



Designation: D7991 – 22

Standard Test Method for Determining Aerobic Biodegradation of Plastics Buried in Sandy Marine Sediment under Controlled Laboratory Conditions¹

This standard is issued under the fixed designation D7991; 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 biodegradation level of plastic materials exposed to laboratory conditions that simulate the environment found in the sandy tidal zone.

1.2 The tidal zone, that is, the part of the coast affected by the tides and movement of the waves, is the borderline between sea and land, frequently a sandy area that is kept constantly damp by the lapping of the waves. Stony and rocky shorelines also exist.

1.3 Plastic marine debris is frequently washed up in this habitat where it must be removed in order to restore the original landscape.

1.4 It is of interest to know the biodegradation behavior of plastics when exposed to conditions simulating this habitat, because this information can help in predicting the time needed for the biodegradation of the litter.

1.5 Biodegradation is determined by measuring the CO₂ evolved by the plastic material when exposed to a sediment kept wet with salt-water in a reactor, to simulate the tidal zone.

1.6 Marine fresh-water habitats (for example, those found in brackish waters and estuaries) are not considered by this standard.

1.7 Reports shall clearly state the percentage of net CO₂ generation for both the test and reference samples at the completion of the test. Furthermore, in the laboratory reports, the results shall not be extrapolated beyond the actual duration of the test.

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

1.8 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

1.10 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 *ASTM Standards:*²

D5988 Test Method for Determining Aerobic Biodegradation of Plastic Materials in Soil

2.2 *ISO Standards:*³

ISO 8245 Water quality Guidelines for the determination of total organic carbon (TOC) and dissolved organic carbon (DOC)

3. Terminology

3.1 *Definitions:*

3.1.1 *tidal zone, n*—the part of the marine environment that extends from the high tide line, which is rarely inundated with water, to the low tide line, which is typically always covered with water.

3.1.1.1 *Discussion*—Synonyms are: eulittoral zone, midlittoral zone, mediolittoral zone, intertidal zone, foreshore.

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 aerobic biodegradation in a controlled laboratory system.

4.1.2 Obtaining sediment and seawater from the shoreline.

¹ This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.96 on Environmentally Degradable Plastics and Biobased Products.

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² 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 Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

4.1.3 Exposing the plastic material to the wet sediment under controlled conditions.

4.1.4 Measuring CO₂ evolved as a function of time.

4.1.5 Assessing the degree of biodegradation by determining the percentage of organic carbon in the plastic material that is converted to CO₂ during the duration of the test. This percentage represents the percentage of mineralization and will not include the amount of carbon converted to cell biomass that is not in turn metabolized to CO₂ during the course of the test.

4.1.6 Estimating the qualitative disintegration of the test material by visual inspection at the end of the test.

5. Significance and Use

5.1 Plastic is sometimes carried by rivers or accidentally discharged by ships into the sea; this plastic can then reach different parts of the marine environment. Tides and waves also frequently deliver plastic marine debris into the sandy tidal zones.

5.2 This test method simulates the environmental conditions found in the tidal zone. Plastic debris that reaches the sandy tidal zone can settle there and become partially or totally buried by sand and kept wet by waves or tides. It is of interest to assess the biodegradation behavior of plastic materials under these conditions to predict the removal time of this waste in the environment.

5.3 This test method is applied to determine the extent of biodegradation of a plastic exposed in the laboratory to a sandy sediment kept wet with seawater. Both sediment and seawater are collected from a sandy beach in the tidal zone. If the natural microbial population present in the sediment is able to biodegrade the plastic, there will be an evolution of CO₂ as a consequence of the aerobic microbial respiration. The level of biodegradation at any given time is the ratio between the cumulative amount of the evolved net carbon dioxide and the theoretical amount produced in the case of total conversion of the organic carbon present in the plastic into carbon dioxide.

5.4 This test method does not measure the amount of organic carbon that is converted into biomass, but only the biodegradation that leads to mineralization (that is, the formation of CO₂).

6. Apparatus

6.1 *Reactor*—Glass vessel approximately 2 to 4-L internal volume that can be sealed air-tight, such as 150-mm desiccators, with an airtight opening, large enough to allow the handling of the content. Biometer flasks are also appropriate. A suitable apparatus is shown in Figure 1 in Test Method D5988. Reactors with higher volumes can be used, if environmental conditions are not affected.

6.2 *Container for the CO₂ Absorber*—A glass beaker to be located in the headspace of the reactor and filled with 100 mL of Ba(OH)₂ 0.025 N or with 30 mL of KOH 0.5 N.

6.3 *Darkened Chamber or Cabinet*, in which the temperature can be maintained at a constant level within a $\pm 2^\circ\text{C}$ range.

NOTE 2—Incubator with either built in lights that can be programmed or else plug in lights that can be operated with a timer power strip can be used to better simulate the environment. The lighting in that case need to

be 12:12 day/night. Details on the lighting regime, light intensity, wave length, incubator type, etc. shall be provided in the report.

6.4 *Analytical Balance*, to weigh the test specimen.

6.5 *Technical Balance*, to weigh reactors and sediment.

6.6 *pH Meter*.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁴ It is acceptable to use other grades provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Barium Hydroxide Solution (0.025 N)*, prepared by dissolving 4.0 g anhydrous Ba(OH)₂/L in distilled water. Filter free of solid material and store sealed as a clear solution to prevent absorption of CO₂ from the air. It is recommended that 2 to 4 L be prepared at a time when running a series of tests. Confirm normality by titration with standard acid before use. When using Ba(OH)₂, however, care must be taken that a film of BaCO₃ does not form on the surface of the solution in the beaker, which would inhibit CO₂ diffusion into the absorbing medium. Alternatively, potassium hydroxide solution (KOH, 0.5 N) could be used and is prepared by dissolving 28 g of anhydrous KOH/L in distilled water and proceeding in the same way as for the Ba(OH)₂ solution.

7.3 *Hydrochloric acid*, 0.05 N HCl when using 0.025 N Ba(OH)₂ or 0.3 N HCl when using 0.5 N KOH.

7.4 *Sediment*—Collect seawater and sediment samples from the shoreline of a sandy beach, where the sediment is submerged in the shallow water. Collect top sediment (the layer from surface till about 20 cm depth). It is important to obtain sediment from multiple samples from the same location (at least 3). Collect the seawater with a bucket and then collect sediment samples with a shovel in separate containers overlain with water, then transfer all samples to a watertight container and quickly deliver it to the laboratory. Remove any obvious plant material, shells, pieces of driftwood, petroleum tar, and other large material. Store the sediment and seawater at approximately 4°C until use. Allow air exchange at time to avoid anaerobiosis. Use preferably within four weeks of sampling. Report the storage times. Before use, perform gravity filtration on the sediment in a funnel with a coarse filter paper to remove excess water. Sediment is ready for testing when seawater is no longer recovered from the filtration. Nitrogen sources (such as NH₄Cl or NaNO₃) can be added to the sediment if this is considered as a factor limiting biodegradation. These additions shall be reported in the test report.

NOTE 3—No data are available at this stage indicating that a specific

⁴ Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Anal. Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.