matter (compost or humus).

3.1.3.1 *Discussion*—Composting uses a natural process to stabilize mixed decomposable organic material recovered from municipal solid waste, yard trimmings, biosolids (digested sewage sludge), certain industrial residues and commercial residues.

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[1] This specification 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 08.03.

[4] Available from Organization for Economic Development, Director of Information, 2 rue Andre' Pascal, 75775 Paris Cedex 16, France.

[5] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[6] *Code of Federal Regulations*, available from U.S. Government Printing Office, Washington, DC 20402.

[7] *Compost Facility Operating Guide*, Composting Council, Alexandria, VA, 1995.

3.1.4 *degradable plastic*—a plastic designed to undergo a significant change in its chemical structure under specific environmental conditions, resulting in a loss of some properties that may be measured by standard test methods appropriate to the plastic and the application in a period of time that determines its classification.

3.1.5 *plastic*—a material that contains as an essential ingredient one or more organic polymeric substances of large molecular weight, is solid in its finished state, and, at some stage in its manufacture or processing into finished articles, can be shaped by flow.

3.1.6 *polymer*—a substance consisting of molecules characterized by the repetition (neglecting ends, branch junctions, other minor irregularities) of one or more types of monomeric units.

4. Classification

4.1 The purpose of this specification is to establish standards for identifying products and materials that will compost satisfactorily in commercial and municipal composting facilities. Products meeting the requirements outlined below are appropriate for labeling as "compostable" in accordance with the guidelines issued by the Federal Trade Commission.[8]

5. Basic Requirements

5.1 In order to compost satisfactorily, a product or material must demonstrate each of the characteristics found in 5.1.1-5.1.3, and which are quantified in Section 6.

5.1.1 *Disintegration During Composting*—A plastic product or material will disintegrate during composting such that any remaining plastic residuals are not readily distinguishable from the other organic materials in the finished product. Additionally, the material or product must not be found in significant quantities during screening prior to final distribution of the compost.

5.1.2 *Inherent Biodegradation*—A level of inherent biodegradation shall be established by tests under controlled conditions, that are comparable to known compostable materials.

5.1.3 *No Adverse Impacts on Ability of Compost to Support Plant Growth*—The tested materials shall not adversely impact on the ability of composts to support plant growth, when compared to composts using cellulose as a control, once the finished compost is placed in soil. Additionally, the polymeric products or materials must not introduce unacceptable levels of heavy metals or other toxic substances into the environment, upon sample decomposition.

NOTE 2—For a better understanding of why these criteria are important, the reader should consult Guide D 6002 *Compost Facility Operating Guide*,[7] and CEN/TC 261/SC 4 N 99.

6. Detailed Requirements

6.1 In order to be identified as compostable, products must pass the requirements of 6.2, 6.3, and 6.4 using the appropriate laboratory tests, representative of the conditions found in aerobic composting facilities. Products and finished articles should be tested in the same form as they are intended to be used. For products that are made in multiple thicknesses or densities, such as films, containers and foams, only the thickest or most dense products need to be tested as long as the chemical composition and structure remains otherwise the same. It is assumed that thinner gages and lower densities will also compost satisfactorily. Similarly, if additives are present in test samples that pass testing, lower levels of the same additives are similarly passed.

6.2 A plastic product is considered to have demonstrated satisfactory disintegration if after controlled laboratory-scale composting, found in 7.2.1 of Guide D 6002, no more than 10 % of its original dry weight remains after sieving on a 2.0-mm sieve.

6.3 A plastic product must demonstrate a satisfactory rate of biodegradation by achieving one of the following ratios of conversion to carbon dioxide found in 6.3.1, 6.3.2 or 6.3.3, within the time periods specified in 6.3.3.1 or 6.3.3.2, using Test Method D 5338 as outlined in 7.3.1 and 7.3.3 of Guide D 6002:

6.3.1 For products consisting of a single polymer (homopolymers or random copolymers), 60 % of the organic carbon must be converted to carbon dioxide by the end of the test period (see 6.3.4), when compared to a known reference material as outlined in 7.3.2 of Guide D 6002.

6.3.2 For products consisting of more than one polymer (block copolymers, segmented copolymers, blends, or addition of low molecular weight additives), 90 % of the organic carbon must be converted to carbon dioxide by the end of the test period (see 6.3.4), when compared to a known reference material as outlined in 7.3.2 of Guide D 6002.

6.3.3 For products consisting of more than one polymer, each individual polymer component, present at more than 1 % concentration, must achieve the 60 % specification for homopolymers, as described in 6.3.1.

6.3.3.1 For materials that are not radiolabeled, the test period shall be no greater than 180 days.

6.3.3.2 If radiolabeled materials are used, then the test period may be as long as 365 days.

NOTE 3—While the end points of biodegradation may include incorporation into biomass or humic substances as well as carbon dioxide, no recognized standard test methods and specifications exist to quantify these outcomes. When these tests and specifications become available, this standard will be revised.

6.4 A plastic product can demonstrate satisfactory terrestrial and aquatic safety if it fulfills the following requirements:

6.4.1 The plastic or product shall have concentrations of heavy metals less than 50 % of those prescribed in 40 CFR Part 503.13, and

6.4.2 The plastic or product shall fulfill the requirements of the tests found in 7.5.2.2 and 7.5.2.3 of Guide D 6002, including the cress seed test for plant germination and a plant growth test following OECD Guideline 208.

7. Sampling

7.1 Sampling shall be conducted as indicated in the specified test method.

[8] *Guidelines for the Use of Environmental Marketing Claims*, Federal Trade Commission, Washington, DC, 1992.

8. Specimen Preparation

8.1 Specimen preparation shall be in accordance with the specified test method.

9. Marking and Labeling

9.1 Marking and labeling shall conform to national and local regulations.

10. Keywords

10.1 biodegradable; compostable plastic; composting; degradable plastics; labeling

Designation: D 6002 - 96

Standard Guide for Assessing the Compostability of Environmentally Degradable Plastics[1]

This standard is issued under the fixed designation D 6002; 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 guide covers suggested criteria, procedures, and a general approach to establish the compostability of environmentally degradable plastics.

1.2 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 638 Test Method for Tensile Properties of Plastics[2]

D 882 Test Methods for Tensile Properties of Thin Plastic Sheeting[2]

D 883 Terminology Relating to Plastics[2]

D 3593 Test Method for Molecular Weight Averages/ Distribution of Certain Polymers by Liquid Size-Exclusion Chromatography (Gel Permeation Chromatography (GPC)) Using Universal Calibration[3]

D 5152 Practice for Water Extraction of Residual Solids from Degraded Plastics for Toxicity Testing[4]

D 5209 Test Method for Determining the Aerobic Biodegradation of Plastic Materials in the Presence of Municipal Sewer Sludge[4]

D 5247 Test Method for Determining the Aerobic Biodegradability of Degradable Plastics by Specific Microorganisms[4]

D 5338 Test Method for Determining Aerobic Biodegradation of Plastic Materials Under Controlled Composting Conditions[4]

D 5509 Practice for Exposing Plastics to a Simulated Compost Environment[4]

D 5512 Practice for Exposing Plastics to a Simulated Compost Environment Using an Externally Heated Reactor[4]

D 5951 Practice for Preparing Residual Solids Obtained After Biodegradability Standard Methods for Plastics in Solid Waste for Toxicity and Compost Quality Testing[4]

D 5988 Test Method for Determining the Aerobic Biodegradation in Soil of Plastic Materials or Residual Plastic Materials after Composting[4]

E 1440 Guide for an Acute Toxicity Test with the Rotifer Brachionus[5]

E 1720 Test Method for Determining Ready, Ultimate, Biodegradability of Organic Chemicals in a Sealed Vessel CO_2 Production Test[5]

G 22 Practice for Determining Resistance of Plastics to Bacteria[6]

2.2 *ORCA Document:*

Guidelines for the Evaluation of Feedstock for Source Separated Biowaste Composting and Biogasification[7]

2.3 *OECD Guidelines:*[8]

OECD Guideline 207 Earthworm, Acute Toxicity Tests

OECD Guideline 208 Terrestrial Plants, Growth Test

3. Terminology

3.1 *Definitions:*

3.1.1 *biodegradable plastic*—a degradable plastic in which the degradation results from the action of naturally occurring microorganisms such as bacteria, fungi, and algae. **D 883**

3.1.2 *compostable*—capable of undergoing biological decomposition in a compost site as part of an available program, such that the material is not visually distinguishable and breaks down into carbon dioxide, water, inorganic compounds, and biomass, at a rate consistent with known compostable materials.

3.1.3 *composting*—a managed process that controls the biological decomposition and transformation of biodegradable material into a humus-like substance called compost; the aerobic mesophilic and thermophilic degradation of organic matter to make compost; the transformation of biologically decomposable material through a controlled process of bio-oxidation that proceeds through mesophilic and thermophilic phases and results in the production of carbon dioxide, water, minerals, and stabilized organic matter (compost or humus). Composting uses a natural process to stabilize mixed decomposable organic material recovered from municipal solid waste, yard trimmings, biosolids (digested sewage sludge), certain industrial residues, and commercial residues (**1**).[9]

3.1.4 *degradable plastic*—a plastic designed to undergo a significant change in its chemical structure under specific

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

[3] *Discontinued*—See 1992 *Annual Book of ASTM Standards*, Vol 08.03.

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

[5] *Annual Book of ASTM Standards*, Vol 11.05.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

[7] Available from Organic Reclamation and Composting Association, Avenue E. Mounier 83, Box 1, B-1200 Brussels, Belgium.

[8] Available from Organization for Economic Development, Director of Information, 2 rue Andre' Pascal, 75775 Paris Cedex 16, France.

[9] The boldface numbers in parentheses refer to the list of references at the end of this guide.

environmental conditions, resulting in a loss of some properties that may be measured by standard methods appropriate to the plastic and the application in a period of time that determines its classification. D 883

3.1.5 *mesophilic phase*—the phase of composting that occurs between 20 and 45°C (68 and 113°F) (1).

3.1.6 *plastic*—a material that contains as an essential ingredient one or more organic polymeric substances of large molecular weight, is solid in its finished state, and, at some stage in its manufacture or processing into finished articles, can be shaped by flow. D 883

3.1.7 *polymer*—a substance consisting of molecules characterized by the repetition (neglecting ends, branch junctions, and other minor irregularities) of one or more types of monomeric units. D 883

3.1.8 *thermophilic phase*—the phase in the composting process that occurs between 45 and 75°C (113 and 167°F); it is associated with specific colonies of microorganisms that accomplish a high rate of decomposition (1).

4. Summary of Guide

4.1 This guide uses a tiered criteria-based approach to assess the compostability of environmentally degradable plastic products (processed material containing polymeric materials, processing additives, and other additives required to meet performance requirements).

4.1.1 This guide includes methods that simulate mesophilic and thermophilic conditions that are representative of composting processes and compost end use.

4.1.2 The tiers progress from rapid screening of polymeric materials and other organic components to relatively long-term, more complex/higher cost evaluations. This guide will allow one to focus the correct level of resources on materials of greatest interest and potential.

4.1.3 Each tier in this guide includes objectives and a summary that presents potential test methods, method principles, test duration, implication of results, and suggested priority.

NOTE 1—The availability of other test methods appropriate for this guide is acknowledged.

NOTE 2—See Fig. 1 for a description of this guide in flow-chart form.

5. Significance and Use

5.1 Plastics that are designed to degrade after use have been developed. These materials are intended to enhance existing solid waste landfill diversion programs by allowing difficult to recycle materials to be collected and processed in alternative solid waste disposal systems. Composting has emerged as a viable approach to process these materials and the organic fraction of municipal solid waste (MSW). A comprehensive testing program is needed to establish the compostability (for example, fragmentation rate, biodegradation rate, and safety) of these materials.

5.2 This guide can be adapted to generate product-specific evidence for the substantiation of compostable claims to obtain classification as a compostable product.

NOTE 3—State and local regulations should also be considered.

6. Tier 1: Rapid Screening Tests

6.1 In this tier, rapid screening level studies are performed, under mesophilic conditions, to obtain information unavailable from literature review. The objectives are as follows:

6.1.1 To determine whether biodegradation of polymeric materials and other organic components in the plastic product can occur. Biodegradation is based on carbon dioxide production.

6.1.2 To expand understanding of the degradation mechanism.

NOTE 4—A positive result in Tier 1 tests is not required to demonstrate the compostability of product components. Components which fail Tier 1 tests might prove successful in Tier 2 composting tests. If a component fails Tier 1, but is still considered promising, it should advance to Tier 2. Likewise, a promising component could enter the test strategy directly at Tier 2.

NOTE 5—Chemical analysis, (for example, regulated heavy metals) of product component may be appropriate prior to initiation of testing.

6.2 The following test methods are suggested for initial screening of polymeric materials, monomeric subunits of the polymer, and other organic components.

6.2.1 *Test Method D 5209 (Sturm Test)*—This aqueous test method uses a fresh sample of activated sewage sludge that has been aerated, homogenized, and settled. The supernatant is used as the inoculum. It contains primarily a mixed bacterial population that promotes rapid biodegradation under mesophilic conditions. The metabolism of test materials produces CO_2, which is trapped in alkali solution and quantitated by titration. The test length is typically 30 days, but it can be extended if the medium is reinoculated. A positive result (recovery of 60 % + of theoretical CO_2) usually indicates that the material will also biodegrade in a composting environment. A negative result should be confirmed by a laboratory thermophilic composting test such as Test Method D 5338. The contribution of nonmicrobial degradation can be quantified by including sterile or poison controls and comparing changes in molecular weight or mass.

6.2.2 *Soil Contact Test (Test Method D 5988)*—This static test uses a defined sand/soil/mature compost matrix to provide a consortium of mesophilic and thermophilic bacteria and fungi. Biodegradation is measured in a manner similar to the Sturm test, based on the amount of material carbon converted to gaseous carbon (CO_2). Readily biodegradable materials can be screened in 30 to 60 days. A negative result should be confirmed under thermophilic composting conditions (Test Method D 5338).

6.3 The following test methods can be used to obtain additional information regarding the inherent biodegradability or degradability of materials.

6.3.1 *Test Method D 5247 (Specific Microbe Test)*—This aqueous test method uses pure microbial cultures to assess the biodegradability of materials under mesophilic conditions, based on weight loss or molecular weight changes. The test duration is 7 to 14 days. Microbes indigenous to the composting or soil environment can be evaluated with this test method.

6.3.2 *Practice G 22 (Bacteria Growth Resistance)*—With this test, solid materials are placed in inoculated molten agar, and the extent of microbial growth is rated. The test duration is approximately 14 days. A positive result indicates that the test material is potentially biodegradable.

6.3.3 *Clear Zone Assays*—Opaque test material is dispersed into solid agar. A given quantity of microorganisms is applied to form a lawn. Degradation of a material is indicated by the formation of clear zones in the solid medium. The test duration is 3 to 14 days. A positive result indicates that the test material is potentially biodegradable. Microbes indigenous to the composting or soil environment can be evaluated with this test method. The biodegradability of nonopaque organic materials can be assessed by adding the indicator 2,3,5-triphenyl-tetrazolium chloride (TTC) to the media. If microbial colonies can oxidize the material, their electron transport pathways will reduce the TTC. Reduced TTC is detected by its deep red color, whereas oxidized TTC is colorless (2).

6.3.4 *Sealed Vessel Test (Test Method E 1720)*—Ready aerobic biodegradability of organic materials is assessed in small, sealed vessels inoculated with sewage microbes. Gaseous CO_2 is monitored by head space analysis. This test method represents a simpler approach relative to Test Method D 5209 (Sturm). A positive result (60 % +) usually indicates that the material will also biodegrade in a composting environment.

6.4 If it appears that a material is being colonized or used as a growth substrate by microorganisms, a more fundamental understanding of the degradation process can be obtained. This typically involves the preparation of purified microbial cultures capable of using the material as a carbon source. The pure cultures can then be used for the isolation and characterization of cellular enzyme systems contributing to degradation of the material (3).

6.5 The potential effect of materials on plant germination may be assessed with the cress seed test. This step may be especially valuable for screening processing additives used at 1 % or less in the plastic. Soils from the above soil contact test (6.2.2) may be evaluated at the beginning and end of the test to establish the potential effect of microbial degradation products. In the cress test, soil or compost is extracted with water and filtered. The supernatant is used for the germination test. Various dilutions of the supernatant are prepared, and aliquots are added to petri dishes lined with filter paper. Cress seeds are placed on the wet paper and left to germinate in the dark over 4 days at room temperature. The percentage of germinated seeds is determined after 4 days and compared to a water control. Soils containing test materials should not be significantly different from the blank soil at 95 % confidence interval.

7. Tier 2: Laboratory and Pilot Scale Composting Assessment

7.1 The objectives of this tier are as follows:

7.1.1 To establish the degradation rate (change in chemical structure, decrease in mechanical strength, fragmentation, or weight loss) of the polymeric material or plastic product under laboratory-scale thermophilic composting conditions.

7.1.2 To confirm the biodegradability of the plastic product and other organic components in the product under laboratory scale thermophilic composting conditions.

7.1.3 To determine whether organic residues continue to biodegrade in a laboratory-scale simulation of compost-amended soil.

7.1.4 To obtain additional evidence with regard to a plastic product's or component's environmental safety using compost obtained from laboratory-scale studies.

7.1.5 To establish the degradation rate of a plastic product or finished article under pilot scale composting conditions prior to the full-scale composting studies described in Tier 3.

7.2 The following test methods are suggested for establishing the degradation rate of polymeric materials or plastic products under laboratory-scale composting conditions.

7.2.1 The degradation rate of test materials under laboratory thermophilic composting conditions may be obtained by performing Test Method D 5338 without the CO_2 trapping component. The test materials are exposed to an inoculum that is derived from stabilized compost from municipal solid waste.

7.2.1.1 Aerobic composting occurs in an environment in which temperature, aeration, and humidity are monitored and controlled closely. The degradation rate of materials may be established with the current Test Method D 5338 temperature profile or constant 58°C, which has been adopted by the European standards organization, CEN. The test duration is 45 days, but it may be extended to simulate field conditions. At various time intervals, materials may be removed from the compost, cleaned, and dried.

7.2.1.2 Changes in material chemical structure may be quantitated based on molecular weight distribution (Test Method D 3593). More sophisticated techniques such as Fourier transform infrared (FTIR) and nuclear magnetic resonance may also be appropriate (4).

7.2.1.3 Loss of material integrity due to material degradation may be quantitated by using Test Methods D 882 for thin films or Test Method D 638 for sheet. Material degradation may also be established based on weight loss. Surface damage may be evaluated using tools such as Scanning Electron Microscopy (SEM).

7.2.1.4 Degradation rates of materials may also be established using simulated MSW matrixes in externally heated and self-heating controlled laboratory-scale composting environments in accordance with Practices D 5509 and D 5512.

7.2.1.5 Sieve analysis can be included in the above tests to obtain additional fragmentation information. Compost containing fragmented material may be passed through a U.S. Standard Sieve[10] with a 3/8-in. (9.51-mm) opening. This simulates the final screening step used to produce high-quality compost products. National, state, and local regulatory requirements should also be consulted.

NOTE 6—Agitation from compost turning equipment at full-scale facilities may give faster fragmentation rates relative to laboratory-scale methods.

7.3 The following test methods are suggested for establishing the biodegradation rate of a plastic product, polymeric materials in the product, and other organic components in a composting environment.

7.3.1 Test Method D 5338 is suggested for establishing the biodegradability of organic components in a plastic product in a composting environment. Material biodegradability is based on the amount of material carbon recovered as gaseous carbon (CO_2) relative to the amount of material carbon

[10] Available from W. S. Tyler Co., Cleveland, OH.

originally added to the compost. Product organic components, at levels of 1 % or less, generally do not require retesting in this step if a positive result was obtained in Tier 1 (6.2). This test can be performed separately or concurrently with (7.2). Biodegradation rates or end points should meet national, state, or local regulations or be compared to the reference materials described in 7.3.2.

7.3.1.1 If a negative result is obtained, check the controls described in the test method or repeat the test method with a lower dose closer to field-use levels (assuming that an acceptable signal:noise ratio is possible).

7.3.2 Products or components may be compared under identical conditions to natural reference materials known to be biodegradable in a composting environment (for example, cellulose or starch (see Guidelines for the Evaluation of Feedstock for Source Separated Biowaste Composting and Biogasification)). Other materials regarded as biodegradable in a composting environment are oak, maple, and corn leaves and kraft paper **(5)**. Unmodified polyethylene film, typically used to collect yard trimmings, is generally considered a negative reference material.

7.3.3 The recovery of all material carbon as gaseous carbon (CO_2) may be impractical due to the incorporation of material carbon into microbial biomass or stable humic substances. ^{14}Carbon labelled materials may allow carbon to be partitioned into CO_2-C, residue-C, water soluble-C, and microbial biomass-C to obtain a complete mass balance. The use of radio-labelled materials allows testing at field-use levels in composts with high background CO_2. However, these definitive studies are comparatively expensive.

NOTE 7—An ASTM standard method for ^{14}C-labelled materials is not available.

7.3.4 The effect of a material on compost microorganisms may be evaluated as described by Schwab, et al **(6)**.

7.4 The following test methods are suggested for establishing the rate at which plastic product organic components continue to biodegrade in compost conditioned soil.

7.4.1 If incomplete biodegradation is indicated in 7.3, the biodegradability of product or component residue in soil may be established with the soil contact method cited in 6.2.2. The test duration should be a minimum of 6 months or until a regulatory specification is attained or results support the calculation of a rate as indicated by the lack of a plateau.

7.4.2 Materials from 7.3.2 can also be evaluated in soil to obtain additional comparative data.

7.4.3 Composts should be prepared in accordance with the Bridging Practice of Practice D 5951 prior to the soil studies.

7.5 The plastic product should not cause any negative ecotoxilogical effects on the resulting compost. The following terrestrial and aquatic ecotoxicity tests are suggested for obtaining evidence regarding product effects on plant and animal life. National, state, and local regulatory requirements should be considered.

NOTE 8—The test material dose specified in laboratory methods, such as Test Method D 5338, is much higher than levels expected to be released into the environment. Ecotoxicity is concentration dependent. If a negative effect is observed, additional testing is suggested based on predicted exposure levels.

7.5.1 Compost from 7.3 should be prepared in accordance with Practice D 5152 or D 5951 prior to performing ecotoxicity tests.

7.5.2 The following ecotoxicity tests are suggested as a minimum prior to proceeding to pilot and full-scale testing:

7.5.2.1 Aquatic toxicity test with rotifer Brachionus in accordance with Guide E 1440. The test duration is one day.

7.5.2.2 Plant germination as described by the cress seed test in 6.5. The test duration is four days.

7.5.2.3 Plant growth test as described by OECD Guideline 208. This procedure determines phytotoxicity by mixing the compost containing the material with soil. The plant emergence survival and growth is evaluated. Three plant species are generally tested. The test duration is approximately 1 month. The results from compost containing material are compared to compost without material and a soil control.

7.5.2.4 Earthworm test in accordance with OECD Guideline 207. This procedure determines possible toxicity by mixing the compost containing the material with a specified soil. The earthworm weight change and survival are measured. The results from compost containing material are compared to compost without material and soil controls.

7.6 Pilot-scale investigations are intended to confirm the results from laboratory-scale composting tests. These tests may be used to evaluate the practical processibility, at anticipated field use levels, of a plastic product or full-sized article by simulating larger-scale operating conditions (see Guidelines for the Evaluation of Feedstock for Source Separated Biowaste Composting and Biogasification). Pilot-scale tests may also be used to establish the impact of different waste matrixes on the degradation of a material **(6)**.

7.6.1 A standard ASTM pilot-scale test method has not been developed. Pilot-scale systems ranging from relatively simple to complex have been constructed by industry **(6)** and commercial testing laboratories. Some systems include rotating drums (manual or mechanical) to simulate full-scale feedstock homogenization and composting process initiation. Some systems control feedstock aeration and temperature. Vessel sizes range from 30 to 200 L. All systems are self-heating. The duration of the thermophilic composting phase ranges from a few days to a few weeks.

7.6.2 Externally heated pilot-scale systems may be required to simulate thermophilic conditions characteristic of full-scale processes.

7.6.3 Product degradation, safety, and microflora changes may be measured with the techniques described in 7.2, 7.3.4, and 7.5.

7.7 In addition to ecotoxicity, a product may not have a negative effect on the quality of the compost based on standard chemical and physical tests. National, state, and local regulation should be consulted.

7.7.1 The quality of pilot-scale composts containing degraded plastic should be compared to pilot-scale plastic-free composts based on chemical analysis. Suggested analyses include Environmental Protection Agency (EPA) 503 heavy metals, pH, compost maturity, density, porosity, and conductivity as described in Refs **(1, 7)**.

8. Tier 3: Field/Full-Scale Assessment

8.1 In this tier, the compostability of products in the field is established based on full-scale composting studies and backyard composting environments. The backyard studies

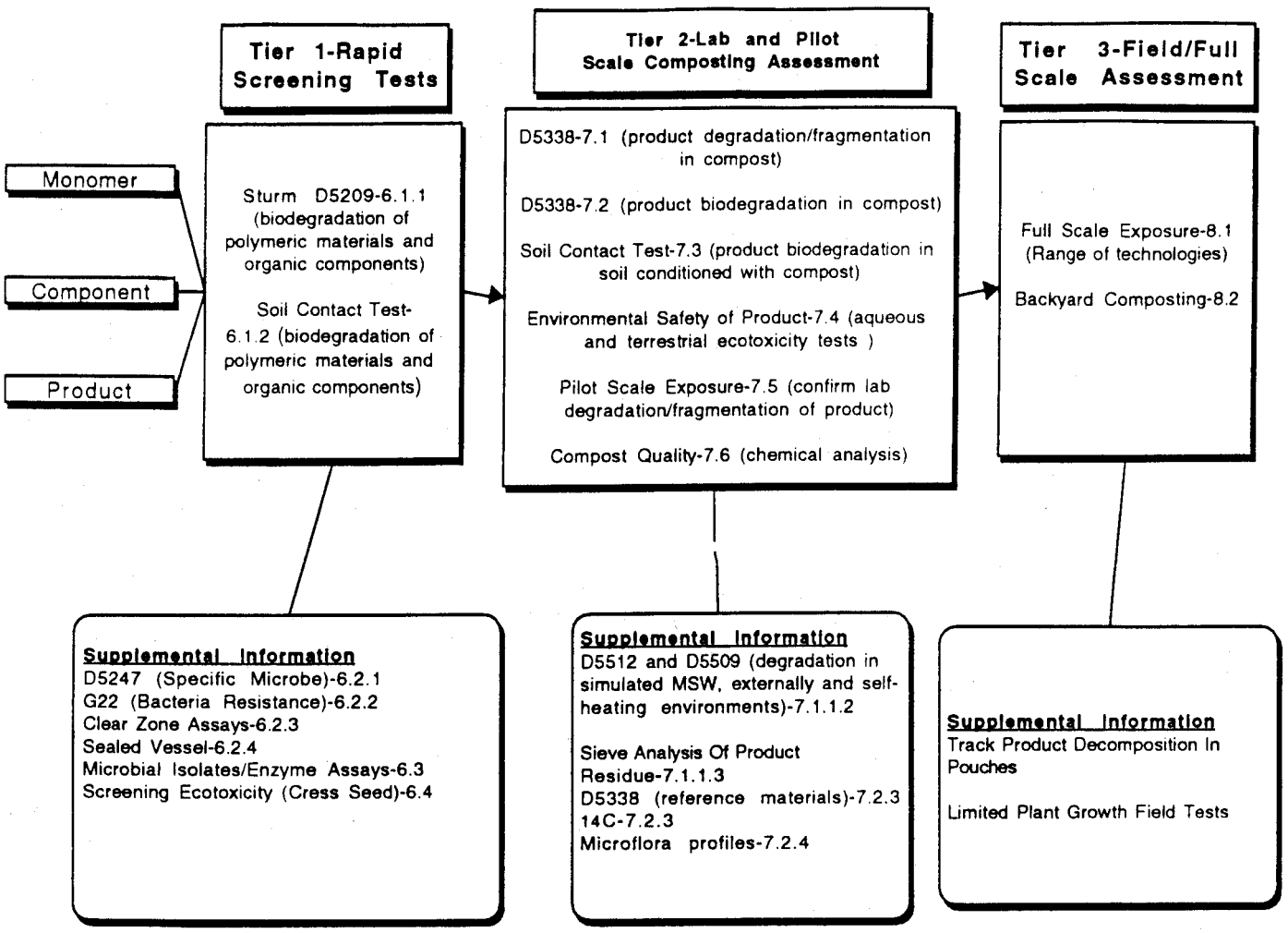

FIG. 1 Flow-Chart of Guide D 6002

have been included in response to current Federal Trade Commission (FTC) marketing guidelines **(8)**.

8.2 The field assessment of products in full-scale systems should include a range of technologies. Technologies range from unmanaged piles (municipal yard waste) to turned aerated static piles with temperature control to tunnel/agitated bay systems with temperature control. Consult Ref **(9)** to obtain descriptions of facility technologies in the United States. The need for full-scale assessment may be reduced as composters, solid waste managers, and degradable plastic product suppliers gain experience with their products.

8.2.1 Ideally, product should be added to the feedstock at anticipated exposure levels and be exposed to the entire process to establish the compatibility with turning equipment and to ensure that the product is not screened off early in the process. Other goals are to ensure that the product does not have an adverse effect on the process (that is, biological activities, litter, odor, pH, etc.) and that the product is not visually distinguishable after curing and final processing is completed.

8.2.2 A useful technique for quantitating the degradation rate in full-scale systems that do not grind feedstock is the placement of fiberglass pouches containing the product in the feedstock. The pouches may be removed periodically to measure the fragmentation rate and quantify product degradation as described in 7.2.

8.2.2.1 A full-scale procedure that includes use of the pouches has been developed by the ASTM Institute for Standard Research Degradable Polymer Advisory Committee. The procedure may be submitted to ASTM for standardization.

8.2.3 Limited plant growth studies are also suggested using compost containing degradable products. The intent of these studies is to confirm previous laboratory/pilot-scale results.

8.3 According to the FTC marketing guidelines **(8)**, an unqualified compostable claim is considered deceptive if the product is not compostable in a "home" or "backyard" environment.

8.3.1 The compostability of products in backyard composting environments can be established if desired. The composting process tends to be slower due to a relatively short thermophilic composting phase. Loss of heat due to the relatively small pile or bin size is a significant factor. The approach described in 7.6 and 7.7 will probably provide sufficient evidence.

8.3.2 The compostability of products should be established in both bins and freestanding piles based on typical home composting practices.

NOTE 9—Guidelines for best management practices under backyard composting environments can be obtained from the Composting Council (1).

9. Report

9.1 The report should summarize the results from all three tiers. The report should contain a conclusion regarding the compostability (fragmentation, biodegradation, and safety) of the product based on the "weight of evidence."

10. Keywords

10.1 biodegradation; compostable; composting; degradable; plastic; polymer; strategy; toxicity

REFERENCES

(1) *Compost Facility Operating Guide*, Composting Council, Alexandria, VA, 1995.

(2) Skipper, H. D., et al, "Microbial Degradation of Herbicides," *Research Methods in Weed Science*, 1989, pp. 457–462.

(3) Jendrossek, D., et al, "Degradation of Poly(3-hydroxybutyrate), PHB, by Bacteria and Purification of a Novel PHB Depolymerase from *Comamonas* sp.," *Journal of Environmental Polymer Degradation*, Vol 1, 1993, pp. 53–63.

(4) Seal, K. J., "Test Methods and Standards for Biodegradable Plastics," *Chemistry and Technology of Biodegradable Polymers*, G. L. Griffin, ed., Blackie Academic and Professional, Bishopbriggs, Glasgow, 1994, pp. 116–134.

(5) "Toward Common Ground," *Proceedings of the International Workshop on Biodegradability*, Institute for Local Self-Reliance, Washington, DC, 1992.

(6) Schwab, et al, "Characterization of Compost from a Pilot Plant-Scale Composter Utilizing Simulated Solid Waste," *Waste Management and Research*, Vol 12, 1994, pp. 289–303.

(7) *Recommended Test Methods for the Examination of Compost and Composting*, Composting Council, Alexandria, VA, 1993.

(8) *Guidelines for the Use of Environmental Marketing Claims*, Federal Trade Commission, Washington, DC, 1992.

(9) *U.S. Solid Waste Composting Facility Profiles*, Vol II, National Composting Program, United Conference of Mayors, Washington, DC, 1993.

(10) Zucconi, et al, "Cress Seed Germination Bioassay," *Bicycle*, March/April 1981.

Designation: D 5338 – 98

An American National Standard

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

This standard is issued under the fixed designation D 5338; 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. This test method is designed to yield reproducible and repeatable test results under controlled conditions that resemble composting conditions. 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.

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 that are not inhibitory to the microorganisms present in aerobic composting piles.

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

1.6 This test method is equivalent to ISO 14852.

2. Referenced Documents

2.1 *ASTM Standards:*

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

D 883 Terminology Relating to Plastics[2]

D 1293 Test Methods for pH of Water[3]

D 1888 Test Methods for Particulate and Dissolved Matter, Solids, or Residue in Water[4]

D 2908 Practice for Measuring Volatile Organic Matter in Water by Aqueous-Injection Chromatography[5]

D 3590 Test Methods for Total Kjeldahl Nitrogen in Water[3]

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

E 260 Practice for Packed Column Gas Chromatography[7]

E 355 Practice for Gas Chromatography Terms and Relationships[7]

2.2 *APHA—AWWA—WPCF Standards:*

2540 D Total Suspended Solids Dried at 103 to 105°C[8]

2540 E Fixed and Volatile Solids Ignited at 550°C[8]

2.3 *ISO Standard:*

ISO 14852 Plastics—Evaluation of the Ultimate Aerobic Biodegradability and Disintegration Under Controlled Composting Conditions—Method by Analysis of Released Carbon Dioxide[9]

3. Terminology

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

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

[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.

Current edition approved July 10, 1998. Published September 1998. Originally published as D 5338 – 92.

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

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

[4] Discontinued; see *1991 Annual Book of ASTM Standards*, Vol 11.01.

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

[6] *Annual Book of ASTM Standards*, Vol 11.04.

[7] *Annual Book of ASTM Standards*, Vol 14.02.

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

[9] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

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D-20

APPLICATION

D-20 ON PLASTICS

This application gives you information about the committee, its scope, officers, and structures. If you would like to become active in the work of this committee, please fill out and mail the application to ASTM Headquarters or fax to 610-832-9667, attention Nancy McAvey, Manager, Member and Committee Services.

SCOPE: The development of test methods, specifications, recommended practices, nomenclature, definitions, and the stimulation of research relating to plastics, their raw materials, components, and compounding ingredients, and to finished products made from plastics such as sheets, rods, tubes, pipes, cellular materials, and molded or fabricated articles.

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polymer is inherently non-biased since radioactivity is a direct measure of the polymer carbon. However, an accurate true value is determined only when detection efficiencies are high, interferences are avoided, and proper calibration is performed. This can be accomplished routinely with present scintillation counting techniques.

15.2 Data on the precision and bias of these test methods between laboratories are being determined by a round-robin test, and will be available on or before December 2003.

10. Test Method A—Aqueous Environment

10.1 Prepare incubation flasks by adding 75 mL of limited BAM, 2 to 5 mL of inoculum and 50 to 100 mg (>500 000 dpm) of the radiolabeled plastic sample to a 125-mL Erlenmeyer flask.

10.2 Pass oxygen through the water flask and the incubation flask (each maintained at 58 ± 5°C) at a rate of 15 mL/min ± 2 mL/min.

10.3 Shake the flasks at 100 ± 10 rotations/oscillations per min.

10.4 The gasses leaving the incubation flask pass through the acid trap and then through the CO_2 absorption column containing 4 to 5 mL of methoxyethyl amine cooled to >15°C or through a column containing 8 to 10 mL of methoxyethyl amine without cooling.

10.5 Switch the gas flow to another absorption column in intervals from 4 to 8 h, depending on the objectives of the experiment and the methoxyethyl amine from the original column is drained into a scintillation vial.

10.6 Add 10 to 15 mL of scintillation cocktail to the scintillation vial and count the sample in the liquid scintillation counter set to measure the beta radiation of carbon-14.

11. Test Method B—Compost Environment

11.1 Uniformly distribute the radiolabeled plastic sample, cut in pieces approximately 2 cm^2(for film), in approximately 300 g of compost as the compost vessel is filled. Addition of 200 to 300 mg of plastic is sufficient for each composting vessel. Comparative samples should have similar surface areas.

11.2 Partially fill acid traps with concentrated sulfuric acid so as to accommodate the water removed from the airflow.

11.3 Keep the compost vessel and humidifying trap in a 58 ± 5°C chamber. Record the temperature of each composter daily and ensure that it is within 5°C of the chamber. Raise the temperature of the air to 58°C by passing it through a coil in the heated chamber prior to entering the water trap (humidifying trap). Bypass these humidifying traps during the first 2 to 3 days due to the production of large amounts of metabolic water. Sample the composters at 3 days and weekly thereafter to determine the percent moisture of the compost. If the percent moisture drops to 50 % pass the incoming air through the water traps. Control moisture in the compost at 50 to 60 %.

11.4 Fill the CO_2 absorption columns with >200 mL of methoxyethyl amine. Drain and measure the volume, and place a 2-mL aliquot in a scintillation vial with 15-mL scintillation fluid. Count in a Beta counter calibrated to count carbon-14.

NOTE 1—It is important in the early days of the composting process to monitor the viscosity of methoxyethyl amine by observing the bubble flow. The large quantity of CO_2 evolving during this period can cause the methoxyethyl amine to solidify. Usually a daily collection and refill of methoxyethyl amine will be sufficient. In order to reduce the volatility of this short-chain amine, the column can be cooled by circulating refrigerant at temperatures< 15°C through the jacket of the column. If the absorption column is not cooled, an additional 20 % of the absorbant must be used. Generally, absorption columns are completely drained and refilled once a day.

NOTE 2—Occasionally two phases develop due to larger quantities of water trapped in the methoxyethyl amine, and these two phases can be blended into one by the addition of several millilitres of methanol. During prolonged composting trials (over 20 days) the sampling interval can be extended depending upon the objectives of the experiment.

11.5 In order to test whether the oxidation of the carbon in the plastic could occur chemically under these composting conditions, it may be necessary to use a sterile control. This is not necessary if the chemistry of the compound being tested is well documented and it is known that chemical oxidation does not occur under these composting conditions.

12. Calculation

12.1 Total dpm in a composting vessel at the start of the test (ST, dpm) is calculated as follows:

$$ST, \text{dpm} = (SA)(LP) \quad (1)$$

where:
SA = specific activity in dpm/mg, and
LP = mg of labeled plastic added to the vessel.

12.2 To calculate the quantity of radioactivity (dpm) detected in CO_2 absorbent, measure the volume of methoxyethyl amine drained from the absorption column. Then, measure the radioactivity in a 2-mL aliquot. Employ the following equation:

$$C, \text{dpm} = [(Al - B)/Cv]/2 \quad (2)$$

where:
C, dpm = total dpm per collection,
Al = dpm in the 2-mL aliquot,
B = background dpm, and
Cv = millilitres from each column collection.

12.3 Calculate the cumulative percent production of carbon-14 carbon dioxide ($^{14}CO_2$) or percent of plastic biologically oxidized (P_x, %) using the following equation:

$$P_x, \% = \Sigma_{1-n}(C, \text{dpm/ST, dpm})\ 100 \quad (3)$$

13. Interpretation of Results

13.1 The $^{14}CO_2$ measured in these test methods are a direct indication of the oxidation of the sample. However, the extent and the rate of oxidation are related to the compost mixture made for that individual test and the form of the sample. Although different batches of compost can produce different results, the compost formula for the simulation of MSW in these test methods will generally give repeatable results. This is due to the selection of common feed ingredients that are standardized in the trade and tend to have a consistent composition. Depending upon the objectives of the test, it is generally wise to include within each test series a standard preparation of known degradation rates and to make test comparisons.

14. Report

14.1 Report the following information:

14.1.1 Data regarding compost temperature and moisture,

14.1.2 Daily (or periodic) production of $^{14}CO_2$,

14.1.3 Cumulative periodic summation of $^{14}CO_2$,

14.1.4 Graphic display of $^{14}CO_2$ summation data comparing treatments, and

14.1.5 Average and standard deviation of replicates if there are sufficient numbers.

15. Precision and Bias

15.1 Measurement of radioisotope labeled (carbon-14)

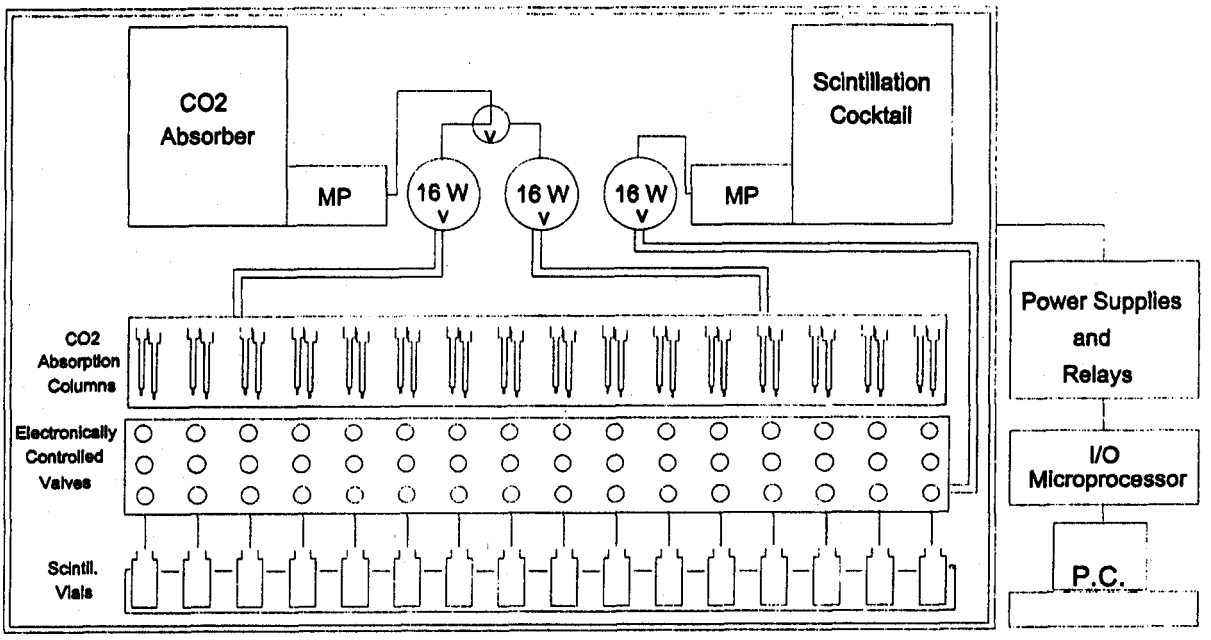

FIG. 4 Fully Automated ^{14}C CO_2 Collection System for Sixteen Units (MP = metering pump, v = valves, 16Wv = sixteen way valves)

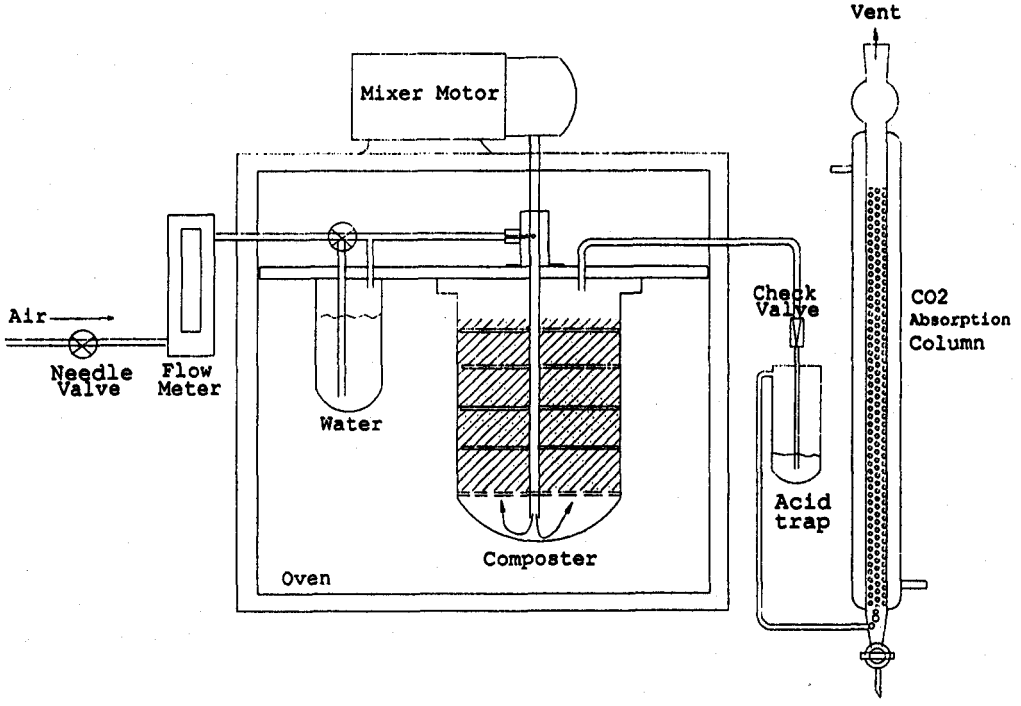

FIG. 5 Automated Radiochemical Composting Apparatus

lies in the ability to detect and distinguish that carbon-14 isotope from a virtual sea of carbon-12 in the compost. This ability to follow or trace that carbon due to its radioactivity requires that the radiochemical purity of the compound be established, that is, verify that only the compound of interest is labeled and no label is present in any other material. This verification requires an analytical technique that can separate the polymer and determine that the radioactivity resides solely in the polymer, and that the structure (NMR analysis) and molecular weight distribution (see D 5296) are the same as the polymer intended for commercial production.

9.2 Thickness of polymer film can be an important variable for any biodegradation test. Thus, the thickness should be measured by a micrometer and included with any data describing biodegradation rates of polymeric material. Further, in the preparation of such films, take care to produce a film of uniform thickness.

9.3 The radiolabeled plastic should be prepared with an SA of at least 7000 dpm/mg. ASA of 10 000 dpm/mg provides sufficient counts for extended test.

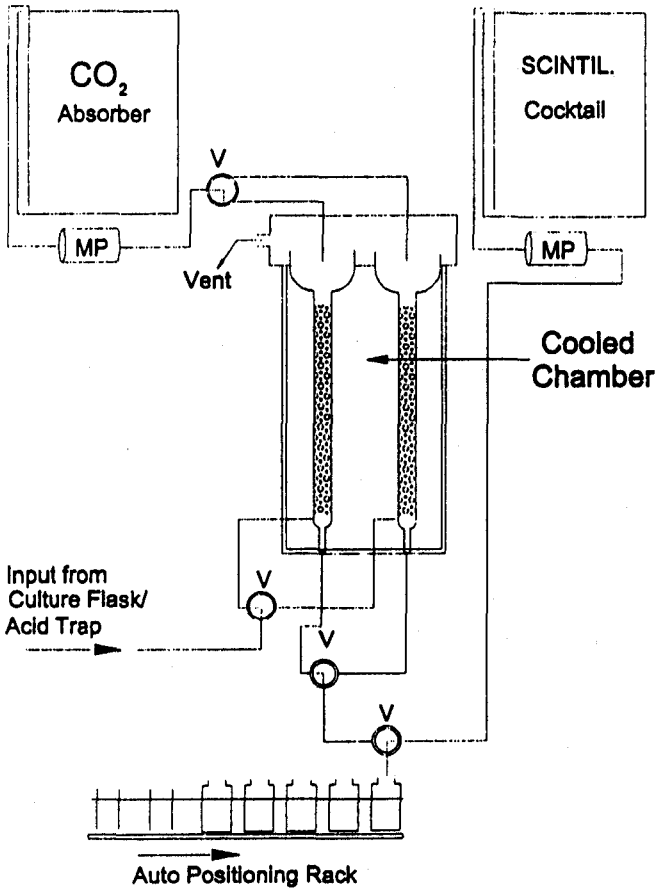

FIG. 3 Single Unit from an Automated ^{14}C CO_2 Collection System (V = valve and MP = metering pump)

in series and insuring that no radioactivity is trapped in the second column.

6.3.1.7 The CO_2 absorbent must not interfere with the accurate measurement of radioactivity in the liquid scintillation counter by incompatibility with any other reagent.

7. Reagents and Materials

7.1 Compost for this testing may be made up in accordance with either of the following recipes, municipal solid waste (MSW) or woody compost, or obtained from an active municipal solid waste or yard waste composting center.

7.1.1 Compost, designed to stimulate municipal solid waste (MSW) organic matter, is prepared by combining the following materials (on a dry matter basis): alfalfa meal, 35.7 %; shredded newspaper, 27.2 %; garden soil, 13.2 %; poplar sawdust, 10.4 %; cottonseed meal, 6.4 %; cow manure, 1.6 %; calcium carbonate; 5.3 %; sodium bicarbonate, 0.2 %. The mixture is designed to contain the biochemical ingredients found in MSW (lignified cellulose (newspaper, poplar sawdust and alfalfa meal), protein (cottonseed meal and alfalfa meal), natural inoculum (garden soil and cow manure); soluble carbohydrates and buffering capacity sufficient to maintain a neutral to slightly basic pH). The particle size of the components of the compost mix are sized to pass through a 6-mm screen. Moisture content should be adjusted (see 7.1.3) to 55 to 60 % for testing.

7.1.2 An alternate compost (used to simulate yard composting activities) was used in an ASTM-ISR study[4] and has the following composition on a dry matter basis (made up to 55 to 60 % moisture for testing): wood chips (brush), 41.4 %; chapped leaves, 25.4 %; grass clippings, 25.8 %; aged compost, 7.3 %. Different compost preparations can produce different rates of biodegradation. A yard and garden waste compost can, with certain plastics, result in slower rates of degradation.[4]

7.2 The compost is made up to 55 to 60 % moisture by measuring the moisture content of each component, calculating the moisture content of the mix, and adding water to bring the mix to near target moisture percentage. The moisture content can be checked by taking a sample of the compost, weighing it, drying it, reweighing it, and calculating the moisture from the difference in weight. Water can then be added as needed. During the experiment, testing of the moisture content should be checked weekly and maintained at 55 to 60 % by humidifying (bubbling the air through water) the air as needed.

7.3 Liquid culture inoculum are prepared from the target aqueous environment. They can be used directly, that is, sewage sludge or waste water treatment sludge, or eluant from compost, or they can be enriched by growing the mixed culture on the carbon source of interest.

7.4 Media used for liquid culture is related to the environment being simulated. For waste water or sewage sludge, a limited basal media (BAM broth minus glucose) is prepared according to the Handbook of Microbial Media.[5] This media relies on the plastic for the major source of carbon bond energy.

7.5 The carbon dioxide absorber, methoxyethyl amine[6] is used to capture CO_2 in the scrubbing columns.

7.6 A liquid scintillation cocktail designed for counting carbon-14 is required. Any commercial scintillation cocktail reagent that is compatible with the CO_2 absorber is acceptable.[7]

8. Hazards

8.1 Compost presents well-known health risks that can be avoided by the use of appropriate protective equipment such as gloves and masks.

8.2 Strong bases used for CO_2 absorption are particularly hazardous and instructions in Material Safety Data Sheets must be followed.

8.3 Radioactive carbon-14 compounds must be handled in accordance with federal and state regulations.

9. Radiolabeled Preparations

9.1 The utility of tagging the carbon in a molecule or a portion of the molecule with the radioactive isotope of carbon,

[4] ISR Degradable Polymeric Materials Program, *Compilation of ISR Contractor Compost Test Report*, ASTM, 1993.

[5] R.M. Atlas, *Handbook of Microbiological Media*, CRI Press, Inc., 1993, p. 113.

[6] Carbosorb® (Packard Instrument Co.) is a strongly basic short-chain amine is a highly efficient CO_2 absorber and is compatible with many scintillation cocktails. Other organic bases can be used for trapping CO_2 but they generally lack the efficiency of methoxyethyl amine. Inorganic bases have several disadvantages for example, they are strong quenching agents, produce severe color or turbidity interference, and have low trapping capacities.

[7] Although the manufacturer of Carbosorb®, recommends their product Permaflour E+®, other scintillation cocktails are also compatible, including the Beckman Environmental Cocktail Ready Safe®.

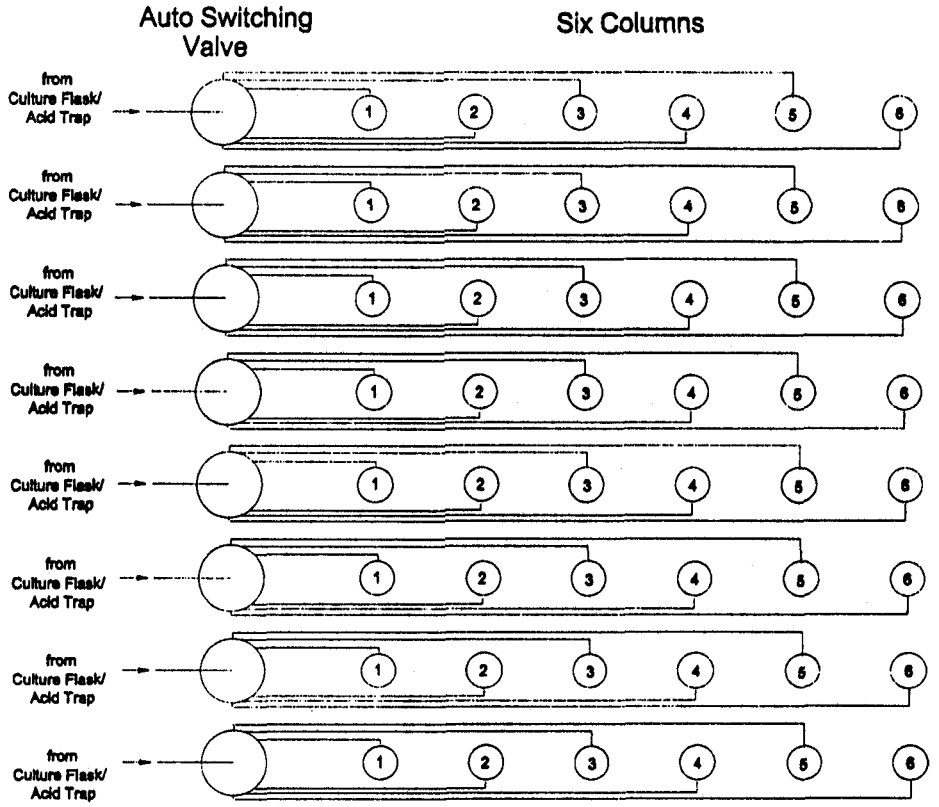

FIG. 2 Semiautomated ^{14}C CO_2 Collection System for Eight Units Over Six Sampling Periods

controlled temperature chamber, a sulfuric acid trap, and a CO_2 absorption column.

6.2.2 The composting vessel is a 1-L borosilicate glass reaction kettle with a glass flange tooled to receive an "O" ring, clamped against an inert plastic surface. Pressurized air, controlled by a needle valve, is passed through a flow meter and then either through a water trap, maintained at the same temperature as the compost, or directly to the compost (25 ± 3 cc/min). The composting vessel is fitted with a central hollow stainless steel shaft that protrudes through a perforated distributor plate at the bottom of the vessel (Fig. 5). The air is passed down the shaft to the space below the distributor plate and then passes up through the compost to the top of the compost where it exits from the vessel. The shaft contains rods projecting perpendicular from the shaft in a radiating fashion. The shaft is connected to a motor that turns the shaft at rate of about 6 r/min. The mixing motion is designed to mix, break up clumps and convey the compost upward. The resultant action tends to circulate the compost in the composting vessel and maintains an even flow of air through the compost.

6.2.3 As air exits the composting vessel, it passes through a check valve and then proceeds through a sulfuric acid trap. The trap dehydrates the air and insures that the CO_2 stays in the gas phase.

6.2.4 The air then passes into a glass column filled with glass helixes and a commercial CO_2 absorber, methoxyethyl amine. The glass helixes break up the gas bubbles and provide greater surface area for the absorption (scrubbing) of CO_2.

6.2.5 The column is jacketed (has an outer glass chamber) where a refrigerant (propylene glycol) is circulated.

6.2.6 A liquid scintillation counter, capable of counting the low-energy beta emitted by the radioactive isotope carbon-14 is used to measure the quantity of radioactivity in the trapped CO_2. An instrument that can automatically measure counting efficiency and correct for quenching is preferred.

6.2.7 It is important to test the system for leaks and insure that the radioactive CO_2 does not escape from the apparatus both for accurate results and safety of personnel.

6.2.8 Vent columns to a radiochemical hood.

6.2.9 Place check valves, that will allow the air flow to travel in only one direction, between the test flasks and the acid and between the acid and absorber.

6.3 *Alternate Composting Apparatus*:

6.3.1 Alternative compost apparatus can be used if it conforms to the following requirements:

6.3.1.1 Although compost vessels can be larger than 1-L, it is generally not practical to exceed 1-L, due to the large volume of CO_2 that is produced and must be completely absorbed.

6.3.1.2 The compost temperature must be controlled.

6.3.1.3 The air supply flow must be controlled and humidified.

6.3.1.4 The air must be stripped of moisture prior to scrubbing to eliminate the two phases that occur when the scintillation cocktail is added to CO_2 absorber that has accumulated too much water.

6.3.1.5 The scrubbing column must be designed to permit enough air/liquid contact to completely capture the CO_2.

6.3.1.6 The efficiency of the absorption column must be verified. This can be accomplished by placing another column

4. Summary of Test Method

4.1 Test Method A involves the characterization of the test material, the preparation of the natural mixed culture inoculum, the control of the culture environment, the collection and measurement of radioactive carbon dioxide (CO_2) over time, and the calculation and interpretation of the results. The results may be compared to those obtained from Test Method D 5209.

4.2 Test Method B involves the characterization of the test material, the preparation of the compost matrix, the control of the composting process, the collection and measurement of radioactive CO_2 over time, and the calculation and interpretation of the results. The results may be compared to those obtained from Practice D 5512 as well as Test Method D 5338.

5. Significance and Use

5.1 These test methods can provide direct and unequivocal evidence of aerobic biodegradability. This requires that the radiochemical purity of the plastic is verified using Test Method D 5296.

5.2 These methods also provide the opportunity to determine the rate of biological oxidation in a complete composting environment or aqueous environment by frequent periodic sampling of carbon dioxide.

5.3 These methods provide biodegradation data at use levels of the plastic in a full cycle composting process or an aqueous system.

6. Apparatus

6.1 *Liquid Culture Apparatus*:

6.1.1 Fig. 1 is a diagrammatic representation of a single unit for measuring the carbon-14 carbon dioxide (CO_2) production from the biodegradation of a labeled polymer in aqueous culture. It consists of a fine needle valve for the sensitive control of oxygen flow, a water and culture flask in a controlled temperature environment, a trap to remove water from the gas stream and to insure the carbon monoxide (CO) stays in the gas phase, and a CO_2 absorption column: Periodic CO_2 production over a chosen period of time can be sampled by collecting the CO_2 absorbent from the column at the end of each period by hand, or by automating the CO_2 collection.

6.1.2 Fig. 2 illustrates an eight-unit system with a semi-automated CO_2 collection system based on a timed, automated six-way valve. The gas effluent from the culture flask and acid trap is continuously passed through an absorption column and periodically switched to the next column. Just before the sixth column is due to switch, the five columns are drained and refilled. Soon after the sixth column switches, it is drained and refilled.

6.1.3 Fig. 3 represents a single unit from a fully automated CO_2 collection system where two absorption columns are alternately used to capture the CO_2. While one column is collecting CO_2 from the effluent, the other is drained into a scintillation vial, scintillation cocktail is added to the vial, and the column is refilled with the CO_2 absorbent automatically.

6.1.4 Fig. 4 is a diagrammatic representation of a sixteen-unit, fully automated system. The system is controlled by a personal computer and an 1/0 microprocessor. Valves and metering pumps are powered by electronically-controlled power supplies and relays. Reservoirs of CO_2 absorbent and scintillation cocktail serve all sixteen units. The scintillation vials are in a rack that positions the vials for each sampling period.

6.1.5 Alternative apparatus can be used if it has the capability of maintaining the appropriate temperature, controlling the oxygen flow, humidification of gas flow, and complete collection of CO_2.

6.1.6 Alternate apparatus can be manually operated or controlled by computer interface.

6.2 *Composting Apparatus*:

6.2.1 Fig. 5 is a diagrammatic representation of the radiochemical composting apparatus. The radiochemical composting apparatus consists of a glass composting vessel capped by an inert plastic surface, a controlled humidified air flow, a

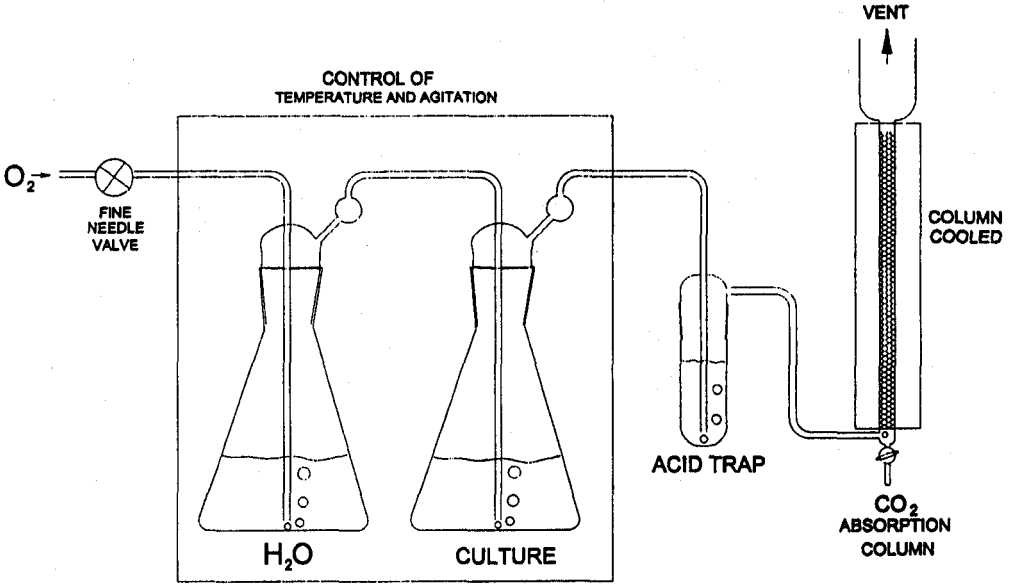

FIG. 1 Single Unit for Measuring ^{14}C CO_2 Production from the Biodegradation of a ^{14}C-Labeled Polymer

Designation: D 6340 – 98

Standard Test Methods for Determining Aerobic Biodegradation of Radiolabeled Plastic Materials in an Aqueous or Compost Environment[1]

This standard is issued under the fixed designation D 6340; 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 These test methods directly determine the rate and degree of biological oxidation of carbon in plastic materials when placed in a composting environment containing simulated municipal solid waste or an aqueous environment under laboratory conditions.

1.2 Test Method A utilizes a mixed culture derived from the target environment (waste water, sewage sludge, compost eluant, and other environmental sources). Temperature, mixing, and aeration are monitored and controlled.

1.2.1 This method has the sensitivity to determine biodegradation at concentrations commonly found in these environments.

1.3 Test Method B starts with fresh compost and proceeds through the normal composting process to an early mature stage. Temperature, aeration; and moisture are monitored and controlled.

1.3.1 This method can determine biodegradation at levels of the plastic commonly expected in municipal solid waste.

1.4 These test methods require that the target component of the plastic material be synthesized using the radioactive isotope carbon-14. Depending upon the objective, either a portion of the components of the plastic or all of the carbon can be uniformly labeled with carbon-14. The test method will determine how that labeled portion will be metabolized and biologically oxidized by the microorganisms in the system tested.

1.5 These test methods can be applied to any carbon-14 labeled compound as well as for plastic materials that have been formulated to biodegrade in a natural aerobic environment.

1.6 The synthesis and preparation of the radiolabled plastic is beyond the scope of these methods. Carbon-14 labeled polymers may be purchased from a number of commercial labs.

1.7 There are no ISO test methods that are equivalent to the test methods in this standard.

1.8 The safety problems associated with compost and radioactivity are not addressed in this standard. It is the responsibility of the user of this standard to establish appropriate safety and health practices. It is also incumbent on the user to conform to all the regulatory requirements, specifically those that relate to the use of open radioactive sources.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 883 Terminology Relating to Plastics[2]

D 5209 Test Method for Determining the Aerobic Biodegradation of Plastic Materials in the Presence of Municipal Sewage Sludge[3]

D 5296 Test Method for Molecular Weight Averages and Molecular Weight Distribution of Polystyrene by High Performance Size Exclusion Chromatography[3]

D 5338 Test Method for Determining Aerobic Biodegradation of Plastic Materials Under Controlled Composting Conditions[3]

D 5512 Practice for Exposing Plastics to Simulated Compost Environment Using an Externally Heated Reactor[3]

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods as they relate to composting, see Terminology D 883.

3.1.1 *specific activity, SA, n*—refers to the quantity of radioactivity per mass unit of compound (polymer, etc.), that is *dpmh%*.

3.2 Acronyms:

3.2.1 *Bq, n*—becquerel; SI unit where 1 curie (Ci) = $3.7 \cdot 10^{10}$ Bq.

3.2.2 *dpm, n*—disintegrations per minute, used to measure the quantity of radioactivity.

3.2.2.1 *Discussion*—The measure dpm is derived from counts per minute (cpm) where dpm = cmp-bkgd/counting efficiency. There are $2.2 \cdot 10^6$ dpm/μCi.

3.2.3 *mCi, n*—millicurie; 1/1000th of a curie (standard unit).

3.2.4 *μCi, n*—microcurie; 1/1000th of a millicurie.

3.2.5 *MSW, n*—municipal solid waste (organic matter).

[1] These test methods are under the jurisdiction of Committee D20 on Plastics and are the direct responsibility of Subcommittee D20.96 on Degradable Plastics.

Current edition approved Nov. 10, 1998. Published February 1999.

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

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

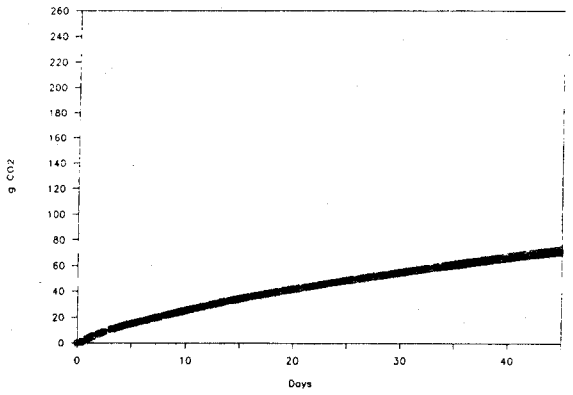

FIG. 3 Cumulative CO_2 Production from Inoculum

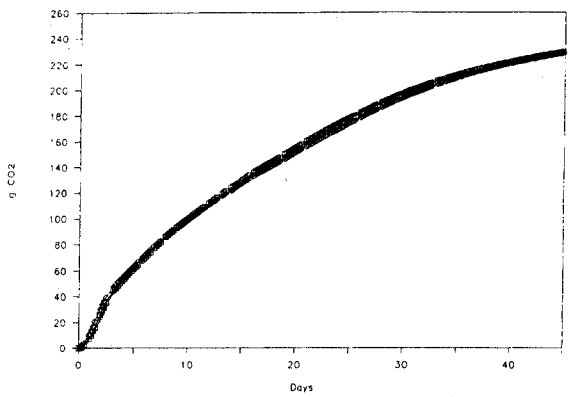

FIG. 4 Cumulative CO_2 Production from Inoculum Plus Cellulose

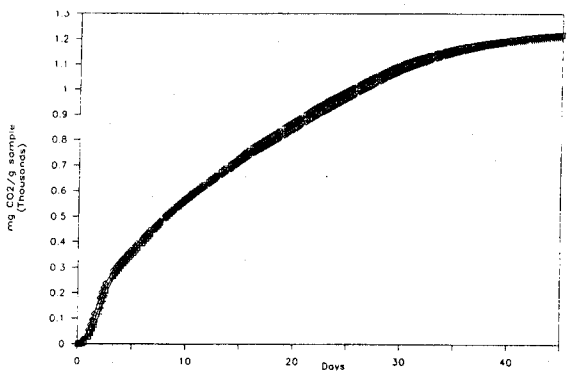

FIG. 5 Net Cumulative CO_2 Production from Cellulose

activity (CO_2 production in the first ten days), date of collection, storage, and handling.

14.1.2 Carbon content of the plastic materials, positive and negative control, and calculation of the theoretical maximum carbon dioxide production. Report specific information on the size, shape, volume, and thickness of the plastic materials tested, along with the form of the plastic material, that is, sheet, powder, pellet, etc.,

14.1.3 Weight of vessels with contents before and at the end of the test,

14.1.4 Cumulative carbon dioxide evolution and oxygen consumption over time and display graphically. Report apparatus used for carrying out the test method,

14.1.5 Percentage of aerobic biodegradation for each plastic material tested, the standard deviation and 95 % confidence interval for each material or control substance tested for the percent of biodegradation,

14.1.6 Percentage of biodegradation relative to the positive reference (cellulose = 100 %),

14.1.7 Temperature range of test, and

14.1.8 pH of compost inoculum and pH of final residues. Volatile fatty acids concentration for vessels with a final pH of less than 7.

15. Precision and Bias

15.1 The precision and bias of the procedure in this test method is being determined.

15.2 Preliminary results for within-laboratory repeatability testing using a controlled composting set-up with gas chromatograph are presented in Table 1. These data represent three different determinations of the degradation of cellulose as a positive reference. The average degradation of cellulose after 45 days of composting at a constant temperature of 50°C (±2°C) was 75.3 %, with an average standard deviation of 2.5 % and an average 95 % confidence limit interval of 5 %. All three runs were carried out within a seven-month period by the same operators. Figures 3, 4, and 5 represent a graphical view of the third run in which a biodegradability of 78.9 % was obtained as the mean for the three replicates containing cellulose as the positive control, with a standard deviation of 0.3 % and a 95 % confidence limit interval of 2 %. The graphs represent the results from three replicates for the inoculum only as the blanks (see Fig. 3), the cellulose as the positive control (see Fig. 4) and the net CO_2 production per gram of cellulose added (see Fig. 5).

16. Keywords

16.1 aerobic biodegradation; biodegradation; composting; plastics

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.

11.4 *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.

11.4.2 Measure the pH in conformance with Test Methods D 1293. 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 D 2908. 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:

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

where:

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

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

$$S_e = SQRT((s^2_{\text{test}}/n1) + (s^2_{\text{blank}}/n2)) \times 100/C_i$$

$n1$ and $n2$ are the number of replicate test and control digesters respectively; s is the standard deviation of the total gaseous carbon produced.

12.5 Calculate the 95 % confidence limits as follows:

$$95\ \%\ CL = \%\ \text{biodegradation} \pm (t \times s_e)$$

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

13. Interpretation of Results

13.1 Information on the toxicity of the plastic material may be useful in the interpretation of inhibitive effects.

13.2 In most instances when investigating a plastic material, a reference or control substance known to biodegrade is necessary in order to check the activity of the inoculum. If sufficient biodegradation (a minimum of 70 % for cellulose within 45 days) is not observed with the positive reference, the test must be regarded as invalid and should be repeated, using new inoculum.

14. Report

14.1 Report the following data and information:

14.1.1 Information on the inoculum, including source, percent dry solids, percent volatile solids, total Kjeldahl nitrogen,

TABLE 1 Results from Within-Laboratory Testing for the Aerobic Biodegradability of Cellulose as a Positive Control Under Controlled Composting Conditions

	Biodegradability after 45 days, %	Standard Deviation, %	95 % Confidence Limit, %
Run 1	76.7	4.9	8.2
Run 2	70.3	2.4	4.8
Run 3	78.9	0.3	2.0
Mean of Three Runs	75.3	2.5	5.0

7.2 *Analytical-Grade Cellulose*, for thin-layer chromatography with a particle size of less than 20 μm as positive control.[10]

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.

10. Test Specimens

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 D 618.

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 D 3590, D 1888, 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 D 4129.

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.

11.2 *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 for a period of 45 days. Initially, keep the incubation temperature at 35°C (±2°C) for a period of one day to simulate a mesophilic start-up phase. Subsequently, raise the temperature to 58°C (±2°C) for a period of four days. After this sanitizing period, reduce the temperature to 50°C (±2°C) for optimum composting conditions and maintain until day 28. Reduce the temperature then to 35°C (±2°C) for the remainder of the test period to simulate a mesophilic curing phase. The incubation time of 45 days may be extended until no significant CO_2 production in excess of the inoculum is recorded for a period of one week.

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

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

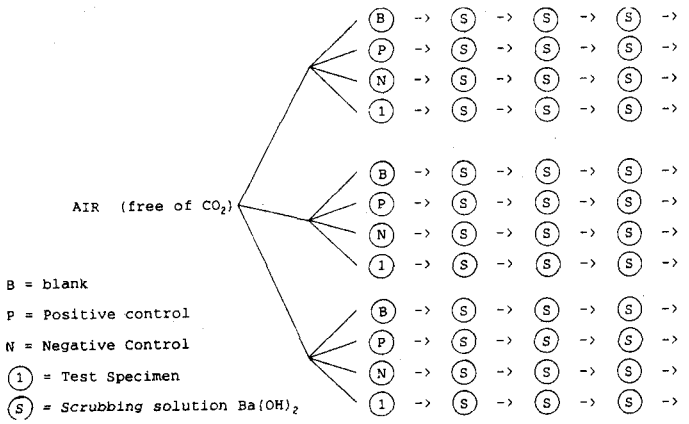

FIG. 1 Set-Up Using Carbon Dioxide-Trapping Apparatus

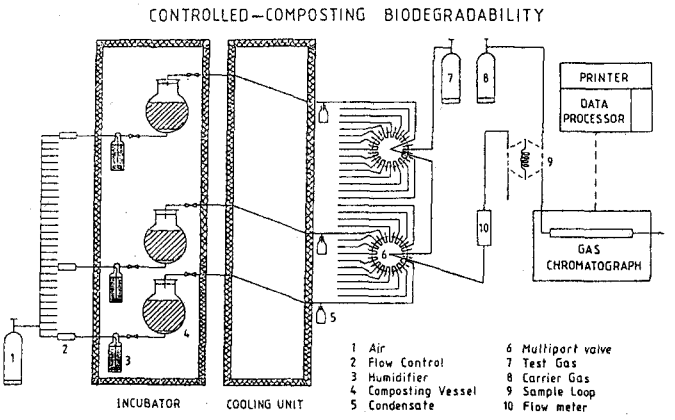

FIG. 2 Optional Set-Up Using a Gas Chromatograph

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 will 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 solid-waste composting plants. 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 systems 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. 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.

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 (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*, (±0.1 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 E 260 and E 355 (see Fig. 2).

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.