Standard Test Method for Determining Biodegradability of Materials Exposed to Municipal Solid Waste Composting Conditions by Compost Respirometry[1]

1. Scope

1.1 This test method covers the biodegradation properties of a material by reproducibly exposing materials to conditions typical of municipal solid waste (MSW) composting. A material is composted under controlled conditions using a synthetic compost matrix and determining the acclimation time, cumulative oxygen uptake, cumulative carbon dioxide production, and percent of theoretical biodegradation over the period of the test. This test method does not establish the suitability of the composted product for any use.

1.2 The values stated in both inch-pound and SI units are to be regarded separately as the standard. The values given in parentheses are for information only.

1.3 *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]

D 513 Test Methods for Total and Dissolved Carbon Dioxide in Water

D 1129 Terminology Relating to Water

D 1293 Test Methods for pH of Water

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

2.2 *APHA-AWWA-WEF Standard Methods:*

2540G Total, Fixed, and Volatile Solids in Solid and Semisolid Samples[3]

3. Terminology

3.1 *Definitions*—Definitions of terms applying to this test method appear in Terminology D 1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *acclimation time*, *n*—the time required for the oxygen uptake to reach 10 % of the total measured cumulative oxygen uptake.[4]

3.2.2 *oxygen uptake*, *n*—the cumulative oxygen consumed by the organisms during the test.

3.2.3 *theoretical carbon dioxide production (ThCDP)*, *n*—the maximum carbon dioxide that can be produced by a material as calculated by the carbon content of the material.

3.2.4 *theoretical oxygen uptake (ThOU)*, *n*— the maximum oxygen consumption required to fully oxidize a material based on the elemental content of the material.

3.2.5 *virgin newsprint*—nonprinted newspaper roll stock.

4. Summary of Test Method

4.1 This test method consists of the following:

4.1.1 The samples are prepared by cutting or forming the material into the form it would most likely be seen in the waste stream. A theoretical maximum carbon dioxide production and oxygen uptake are determined from an elemental analysis.

4.1.2 An inoculum is obtained from a municipal MSW or yard waste compost facility. It is procured from a static pile that has been composting for at least two months.

4.1.3 The synthetic MSW is prepared from virgin newsprint, pine bark or wood chips, corn starch, corn oil, bovine casein, and urea. A buffer/dilution water is prepared from magnesium, calcium, iron and a phosphate buffer.

4.1.4 The test material, synthetic compost, inoculum, and dilution water are combined and placed in a highly insulated reactor which monitors oxygen consumption and temperature and captures all evolved carbon dioxide.

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[1] This test method is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.07 on Municipal Solid Waste.

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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 Public Health Assoc., 1015 15th Street, NW, Washington, DC 20005, Standard Methods for the Examination of Water and Waste Water, 18th ed., 1992.

[4] Tabak, Henry H. and Lewis, Ronald F., *CEC/OECD Ring Test of Respiration Method for Determination of Biodegradability*, U. S. Environmental Protection Agency, pp. 1–3.

4.1.5 The system is monitored, and oxygen uptake rates, temperature profiles, and total carbon dioxide produced are recorded.

4.1.6 The total oxygen uptake and carbon dioxide produced are compared with the theoretical values obtained from the elemental analysis, and a percent of biodegradation is generated. Possible negative effects of the material are evaluated by observing the acclimation time of the synthetic MSW and evaluating the oxygen uptake rate.

5. Significance and Use

5.1 As the crisis in solid waste continues to grow, MSW composting is increasingly being considered as one component in the overall solid waste management strategy. The volume reduction achieved by composting, combined with the production of a usable end product, is resulting in increasing numbers of municipalities analyzing and selecting MSW composting as an alternative to incineration or to reduce reliance on landfill disposal. This test method will help determine the effect of materials on the compost process and establish if the material can be properly disposed through solid waste composting facilities.[5]

5.2 This test method attempts to provide a simulation of the overall compost process while maintaining reproducibility. Exposing the test material with several other types of materials that are typically in MSW provides an environment which provides the key characteristics of composting: material not in a sole carbon source environment which allows co-metabolism, compost system is self heating, and provides a direct measurement of organism respiration.

6. Apparatus

6.1 *Compost Respirometry Apparatus (see* Fig. 1*)*:

6.1.1 A minimum of six reactors, 2 to 6-L volume, with the test material in triplicate and the controls in triplicate. The reactors should be surrounded with efficient insulation to minimize heat loss and be gastight. Insulation should be 8 cm of urethane foam or equivalent.

6.1.2 *Tubing*, with high resistance to gas permeation.

6.1.3 *Peristaltic Pump*, to control and maintain gas flow through each reactor.

6.1.4 *4-L Scrubber Vessel*, for each reactor fitted with a scrubber solution sampling port.

6.1.5 *Differential Pressure Switch*, for each reactor that actuates between 2 and 5 in. (51 and 127 mm) of water.

6.1.6 *Solenoid and Mass Flowmeter*, to control and measure the addition of pure (99.997 +) oxygen to system.

6.1.7 *Temperature Probe*, situated in the middle of the compost.

6.1.8 *Data Acquisition and Control System*, for the measurement of temperature and the control and measurement of the oxygen addition.

6.2 *Miscellaneous*:

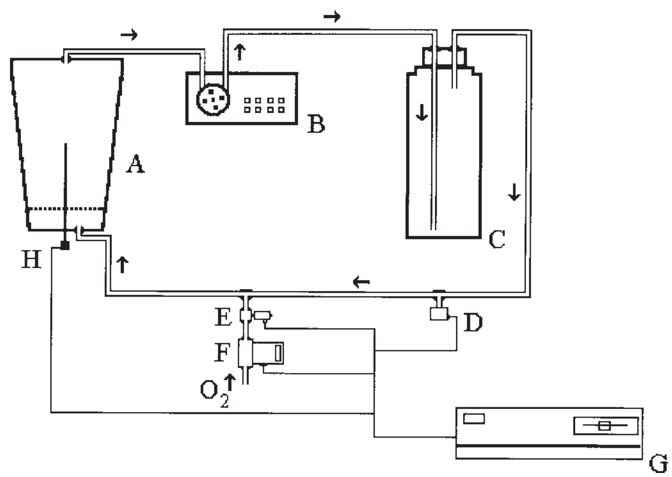
6.2.1 *Temperature Control Room*, or hood to maintain the external temperature of the apparatus at 40°C.

6.2.2 *Flow Meter*, to measure recirculation flow in each reactor (optional).

6.2.3 *Computer Control of Peristaltic Pump*, for automatic recirculation flow control (optional).

6.3 Suitable devices for the measurement of pH, dry solids (105°C), elemental analysis of material, carbon dioxide content of scrubbers, weight, and volume of the final compost material.

7. Test Materials



NOTE 1—The compost respirometer features a 4-L reactor vessel (A) insulated with 8 cm of urethane foam. The atmosphere is drawn through the reactor by a peristaltic pump (B) to maintain aeration. The effluent gases are passed through a 4-L scrubber vessel (C) containing 1.5 L of 5 *M* NaOH to remove any carbon dioxide from the effluent gas stream. Samples are drawn from this scrubber solution during the evaluation to determine the carbon dioxide released by the compost. As the microorganisms consume the oxygen in the system, a pressure drop occurs and is detected by a highly sensitive pressure switch (D). This signals the data acquisition and control system (G) and the oxygen is replaced with pure bottled oxygen by a solenoid (E) and the amount added is measured by a mass flowmeter (F). The gasses are then returned to the reactor. A thermocouple (H) is centered in the test reactor to monitor the temperature of the compost. The system is sealed to prevent interference from barometric fluctuations.

FIG. 1 Compost Respirometer Functional Diagram

7.1 The test materials can be in any form as long as it's dimensions do not exceed 3 by 3 by 12 cm. The test materials should be in the form that they would be seen in the waste stream. A representative sample must be obtained by using appropriate ASTM methods or other documented method.

7.2 Analyze the test materials for carbon, hydrogen, nitrogen, oxygen, phosphorus, sulfur, and any other elements that are suspected to be present at a level to effect oxygen uptake. The ThOU must be calculated for each material.

7.3 Calculate the ThCDP from the carbon content of the test material.

7.4 The nitrogen content of the synthetic MSW should be adjusted if the C/N ratio is greater than 40:1. This is accomplished by adjusting the urea content of the synthetic MSW. The synthetic MSW has adequate nitrogen to support the addition of up to 35 g of carbon before the ratio exceeds 40:1.

[5] Biocycle: Journal of Waste Recycling Staff, eds., *The Biocycle Guide to Composting Municipal Wastes*, JG Press, Inc., 1989.

If the urea content is adjusted, all reactors including controls must contain the same concentration of urea.

8. Reagents and Materials

8.1 *Scrubber Solution*, containing 3.25 *N* NaOH in distilled water. Store in a gas-tight plastic container. Add 30 mg of phenolphthalein to the solution to indicate scrubber exhaustion.

8.2 *Dilution/Buffer Solution*, containing the following:

Chemical	Purpose	Concentration, g/L
KH_2PO_4	phosphate buffer	6.8
$Na_2HPO_4 \cdot 7H_2O$	phosphate buffer	55.6
$MgSO_4 \cdot 7H_2O$	nutrient	0.0225
$CaCl_2$	nutrient	0.0275
$FeCl_3 \cdot 6H_2O$	nutrient	0.00025

8.3 *Synthetic Municipal Solid Waste*, containing the following:[6,7]

Constituent	Chemical Used	Dry Weight, %
Cellulosics	shredded, virgin newsprint	41
Inerts	pine bark or wood chips	39
Carbohydrates	corn starch	5.2
Lipids	corn oil	5.4
Proteins	bovine casein	2.0
Organic nitrogen	urea	1.4
Buffer/Nutrient	as listed	5.8

8.4 *Polyethylene*, or another nonbiodegradable material is the negative control material. It should be in the same form as the test materials to provide the same physical conditions in all reactors. The synthetic MSW acts as a positive control to verify the viability of the inoculum, see 13.4 for requirements.

9. Hazards

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

9.2 This test method does not address all of the health and safety issues related to it's use. It is the responsibility of the user to establish appropriate safety measures.

9.3 High-purity high-pressure gases can be dangerous if not handled correctly. Follow all safety precautions and monitor the system often to ensure proper operation.

10. Inoculum

10.1 The inoculum should be obtained from MSW or yard waste that has properly composted for two to four months. The compost should be screened with a <3-mm screen.

10.2 The compost can be stored at room temperature for up to 48 h before use. It should not be allowed to dry.

11. Procedure

11.1 This procedure is for twelve 4-L reactors with 4-L scrubber vessels. Other configurations will need to adjust weights and volumes to maintain proportional liquid:solid ratios of components.

11.2 *Synthetic Municipal Solid Waste*:

11.2.1 *Dilution Water*— Weigh out the ingredients for 3600 mL of dilution water. This will make enough dilution water for 13 reactors:

Compound	Quantity per Reactor	Per 3600 mL
KH_2PO_4	1.87 g	24.5 g
$Na_2HPO_4 \cdot 7H_2O$	15.29 g	200 g
$MgSO_4$	0.003 g	0.039 g
$CaCl_2 2H_2O$	0.0076 g	0.099 g
$FeCl_3$	0.0002 g	0.003 g
Urea	4.0 g	52 g

11.2.2 *Dry Ingredient Preparation*:

11.2.2.1 Weigh out 120 g of shredded virgin newsprint and place in a sealed plastic bag. Mark each bag with the actual weight of newsprint.

11.2.2.2 Weigh out 115 g of 2.5 by 2.5 by 0.6 cm (approximately) fresh wood chips or pine bark into a 1-L plastic beaker with 15.1 g of corn starch and 5.95 g of bovine casein. Repeat until twelve sets of dry ingredients are prepared.

11.2.3 *Inoculum Preparation*:

11.2.3.1 Obtain approximately 1 kg of mature compost from a municipal MSW or yard waste compost facility.

11.2.3.2 Screen compost with 3-mm wire mesh screen and retain the <3-mm portion that is used as the inoculum.

11.2.3.3 Weigh out 12 g of inoculum into each of 12 weighing trays.

11.3 *Sample Preparation*:

11.3.1 Determine the dry solids of the test materials and obtain the elemental analysis. Calculate the amount of test material required to provide 50 g of ThOU.

11.3.2 Prepare the control samples by using polyethylene as the material and form or cut it into the same physical size and shape as the test material.

11.4 *Reactor Loading*:

11.4.1 Mix the shredded newsprint and the dry ingredients and add the dilution water. Thoroughly mix until there are no clumps of paper or chemical.

11.4.2 Add 15.8 g of corn oil by dispensing directly to the mixture. Mix the ingredients until the oil is evenly distributed.

11.4.3 Add control or test product and mix until products are evenly distributed.

11.4.4 Add inoculum and thoroughly mix into compost. Load into reactor, taking care not to compact the compost mixture.

11.5 *Scrubber Preparation*:

11.5.1 Fill the scrubber vessels with 1.5 L of 3.25 *N* NaOH solution.

11.5.2 Add 30 mg of phenolphthalein indicator to the scrubber solution.

11.5.3 Seal the scrubber vessel to minimize atmospheric carbon dioxide absorption.

11.6 *Run Startup*:

11.6.1 Assemble test reactor system and allow system to reach ambient temperature and stabilize.

11.6.2 Sample the scrubber vessels and analyze for carbon dioxide content by using Test Method D 513 or other suitable method.

[6] Clark, C. S., et al., "Laboratory Scale Composting: Techniques," *Journal of the Environmental Engineering Division-ASCE*, October, 1977.

[7] U. S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, *Characterization of Municipal Solid Waste in the United States, 1960 to 2000 (update 1988)*, EPA/530-SW-88-033, March 1988.

11.6.3 Set the recirculation pump to 100 mL/min or at a level that will allow adequate aeration without excessive heat loss. If the test reactor temperatures exceed 60°C increase the aeration to keep the reactor temperature below 65°C.

11.7 *Run Operation*:

11.7.1 The reactors are operated for a period of 45 days. If the environment control option is implemented, maintain ambient temperature at 40°C.

11.7.2 Set the computer data acquisition system to collect and store the temperature and oxygen uptake data a minimum of once every 2 h.

11.7.3 To prevent channeling, shake the reactors vigorously once a week.

11.7.4 There must be NO leaks in the system. The tubing and fittings must be checked thoroughly at the beginning of the test and after each shaking with a soap solution. Any leaks must be corrected immediately. The test apparatus should be monitored daily to ensure proper operation.

11.8 *End of Run*:

11.8.1 Sample the scrubber vessels in duplicate and analyze for carbon dioxide content.

11.8.2 Determine the total weight of the compost material and determine the dry solids concentration.

11.8.3 Measure the pH by diluting 10 g of sample with 50 mL of distilled water (refer to Test Methods D 1293). Shake and measure pH immediately. If the pH of the compost is below 7, determine the volatile fatty acids (VFA) content of the compost (see Practice D 2908). If the VFA is >2g/kg, the reactors have soured and the results are invalid.

12. Calculation

12.1 Calculate the theoretical oxygen uptake from the percent by weight of each of the elements:

$$ThOU\ (\text{mg } O_2/\text{mg}) = C/37.5 + H/12.5 + N/29.2 + P/48.4 + S/66.7 - O/100 \quad (1)$$

12.2 The theoretical carbon dioxide production is also determined from the elemental analysis and is calculated as follows:

$$ThCDP\ (\text{mg } CO_2/\text{mg}) = (\%\ \text{by } weight\ carbon)\ (3.667) \quad (2)$$

12.3 Calculate the oxygen uptake due to the test material by subtracting the mean of the control reactors from each of the test reactors at each data point.

12.4 Calculate the total carbon dioxide produced by subtracting the mean of the control analytical results from each of the test reactors.

12.5 Calculate the percent of theoretical of the total oxygen uptake and carbon dioxide by dividing the measured totals by the calculated ThOU or ThCDP and multiplying by 100.

12.6 The acclimation time for the test material and compost matrix can be determined at the point which the oxygen uptake exceeded 10 % of the total oxygen uptake for that reactor.

12.7 If the test materials oxygen uptake curve shows a negative slope at the beginning of the curve, the test material acclimation time is calculated at the point were the slope of the oxygen uptake curve becomes positive for a minimum of 24 h.

13. Interpretation of Results

13.1 The acclimation time can provide valuable information on the toxicity or inhibition effects of the test material.

13.2 The percent of theoretical oxygen uptake is an excellent measure of the biodegradability of the test material since it represents the mineralization of carbon in addition to all other oxidative reactions.

13.3 The percent of theoretical carbon dioxide production can be used to measure biodegradability (with limitations) and can be used to correlate results with other carbon dioxide based test methods.

13.4 To ensure an active and viable inoculum, the total oxygen uptake for the control reactors should exceed 80 g. If this is not observed over the 45 days then the test must be regarded as invalid and should be repeated with new inoculum.

14. Report

14.1 Report the following information and data with standard deviations and 95 % confidence intervals when appropriate:

14.1.1 Information on source, dry solids, storage conditions, and age of the inoculum,

14.1.2 The ThOU and ThCDP of test materials with elemental analysis results,

14.1.3 Test material physical characterization including size, shape, weight, dry weight, initial and final reactor observations, and sampling technique used,

14.1.4 Percent of ThOU and ThCDP for each test material,

14.1.5 Total oxygen uptake and oxygen uptake versus time plots for entire reactors and test materials only. Rate plots if applicable,

14.1.6 Acclimation times for test materials,

14.1.7 Total carbon dioxide produced and carbon dioxide versus time plots if multiple samples were analyzed during the test,

14.1.8 Temperature plots of each reactor,

14.1.9 Total weight loss of reactor contents and dry weight of end reactor contents,

14.1.10 Final pH of compost, investigative results if pH is below seven, and

14.1.11 Full description of test apparatus and documentation of any variations from this test method.

15. Precision and Bias

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

15.2 Typical oxygen uptake and temperature plots are presented in Fig. 2 and Fig. 3. Within-test reproducibility from three different test runs are presented in Table 1. Run 1 is the control reactors with pine bark, Run 2 is with fresh wood chips, and Run 3 is with previously used wood chips.

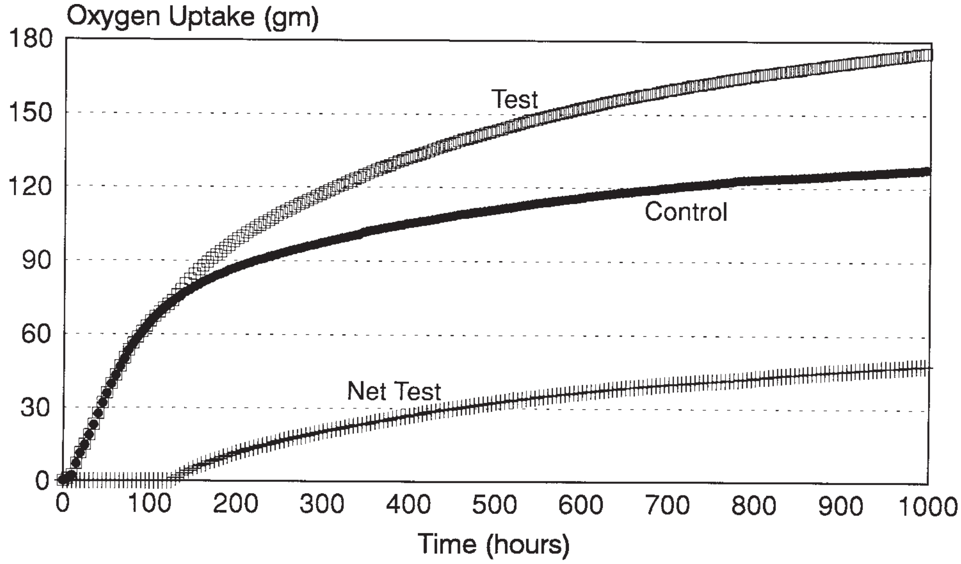


FIG. 2 Typical Control Reactor, Test Reactor, and Net Test Reactor

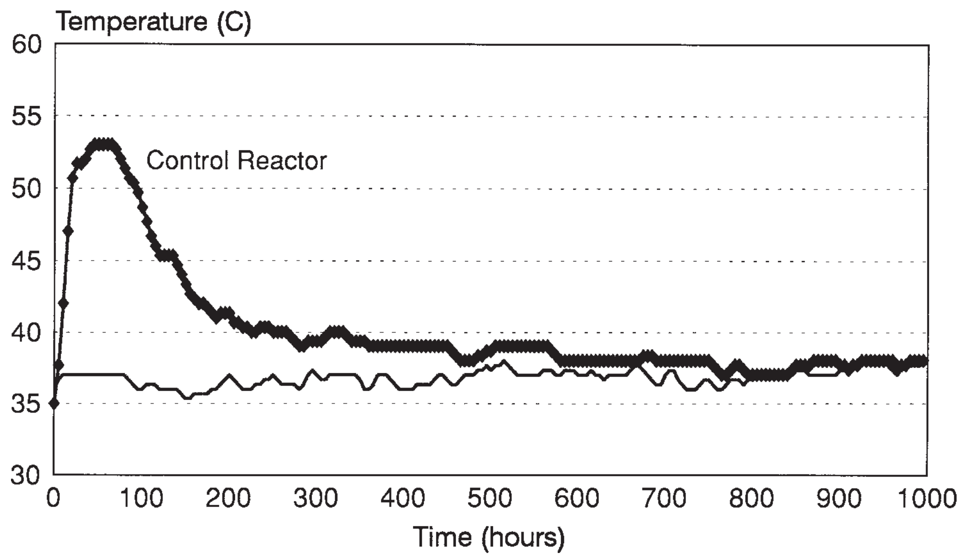


FIG. 3 Typical Temperature Plots, Ambient Set at 37°C

TABLE 1 Within-Test Reproducibility for Three Synthetic MSW Compositions

	O_2 Uptake, g	Standard Deviation, %	CO_2 Produced, g	Standard Deviation,%
Run 1, pine bark	131	2.4	111	6.2
Run 2, new chips	134	0.82	115	4.0
Run 3, used chips	115	0.87	91.4	3.7