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Standard Practice for Preparing Residual Solids Obtained After Biodegradability Standard Methods for Plastics in Solid Waste for Toxicity and Compost Quality Testing[1]

This standard is issued under the fixed designation D 5951; 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 practice covers a standard procedure for preparing the residual solids obtained at the end of standard methods for biodegradability testing of plastics in solid waste, for subsequent toxicity and compost quality testing. The practice yields mixtures that can be used as such for terrestrial toxicity testing or that can be submitted to water extraction for further aquatic toxicity tests, in accordance with Practice D 5152, and in conjunction with Method D 4229, Guides E 729, E 1192, E 1295, and E 1440, or other currently accepted toxicity test methods (see OECD Guidelines 201, 202, 203, 207, and 208 or U.S. EPA 40FR797A, as well as other documents such as *A New Manual for Conducting Microtox Test with the Model 500 Analyzer*[2] the work on cyst-based toxicity tests by Centeno, et al[3]). The mixtures can also be used for further soil contact biodegradation testing.

1.2 This practice provides for storage and drying of the mixtures obtained at the end of the test methods for determination of the biodegradability of plastics under controlled composting conditions (Test Method D 5338), and under high-solids anaerobic digestion (Test Method D 5511). The mixtures contain the biologically decomposed residuals from solid waste and from the plastic materials. For the blanks, the residuals will be derived only from the biologically decomposed solid waste. In the event that a particular sample does not pass the toxicity test, chemical characterization of the degradation products can be performed on the sample to determine the source of the toxicity. Description of the performance of these analyses is beyond the scope of this practice.

1.3 There is no ISO standard that is equivalent to this practice.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 883 Terminology Relating to Plastics[4]

D 3987 Test Method for Shake Extraction of Solid Waste with Water[5]

D 4229 Method for Conducting Static Acute Toxicity Tests on Waste-Waters with Daphnia[6]

D 5152 Practice for Water Extraction of Residual Solids from Degraded Plastics for Toxicity Testing[7]

D 5338 Test Method for Determining Aerobic Biodegradation of Plastic Materials Under Controlled Composting Conditions[8]

D 5511 Test Method for Determining Anaerobic Biodegradation of Plastic Materials Under High-Solids Anaerobic-Digestion Conditions[8]

E 729 Guide for Conducting Acute Tests on Test Materials with Fishes, Macroinvertebrates, and Amphibians[9]

E 1192 Guide for Conducting Acute Toxicity Test on Aqueous Ambient Samples and Effluents with Fishes, Macroinvertebrates, and Amphibians[9]

E 1295 Guide for Conducting Three Brood, Renewal Toxicity Tests with Ceriodaphnia Dubia[9]

E 1440 Guide for an Acute Toxicity Test with the Rotifer Brachionus[9]

2.2 *OECD Guidelines:*[10]

OECD Guideline 201 Alga, Growth Inhibition Test

[1] This practice is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.96 on Environmentally Degradable Plastics.

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[2] Microbics Corporation, Carlsbad, CA.

[3] Centeno, M. D., Brendonck, L., and Persoone, G., "Cyst-Based Toxicity Tests III: Development and Standardization of an Acute Toxicity Test with the Freshwater Anostraean Crustacean Streptocephalus probosciceus," *Progress in Standardization of Aquatic Toxicity Tests*, A. M. V. M. Soares and P. Calow, eds., Lewis Publishers, London, United Kingdom, pp. 37–56.

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

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

[6] Discontinued; see *1987 Annual Book of ASTM Standards*, Vol 11.04.

[7] Discontinued; see *1998 Annual Book of ASTM Standards*, Vol 08.03.

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

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

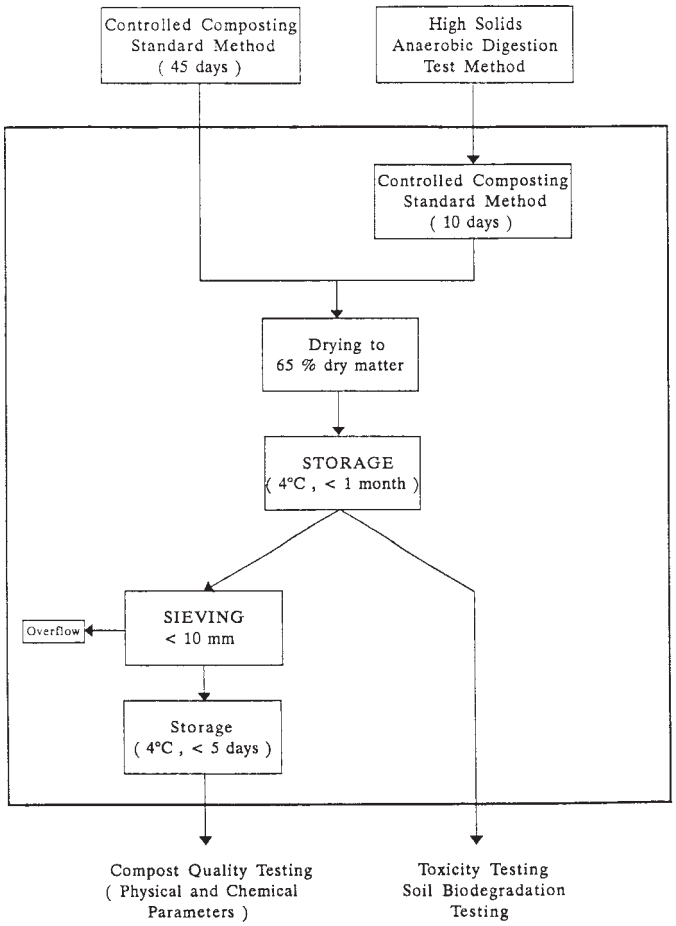

FIG. 1 Schematic Overview of Practice

OECD Guideline 202 Daphnia sp., 14-day Reproduction Test
OECD Guideline 203 Fish, Acute Toxicity Test
OECD Guideline 207 Earthworm, Acute Toxicity Tests
OECD Guideline 208 Terrestrial Plants, Growth Test

2.3 *Other Standards:*

U.S. EPA 40FR797A A Variety of Toxicity Testing Procedures[11]
APHA 2540E Fixed and Volatile Solids Ignited at 550°C[12]

3. Terminology

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

4. Significance and Use

4.1 Standard methods for determining the biodegradation of plastics simulating solid waste treatment systems have been developed. In addition to the rate and degree of biodegradation, the impact of the biologically decomposed plastics on the quality of the compost is important, in particular with regard to any toxicity or biodegradation that the residual plastics may exhibit when the final end-product, compost, is applied to the soil. Therefore, the effects of residual plastics on toxicity need to be measured as well as the impact on the compost quality, in order to evaluate the safe biological treatment through aerobic or anaerobic degradation.

4.1.1 This practice provides a way of preparing residual solids from tests simulating conditions of solid waste degradation for subsequent toxicity, biodegradation, or compost quality testing.

4.2 Several methods have been developed for the determination of toxicological effects. A distinction can be made between aquatic toxicity tests and terrestrial toxicity tests.

4.3 At the end of a composting process for municipal solid waste, a post-treatment is performed to transform the rough process compost into an upgraded market compost. This post-treatment consists primarily of a drying of the compost followed by a sieving over a 10-mm screen.

4.4 This practice provides for a standardized post-treatment at the end of a standard method for biodegradation under controlled composting conditions. The practice consists of drying, storage, and sieving to be followed by further compost quality testing. No sieving is applied to the dried residual solids for subsequent toxicity and soil biodegradation testing.

4.5 At the end of an anaerobic digestion process for municipal solids waste, a post-treatment is performed to transform the digested material into an upgraded compost. This post-treatment consists of a 10-day aerobic maturation and drying phase, followed by a screening over a 10-mm screen.

4.6 This practice provides for a standardized post-treatment at the end of the high solids anaerobic digestion method and consists of preparing a mixture of the digested residue with a compost, in order to submit the mixture to a ten-day controlled composting phase. The mixtures retrieved after the ten-day controlled composting steps are then treated in the same way as the mixtures retrieved from an aerobic composting process and prepared for subsequent toxicity testing by drying, storage, and sieving.

4.7 This practice is end-point specific as written, in that it is focused on a post-degradation analysis after the completion of a standard method. Sample preparation and storage as recommended herein allow for further qualitative and quantitative toxicological, chemical, and physical testing procedures that are beyond the scope of this practice. These procedures may be useful in the characterization of degradation products derived from biodegradable plastics. In case toxicity is observed, the material may be tested and prepared in the standard test methods in a concentration of 1 % (weight over weight) added to the inoculum or to a standardized fresh waste matrix and evaluated again for toxicity and compost quality testing.

4.8 *Limitations*—Because there is a wide variation in the construction and operation of composting and anaerobic digestion systems and because regulatory requirements vary, this practice is not intended to simulate the post-treatment and compost refining of any particular composting or digestion

[10] Available from Director of Information, OECD, 2 rue André Pascal, 75775 Paris Cedex 16, France.

[11] Available from Superintendent of Documents, U.S. Government Printing Office, Washington DC 20402.

[12] *Standard Methods for the Examination of Water and Wastewater (APHA)*, 17th Ed., available from American Public Health Association, 1740 Broadway, New York, NY 19919.

system. However, it is expected to resemble a common and simplified approach to the post-treatment and refining of the compost.

5. Apparatus

5.1 *Analytical Equipment*, for measuring dry solids (105°C).

5.2 *Suitable Services and Analytical Equipment*, for measuring the aerobic degradation of plastic materials under controlled composting conditions.

5.3 *Sieve*, with a mesh size of 10 mm.

5.4 *Precision Balance (±0.1 g), Analytical Balance (±0.1 mg), and Thermometer*.

6. Reagents

6.1 *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.[13] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Prepare as specified in the toxicity test to be used.

7. Procedure

7.1 At the end of the high-solids anaerobic digestion standard test method (Test Method D 5511), 500 to 1000 g of digested sample materials coming from each replicate are mixed with the compost inoculum, as described in Test Method D 5338, in a ratio of 1:1 on a wet-weight basis, and submitted subsequently to controlled composting for a period of ten days at a temperature of 58°C and a dry matter content between 45 and 60 %.

7.1.1 Biodegradation is followed in accordance with Test Method D 5338 by comparing additional CO_2 production from the material containing the digested plastics with the CO_2 production coming from the blank. The same is done for the replicates containing the positive reference.

7.2 After composting residual solids from high-solids anaerobic digestion as described in 7.1, or after controlled composting as described in Test Method D 5338 at a uniform temperature of 58°C, remove and mix the contents of each replicate compost vessel carefully. Continue mixing until the contents are mixed thoroughly. The resulting homogeneous mixture should have a uniform moisture content and appearance. Repeat the same procedure for positive reference and blank replicates.

7.3 Three subsamples are taken from the homogeneous mixture, on which the dry solids are determined according to APHA 2540E.

7.4 The rest of the mixture is dried at a temperature of 20 to 45°C until a dry solids content of 65 ± 2 % is reached. The dry solids content is determined after drying.

7.5 The dried mixtures can be stored for a maximum of four weeks at 4°C. The mixtures should be opened on a weekly basis in order to prevent slow accumulation of acids in the mixtures due to anaerobic conditions.

7.6 For the terrestrial toxicity tests and the determination of further biodegradation of the plastic materials in the soil, the final dried mixture at 65 % dry solids is used as is. For the aquatic toxicity tests, an extraction is performed according to Test Method D 3987.

7.7 For the compost quality analyses, the dried mixture is weighed and sieved for a period of 30 min over a screen of 10 mm, and the wet and dry weights are determined on the fraction retained by and the fraction passing through the screen. Sieving is accomplished by means of a laboratory sieving machine (sieve shaker plus horizontal amplitude). The fraction passing through the screen can be submitted for further appropriate chemical and physical analyses, such as water holding capacity, salt content, volatile solids, and other appropriate compost parameters.

8. Report

8.1 Report the following information:

8.1.1 Source and type of sample.

8.1.2 Final results (final percentage of aerobic biodegradation and graphic display) of the previous standard-controlled composting test (Test Method D 5338) that generated the composts to be processed according to this practice.

8.1.3 Final results of the previous standard high solids anaerobic digestion test (Test Method D 5511), with the final percentage of anaerobic degradation and biodegradation obtained in the ten-day aerobic controlled composting test (the total cumulative biodegradation with graphic display), which generated the composts to be processed according to this practice.

8.1.4 Dry solids content of the compost mixture at the start of the practice and the dry solids content of the mixture after drying.

8.1.5 Storage conditions and duration.

8.1.6 Wet-weight distribution between throughput and the fraction retained by sieving. Visual characteristics of the residual plastic (if any) before and after sieving.

9. Keywords

9.1 biodegradation; compost; composting; digestion; plastics; toxicity

[13] *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 *Analar 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.