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## 05.08 RESPIROMETRY

### DISCLAIMERS

- (1) The methodologies described in TMECC do not purport to address all safety concerns associated with their use. It is the responsibility of the user of these methods to establish appropriate safety and health practices, and to determine the applicability of regulatory limitations prior to their use.
- (2) All methods and sampling protocols provided in TMECC are subject to revision and update to correct any errors or omissions, and to accommodate new widely accepted advances in techniques and methods. Please report omissions and errors to the U.S. Composting Council Research and Education Foundation. An on-line submission form and instructions are provided on the TMECC web site, <http://www.tmecc.org>.
- (3) Process alternatives, trade names, or commercial products as mentioned in TMECC are only examples and are not endorsed or recommended by the U.S. Department of Agriculture or the U.S. Composting Council Research and Education Foundation. Alternatives may exist or may be developed.

### 1. Scope

1.1 This test covers the indirect determination of microbial activity in compost by measuring respiration rates in a compost sample. It is used as an indicator of compost stability.

1.1.1 *Method 05.08-A Specific Oxygen Uptake Rate (SOUR).*

1.1.2 *Method 05.08-B Carbon Dioxide Evolution Rate.*

1.1.3 *Method 05.08-C In-Situ Oxygen Refresh Rate*—Modified after USAEC Report ENAEC-TS-CR-93208.

1.1.4 *Method 05.08-D Dewar Self-Heating Test.*

1.1.5 *Method 05.08-E Solvita<sup>®</sup> Maturity Index.*

1.1.6 *Method 05.08-F Biologically Available Carbon.*

1.2 Values stated in SI units are to be regarded as the standard. Values given in parentheses are provided for information only.

### 2. Referenced Documents

#### 2.1 TMECC:

Method 02.01-A Compost Sampling Principles and Practices

Method 02.01-B Selection of Sampling Locations for Windrows and Piles

Method 02.02-C Man Made Inert Removal and Classification

Method 03.02-C Unmilled Material Ignited at 550°C with Inerts Removal.

Method 03.09 Total Solids and Moisture

Method 04.01 Organic Carbon

Method 05.03-A Field Assessment of Compost Color and Odor

Method 05.07-A Loss on Ignition Organic Matter

#### 2.2 Apparatus Manuals:

Cole-Parmer Instrument Company Inc., 7425 N. Oak Park Avenue, Miles, IL 60714 USA; URL: <http://www.coleparmer.com>.

YSI Incorporated, 1725 Brannum Lane, Yellow Springs, OH 45387 USA; URL: <http://www.yxi.com>.

Columbus Instruments (MicroOxymax), 950 North Hague Avenue; Columbus, OH 43204-2121 USA; URL: <http://www.colinst.com>.

#### 2.3 Other References:

Carlsbaek, M. and M. Broegger. 1999. Danish soil improvement: new standardized product sheet for compost. Report to Danish EPA. *In* Proceedings of ORBIT99 Organic Recovery & Biological Treatment Symposium. Weimar, Germany.

Iannotti, D. A., M. E. Grebus, B. L. Toth, L. V. Madden, and H. A. J. Hoitink. 1994. Oxygen Respirometry to Assess Stability and Maturity of Composted Municipal Solid Waste. *J. Environ. Qual.* 23:1177-1183.

Iannotti, D. A., T. Pang, B. L. Toth, D. L. Elwell, H. M. Keener and H. A. J. Hoitink. 1993. A Quantitative Respirometric Method for Monitoring Compost Stability. *Compost Science & Utilization* 1:52-65.

Jourdan, B. 1982. Standardizing Selected Methods for Determining the Degree of Maturity Decomposition of Municipal Composts. Abfall Wirtschaft Forschungsbericht. Univ. Stuttgart.

Standard Methods for the Examination of Water and Wastewater. 1992. Part 2000, Physical and Aggregate Properties. Method 2710 A. Oxygen-Consumption Rate.

Zibilske, L.M. 1994. Carbon Mineralization. pp. 835-863. In R.W. Weaver (ed.). Methods of Soil Analysis. Part 2. SSSA Book Series 5. SSSA, Madison, WI.

### 3. Terminology

3.1 *aerobic, adj*—Living or occurring only in the presence of oxygen, (e.g., aerobic bacteria).

3.2 *ammonia (NH<sub>3</sub>), n*—A volatile gas that contains nitrogen, ammonia gas.

3.3 *anaerobic, adj*—Living or occurring only in the absence of oxygen, (e.g., anaerobic bacteria).

3.4 *biodegradable volatile solids, n*—The biodegradable portion of total solids that volatilizes to gasses when a compost or feedstock is combusted at 550°C in the presence of excess air, % g.g<sup>-1</sup>.

3.5 *biologically available carbon (BAC)*—CO<sub>2</sub>-C as a product of microbial respiration where carbon is the limiting factor of a nutrient-enriched, and physically and chemically optimized compost sample matrix. A parameter proposed to emulate managed agricultural soil conditions.

3.6 *carbon-dioxide evolution, n*—The amount of CO<sub>2</sub> gas generated from the decomposition of organic matter during composting and detected in the headspace as described in Methods 05.08-C and 05.08-E. Determinations of the rate of decomposition as indicated by CO<sub>2</sub> evolution from a compost sample is a reliable means to assess compost stability and one of the indicators of compost maturity. Like other bioassay methods, respirometry can be used as a screening tool to indirectly assess the relative phytotoxicity of compost products.

3.7 *decomposition, n*—*Biological*, The act or result of decomposing; disintegration and breakdown or decay of organic materials into simpler compounds. *Chemistry*, Separation into constituents by chemical reaction.

3.8 *Dewar vessel*—a super-insulated vessel, invented by Sir James Dewar in 1893. The vacuum lined vessel was intended to keep cool (or hot) materials in a stable state. It was Bernd Jourdan (1982), working at the Institute of Wastewater Management of the University of Stuttgart who first applied the vessel to evaluating compost maturity and self-heating.

3.9 *facultative anaerobes, adj*—Bacteria that are capable of functioning with or without oxygen.

3.10 *maturity index, n*—A rating system devised to categorize compost relative to aging; it is based upon three or more relevant parameters as determined from compost analytical data, (e.g., carbon:nitrogen ratio; respiration rate; and ammonium:nitrate ratio, etc.).

3.11 *respiration, n*—An energy generating process by which an inorganic molecule such as O<sub>2</sub>, NO<sub>3</sub>, SO<sub>4</sub> or CO<sub>2</sub> is reduced through a series of metabolic steps to form water (H<sub>2</sub>O), diatomic nitrogen (N<sub>2</sub>), hydrogen sulphide (H<sub>2</sub>S) or methane (CH<sub>4</sub>). The reducing agent or substance that is first oxidized can be either an organic, (e.g., glucose), or inorganic, (e.g., NH<sub>4</sub>Cl), compound. This process occurs within the mitochondria of living cells and in various microorganisms. At the mitochondria of living cells and in heterotrophic microorganisms organic molecules are the energy source and O<sub>2</sub> is reduced. Anaerobic heterotrophs use organic molecules as their energy source, but reduce nitrate and sulfate. Aerobic autotrophs use inorganic molecules as their energy source and reduce O<sub>2</sub>.

3.12 *Solvita<sup>®</sup> Maturity Index, n*—An index that incorporates two test parameters (NH<sub>3</sub> and CO<sub>2</sub>) to yield color-coded test results. The test ranks compost on an index scale [1-8] of increasing compost maturity, i.e., a value of 8 indicates that the compost is resistant to further decomposition, biologically stable, and free of ammonia which can be toxic to plant growth.

3.13 *stability index, n*—the level of microbial activity in a sample of compost as determined by a respiration test; assumes a balanced nutrient status that favors microbial activity and the absence of toxins or other compounds that inhibit microbial respiration.

### 4. Summary of Test Methods

4.1 Although O<sub>2</sub> consumption and CO<sub>2</sub> evolution are related, the measurements are not always equivalent. Not all biological activity results in the complete mineralization of carbon to CO<sub>2</sub>. Oxygen consumption rates may approximate aerobic biological activity more closely than CO<sub>2</sub> evolution rates.

4.2 Apparatus for CO<sub>2</sub> measurements are generally less expensive than those needed for measuring O<sub>2</sub>. Additionally, CO<sub>2</sub> measurements are precise and simple, whereas O<sub>2</sub> consumption measurements are tedious and precision across replicates is difficult to maintain.

4.3 *Method 05.08-A Specific Oxygen Uptake Rate*—The rate of O<sub>2</sub> consumption is quantitatively measured using manometric and electrolytic respirometers, by measuring changes in O<sub>2</sub> concentrations with gas

chromatography or O<sub>2</sub> electrodes (Zibilske, 1994). The method for measuring changes in headspace O<sub>2</sub> concentrations with an O<sub>2</sub> electrode is described in this section.

4.3.1 The relative O<sub>2</sub> concentration in the head space of a closed flask containing a moist compost sample of known volume and mass, at known temperature and barometric pressure is monitored. The O<sub>2</sub> consumption rate per day is determined and reported relative to the total solids and organic matter contents of the material tested.

4.3.2 Details of the method are given by Ianotti et al., (1993 and 1994). Modifications of the published method (Department of Soil, Water, and Climate - Research Analytical Laboratory, University of Minnesota, St. Paul) are included to compensate for different types and conditions of MSW composts analyzed by that laboratory. All modifications and deviations from the published method are noted.

4.4 *Method 05.08-B Carbon Dioxide Evolution Rate*—The amount of CO<sub>2</sub> released biologically from a compost sample as a result of standardized incubation is reported per unit of volume or weight. This test is used to estimate the relative stability (biological activity) and maturity index of compost.

4.4.1 CO<sub>2</sub> evolution is an index of biological activity. Rates are measured in the headspace gas of static or dynamic systems with gas chromatography, infrared spectroscopy and alkali trapping and analysis, (e.g., manometric, titrimetric, conductimetric, or infrared spectrometric; Zibilske, 1994). The method for measuring CO<sub>2</sub> evolution rates in static chambers with alkali trapping and titration is described in this section.

4.4.2 Microorganisms utilize O<sub>2</sub> and generate CO<sub>2</sub> and water vapor during aerobic decomposition of organic matter. Microorganisms respire at high rates in biologically unstable compost and consume more O<sub>2</sub> and generate more CO<sub>2</sub> and water vapor than in more stable composts.

4.4.3 During anaerobic decomposition of feedstock materials, CO<sub>2</sub> and methane, CH<sub>4</sub>, are generated.

4.5 *Method 05.08-C In-Situ Oxygen Refresh Rate*—Managed compost piles are turned or aerated to replenish O<sub>2</sub> that is consumed by microorganisms during decomposition of organic matter. Immediately after turning, or other aeration activity, one or more O<sub>2</sub> probes are inserted into the pile at various depths, and O<sub>2</sub> concentration is recorded at 10 minute intervals until the values stabilize. Results are expressed as percentage O<sub>2</sub> per interval of time.

4.6 *Method 05.08-D Dewar Self-Heating Test*—A standardized procedure used to measure self-heating as

an indicator of biological activity. The difference in the maximum temperature produced by a sample of compost incubated for 10 days in a special, calibrated vessel relative to ambient temperature is measured.

4.7 *Method 05.08-E Solvita<sup>®</sup> Maturity Index*—A semi-quantitative (scaling) procedure used to determine carbon-dioxide (CO<sub>2</sub>) and ammonia (NH<sub>3</sub>) release into the closed headspace above a volumetric compost sample. The test provides a rapid and accurate determination of compost maturity.

4.7.1 Two determinations are performed simultaneously on one sample during a 4-h test period. The relative concentrations of evolved CO<sub>2</sub> and NH<sub>3</sub> are expressed on two corresponding color-indicator paddles. The color-coded paddles are pre-calibrated for a range of CO<sub>2</sub> evolution rates from approximately 2 through 30 mg CO<sub>2</sub>-C per g OM per day, and a range of NH<sub>3</sub> concentrations equivalent to 200 through 20,000 mg of NH<sub>3</sub>-N + NH<sub>4</sub>-N per kg of compost (dw basis).

4.7.2 Color changes occur during the 4-h test period and express the relative concentrations of CO<sub>2</sub> and NH<sub>3</sub> in the compost sample. The *Solvita<sup>®</sup> Maturity Index* is derived from results of both tests and normally increases as both the CO<sub>2</sub> rate and NH<sub>3</sub> levels decline.

4.8 *Method 05.08-F Biologically Available Carbon*—A compost sample is prepared by optimizing moisture, pH, porosity, nutrients and temperature. The prepared sample is incubated for three days to reestablish a microbial community. One measure of CO<sub>2</sub>-C is made in the headspace of the sample vessel to determine BAC-Respiration.

## 5. Significance and Use

5.1 *Methods 05.08-A and 05.08-B*—Respirometry is the measurement of CO<sub>2</sub> evolved or O<sub>2</sub> consumed by heterotrophic microorganisms within the compost and provides an estimate of biological activity of a composted material. Oxygen consumption during composting is influenced primarily by the rate of aerobic biological activity. Since aerobic activity is a function of compost stability, respiration rates are also related to compost stability.

5.1.1 Microorganisms utilize O<sub>2</sub> and generate CO<sub>2</sub> and water vapor during aerobic decomposition of organic matter. Microorganisms respire at high rates in biologically unstable compost and consume more O<sub>2</sub> and generate more CO<sub>2</sub> and water vapor than in more stable composts.

5.1.2 During anaerobic decomposition of feedstock materials, CO<sub>2</sub> and CH<sub>4</sub>, methane, are generated.

**5.2 Method 05.08-C In-Situ Oxygen Refresh Rate**—This test is used to monitor or evaluate the relative aerobic status of compost under field conditions.

**5.3 Method 05.08-D Dewar Self-Heating Test**—The test was first introduced in Europe in 1982 by Jourdan and recently re-evaluated. Numerous workers have reported investigations on compost maturity and the heating traits of composts. The Dewar self-heating method was adopted as an official standard for stability by the German Department of the Environment in 1984 as a follow up to the 1982 Sewage Sludge Order.

5.3.1 The self-heating test based on Dewar flask measurement has merit as a general technique to evaluate compost stability and maturity, provided the general conditions of the test and the specific equipment are applied. The method may be utilized by producers under field conditions where a relatively stable room temperature of 20-25°C (but no more than 25°C) can be maintained around the vessel. In the laboratory, the Dewar method aids researchers in understanding the differences in idealized laboratory technique versus field observations. Teachers and environmental monitoring programs have found the test useful to demonstrate principles of compost aging. Because the results are expressed as temperature, they are easily understood and accepted by users and consumers.

5.3.2 The Dewar method is simple to use, applicable to the majority of composts produced, and only requires a small, standardized vessel and min/max thermometer.

5.3.3 The Dewar test integrates a number of factors present in normal composts and can reflect well with field observations about the stability status of compost. It does not provide the same type of data as the more precise laboratory respirometry procedures, but, like all respiration methods, (Dewar self-heating, CO<sub>2</sub>-evolution, O<sub>2</sub> consumption), it gives a relative indication of the biological activity status of the compost as it pertains to biological stability.

**5.4 Method 05.08-E Solvita<sup>®</sup> Maturity Index**—An indexing system devised to rank compost maturity by indirectly measuring biological activity, or respiration, and chemical stability. The test is used to evaluate unknown compost products to help verify marketing claims for compost product shipments.

**5.5 Method 05.08-F Biologically Available Carbon**—Optimization of all edaphic parameters, except carbon, promotes the highest possible respiration rate, where carbon remains the only limiting factor. This test determines whether the compost respiration process will reactivate after compost is introduced to agricultural conditions.

## **6. Interference and Limitations**

6.1 Many adiabatic factors affect the activity of microorganisms in composts and must be carefully maintained to obtain precise and accurate readings for stability. Respirometry tests require a balanced nutrient status that favors microbial activity and the absence of toxins or other compounds that inhibit microbial respiration.

6.1.1 Microbiological respiration depends on moisture relative to the WHC, rather than to moisture content based upon sample wet-weight.

6.1.2 Generally, test compost samples that have a moisture content below 35%, wet weight basis, will be biologically dormant and a respiration rate determination will be artificially low. Samples must be moistened to 70% to 85% of WHC (typically 45% to 50%, wet weight basis), and allowed to equilibrate for at least 24 h at a specified temperature as per the method, prior to analysis. Incubation for up to three days may be necessary with some compost samples.

6.1.3 Samples removed from high temperature zones in compost piles (55-65°C) harbor thermophilic microorganisms that may not be active at low, mesophilic temperatures (below 37°C). Compost samples must contain subsamples from various temperature zones of a pile and be tested at a temperature of 34°C to avoid temperature pitfalls. Iannotti et al. (1993, 1994) provides more details on this potential interference.

COMMENT—If the compost is still at high temperatures, then it may be a waste of money to test for stability if the goal is testing for compost quality. This may be important if testing for other purposes, i.e., tracking metabolic activity during the course of compost production.

NOTE 1—Testing compost for stability at 37°C may be selectively testing for organisms in the upper range of the mesophilic organisms and may not be indicative of what can happen in the soil after the compost is incorporated. It is very possible to get a misleading analysis of compost stability when testing at 37°C. It is suggested by leading compost stability researchers that pre-incubation at 25-28°C and testing at 34°C would be more reflective of the actual compost metabolic activity potential.

6.1.4 Samples that are over-moist, tightly packed in a sealed container and shipped at temperatures above 4°C, usually arrive in an anaerobic state that is unrepresentative of the sample source and are not suitable for analysis.

6.1.5 Toxic compounds and some heavy metals that occasionally contaminate compost can inhibit microbial respiration.

**6.2 Method 05.08-A Specific Oxygen Uptake Rate**—Saprophytic fungi may heavily colonize properly moistened samples. This condition is usually

associated with very high levels of  $\text{NH}_4$  ( $> 500 \text{ mg kg}^{-1}$ ) and unstable material. Fungal mycelium serve as a food source for bacteria and will induce a flush of bacterial activity during incubation and upon aeration. If the presence of these fungi is not diminished through incubation prior to respirometry measurements, respiration measures will indicate high  $\text{O}_2$  uptake rates. If this condition persists, the presence of mycelium must be noted in the laboratory report.

6.2.1 In addition to the other issues for Method 05.08-A, the presence of inert material in the sub-sample used to determine sample organic matter must be evaluated. It is recommended that an inert content estimate be made on a parallel sub-sample and that organic matter content (biological volatile solids) be estimated using a clean sample, free from inerts. Refer to *Method 02.02-C Man Made Inert Removal and Classification* for a detailed description of this step in sample preparation.

### 6.3 *Method 05.08-B Carbon Dioxide Evolution Rate:*

6.3.1 Depletion of  $\text{O}_2$  in the headspace of incubation vessels may result in decreased biological activity. This can be avoided with 4-L containers and approximately 25-g samples, except for very unstable composts.

6.3.2 Alkali trapping of  $\text{CO}_2$  in the headspace of the incubation container may reduce the partial pressure of  $\text{CO}_2$  enough to upset the carbonate equilibrium in the compost sample. For this reason, some of the measured  $\text{CO}_2$  may be derived from inorganic sources such as carbonates.

6.4 *Method 05.08-C In-Situ Oxygen Refresh Rate*—Frequent equipment re-calibration and cleaning is imperative to attain reliable readings. Standard gases of fixed  $\text{O}_2$  content may be blended to achieve 1, 5, 10 and 15%  $\text{O}_2$  for exact calibration trials. Apparatus not adapted with temperature compensation can provide unpredictable results.

6.5 *Method 05.08-D Dewar Self-Heating Test*—A compost sample passed through a 20-mm sieve improves test precision, but results in slightly higher temperatures.

6.5.1 Optimal compost sample moisture conditions must be maintained for successful application of the Dewar self-heating procedure. As with other respiration tests, if sample moisture is too low (~30%), or too high (~65%), the Dewar class maturity determination will result in false positive. Originally, the European procedure called for optimizing moisture by partial pre-drying and remoistening to a set point of ~30% moisture. This moisture level is too low for

compost with a high WHC and correspondingly low bulk density.

6.5.2 The optimal moisture to conduct a Dewar test often depends on porosity of the material. The less porous the compost material, the more air transfer and heating are limited. Since moisture absorbs heat, it is understandable that the lowest optimal amount of water will produce the highest heating in the Dewar test. With experience, specific users will evolve appropriate methods that give reliable results.

### 6.6 *Method 05.08-E Solvita® Maturity Index:*

6.6.1 *Volatile Fatty Acids*—High levels of volatile fatty acids (VFA) may interfere positively with Solvita® and negatively with other respiration tests. Compost samples that produce a Solvita® #1 [bright yellow] commonly contain high levels of VFA. The maximum interference observed for VFA-containing samples is approximately one color change for Solvita®, but must be separately determined for other respiration procedures.

6.6.2 *Immature Composts*—High levels of ammonia ( $\text{NH}_3$ ) in compost may lower the  $\text{CO}_2$ -evolution rate (interfere negatively) as indicated by Solvita®. This interference is factored out by the ammonia-test result of the second gel-paddle.

6.6.3 *Active Denitrification*—In certain cases of composts that are anaerobic or undergoing active denitrification, nitrous-oxide can be produced resulting in an off-coloring of the Solvita® gel. Such samples invariably give high test results for nitrite, a phytotoxic intermediate of nitrification or denitrification.

6.6.4 *Temperature*—The Solvita® test is normally run at room-temperature for 4 hours. If the test is run at temperatures outside this range (20-25°C), results should be read at more or less than four hours. Compost samples collected from active piles must be re-equilibrated at room temperature before testing is started.

6.6.5 *Shelf-life*—The Solvita® gel-pack can be stored for approximately one year. Shelf-life can be extended with refrigerated storage (4°C).

## 7. **Sample Handling**

7.1 *Methods 05.08-A and 05.08-B*—See methods for pre-incubation requirements.

7.1.1 *Sample Moisture Status*—The moisture level should be judged by the squeeze test at sampling. If the compost is too wet or too dry, it is advisable to postpone sample collection and to adjust pile moisture. Changes made to a sample after collection may unpredictably bias the test result.

7.1.1.1 To ensure test results that more accurately represent compost material stability, the sample must be in a condition where aerobic microbes flourish. If samples are too wet (potentially anaerobic) or too dry, they need to be brought to the proper moisture content (70% to 85% of WHC). For most samples this is between 40-50% moisture. Samples with a high bulk density, approximately  $0.75 \text{ g cm}^{-3}$ , and low organic matter are usually over-saturated at 40-50% moisture and require less water (30-40% moisture, wet weight basis). Conversely, samples with a low bulk density and a very high WHC may be too dry with only 40-50% moisture.

7.1.1.2 *Excessively Dry Samples*—Spread a  $600 \text{ cm}^3$  sample aliquot uniformly onto a clean plastic lab tray ( $18 \times 22 \text{ in.}$ ). Sprinkle with deionized water while thoroughly mixing by hand until moisture content of 70% to 85% of WHC is attained. Use caution to maintain a loose texture and avoid aggregating the compost test aliquot into clumps or balls. Unwanted aggregates form most easily when handling material that is too wet. Transfer two  $300 \text{ cm}^3$  aliquots of moistened material to two 1 quart vegetable Ziploc<sup>®</sup> brand plastic vegetable bags. Place the bags in a high humidity incubator set at  $34^\circ\text{C}$  overnight to continue moisture equilibration.

7.1.1.3 *Excessively Wet Samples*—A sample is too wet if water can be squeezed from a fist-full of material. This precautionary observation should be performed when the sample is received at the laboratory, before sample splitting, sieving or initial sample preparation. If too wet, spread a  $600 \text{ cm}^3$  sample aliquot uniformly on a tray and allow to dry until no free water is evident. If the sample is left in open air for extended periods (over night), the sample may become excessively dry and will require re-moistening. To prevent this problem, a perforated sheet of aluminum foil, paper or plastic may be used to cover the tray containing the excessively wet sample.

7.2 *Method 05.08-C In-Situ Oxygen Refresh Rate*—This test is initiated immediately after turning (windrow systems) or during a complete aeration cycle (closed, static systems).

7.3 *Method 05.08-D Dewar Self-Heating Test*—A well blended representative compost sample with a moisture content of 70-85% of WHC is cooled to ambient temperature ( $18\text{-}22^\circ\text{C}$ ). Sieving compost through a 20-mm sieve improves test precision, but results in slightly higher temperatures.

7.4 *Method 05.08-E Solvita<sup>®</sup> Maturity Index:*

7.4.1 *Composite Sample*—A well-blended composite sample representing the average of the whole pile to be tested (or any specified portion thereof) is gathered by collecting several sub-samples throughout the pile with a shovel or other sampling device. Homogeneous samples are most easily collected immediately after turning a pile. Large fragments such as wood chips and other bulking agents ( $> \frac{1}{2} \text{ in.}$ ) are too large for the Solvita<sup>®</sup> jar and should be removed or screened from the compost sample before testing.

7.4.2 *Sample Temperature*—Hot (thermophilic) samples must be cooled to room temperature before testing.

7.4.3 *Sample Moisture Content*—Optimal moisture is absolutely necessary. The moisture level is judged by the squeeze test at sampling. If the compost is too wet or too dry, it is advisable to postpone sample collection for the Solvita<sup>®</sup> test and to adjust pile moisture. Changes made to a sample after collection may unpredictably bias the Solvita<sup>®</sup> test result.

NOTE 3—A squeeze test is performed with a handful of compost. A moist sample will clump when tightly squeezed. A sample with optimal moisture will feel wet, but not produce free water. A sample that is too dry is dusty and will not clump with hard squeezing.

7.5 *Method 05.08-F Biologically Available Carbon*—Store at  $4^\circ\text{C}$  for no more than three days until tested. Samples should be prepared for analysis upon receipt. See method.

<b>Test Method:</b> Respirometry: SOUR: Specific Oxygen Uptake Rate						<b>Units:</b> mg O <sub>2</sub> g <sup>-1</sup> (TS, OM) d <sup>-1</sup>		
<b>Test Method Applications</b>								
<b>Process Management</b>						<b>Product Attributes</b>		
<i>Step 1:</i> Feedstock Recovery	<i>Step 2:</i> Feedstock Preparation	<i>Step 3:</i> Composting	<i>Step 4:</i> Odor Treatment	<i>Step 5:</i> Compost Curing	<i>Step 6:</i> Compost Screening and Refining	<i>Step 7:</i> Compost Storing and Packaging	<i>Safety Standards</i>	<i>Market Attributes</i>
		05.08-A		05.08-A		05.08-A		05.08-A

## 05.08-A SOUR: SPECIFIC OXYGEN UPTAKE RATE

LOOK—Interference and Limitations, and Sampling Handling issues are presented as part of the introduction to this section.

COMMENT—Automated systems used in place of the apparatus described in this section provide significantly lower outcomes for moderate to highly active compost samples, (e.g., Columbus Instruments describes a system, MicroOxymax, at <http://www.colinst.com>, 950 North Hague Avenue; Columbus, OH 43204-2121 USA).

### 8. Apparatus for Method A

8.1 *Oxygen meter*—(e.g., YSI oxygen meter, digital model no. 58, or equal).

8.2 *Oxygen sensor*—(e.g., YSI oxygen probes, model no. 5718, or equal).

8.3 *Data logger*—(e.g., A/D conversion board for PC computer, No. G-08109-25; software, No. G-08109-32. Cole-Parmer Instruments, or equal).

8.4 *Computer*—IBM PC-XT, minimum.

8.5 *Incubator*—for setting at 34°C, with humidity control system.

8.6 *Water bath*—set at 34°C, with cover.

8.7 *Vaporizer-humidifier*—cool, (e.g., Hanksraft No. 240), if incubator is not equipped with humidity control system.

8.8 *Erlenmeyer flask*—1-L, fitted with a two-hole stopper and one short glass delivery tube, and a fritted sparger tube.

8.9 *Erlenmeyer flasks*—500-mL (one per sample).

8.10 *Evaporation dish*—borosilicate glass beakers, 100-mL and 250-mL (per sample).

8.11 *Pinch clamps*—for 0.94 cm (<sup>3</sup>/<sub>8</sub> in.) o.d. flexible plastic tubing.

8.12 *Tubing*—0.94 cm (<sup>3</sup>/<sub>8</sub> in.) o.d. flexible rubber or plastic.

8.13 *T-Connectors*—plastic T-fittings for 0.94 cm (<sup>3</sup>/<sub>8</sub> in.) o.d. flexible rubber or plastic.

8.14 *Stoppers*—rubber, No. 5.

8.15 *Pipettes*—Pasteur, 15 cm (6 in.).

8.16 *Mesh*—firm nylon, 1 to 2 mm; cut in approximately 6.25 cm (2.5 in.) diameter disc to fit on inside base of a 500-mL Erlenmeyer flask.

8.17 *Vinyl tubing*—0.94 cm (<sup>3</sup>/<sub>8</sub> in.) flexible tubing shaped as ring and attached to mesh disc.

8.18 *Cotton cloth*—45 cm × 60 cm tea towel or equal.

8.19 *Beakers*—200-mL.

8.20 *Hypodermic needle*—1-cc tuberculin syringe, 25 × <sup>5</sup>/<sub>8</sub> in., (e.g., Pharmasela. Catalog No. 7021D, or equivalent).

8.21 *Check valve*—for water bath aeration tubing.

8.22 *Marking pen*—for glass.

8.23 *Weight rings*—to anchor Erlenmeyer flasks in water bath.

8.24 *Drying oven*—forced-air, vented, set at 70±5°C.

8.25 *Muffle furnace*—vented, set at 550°C.

8.26 *Beakers*—100-mL, borosilicate glass.

8.27 *Analytical balance*—accurate to ± 0.001 g.

### 9. Reagents and Materials for Method A

9.1 *Water*—deionized, 17 MΩ·cm minimum resistivity.

9.2 *Glycerol*.

9.3 *Tween*<sup>®</sup> 20—polyoxyephene (20) sorbitan monolaurate, (available through JT Baker Chemical Co, NJ, and others).

9.4 *Bags*—plastic, with vents or perforations, 0.25 L (1 qt) size, (e.g., Ziploc<sup>®</sup> brand vegetable bags with freshness vents).

### 10. Apparatus Assembly and Sample Preparation for Method A

10.1 *Sample Cleaning*—remove all large pieces of plastic (>4.0-mm) and other non-biodegradable combustibles from the compost sample.

10.2 *Pre-Incubation*—after adjusting sample moisture, transfer 300 cm<sup>3</sup> aliquots into two bags and close the bags, (e.g., one qt size Ziploc<sup>®</sup> plastic vegetable bags). Place the bags on a lower shelf of an

incubator set at 34°C and cover loosely (as a blanket) with a wet cotton cloth (wring out after soaking in deionized water). The wet cotton tea towel minimizes evaporative water loss near the vents in the vegetable bag.

10.2.1.1 *Incubator Humidity*—Set humidity at approximately 99% relative humidity. If incubator is not equipped with humidity control system, place a humidifier on a laboratory tray on the top shelf of the incubator. Plug the humidifier into the 24 h timer set to repeat periods of 3 h *ON* and 1 h *OFF*.

NOTE 1A—Use of vented bags, wet covering cloth and humidifier is an addition to the method as described by Iannotti, et al.

10.2.2 *Pre-Incubation*—Incubate samples for approximately 24 h, pending condition of the samples. During the incubation period check the samples daily for signs of anaerobic conditions.

10.2.2.1 If problems are observed, carefully mix the sample by gently shaking or stirring. This action assists aeration by breaking and blending anaerobic pockets throughout the sample. The presence of white fungal mycelium is usually associated with high  $\text{NH}_4$  and unstable material.

CAUTION—If samples are too moist, any handling of the sample can result in the formation of clumps and balls. If this condition develops, prepare a new sample and reinitiate the incubation process.

10.3 *Flask Preparation*—before adding a compost aliquot to the flask, assemble a mesh support that will promote free movement of air below and through the sample. Refer to Fig 05.08-A1 for illustration.

10.3.1 Form a ~5-cm (2-in.) i.d. ring with flexible 0.94 cm ( $\frac{3}{8}$  in.) tubing.

10.3.2 Cut a disc of nylon mesh to fit inside bottom of 500-mL Erlenmeyer flask

10.3.3 Attach the flexible ring and disc of mesh using nylon string.

10.3.4 Push the assembled disc with tubing into the mouth of flask and force it to fit flat on the flask bottom with the ring of tubing facing down.

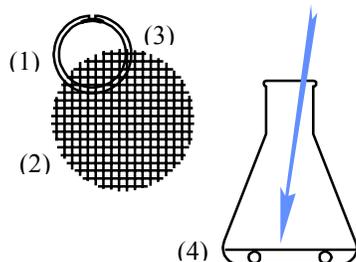


Fig 05.08-A1 Flask preparation.

10.3.5 Weigh and record the mass of the flask fitted with nylon mesh and tubing ring.

10.4 *Flask Stopper Assemblies*—Two separate stoppers are used during this test, during aeration, and during  $\text{O}_2$  measurement. Refer to Fig 05.08-A2 for illustration.

10.4.1 Bore two 7.5-mm adjacent holes into the first No. 5 rubber stopper and insert a pasteur pipette and straight glass tubing into the holes.

10.4.2 Attach a 20-cm (8-in.) section of flexible tubing fitted with check valve onto the straight glass tubing.

10.4.3 Connect the pipette and air-feed from the fritted glass sparger tube into the 1-L flask (not illustrated).

10.4.4 Bore one 1.8-cm hole into the second No. 5 rubber stopper and insert the  $\text{O}_2$  sensor.

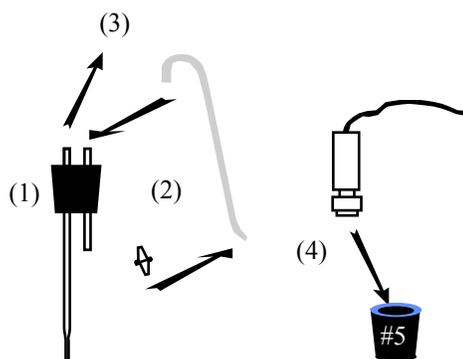


Fig 05.08-A2 Flask stopper assemblies.

10.5 Gently transfer 250  $\text{cm}^3$  aliquot of pre-incubated and properly moistened compost into the flask through a funnel with a 200-mL beaker. Refer to Fig 05.08-A3 for illustration.

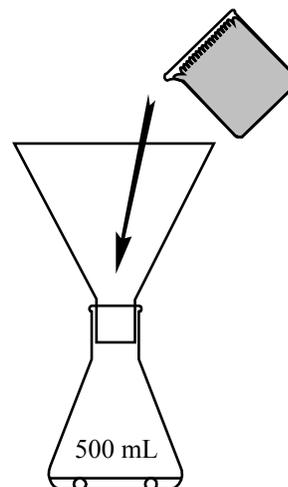


Fig 05.08-A3 Transferring compost aliquot to incubation flask.

10.5.1 Weigh and record the mass of the sample-filled flask.

NOTE 2A—The published method calls for a 60 g dw equivalent sample of moist compost. This method calls for 250 cm<sup>3</sup> of pre-incubated and properly moistened compost. Use of the volume measure improves experimental precision of the respiration test. This modification improves segregation of samples with similar solids content, but dissimilar physical characteristics, i.e., different bulk densities.

10.6 *Test Aliquot Moisture*—Transfer the remaining incubated compost material (approximately 50 cm<sup>3</sup>) into tared 100-mL beaker, record the gross weight.

10.6.1 Oven dry at 70±5°C until weigh change diminishes to nil. Record the oven dry weight for determination of sample total solids content (TS, wet basis) as described in Method 03.09 Total Solids and Moisture.

10.6.2 Ash the oven-dried aliquot at 550°C for 2 h as described in Method 05.07 LOI Organic Matter.

10.7 *Equilibrate Temperature and Aerate Sample*—Refer to Fig 05.08-A4 for illustration.

10.7.1 Fill water bath with tap water to a depth of about 6 cm and pre-heat to 34°C.

10.7.2 Place flasks containing pre-incubated compost in the pre-heated water bath and anchor with weight rings.

10.7.3 Insert aeration assembly into mouth of flask.

10.7.4 Position tip of pipette between base of flask and support mesh.

10.7.5 Place flask fitted with weight ring into 34°C water bath.

10.7.6 Attach tubing from sparger to inlet end of pipette.

10.7.7 Adjust aeration rate to allow approximately one bubble per s to pass from outlet positioned below water line.

10.7.8 Aerate using sparged air for one h to equilibrate sample temperature.

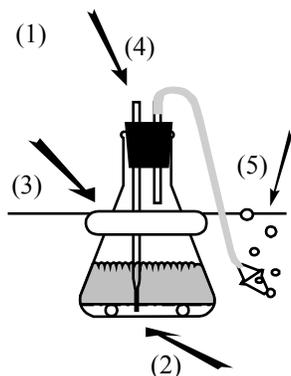


Fig 05.08-A4 Equilibrate temperature and aerate sample.

10.8 *Equilibrate Flask Air Pressure:*

10.8.1 Insert O<sub>2</sub> sensor assembly into the flasks without removing them from the water bath. To minimize air leakage use glycerol, to seal the O<sub>2</sub> sensor into the stopper and to seal the stopper into the flask.

10.8.2 Equilibrate the air pressure within flask to that outside the flask. Insert a hypodermic needle through the stopper fitted with the O<sub>2</sub> sensor before placing the stopper into the flask. Remove the needle after the stopper is securely in place and pressure within the flask equilibrates to that outside of the flask, approximately five min.

10.8.3 Check electronic cable connection from O<sub>2</sub> sensor assembly to computer.

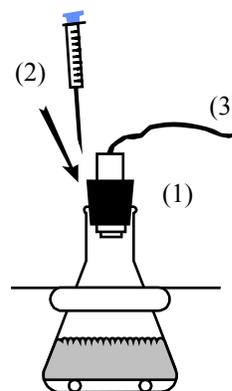


Fig 05.08-A5 Equilibrate barometric pressure inside flask.

NOTE 3A—The hypodermic needle relieves pressure due to displacement that occurs while inserting the stopper into the flask. The needle allows the air pressure inside the flask to equilibrate with that outside the flask, i.e., a parameter representing atmospheric pressure in equation 13.1. The pin-hole created by the needle seals itself. Monitor the texture of the stopper with continued use. If the stopper becomes slightly brittle, leaks will occur.

10.9 *Water Blank*—Run a parallel deionized H<sub>2</sub>O blank to detect systematic errors. Transfer 250 mL deionized H<sub>2</sub>O into a clean flask, treating it as a compost sample to measure respiration.

NOTE 4A—The original method does not call for a blank. The blank reveals non-linear changes in measured O<sub>2</sub> percent that may be attributed to systematic error, (e.g., variations in water bath temperature, voltage fluctuation and other factors not attributable to compost stability).

## 11. Procedures for Method A

11.1 *Apparatus*—Assemble apparatus as described in step 10.

11.2 *Pre-Incubation*—Perform all incubation and equilibration steps as described in step 10, above.

11.3 *Record Headspace Oxygen Concentration Change Over Time*—Set the data logger to record O<sub>2</sub> uptake (%) at 1 min intervals. Record changes in

percent O<sub>2</sub> within each flask for at least 90 min. Avoid long runs (>10 h) that promote anaerobic conditions in the flask. Anaerobic conditions will damage the O<sub>2</sub> sensors.

NOTE 6A—The original method calls for longer measurement intervals (10 min) over a shorter experiment duration (1 h). More consistent results and improved ease in diagnosing systematic errors are possible when measurements are recorded at short intervals (1 min) over a longer period of time (1.5 h).

11.4 *Determine Volume of Air in Flask (mL)*—Mark the neck of the flask at a point corresponding to the bottom or base of the O<sub>2</sub> probe assembly. Remove the probe assembly and flask from the water bath. Partially fill the flask (~80%) with deionized H<sub>2</sub>O.

NOTE 7A—Avoid formation of bubbles and foam. Partially fill the flask with water to saturate sample. Allow the sample to rest for approximately 2 h; gently stir using a nylon or glass stirring rod; then fill to volume with water, i.e., to the mark with water. Weigh and record the gross weight of the flask, water and compost sample. Add two drops of Tween® 20 to diminish the formation of bubbles and to increase absorption. Calculate the net weight of water by subtracting the flask tare weight and the calculated oven dry weight of the compost sample from the gross weight. Assume 1 g of H<sub>2</sub>O is equivalent to 1 mL of air.

11.5 *Data Analysis and Calculations*—Upon completion of the 90 min run, transfer the logged O<sub>2</sub> uptake data to a spreadsheet and create a scatter chart of O<sub>2</sub>% versus time (t, min). Select a linear segment on the chart (at least 30 min from data set) to calculate the slope for O<sub>2</sub> uptake. Usually, the rate becomes relatively linear (r > 0.99) after the 20-30 min mark. The slope of deionized water blanks should be between -0.01 and -0.02 (Δ[O<sub>2</sub>] Δt<sup>-1</sup>).

## 12. Trouble Shooting for Method A

12.1 *Aeration*—Sample aeration during temperature equilibration must be uniform across all samples (flasks) in water bath. Periodically check bubble rate (1 bubble per s). Correct any deviations by either tightening or loosening clamps on air delivery tubes.

12.2 *Temperature and Pressure Fluctuations*—Increasing temperature and pressure accelerate the rate of O<sub>2</sub> uptake and decreasing temperature and pressure decelerates uptake rates.

## 13. Calculations and Corrections for Method A

13.1 *Determine Specific Oxygen Uptake Rate, mg O<sub>2</sub> g<sup>-1</sup> (TS, OM) d<sup>-1</sup>:*

$$\text{SOUR} = \frac{0.196 \times [V \div 1000] \times P \times [\text{MW} \times 1000] \times S \times 1440}{[R \times T \times X \times M]}$$

*Equation 13.1*

13.2 *The simplified form of the SOUR formula combining all constants, mg O<sub>2</sub> g<sup>-1</sup> (TS, OM) d<sup>-1</sup>:*

$$\text{SOUR} = \frac{[(1.1 \times 10^5) \times V \times P \times S] \div [T \times X \times M]}$$

*Equation 13.2*

where:

SOUR = specific oxygen uptake rate, mg O<sub>2</sub> g<sup>-1</sup> (TS, OM) d<sup>-1</sup>

V = volume of air in flask, mL,

P = atmospheric pressure at elevation of measurement, atm,

MW = molecular weight of O<sub>2</sub> = 32 g mol<sup>-1</sup>,

R = ideal gas constant, 0.08206 L atm mol<sup>-1</sup> K<sup>-1</sup>,

T = temperature in degrees, °K (°C+273)

S = slope of change in percent O<sub>2</sub> saturation per minute divided by 100, (e.g., the change from 100% to 90% in one minute, S = 0.1 min<sup>-1</sup>),

X = wet weight of compost test aliquot, g

M = mass unit, fraction of total solids (TS) and organic matter (OM) of a parallel sample aliquot, i.e., 0.00-1.00, g g<sup>-1</sup> wet basis, at 70±5°C, and 550°C dw basis.

1440 = conversion of minutes to days,

1000 = conversion of mL to L and mg to g, unitless, and

0.196 = the fraction of O<sub>2</sub> in saturated air at 34°C.

NOTE 8A—The formula presented above was re-derived by F. Michel and modified from that originally published by Iannotti, et al., to allow accurate determinations of specific O<sub>2</sub> uptake rates at any location or elevation.

<b>Test Method:</b> Respirometry. Carbon Dioxide Evolution Rate						<b>Units:</b> mg CO <sub>2</sub> -C g <sup>-1</sup> (TS, OM) d <sup>-1</sup>		
<b>Test Method Applications</b>								
<b>Process Management</b>						<b>Product Attributes</b>		
<i>Step 1:</i> Feedstock Recovery	<i>Step 2:</i> Feedstock Preparation	<i>Step 3:</i> Composting	<i>Step 4:</i> Odor Treatment	<i>Step 5:</i> Compost Curing	<i>Step 6:</i> Compost Screening and Refining	<i>Step 7:</i> Compost Storing and Packaging	<i>Safety Standards</i>	<i>Market Attributes</i>
		05.08-B		05.08-B		05.08-B		05.08-B

## 05.08-B CARBON DIOXIDE EVOLUTION RATE

LOOK—Interference and Limitations, and Sampling Handling issues are presented as part of the introduction to this section.

COMMENT—Automated systems used in place of the apparatus described in this section provide significantly lower outcomes for moderate to highly active compost samples, (e.g., Columbus Instruments describes a system, MicroOxymax, at <http://www.colinst.com>, 950 North Hague Avenue; Columbus, OH 43204-2121 USA).

### 14. Apparatus for Method B

14.1 *Incubator*—capable of sustaining constant temperature of 25–28°C with near-100% relative humidity.

14.2 *Beakers*—50-mL, glass.

14.3 *Incubation Vessel*—200-mL Erlenmeyer flask.

14.4 *Respiration Flask*—4-L Containers, mason jars or equivalent that can be readily sealed.

14.5 *Magnetic Stirrer*.

14.6 *Titration Burette*.

14.7 *Dispensing Pipettes*.

14.8 *Flask*—volumetric, 1-L.

### 15. Reagents and Materials for Method B

15.1 *Sodium Hydroxide (1M)*—Place 40 g of NaOH pellets into a 1-L volumetric flask. Add approximately 500 mL deionized water. Dissolve completely, cool, and add deionized water to bring to 1 L and stopper tightly.

15.2 *Barium chloride (~0.5N)*—Place 120 g of BaCl<sub>2</sub>·2H<sub>2</sub>O into 1 L of CO<sub>2</sub> free distilled water. Mix well with a magnetic stirrer until dissolved and stopper tightly.

15.3 *Phenolphthalein Indicator Solution*—Dissolve 5 g solid phenolphthalein in 500 mL 95% ethyl or isopropyl alcohol, and add 500 mL distilled water. Mix well with a magnetic stirrer. If necessary, add 0.02N NaOH dropwise until a faint pink color appears in solution.

15.4 *Hydrochloric Acid (0.5M)*—Make 6M HCl, by placing 500 mL distilled water into a 1-L volumetric flask and slowly add 500 mL concentrated HCl. Make 0.5M HCl, by placing 500 mL distilled water into a 1-L

volumetric flask, adding 83 mL 6N HCl, and filling to volume mark with distilled water. Store in a glass carboy.

15.5 *Acid Normalization (mol L<sup>-1</sup>)*—Hydrochloric acid for respiration titrations should be standardized after preparation and on a monthly basis thereafter. Over time, the evaporation of water, absorption of CO<sub>2</sub> from the air and other factors may change the normality of the acid.

15.5.1 Weigh between 0.400 and 1.000 g of THAM (tris hydroxymethyl aminomethane) in a 50-mL beaker. Add approximately 20 mL of distilled water. Add two drops of bromocresol green/methyl red mixed indicator. Titrate with HCl to the endpoint denoted by a color change from green to red.

15.5.2 *Determine Normality of Acid*—Follow equation.

$$A = B \div [C \times D] \quad \text{Equation 15.5.2}$$

where:

A = normality of HCl, mol L<sup>-1</sup>,

B = 0.400 to 1.000 g of THAM (tris hydroxymethyl aminomethane), g,

C = molecular weight (gmw) THAM, 121.14 g mol<sup>-1</sup>, and

D = volume of HCl, L.

15.5.3 Repeat the standardization procedure two times and average results. Record the normality and the date in the lab notebook and on the carboy.

### 16. Procedure for Method B

16.1 *Pre-Incubation*—Prepare approximately 30 g of as-received moist material as described.

16.1.1 *Sample Moisture Adjustment*—Use the squeeze test to approximate the moisture status of each sample. Optimal moisture of approximately 70% to 85% of WHC is absolutely required. It is important to clearly report sample moisture adjustment. The moisture adjustment step must be included as commentary in the data reporting process. This is most readily accomplished by reporting the sample moisture before and after adjustment.

NOTE 1C—A squeeze test is performed with a handful of compost. A moist sample will clump when tightly squeezed. A sample with optimal moisture will feel wet, but not produce free water. A sample that is too dry is dusty and will not clump with hard squeezing.

16.1.2 Allow the samples to set or pre-incubate at room temperature (25-28°C) for a specified period of time but not less than 24 h and not more than three days. Use care to minimize sample moisture loss by maintaining high humidity conditions in the incubator, or other large, closed container.

16.1.3 The purpose of a pre-incubation period is to allow microorganisms in the compost to adapt to the mesophilic environment in which the test is conducted.

16.2 *Determination of Total Solids and Organic Matter Content*—Determine the total solids and moisture content of the sample in preparation for this respirometry test. Refer to Method 03.09 Total Solids and Moisture. Determine the organic matter content of the parallel sample using Method 05.07-A LOI Organic matter.

16.3 *Incubation*—Transfer 25.0 g pre-incubated compost sample into the incubation vessel set at 34°C. Record the weight of this sample to the nearest 0.01 g.

NOTE 2C—A 30-mL NaOH trap should be used initially for unstable materials to insure all the NaOH is not neutralized.

16.3.1 Transfer 20 mL of 1M NaOH to a 50-mL beaker. Place the NaOH and the compost sample into an incubation vessel. Close the lid tightly and place it in an incubator set at 34°C. Report the incubation temperature selected if different than that called for in this protocol.

16.3.2 Set up a blank by placing a 20 mL aliquot of 1M NaOH into an incubation vessel without a compost sample.

16.3.3 Record the date and time the first sample was prepared.

16.4 *Titration*—The amount of CO<sub>2</sub> absorbed by each NaOH trap is determined daily over a four day period by back titration of the residual with normalized HCl according to the procedure outlined below.

16.4.1 Open the incubation vessel and remove the sample container and beaker containing NaOH.

16.4.1.1 *Optional Step*—Remove and weigh the beaker containing the compost sample. This step is included to track sample moisture through the 4-day experiment. Calculate sample moisture for each of the four titrations.

16.4.2 Transfer the NaOH to a 200-mL Erlenmeyer flask rinsing with distilled water and add approximately 20 mL of 0.5N BaCl<sub>2</sub>·2H<sub>2</sub>O.

NOTE 3C—If a 30-mL NaOH trap is used, 40 mL BaCl<sub>2</sub>·H<sub>2</sub>O should be added.

16.4.3 Add two to three drops of phenolphthalein indicator.

16.4.4 After zeroing the burette, add HCl until the solution begins to turn clear. Use a magnetic stirrer to mix the solution while adding the acid. The endpoint has been reached when addition of one drop of HCl turns the solution from pink to clear.

16.4.5 Record the date and time the first sample was titrated, the normality of the HCl used and the volume of HCl required to achieve the endpoint.

16.4.6 Place the sample back into the incubation vessel with a fresh amount of NaOH.

16.5 Perform calculations for each of the four titrations. Report the average CO<sub>2</sub> evolution rate on the basis of both totals solids and organic matter, as mg CO<sub>2</sub>-C g<sup>-1</sup> TS d<sup>-1</sup> and mg CO<sub>2</sub>-C g<sup>-1</sup> OM d<sup>-1</sup>.

## 17. Calculations for Method B

17.1 Calculate CO<sub>2</sub> Evolution for each titration:

$$A = [(B - C) \times (D \times E)] \div [F \times G] \quad \text{Equation 17.1}$$

where:

- A = mg CO<sub>2</sub>-C g<sup>-1</sup> (TS, OM) d<sup>-1</sup>,
- B = volume of standardized HCl used for blank titration, mL,
- C = volume of standardized HCl used for sample titration, mL,
- D = normality of standardized HCl, mol<sub>c</sub> L<sup>-1</sup>,
- E = 6 = equivalent weight of CO<sub>2</sub>-C in NaOH,
- F = moist weight of sample in container, g, and
- G = mass unit, fraction of total solids (TS) and organic matter (OM) determined on a parallel sample, 0.00-1.00, g g<sup>-1</sup> wet basis determined at 70±5°C or 550°C dw basis, respectively.

17.2 Calculate the average rate of CO<sub>2</sub> evolution:

$$H = \Sigma A \div I \quad \text{Equation 17.2}$$

where:

- H = average mg CO<sub>2</sub>-C g<sup>-1</sup> (TS, OM) d<sup>-1</sup>,
- ΣA = tally CO<sub>2</sub> evolution measures from days one through four, from Equation 17.1, and
- I = duration of experiment, four d.

<b>Test Method:</b> Respirometry. In-Situ Oxygen Refresh Rate						<b>Units:</b> % O <sub>2</sub> hr <sup>-1</sup> chart		
<b>Test Method Applications</b>								
<b>Process Management</b>							<b>Product Attributes</b>	
<i>Step 1:</i> Feedstock Recovery	<i>Step 2:</i> Feedstock Preparation	<i>Step 3:</i> Composting	<i>Step 4:</i> Odor Treatment	<i>Step 5:</i> Compost Curing	<i>Step 6:</i> Compost Screening and Refining	<i>Step 7:</i> Compost Storing and Packaging	<i>Safety Standards</i>	<i>Market Attributes</i>
		05.08-C		05.08-C		05.08-C		05.08-C

### 05.08-C IN-SITU OXYGEN REFRESH RATE

LOOK—Interference and Limitations, and Sampling Handling issues are presented as part of the introduction to this section.

SUBMITTED BY William F. Brinton, Woods End Research.

#### 18. Apparatus for Method C

18.1 *Oxygen probe*—configuration adapted to field data collection, or equivalent manufactured device

18.1.1 *Oxygen sensor*—capable of  $\pm 0.5\%$  O<sub>2</sub> readings, with temperature compensator.

18.1.2 *Probe*—galvanized steel pipe, 0.3-cm (1/8 in.) I.D. and ~1.3-m length.

18.1.3 *Filter*—in-line, for water vapor.

18.1.4 *Squeeze bulb*—with a per squeeze volume of five mL, to extract air.

18.2 *Data logger (optional)*—digital data logger, minimum specification to log at a ten-minute interval for two h.

#### 19. Procedures for Method C

19.1 Pre-calibrate the O<sub>2</sub> probe to ambient at 20.9% O<sub>2</sub>.

19.2 Perform aeration procedure, (e.g., turn windrow, cycle fans, etc., pending composting system).

19.3 Insert probe to a specified depth. The probe remains in position until the end of reading the refresh test.

NOTE 1C—It is advisable to repeat this test at various pile positions and depths to test for uniformity of pile aeration characteristics.

19.3.1 *Sampling Strategies*—stratify the compost pile or bin into spatial zones, where each zone represents a relative position within the compost, (e.g., core of the bulk and margins at various depths).

Perform the test at all positions to evaluate aeration uniformity.

19.4 Record percent O<sub>2</sub> at a ten min interval for two h or until readings level off. Round the O<sub>2</sub> reading to the nearest whole unit.

19.5 Graph results with Y-axis as O<sub>2</sub> percent and X-axis as time in minutes.

#### 20. Interpretation of Results for Method C

20.1 Refresh rate is considered excellent if pile O<sub>2</sub> does not fall below 5% within two h. If pile O<sub>2</sub> falls under 2% in thirty min, then odor events are likely. The interpretation must consider the feedstock types, (e.g., a higher minimum O<sub>2</sub> reading may be appropriate for feedstocks that are predisposed to produce odor).

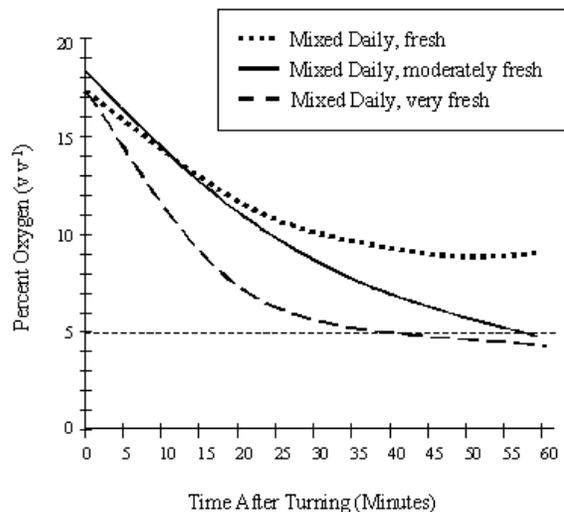


Fig 05.08-C1 Idealized oxygen refresh rate test results from monitoring three piles of varying condition during active phases of the composting process.

<b>Test Method:</b> Respirometry. Dewar Self-Heating Test						<b>Units:</b> Δ°C		
<b>Test Method Applications</b>								
<b>Process Management</b>						<b>Product Attributes</b>		
<i>Step 1:</i> Feedstock Recovery	<i>Step 2:</i> Feedstock Preparation	<i>Step 3:</i> Composting	<i>Step 4:</i> Odor Treatment	<i>Step 5:</i> Compost Curing	<i>Step 6:</i> Compost Screening and Refining	<i>Step 7:</i> Compost Storing and Packaging	<i>Safety Standards</i>	<i>Market Attributes</i>
		05.08-D		05.08-D				05.08-D

## 05.08-D DEWAR SELF-HEATING TEST

LOOK—Interference and Limitations, and Sampling Handling issues are presented as part of the introduction to this section.

ADAPTED FROM—Woods End Research Laboratory, Inc. in Mt. Vernon, ME. Dr. William Brinton, President of Woods End Research Laboratory; Dr. Mary Droffner, Director of Microbiology; Eric Evans, Laboratory Director; and Richard Brinton, director of Woods End UK, Stroud, England office. A Dewar Self-heating Kit and set of instructions are available by writing to Woods End at the following address: Woods End Research Laboratory, Inc.; P.O. Box 197 Mt. Vernon, ME 04352. Tel: 207.293.2457. The Dewar test was first formulated in Stuttgart Germany by Jourdan (1982).

### 21. Apparatus for Method D

NOTE 1D—The Dewar kit presented here consists of three parts, each replaceable separately. The proper materials may be readily obtained from major scientific supply houses.

21.1 *Dewar vessel*—2-L, 100 mm i.d., steel-encased,

NOTE 2D—The inner diameter and volume specifications of the Dewar vessel must be correct.

21.2 *Thermometer*—dual scale min-max inside/outside digital thermometer with ±1°C increments over a range of 10°C through 80°C, and

21.3 *Thermocouple probe*—30 cm, attached to a PVC wand for insertion into vessel.

### 22. Reagents and Materials for Method D

22.1 *None required.*

### 23. Procedure for Method D

23.1 Separate approximately 2 L of the representative compost sample.

23.2 Determine sample moisture.

23.2.1 Add or remove moisture if the sample is too dry or wet.

23.3 Equilibrate compost sample to ambient temperature (18-22°C).

23.4 Fill the Dewar flask with sample material (~2 L). Gently shake the filled flask to simulate natural settling.

23.5 Insert the high-point reading thermocouple probe into the flask to a point about 5 cm (2 in.) from the bottom of the flask.

NOTE 3D—Do not push against the bottom of the flask.

23.5.1 The thermometer records both maximum ambient and sample temperatures.

23.5.2 Maintain ambient temperature and vessel at 18°C - 22°C for the duration of the test.

23.6 Record ambient and sample temperatures on a daily basis, and days of readings.

23.6.1 Compost will normally achieve its highest temperature within three to five days. If the compost sample has been exposed to very cold conditions or requires remoistening, maximum temperature may not be achieved until days five to ten.

23.6.2 Continue recording temperatures for at least two days after maximum temperature is reached.

### 24. Calculations for Method D

24.1 *Net Temperature Rise:*

$$R = H - A \quad \text{Equation 24.1}$$

where:

R = net temperature rise, Δ°C,

H = highest temperature recorded over test period, °C, and

A = ambient temperature recorded, °C.

### 25. Interpretation for Method D

25.1 Interpretation of the results is based on division into five-levels of 10°C increments of the compost heating (Refer to Table 05.08-D2). For example, Class I refers to 10°C, II is 20°C and the highest grade V is 50°C heating over ambient. Heating past this high point can occur but is unlikely owing to obvious self-limitation around 70°C. The results require about 2-9 days to record; fresh composts achieve elevated temperatures sooner than stable composts.

25.1.1 The five categories on the interpretation scale are often grouped by practitioners and European agencies into three major classes, where the lowest grade (I) is called "fresh-compost", the middle two (II-III) is referred to as "active compost", and the upper two (IV-V) are termed "finished compost". Compost marketers expect compost to be grade IV or V. The basis of this classification of ripeness is shown in Table

05.08-D1 Classification into five groups rather than three is arbitrary, and has been frequently debated in official circles. Essentially, the system has been upheld by more recent European work.

to interpret the results of such anomalous samples. Woods End experience shows that heat or moisture damaged composts behave in this manner, appearing to be stable but re-heating significantly later, presumably due to re-establishment of indigenous microflora.

25.2 Some Dewar runs give inexplicable heat rise after a week or more in the vessel. Care must be taken

Table 05.08-D1 Example format for data collection.

<i>Term</i>	<i>Date/Time</i>	<i>Flask Temp. (°C)</i>	<i>Ambient Temp.(°C)</i>	<i>Net Rise (°C)</i>
<i>Day 0</i>				
<i>Day 1</i>				
<i>Day 2</i>				
<i>Day 3</i>				
<i>Day 4... etc.</i>				
				<i>Maximum:</i>
				<i>Stability Rating:</i>

Table 05.08-D2 Dewar self-heating increments, rating and description of stability classification based on the European system.

<i>Temperature Rise Above Ambient</i>	<i>Official Class of Stability</i>	<i>Descriptors of Maturity Class or Group</i>	<i>Major Group of Compost</i>
< 10°C	V	Finished Compost; stable to very stable compost	Finished
10° – 20°C	IV	Maturing; moderately unstable, curing compost	Curing
20° – 30°C	III	Active Compost, material decomposing and unstable	Active
30° – 40°C	II	Immature Compost, young or very active compost	Active
> 40°C	I	Raw Feedstock; fresh compost, mixed ingredients	Raw or Fresh

25.3 *Dewar Self-Heating versus CO<sub>2</sub> Respirometry*— Compost self-heating in a Dewar vessel is a respiration technique, and provides similar results to CO<sub>2</sub> respirometry measured over a 3-day to 7-day period. The Dewar test measures heat released during microbial respiration associated with the composting process. Table 05.08-D3 illustrates the relative relationships between heat rise measured in Dewar vessel and corresponding measures of CO<sub>2</sub> respiration levels.

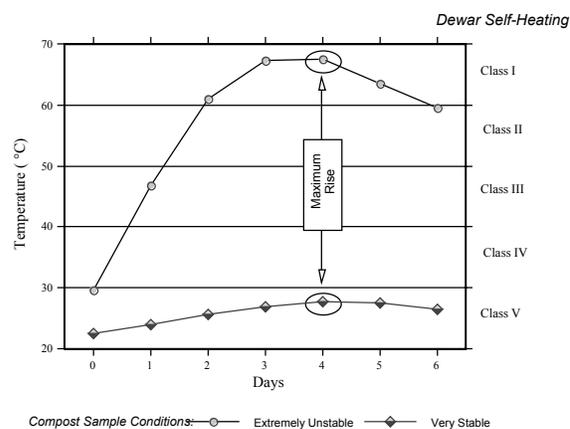


Fig 05.08-D1 Dewar Self-Heating illustration of test results for two distinct composts across a testing period of six days.

25.3.1 The data in the left and middle columns in Table 05.08-D3 indicate the respiration level and maturity classification. The right column of Table 05.08-D3 indicates the corresponding Dewar maturity classification that approximates accords to these levels.

It may be seen that Dewar saturates at a lower respiration rate than with CO<sub>2</sub> respirometry methods.

25.3.2 The difference between laboratory respiration and Dewar methods is that the Dewar vessel simulates heat take-off simultaneous with the experiment, and may quickly reach a self-limiting temperature, i.e., temperatures greater than 65°C. Only a very sophisticated laboratory feed-back apparatus which generates a heat rise proportional to carbon released during respiration could produce data which is truly comparable to the same as data obtained from a Dewar test.

25.3.3 The Dewar classification range is narrower than that provided by CO<sub>2</sub> respirometry alone. The Dewar method is driven by temperature increases induced through accelerated microbial respiration, most notably within Dewar classes III and IV. Unlike the Dewar self-heating procedure, conventional CO<sub>2</sub> or O<sub>2</sub> respirometric methods monitor microbial respiration at a fixed temperature in comparison to temperatures found in an actual composting conditions (Iannotti, 1993).

Table 05.08-D3 Relationship of CO<sub>2</sub> respiration to Dewar self-heating test and equivalent classes.

mg CO <sub>2</sub> -C g TS d <sup>-1</sup>	mg CO <sub>2</sub> -C g OM d <sup>-1</sup>	Rating of Respiration	Equivalent Dewar Maturity Class
0 – 6	0 – 4	▪ very low rate	V
6 – 25	4 – 16	▪ moderately low	IV-III
25 – 46	16 – 30	▪ medium rate	II-I
46 – 77	30 – 50	▪ medium-high rate	I
> 77	> 50	▪ high rate	

NOTE 1D—rating developed and used by Woods End scientists since 1980 and is based on screening several thousand composts

25.4 *Marketing Considerations*—European field data for biosolids composts suggests several ways to interpret the Dewar data. Table 05.08-D4 relates “Best Use” for source-separated residential food residue blended with yard-waste compost to the Dewar classifications. Different and possibly more conservative use guidelines may be applicable for other composts with different feedstocks.

Table 05.08-D4 Proposed relationship of Dewar class to best use of compost.

Class of Stability Dewar Test	Best Use of Compost Class
V	Potting Mixes, seedling starters
IV	General Purpose Gardening, Greenhouse cultivation
III	All field Crops, Grapes, Fruit, Apples
II	Limited Field Cultivation with wait-period, (e.g., corn, tomatoes, broccoli, etc.)
I	Compost Raw Feedstock only

25.5 *Pathogen Reduction*—The Dewar method may also be useful for assessing pathogen reduction. In one study, preliminary findings with biosolids composts suggest consistent pathogen removal by USEPA 40CFR503 standards after the compost achieves Dewar class IV and fails to re-heat after disturbance.

<b>Test Method:</b> Respirometry. Solvita <sup>®</sup> Maturity Index						<b>Units:</b> index		
<b>Test Method Applications</b>								
<b>Process Management</b>							<b>Product Attributes</b>	
<i>Step 1:</i> Feedstock Recovery	<i>Step 2:</i> Feedstock Preparation	<i>Step 3:</i> Composting	<i>Step 4:</i> Odor Treatment	<i>Step 5:</i> Compost Curing	<i>Step 6:</i> Compost Screening and Refining	<i>Step 7:</i> Compost Storing and Packaging	<i>Safety Standards</i>	<i>Market Attributes</i>
		05.08-E	05.08-E	05.08-E		05.08-E		05.08-E

## 05.08-E SOLVITA<sup>®</sup> MATURITY INDEX

LOOK—Interference and Limitations, and Sampling Handling issues are presented as part of the introduction to this section.

CONTRIBUTION—Dr. William F. Brinton, Woods End Research Laboratory, Inc.

### 26. Apparatus for Method E

26.1 *Solvita<sup>®</sup> jar*—sample container provided in the test kit package by the manufacturer.

### 27. Reagents and Materials for Method E

27.1 *Gel strips*—two color-coded paddles provided in the test Kit package by the manufacturer.

### 28. Procedure for Method E

28.1 *Transfer compost to the Solvita<sup>®</sup> jar*—Fill the jar to the fill line (Fig 05.08-E1). To obtain proper sample density, sharply tap the bottom of the jar on a counter. Fluffy or coarse composts should be compacted by pressing firmly into the jar.



Fig 05.08-E1 *Solvita<sup>®</sup>* test kit jar.

NOTE 1E—Equilibration Step. Compost in a sub-optimal state, as described under sample handling, may require equilibration for one to three d prior to testing. Equilibration may be necessary for thermophilic samples, for samples whose moisture is adjusted after collection, and for frozen samples.

28.2 *Remove gel paddles from their packaging*—Open each package by tearing along the top strip. Carefully remove each paddle by grasping its handle. Do not touch the special gel surface nor allow compost to physically contact the gel. Start the test within 30 min after the *Solvita<sup>®</sup>* gelpacks are opened.

28.3 *Insert the paddles into the sample*—Orient the two paddles as indicated by the color squares on the jar label. Labels must be seen through their respective viewing-window. Push the paddle tips into the compost to the bottom of the jar. The paddles should be

positioned at right angles to each other. Edges of the paddles can touch each other in the middle of their gel strips without affecting the results. Be careful not to jostle or tip the jar. Do not use a paddle if the gel is dry or discolored. The gel color should be that same as the “Control Color” indicated on the respective color charts.



Fig 05.08-E2 Gel paddle.

CAUTION—The gel is not harmful to touch, but should not physically contact the mouth or eyes.

28.4 *Screw the lid tight*—Allow the sample to incubate for four h out of contact with direct sunlight at an ambient temperature of 20-25°C (68-77°F).



Fig 05.08-E3 Test period is four h.

28.5 *Read the gel color*—Read and interpret the *Solvita<sup>®</sup>* paddle colors four h after the lid is secured and the test is started. With the lid in place, view the paddle colors through their respective viewing windows at the side of the jar. Illuminate the paddles from the front with moderate-intensity, fluorescent lighting. Compare to the color charts provided with the kit, and record the color numbers that most closely match (Refer to Fig 05.08-E4).



Fig 05.08-E4 Color indicators.

NOTE 2E—The Solvita<sup>®</sup> test is based on a 4-h reading. Gel color may continue to change after the 4-h incubation period, so it is imperative that color interpretation be performed at 4-h to ensure proper interpretation. Color is ideally interpreted under bright fluorescent lighting.

## 29. Interpretation of Results for Method E

29.1 The Solvita<sup>®</sup> Maturity Index of the compost sample is determined in Table 05.08-E1 from the test result color numbers for CO<sub>2</sub> and NH<sub>3</sub> corresponding to the color charts (Fig 05.08-E4). This index value is

used to determine the level of compost maturity with the use of Table 05.08-E2. For composts with low ammonia (chart value 4 or 5) the Solvita<sup>®</sup> Maturity Index is the same as the CO<sub>2</sub>-color number. For high ammonia levels, the Solvita<sup>®</sup> Maturity Index will be less than it appears from the CO<sub>2</sub> result. The reason is that ammonia can inhibit microbial activity and interfere with the CO<sub>2</sub> test.

29.2 As compost ages, it normally goes from a fresh condition (Solvita<sup>®</sup> Index 1 to 2) to a mature state (Solvita<sup>®</sup> Index 7 to 8). This can take weeks to months, depending on the materials and method of composting. Table 05.08-E1 presents an overview of this aging process and shows how other tests that are used to characterize stability can be compared to the Solvita<sup>®</sup> test.

Table 05.08-E1 Solvita<sup>®</sup> Maturity Index Computation Table.

		Paddle C								
		1	2	3	4	5	6	7	8	
Paddle A	5	Very Low NH <sub>3</sub>	1	2	3	4	5	6	7	8
	4	Low NH <sub>3</sub>	1	2	3	4	5	6	7	8
	3	Medium NH <sub>3</sub>	1	1	2	3	4	5	6	7
	2	High NH <sub>3</sub>	1	1	1	2	3	4	5	6
	1	Very High NH <sub>3</sub>	1	1	1	1	1	2	3	4

Table 05.08-E2 Solvita<sup>®</sup> Maturity Index and other Indexes.

Solvita <sup>®</sup> Maturity Index	STAGE OF THE COMPOSTING PROCESS	MAJOR CLASS	Approximate Equivalencies <sup>1</sup>		
			Dewar	CO <sub>2</sub> -C Rate	O <sub>2</sub> Rate
8	Inactive, highly matured compost, very well aged, possibly over-aged, like soil; no limitations for usage	FINISHED COMPOST	V	1	< 3
7	Well matured, aged compost, cured; few limitations for usage			2	5
6	Curing; aeration requirement reduced; compost ready for piling; significantly reduced management requirements	Curing		4	11
5	Compost is moving past the active phase of decomposition and ready for curing; reduced need for intensive handling	ACTIVE COMPOST	IV	6	16
4	Compost in medium or moderately active stage of decomposition; needs on-going management	Very Active	III	8	21
3	Active compost; fresh ingredients, requires intensive oversight and management		II	10	27
2	Very active, putrescible fresh compost; high-respiration rate; requires very intensive aeration and/or turning	RAW COMPOST	I	12	32
1	Fresh, raw compost; typical of new mixes; extremely high rate of decomposition; putrescible or very odorous material			> 15	> 40

<sup>1</sup> Interpretations provided by Woods End Research

Column 1: Dewar Self Heating test using standard Dewar Flask, grades as per interpretation (see TMECC Method 05.08-D)

Column 2: CO<sub>2</sub> Rate is the total mg CO<sub>2</sub>-C evolved per g (OM) per day at 34°C (see TMECC Method 05.08-B)

Column 3: O<sub>2</sub> Respiration Rate (SOUR) as mg O<sub>2</sub> consumed per g (OM) per day (calculated from Column 2)

<b>Test Method:</b> Respirometry. Biologically Available Carbon						<b>Units:</b> mg CO <sub>2</sub> -C g <sup>-1</sup> OC d <sup>-1</sup>		
<b>Test Method Applications</b>								
<b>Process Management</b>						<b>Product Attributes</b>		
<i>Step 1:</i> Feedstock Recovery	<i>Step 2:</i> Feedstock Preparation	<i>Step 3:</i> Composting	<i>Step 4:</i> Odor Treatment	<i>Step 5:</i> Compost Curing	<i>Step 6:</i> Compost Screening and Refining	<i>Step 7:</i> Compost Storing and Packaging	<i>Safety Standards</i>	<i>Market Attributes</i>
		05.08-F	05.08-F	05.08-F		05.08-F		05.08-F

## 05.08-F BIOLOGICALLY AVAILABLE CARBON

LOOK—Interference and Limitations, and Sampling Handling issues are presented as part of the introduction to this section.

CONTRIBUTION—**PROPOSED METHOD** by Frank Shields; Soil Control Lab; Watsonville, CA

### 30. Apparatus for Method F

30.1 *CO<sub>2</sub> analyzer and integrator*—(e.g., FUJI Electric and HP3393A or equal).

30.2 *Incubator*—capable of maintaining 36°C.

30.3 *Analytical balance*—with ±0.005 g precision.

30.4 *Air tank*

30.5 *CO<sub>2</sub> tank*

30.6 *Syringes*—250, 100 and 25 µL.

30.7 *Oven*—forced-air, capable of sustaining 70°C.

30.8 *Sieve*—stainless steel or plastic, 4-mm (#5) mesh.

30.9 *Crucibles*—porcelain, 70 cm<sup>3</sup>.

30.10 *Volatile organic acid bottles*—40-mL, typically used in GC analysis, (e.g., I-Chem 40-mL clear VOA).

30.11 *Rubber stopper*—2 hole, #1, to fit VOA bottle listed above.

30.12 *Vinyl tubing*—4-mm i.d.

30.13 *T-fitting*—4-mm o.d.

30.14 *Timer*—2 h.

### 31. Reagents and Materials for Method F

31.1 *Plastic bags*—to cover plastic cups, (6 in. × 9 in.).

31.2 *Plastic cups*—500-mL, (e.g., Solo P-16, 16-oz).

31.3 *Sand*—quartz, #20, soaked in 1N HCl two h, DI water wash, Heated 500°C).

31.4 *Water*—deionized, minimum resistivity 17 MΩ·cm, minimum standard.

31.5 *Stock Nutrient Solution (Hoagland Solution)*—Dissolve potassium phosphate in approximately 750 mL of water. Add and dissolve the other chemicals in the order presented (below). Dissolve each chemical before adding the next. Warm water will speed up the

process. Bring to 1 L with DI water. Dilute 5× for working solution.

31.5.1 *potassium phosphate*—KH<sub>2</sub>PO<sub>4</sub>, 1.4 g.

31.5.2 *potassium nitrate*—KNO<sub>3</sub>, 5.7 g.

31.5.3 *calcium nitrate*—  
5Ca(NO<sub>3</sub>)<sub>2</sub>·NH<sub>4</sub>NO<sub>3</sub>·10H<sub>2</sub>O, 8.5 g.

31.5.4 *magnesium sulfate*—MgSO<sub>4</sub>·7H<sub>2</sub>O, 4.3 g.

### 32. Procedure for Method F

#### 32.1 Sample Preparation:

32.1.1 Screen an as-received moist compost sample through a 4-mm sieve.

32.1.2 Determine total solids and organic carbon content on a parallel aliquot. Use Method 04.01 Organic Carbon.

32.1.3 Transfer 10.0 to 20.0 g of compost to a 500-mL plastic cup.

32.1.4 Add 90 g of sand to the same cup and mix thoroughly.

32.1.5 Stir in 20 mL of nutrient solution.

32.1.6 *Blank*—Prepare sand plus nutrient solution in a second 500-mL plastic cup.

32.1.7 Place the plastic bag over the top of each prepared 500-mL sample cup and incubate at 35°C for three d.

#### 32.2 Experimental:

32.2.1 The incubated sand/compost and blank are re-mixed in their cups.

32.2.2 Sub-samples are removed for total solids and organic carbon determinations.

32.2.3 Place 4 to 10 grams of prepared sample in each of five 40-mL VOA bottles; include both samples and blank.

32.2.4 Zero the timer.

32.2.5 A light flow of air is introduced into the VOA bottle #1 for 8 sec, which is then capped and shaken.

32.2.6 At fifteen sec, air is introduced to the VOA bottle #2 until all ambient air is replaced with

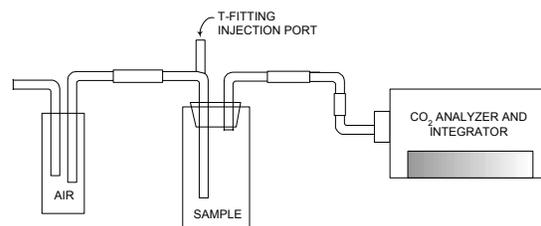
compressed air. The VOA bottle is then capped and the sample allowed to rest (respire) for one h.

32.2.6.1 *Calibrate the CO<sub>2</sub> Analyzer*—During the one-hour resting period, calibrate the CO<sub>2</sub> analyzer by injecting known concentrations of CO<sub>2</sub> into the T-fitting and plotting a line. The T-fitting is capped with tape between injections.

32.2.7 At exactly one h, the first VOA bottle is uncapped and quickly positioned in place of the sample VOA bottle #1 in the set-up. The CO<sub>2</sub> produced in one h by that sample is recorded.

32.2.7.1 After fifteen sec the next sample VOA bottle is positioned in the setup to replace the previous sample. This process continues until all samples are measured.

### 32.3 Preparation of CO<sub>2</sub> Apparatus:



### 33. Calculations for Method F

33.1 Calculate the amount of CO<sub>2</sub>-carbon (mg CO<sub>2</sub>-C) per gram of compost organic carbon per day as follows:

$$X = (A \times 0.01286) \div OC \quad \text{Equation 33.1}$$

where:

$$X = \text{mg CO}_2\text{-C g}^{-1} \text{OC d}^{-1}$$

A = CO<sub>2</sub> per hour,  $\mu\text{L}$ , and

OC = organic carbon, % dw, determined using Method 04.01-A.

## 05.08 METHODS SUMMARY

### 34. Report

34.1 Report sample condition, including as-received moisture content, presence or absence of fungal mycelium, and sample color and odor as indicated in Method 05.08. Indicate whether sample is in an anaerobic or aerobic state upon testing, both before and after pre-incubation steps.

34.2 *Method 05.08-A Specific Oxygen Uptake Rate*—Report respiration rate as a function of O<sub>2</sub> consumption and the corresponding rating and characteristic, following the stability indexing system listed in Table 05.08-1; column 1 - SOUR OM.

34.2.1 *Units*—±1 mg O<sub>2</sub> g<sup>-1</sup> TS d<sup>-1</sup>, and

34.2.2 *Units*—±1 mg O<sub>2</sub> g<sup>-1</sup> OM d<sup>-1</sup>.

34.2.3 Report methods for determining mass units, TS and OM. Use Method 05.07 LOI Organic Matter for OM, sometimes referred to as BVS, as the unit mass and Method 03.09-A Total Solids and Moisture at 70±5°C for TS as the unit mass.

NOTE—OM represents the organic matter fraction of a sample and assumes that man-made volatile inerts, if present, do not exceed a sieve size of 4 mm.

34.3 *Method 05.08-C Carbon Dioxide Evolution Rate*—Report respiration rate as a function of CO<sub>2</sub> evolution and the corresponding rating and characteristic, following the stability indexing system listed in Table 05.08-1; column 2 - CO<sub>2</sub> Evolution.

34.3.1 *Units*—±1 mg CO<sub>2</sub>-C g<sup>-1</sup> OM d<sup>-1</sup>, and

34.3.2 *Units*—±1 mg CO<sub>2</sub>-C g<sup>-1</sup> TS d<sup>-1</sup>.

34.3.3 Report method for determining mass unit basis, TS or OM. Use Method 05.07 LOI Organic Matter for OM, sometimes referred to as BVS, as the unit mass and Method 03.09-A Total Solids and Moisture at 70±5°C for TS as the unit mass.

34.4 *Method 05.08-B In-Situ Oxygen Refresh Rate:*

34.4.1 *Units*—±0.5 %, % O<sub>2</sub>.

34.4.2 Report description of apparatus and calibration technique employed at time of sampling.

34.5 *Method 05.08-D The Dewar Self-Heating*—Report method name and apparatus used; total solids content; source material of compost, (e.g., municipal solid waste, biosolids, yard waste, etc.); net temperature rise (Δ°C); maximum sample temperature (°C); ambient temperature (°C) under test conditions, the number of days (d) required to reach maximum temperature rise; and stability class as determined using Table 05.08-D2.

34.6 *Method 05.08-E The Solvita<sup>®</sup> Maturity Index*—Report on a scale of 1 to 8 determined using the 2-way table, Table 05.08-E1.

34.6.1 Report the relative CO<sub>2</sub> level using the *Solvita<sup>®</sup>* scale of 1 to 8.

34.6.2 Report the relative NH<sub>3</sub> level using the *Solvita<sup>®</sup>* scale of 1 to 5.

34.6.3 Report all sample pretreatment steps beyond those described under paragraphs 7.4 and 28 of this protocol, (e.g., extended incubation times, temperature equilibration, moistening of sample, drying of sample, etc.).

34.7 *Method 05.08-F Biologically Available Carbon:*

34.7.1 *Units*—±1 mg CO<sub>2</sub>-C g<sup>-1</sup> OC d<sup>-1</sup>, where OC, is the organic carbon content determined using Method 04.01.

### 35. Interpretation of Results

35.1 The Compost Stability Index is based upon results of respiration monitoring to measure the relative level of microbial activity in a sample (Table 05.08-1).

35.2 The level of microbial activity in a sample is determined using results of respiration monitoring; however, the index (below) assumes optimized moisture, temperature and nutrient status that favor microbial activity, and insignificant concentrations of

toxins and other compounds that inhibit microbial respiration.

35.3 Generally, it is not appropriate to report respirometry test results as the sole measure of compost stability. Always review analytical results for nutrient content, pH, electrical conductivity, etc., and screen for the presence of phytotoxins with a biological assay when establishing compost use guidelines or restrictions.

Table 05.08-1 Compost Stability Index—Ranges indicate relative compost stability for respiration methods described in TMECC. The level of microbial activity in a sample is based primarily upon results of respiration monitoring. The index assumes optimized moisture, temperature, pH, and nutrient status that favor microbial activity, and insignificant concentrations compounds that inhibit microbial respiration.

<i>SOUR (OM)</i> <i>05.08-A</i>	<i>CO<sub>2</sub>-C</i> <i>05.08-B</i>	<i>DEWAR</i> <i>05.08-D</i>	<i>SOLVITA</i> <sup>®</sup> <i>05.08-E</i>	<i>BAC OC</i> <i>05.08-F</i>	<i>STABILITY</i> <i>RATING</i>	<i>GENERAL CHARACTERISTICS</i>
< 3	1	V	8	< 2	very stable	<ul style="list-style-type: none"> <li>▪ well cured, finished compost</li> <li>▪ no continued decomposition</li> <li>▪ no odors</li> <li>▪ no potential for VFA phytotoxicity and odor</li> </ul>
3 – 10	2 – 4		7	2 – 4	stable	<ul style="list-style-type: none"> <li>▪ moderately well cured compost</li> <li>▪ odor production not likely</li> <li>▪ limited potential for VFA phytotoxicity and odor</li> <li>▪ minimal to no impact on soil carbon and nitrogen dynamics</li> </ul>
11 – 20	5 – 7	IV	5 – 6	5 – 8	moderately unstable, curing compost	<ul style="list-style-type: none"> <li>▪ curing compost</li> <li>▪ odor production not likely</li> <li>▪ aeration requirement reduced</li> <li>▪ limited potential for VFA phytotoxicity and odor</li> <li>▪ minor impact on soil carbon and nitrogen dynamics</li> </ul>
21 – 26	8 – 9	III	4	9 – 12	unstable raw compost	<ul style="list-style-type: none"> <li>▪ active, uncured compost</li> <li>▪ minimal odor production</li> <li>▪ high aeration requirement</li> <li>▪ moderate to high potential for VFA phytotoxicity</li> <li>▪ moderate potential for negative impact on soil carbon and nitrogen dynamics</li> </ul>
27 – 31	10 – 11	II	3	13 – 20	raw compost, raw organic products	<ul style="list-style-type: none"> <li>▪ highly active, uncured compost</li> <li>▪ odor production likely</li> <li>▪ high aeration requirement</li> <li>▪ high potential for VFA phytotoxicity and odor</li> <li>▪ high potential for negative impact on soil carbon and nitrogen dynamics</li> </ul>
> 32	> 11	I	1 – 2	> 20	raw feedstock, unstabilized material	<ul style="list-style-type: none"> <li>▪ raw, extremely unstable material</li> <li>▪ odor production expected</li> <li>▪ high aeration requirement</li> <li>▪ probable VFA phytotoxicity with most materials</li> <li>▪ negative impact on soil carbon and nitrogen dynamics expected</li> <li>▪ generally not recommended for use as compost</li> </ul>

**REPORTING UNITS:**

*SOUR OM* : mg O<sub>2</sub> g<sup>-1</sup> OM d<sup>-1</sup>; *CO<sub>2</sub>-C* : mg CO<sub>2</sub>-C g<sup>-1</sup> OM d<sup>-1</sup>; *Dewar and Solvita*<sup>®</sup> : refer to respective indices; *BAC OM* : mg CO<sub>2</sub>-C g<sup>-1</sup> OC d<sup>-1</sup>. It is not recommended to report a respirometry test result as the sole measure of compost stability.

NOTE—Anticipate refinement of the compost stability index with advances in compost stability research.

### **36. Precision and Bias**

*36.1.1 Method 05.08-A Specific Oxygen Uptake Rate*—The precision and bias of this test have not been determined. Data are being sought for use in developing a precision and bias statement.

*36.2 Method 05.08-B Carbon Dioxide Evolution Rate*—The precision and bias of this test are not documented. Data are being sought for use in developing a precision and bias statement.

36.2.1 Precision of the titrimetric method is approximately  $\pm 10\%$  of the known CO<sub>2</sub> concentration.

*36.2.2 Method 05.08-C In-Situ Oxygen Refresh Rate*—The precision and bias of this test have not been determined. Data are being sought for use in developing a precision and bias statement.

*36.3 Method 05.08-D The Dewar Self-Heating Method*—The precision and bias of this test have not

been determined. Data are being sought for use in developing a precision and bias statement.

*36.4 Method 05.08-E Solvita<sup>®</sup> Maturity Index*—Color change is linear over the range of CO<sub>2</sub> or NH<sub>3</sub> tested. The tonal range of the color indicator is accurate even for color-blind perception. Half-tone color changes can be accurately interpolated by the trained eye.

*36.5 Method 05.08-F Biologically Available Carbon*—The precision and bias of this test have not been determined. Data are being sought for use in developing a precision and bias statement.

### **37. Keywords**

37.1 carbon dioxide (CO<sub>2</sub>) evolution; microbial activity; oxygen (O<sub>2</sub>); refresh rate; respiration; stability; specific oxygen uptake rate; SOUR; respirometry; self-heating; Dewar; Solvita<sup>®</sup>; ammonia; biologically available carbon

