Designation: D6340 – 98 (Reapproved 2007)

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 D6340; 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:*[2]

D883 Terminology Relating to Plastics

D5209 Test Method for Determining the Aerobic Biodegradation of Plastic Materials in the Presence of Municipal Sewage Sludge (Withdrawn 2001)[3]

D5296 Test Method for Molecular Weight Averages and Molecular Weight Distribution of Polystyrene by High Performance Size-Exclusion Chromatography

D5338 Test Method for Determining Aerobic Biodegradation of Plastic Materials Under Controlled Composting Conditions, Incorporating Thermophilic Temperatures

D5512 Practice for Exposing Plastics to a Simulated Compost Environment Using an Externally Heated Reactor (Withdrawn 2002)[3]

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods as they relate to composting, see Terminology D883.

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

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

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

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).

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 D5209.

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 D5512 as well as Test Method D5338.

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 D5296.

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

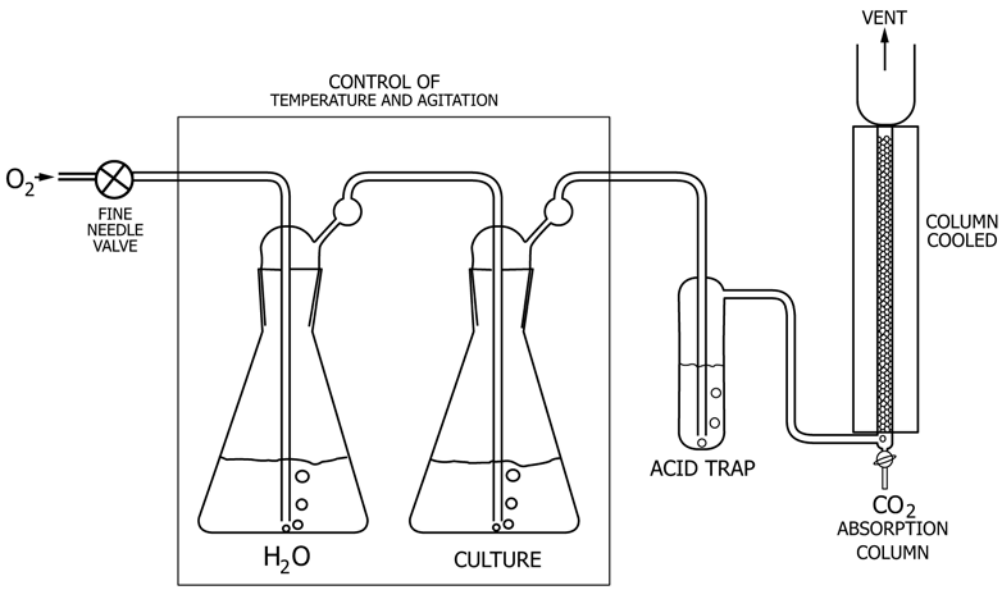

FIG. 1 Single Unit for Measuring ^{14}C CO_2 Production from the Biodegradation of a ^{14}C-Labeled Polymer

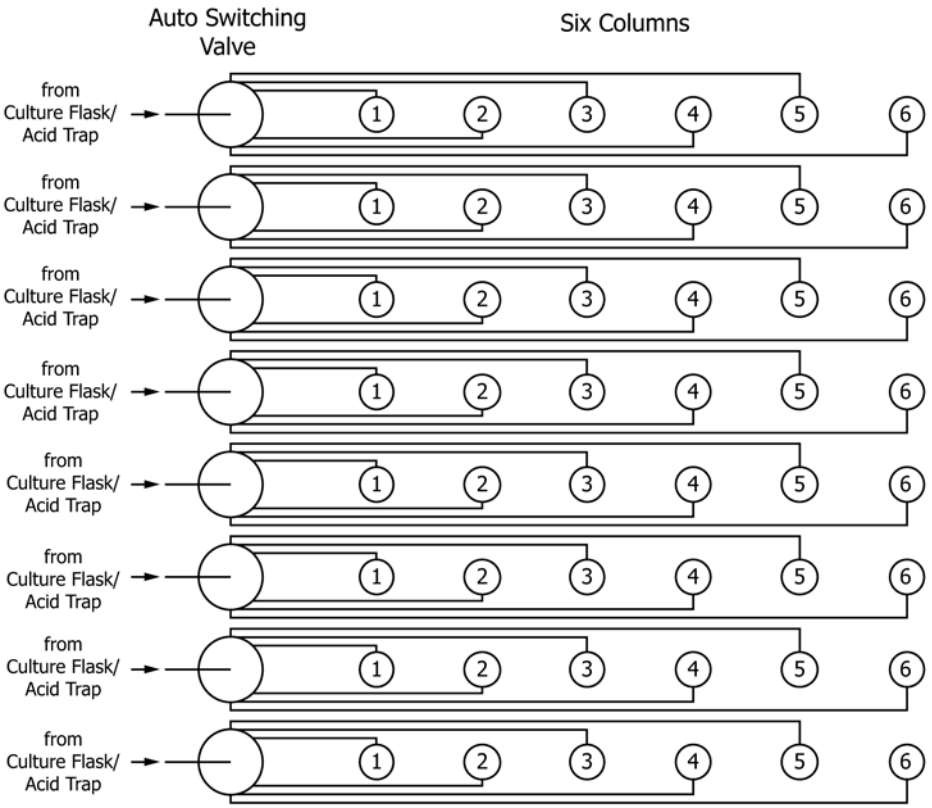

FIG. 2 Semiautomated ^{14}C CO_2 Collection System for Eight Units Over Six Sampling Periods

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 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:*