

|   |   |                              |                                  |                                  |  |   |                           |                          |
|---|---|------------------------------|----------------------------------|----------------------------------|--|---|---------------------------|--------------------------|
| <b>Test Method:</b> Field Sampling of Compost Materials. Five Protocols |   |                              |                                  |                                  |  |   | <b>Units:</b> NA          |                          |
| <b>Test Method Applications</b>   |   |                              |                                  |                                  |  |   |                           |                          |
| <b>Process Management</b>   |   |                              |                                  |                                  |  |   | <b>Product Attributes</b> |                          |
| <i>Step 1:</i><br>Feedstock Recovery                                    | <i>Step 2:</i><br>Feedstock Preparation | <i>Step 3:</i><br>Composting | <i>Step 4:</i><br>Odor Treatment | <i>Step 5:</i><br>Compost Curing | <i>Step 6:</i><br>Compost Screening and Refining | <i>Step 7:</i><br>Compost Storing and Packaging | <i>Safety Standards</i>   | <i>Market Attributes</i> |
| 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.

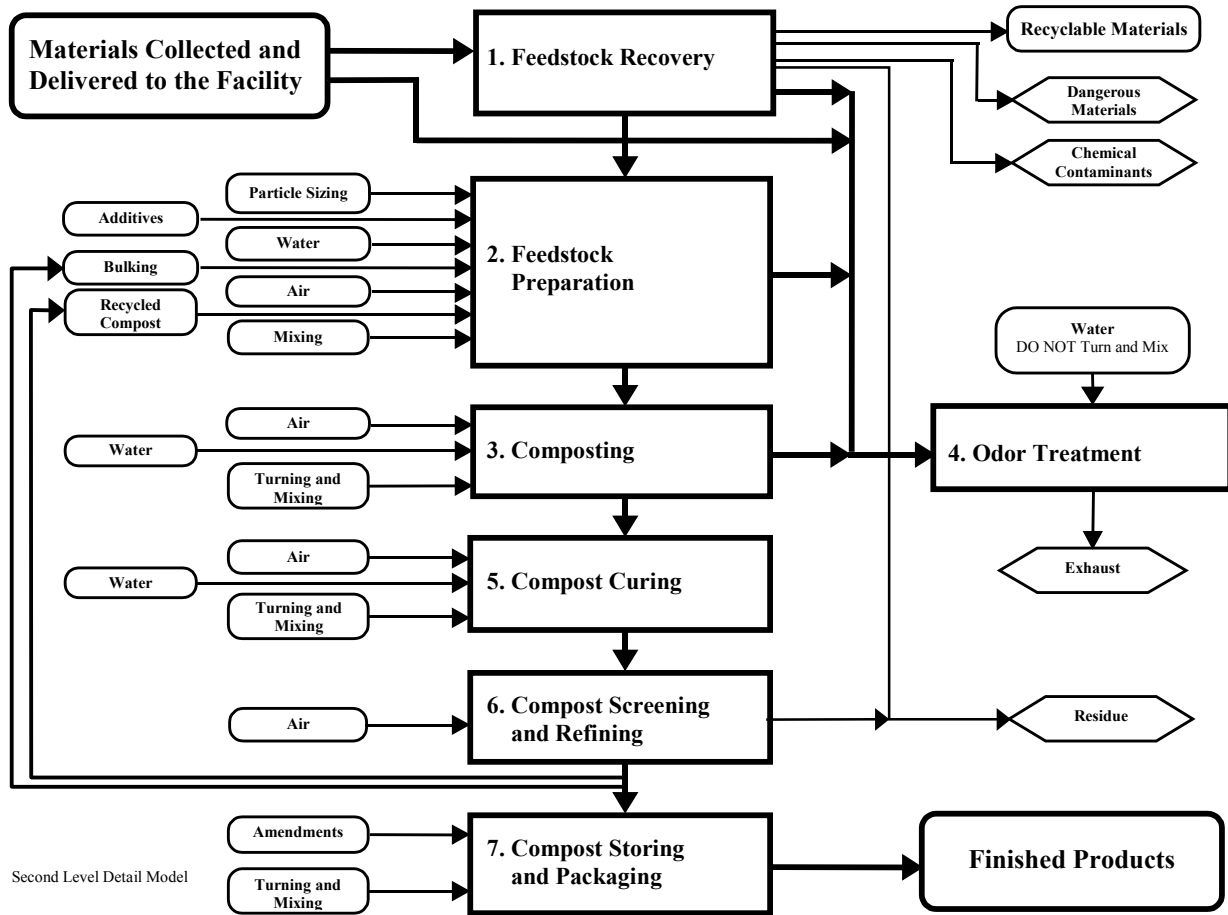


Fig 02.01-1 Composting Unit Operations Model.

## 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**  
*Field Sampling of Compost Materials 02.01*

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<br>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†                     |

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

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

|   |   |                              |                                  |                                  |  |   |                           |                          |
|---|---|------------------------------|----------------------------------|----------------------------------|--|---|---------------------------|--------------------------|
| <b>Test Method:</b> Compost Sampling Principles and Practices |   |                              |                                  |                                  |  |   | <b>Units:</b> NA          |                          |
| <b>Test Method Applications</b>                               |   |                              |                                  |                                  |  |   |                           |                          |
| <b>Process Management</b>                                     |   |                              |                                  |                                  |  |   | <b>Product Attributes</b> |                          |
| <i>Step 1:</i><br>Feedstock Recovery                          | <i>Step 2:</i><br>Feedstock Preparation | <i>Step 3:</i><br>Composting | <i>Step 4:</i><br>Odor Treatment | <i>Step 5:</i><br>Compost Curing | <i>Step 6:</i><br>Compost Screening and Refining | <i>Step 7:</i><br>Compost Storing and Packaging | <i>Safety Standards</i>   | <i>Market Attributes</i> |
| 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.

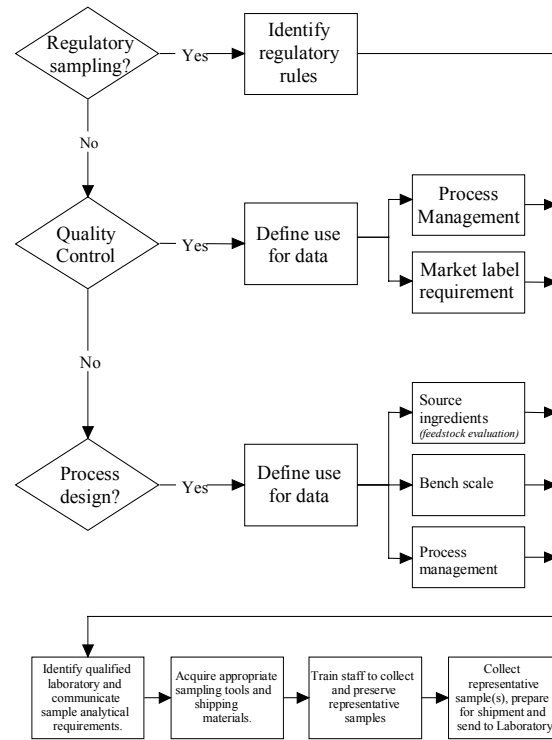


Fig 02.01-A1 Compost sampling decision tree, overview of sampling objectives.

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.

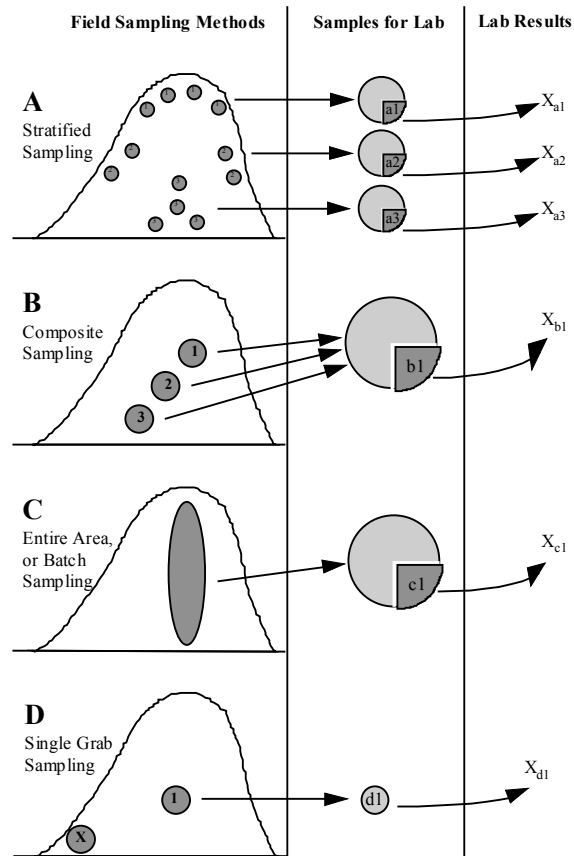


Fig 02.01-A2 The sampling schematic.

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.

| Type                    | Sampling Action  | Constraints   | Preferred Tools   |
|-------------------------|--|---|---|
| A. Home Bins            | Must open bin, remove cover and sides, and mix by hand                                       | Not homogenous, may be hard or impossible to open   | Pail and spading fork                                     |
| B. Turned Windrows      | 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                         |
| C. Static Piles         | 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 |
| D. Agitated-Bed         | 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,                               |
| E. Enclosed Vessel      | Sample from side doors or top port after agitation   | Very difficult or impossible access; potential layering   | 5-gal pail, spading shovel, corer, auger                  |
| F. Rotating Vessels     | 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                               |
| G. Compost Curing Piles | 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   |
| H. Bagged Product       | Sample multiple bags, cores drawn  | Bag damage, difficult access  | 5-gal pail, trowel or soil-corer                          |
| I. Source Ingredients   | Composite from each pile separately, remove surface  | Non-uniformity may be great, poorly mixed, difficult access   | Large pail, shovel; bucket loader                         |
| J. Lab Systems          | 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.

| Amount produced<br>(metric tons of biosolids<br>compost per 365-day period) | Monitoring Frequency for<br>Pathogens and Trace Elements |
|---|--|
| < 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.

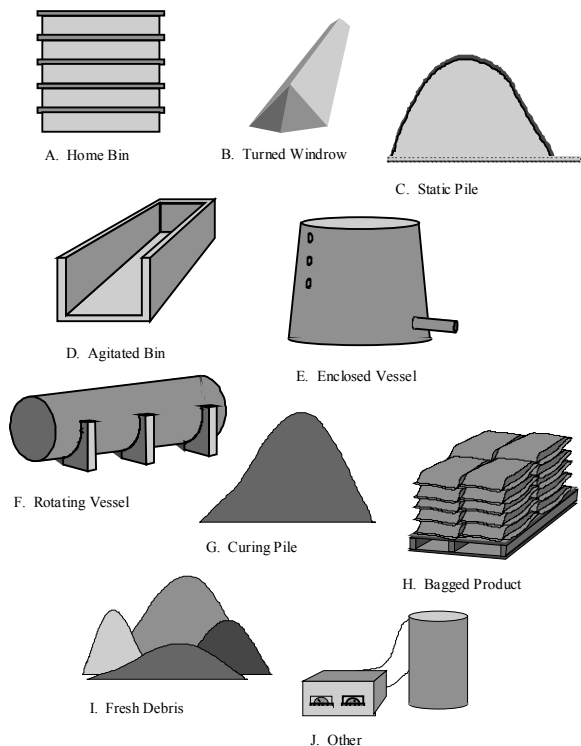


Fig 02.01-A3 Composting technologies.

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><br>Feedstock Recovery                                       | <i>Step 2:</i><br>Feedstock Preparation | <i>Step 3:</i><br>Composting | <i>Step 4:</i><br>Odor Treatment | <i>Step 5:</i><br>Compost Curing | <i>Step 6:</i><br>Compost Screening and Refining | <i>Step 7:</i><br>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

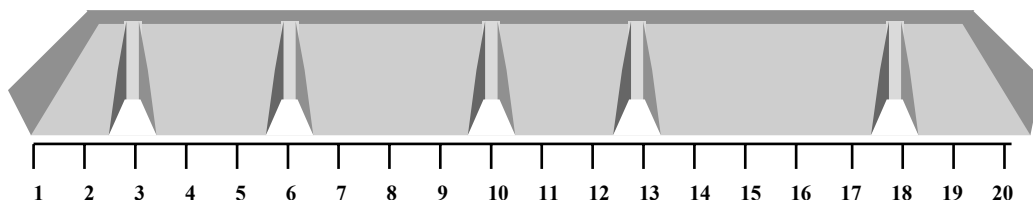


Fig 02.01-B1 Hypothetical sample collection pattern from a compost windrow.

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.

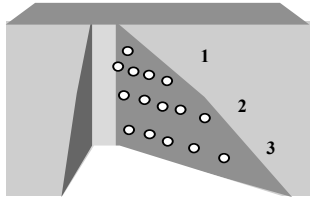


Fig 02.01-B2 Five horizontally dispersed point-samples are collected from each of three depths or zones within each cutout.

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

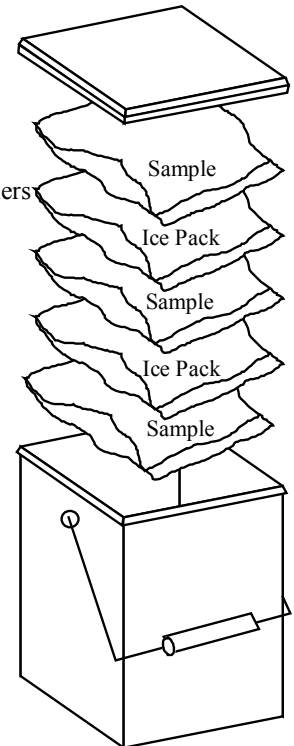


Fig 02.01-B3 Preparation for shipment.

|  |   |                              |                                  |                                  |  |   |                           |                          |
|--|---|------------------------------|----------------------------------|----------------------------------|--|---|---------------------------|--------------------------|
| <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><br>Feedstock Recovery                           | <i>Step 2:</i><br>Feedstock Preparation | <i>Step 3:</i><br>Composting | <i>Step 4:</i><br>Odor Treatment | <i>Step 5:</i><br>Compost Curing | <i>Step 6:</i><br>Compost Screening and Refining | <i>Step 7:</i><br>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.

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

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

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

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

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.

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

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

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

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

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 EP toxicity).

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.

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

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><br>Feedstock Recovery                                  | <i>Step 2:</i><br>Feedstock Preparation | <i>Step 3:</i><br>Composting | <i>Step 4:</i><br>Odor Treatment | <i>Step 5:</i><br>Compost Curing | <i>Step 6:</i><br>Compost Screening and Refining | <i>Step 7:</i><br>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><br>Feedstock Recovery                                    | <i>Step 2:</i><br>Feedstock Preparation | <i>Step 3:</i><br>Composting | <i>Step 4:</i><br>Odor Treatment | <i>Step 5:</i><br>Compost Curing | <i>Step 6:</i><br>Compost Screening and Refining | <i>Step 7:</i><br>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.

|   |  |
|---|--|
| STA Laboratory: _____<br>Address: _____<br>City, State Zip code: _____  | Tel: _____<br>FAX: _____<br>Email: _____ |
| Client/Reporting Company: _____<br>Contact Name: _____<br>Billing Address: _____<br>City, State Zip code: _____ |  |
| Tel: _____<br>FAX: _____<br>Email: _____  |  |
| City, State Zip code: _____<br>Send Results to: _____<br>City, State Zip code: _____                            |  |
| Name or Source of sample(s): _____<br>Name of Sample Collector: _____   |  |

|  |                 |                             |                                  |
|--|-----------------|-----------------------------|----------------------------------|
| <b>LABORATORY USE ONLY</b>   |                 | <b>Storage Locations</b>    |                                  |
| Freezer _____  | Cold Room _____ | Storage Shelf _____         |                                  |
| Sample Condition: _____  |                 |                             |                                  |
| Sample Type: <input type="radio"/> COMPOSITE   |                 | <input type="radio"/> POINT | <input type="radio"/> STRATIFIED |
| P. O. Number: _____  |                 |                             |                                  |
| Client STA ID: _____   |                 |                             |                                  |
| USCC Member: <input type="radio"/> YES <input type="radio"/> NO ID: _____  |                 |                             |                                  |
| <b>SELECTION OF ANALYSIS. Refer to <a href="http://timecc.org/sta">http://timecc.org/sta</a> for details.</b>  |                 |                             |                                  |
| STA Suite: All 503 Rule Tests. Other – Specify additional tests in fields A through D (below).   |                 |                             |                                  |
| <b>NOTE 1</b> Your selection of STA Suite (below) authorizes laboratory personnel to disclose all analytical results AND submit the STA Compost Technical Data Sheet directly to STA program management. |                 |                             |                                  |
| A  | B               | C                           | D                                |

| Client Sample Identification<br>(and special instructions) | Collection Date/Time                          | Sample Matrix  | Sample Container  | Shipping Temp.  | Selected Analysis     |                       |                       |                       | Lab/Job Number |
|--|---|--|---|---|-----------------------|-----------------------|-----------------------|-----------------------|----------------|
|  |   |  |   |   | STA                   | 503                   | A                     | B                     |                |
| 1  | Date: _____<br>Time: _____<br>Initials: _____ | Compost <input type="radio"/><br>Feedstock <input type="radio"/> | Plastic Bag <input type="radio"/><br>Pail <input type="radio"/> | Ambient <input type="radio"/><br>Wet Ice <input type="radio"/><br>Dry Ice <input type="radio"/> | <input type="radio"/> | <input type="radio"/> | <input type="radio"/> | <input type="radio"/> | 1              |
| 2  | Date: _____<br>Time: _____<br>Initials: _____ | Compost <input type="radio"/><br>Feedstock <input type="radio"/> | Plastic Bag <input type="radio"/><br>Pail <input type="radio"/> | Ambient <input type="radio"/><br>Wet Ice <input type="radio"/><br>Dry Ice <input type="radio"/> | <input type="radio"/> | <input type="radio"/> | <input type="radio"/> | <input type="radio"/> | 2              |
| 3  | Date: _____<br>Time: _____<br>Initials: _____ | Compost <input type="radio"/><br>Feedstock <input type="radio"/> | Plastic Bag <input type="radio"/><br>Pail <input type="radio"/> | Ambient <input type="radio"/><br>Wet Ice <input type="radio"/><br>Dry Ice <input type="radio"/> | <input type="radio"/> | <input type="radio"/> | <input type="radio"/> | <input type="radio"/> | 3              |
| 4  | Date: _____<br>Time: _____<br>Initials: _____ | Compost <input type="radio"/><br>Feedstock <input type="radio"/> | Plastic Bag <input type="radio"/><br>Pail <input type="radio"/> | Ambient <input type="radio"/><br>Wet Ice <input type="radio"/><br>Dry Ice <input type="radio"/> | <input type="radio"/> | <input type="radio"/> | <input type="radio"/> | <input type="radio"/> | 4              |
| 5  | Date: _____<br>Time: _____<br>Initials: _____ | Compost <input type="radio"/><br>Feedstock <input type="radio"/> | Plastic Bag <input type="radio"/><br>Pail <input type="radio"/> | Ambient <input type="radio"/><br>Wet Ice <input type="radio"/><br>Dry Ice <input type="radio"/> | <input type="radio"/> | <input type="radio"/> | <input type="radio"/> | <input type="radio"/> | 5              |
| Releasing Signature 1                                      | Date  | Time   | Receiving Signature 1   | Date  | Time                  |                       |                       |                       |                |
| Releasing Signature 2                                      | Date  | Time   | Receiving Signature 2   | Date  | Time                  |                       |                       |                       |                |
| Releasing Signature 3                                      | Date  | Time   | Receiving Signature 3   | Date  | Time                  |                       |                       |                       |                |
| Releasing Signature 4                                      | Date  | Time   | Receiving Signature 4   | Date  | Time                  |                       |                       |                       |                |

Fig 02.01-E1 Chain of Custody. Compost sample chain of custody log

