

# Compost Sampling Plan

## Introduction

Analytical sampling and analysis of biosolids compost is a critical component to regulatory compliance and process efficiency for the permit under which the Hornsby Bend Biosolids Management Plant (HBBMP or the “facility”) operates. This document details the way samples shall be collected as a part of the biosolids reuse contract. This plan is to be followed closely, but with the understanding that complications arise, and flexibility is necessary to be successful in an industrial composting process. Thus, this document will be evaluated and revised by the contractor and CoAWU as necessary with an official review and revision to take place by the anniversary of the execution of the contract (**Appendix A**).

## Definitions

- **AWL** – City of Austin Water Laboratory
- **CoA** – City of Austin
- **CoAW** - City of Austin Water
- **CiCM** - City Compost Manager – person responsible for administrating CoA’s interests regarding the CoAWU biosolids contract. The CiCM is responsible for reviewing contractor work practice, monitoring budgets and ensuring regulatory compliance. The CiCM is the CoA’s primary manager of the biosolids contract.
- **CoCM** - Contactor Compost Manager – person responsible for contractor operational management of the biosolid contract. The CoCM ensures smooth day-to-day operations, manages contractor staff and ensures work is completed efficiently, safely and properly.
- **PFRP** – Process to Further Reduce Pathogens (40 CFR 503 Appendix B)
- **STA** – USCC Seal of Testing Assurance
- **T3** – Time, temperature and turn criteria for PFRP
- **USCC** – United States Composting Council
- **QCS** - Qualified Compost Sampler

## Sampling Personnel

A Qualified Compost Sampler (QCS) is a person who has demonstrated both the technical knowledge and capabilities of compost sampling to the CiCM. Only QCS may sample for laboratory analysis on non-compost materials or compost generated within the biosolids reuse contract. Only the CiCM has the authority to approve a QCS. The CoAWU will train two (2) QCSs, one of which is the CiCM. The contractor will provide three (3) QCSs, one of which is the CoCM.

With proper training, a QCS can be a CoA employee, contractor employee or CiCM-approved 3<sup>rd</sup> party sampler.



## Training

To be considered trained, a QCS must have completed the following action items:

- Read and gain basic comprehension of the City of Austin Water Utility Compost Sampling Plan (this document)
- Read and gain basic comprehension of the TMECC *Field Sampling of Compost Materials* (**Appendix B**)
- Read and gain basic comprehension of TCEQ compost sampling methods in TAC §332.71 (**Appendix C**)
- Pass an informal oral evaluation of sampling protocol by the CiCM
- Observe a QCS conducting two (2) compost sampling events
- Conduct two (2) compost sampling events under the supervision of a QCS without incident or analytical issues.

A log of the trained and approved QCSs is available in **Appendix D**.

Sampling without proper training or qualification will result in invalidation of sample results and necessitate resampling at the contractor's expense. Repeated issues (i.e., two or more) with sample quality may result in the revocation of QCS status by the CiCM and necessitate retraining.

## Pre-sampling Communication/Planning

Effective sampling requires planning and coordination on the part of the contractor, city staff and lab. Due to the batch-specific nature of windrow composting, it is not likely that analytical sampling will occur on a regularly scheduled basis. However, some compost may undergo regular sampling (e.g., monthly STA-compliance sampling)

Temperature, moisture and field-scale stability and maturity (i.e., Solvita) are routine process analysis and does not require scheduling between contractor and CiCM.

Prior to sampling, contractor shall consider current short-term weather, upcoming holidays, employee availability and laboratory availability prior to planning a sampling event.

Upon a compost batch reaching T3 (i.e., 15 days of the three-point temperature average of each windrow > 55 C° and >=5 full windrow turns) contractor shall notify CiCM and AWL contact that the batch is ready for sampling. The full list of analytes for the sampling events are available in **Appendix D**. Contractor will request prospective sampling date via email from CiCM. After approval of date and selected QCS, CiCM will provide contractor QCS with chain of custody, sample containers, and sample cooler. CiCM may choose to observe contractor QCS during sampling or may opt to be the QCS taking the sample. Personnel selection for the sampling process is at the discretion of the CiCM. Sample shall be delivered to the laboratory on ice immediately after sampling has taken place. A post-sampling email will be sent to the CiCM, CoCM and AWL lab contact after the sample has been delivered.



Barring unforeseen problems, no additional communication is necessary until the analytical results have been received from AWL. This typically takes one (1) calendar month from the date of sample submittal. During this interim period, it is advised that the contractor QCS begin the process of Solvita sampling for field-screening of stability and maturity. This is also a routine process analysis and does not require scheduling with the CiCM.

When the batch is conditionally reported as stable and mature by Solvita Sampling, the contractor will notify the CiCM via email to request compost sampling for stability and maturity. This sample may be taken by a 3<sup>rd</sup> party sampler or CiCM QCS for STA analysis. In this case, the CiCM, 3<sup>rd</sup> party sampler and CoCM will arrange a sampling date via email such that all parties may observe if they choose.

## Sampling Methodologies

### Compost Amendment

Any amendments added to the compost for any purpose must be approved by the CiCM prior to addition to the compost process. Contractor shall provide CiCM with Safety Data Sheets or source certification statement for the amendment. Contractor shall provide sample of amendment to CiCM for analytical testing at AWL **at the contractor's expense**. Contractor shall provide written justification detailing process usage and product quality assurance related to amendment. Any change in formulation or type of amendment will require additional approval by CiCM.

### Compost Sampling

Sampling will take place from a compost windrow. Sampling shall only take place after compost piles are sufficiently dry for sampling (e.g., 2-3 days without rain). If prospective material is not in a windrow (e.g., curing pile), CoCM will request that contractor staff take compost material from materials that have met T3 criteria and place in a small windrow (i.e., 100' long). If windrow sampling is not technically feasible, refer to *Test Methods for the Examination of Composting and Compost - Field Sampling of Compost Materials, 2001* and discuss alternate sampling directive with CiCM.

The QCS will generate a set of random numbers from [1 to the length of the compost pile] ( e.g., [www.random.org](http://www.random.org)). These numbers will correspond to the sample cuts locations within the windrow. Even number cuts will be taken from one side of the pile, odds from the other side to reduce sample selection bias. For example, with a 100' wind row running east to west, five random numbers would be generated [2, 37, 43, 49, 86]. QCS will cut into the windrow at 2' and 86' on the north side and 37', 43' and 49' on the south side.

A sharpshooter shovel, precleaned with soapy water and dried will make the pre-measured cuts into the windrow pile from the peak of the pile down to the bottom (See Figure 2.01-B from *TMECC Field Sampling of Compost Materials*).

## 02.01-B SELECTION OF SAMPLING LOCATIONS FOR WINDROWS AND PILES

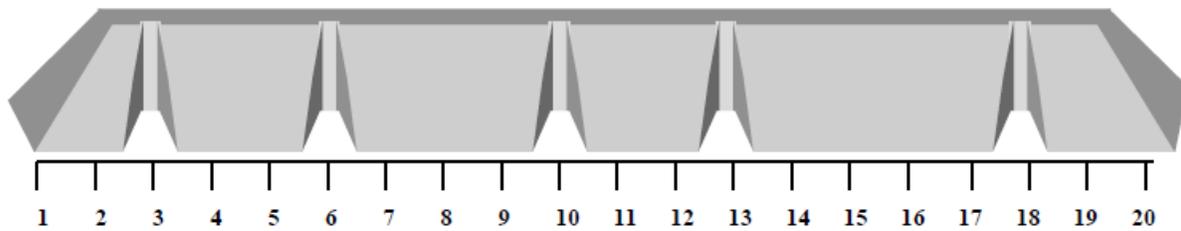


Fig 02.01-B1 Hypothetical sample collection pattern from a compost windrow.

Point samples will be taken from within the sample cuts from the top 1/3, middle 1/3 and lower 1/3. Five samples should be taken from each depth interval using a clean plastic scoop or by hand with nitrile glove. Each point sample will be put into a clean 5-gallon plastic bucket. Upon completion of a sample cut with 15 point samples, mix samples with clean wooden dowel until homogenous. Cap bucket and shake vigorously. Repeat three more times.

Repeat process with remaining cuts until 5 mixed buckets of compost have been produced. Place all 5 buckets worth of sample into large container or on tarp and mix together. This mixture is representative of the compost in the windrow. Recursively quarter sample pile, mixing each subset and re-quartering until sample volume is sufficient to fill sample containers provided by laboratory. Retain approximately 1 gallon of sample for baseline Solvita testing. Discard unused sample back into windrow.

Sample containers shall be labeled using a permanent marker to generate a unique sample name as follows:

**BatchXX\_Year\_Month\_Day\_Time.**

QCS shall complete a chain of custody (**Appendix E**). Samples will be placed in an iced cooler and delivered to the laboratory as soon as possible.

A minimum of two separate sets of compost samples will be performed for each batch. The first sample will be acquired after T3 to ensure PFRP. The second sampling will be after conditional stability and maturity field-screening (i.e., Solvita) have been reported for analytical confirmation of stability and maturity.

An annual compost sample event will take place in September of each year which will be composed of a composite compost sample of all PFRP-compliant compost available from the contractor compost inventory.

Samples sent to a 3<sup>rd</sup> party laboratory will be acquired in the same manner, but may require different sample containers, chains of custody and will likely need to be shipped. Refer to TMECC *Field Sampling of Compost Materials* for shipping guidelines.

Data will be reported to CiCM by AWL and sent to CoCM upon receipt.



## Bulking Agent

Bulking agent shall be sampled by a QCS at the discretion of the CiCM prior to entering the composting process. Contractor should consider the lag between sourcing, sampling and analytical work as it may affect the process workflow. Plan accordingly to prevent disruption. Sampling methodology should be modeled after compost sampling method. Contractor will provide a source certification statement for new or modified bulking agents.

## Temperature & Moisture

Temperature and moisture measurement shall be taken by the contractor daily after activation on every windrow at three locations along the windrow. The temperature and moisture probes will be inserted halfway between the top and bottom of the windrow downward at a 45° angle to the maximum probe depth (36"-48" preferable probe length). The contractor will wait for the readings to equilibrate prior to recording the parameters (on the order of 15-30 seconds). Contractor shall input temperature and moisture data into WEIRS database for compliance tracking (or other CiCM approved data management solution). Contractor shall consistently sample the same side of the material pile (e.g., sample the east side of piles running north-south or equivalent).

To comply with odor, fire and dust plans the contractor shall take representative temperature and moisture samples of non-compost materials in the contractor process areas on a weekly basis. The definition of representative is variable because the configuration of these piles can also be varied. Representative sampling will be determined by a QCS. Non-compost materials include overs, ground yard waste, and any other material stored in large quantities with combustion potential. These measurements shall be recorded in a logbook. Exceedance of safe temperatures and moisture limits shall be immediately reported to the CiCM and CoCM.

Contractor is responsible to maintaining measurement equipment with calibration in addition to analyzing data to check for anomalous readings or trends indicating process problems or safety hazards.

## Field Screening of Stability and Maturity - Solvita Sampling

Laboratory analysis of stability and maturity can be time consuming and expensive. Solvita testing provides a cheap, quick method of semi-quantitative compost maturity and stability testing. This testing shall be completed by a QCS after PFRP has been met both by T3 and pathogen reduction requirements. QCS shall acquire representative sample using the compost sampling method detailed above and take approximately 1 gallon of compost to a sheltered area to process for 24 hours prior to sampling. QCS shall perform ball moisture test (i.e., squeeze compost with gloved hand to see if it retains ball shape and does not extrude water) to determine readiness for analysis. If sample passes ball test, follow TMECC 5.08-E and Solvita manufacturers guide (**Appendices F and G**, respectively) for specific instructions on methodology and operations of the Solvita analysis.



These data shall be tracked within WEIRS or other CiCM-approved tracking mechanism. This analysis shall be completed at a contractor defined interval until compost is shown to be stable and mature. When this occurs, the final analytical sampling may proceed by the QCS so that compost may be marketed and distributed.

## Appendices





## Appendix B - TMECC Field Sampling Guide

Test Method: Field Sampling of Compost Materials. Five Protocols							Units: NA	
Test Method Applications								
Process Management							Product Attributes	
Step 1: Feedstock Recovery	Step 2: Feedstock Preparation	Step 3: Composting	Step 4: Odor Treatment	Step 5: Compost Curing	Step 6: Compost Screening and Refining	Step 7: Compost Storing and Packaging	Safety Standards	Market Attributes
02.01-A	02.01-A	02.01-A	02.01-A	02.01-A	02.01-A	02.01-A	02.01-A	02.01-A
02.01-B	02.01-B	02.01-B	02.01-B	02.01-B	02.01-B	02.01-B	02.01-B	02.01-B
02.01-C	02.01-C							
		02.01-D	02.01-D	02.01-D	02.01-D	02.01-D	02.01-D	02.01-D
02.01-E	02.01-E		02.01-E			02.01-E	02.01-E	02.01-E

## 02.01 FIELD SAMPLING OF COMPOST MATERIALS

### 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/addenda>.
- (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. Source

1.1 This section covers sampling procedures for compost and composting feedstock.

1.1.1 *Method 02.01-A Compost Sampling Principles and Practices* adapted from sampling procedure documents provided by Dr. William F. Brinton, Woods End Research Laboratory, 1996.

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

1.1.3 *Method 02.01-C Sampling Plan for Composted Material*—adapted from the US EPA’s Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition, September, 1986. Consideration and importance was placed on sampling composted solid waste rather than sampling sediments, sludges, or soils for waste analysis. Most information remained unchanged. The majority of the information on sampling was taken from Chapter Nine, Volume II of the U.S. EPA Solid Waste - 846 Manual.

1.1.4 *Method 02.01-D Composting Feedstock Material Sampling Strategies.*

1.1.5 *Method 02.01-E Data Quality Management and Sample Chain of Custody.*

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

ASTM D 5231-92, Determination of the Composition of Unprocessed Municipal Waste. *In Annual Book of ASTM Standards*, Vol. 04.08

ASTM D 4547-91, Sampling Waste and Soils for Volatile Organics. *In Annual Book of ASTM Standards*, Vol. 04.08

A Plain English Guide to the EPA Part 503 Biosolids Rule. US EPA Office of Wastewater Management. EPA/832/R-93/003, September 1994.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. US EPA SW-846. 3rd Edition, September, 1986.

Statistical Quality Control Handbook. Western Electric Company, Inc. 2nd Edition. 1958.

### 3. Terminology

3.1 *aliquot, n*—a sub-sample of a material prepared for, and subjected to laboratory analysis. A sub-sample size smaller than 1 g may be used to represent more than 1000 kg of compost.

3.2 *attribute verification, n*—a laboratory protocol that includes standard reference materials, checks and blanks to validate analytical determinations.

3.3 *confidence interval, n*—a statistical range with a specified probability that a given parameter lies within that range. The magnitude of the range increases as the specified probability is increased.

3.4 *process monitoring, n*—samples collected at predetermined intervals within the composting process to track the targeted changes in biological, chemical and physical characteristics; key process variables in compost piles that should be monitored include porosity, oxygen percent, moisture percent, temperature, retention time or age.

3.5 *process variability, n*—deviations from optimal management procedures of compost production that

may induce deviations in the desired result and sub-optimal finished compost.

3.6 *product variability, n*—heterogeneity of the chemical, biological and physical characteristics of a compost product attributable to both the composting process and the heterogeneity of input feedstocks.

3.7 *representative sample, n*—a sample that accurately reflects the average chemical, biological and physical characteristics of interest from the source of feedstock, bulk material or compost batch in question.

3.8 *sample collection frequency, n*—retrieval of representative samples at intervals that accurately represent the status within the process step of interest for the bulk of compost in question or batch of concern.

3.9 *statistical validity, n*—determinations made from a sample that accurately represent the average characteristics of the compost of interest.

#### **4. Sampling Collection and the Composting Process**

4.1 A generalized model developed to represent the aerobic composting process is presented in Fig 02.01-1 Composting Unit Operations Model.

4.1.1 Market attribute analytical values for a finished compost vary according to the type or blend of composting feedstocks and composting process. Value-added compost products are illustrated in Chapter 01.00 Fig 01.02-A2 Composting Products Model. Sampling and testing plans must be designed to suit the feedstock used in composting, the specific approach to feedstock preparation and composting process management in each composting project, and specifically for each finished product.

##### *4.2 Selection of Sampling Method:*

4.2.1 *Feedstock Sampling Location*—The sampling location for composting feedstock is after feedstock recovery (step 1) has been completed. Feedstock sampling is performed after routine removal of recyclable and/or problem materials. Samples should be taken before feedstock preparation (step 2), i.e., before shredding or size reduction, and before supplemental nutrients, bulking agents or water have been added. The facility operators can provide the best information for the locations to obtain feedstock samples.

NOTE 1—Once the feedstock preparation, (step 2 of the composting process model), is completed, the actual

composting process begins with the material placed in piles, windrows or reaction vessels for composting.

4.2.2 *Prepared Feedstock Sampling*—Samples should be taken after feedstock preparation before composting. Facility operators can provide the best information for the locations to obtain feedstock samples.

4.2.3 *Composting and Compost Curing Process Control Sampling Locations*—The sampling location for process monitoring during composting, step 3, and compost curing, step 6, is indicated in Fig 02.01-B1 Hypothetical Sample Collection Pattern from a Compost Pile.

4.2.4 *Finished Compost Sampling Locations*—Finished compost is expected to match the needs of the customers, and may be obtained from step 3, Composting; step 5, Compost Curing; step 6, Compost Screening and Refining; and step 7, Compost Storing and Packaging as indicated in Chapter 01.00 Fig 01.02-A2 Composting Products Model. Finished compost samples are taken from the actual product that is released for distribution to an end-user.

#### **5. Summary of Methods**

5.1 *Method 02.01-A Compost Sampling Principles and Practices*—Review of sampling design schemes adapted from sampling procedure documents provided by Dr. William F. Brinton, Woods End Research Laboratory, Inc.

5.2 *Method 02.01-B Selection of Sampling Locations for Windrows and Piles*—Descriptions of sample collection as sets of compost sub-samples collected and combined to represent the average chemical, physical and biological characteristics of the compost material for a batch windrow or pile of cured or curing compost.

5.3 *Method 02.01-C Sampling Plan for Composted Material*—Review of US EPA SW-846 sampling plan guidelines and statistical procedures for estimating required minimum number of samples.

5.4 *Method 02.01-D Composting Feedstock Material Sampling Strategies*—A representative sample of feedstock is collected to identify its chemical and physical characteristics.

5.5 *Method 02.01-E Data Quality Management and Sample Chain of Custody*—Consideration for third-party sample collection and preparation. Also, an example form and description of the parameters needed for a chain of custody report.

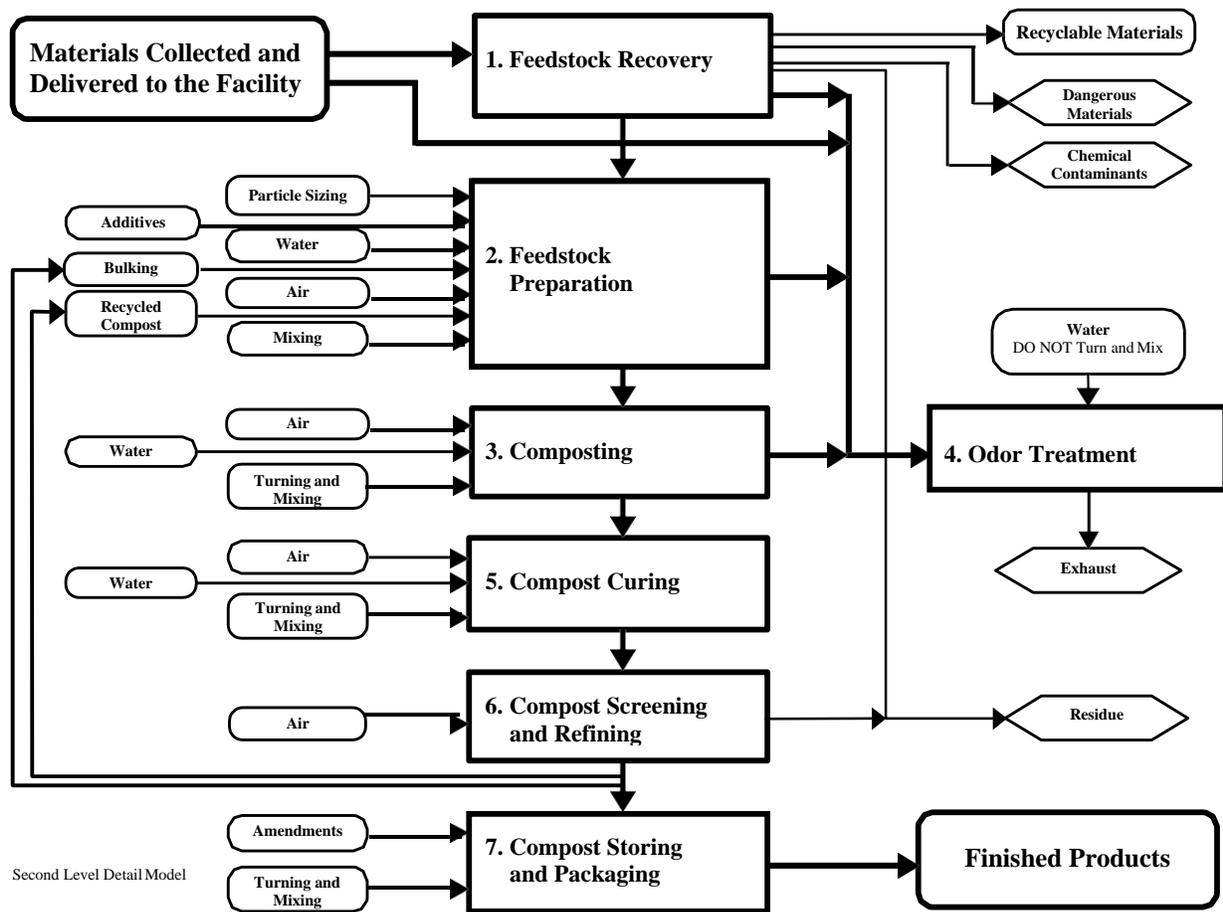


Fig 02.01-1 Composting Unit Operations Model.

## 6. Significance and Use

6.1 *Method 02.01-A Compost Sampling Principles and Practices*—Source of general guidelines and considerations needed to develop an appropriate compost sampling plan.

6.2 *Method 02.01-B Compost Material Sampling Strategies*—A general guide for compost sample collection and preservation from compost curing piles.

6.3 *Method 02.01-C Sampling Plan for Composted Material (from SW-846 Chapter Nine, part 1)*—The initial, and perhaps most critical element in a program designed to evaluate the physical, chemical and biological properties of a compost is the plan for sampling the material in question. It is understandable that analytical studies, with their sophisticated instrumentation and high cost, are often perceived as the dominant element in a characterization program. Yet, despite that sophistication and high cost, analytical data generated by a scientifically defective sampling plan have limited utility.

6.4 *Method 02.01-D Composting Feedstock Material Sampling Strategies*—A general guide for feedstock sample collection. Specific methods should be modified for differing feedstock materials.

6.5 *Method 02.01-E Data Quality Management and Sample Chain of Custody*—A method of tracking a collected sample from date, time and location of sampling through completion of laboratory analysis.

## 7. Interference and Limitations

7.1 Analytical error associated with sampling and handling is compounded when multiple properties with conflicting sampling needs are measured from the same sample. For example, it is a good idea to subdivide and remix samples repeatedly if mineral and metal tests are being performed. This improves homogeneity and reduces sample variance. Unfortunately, this same method induces excessive volatilization of some of the compounds, and causes microbial cross-contamination. Therefore, the sampling plan must specify a separate sampling and handling scheme for each test parameter that requires special sampling.

**Sample Collection and Laboratory Preparation**  
*Field Sampling of Compost Materials 02.01*

**7.2 Method 02.01-B Compost Material Sampling Strategies**—As compost heterogeneity increases, the number of sub-samples should be increased. If insufficient numbers of samples are collected, analytical results will not represent the compost in question.

7.2.1 Moisture loss or gain during sample handling and splitting may become significant. It is therefore necessary to mix and split a sample under sheltered conditions, such as inside a building where wind, temperature and sunlight or precipitation will not distort the compost moisture.

**7.3 Method 02.01-C Sampling Plan for Composted Material**—Knowledge of or access to statistical procedures is required.

**7.4 Method 02.01-D Composting Feedstock Material Sampling Strategies**—Sample heterogeneity of feedstock may be much higher than that of the finished composted product. It is crucial that all sampling plan collection procedures are followed to maximize the reliability and accuracy of the feedstock sample analytical results.

7.4.1 Moisture loss or gain during sample handling and splitting may become significant. It is therefore necessary to mix and split a sample under sheltered conditions, such as inside a building where wind, temperature and sunlight or precipitation will not distort the feedstock moisture.

**8. Sample Handling**

8.1 Collect samples from areas of the compost pile that are representative of the general appearance, and avoid collecting atypically moist samples (> 60% moisture, wet basis). If balls form during the process of blending and mixing of point-samples, the compost sample is too wet. Excessively moist compost will cause unreliable physical and biological evaluation.

8.2 For most feedstock or compost samples, use containers made of stainless steel, plastic, glass or Teflon. These materials will not change compost chemical quality. Laboratories provide advice on appropriate sample containers, preservatives and shipping instructions when requested.

8.3 A representative compost sample must be collected from appropriate sampling locations and consist of no less than 15 point-samples. Sampling locations along the perimeter of the compost pile where compost point-samples will be extracted and vertical distances from the ground or composting pad surface shall be determined at random, and shall be representative of the compost on the site.

8.3.1 Determine the number and types of sampling and shipping containers to be used. The composite sample is placed in a sanitized container and thoroughly mixed. Follow proper quality assurance/quality control procedures for sample preservation, storage, transportation and transfer. Sample the cured compost and aliquot 12 L (3 gal) sub-samples from the composite sample and place in a sanitized plastic container and seal.

8.3.2 Utilize the Student's "t"-test with a confidence interval of 80% to statistically analyze the test data. Refer to TMECC 02.01-A, paragraph 9.10 *Sampling Intervals* for guidance in determining sample collection frequency.

8.4 *Test Methods versus Sampling Methods*—The laboratory test method and analytical parameter of interest dictate the method of sample collection, type of container for shipping and storage of samples and sample handling procedures required. Table 02.01-1 provides a partial list of analytical traits that are affected by sample collection and handling. In general, volatile compounds and elements, physical bulk factors and microbiological samples require special considerations when developing the sampling plan.

Table 02.01-1 Partial list of test parameters that require special sampling and handling considerations.

<i>Test Parameter</i>	<i>Principle Constraint</i>	<i>Associated Error</i>	<i>Alteration of Sampling for Corrective Action</i>
Total-N	Volatilization loss of NH <sub>3</sub> during sample handling	Underestimation of total N and volatile N	Place in container quickly with minimal stirring
Volatile fatty acids (VFA)	Volatilization loss of VFA during sample handling	Underestimation of VFA content	Place in container quickly with minimal stirring
Microbiology (pathogens)	Contamination from tools, buckets, air	Over or under estimation of pathogens	Use only clean, sterile containers and implements
Bulk Density	Excess sample moisture	Overestimation of volume/weight	Take large, oversized samples

8.4.1 In each case the determination for a trait of interest can be changed adversely by improper sample collection and handling, and consequently lead to erroneous conclusions. Analytical precision or relative variability may not be affected by inappropriate

sampling, but accuracy of the expected determination may be biased and incorrect.

8.5 *Containers, Post-Sample Handling*—For each type of parameter measured after sampling specific containers and holding times should be observed prior

to and during transport to a laboratory (see Tables 02.01-2 through 02.01-6). Use multiple containers to preserve sample integrity as necessary.

8.5.1 Despite the wide variation in sample holding times and condition requirements, all compost samples targeted for general testing should be chilled immediately upon collection and preparation. Refer to Tables 02.01-2 through 02.01-6 to find the most appropriate storage temperature for each test parameter of interest.

8.5.2 When plastic containers are acceptable, use double Ziploc®-type 4-8 L (1-2 gal) bags marked on the exterior with a marking pen with insoluble ink, and placed with several cool-packs in a large polystyrene cooler or similar insulated container.

8.5.3 Ship the samples to the laboratory for delivery within 24 h or less. Request that the laboratory staff

store samples at 4°C when delays in lab preparation are anticipated.

8.5.4 Collection and storage of samples for organic compound analysis - polychlorinated biphenyls (PCBs), polynuclear aromatic hydrocarbons (PAHs) or volatile fatty acids (VFAs) - require glass containers with Teflon lids, or exclusively Teflon containers. Sample containers should be filled to overflowing with material to minimize airspace in the container and reduce volatilization of organic compounds during storage.

8.5.5 Include proper *Chain-of-Custody* information: date, time, name of the sampling entity and name individual responsible for sample. Refer to *Method 02.01-E Data Quality Management and Sample Chain of Custody* for an example form and description of parameters needed to complete a chain of custody report.

Table 02.01-2 *Physical Parameters: Sampling containers and conditions for compost and source ingredient testing.*

<i>Test Parameter of Interest</i>	<i>Container</i>	<i>Conditions</i>	<i>Maximum Holding Time Allowed in Lab</i>
Bulk Density, Hydraulic Conductivity, Porosity, Water Holding Capacity	P, G	4°C	7 d
Temperature	NA	NA	Immediate, no delay
Total Solids	P, G	4°C	24 h

NOTE 2—P=Plastic; G=Glass

Table 02.01-3 *Organic and Biological Properties: Sampling containers and conditions for compost and source ingredient testing.*

<i>Test Parameter of Interest</i>	<i>Container</i>	<i>Conditions</i>	<i>Maximum Holding Time Allowed in Lab</i>
Respirometry	P, G	4°C	24 h
Organic Carbon	P, G	4°C	14 d
Volatile Fatty Acids	G (2 L CWM)	4°C	14 d
Volatile Solids	P, G	4°C	14 d

NOTE 3—P=Plastic; G=Glass

Table 02.01-4 *Chemical Parameters: Sampling containers and conditions for compost and source ingredient testing.*

<i>Test Parameter of Interest</i>	<i>Container</i>	<i>Conditions</i>	<i>Maximum Holding Time Allowed in Lab</i>
Acidity/Alkalinity (pH), Electrical Conductivity, Kjeldahl Nitrogen, Nitrate Nitrogen (NO <sub>3</sub> -N), Nitrite Nitrogen (NO <sub>2</sub> -N), Ammonia Nitrogen and Ammonium Nitrogen (NH <sub>3</sub> -N, NH <sub>4</sub> -N), Sulfide	P, G	4°C	48 h
All other Metals	P, G	4°C	6 months
Chloride, Sulfate	P, G	4°C	28 d
Chromium VI	P, G	4°C	24 h
Mercury	P, G	4°C	28 d

NOTE 4—P=Plastic; G=Glass

**Sample Collection and Laboratory Preparation**  
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Table 02.01-5 *Pathogens*: Sampling containers and conditions for compost and source ingredient testing.

<i>Test Parameter of Interest</i>	<i>Container</i>	<i>Conditions</i>	<i>Maximum Holding Time Allowed in Lab</i>
Enteric Virus	G	-70°C	> 8 h
Enteric Virus	SP, G	4°C	8 h
Coliforms and other bacteria	SP, G	4°C	48 h
Helminth Ova	SP, G	4°C	1 month

NOTE 5—SP=Sterilized Polypropylene; G= Sterilized Glass

Table 02.01-6 *Synthetic Organic Compounds*: Sampling containers and conditions for compost and source ingredient testing.

<i>Test Parameter of Interest</i>	<i>Container</i>	<i>Conditions</i>	<i>Maximum Holding Time Allowed in Lab</i>
Chlorinated Herbicides, and Chlorinated Hydrocarbons, PCB	G, Teflon lined cap (2-1/2 L.A.J.)	4°C	7 d until extraction
Chlorinated Pesticides	16 oz B.R. (2-1/2 L.A.J.)	4°C	7 d until extraction
Dioxins & Furans, Nitroaromatics and Isophorone, and Polycyclic Aromatic Hydrocarbons, PAH	G, Teflon lined cap (2-1/2 L.A.J.)	4°C store in dark	7 d until extraction
Phthalate esters	G, Teflon lined cap	4°C	7 d until extraction
Purgeable aromatic hydrocarbons	G, Teflon lined septum (40-mL Glass V)	4°C	14 d prior lab testing
Semi-Volatile Organics	G, Teflon-lined Septum (2.5-L Jug)	4°C	7 d
TCLP Sample	G, Teflon-lined Septum (2.5-L Jug)	4°C	7 d until extraction
Volatile Organic Compounds (VOC)	G, Teflon lined septum (40-mL Glass V)	4°C	14 d preserved in HCl <sup>†</sup>

NOTE 6—P=Plastic; G=Glass, HDPE=High Density Polyethylene

†—Evaluation data is being sought to confirm this requirement for curing and finished composts.

Test Method: Compost Sampling Principles and Practices							Units: NA	
Test Method Applications								
Process Management							Product Attributes	
Step 1: Feedstock Recovery	Step 2: Feedstock Preparation	Step 3: Composting	Step 4: Odor Treatment	Step 5: Compost Curing	Step 6: Compost Screening and Refining	Step 7: Compost Storing and Packaging	Safety Standards	Market Attributes
02.01-A	02.01-A	02.01-A	02.01-A	02.01-A	02.01-A	02.01-A	02.01-A	02.01-A

## 02.01-A COMPOST SAMPLING PRINCIPLES AND PRACTICES

COMMENT—This section was adapted from sampling procedure documents provided by Dr. William F. Brinton, Woods End Research Laboratory, 1996.

### 9. Justification for Compost Sampling

9.1 Sampling of compost and compost products is an essential aspect of process monitoring, quality control, marketing and labeling, and regulatory compliance. Like other functions of site management, sample collection involves carefully planned and often labor intensive activities. Four common reasons for compost sampling are described:

9.1.1 *Ingredient Analysis*—basic data on source ingredients are needed for the design of a composting process or identification of an optimal composting feedstock recipe.

9.1.2 *Process Design and Monitoring*—composting process evaluation requires information on material characteristics and process benchmarks. Specific sample collection protocol is designed for each parameter of interest.

9.1.3 *Marketing and Labeling*—specification sheets or product labels for compost are needed to compare product with others in the marketplace.

9.1.4 *Regulatory Compliance*—compost process and product requires periodic testing for compliance with specified traits including certain metals, pathogens, stability and maturity.

9.2 *Use of Sampling Data*—Sampling decisions require an understanding of the need for data collection, specifically how to sample and when to collect samples. The sampling decision tree presented in Fig 02.01-A1 illustrates a decision process to assist in the development of proper sample collection methods, to identify sampling interval and sample size, and the end use of sample data. When regulations do not apply, as is the case for recipe formulation, process monitoring for quality assurance (QA) and internal quality control (QC), it is important to clearly understand the intended use of the data and to determine the appropriate sampling procedures. For example, if C:N ratio interpretation is considered very important, then very low variations in sample carbon and nitrogen determinations become a major

consideration and a sample collection process must be designed to support to this requirement.

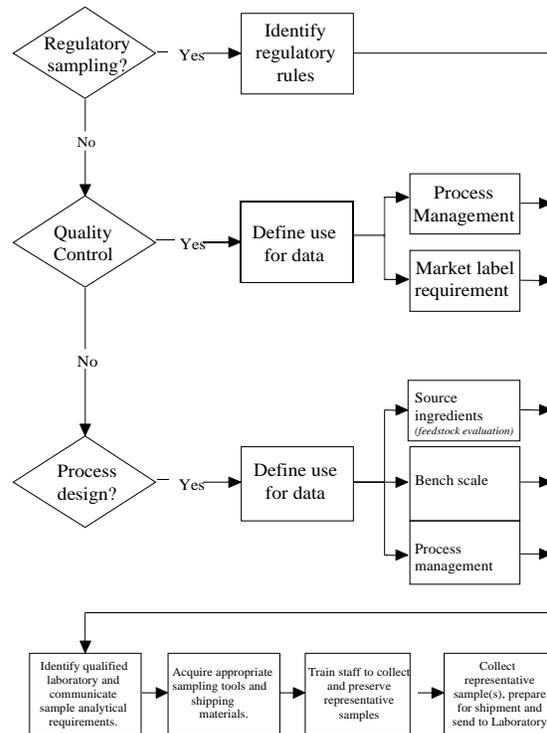


Fig 02.01-A1 Compost sampling decision tree, overview of sampling objectives.

9.3 *Types of Sampling*—Two types of sample collection are used: point-sampling and composite-sampling:

9.3.1 *point sampling*—site-specific sample collection from within the general mass is used to identify and quantify points of extreme variability, hot spots or problem zones. Point-sampling alone should not be used unless special conditions exist.

9.3.2 *composite-sampling*—a single sample for laboratory analysis composed of multiple, well-blended point- or sub-samples uniformly distributed throughout the entire volume that, after mixing, accurately represents an average or median value of the property or trait of interest for a batch or general mass. Properly implemented composite sampling is preferable for most sampling plans because it provides a reliable estimate

of the average or median property or trait of a batch or segment of a continuous stream, rather than a specific spot trait.

9.3.2.1 *stratified sampling*—a modified composite sampling scheme is used to document gradients and define heterogeneity as a function of position within the bulk or general mass of sampled material, where the general mass is subdivided into separate zones and a series of point-samples are collected and composited within each zone. Stratified sampling should be used when heterogeneity of compost is unknown and when regulatory constraints require knowledge of the relative spatial and temporal variability. This is most often based upon the standard deviation and mean; refer to Method 02.01-B for equations applied in calculations for approximating the required number of sub-samples to accurately estimate the average value for the parameter or trait of interest.

9.3.2.2 *interval sampling*—sampling from moving conveyor belts.

9.4 *Sampling Plan*—The constraints of the material and the composting technology must be considered when an optimal sampling plan is designed. Combinations of composite and point sampling are illustrated within the four sampling schemes presented in Fig 02.01-A2. The sampling scheme selected must address limitations of the selected test parameter and should not distort the analytical result.

9.4.1 Stratified sampling (Scenario A, Fig 02.01-A2) is used to determine variability, profile gradients and spatial uniformity characteristics. In most cases, composite sampling (Scenario B, Fig 02.01-A2) is satisfactory when the amount of variability within the mass is known to be insignificant. It involves combining several representative sub-samples into one composite sample that is then thoroughly mixed, then split for shipment to the laboratory. Area or batch sampling (Scenario C, Fig 02.01-A2) and single grab-or point-sampling (Scenario D, Fig 02.01-A2) are for special cases where one sample is collected at one location. Area or batch sampling is typified by a whole mass collected as one sample unit. This method is most appropriate when moving the mass from a vessel to a curing pile. A single point-sample does not provide a representative sample for the bulk mass. Batch sampling and point sampling should be employed to characterize an obvious or potential anomaly at one specific point, time or location within a process. A good example of a single point sample to detect anomalies is shown as X in Fig. 02.01-A2 D, a location referred to as the “toe” of a static aerated pile, and one which is vulnerable to suboptimal temperatures needed to achieve pathogen reduction. For this reason, it is sometimes specifically included to verify pathogen content of compost that has finished the thermophilic phase.

9.5 *Importance of Representative Sampling*—A representative sample defines a material’s average characteristic, typical for the entire material being sampled. Under virtually all composting conditions, the mass of compost material is large and heterogeneous. A representative sample of compost is not easily obtained; and sampling must be repeated over time to compensate for naturally high variations. Under proper management and as compost-curing advances, variability within a curing pile or windrow will decrease.

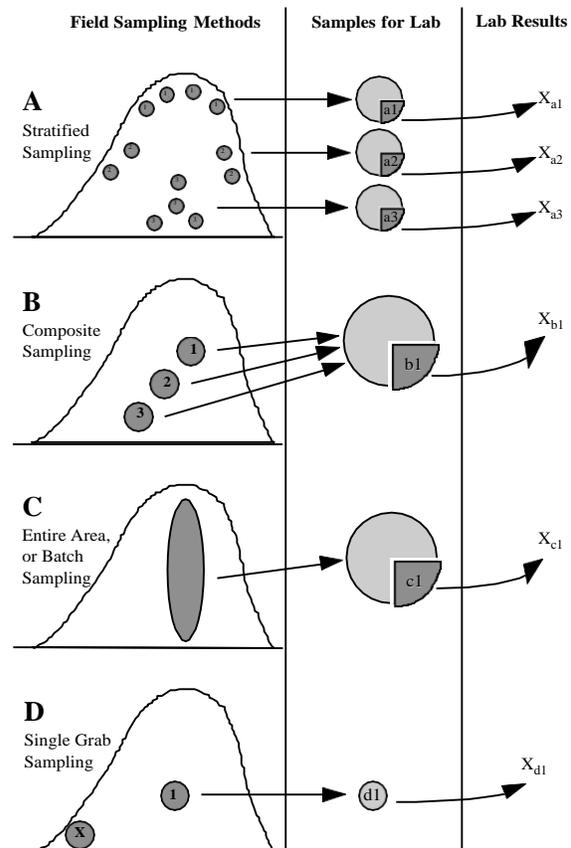


Fig 02.01-A2 The sampling schematic.

9.6 *Variables that Compromise Quality of Sampling*—Sample collection technique and variability of compost and cured compost affect the relative accuracy of sampling and the reliability of laboratory analytical determinations. Failure to adjust sampling protocols according to the nature and source of variations may invalidate test results and lead to inappropriate management or marketing decisions.

9.6.1 *Bias Introduced by the Sampler*—Inaccurate sample collection is often due to systematic or intentionally selective sampling introduced by the sampler. Significant error will result from attempts by the sample collector to counteract perceived variability. Examples include avoiding the collection of sub-

samples from wet pockets or systematically excluding large particles from the composite sample. **Deliberate bias results from an attempt by the sampler to prepare samples that appear superior in a perceived physical trait that does not actually represent the bulk or batch of interest.**

9.6.2 *Sample Heterogeneity*—The following are key sources of non-uniformity that can give rise to significant sampling errors.

9.6.2.1 *Sub-sample size* affects sampling accuracy. In general, a representative composite sample contains large (> 1000 cm<sup>3</sup>) and plentiful sub-samples (>15 samples).

9.6.2.2 *Complete and thorough mixing* throughout the composting process improves the quality and ease of sampling. Poor initial mixing effects variability of the parameters throughout the composting process. Repeated use of turning machinery during composting improves homogeneity. However, within days or even hours after turning, mixing or re-piling, the composting mass may develop gradients of stability, moisture, bacteria and ammonia. When pre-mixing, blending or turning are not employed, as in static pile composting or compost curing, the sampling plan should include more sub-samples per composite sample to compensate for inherently high variability within the mass.

9.6.2.3 *Soil and stones* are frequently picked up during routine compost production operations. These pose problems for good sampling. In some cases, the sampler may bias the sample by deliberately excluding gravel and stones present in a compost (soil can not be easily seen). On the other hand, a laboratory that receives a sample containing stones or small gravel may not sub-sample, pre-screen, and grind, resulting in variable results. Staff responsible for sampling must correctly diagnose the situation and advise the analytical laboratory about it. In some cases, laboratories must issue disclaimers about their own sub-sampling technique.

9.6.2.4 *Foreign and non-compostable matter* almost invariability poses problems to the sampler, and also the laboratory. This is most likely the case with municipal solid waste (MSW) and certain industrial by-products where large and variable amounts of such substances are present. The best approach is to take large sub-samples and blend frequently before removing the final sub-sample for examination or testing. There is presently no generally accepted or standard practice for gauging the minimum sample size required in such situations.

9.6.2.5 *Varying particle size* is one of the most common sources of sample variability. For example, a composting feedstock mix may have exactly 27% wood chips, but inability to sub-sample adequately could result in finding anywhere from 11 to 38% wood chips.

The error introduced to C:N values for samples of this range is significant.

9.6.2.6 *Layering, compaction and gradients* of composts arise as a result of inadequate initial mixing, infrequent or excessive turning/mixing during feedstock preparation, or during the composting process because of equipment/ventilation actions such as inappropriate selection and use of bulking materials. Any one or more of these can easily confound sampling attempts.

9.7 *Sampling Practice*—Sampling begins with the decision to evaluate materials and proceeds to determining how and in what time frame the sample is needed. Practical steps include identifying the important parameters to be analyzed and working backwards through the decision tree to identify how to obtain a suitable sample for the specific technology and parameter of interest. Following this process, a sampling protocol and sample log is constructed. Technological constraints sometimes present significant challenges for sampling, however, in most cases, reliable samples can be obtained once a thorough analysis of the process plan is conducted.

9.8 *Composting Technology Systems and Sample Collection*—The physical/mechanical nature of the feedstock preparation and composting operation may impose constraints on sampling. Each composting technology imposes specific limitations on sampling. Representative samples may not be obtainable with some technologies. Therefore, a facility's sampling plan must take into account the realistic strategy for obtaining representative samples. In general, highly engineered compost processes impose more constraints on sampling than a simple composting process. For example, outdoor windrows are more easily sampled than large rotating drums.

9.8.1 Ten basic types of composting systems are presented in Fig 02.01-A3 and their associated sampling constraints are outlined in Table 02.01-A1. Each system introduces particular traits or constraints that impact how (and why) samples are collected. New forms of compost technology under development may expand the list, but the generic form of the prescribed models cover most existing composting technologies.

9.8.2 *Sampling Plan Basics*—The two process-focused modes of compost sampling are: i) In-Process sampling for monitoring during a specific composting technology process; and ii) End-Process sampling. There may be multiple steps or multiple processes involved in an overall system. Sample collection for testing commonly occurs at the end of a specific step of the composting process, mostly for convenience and to be certain that the sample is representative of the batch. Sample collection during a process imposes significant constraints because of the inherent variability of in-process materials. Sampling at these points must be

carefully designed to sample across any existing gradient of non-uniformity.

9.8.3 Discussion in the following section identifies technologies and primary constraints or requirements for representative sampling.

9.8.3.1 *Type A. Home Bins* come in many shapes and sizes, from fixed solid containers, loose wooden structures to rotating solid-tanks. The appropriate framework for sampling is to select the material representing the finished product. Some systems provide doors at the bottom of a bin from which samples may be easily removed; other bins require disassembling or removal from the pile and hand-mixing of the mass. Precaution must be taken to assure a homogenous mixture under any circumstance.

NOTE 7—The inclusion of home composting bins in TMECC is not a suggestion or endorsement for regulatory control, but for information and perspective only. While home composting bins are not a mainstay of commercial composting and not currently or likely to be regulated by state or local jurisdictions when the end product is used by the home generator and producer, the principles described in TMECC for assessing overall quality of compost are suitable for use on such products.

9.8.3.2 *Type B. Turned Windrows* are either batch or continuous piles. In the former common case, the entire windrow is made from similar ingredients at about the same time (e.g., within 3 d). In the latter case, materials are added lengthwise over time. In both cases, non-uniformity is observed down the length of the pile and is greatest with continuous modes of composting. Sampling of windrows requires compositing over a discrete length, either the entire pile, or a sub-section identified to have similar age or other characteristics. Windrow turning machines are useful for preparing uniform mixtures suitable for composite sampling; however, a single pass with a turning machine will not result in an evenly mixed pile, 3-4 passes commonly are required. If turning is performed frequently, the need for multiple turns prior to sampling diminishes.

9.8.3.3 *Type C. Static Piles* are recognized for their non-uniformity. These piles exhibit gradients of temperature, aeration and exposure to elements that reduce homogeneity over time. To obtain a representative sample from a static pile, extreme disruption and mixing is required. Breaking down the pile with a bucket loader and re-mixing after removal of the outer cover may be necessary. If mixing is not complete, sub-samples should be taken from each region during pile breakdown, or from the bucket as material is removed. However, if the purpose of sampling is to characterize non-uniformity, then effort must be made to get to the region of concern where a representative sample can be collected. This could be performed using a core sampler, or by breaking open the pile with heavy equipment.

9.8.3.4 *Type D. Agitated-Bed* systems generally move compost along the length of the system at a fixed rate per day. Should sampling be necessary during the process, care must be taken to understand the variability imposed by nature of daily additions to the system. In some cases, the actual technology physically restricts access for various reasons including worker safety. In such situations, samples can be collected at the discharge end where material comes off the bin. Several sub-samples should be taken each day, cooled immediately; and several days' accumulated samples (except for bacteriological and others parameters limited by a 48 h holding time) can be composited to form a bulk sample.

9.8.3.5 *Type E. Enclosed Vessel* reactors are either circular or oblong containers, bins or towers (these systems may or may not contain internal moving parts) and cannot be easily accessed for sampling. Sample collection is best performed at the vessel's discharge end. In-process sampling for quality control and process monitoring is not always practical with these systems.

9.8.3.6 *Type F. Rotating Vessels* are horizontal tanks, usually positioned on a gradient. They are used for continuous and sometimes for batch composting. Most systems do not have ports to access the material during processing, making in-process sampling impractical. As with the enclosed vessel design, sampling is usually performed at the discharge end of the vessel. Rotating vessels are often used during "Feedstock Preparation" for many technology types, and sampling is performed on the download conveyor.

9.8.3.7 *Type G. Cure Piles* are frequently very large and may contain material composited from several piles. Because of their heterogeneity and size, and the typical lack of turning and mixing, they usually display extreme gradients of moisture, maturity and bulk density. Under these circumstances, one effective way to adequately sample is to use a large tractor loader to break into the pile, moving and mixing the materials in the process. The sampling plan must incorporate a stratified sampling scheme and point sampling to distinguish gradients and map spatial non-uniformity.

9.8.3.8 *Type H. Bagged Product* results from a mixing and screening process that is assumed to produce uniform material prior to bagging. Additional mixing of the bulk mass after bagging and prior to sampling is precluded. Therefore, a statistically representative sample must consist of many sub-samples collected from different bags. Additionally, the physical constraint of extracting small sample cores from separate bags that are palletized compounds the problems of collecting proper samples.

9.8.3.9 *Type I. Source Ingredients* are notorious for non-uniformity. Large sub-samples that accurately

represent the distribution of ingredients must be well mixed, and if possible (when appropriate), shredded to reduce the sample size while retaining sample integrity. Large mechanical equipment may improve the sample collection and preparation process.

9.8.3.10 *Type J. Lab Systems* are a special case of composting and are usually handled as a discrete sampling problem on an individual institutional basis. However, with the increasing popularity of bench scale testing, particularly for bioremediation composting, the value of describing sample units and types becomes

more obvious. In general, these units contain highly uniform materials and are sometimes so small that the entire unit becomes the sample from which sub-samples are drawn for separate analyses. Because non-uniformity increases with miniaturization, lab systems are usually designed with small openings into discrete sections of tanks to facilitate extraction of small sub-samples. This allows the operator to monitor the formation of gradients and non-uniformity in miniature lab systems.

Table 02.01-A1 Sampling operations, constraints and required tools for ten types of composting technologies.

<i>Type</i>	<i>Sampling Action</i>	<i>Constraints</i>	<i>Preferred Tools</i>
<i>A. Home Bins</i>	Must open bin, remove cover and sides, and mix by hand	Not homogenous, may be hard or impossible to open	Pail and spading fork
<i>B. Turned Windrows</i>	Sample after turning with machine from surface of pile if well mixed	Pile varies along length, turning machine may not homogenize in one pass	5-gal pail, spading shovel, corer
<i>C. Static Piles</i>	Remove chip cover, and dig into depth, may require bucket loader and multiple depth sampling	Extreme non-uniformity, layering and clumping, inadvertent mixing with cover or surface residues; may be sealed inside tube	5-gal pail, spading shovel, corer or auger, bucket loader
<i>D. Agitated-Bed</i>	Sample after turning or agitation event, or sample discharge	Difficult access except at discharge, piles vary along length with age of source	5-gal pail, spading shovel,
<i>E. Enclosed Vessel</i>	Sample from side doors or top port after agitation	Very difficult or impossible access; potential layering	5-gal pail, spading shovel, corer, auger
<i>F. Rotating Vessels</i>	Sample from discharge/output end or take-away conveyor	Difficult or impossible to sample except at discharge; output varies with time	5-gal pail, shovel or scoop
<i>G. Compost Curing Piles</i>	Remove chip cover, and dig into depth, may require bucket loader and multiple depth sampling	Very large piles, non-uniformity, difficult access, compaction and layering; surface cover mixing	5-gal pail, spading shovel, corer, auger, bucket loader
<i>H. Bagged Product</i>	Sample multiple bags, cores drawn	Bag damage, difficult access	5-gal pail, trowel or soil-corer
<i>I. Source Ingredients</i>	Composite from each pile separately, remove surface	Non-uniformity may be great, poorly mixed, difficult access	Large pail, shovel; bucket loader
<i>J. Lab Systems</i>	Open system and remove with core sampler	Small scale, difficult access	5-gal pail, Spatula, trowel, soil-corer

9.9 *Sampling Interval*—There are no process-specific formulas that dictate sampling intervals for source ingredients and compost, except when biosolids are composted (Table 02.01-A2). Sampling intervals of composting materials for reporting purposes may be fixed by certain regulations. It is advisable to consult local or state sampling guidelines. As a general rule, incoming feedstocks should be sampled every two weeks, or every 3,000 to 5,000 tons of finished product.

9.9.1 *Formula to estimate sampling interval, d:*

$$S = T \div F \times R \quad \text{Equation 9.9.1}$$

where:

- S = sampling interval in days, d
- T = sampling threshold in tons (e.g., 4,000 t), t,
- F = tons of incoming feedstock per day, t d<sup>-1</sup>, and
- R = weight reduction factor of incoming feedstock, %.

9.9.2 *Weight Reduction Factor, R:*

$$R = C \div F \quad \text{Equation 9.9.2}$$

where:

- R = weight reduction factor of incoming feedstock, %,
- C = mass of finished compost per week, t dw, and
- F = mass of incoming feedstock per week, t dw

NOTE 1A—If the actual weight reduction factor is unknown, use 0.70 until the actual value can be determined. Refer to Method 03.09 Total Solids and Moisture for a description of how to determine dry weight of compost and feedstocks.

Table 02.01-A2 Sampling intervals for composted biosolids.

<i>Amount produced</i> (metric tons of biosolids compost per 365-day period)	<i>Monitoring Frequency for</i> <i>Pathogens and Trace Elements</i>
< 290	Once per year (1 yr <sup>-1</sup> )
≥ 290 to < 1,500	One per quarter (4 times yr <sup>-1</sup> )
≥ 1,500 to < 15,000	Once per 60 days (6 times yr <sup>-1</sup> )
≥ 15,000	Once per month (12 times yr <sup>-1</sup> )

*Adapted from US EPA 40CFR503*

**Sample Collection and Laboratory Preparation**  
*Field Sampling of Compost Materials 02.01*

9.9.3 *Sampling raw source ingredients—Example 1.* Samples shall be taken from incoming material that has been shredded, tumbled or otherwise reduced in particle size. From the material exiting the shredder/mixer, one point-sample shall be obtained every 2 h, over an operational period of 6-8 h, for a total of 4 samples. Sample size should be approximately 1000 cm<sup>3</sup> (~ 1 qt) per sample. The four samples shall then be thoroughly mixed together (composite), and a portion of the mixture (composite sub-sample) taken for analysis. If point-sampling directly from the shredder or mixing mill is not possible, the incoming material shall be sampled no more than 24 h after passing through the shredding equipment.

9.9.4 *Example 2—Sampling compost materials.* For each sampling event, a single composite sample shall be made up of multiple sub-samples for each pile or batch, unless otherwise directed.

9.9.5 *Example 3—Sample locations.* Construct and label a diagram of sample locations for your composting system. The example provided in TMECC 02.01-B indicates a minimum of fifteen sub-samples per pile. This procedure establishes a composite or general characterization of the attributes in a compost pile.

9.9.5.1 Refer to section 02.01-B for a strategy to sample generic windrows of compost.

9.9.5.2 Samples collected during the composting process are not composited in the same manner as finished samples because point-specific problems must be identified and monitored. Factors such as anaerobic materials and volatile fatty acids (VFA) may need to be determined from point-samples extracted from multiple locations in the same pile.

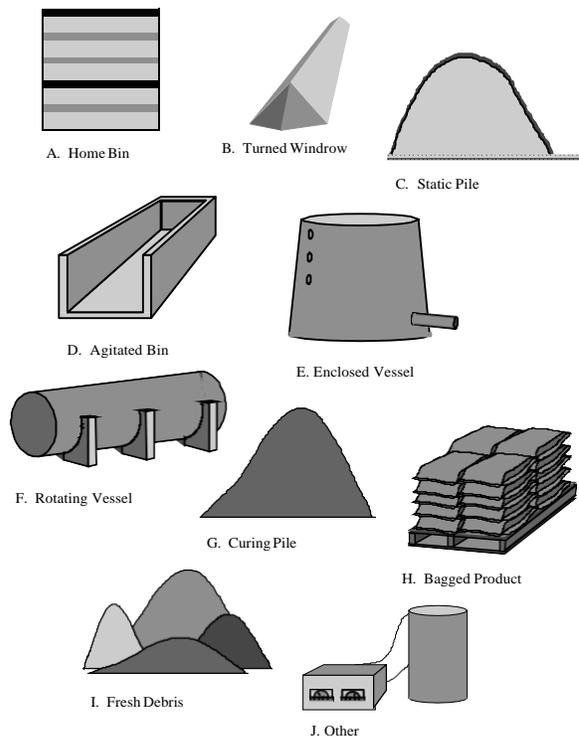


Fig 02.01-A3 Composting technologies.

9.9.6 *Example 4—Sample Variance Exercise.* The coefficient of variation (CV) expresses the relative variability for a parameter of interest across multiple samples. The CV is expressed as a percentage and calculated by dividing the sample standard deviation by the sample mean and multiplied by 100.

9.9.6.1 The ability to distinguish differences between arithmetically similar sample values decreases as the CV increases. It is difficult to draw specific conclusions about analytical results when variability is high. Under circumstances where variability is consistently high either the sampling plan must be redesigned to account for the excessively high variability, or the parameter should be discarded as a standard measure.

9.9.6.2 Consider a hypothetical case where two standard parameters are used to evaluate compost stability, C:N and VFA. Assume that the upper limit of acceptable variability for the parameters are set at 15% for C:N, and 45% for VFA. Low CV thresholds are generally assigned to system and process critical measures, and high CV thresholds are assigned to less critical standard measures.

NOTE 2A—This is a hypothetical case. It may be very difficult to establish meaningful CV limits without a large amount of data from many composts across time for a given test parameter. In addition, depending on the test, an individual test parameter may show a very large CV for repeated analysis of one sample.

9.9.6.3 In the example given in Table 02.01-A2, the CV for VFA testing is greater than the CV for C:N analysis, but the latter is unacceptable, given the use of the data, whereas the former is acceptable. In this hypothetical case, large variations across VFA samples are less significant than smaller variations associated with C:N. This is because variations in VFA's are transient and either readily corrected or do not diminish compost quality relative to its intended use, whereas highly variable C:N ratios indicate potentially serious problems with the composting process and product quality.

Table 02.01-A3 Compost sample data analyzed for variability

<i>Sample</i>	<i>C:N Ratio</i>	<i>VFA mg kg<sup>-1</sup></i>
1	35	12,000
2	19	18,000
3	39	19,000
4	22	25,000
5	42	9,000
Average:	31.4	16,600
Standard Deviation:	10.3	6,268
%CV:	33	38
<i>Acceptable CV:</i>	15%	45%
<i>Suitability of Data:</i>	REJECT	ACCEPT

9.10 *Sampler Devices*—There is no single standardized compost sampling device. Tools and devices for soil and forage sampling are relatively simple and efficient and are useful for compost sampling, but they have severe limitations.

9.10.1 *Slotted Tube Sampler*—Single or double, slotted tube and rod, all slotted ends and a minimum 5-cm (2-in.) diameter. The Pennsylvania State Forage Sampler, or equivalent, is a satisfactory core sampler for composts that do not contain significant foreign objects.

9.10.2 *Shovel*—Standard long, handled, pointed tip; typical horticultural narrow shovel, cleaned well with soapy water, rinsed, and dried between samples.

9.10.3 *Thief Sampler,*

9.10.4 *Trier,*

9.10.5 *Pipe*—PVC or plastic,

9.10.6 *Tarpaulin*—plastic,

9.10.7 *Pail*—16- to 20-L (4- to 5-gal), square pails. Use standard 5-gal plastic pails only when square pails are not available (e.g., square pails are available through Cleveland Bottle & Supply Co.; 850 East 77th Street; Cleveland, OH 44103; telephone: 216 881 3330; FAX: 216 881 7325; URL: <http://www.clevelandbottle.com/squrpail.html>). Pails must be cleaned well with soapy water, rinsed, and dried between samples.

9.10.8 *Trowel*—Standard garden, high-density polypropylene (HDPP) for sub-sample mixing and bag-filling; trowels must be cleaned well with soapy water, rinsed, and dried between samples.

9.10.9 *Sample Containers*—Use a container that is appropriate for the laboratory analysis to be performed on the collected compost sample. Refer to Tables 02.01-2 through 02.01-6, and Figure 02.01-B3.

9.10.10 *Labels and Logbook*

9.10.10.1 *Labels*—Name of technician, operator, inspector, facility name, pile identification, date, time, sample number and location in pile using length, width and height coordinates from an identified end and depth from surface measured perpendicular to surface, purpose of sample/test, method of sample preservation.

9.10.10.2 *Logbook*—Name of technician, operator, or inspector; and facility name. Pile data including: pile identification; feedstock-mix; type of pile; date started; weather conditions at time of sampling (for exposed piles only); pile orientation relative to natural drainage. Sample data including: date and time of sample collection; location where samples were collected in pile using length, width and height coordinates from an identified end and depth from surface measured horizontally; description of the sampling point; purpose of sample/test, method of sample preservation, point or composite sample; number and volume of the samples taken; date and time samples were shipped.

<b>Test Method:</b> Selection of Sampling Locations for Windrows and Piles							<b>Units:</b> NA	
<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>
		02.01-B	02.01-B	02.01-B	02.01-B	02.01-B	02.01-B	02.01-B

## 02.01-B SELECTION OF SAMPLING LOCATIONS FOR WINDROWS AND PILES

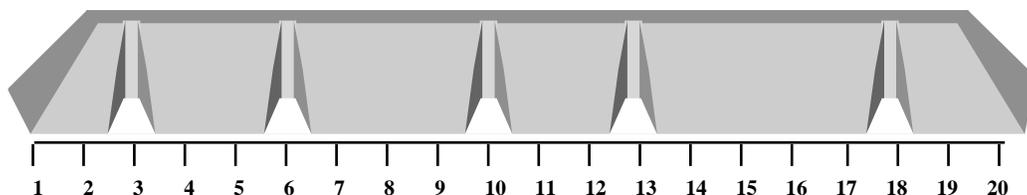


Fig 02.01-B1 Hypothetical sample collection pattern from a compost windrow.

NOTE 1B—In this example, a scale from 1-20 is superimposed on the long dimension of a compost windrow. Five distances (3, 6, 10, 13 and 18 m) are randomly selected to each side of the windrow, (e.g., numbers randomly pulled from a hat), to assign sample collection locations. Point-samples are collected from within three zones at each cutout.

NOTE 2B—The illustrated cut-outs are depicted on one side of the windrow; in a real operation, the cut-outs must be randomly assigned to each side of the windrow. Cone-shaped piles have a circular base. Measure around the base of a cone-shaped pile and randomly assign cutout positions along the pile's meridian, or circumference.

### 10. Apparatus for Method B

10.1 *Sampling Container*—five 16- to 20-L (4- to 5-gal), plastic (HDPP), glass.

10.1.1 *Organic Contaminant Tests*—For samples to be analyzed for the presence of organic contaminants, please refer to Table 02.01-6 Organic Contaminant Tests: Sampling containers and conditions for compost and source ingredient testing. Modify sample packaging steps presented in this section accordingly.

10.2 *Sampling Device*—silage auger, tilling spade, or other appropriate sampling device.

10.3 *Tractor Loader*—with loader, (e.g., Bobcat, etc.).

10.4 *Trowel*—high-density polypropylene (HDPP), for stirring and mixing composite sample.

10.5 *Pail*—16- to 20-L (4- to 5-gal), square pails. Use standard 5-gal plastic pails for shipping only when square pails are not available (e.g., square pails are available through Cleveland Bottle & Supply Co.; 850 East 77th Street; Cleveland, OH 44103; telephone: 216 881 3330; Fax: 216 881 7325; URL: <http://www.clevelandbottle.com/sqrpail.html>).

### 11. Reagents and Materials for Method B

11.1 *Plastic Bags*—three 4-L (1 gal) durable bags with seal, (e.g., Ziploc® Freezer bags).

11.2 *Plastic Gloves*.

11.3 *Tarp*—clean plastic, canvas, or other type of mixing surface if feedstock is liquid sludge.

11.4 *Cold Packs*—chemical ice packs, or 4-L plastic bags (e.g., heavy duty Ziploc® freezer bags) filled with approximately 0.5 L of water and frozen flat. One ice pack per 4-L sample container of compost to be shipped, (e.g., three ice packs are recommended for three compost 4-L samples).

11.5 *Aluminum Foil*—lining for plastic shipping pail, and

11.6 *Packing Material*—newspaper or other appropriate bulking material to be used as packing or fill to minimize sample movement within the shipping container (square pail) during shipping.

11.7 *Adhesive Tape*—duct tape, 5-cm (2-in.) width.

### 12. Procedures for Method B

12.1 *Cut into Finished Compost*—Using tractor skid-loader, bobcat or shovel, or sample boring device, cut into the finished compost pile or windrow at five or more randomly selected positions. Collect samples from the full profile and breadth of the compost windrow or pile. Refer to Fig 02.01-B1.

12.2 *Collect Point-Samples*—Samples of equal volume are extracted from the compost pile at three depths or zones measured from the pile's uppermost surface. Collect no less than five point-samples from each of the three depths or zones illustrated in Fig 02.01-B2. The five point samples for each zone must be collected in a manner to accurately represent the horizontal cross-section of the windrow or pile. Use a sanitized sampling tool (a gloved hand, clean shovel or auger) when collecting samples and when transferring samples to the 5-gal sample collection pail.

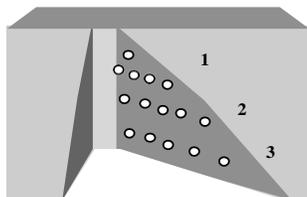


Fig 02.01-B2 Five horizontally dispersed point-samples are collected from each of three depths or zones within each cutout.

NOTE 3B—(1) upper  $\frac{1}{3}$  of compost profile height; (2) middle  $\frac{1}{3}$  of compost profile height; and (3) lower  $\frac{1}{3}$  of compost profile height, where compost pile does not exceed the recommended overall height of 3 m. Create more than three sampling depths or zones within each cutout when the curing pile exceeds a height of 3 m, relative variability is high or the property of interest is found at very low concentrations, near the laboratory detection limit.

12.3 Composite Point-Samples—Place all 15 point samples from one cutout together into one sanitized plastic pail. Completely mix the point samples by stirring thoroughly with a sanitized wooden stick or lath, and by covering and shaking the pail to further mix the samples.

12.3.1 Repeat the blending process at least four times until all point samples are thoroughly blended to form one composite sample that accurately represents the compost for the cutout.

12.3.2 Proceed to the next compost sample cutout and repeat this process to collect one thoroughly blended composite sample from each of the five cutouts.

12.3.3 Composite Sample—Transfer the five composite samples from the sample collection pails onto a mixing tarp or other appropriately sanitized surface or container, such as into a large pail where all samples can be mixed, blended and then covered to minimize moisture loss. Thoroughly blend the five composite samples to form one large sample that represents the average condition of the entire batch or windrow in question.

12.3.3.1 Quarter the composite sample and thoroughly mix and quarter again. Continue to subdivide and split the sample into quarters and mix as described until sample size reaches approximately 12 L (3 gal).

12.4 Stratified Sampling—This sample collection strategy is used to evaluate for the presence of spatial variations or gradients in compost characteristics across and through a windrow or pile.

12.4.1 Stratified Samples across Cutouts—Use this sampling strategy to test for differences in compost characteristics between sample cutouts and along the longer dimension of a windrow. Do not composite materials from the five separate cutouts when

monitoring for the presence of gradients along the longer dimension of a windrow. Pack and prepare five separate samples (i.e., five separate composite samples, one from each cutout) for shipment as described in step 12.5.

12.4.2 Stratified Samples within Cutouts—Use this sampling strategy to evaluate for the presence of spatial variations or gradients that occur with changes in pile depth or distance from the windrow core to its surface.

## 12.5 Prepare for Shipment and Storage:

12.5.1 Transfer the blended compost to three 4-L (1-gal) sample bags, (e.g., plastic Ziploc® freezer bags).

12.5.2 Line the shipment pail with aluminum foil or other reflective material to minimize sample heat-gain. Place the sample bags containing the compost sample into the plastic pail and interleave with ice packs for shipping (refer to Fig 02.01-B3).

12.5.3 Cover the pail with its lid. Seal and secure the lid with a packing tape. Send the sample pail by one-day express delivery service to your selected laboratory for analysis. Include a chain of custody information sheet with environmental regulatory samples (Refer to Method 02.01-E).

NOTE 3B—Maintain cool samples at 4°C (39.2°F) to diminish microbial and chemical activity prior to and during sample shipment.

Foil lined plastic pail lid

Three 4-L sample containers

Two 4-L ice packs

Foil lined shipping pail

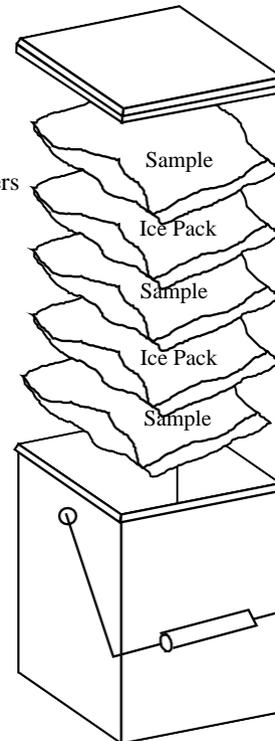


Fig 02.01-B3 Preparation for shipment.

<b>Test Method:</b> Field Sampling Plan for Composted Material							<b>Units:</b> NA	
<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>
02.01-C	02.01-C	02.01-C	02.01-C	02.01-C	02.01-C	02.01-C	02.01-C	02.01-C

## 02.01-C FIELD SAMPLING PLAN FOR COMPOSTED MATERIAL

### 13. US EPA SW-846 Guideline Review and Considerations

13.1 With its hazardous waste management system, the US EPA requires that certain solid wastes be analyzed for physical and chemical properties. In its hazardous waste management system, it is mostly chemical properties that are of concern, and in the case of a number of chemical components, the US EPA has promulgated levels (regulatory thresholds) that cannot be equaled or exceeded.

13.1.1 Regulations pertaining to the management of hazardous waste contain three references regarding the sampling of solid wastes for analytical purposes:

13.1.1.1 Collect representative samples of waste, so that they exhibit average properties of the bulk compost or feedstock.

13.1.1.2 Collect enough samples (but no less than four samples) over a period of time sufficient to represent the variability of the compost or feedstock.

13.2 *Sampling Plan Implementation*—The US EPA manual contains a section on implementation of the sampling plan (SW-846 Chapter Nine, part 2). Within that section there is discussion concerning the sampling program's objectives for evaluating a compost. (Refer to Fig 03.01 Sample fate).

13.2.1 The example suggests the following questions be answered:

13.2.1.1 Is the sampling being performed to comply with environmental regulation?

13.2.1.2 Samples are to be analyzed for which parameters?

13.2.1.3 Why not others?

13.2.1.4 Should samples be analyzed for fewer parameters?

13.2.1.5 What is the end-use of the generated data?

13.2.1.6 What are the required degrees of accuracy and precision?

13.2.2 These questions may or may not be as important for sampling composted solid waste.

13.3 *Sampling Plan Considerations*—The implementation section contains a category entitled

Sampling Plan Considerations. The sampling plan is usually a written document that describes the objectives, and details the individual tasks and how they will be performed. The more detailed the sampling plan, the less opportunity for oversight or misunderstanding during sampling, analysis, and data management.

13.3.1 The SW-846 document suggests that a sampling plan be designed with input from the various sectors involved in the project, including the following personnel:

13.3.1.1 *regulatory sampling*—in many cases may require state permits and consultations with state officials.

13.3.1.2 *end-user*—to use the data to attain program objectives.

13.3.1.3 *field team member*—an experienced member of the field team who actually collects samples.

13.3.1.4 *analytical chemist*—to review analytical requirements for sampling, preservation, and holding times that will be included in the sampling plan.

13.3.1.5 *process engineer or equivalent*—it explain details and constraints of the production process being sampled.

13.3.1.6 *statistician*—to review the sampling approach and verify that the resulting data will be suitable for any required statistical calculations for decisions.

13.3.2 *quality assurance representative*—to review the applicability of standard operating procedures and determine the number of blanks, duplicates, spike samples, and other steps required to document the accuracy and precision of the resulting data.

13.3.3 If no one is familiar with the site to be sampled, then a pre-sampling site visit should be arranged to acquire site-specific information. Some modifications of the sampling plan may be necessary. It is necessary to have at least one experienced sampler as a member of a sampling team.

### 14. Statistical Validity of Sampling Plan

14.1 *Objectives*—The primary objective of a sampling plan for a compost is to collect an appropriate

number of representative samples and subsamples for accurate and precise measurement of the chemical, physical and biological properties of the compost. If the chemical measurements are sufficiently accurate and precise, they will be considered reliable estimates of the chemical properties of the compost.

14.1.1 Generally, high degrees of accuracy and precision are required if one or more chemical components of compost are present at a concentration that is close to the applicable regulatory threshold. Alternatively, relatively low accuracy and low precision can be tolerated if the components of concern occur at levels far below or far above their applicable thresholds. Low sampling precision is often associated with considerable savings in analytical costs, as well as expenses associated with sampling; and is clearly recognizable even in the simplest of statistical tests. However, low sampling accuracy may not entail cost savings and is always obscured in statistical tests (i.e., it cannot be evaluated). Although it is often desirable to design sampling plans for compost to achieve only the minimally required precision (at least two samples are required for any estimate of precision), it is prudent to design the plans to attain the greatest possible accuracy.

14.2 *Composite Sampling*—For composite sampling, a number of random subsamples are initially collected and combined into a single sample, which is analyzed for the chemical constituents of concern. The major disadvantage of composite sampling, as compared with non-composite sampling, is loss of information about the spatial variability of chemical constituents because only a single estimate of the parameter is generated. The benefit is that a credible, general representation of the entire compost pile is generated from a large number of subsamples which are composited.

14.3 *Sampling Quality Assurance/Quality Control (QA/QC)*:

14.3.1 Make sure all sampling equipment and containers are clean. If equipment is used to collect multiple samples, provisions for cleaning and decontamination are required between samples.

14.3.2 Properly label all samples and keep accurate records. Record as much information on sample labels as possible prior to arriving at the site. Sample labels and field notes should include material type, location, date, approximate age of compost, sampler's name, special sampling procedures used, analytical procedures to be performed, preservatives added (if any), and any special observations or incidents during the sampling event.

14.3.3 Point-samples must be stored in a refrigerator (4°C) before analysis when delays in shipment to laboratory are anticipated. This preservation is especially important for feed stock samples, compost to be evaluated for stabilization or maturity, or

microbiological analysis. Chemical quality changes that may take place due to microbiological activity between sample collection and laboratory analysis should be avoided.

14.3.4 Chain of custody forms and procedures should be used with all environmental samples.

14.4 *Other Sampling Considerations*—Compost samples are taken at each facility for a variety of purposes. Varying levels of expertise and quality assurance are required depending on the sampling purpose or objective. A unique sampling protocol should be developed for each specific objective. This information should be detailed in a facility operation and maintenance (O&M) manual and be accessible to all facility staff.

14.4.1 Key process variables including porosity, nutrient balance, oxygen, moisture, temperature and time are monitored and controlled on a continual or daily basis. Measurements of weight and volume of waste arriving and compost leaving the facility are necessary for planning material movements, personnel and transportation requirements, and maintaining facility aesthetics. Although this is the most frequent type of sampling conducted, the sampling quality assurance requirements are the least significant for these activities. Generally, process control and material handling data do not need to be precise to be useful, (e.g., appropriate application of quick-tests). Regulatory compliance and product attribute data must be highly precise and accurate, (e.g., statistically valid sampling program to accurately estimate the average value of interest).

14.5 *Sampling Frequency*—Operating permits for compost sites require that concentrations of certain constituents of environmental concern be evaluated, (e.g., As, Ba, Cd, Cu, Cr, Hg, Mn, Mo, Ni, Pb, Se, Zn, pathogens such as *Salmonella* and fecal coliform, and organic compounds such as PCB's, PCP's, dioxins, furans, organochlorine and organophosphorus pesticides). Regulatory agencies establish compliance using individual sample results. It is, therefore, very important that sample collection and preparation techniques provide representative samples.

NOTE 1C—As much as 20,000 m<sup>3</sup> of compost may be represented by one subsample as small as 1 g. Because of this, it is vital that the sample be representative of the total material. *Quality control and quality assurance for quarterly testing must be greater than that employed for routine daily monitoring.*

14.6 *Statistical Techniques*—Statistical techniques for obtaining accurate and precise samples are relatively simple and easy to implement. Accurate representations of an entire compost pile or batch may be achieved through random sampling. In random sampling, every unit in the population has a theoretically equal chance of being sampled and

measured. Consequently, statistics generated by the sample (e.g. sample mean and to a lesser degree, standard deviation) are unbiased estimators of true population parameters. That is, the sample is representative of the population. A common method of selecting a random sample is to divide the population by an imaginary grid, assign a series of consecutive numbers to the units of the grid, and select the number to be sampled using a random-numbers table.

NOTE 2C—Haphazardly selected samples are not random and therefore not a suitable substitute for a randomly selected sample. That is because there is no assurance that a person performing undisciplined sampling will not consciously or subconsciously favor the selection of certain units of the population.

14.6.1 Sampling precision is achieved by collecting the appropriate number of samples that are uniformly distributed across the entire volume of compost. Precision is improved by increasing the number of samples, while maintaining a sampling pattern to guarantee a spatially uniform distribution.

14.6.2 If a batch of compost is randomly heterogeneous with regard to its chemical characteristics and if that random chemical heterogeneity remains constant from batch to batch, accuracy and appropriate precision can usually be achieved by simple or systematic random sampling. More complex stratified random sampling is appropriate if a batch of compost is known to be non-randomly heterogeneous in terms of its chemical properties and non-random chemical heterogeneity is known to exist from batch to batch. In such cases, the population is stratified to isolate the known sources of non-random chemical heterogeneity. The units in each stratum are numerically identified, and a simple random sample is taken from each stratum. This type of sampling would be advantageous only if the stratification efficiently divides the waste into strata that exhibit maximum between-strata variability and minimum within-strata variability. In composted solid waste that is frequently turned and mixed, little if any stratification is likely to occur. If little or no information is available concerning the distribution of chemical components, simple or systematic random sampling are the most appropriate sampling strategies.

14.7 *Number of Samples*—The appropriate number of samples to collect is the least number required to generate a sufficiently precise estimate of the true mean concentration of a chemical component of a compost. From the compost producer's perspective, this means that the minimum number of samples needed to demonstrate that the upper limit of the confidence interval for the true mean is less than the applicable regulatory threshold value. It is always prudent to collect a greater number of samples than indicated by preliminary estimates of the mean and variance since poor preliminary estimates of those statistics can result

in an underestimate of the appropriate number of samples to collect.

14.8 *Simple Random Sampling*—For convenience, the statistical calculations for simple random sampling (wherein within-batch heterogeneity that may be encountered by a compost producer is low) are provided (adapted from SW-846 Chapter Nine, part 2, pages 13-14).

14.8.1 Obtain preliminary estimate of  $\bar{x}$  for each chemical component of compost that is of concern. The above-identified statistic is calculated by Equation 14.8.1.

$$\sum_{i=1}^n x_i$$

$$\bar{x} = \frac{\sum_{i=1}^n x_i}{n} \quad \text{Equation 14.8.1}$$

where:

- $\bar{x}$  = simple random sample mean,
- $n$  = total number of sample measurements,
- $x$  = variable in question (e.g., mercury),
- $i$  = individual samples ranging from 1 to  $n$ , and

$\sum_{i=1}^n x_i$  = sum of all  $x$ 's (analytical results for individual samples), from  $i = 1$  through  $i = n$ .

14.8.2 Obtain preliminary estimate of variance,  $s^2$ , for each chemical component of concern. The above-identified statistic is calculated by Equation 14.8.2.

$$\frac{\sum_{i=1}^n x_i^2 - \left(\sum_{i=1}^n x_i\right)^2/n}{n-1}$$

$$s^2 = \frac{\sum_{i=1}^n x_i^2 - \left(\sum_{i=1}^n x_i\right)^2/n}{n-1} \quad \text{Equation 14.8.2}$$

where:

- $s^2$  = variance of simple random sample,
- $n$  = total number of sample measurements,
- $x$  = variable in question (e.g., mercury), and
- $i$  = individual samples ranging from 1 to  $n$ .

14.8.3 Estimate the appropriate number of samples ( $n_1$ ) to be collected from the compost through use of Equation 14.8.3 and Table 02.01-C1. Derive individual values of  $n_1$  for each chemical component of concern ( $x$ ). The appropriate number of samples to be taken from the compost is the greatest of the individual  $n_1$  values.

$$\frac{t_{.20}^2 s^2}{\Delta^2}$$

$$n = \frac{t_{.20}^2 s^2}{\Delta^2} \quad \text{Equation 14.8.3}$$

where:

- $n$  = number of samples,
- $t_{.20}^2$  = tabulated "t" value for two-tailed confidence interval and a probability of 0.20,
- $s^2$  = sample variance, and

$\Delta^2$  = the square of the regulatory threshold minus sample average, defined by US EPA, (e.g., 100 mg kg<sup>-1</sup> for barium in elutriate of EPtoxicity).

Table 02.01-C1 Tabulated values of Student's "t" for evaluating compost.

Degrees of freedom (n-1)	Tabulated "t" value	Degrees of freedom (n-1)	Tabulated "t" value
1	3.078	16	1.337
2	1.886	17	1.333
3	1.638	18	1.330
4	1.533	19	1.328
5	1.476	20	1.325
6	1.440	21	1.323
7	1.415	22	1.321
8	1.397	23	1.319
9	1.393	24	1.318
10	1.372	25	1.316
11	1.363	26	1.315
12	1.356	27	1.314
13	1.350	28	1.313
14	1.345	29	1.311
15	1.341	30	1.310
		40	1.303
		60	1.296
		120	1.289

14.8.3.1 Randomly collect at least  $n_1$  (or  $n_2 - n_1$ ,  $n_3 - n_2$ , etc., as will be indicated in step 8) samples from the compost. Maximize the physical size (volume) of all samples that are collected from the strata.

NOTE 3C—Collection of a few extra samples will provide protection against poor preliminary estimates of  $\bar{x}$  and  $s^2$ .

14.8.3.2 Analyze the  $n_1$  (or  $n_2 - n_1$ ,  $n_3 - n_2$ , etc.) samples for each chemical component of concern. Superficially (graphically) examine each set of analytical data from each stratum for obvious departures from normality.

14.8.4 Calculate the standard deviation ( $s$ ) for each set of analytical data by Equations 14.8.1, 14.8.2, 14.8.4 and 14.8.5.

$$s = \sqrt{s^2} \quad \text{Equation 14.8.4}$$

14.8.5 Calculate  $\bar{x}$ ,  $s^2$ , and standard error ( $s_x$ ) for each set of analytical data by, Equations 14.8.1, 14.8.2, and 14.8.5.

$$s_x = \frac{s}{\sqrt{n}} \quad \text{Equation 14.8.5}$$

14.8.5.1 If  $\bar{x}$  for a chemical component is equal to or greater than the applicable regulatory threshold (from Equation 14.8.3) and is believed to be an accurate estimator of  $\mu$  (population mean), the component is considered to be present in the compost at a hazardous concentration, and the study is

completed. Otherwise, continue the study. In the case of a set of analytical data that does not exhibit obvious abnormality and for which  $\bar{x}$  is greater than  $s^2$ , perform the following calculations with non-transformed data. Otherwise, consider transforming the data by the square root transformation (if  $\bar{x}$  is about equal to  $s^2$ ) or the arcsine transformation (if  $\bar{x}$  is less than  $s^2$ ) and performing all subsequent calculations with transformed data.

14.8.6 Determine the confidence interval (CI) for each chemical component of concern by Equation 14.8.6. If the upper limit of the CI is less than the applicable regulatory threshold (applied in Equation 14.8.3), the chemical component is not considered to be present in the compost at a hazardous concentration, and the study is completed. Otherwise, the opposite conclusion is tentatively reached.

$$CI = \bar{x} \pm t_{0.20} s_x \quad \text{Equation 14.8.6}$$

where:

$t_{0.20}$  = referred to in Table 02.01-C1 Tabulated values of Student's "t" for evaluating compost for appropriate degrees of freedom.

14.8.7 If a tentative conclusion of hazard is reached, re-estimate the total number of samples ( $n_2$ ) to be collected from the compost by use of Equation 14.8.3. When deriving  $n_2$ , employ the newly calculated (not preliminary) values of  $\bar{x}$  and  $s^2$ . If additional  $n_2 - n_1$  samples of compost cannot reasonably be collected, the study is completed, and a definitive conclusion of hazard is reached. Otherwise, collect an extra  $n_2 - n_1$  samples of compost.

14.8.8 Repeat the basic operations described in Steps 14.8.3 through 14.8.7 until the compost is judged to be non-hazardous or, if the opposite conclusion continues to be reached, until increased sampling effort is impractical.

14.9 Stratified Random Sampling—For convenience, the statistical calculation steps for stratified random sampling that must be performed in situations that may be encountered by a compost producer where within-batch heterogeneity is high are provided below (from SW-846 Chapter Nine, part 2, pages 18-19).

14.9.1 Obtain preliminary estimate of  $\bar{x}$  for each chemical component of concern. The identified statistic is calculated by Equation 14.9.1.

**Sample Collection and Laboratory Preparation**  
*Field Sampling of Compost Materials 02.01*

$$\bar{x} = \sum_{k=1}^r W_k \bar{x}_k \quad \text{Equation 14.9.1}$$

where:

$\bar{x}$  = stratified random sample mean,

$\bar{x}_k$  = stratum mean, and

$W_k$  = fraction of population represented by stratum  $k$   
(number of strata  $[k]$  range from 1 to  $r$ ).

14.9.2 Obtain preliminary estimate of  $s^2$  for each chemical component of compost that is of concern. The identified statistic is calculated by Equation 14.9.2.

$$s^2 = \sum_{k=1}^r W_k s_k^2 \quad \text{Equation 14.9.2}$$

where:

$s^2$  = stratified random sample variance,

$s_k^2$  = stratum variance, and

$W_k$  = fraction of population represented by stratum  $k$   
(number of strata  $[k]$  range from 1 to  $r$ ).

14.9.3 Estimate the appropriate number of samples ( $n_1$ ) to be collected from the compost through use of Equation 14.8.3 and Table 02.01-A1 Tabulated values of Student's "t" for evaluating compost. Derive individual values of  $n_1$  for each chemical component of concern. The appropriate number of samples to be taken from the compost is the greatest of the individual  $n_1$  values.

14.9.4 Randomly collect at least  $n_1$  (or  $n_2 - n_1$ ,  $n_3 - n_2$ , etc., as will be indicated in step 8) samples from the compost. If  $s_k$  for each stratum (see Equation 14.9.2) is believed to be an accurate estimate, optimally allocate samples among strata (i.e., locate samples among strata so that the number of samples collected from each stratum is directly proportional to the  $s_k$  for that stratum). Otherwise, proportionally allocate samples among strata according to size of the strata. Maximize the physical size (volume) of all samples that are collected from the strata.

14.9.5 Analyze the  $n_1$  (or  $n_2 - n_1$ ,  $n_3 - n_2$ , etc.) samples for each chemical component of concern. Superficially (graphically) examine each set of analytical data from each stratum for obvious departures from normality.

14.9.6 Calculate  $\bar{x}$ ,  $s^2$ , the standard deviation ( $s$ ), and  $s_x$  for each set of analytical data by, respectively, Equations 14.9.1, 14.9.2, 14.8.4 and 14.8.5.

14.9.7 If  $\bar{x}$  for a chemical component is equal to or greater than the applicable regulatory threshold (from Equation 14.8.3) and is believed to be an accurate estimator of  $\mu$  (population mean), the component is considered to be present in the compost at a hazardous concentration, and the study is completed. Otherwise, continue the study. In the case of a set of analytical data that does not exhibit obvious abnormality and for which  $\bar{x}$  is greater than  $s^2$ , perform the following calculations with non-transformed data. Otherwise, consider transforming the data by the square root transformation (if  $\bar{x}$  is about equal to  $s^2$ ) or the arcsine transformation (if  $\bar{x}$  is less than  $s^2$ ) and performing all subsequent calculations with transformed data.

14.9.8 Determine the confidence interval (CI) for each chemical component of concern by Equation 14.8.6. If the upper limit of the CI is less than the applicable regulatory threshold (applied in Equation 14.8.3), the chemical component is not considered to be present in the compost at a hazardous concentration, and the study is completed. Otherwise, the opposite conclusion is tentatively reached.

14.9.9 If a tentative conclusion of hazard is reached, re-estimate the total number of samples ( $n_2$ ) to be collected from the compost by use of Equation 14.8.3. When deriving  $n_2$ , employ the newly calculated (not preliminary) values of  $\bar{x}$  and  $s^2$ . If additional  $n_2 - n_1$  samples of compost cannot reasonably be collected, the study is completed, and a definitive conclusion of hazard is reached. Otherwise, collect an extra  $n_2 - n_1$  samples of compost.

14.9.10 Repeat the basic operations described in Steps 14.9.3 through 14.9.9 of Fig 02.01-1 Composting Unit Operations, until the compost is judged to be non-hazardous or if the opposite conclusion continues to be reached until increased sampling effort is impractical.

<b>Test Method:</b> Composting Feedstock Material Sampling Strategies						<b>Units:</b> NA		
<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	Safety Standards	Market Attributes
02.01-D	02.01-D							

## 02.01-D BATCH FEEDSTOCK MATERIAL SAMPLING STRATEGIES

### 15. Apparatus for Method D

15.1 *Sampling Container*—20-L (5-gal), stainless steel, plastic, glass or Teflon.

15.2 *Sampling Device*—wooden spatula or tiling spade, etc.

15.3 *Trowel*—high-density polypropylene (HDPP).

15.4 *Plastic Storage Pail*—20-L (5-gal), square pails, Use standard 5-gallon plastic pails only when square pails are not available (e.g., Cleveland Bottle & Supply Co.; 850 East 77th Street; Cleveland, OH 44103; telephone: 216 881 3330; Fax: 216 881 7325; URL: [www.clevelandbottle.com/squrpail.html](http://www.clevelandbottle.com/squrpail.html)).

### 16. Reagents and Materials for Method D

16.1 *Plastic Gloves*.

16.2 *Tarp*—clean plastic, canvas, or other type of mixing surface if feedstock is liquid sludge.

16.3 *Plastic Bags*—three 4-L (1 gal) Ziploc® freezer bags.

16.4 *Cold Packs*—chemical ice packs,

16.5 *Aluminum Foil*—lining for plastic shipping pail, and

16.6 *Adhesive Tape*—duct tape, 5-cm (2-in.) width.

### 17. Procedure for Method D

17.1 *Sample Collection*—Identify and collect an appropriate number of subsamples needed to ensure a reliable analytical result as described in Methods 02.01-A, B or C.

17.1.1 Place each subsample into a sampling (subsample) container.

17.1.2 Transfer the contents of the subsample container onto (into) mixing surface (container) and proceed to the next randomly selected sample point.

17.1.3 Repeat steps 17.1.1 and 17.1.2 until the predetermined number of subsamples is obtained.

17.2 *Sample Mixing*—Place subsamples on clean tarp or other similar mixing platform, mix sub-samples thoroughly using a wooden spatula or comparable sampling tool.

17.3 *Sample Splitting*—Subdivide sample into quarters, thoroughly mixed composite sample into fourths. Repeat steps 17.2 and 17.3 until sample size is appropriate for intended analysis.

17.4 *Sample Storage and Shipping*—Place composite sample aliquot in clean container, preferably a Teflon pail or similar inert material.

CAUTION—Do not use galvanized sheet metal collection or mixing tools. The galvanized coating will contaminate the sample with zinc.

17.4.1 Transfer blended feedstock or compost to fill three 4-L (1-gal) plastic Ziploc® freezer bags.

17.4.2 Line the shipment pail with aluminum foil to minimize heat exchange. Place the plastic Ziploc® freezer bags containing the feedstock samples in the plastic pail and interleave with cold packs for shipping (refer to Fig 02.01-B3).

17.4.3 Seal the square pail with its lid. Seal and secure lid with duct tape. Send the square plastic pail containing samples by two-day express service to the selected laboratory for analysis. Include completed chain of custody forms when necessary.

NOTE 1D—If any delay is anticipated, cool sample to 4°C (39.2°F) to diminish microbial and chemical activity prior to sample shipment.

<b>Test Method:</b> Data Quality Management and Sample Chain of Custody						<b>Units:</b> NA		
<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	Safety Standards	Market Attributes
02.01-E	02.01-E		02.01-E			02.01-E	02.01-E	02.01-E

## 02.01-E DATA QUALITY MANAGEMENT AND SAMPLE CHAIN OF CUSTODY

### 18. Aspects of Sampling Quality Assurance for Reported Data

18.1 Three critical steps in the sampling process precede laboratory analysis and often dictate data quality.

- 18.1.1 sample planning and collection;
- 18.1.2 sample handling and preservation; and
- 18.1.3 laboratory sample preparation.

18.2 Each step in the sampling process must be properly executed in a timely manner by well informed, trained individuals to ensure that the collected sample accurately represents a compost batch, windrow or pile.

18.3 *Quality Sample Management*—Regulatory and certification systems may dictate that samples are properly collected, preserved and prepared for analysis. Consider the following hypothetical example of sample management where a certified third party is introduced to manage the sampling plan.

18.3.1 The third party assumes all quality assurance and quality control responsibilities associated with:

- 18.3.1.1 sample planning and collection;
- 18.3.1.2 sample handling and preservation; and
- 18.3.1.3 laboratory sample preparation.

18.3.2 Responsibility for rigorous sample collection is transferred from facility management to the third party. Responsibilities associated with sample storage, preparation and laboratory analysis are also transferred from the analytical laboratory to the third party.

18.3.3 One of the principal benefits of the third party sampling system is to diminish deviations in sampling plan interpretation and implementation across separate facilities and laboratories. Third party control can decrease variability by maintaining consistent field sampling protocols across all participating facilities. Field sample collections would be implemented as described in *TMECC 02.01 Field Sampling of Compost Materials*. Consistent sample preparation protocols would also be followed for laboratory analysis as described in *TMECC 02.02 Laboratory Sample Preparation for Analysis*.

18.4 *Tracking Quality*—A sample must be properly collected and prepared for shipment, and then properly manipulated by laboratory personnel who follow specific preparation protocols designed for each analytical methodology. Previous sections emphasized the importance of properly designed and implemented sampling plans. This section introduces a protocol designed to modify data interpretation to interpret sample variability.

18.4.1 Consider the following hypothetical sampling plan that incorporates an additional step to verify accuracy of reported results using cross-validation techniques. One type of a statistically valid sample management plan requires that samples are properly collected at a very high frequency while the actual number of samples submitted for analysis remains small.

18.4.1.1 *Establish Baseline*—A significant number of samples that represent the composting process of a facility are collected over time and sent to a laboratory for analysis. Results from these samples serve to establish a baseline of information that accurately represents the compost produced by the facility and a given feedstock blend.

18.4.1.2 *Track Deviations from Baseline*—After the baseline is established, samples are collected at specified intervals, over time or per unit of compost produced (refer to *TMECC 02.01-A Equation 9.9.1 Formula to estimate sampling interval*), and held in cold storage. After a specified interval, (e.g., quarterly or monthly) a small but statically representative number of prepared samples are randomly selected from the stored samples and sent to a laboratory for analysis. Because multiple samples would be randomly selected from a larger population of samples, a more reliable statistical inference can be generated than by simply directly submitting monthly or quarterly samples for analysis.

18.4.2 Sampling programs of this nature may require that field samples, or samples prepared for laboratory analysis, are submitted to a secure or bonded cold-storage facility where frequently collected samples are inventoried and properly stored. Samples must be retained in storage for a predetermined time period to

safeguard against cases where a need for re-testing may arise.

18.4.3 *Sampling Costs*—Sampling program maintenance costs should be considered when designing an effective monitoring system. It is difficult to weigh the relative importance of data quality when there is no clear relationship between financial outcome

and monitoring protocol. Successful implementation will increase when data quality relates to an increased financial incentive, either artificially through incentives offered by the governing regulatory agency or through quality assurance certification programs designed to indirectly increase market share.

## 02.01 SUMMARY

### **19. Report**

19.1 Chain of custody forms and procedures should be used with all environmental or regulatory samples. A chain of custody form is used to track sample handling from time of collection through laboratory analysis, and data reporting. Suggested information for the chain-of-custody record includes, at a minimum: Collector's name; Signature of collector; Date and time of collection; Place and address of collection; Requested preprocessing (subsampling, compositing, sieving); Requested analyses; Sample code number for each sample (if used); Signature of the persons

involved in the chain of possession. Refer to Fig 02.01-E1 Chain of Custody form for an example.

### **20. Keywords**

20.1 accuracy; aliquot; attribute verification; bias; chain of custody; closed vessel system; composite; compost; coefficient of variation; %CV, confidence interval; feedstock; grab-sample; point-sample; point-sampling; open vessel system; precision; process monitoring; process variability; product variability; quality control; quality assurance; representative sample; sample collection frequency; sampling; sampling plan; statistical validity; stratified sampling; windrow.



## Appendix C - TCEQ TAC 332.71 Compost Sampling Guidelines

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# Texas Administrative Code

<a href="#">TITLE 30</a>	ENVIRONMENTAL QUALITY
<a href="#">PART 1</a>	TEXAS COMMISSION ON ENVIRONMENTAL QUALITY
<a href="#">CHAPTER 332</a>	COMPOSTING
<a href="#">SUBCHAPTER G</a>	END-PRODUCT STANDARDS
RULE §332.71	Sampling and Analysis Requirements for Final Product

(a) Applicability. Facilities that receive a registration or permit under this chapter, are required to test final product in accordance with this section. Final product derived from municipal sewage sludge at registered facilities is not subject to the requirements of this section, but must comply with the requirements of Chapter 312 of this title (relating to Sludge Use, Disposal, and Transportation).

(b) Analytical methods. Facilities which use analytical methods to characterize their final product must use methods described in the following publications.

(1) Chemical and physical analysis shall utilize:

- (A) "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods" (SW-846);
- (B) "Methods for Chemical Analysis of Water and Wastes" (EPA-600); or
- (C) "Recommended Test Methods for the Examination of Composts and Composting" (Compost Council, 1995).

(2) Analysis of pathogens shall utilize "Standard Methods for the Examination of Water and Wastewater" (Water Pollution Control Federation, latest edition).

(3) Analysis for foreign matter shall utilize "Recommended Test Methods for the Examination of Composts and Composting" (Composting Council, 1995).

(4) Analysis for salinity and pH shall utilize NCR (North Central Regional) Method 14 for Saturated Media Extract (SME) Method contained in "Recommended Test Procedure for Greenhouse Growth Media" North Central Regional Publication Number 221 (Revised), Recommended Chemical Soil Test Procedures, Bulletin Number 49 (Revised), October 1988, pages 34-37.

(5) Analysis of total, fixed and volatile solids shall utilize Method 2540 G (Total, Fixed, and Volatile Solids in Solid and Semi-solid Samples) as described in "Standard Methods for the Examination of Water and Wastewater" (Water Pollution Control Federation, latest edition).

(6) Analysis for maturity shall utilize the reduction of organic matter (ROM) calculation method, as described in the TNRCC "Quality Assurance Program Plan" (QAPP) or a TNRCC approved Quality Assurance/Quality Control (QAQC) plan during the first 18 months of a facility's operation. Reduction in organic matter is calculated by measuring the volatile solids content at two points in the composting process: when compost feedstocks are initially mixed and when the compost is sampled for end-product testing for total metals and PCBs. For purposes of compost maturity analysis, the effect of the addition and removal of volatile solids and fixed solids to the compost shall be included in the ROM calculation procedure. After the completion of the maturity testing protocol described in subsection (d) of this section, or the facility QAQC plan, or 18 months, which ever comes first, the method recommended in the protocol and approved by the TNRCC shall be utilized.

(c) Sample collection. Sample collection, preservation and analysis shall assure valid and representative results pursuant to an Agency-approved QAQC plan.

(d) Maturity Testing Protocol.

(1) A maturity testing protocol shall be described in the facility QAQC. The protocol shall consist of the ROM method or a comparison of the interim ROM method to a minimum of three test methods with one test method selected from each of subparagraphs (A), (B), and (C) of this paragraph, together with any method in subparagraph (D) of this paragraph.

(A) Chemical analyses:

- (i) carbon/nitrogen ratio;
- (ii) water soluble ions;
- (iii) water soluble organic matter;
- (iv) cation exchange capacity;
- (v) electrical conductivity;
- (vi) crude fiber analysis;
- (vii) humification analysis; or
- (viii) ratios of the above measurements.

(B) Physical analyses.

- (i) Dewar self-heating; or
- (ii) color.

(C) Respiration analyses:

- (i) CO<sub>2</sub>; or
- (ii) O<sub>2</sub>.

(D) Other test methods proposed in the facility QAQC plan and approved by the TNRCC.

(2) The test methods used in the maturity test protocol shall be based on methodologies published in peer reviewed scientific journals, the publication entitled "Recommended Test Methods for the Examination of Composts and Composting (Compost Council, 1995), or other methods as approved by the TNRCC.

(3) The completed maturity testing protocol shall lead to a recommended maturity testing method(s) capable of classifying compost into maturity grades described in §332.72 of this title (relating to Final Product Grades) and identifying materials which are stable but not mature. The maturity test protocol shall address seasonal variations in compost feedstock and shall be completed within 18 months of the start of a new compost feedstock mixture.

(4) The results of the protocol and recommendations shall be submitted to the TNRCC for review and approval. The basis of the TNRCC review and approval shall be the demonstration that the recommended method adequately classifies compost into maturity classes. The purpose of the TNRCC review and approval is not intended to provide detailed guidance to end users about the agricultural and horticultural compost uses.

(5) The compost maturity protocol does not need to be repeated unless a significantly new compost feedstock recipe is utilized.

(e) Documentation.

(1) Owners or operators of permitted or registered facilities shall record and maintain all of the following information regarding their activities of operation for three years after the final product is shipped off site or upon site closure:

(A) batch numbers identifying the final product sampling batch;

(B) the quantities, types and sources of feedstocks received and the dates received;

(C) the quantity and final product grade assigned described in §332.72 of this title;

(D) the date of sampling; and

(E) all analytical data used to characterize the final product, including laboratory quality assurance/quality control data.

(2) The following records shall be maintained on-site permanently or until site closure:

(A) sampling plan and procedures;

(B) training and certification records of staff; and

(C) maturity protocol test results.

(3) Records shall be available for inspection by TNRCC representatives during normal business hours.

(4) The executive director may at any time request by registered or certified mail that a generator submit copies of all documentation listed in paragraph (1) of this subsection for auditing the final product grade. Documentation requested under this section shall be submitted within ten working days of receipt of the request.

(f) Sampling Frequencies.

(1) Registered facilities. For those facilities which are required to register, all final product on-site must be sampled and assigned a final product grade set forth in §332.72 of this title (relating to Final Product Grades) at a minimum rate of one sample for every 5,000 cubic yard batch of final product or annually, whichever is more frequent. Each sample will be a composite of nine grab samples as discussed in subsection (g) of this section.

(2) Permitted facilities. For facilities requiring a permit, all final product on-site must be sampled and assigned a final product grade set forth in §332.72 of this title at a minimum rate of one sample for every 3,000 cubic yard batch of final product or monthly whichever is more frequent. Each sample will be a composite of nine grab samples as discussed in subsection (g) of this section.

(3) Alternative testing frequency. One year after the initiation of final product testing in accordance with this section, an operator of a registered or permitted facility may submit to the executive director a request for an alternative testing frequency. The request shall include a minimum of 12 consecutive months of final product test results for the parameters set forth in subsection (h) of this section. The executive director will review the request and determine if an alternative frequency is appropriate.

(g) Sampling Requirements. For facilities subject to sampling and analysis, the operator shall utilize the protocol in the TNRCC QAPP or a TNRCC approved facility QAQC plan shall be followed. The executive director may at any time request that split samples be provided to an agency representative. Specific sampling requirements which must be satisfied include:

(1) Sampling from stockpiles. One third of the grab samples shall be taken from the base of the stockpile (at least 12 inches into the pile at ground level), one third from the exposed surface and one third from a depth of two feet from the exposed surface of the stockpile.

(2) Sampling from conveyors. Sampling times shall be selected randomly at frequencies which provide the same number of subsamples per volume of finished product as is required in subsection (d) of this section.

(A) If samples are taken from a conveyor belt, the belt shall be stopped at that time. Sampling shall be done along the entire width and depth of the belt.

(B) If samples are taken as the material falls from the end of a conveyor, the conveyor does not need to be stopped. Free-falling samples need to be taken to minimize the bias created as larger particles segregate or heavier particles sink to the bottom as the belt moves. In order to minimize sampling bias, the sample container shall be moved in the shape of a "D" under the falling product to be sampled. The flat portion of the "D" shall be perpendicular to the beltline. The circular portion of the "D" shall be accomplished to return the sampling container to the starting point in a manner so that no product to be sampled is included.

(h) Analytical Requirements. Final product subject to the sampling requirements of this section will be tested for all of the following parameters. The executive director may at any time request that additional parameters be tested. These parameters are intended to address public health and environmental protection.

(1) Total metals, to include:

(A) Arsenic;

(B) Cadmium;

(C) Chromium;

(D) Copper;

(E) Lead;

(F) Mercury;

(G) Molybdenum;

(H) Nickel;

(I) Selenium; and

(J) Zinc.

(2) Maturity/Stability by reduction in organic matter on an interim basis and by approved method of maturity/stability analysis after the completion of the maturity/stability method protocol as described in subsections (b) and (d) of this section.

(3) Weight percent of foreign matter, dry weight basis.

(4) pH by the saturated media extract method.

(5) Salinity by the saturated media extract electrical conductivity method.

(6) Pathogens:

(A) salmonella; and

(B) fecal coliform.

(7) Polychlorinated-biphenyls (PCBs)--required only for permitted facilities.

(i) Data Precision and Accuracy. Analytical data quality shall be established by EPA standard laboratory practices to ensure precision and accuracy.

(j) Reporting Requirements.

(1) Facilities requiring registration must report the following information to the executive director on a semiannual basis for each sampling batch of final product. Facilities requiring a permit must report similarly but on a monthly basis. Reports must include, but may not be limited to all of the following information:

(A) batch numbers identifying the final product sampling batch;

(B) the quantities, types and sources of feedstocks received and the dates received;

(C) the quantity of final product and final product standard code assigned;

(D) the final product grade or permit number of the disposal facility receiving the final product if it is not Grade 1 or Grade 2 Compost as established in §332.72 of this title (relating to Final Product Grades);

(E) all analytical results used to characterize the final product including laboratory quality assurance/quality control data and chain-of-custody documentation; and

(F) the date of sampling.

(2) Reports must be submitted to the executive director within two months after the reporting period ends.

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**Source Note:** The provisions of this §332.71 adopted to be effective November 29, 1995, 20 TexReg 9717.

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## Appendix E – Example Chain of Custody





## Appendix F - TMECC 5.05-E – Solvita Sampling Guide

Test Method: Organic Matter. Three Methods						Units: % g g <sup>-1</sup> dw		
<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>
		05.07-A		05.07-A		05.07-A		05.07-A
							05.07-B	05.07-B
05.07-C	05.07-C	05.07-C		05.07-C	05.07-C	05.07-C		05.07-C

## 05.07 ORGANIC MATTER

### 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 determination of organic matter content in compost.

1.1.1 *Method 05.07-A Loss-On-Ignition Organic Matter Method (LOI)*—A direct determination method that indicates organic matter content by quantifying the amount of solid material combusted relative to the original oven dried sample.

1.1.2 *Method 05.07-B Humic Substances*—Proposed Fulvic Acid and Humic Acid Extraction and Characterization.

1.1.3 *Method 05.07-C Calculations for Organic Matter Decomposition*—This method covers the determination of organic matter decomposition of batch process compost. The protocol is not suitable for use with continuous flow-type composting technologies. This approach to measuring compost stability status is not strongly recommended. No practical test method has been developed, except on biosolids where US EPA CFR Chapter 40 Part 503 references a volatile solids reduction test for biosolids.

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

### 2. Review of Organic Matter

2.1 *Background*—Organic matter is an important reservoir of carbon and a dynamic component of soil and the carbon cycle. It impacts the physical, chemical and biological properties of a soil. Addition of organic matter to soil alters its physical characteristics by changing plant available soil water retention, infiltration, drainage and aeration. Structural parameters are optimized for plant growth by lowering soil bulk density, increasing water holding capacity and aeration. Chemically, the soil nutrient status or nutrient carrying capacity is enhanced by organic matter. Biologically, an enhanced soil organic matter fraction serves as a rich nutrient reservoir and energy source for beneficial microbes.

2.2 *Source*—Soil organic matter content can be increased through frequently repeated applications of compost. Organic matter test determinations will correspond to a compost's stability status and aid in defining the commercial value of a compost relative to its organic matter content.

COMMENT—An organic matter management plan would become practical with the development of compost organic matter test method that could be used to help predict the outcome of applying compost to soil. Present methods simply determine the concentration of organic matter in compost. Knowledge of compost organic matter content does not relate directly to a percentage of soil organic matter after compost is applied to the soil. Factors that clearly alter the organic matter concentration in soil include moisture, temperature, and aeration. An organic matter management plan considers the organic matter content of a compost as one parameter to calculate a compost application rate and frequency for a given soil, to raise that soil's organic matter content to a predetermined target level. This requires identification of a common test method (or suite test methods) for both compost and soil.

CAUTION—Careful attention must be given to historical reports to differentiate references of organic matter measurements versus determinations of total organic carbon. Total organic carbon is used when calculating a C:N ratio. Organic matter contains a number of components in addition to carbon, including nitrogen, sulfur, oxygen, and various micronutrients.

2.3 *Occurrence*—Organic matter is the sum of substances containing organic carbon (Schnitzer,

1991), and is defined as the total organic components in soil including undecayed plant and animal tissues, their partial decomposition products, and the soil biomass exclusive of the macrofauna and macroflora (Vaughan et al., 1985). Organic matter or humus consists of two broad categories known as non-humic and humic substances. The non-humic groups are simple compounds such as carbohydrates, aliphatic and aromatic hydrocarbons, amino acids, ethylene, and hydrogen sulfide that are easily degraded by soil organisms. In contrast, the humic fraction is made up of complex organic molecules, usually formed as byproducts of decomposition and resistant to further degradation. The two stable components of humic substances that play a dominant role in soil physical properties are humic and fulvic acids. These weak acids are also present in organic waste and are suggested to be chemically and structurally similar to humic substances in soil (Sposito et al., 1982).

2.3.1 Organic matter acts as both a sink and source in the soil system. It is a large pool for storage of nitrogen, phosphorus, and sulfur, and can supply nutrients for plant growth. Mineralization of organic matter by microorganisms releases nitrogen, phosphorus, and sulfur to plants. The mineralization of organic matter in grassland soils has contributed to much of the nitrogen and phosphorus nutrition of crops (Tiessen and Stewart, 1983). The negatively charged carboxylic and phenolic functional groups of organic matter produce a high cation exchange capacity relative to other soil fractions (Bohn et al., 1985; McBride, 1994). The functional groups attract metals, metal oxides, hydroxides, and clay minerals to reduce trace metal solubility (Emmerich et al., 1982a; Emmerich et al., 1982b; Leita and DeNobili, 1991).

2.3.2 Organic matter can be partitioned into fresh, slightly humified, and humified state of decomposition (Conti et al., 1993). The humified organic matter is chemically stable and mature (that is, free of organic phytotoxins). Humified organic matter releases nutrients slowly, similarly to a slow release fertilizer (Chen and Avnimelech, 1986). The rate of nutrient release varies with soil physical and chemical properties, climate, microbial population, and the degree of maturity.

2.4 *Nitrogen and Carbon Dynamics*—Nutrient cycling involves immobilization and mineralization driven by microbial activity (Duxbury et al., 1989). Nutrient turnover from labile soil organic matter (which includes soil microbial population) is affected by the carbon supply to heterotrophic microorganisms (Theng et al., 1989). Nitrogen mineralization rates are dependent upon the carbon to nitrogen ratio (C:N ratio).

2.4.1 The dynamic of soil carbon and nitrogen with four cropping systems in agroecosystems was studied by Mazzarino et al. (1993). Sources of carbon and nitrogen additions included tree prunings, corn and bean residues, and inorganic fertilizers. The long-term addition of organic matter in the tree alley cropping treatments increased total and microbial carbon and nitrogen, water-soluble carbon, and soil moisture. Ladd et al. (1977) attempted to partition the mineralization potential from the organic nitrogen component in soil and demonstrated that nitrogen mineralization and availability to crops varies with waste type. Bitzer and Sims (1988) studied nitrogen mineralization in soils amended with poultry manure. They found that organic nitrogen from poultry manure mineralizes rapidly and was even enhanced by the addition of inorganic nitrogen. Rees, *et al.* (1993) studied the influence of the rate and type of manure on nitrogen uptake and uptake efficiency in wheat and barley. They found that nitrogen uptake by barley was increased when inorganic nitrogen fertilizer was added with poultry manure. Bremer and Kessel (1992) reported that 40% of the nitrogen in a lentil green manure was potentially available for plant uptake. Tyson and Cabrera (1993) showed that composted poultry litter mineralized less nitrogen than uncomposted poultry litter, reducing the potential of nitrate pollution. Smith, *et al.* (1993) showed that there was a rapid mineralization of organic nitrogen with treatments of 10 MT A<sup>-1</sup> alkaline pasteurized sewage sludge.

2.4.2 Changes in organic matter and net mineralization rate are influenced by the cropping system, type of litter, environmental factors, and microbial populations (Van Vuuren et al., 1993; Mazzarino et al., 1993; Rees et al., 1993; Zak et al., 1993). In plant communities dominated by dwarf shrubs, van Vuuren et al. (1993) found that net nitrogen mineralization rates increase with increasing amounts of organic matter and soil nitrogen. When litter was replaced by grass, no clear effect was seen on net nitrogen mineralization rates.

2.4.3 Residue quality is another factor affecting nitrogen turnover (Honeycutt et al., 1993). Two residue qualities of hairy vetch harvested in the fall and spring had different carbon and nitrogen mineralization rates independent of the residue loading rate. Approximately 35% of the added carbon mineralized 30 days after application of the fall vetch, and 17% of added carbon mineralized 30 days after application of spring vetch. The effect was postulated to be due to lignin or hemicellulose contents of the vetch rather than residue nitrogen content or C:N ratio (Honeycutt et al., 1993).

**2.5 Physical Properties**—Soil organic matter influences physical, chemical, and biological properties of the soil. Physical effects of organic matter on soil include improved soil structure, increased aeration, and increased water holding capacity and decreased density. These physical modifications to soil structure modify conditions for root development. Enhanced root development improves water use efficiencies and nutrient uptake.

2.5.1 Most agricultural cropping systems return relatively low amounts of organic matter to soil as crop residues. Soil structure is damaged under continuous cropping systems. Over time, this reduces root penetration and development, and soil aeration. Crop yields are negatively affected by the decreased soil aeration and drainage, due to the depletion of organic matter and increase in soil bulk density. Compost amendments can reverse many negative factors associated with intensive crop production.

**2.6 Organic Matter and Aggregate Sizes**—Soil aggregates are not random assemblages of small particles, but are stabilized aggregates of increasingly larger units that are held together by different organic binding agents. Aggregate formation is a continuum.

2.6.1 Among the physical properties affected by organic matter, the degree of aggregation is fairly well studied (Piccolo and Mbagwu, 1990). Direct correlation were found between total organic matter and aggregate stability (Christenson, 1986). A recent approach in the evaluation of mineralizable organic carbon and nitrogen is to establish aggregate size distribution of organic matter. Some investigators observed that the different size fractions of organic matter are more important to predict organic matter turnover (Cambardella and Elliott, 1992; Elliott, 1986, Janzen et al., 1992).

2.6.2 Organic matter can be fractionated into light and heavy fractions. The light fraction which includes particulate organic matter (POM) is labile, mineralizable, and plays a role in carbon and nitrogen turnovers (Janzen, 1987; Janzen et al., 1992). The light organic matter fraction consists of organic material in various stages of decomposition and has a density of less than  $1.6 \text{ g cm}^{-3}$  (Janzen, 1987; Janzen et al., 1992; Cambardella, 1994). The relative concentration of carbon and nitrogen in this fraction is high compared to the heavy organic matter fraction (Cambardella et al., 1992; Strickland and Sollins, 1987). Organic matter concentrations may differ within particle size fractions. The enriched labile fraction (ELF) of organic matter is used to bind soil particles and form aggregates. As aggregate size increases, the ELF is protected more and more from microbial attack and remains in the soil for

longer periods of time unless mechanical disturbances occur (Cambardella, 1994). Particulate organic matter (POM) is the organic matter fraction embedded in aggregate structure that is more exposed to microbial attack than ELF. The degree of physical occlusion (i.e. POM occludes ELF) can limit the physical accessibility of carbon and nitrogen sources to microbes. Particulate organic matter which consists primarily of decaying plant roots, is much lighter than ELF and is highly influenced by soil management (Cambardella and Elliott, 1993; Wander et al., 1994). The POM may be a major pool for supplying plant available nutrients. The heavy fraction which can be separated by density or sieving, is mostly associated with the clay fraction (Cambardella and Elliott, 1992; Cambardella and Elliott, 1993).

2.6.3 In a recent study, Cambardella and Elliott (1994) found high organic carbon and nitrogen associated with macroaggregates. Further, they found that 18% of the total carbon and 25% of the total nitrogen in no-till soil was associated with fine-silt size particles having a density of  $2.07$  to  $2.22 \text{ g cm}^{-3}$ . Piccolo and Mbagwu (1990) studied the effect of organic waste (pig slurry, cattle slurry, and sewage sludge) amendments to evaluate their influence on aggregate stability and molecular sizes of humic substances. They separated the surface soil into microaggregates of sizes 250-125, 125-50, and  $< 50 \mu\text{m}$ . The organic waste amendment linked together the fine particles promoting the formation of stable aggregates. Microaggregate stability is well correlated with the humic substance fraction of organic matter (Piccolo and Mbagwu, 1990; Chaney and Benson, 1984).

**2.7 Soil Structure and Stability**—Intensive agricultural management systems that do not return significant quantities of plant residues to a soil cause degradation of soil structure and severe soil erosion (Campbell, 1982; Elliott, 1986). Soil structure is intimately related to soil aggregate stability, which is dependent upon the presence of organic matter and organic binding agents. Organic matter has chemical and biological agents that act to glue soil particles together (Rose, 1991). Proper soil aggregation provides large, structured soil pores. Large aggregates formed in the presence of organic matter are non-capillary pore spaces through which air penetrates and excess water is drained.

2.7.1 Three types of organic binding agents have been classified: i) transient - rapidly decomposable polysaccharide; ii) temporary - roots and fungal hyphae; and iii) persistent - lignin, cellulose, hemicellulose (Tisdall and Oades, 1982). Soil aggregates are categorized into two relative size

classes: i) macroaggregates; and ii) microaggregates. Macroaggregates are bound by temporary binding agents such as roots and fungal hyphae, and may be destroyed with tillage. Microaggregates are bound by persistent organic agents independent of management, and are not destroyed by cultivation (Tisdall and Oades, 1982). Cambardella and Elliott (1993) observed that no-till management can ameliorate the detrimental effects of intensive cultivation by promoting macroaggregate stability and increasing organic carbon and nitrogen accumulation.

2.7.2 The addition of municipal waste to soils decreases soil bulk density (Kreft, 1987; Tester, 1990). The decrease in bulk density is due to both a dilution effect and an increase in non-capillary pore space. In a preliminary analysis, municipal solid waste compost moldboard plowed at 20 cm on a loamy sand soil showed lower bulk density values compared to the unamended control (Mamo et al., 1993).

2.8 *Water Retention and Infiltration*—Soil organic matter increases soil water holding capacity. This is particularly salient for coarse, well drained soils, where water infiltration rates are high and irrigation is required to maintain viable crop production. Kreft (1987) found an increase in soil moisture on a loamy sand soil with additions of municipal solid waste compost. Plant available water and water available for microorganisms may not rise with additions of MSW compost (Pera et al., 1983). Kreft (1987) and Cook et al., (1994) demonstrated that water retention increases upon the addition of MSW compost, but plant available water for the fine soils did not increase. Turner et al. (1994) reported an increase in water holding capacity of sandy soils amended with MSW compost with no apparent increase in plant available water. Stabilized organic matter in soils can retain up to four times its own weight of water but only about one half of this may be available to plants (Simpson, 1983). This is due in part to the higher water tension of the organic matter and the general increased presence of soluble salts.

2.9 *Heat Retention*—The presence of humic substances with their unique colloidal chemistry gives soil a dark brown color - contributing to higher absorption of radiation. The volumetric heat capacity of organic matter is higher than all other components of the soil with the exception of liquid water.

2.9.1 Organic matter can lower the overall soil thermal conductivity of well to excessively well drained

soils and organic matter additions to poorly to very poorly drained soils can increase the overall soil thermal conductivity by enhancing the air capacities of these soils.

2.10 *Variable Rate Compost Applications*—Advantages and disadvantages of compost applications are considered to construct the conceptual model to optimize compost applications to manage spatially variable soil conditions. As soil physical, chemical and biological conditions vary across the landscape, so do the relative benefits of nutrient applications (Malo and Worcester, 1975). Because compost is an expensive, relatively scarce and sometimes toxic soil amendment, it is difficult to justify high rate applications across entire fields and farms. Computer-controlled technologies, global positioning systems (GPS), satellite and low altitude aerial imagery, and geographic information system (GIS) are effective tools for mapping and optimizing variable rate compost applications.

2.11 *Organic Matter Management and Spatial Modeling*—Soil attribute characteristics derived from remotely sensed imagery of bare soil provides high resolution models that accurately express soil texture variability, soil drainage patterns, soil organic matter variations and other soil attributes that influence soil water-holding characteristics. A soil organic matter management plan focuses on manipulations of soil water-holding characteristics by tailoring compost application rates and frequencies to the natural soil patterns in the farming landscape (i.e., high application rates and frequencies of compost in areas with low organic matter content, and low application rates and frequencies in areas with high organic matter content).

2.11.1 Digital imagery, (e.g., remotely sensed near infra-red [NIR] imagery of bare soil), lends itself to modeling spatial variations in soil parameters and can indicate optimal, suboptimal or possibly inappropriate landscape positions or locations for compost applications within a field (Zheng and Schreier, 1988). For example, the light zones in Fig 05.07-1 (high NIR reflectance) indicate low soil organic matter content, low water-holding capacity and coarse soil texture (Fig 05.07-2). At the opposite end of this scale, dark zones (high NIR absorbance) indicate high soil organic matter content, high water-holding capacity and fine soil texture.

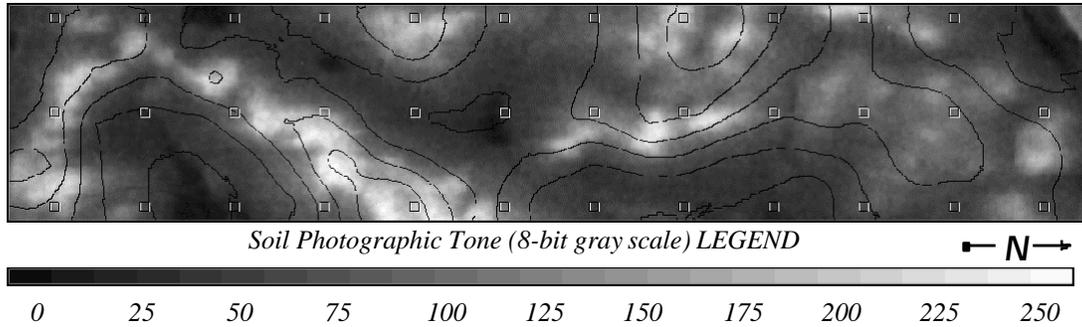


Fig 05.07-1 Gray scale image (8-bit) of a low-altitude color infra-red aerial photograph of bare soil. The uniformly spaced small squares within the image represent soil sample collection points.

ADAPTED FROM—Thompson and Robert, 1995.

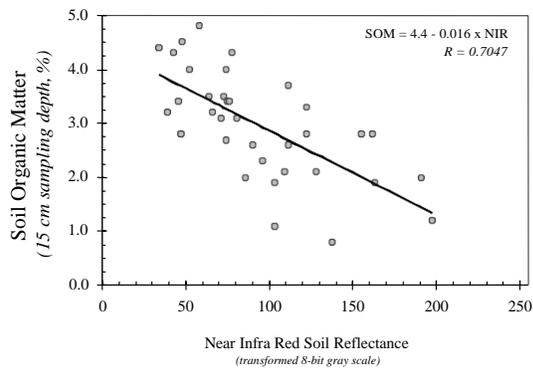


Fig 05.07-2 Soil Organic Matter model used to calibrate a remotely sensed image of bare soil. Calibration technique to transform remotely sensed imagery into spatially variable soil attributes.

**2.12 Practicality of Variable Rate Applications—**Significant research has not yet been conducted to adequately demonstrate the theoretical benefits of variable rate compost applications. Related landscape studies and small plot research on individual soils indicate that appropriately defined compost applications are beneficial and will significantly enhance the productivity of most soils. Throughout the review of organic matter, it is repeatedly reported that compost applications do modify soil physical, chemical and biological characteristics. Extrapolation of these concepts to optimize variable rate applications suggests a viable strategy for efficient and optimized utilization compost products. Bulk application equipment is available that, with minor modifications, will accommodate computer-controlled variable rate compost applications (Fig 05.07-3).



PROVIDED BY—Imagery from Highway Equipment Co., IL (1997).

Fig 05.07-3 Bulk compost spreading equipment can be modified to facilitate variable rate compost applications.

### 3. Referenced Documents

#### 3.1 TMECC Methods:

- Method 02.02-A Sample Mixing and Splitting.
- Method 02.02-C Man-Made Inert Removal and Classification.
- Method 02.02-D Milling and Grinding Samples, Harrison Method, or
- Method 02.02-E Milling and Grinding Samples, Munter Method.
- Method 02.02-F Modifications for Feedstock Sample Preparation.
- Method 03.02-B Milled Material Ignited at 550°C with Inerts Removal.
- Method 03.09-A Total Solids and Moisture.
- Section 04.01 Organic Carbon.

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#### 4. Terminology

4.1 *ash, n*—The inorganic material, or mineral residue of total solids that remains when a compost or feedstock is combusted at 550°C in the presence of excess air; *fixed solids*, % g.g<sup>-1</sup>.

4.2 *biodegradable volatile solids, n*—The organic carbon compounds of total solids that volatilize to carbon dioxide and other gasses when a compost or feedstock is combusted at 550°C in the presence of excess air, % g.g<sup>-1</sup>.

4.3 *compostable, n*—Biodegradable materials that decompose significantly during the retention time of a self-heating composting process; biodegradable materials that readily degrade to carbon dioxide and water when incorporated into a compost pile.

4.4 *fixed solids, n*—The inorganic material, or mineral residue of total solids that remains as ash when a compost or feedstock is combusted at 550°C in the presence of excess air; *Ash*, % g.g<sup>-1</sup>.

4.5 *humic substances, n*—They are complex organic fractions, usually formed as byproducts of decomposition that resist further degradation. Humic

acid, fulvic acid, and humin are humic substances. They are chemically complex substances of high molecular weight, and tend to be amorphous, dark-colored, hydrophilic and acidic. Two stable components of humic substances that play a dominant role in soil physical properties are humic and fulvic acids. These weak acids are also present in organic waste and are suggested to be chemically and structurally similar to humic substances in soil (adapted from Sposito et al., 1982).

4.5.1 *fulvic acids, n (FA)*—fraction of humic substances that solubilize in an alkali solution and is not precipitated by acid. It can form water-soluble complexes at any pH and exhibits a greater affinity for  $\text{Fe}^{3+}$  and  $\text{Al}^{3+}$  than other cations. This affinity varies with pH.

4.5.2 *humic acid, n (HA)*—fraction of humic substances that solubilize in dilute alkali conditions and is precipitated by acid. It can form water-soluble complexes at pH's greater than 6.5, but below this pH humic acid is insoluble.

4.5.3 *humin, n*—fraction of humic substance that does not solubilize in either weak acid or alkaline solution.

4.6 *moisture content, n*—The liquid fraction (percentage) of a compost or feedstock that evaporates at  $70\pm 5^\circ\text{C}$ , %  $\text{g}\cdot\text{g}^{-1}$ .

4.7 *organic carbon, n*—biologically degradable carbon containing compounds found in the soil or compost organic fraction. They originate from sugars, starches, proteins, fats, hemicellulose, cellulose and lignocellulose that are found in composting feedstock and are biologically degraded during composting and curing. Other organic carbon forms that are generally not degraded biologically include petroleum and petroleum byproducts, such as plastics and contaminated oils. They can be degraded by physical means, for example if the temperature is sufficiently high. It does not include inorganic carbonate concretions such as calcium and magnesium carbonates.

4.8 *organic matter fractions, n (e.g., humic substances: fulvic acid; humic acid; and humin)*—complex mixtures of polymeric organic molecules that cannot be separated into homogeneous molecules and cannot be precisely defined in chemical terms. Fraction ratios vary directly with the strength of base and acid employed in the extraction/separation procedure.

4.9 *organic matter, n (OM)*—the sum of solids in compost that contain organic carbon (adapted from Schnitzer, 1991); the total organic components in compost including undecayed plant and animal tissues,

their partial decomposition products, and the compost biomass exclusive of living macrofauna and macroflora (adapted from Vaughan et al., 1985).

4.10 *organic matter, n*—the sum of solids in compost that contain organic carbon; the total organic components in compost including undecayed plant and animal tissues, their partial decomposition products, and the compost biomass exclusive of living macrofauna and macroflora.

4.11 *oxidizable carbon, n*—Equivalent to total organic carbon and relative to oxidant employed. Oxidizable carbon is measured by Walkley Black methods devised for use in mineral soils.

4.12 *total solids, n*—The solid fraction of a compost or feedstock that does not evaporate at  $70\pm 5^\circ\text{C}$ , which consists of fixed solids, biodegradable volatile solids, and volatile solids that are not readily biodegradable, %  $\text{g}\cdot\text{g}^{-1}$ .

4.13 *volatile solids, n*—Materials that volatilize to carbon dioxide and other gasses when a compost or feedstock is combusted at  $550^\circ\text{C}$  in the presence of excess air, %  $\text{g}\cdot\text{g}^{-1}$ . The sum of biodegradable solids that degrade during composting, non-biodegradable solids and biodegradable solids that do not degrade during the retention time allowed for composting.

## 5. Summary of Test Methods

5.1 *Organic Matter Determinations*—Identification and development of a suitable extractant or determination method for organic matter is a major research interest among soil scientists. Procedures commonly used are dichromate oxidation, peroxide oxidation, hot alkali extraction, and loss on ignition (LOI).

5.2 *Method 05.07-A Loss-On-Ignition Organic Matter Method (LOI)*—Organic matter content of a compost sample is determined by igniting an oven-dried sample in a muffle furnace at  $550^\circ\text{C}$ . The volatilized material is the organic matter fraction and the remaining ash is the mineral fraction.

5.2.1 The LOI method is a direct determination of compost organic matter. The method is rapid, easy, precise and accurate for properly prepared samples. The compost method is based upon methods developed for use with peat and organic soils.

5.2.2 In the interest of improving intra-laboratory precision and to decrease the time required to complete analysis,  $550^\circ\text{C}$  was accepted as most appropriate ashing temperature for organic matter determinations on compost and composting feedstock samples.

5.2.2.1 The method adheres to protocols of similar methods provided in ASTM and AOAC: Test Method

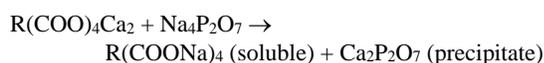
C from ASTM D 2974 - 87 (Reapproved 1995). Standard Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils; Test Method 967.05 (final action 1967) from AOAC Official Methods of Analysis Organic Matter in Peat (1990).

5.2.2.2 The range of temperatures used for soil organic matter determination varies from 360 in mineral soils up to 750°C for some organic materials. The organic material fraction is volatilized and the mineral fraction is retained as ash. Percent organic matter content is obtained by difference relative to the bulk oven-dried sample.

*5.3 Method 05.07-B Humic Substances - Proposed Fulvic Acid and Humic Acid Extraction and Characterization*—Humic substances are solubilized with a strong base and extracted. Fractionation and purification is performed to separate fulvic acids from humic acids. Determinations are performed with spectrophotometers following the principle that organic matter absorbs in the ultra violet, visible, and especially the infrared regions.

5.3.1 Humic and fulvic acids are soluble in basic media and can be extracted from soil and organic materials using aqueous alkali solutions. Fulvic acids are soluble at all pH ranges, while humic acids are soluble in basic media only.

5.3.2 The reaction below shows the principle behind the extraction.



where:

R = aliphatic or aromatic carbon chain skeleton.

5.3.3 Humus makes up a large fraction of organic matter and is important in soil ecology, soil fertility and soil structure. Total organic carbon of compost also contains humic substances that include fulvic and humic acids. The proportion of humus within compost increases with compost stability. In general, the relative proportion of humic carbon to the total organic carbon content of organic matter increases as compost stabilizes. This relationship varies with the nature of the raw materials used to form the compost. Raw materials high in lignin usually yield greater amounts of humus than materials low in lignin.

5.3.4 Humic substances may be beneficial to compost, especially if there are high concentrations of heavy metals within the feedstock. This is because humic acids readily form complexes or chelates with metals e.g., Zn, Mn and Fe reducing the concentration of soluble metals in solution.

*5.4 Method 05.07-C Organic Matter Decomposition Calculations*—The organic matter fraction (OM),

occasionally referred to as the biodegradable volatile solids fraction (BVS), of total solids diminishes during the composting as a function of controlled biological decomposition. The total solids fraction includes inorganic materials that remain as ash after ignition at 550°C, the volatile solids in feedstock that biodegrade, and volatile inorganic materials remaining in a finished product such as sand, stones, carbonate concretions, plastic, metal and glass. As feedstock products are degraded, they become biologically stable; carbon dioxide and water are byproducts under aerobic conditions while methane is the main byproduct under anaerobic conditions. This test provides a mechanism for tracking the decomposition process by measuring and documenting changes in organic matter content of materials at multiple stages of the composting process.

5.4.1 Organic matter content is determined for the same material at different stages of a batch composting process, from feedstock preparation to screening and packaging. Samples are ashed at 550°C as described in Method 05.07-A LOI Organic Matter, and percent reduction in organic matter content due to decomposition during the composting process is calculated.

## **6. Interference and Limitations**

6.1 Samples high in petroleum based inert material (hard plastics) or inorganic carbon (carbonates) may significantly inflate compost organic matter determinations if organic matter content is approximated solely from carbon content.

6.1.1 Film plastics alone cause less error because of their minor impact on overall sample mass.

6.1.2 It is imperative to measure inert plastic content of a compost with a parallel sample and correct for carbon contributed by petroleum-based plastics.

*6.2 Method 05.07-A Loss-On-Ignition Organic Matter Method (LOI):*

6.2.1 Deviation from the recommended ashing temperature of 550°C will introduce significant error. Lower combustion temperature can produce a significantly lower LOI OM result.

*6.3 Method 05.07-B Humic Substances - Proposed Fulvic Acid and Humic Acid Extraction and Characterization*—Alkali solutions employed with this method, namely sodium hydroxide and sodium pyrophosphate cause slight oxidation of organic matter, dissolve cellular components of plant residues and other lignins of organic matter that are not yet humified. This tendency alters the expected value representing humic substances. When sodium pyrophosphate is used as an extractant, removal of

phosphate from extracted organic matter is not practicable and will interfere with the analysis.

**6.4 Method 05.07-C Calculations for Organic Matter Decomposition**—Reduction in organic matter is one of the original test methods used to approximate biosolids stability. The reduction of organic matter in compost is not a stand-alone indicator of compost stability or maturity; other indicators must be considered such as C:N ratio, respirometry, pH, bulk density, ammonium to nitrate ratio, etc.

6.4.1 This protocol was designed for compost samples and accounts for the inert content of compost.

6.4.2 The protocol is valid only in batch composting processes when samples are taken on the same composting materials, after initial screening, in-process, and again before final screening.

6.4.3 This test is not applicable for continuous composting processes. By virtue of the continuous blending and multiple screening steps built into most continuous systems, tracking a batch through the process is not practical and prone to significant systematic error.

## **7. Sample Handling**

**7.1 Method 05.07-A Loss-On-Ignition Organic Matter Method (LOI)**—Compost samples should be air-

dried at 36°C and sieved through 9.5-mm sieve. Inert materials, especially plastics and plant debris should be removed. If the sample is high in carbonate, an acid wash treatment may be necessary to remove carbonates.

**7.2 Method 05.07-B Humic Substances - Proposed Fulvic Acid and Humic Acid Extraction and Characterization**—Samples must be air dried at 36°C.

**7.3 Method 05.07-C Calculations for Organic Matter Decomposition**—Follow sample collection protocols as described in 02.01 Field Sampling of Compost Materials.

### **7.4 Test Sample Aliquot Size:**

7.4.1 *Compost Samples*—150 cm<sup>3</sup>;

7.4.2 *In-Process Samples*—250 cm<sup>3</sup>; or

7.4.3 *Feedstock Samples*—750 cm<sup>3</sup>.

7.5 Prepared samples are air-dried, inerts are separated, the compostable materials are milled to a fine powder (< 0.5 mm) and thoroughly mixed. The milled sample shall not contain materials that are not compostable.

Test Method: Organic Matter. Loss On Ignition Method						Units: % g <sup>-1</sup> dw		
Test Method Applications								
Process Management						Product Attributes		
<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>
		05.07-A		05.07-A		05.07-A		05.07-A

## 05.07-A LOSS ON IGNITION METHOD

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

### 8. Apparatus for Method A

8.1 *Oven*—forced air, set at 70±5°C.

8.2 *Muffle Furnace*—set at 550°C.

8.3 *Sieves*—1-mm stainless mesh sieve.

8.4 *Analytical Balance*—accurate to ± 1.0 mg (e.g., Mettler instruments, or equal).

8.5 *Sample Containers*

8.5.1 *Crucibles (for small sample aliquots)*—ceramic, carbon free (alundum, zircon, or equal).

8.5.2 *Beaker (for large sample aliquots)*—150 mL, Pyrex or equivalent (optional, if larger sample size is preferred).

8.6 *Desiccator*—equipped with calcium chloride as a desiccant (Fisher Scientific, or equal).

### 9. Reagents and Materials for Method A

9.1 *Hydrochloric Acid*—0.05 N HCl.

### 10. Procedure for Method A

10.1 Oven dry a 10-g compost sample in a forced-air oven set at 70±5°C until sample weight change diminishes to nil, approximately 2 h for air-dried samples and up to 24 h for as-received moist material.

NOTE 1A—Use a larger sample (approximately 100 cm<sup>3</sup>) if within sample heterogeneity is significant. This will minimize error associated with sample heterogeneity.

10.2 Cool the oven-dried sample in a desiccator and record the oven dry weight, dw (±0.001 g).

10.3 Remove carbonates by wetting the sample with excess 0.05 N HCl. Add acid until foaming ceases.

10.3.1 Dilute the excess acid with distilled water.

10.3.2 Drive off excess moisture from the carbonate-free sample aliquot by oven-drying at 75°C until weight change due to moisture loss diminishes to nil. Measure and record the oven-dry weight of the sample aliquot.

10.4 Place the sample in a muffle furnace. Slowly ramp the furnace temperature to 550°C. Combust the sample at 550°C for 2 h and then slowly ramp the furnace temperature down to approximately 200°C.

10.5 Remove the ashed samples from the furnace, transfer them to a desiccator and allow them to cool to ambient laboratory temperature.

10.6 Measure and record net ashed weight, AshW (±0.001 g) of each sample.

### 11. Calculations for Method A

11.1 *Organic matter using Loss On Ignition:*

$$OM = (1 - \text{AshW} \div \text{dw}) \times 100 \quad \text{Equation 11.1}$$

where:

OM = percent LOI organic matter, %.

AshW = sample net weight after ignition at 550°C, g, and

dw = sample net weight after drying at 70±5°C before ignition, g.

<b>Test Method:</b> Organic Matter. Humic Substances						<b>Units:</b> mg kg <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>
		05.07-B	05.07-B	05.07-B				05.07-B

## 05.07-B HUMIC SUBSTANCES - PROPOSED FULVIC ACID AND HUMIC ACID EXTRACTION AND CHARACTERIZATION

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

### 12. Apparatus for Method B

- 12.1 *Drying Oven*—forced air, vented drying set at 70±5°C.
- 12.2 *Sieves*—4-mm and 6.3-mm mesh plastic sieves.
- 12.3 *Mechanical Shaker*—reciprocal or equivalent, (e.g., Tyler Model RX-86).
- 12.4 *Sample Bottles*—200-mL, polypropylene.
- 12.5 *Centrifuge*—capable of 20,000 g.
- 12.6 *Spectrophotometer*—double beam, Perkin-Elmer or equivalent.
- 12.7 *Infrared Spectrometer*—Perkin-Elmer or equivalent.

### 13. Reagents and Materials for Method B

- 13.1 *Water*—deionized, minimum resistivity 17 MΩ·cm minimum standard.
- 13.2 *Sodium Hydroxide*—0.1 N NaOH; or *Sodium Pyrophosphate*—0.1 N Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub>.
- 13.3 *Hydrochloric Acid*—0.05 N and 2.0 N HCl.
- 13.4 *Sulfuric Acid*—0.05 N H<sub>2</sub>SO<sub>4</sub>.
- 13.5 *Cation Exchange Resin*—Amberlite IR-20 or Dowex -50 hydrogen form.
- 13.6 *Sodium Bicarbonate*—0.05 N NaHCO<sub>3</sub>.
- 13.7 *Potassium Bromide*—KBr, spectroscopic purity.

### 14. Procedure for Method B

#### 14.1 Extraction:

- 14.1.1 Leach sample with excess 0.05 N HCl to remove carbonates until foaming ceases.
- 14.1.2 Decant excess acid and wash residue with distilled water.
- 14.1.3 Air-dry the sample and transfer 10 g of treated sample into 200-mL polypropylene flask.
- 14.1.4 Add 100 mL of 0.1 N NaOH.

14.1.5 Replace headspace air in the flask with N<sub>2</sub> gas, stopper and shake flask for 24 h.

14.1.6 Centrifuge mixture at 10,000 revolutions per min for 10 min.

14.1.7 Decant supernatant into polypropylene container.

14.1.8 Repeat steps 14.1.4 through 14.1.7 two or three times [2× - 3×].

#### 14.2 Fractionation:

14.2.1 Suspend the residue in 50 mL of distilled water.

14.2.2 Collect washing in same polypropylene container used in step 14.1.7.

14.2.3 Acidify alkaline extract to pH 2 with 2 N HCl, leave extract at room temperature (25°C) for 24 h.

14.2.4 Separate soluble material by centrifugation, centrifuge mixture at 10,000 revolutions per min for 10 min.

NOTE 1B—The soluble material contains fulvic acid (FA), and coagulated contains humic acid (HA). Centrifugation separates supernatant from precipitate.

14.2.5 Freeze dry both fractions.

#### 14.3 Purification of Fulvic Acid (FA):

14.3.1 Apply aqueous solution of FA 2× to 3× in succession over hydrogen form resin.

14.3.2 Pass 1 N NaOH through resin and collect elute.

14.3.3 Freeze dry residue.

#### 14.4 Purification of Humic Acid (HA):

14.4.1 Weigh 1 g of HA in polypropylene bottle; add 100 mL of HCl-HF to bottle; shake mixture for 24 h at 25°C; filter extract through sieve.

14.4.2 Repeat step 14.4.1 3× or 4×.

14.4.3 Wash residue with distilled water and dry.

#### 14.5 Absorption Method for Characterization of Humic Materials (HA or FA):

14.5.1 Dissolve 2 to 4 mg of FA or HA in 10 mL of 0.05 *N* NaHCO<sub>3</sub>.

NOTE 2D—pH should be near 8.0.

14.5.2 Measure absorption at 465 and 665 nm.

NOTE 3D—Use 0.05 *N* NaHCO<sub>3</sub> in the reference cell.

14.5.3 Obtain ratio of absorption,  $E/E_0$ .

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*14.6 Infrared Spectrometry for Characterization of Humic Materials (HA or FA):*

14.6.1 Mix 1.0 mg of FA or HA with 400 mg of dry KBr pellets.

14.6.2 Press into suitable die under vacuum at pressure of 7,500 kg cm<sup>-2</sup> for 20 min.

14.6.3 Measure frequency bands of functional groups.

<b>Test Method:</b> Organic Matter. Calculations for Organic Matter Decomposition						<b>Units:</b> % g <sup>-1</sup> dw		
<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>
05.07-C	05.07-C	05.07-C		05.07-C	05.07-C	05.07-C		05.07-C

## 05.07-C CALCULATIONS FOR ORGANIC MATTER DECOMPOSITION

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

### 15. Apparatus for Method C

15.1 *Balance*—analytical, with accuracy of  $\pm 0.001$  g.

15.2 *Desiccator Cabinet*—vacuum with desiccant tray containing a color indicator of moisture concentration or an instrument indicator.

15.3 *Drying Ovens*—two, forced-air, vented, set at  $70 \pm 5^\circ\text{C}$  and  $36^\circ\text{C}$ .

15.4 *Sample Containers*—glass beakers capable of withstanding temperatures above  $550^\circ\text{C}$ , (e.g., Pyrex, etc.); use 150-mL crucibles or beakers with compost samples, and 500-mL beakers with in-process and feedstock samples.

15.5 *Furnace*—forced air muffle, set at  $550^\circ\text{C}$ .

15.6 *Mill or Grinder*—capable of milling feedstocks to a fine power, i.e., particle size of  $<0.5$ -mm.

15.7 *Sieve*—4-mm mesh, plastic or stainless steel, approximately 30-cm diameter, with capture pan.

15.8 *Watch Glass*—5-cm (2-in.) diameter for 150-mL beakers, and 10-cm (4-in.) diameter for 500-mL beakers.

### 16. Reagents and Materials for Method C

16.1 *None required.*

### 17. Procedure for Method C

17.1 *Sample Aliquot Preparation:*

17.1.1 Dry sample aliquots in a forced-air, vented oven until weight change due to moisture loss diminishes to nil:

17.1.1.1 *Compost Samples*—air dry a 150 cm<sup>3</sup> sample aliquot at  $36^\circ\text{C}$ ;

17.1.1.2 *In-Process Samples*—air dry a 250 cm<sup>3</sup> sample aliquot at  $36^\circ\text{C}$ ; or

17.1.1.3 *Feedstock Samples*—oven dry a 750 cm<sup>3</sup> sample aliquot at  $70 \pm 5^\circ\text{C}$ .

17.1.2 Separate the sample into two size fractions with the 4-mm sieve. Gently rub as much material as

practical through the 4-mm sieve. Retain each size fraction for further processing.

NOTE 1A—Inert materials that adhere to aggregates of composted particles are more easily separated when samples are air-dried rather than oven-dried. Oven-drying often causes the fragments to strongly adhere, making the segregation process very difficult.

17.1.3 Spread the  $>4$ -mm sample onto a clean laboratory tray. Separate the non-compostable materials from the compostable materials. Non-compostable materials do not readily humify. Retain all compostable and non-compostable material separately for further processing.

17.1.4 Recombine the  $>4$ -mm compostable fraction with the  $<4$ -mm fraction. Grind or mill the recombined compostable fraction to a fine powder ( $<0.5$  mm).

17.2 *Preparation of Evaporating Dish:*

17.2.1 Heat the clean crucibles or beakers to  $105^\circ\text{C}$  for approximately 1 h to drive off all hygroscopic moisture.

17.2.2 Place heated beakers or crucibles in a desiccator cabinet to cool to ambient laboratory temperature.

17.2.3 Weigh the crucibles or beakers and record the dry tare weights immediately prior to use.

17.3 *Oven Dry Each Fraction:*

17.3.1 Oven dry the milled compostable fraction at  $70 \pm 5^\circ\text{C}$  in a forced-air oven for 18 h to 24 h, until weight change diminishes to nil. Cool the sample to ambient laboratory temperature in a desiccator cabinet. Record the oven dry weight, i.e., mass of the compostable fraction solids ( $S_c$ ).

17.3.2 Oven dry the non-compostable fraction at  $70 \pm 5^\circ\text{C}$  as described above and obtain the mass of non-compostable solids ( $S_N$ ).

**17.4 Organic Matter Content:**

17.4.1 *Compostable Fraction Test Aliquot*—Transfer a representative aliquot of the milled compostable fraction to a beaker or crucible:

17.4.1.1 *Compost Test Aliquot Size*—50 cm<sup>3</sup> test aliquot;

17.4.1.2 *In-Process Test Aliquot Size*—150 cm<sup>3</sup> test aliquot; or

17.4.1.3 *Feedstock Test Aliquot Size*—250 cm<sup>3</sup> test aliquot.

17.4.2 Weigh and record the mass of the crucible or beaker, and test aliquot. Subtract the tare weight to determine the mass of the test aliquot (S<sub>A</sub>).

17.4.3 Place a watch glass over the mouth of each crucible or beaker; place the crucibles or beakers containing the compostable fraction test sample aliquot in the muffle furnace. Ramp the muffle furnace temperature to 550°C and ash the samples at 550°C for two h.

17.4.4 Allow the muffle furnace to cool by ramping the furnace temperature down to approximately 200°C; transfer the ashed sample crucibles or beakers with watch glass in place to a desiccator and cool to ambient laboratory temperature.

17.4.5 Remove the watch glass. Weigh and record the gross mass of the sample containers and ash; calculate the net weight of ash (A<sub>A</sub>) in the test aliquot.

17.5 Track organic matter decomposition through the composting process.

17.5.1 Repeat the determination of organic matter content (Equation 18.1.1) for samples collected at each stage of the composting process and for each batch of interest; repeat steps 17.1 through 17.4 for each organic matter decomposition sample.

**18. Calculation for Method C**

18.1 *Calculate the organic matter content for each test sample:*

$$OM = V_C \div S_T \times 100 \quad \text{Equation 18.1.1}$$

where:

- OM = organic matter content, % dw basis,
- V<sub>C</sub> = compostable material volatilized from the sample, calculated g, and
- S<sub>T</sub> = combined mass of solids, calculated g,

and:

$$V_C = S_C \times VS_A \quad \text{Equation 18.1.2}$$

$$S_T = S_C + S_N \quad \text{Equation 18.1.3}$$

$$VS_A = 1 - A_A \div S_A \quad \text{Equation 18.1.4}$$

where:

- S<sub>A</sub> = dry mass of the milled test aliquot before ashing, measured g,
- A<sub>A</sub> = dry mass of the milled test aliquot after ashing, measured g,
- VS<sub>A</sub> = fraction of dry solids volatilized from test aliquot, calculated unitless ratio,
- S<sub>C</sub> = mass of dry solids for the milled compostable fraction of the original sample, dw basis, measured g, and
- S<sub>N</sub> = mass of dry solids for the non-compostable fraction of the original sample, dw basis, measured g.

18.2 *Calculate organic matter decomposition (D) for finished compost relative to original feedstock blend:*

$$D_3 = C \div F \times 100 \quad \text{Equation 18.2}$$

18.3 *Calculate D for in-process material relative to original feedstock blend:*

$$D_2 = P \div F \times 100 \quad \text{Equation 18.3}$$

18.4 *Calculate D for finished compost relative to in-process material:*

$$D_1 = C \div P \times 100 \quad \text{Equation 18.4}$$

where:

- D<sub>1</sub> = stage one decomposition, ratio of organic matter of finished compost versus the organic matter of its in-process material, %,
- D<sub>2</sub> = stage two decomposition, ratio of organic matter of in-process material versus the organic matter of its feedstock, %,
- D<sub>3</sub> = stage three decomposition, ratio of organic matter of finished compost versus the organic matter of its feedstock, %,
- C = organic matter content of finished compost, %,
- P = organic matter content of in-process material, %,
- and
- F = organic matter content of original compost feedstock blend, %.

## 05.07 METHODS SUMMARY

### 19. Report

#### *19.1 Method 05.07-A Loss-On-Ignition Organic Matter Method (LOI)*

19.1.1 Report LOI organic matter content as a percentage on an oven-dried basis ( $70\pm 5^{\circ}\text{C}$ ) with three significant figures.

19.1.2 Report any deviation from the recommended procedures, (e.g., different ashing temperature, etc.).

19.1.3 If present, report the removal of carbonates from sample.

*19.2 Method 05.07-B Humic Substances - Proposed Fulvic Acid and Humic Acid Extraction and Characterization*—Data for samples are reported as ratios to three significant figures.

*19.3 Method 05.07-C Calculation for Organic Matter Decomposition*—Report organic matter decomposition percentage for each stage of the composting process. Report source material, (e.g., municipal solids waste, yard waste, biosolids, etc.), and feedstock blend components.

19.3.1 Never report organic matter decomposition as a stand-alone indicator of compost stability or maturity; other indicators must be considered such as C:N ratio,

respirometry, pH, bulk density, ammonium to nitrate ratio, etc.

### 20. Precision and Accuracy

*20.1 Method 05.07-A Loss-On-Ignition Organic Matter Method (LOI)*—The precision and bias of this test are being determined. Data are being sought for use in developing a precision and bias statement.

*20.2 Method 05.07-B Humic Substances - Proposed Fulvic Acid and Humic Acid Extraction and Characterization*—The precision and bias of this test have not been determined. Data are being sought for use in developing a precision and bias statement.

*20.3 Method 05.07-C Proposed Calculation for Organic Matter Reduction*—The precision and bias of this test is not determined. Data are being sought for use in developing a precision and bias statement.

### 21. Keywords

21.1 ash; feedstock; in-process compost; finished compost; humus; humic acid; fulvic acid; humin; organic carbon; organic constituents; organic matter; oxidizable carbon; loss on ignition; LOI; organic matter reduction; ash; solids; total solids; volatile solids;





## Attachment G - Solvita Operations Manual



**UPDATED**  
**Version 9.0**

## OFFICIAL SOLVITA<sup>®</sup> GUIDELINE COMPOST EMISSIONS TEST

The Solvita<sup>®</sup> compost test is a widely recognized and easy-to-perform procedure to measure evolution of carbon-dioxide (CO<sub>2</sub>) and volatile ammonia (NH<sub>3</sub>), the two most prominent gaseous emissions of active composts. These indicators are used together to gauge stability and maturity, important co-dependent traits relating to compost quality.

### **THIS MANUAL UPDATES THE FOLLOWING PROTOCOLS from Vers 8.0:**

- Carbon sequestration table added for interpretation of compost value
- New figure added on relationship of Solvita color to CO<sub>2</sub>% in compost sample.
- Updated info on the joint role of CO<sub>2</sub> and Ammonia in determine true maturity.

Solvita<sup>®</sup> is employed with composts and manures for the following purposes:

- 1) To comply with maturity standards (**Table 1** - Maturity Index)
- 2) To evaluate compost status (**Table 2 - 3**) and to determine aeration needs (**Table 4**).
- 3) To determine product best-use (**Table 5**) and ammonia situation (**Table 6**).

### **Scope of Test and Obtaining Satisfactory Results**

The Solvita<sup>®</sup> test may be used to obtain several types of information regarding stability & maturity (co-dependent factors), potential nitrogen-loss, and quantitative respiration rates. Solvita is designed to be a volumetric test and is run at standard sample density (see Chart 1 on next page). Composts are rarely uniform and special attention to proper sampling is recommended. A troubleshooting key is included in the Appendix.

### **QUALITY CONTROL & STORAGE OF SOLVITA KITS**

Solvita<sup>®</sup> kits are pre-calibrated and packaged for highest quality prior to shipping. The sealed probes should be the "Control Color" when the foil pack is opened (see color chart). If the foil packs are damaged or the jar is cracked then the test may not work properly. The probes show Lot No and Expiration Dates on the package. The plastic jars may be reused 4 times, then discarded. Shelf-life is improved by refrigeration. Do not allow gels to freeze.

Solvita<sup>®</sup> is a trademark of Woods End Laboratories, Inc.  
Protected by one or more of the following patents:  
5,320,807 - 6,391,262 - 6,780,646



Vers. 9.0

Detailed sampling instructions are available at:  
[www.woodsend.org](http://www.woodsend.org)

## SAMPLE PREPARATION

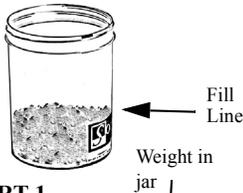
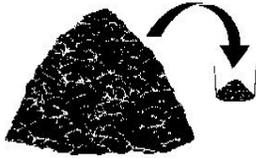


CHART 1.  
Field Density

lbs/ yd <sup>3</sup>	kg/m <sup>3</sup>	g/ jar
500	300	30
800	475	50
1,000	600	60
1,200	700	70



- OBTAIN and PREPARE SAMPLE:** Take several grab samples to prepare a composite by mixing all sub-samples representative of the entire compost. *Remove large wood chips and other objects.* A  $3/8$ " (10mm) sieve is recommended before loading test jar.
- CHECK MOISTURE:** For proper respiration, moisture should be at optimum. A sample that is too dry may give a false positive maturity test (ammonia is still volatile). It may be acceptable to test without moisture adjustment if as-is results are desired (e.g. bagged compost). To determine ideal moisture use the squeeze test<sup>1</sup>. If too dry, carefully add water while mixing and repeat the squeeze test until proper moisture is achieved. Then, *allow to stand for several hours or overnight loosely covered* so the sample equilibrates to the new condition.
- LOADING SAMPLE:** Carefully fill the Solvita jar to the fill line. To obtain proper density tap the jar gently while filling. The proper weight in grams per jar corresponding to field density is found in Chart 1.
- EQUILIBRATION STEP:** Let the sample "air-out" loosely covered in the jar for up to one-hour prior to starting the Solvita test. *If the sample was taken directly from a very hot or frozen pile, it is advisable to allow it to stand at least for 24hrs before starting the test.*
- STARTING TEST:** The Solvita maturity method requires two tests carried out together in the same 4-hr period. Tear open both the pouches marked "High CO<sub>2</sub>" and "Ammonia" and carefully remove each probe. The gel in the probe is color-coded: the carbon-dioxide probe is purple and the ammonia probe is yellow. *Do not touch the gel surface, and don't allow compost to touch it.* Once the pouch is open, the test should be started immediately.
- INSERT PROBES:** Both probes are pushed into the sample in the jar, visible through the clear back panel. The edges of the probes can be touching in the middle at about right angles. Push the probe all the way into the compost to the bottom of the jar. Do not jostle or tip the jar which may coat the sensitive gel probes with compost!
- ATTACH LID:** place the lid containing a red gas-tight gasket on the jar and screw firmly. Keep the jar at room temp. for 4 hrs (68-77°F or 20-25°C) preferably *out of sunlight*.
- READ THE GEL COLOR:** At 4 hours after starting the test, remove probes one at a time and hold next to the proper color chart or use the Digital Color Reader. Compare the gel color to the numbered color scales, finding the closest match (half shades of color may also be read). *Read the color immediately after removing from the jar.* Color matching generally works best under fluorescent lighting.

1. **Squeeze test:** make a fistful of compost. Squeeze very hard. Moisture should appear between fingers but not drip out if compost is at the proper moisture content.

# CTIONS



The Digital Color Reader (DCR) eliminates the subjectivity of color discrimination and notably improves the scale of readability.

9. THE LID LABEL is removable and may be affixed to a notebook page as a record and official proof-of-testing.

10. DETERMINE COMPOST MATURITY INDEX: Using the CO<sub>2</sub> and NH<sub>3</sub> test results, consult **Table 1** below to find the intersection of the two values. It should be noted that composts with no free ammonia (or compost with pH < 7.5) the Index is usually same result as the CO<sub>2</sub> probe. Table 1 corrects for the competition of volatile ammonia with CO<sub>2</sub> rate.

11. TO ESTIMATE GAS EMISSIONS, consult later sections. For aerobic compost, *the inverse of CO<sub>2</sub> (from 21%) is the amount of oxygen consumed in 4 hours.* This can be used to estimate air needs to maintain aerobic respiration (see Table 4).

### FINDING THE CORRECT COMPOST MATURITY INDEX

CO<sub>2</sub> respiration and NH<sub>3</sub> ammonia emissions jointly determine the stability and maturity. Therefore to properly identify compost maturity using the Solvita system both test results must be performed together.

1) As shown in **Table 1** below, cross-reference the two numbers from the test to find the common intersection value which is the *Solvita Maturity Index* (red arrows show example).

2) **Table 2** is a visual guide to aid understanding overall composting status.

**ALWAYS REFER TO THE CURRENT SOLVITA TEST MANUAL provided with each kit for the current interpretation.**

**Table 1. Compost Maturity Index Calculator<sup>a</sup>**

use the Ammonia and CO<sub>2</sub> probe color numbers and read across and down to where the columns meet

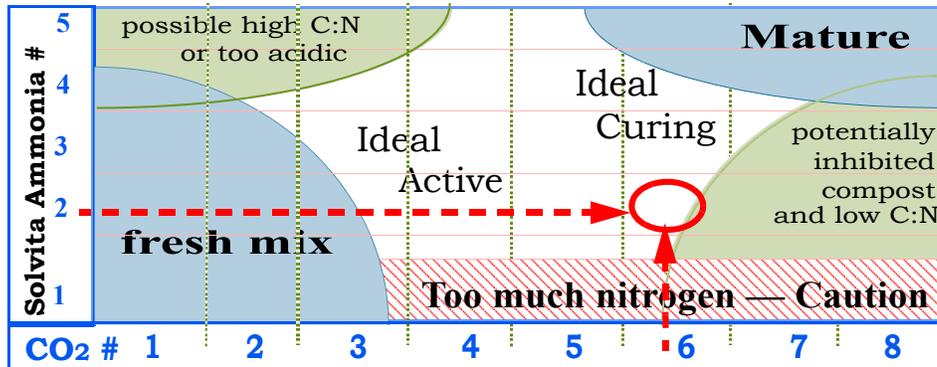
		SOLVITA CO <sub>2</sub> Test Result is:								
		1	2	3	4	5	6	7	8	
Solvita Ammonia Test Result is:	5	VLow / No NH <sub>3</sub>	1	2	3	4	5	6	7	8
	4	Low NH <sub>3</sub>	1	2	3	4	5	6	7	8
	3	Medium NH <sub>3</sub>	1	1	2	3	4	5	6	7
	2	High NH <sub>3</sub>	1	1	1	2	3	4	5	6
	1	Very High NH <sub>3</sub>	1	1	1	1	1	2	3	4

a. Example (red arrows): If the NH<sub>3</sub> result is 2, and the CO<sub>2</sub> result is 6, then the Maturity Index is: 4

## STATUS AND CONDITION OF COMPOST PROCESS

Using both Solvita results Table 2 indicates where in the general process compost may be. Table 3 based on the Maturity Index can be used to infer the overall condition.

**Table 2 STATUS OF COMPOSTING PROCESS**



Example: If the NH<sub>3</sub> result is 2, and the CO<sub>2</sub> result is 6, then the process is *Active* bordering on too low C:N

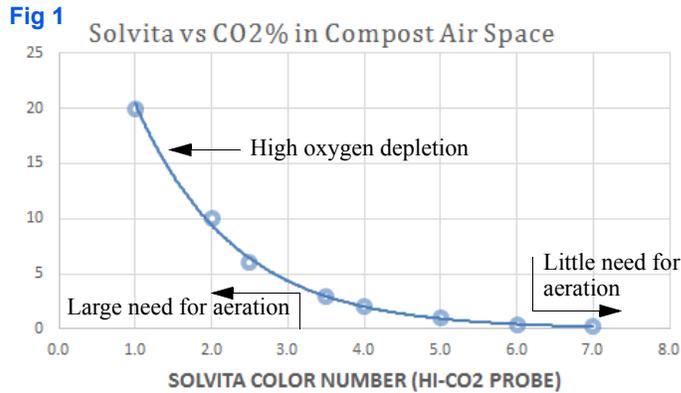
**Table 3 CONDITION OF COMPOST BASED ON MATURITY INDEX**

<b>Maturity Index from Table 1</b>	<b>8.</b>	Inactive, highly matured compost, very well aged, possibly over-aged, like soil; no limitations for usage	“FINISHED” COMPOST
	<b>7.</b>	Well matured, aged compost, cured; few limitations for usage	
	<b>6.</b>	Curing; aeration requirement reduced; compost ready for piling; reduced management requirements. <i>Solvita 6 and above is commonly recognized as suitable maturity for official uses.</i>	Curing
	<b>5.</b>	Compost is moving past the active phase of decomposition and ready for curing; reduced need for intensive handling	“ACTIVE” COMPOST
	<b>4.</b>	Compost in medium or moderately active stage of decomposition; needs on-going management	
	<b>3.</b>	Active compost; fresh ingredients, still needs intensive oversight and management	Very Active
	<b>2.</b>	Very active, putrescible fresh compost; high-respiration rate; needs very intensive aeration and/or turning	“RAW” COMPOST
	<b>1.</b>	Fresh, raw compost; typical of new mixes; extremely high rate of decomposition; putrescible or very odorous material	



## MANAGING PROPER AERATION

Solvita® CO<sub>2</sub> shows the accumulation of CO<sub>2</sub> in the airspace of the compost (see **Fig 1**). The CO<sub>2</sub> gain is proportional to O<sub>2</sub> depletion. Therefore, the test enables estimation of the need for air exchange to maintain aerobic conditions. Aeration also depends on porosity of the mixture.



**Table 4 OXYGEN DEPLETION AND NEED FOR AERATION**

Solvita Rate†	CO <sub>2</sub> produced / O <sub>2</sub> consumed in Solvita test ‡	In-vessel and large pile systems	Open windrows; short, loosely covered piles; home compost bins §
<b>8.</b>	<b>≤0.4%</b>	Refresh air in 4-days	Essentially self-aerating unless material is very wet or very dense
<b>7.</b>	<b>0.7%</b>	Refresh air in 2-days	
<b>6.</b>	<b>1.2%</b>	Refresh air daily (every 24 hrs)	
<b>5.</b>	<b>2.0%</b>	Refresh air twice per day (every 12 hrs)	The need to turn should be determined by pile size and temperature in core; if hot it should be turned at least monthly
<b>4.</b>	<b>3.0%</b>	Refresh air 4x per day (every 6 hours)	
<b>3.</b>	<b>5.0%</b>	Refresh air 6x per day (every 4 hours)	Should be regularly turned on a scheduled basis until the pile temperature peaks and starts to decline and maturity improves.
<b>2.</b>	<b>8.0%</b>	Refresh air 10x per day (every 2.5 hours)	
<b>1.</b>	<b>≥13%</b>	Refresh air 16x per day (every 1.5 hour)	

† Solvita® Maturity Index (Table 1) or Solvita® CO<sub>2</sub> probe if the Solvita® ammonia is  $\geq 4$

‡ Ambient air is 20.9% O<sub>2</sub> and < 0.04% CO<sub>2</sub>

§ Natural air diffusion in open compost piles may be sufficient for full aeration and depends on the texture, wetness and surface:volume ratio.



## COMPOST CARBON SEQUESTRATION VALUE

Compost may have carbon sequestration potential in dependence on its level of stability or “decay rate” (often used to determine product half-lives). This value can be inferred from the rate of CO<sub>2</sub> respiration during composting. Carbon-storage value is the inverse of the loss rate. Composts with high respiration are losing carbon as CO<sub>2</sub> to the atmosphere at a faster rate than can be justified for sequestration value. Use **Fig 2** with the Solvita test to see both the CO<sub>2</sub> release quantities and categories of storage potential.

**Fig 2**

SOLVITA COLOR #	lbs CO <sub>2</sub> per day per yd <sup>3</sup> ‡	kg CO <sub>2</sub> - C per day per t TS	tons CO <sub>2</sub> per day per 10,000yd <sup>3</sup>	Sequestration value of carbon
1.0	0.71	4.28	3.57	NONE
2.0	0.36	2.14	1.78	slight
3.0	0.21	1.28	1.07	v-low
4.0	0.11	0.64	0.54	low
5.0	0.07	0.43	0.36	m-low
6.0	0.04	0.21	0.18	medium
7.0	0.01	0.09	0.07	m-high
8.0	0.01	0.04	0.04	high

‡ compost bulk density 0.5 g/cc or 850 lb/yd

## SELECTING BEST USE OF COMPOST BASED ON MATURITY

The favorable relationship of compost maturity to plant performance is well known<sup>1</sup>. Table 5 provides general best-use categories in relation to maturity (see note).

**Table 5 COMPOST BEST USE GUIDELINE**

SOLVITA® MATURITY INDEX	Class of these materials similar to:									
	Raw Feestock	Landspreading Mulch	Farm Crops, Field Cultivation	Greenhouses	Pastures, Hay Crops, Turf	Topsoil Substitutes	General Gardening	Container Media	Planting Mixes	
Increase wait time prior to planting <b>7 - 8</b>						✓	✓	✓	✓	Soil & peat-based mixes; growing media
<b>6 - 7</b>				✓	✓	✓	✓	✓		Soil blends, filter berms
<b>4 - 5</b>			✓	✓	✓					dried organic fertilizers, processed material
<b>1 - 3</b>	✓	✓	✓							fresh & dehydrated manures, raw wastes

**Note:** There are other key factors that determine how well a compost performs. A compost can be examined for other traits by a laboratory experienced in compost analysis. See [www.woodsend.com](http://www.woodsend.com) for up-to-date information on compost testing.

1. Literature about Solvita® validation and field testing can be found at [www.solvita.com/publications](http://www.solvita.com/publications)



## AMMONIA EMISSIONS OF MANURES & COMPOSTS

The Solvita® ammonia test can be applied alone to measure the gaseous ammonia present in the air space for any given material. To measure the true maturity of compost the ammonia and CO<sub>2</sub> tests are run together. Ammonia affects respiration directly and indirectly and is also the primary mode of loss of nitrogen from active composts and manure. It is classed as a hazardous odor and may be toxic to macro-organisms such as earthworms and may inhibit microbial respiration.

Table 6 provides guidelines for results of ammonia present in compost and bedding manure or animal litter layers. If a sample persists in high ammonia values (1 - 3) for any period of time, measures should be considered to control it.

**Table 6 Solvita® Ammonia Gas Content, Plant-Toxicity, N-losses**

Ammonia Color No:	1	2	3	4	5
<b>Material Condition</b>	----- Extremely Active -----		Active	Curing	Stable
<b>Potential Plant toxicity:</b>	Very High	High	Medium	Slight	None
<b>Noxious Hazard</b>	Extreme	Severe	Moderate	Slight	None
<b>ppm NH<sub>3</sub> in headspace‡</b>	<b>&gt;25,000</b>	<b>15,000</b>	<b>8,500</b>	<b>3,500</b>	<b>&lt;100</b>
<b>N-loss potential §</b>	V. High	M High	Moderate	Low	None

‡ Concentration of ammonia gas in headspace of Solvita® test-jar. The concentration expected for an enclosed compost system will vary based on the specific ratio of the composting material to the total volume of the container. A web-based tool is available to show conversion of ambient Solvita ammonia to free NH<sub>3</sub> in any building space.

§ Based on amount of NH<sub>3</sub>-N absorbed in 4-hr test in Solvita® jar. The actual losses during composting will depend on aeration frequency, moisture and pH.

### Technical Notes

**Interferences:** CO<sub>2</sub>-Probe. Nitrous oxide and VOC may cause a pinkish tinge or a positive color error (lower apparent stability). Volatile Fatty Acids > 10,000 ppm cause a positive error (lower apparent stability). Gases of NH<sub>3</sub> and CO<sub>2</sub> form unstable ammonium carbonate in presence of water. Therefore volatile ammonia > 3,500ppm (Solvita NH<sub>3</sub> colors 1 - 3) may impede CO<sub>2</sub> color development resulting in a negative error (corrected for in the Maturity Index chart Table 1).  
NH<sub>3</sub> Probe: No known interferences.

**Bagged-Samples:** Cumulative saturation by CO<sub>2</sub> or NH<sub>3</sub> of samples held for any period inside bags or jars may result in a positive error (more apparent CO<sub>2</sub> or NH<sub>3</sub>). This is corrected by allowing samples to air-out for a brief time (1 hour) before testing.

**DCR (Digital Color Reader):** The DCR available to read Solvita gives the same visual color scale as expected but over a wider and more precise range 0.2 - 8.00. The DCR additionally gives quantitative CO<sub>2</sub> and NH<sub>3</sub> data. DCR's require upgrading occasionally as provided by Solvita information.

*Reprints of independent validation studies for Solvita® are available at [www.solvita.com/publications](http://www.solvita.com/publications)*



## APPENDIX - Troubleshooting Guideline

Indicated Problem or Result	Possible Explanation	Possible Remedy
Compost is very fresh but test results indicate “mature”	Compost may be very low in organic matter or mixed with too much soil	Check organic content; check available-N; add fresh ingredients;
	Compost is inhibited by low pH (food scraps); or very dry and hot prior conditions; check ammonia	Check pH and VOA level; correct moisture; test again 1-2 days later
Compost is old but Solvita® results indicate “active” and/or high ammonia levels	Material has composted improperly or is very dense, too wet or too dry, too compacted, poor mix of ingredients, not enough air	Turn pile, loosen material, add moisture or “green” materials if needed; if high in ammonia select for field rather than seedling use
Compost has given the same Solvita® colors on several tests at 1-2 weeks apart	Compost is not progressing properly— it may be too dry or too compacted, not well mixed; C:N or pH is too high or too low	If pile looks woody add green matter; add moisture if too dry; loosen if too dense
Different parts of the pile give different Solvita® colors	Pockets of poorly mixed or poorly aerated material exist	Re-mix entire pile and re-sample and test again
Core is always #1 or #2 on Maturity Scale	Core is anaerobic and/or is not composting properly	Provide coarse structural materials, mix pile or add air; pile may be too large
Solvita indicates “mature” but plants were hurt by compost	Compost may be acidic, contains high levels of salts, or VOA, or has no available nutrients.	Check pH and conductivity; allow more composting; allow curing time in soil before planting
Color doesn’t match the color chart	Package may have leaked air prior to the test or is defective; or sample is high in nitrite	Discard probe and request replacement product; aerate compost, test again
Unexpected CO <sub>2</sub> vs. ammonia probe results	unusual or extreme conditions persist; check probe quality	See table 2 and table 6
DCR color result is different from visual color	high emissions of VOC or nitrous oxide detected by spectrometer but not visible to eye	allow compost to air out; allow compost to mature
Solvita CO <sub>2</sub> differs from a lab CO <sub>2</sub> assay	Solvita test reflects <i>volumetric</i> emissions, not weight-based	convert Solvita readings to weight basis with DCR

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