

**Test Methods
for the Examination of**

**Composting
and
Compost**

Prepared for:

THE US COMPOSTING COUNCIL RESEARCH AND EDUCATION FOUNDATION, AND
THE UNITED STATES DEPARTMENT OF AGRICULTURE

Editor in Chief:

Wayne H. Thompson

Co-Editors:

Philip B. Leege
Patricia D. Millner
Maurice E. Watson

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MANUAL DEVELOPMENT

This manual of test methods has benefited from the expertise and input of numerous individuals. In addition, several groups cooperated and many contributors provided valuable suggestions for improvement. Throughout its development, the focus always remained on producing a technically sound manual of test methods and protocols. The development stages of the TMECC project are described below.

Stage 1. Project Concept Development

The US Composting Council's Standards and Practices Committee and Markets Development Committee developed a list of key process variables to measure and manage, and attributes to report for the following: composting feedstock; the composting process; finished compost, product safety and regulatory compliance, and marketing claims.

Stage 2. Minnesota Compost Utilization Project (MN-CUP)

The Standards and Practices Committee worked cooperatively with the Minnesota Office of Environmental Assistance, the University of Minnesota's Department of Soil, Water, and Climate Research Analytical Laboratory, and Malcolm Pirnie for two years to survey eight municipal solid waste composting facilities in Minnesota. This was an observational study designed to document feedstock, in-process and finished compost sampling and preservation protocols, laboratory preparation steps and analytical methodologies. The documented sampling and analytical methods are presented along with others in this manual in the form and style of ASTM methods.

The MN-CUP study was divided into three phases:

Phase I—Sample Variability. For the first two months, three separate compost samples were collected at each of three locations to document variability within one batch at one site. One of the three samples from each location was subdivided into ten subsamples during laboratory sample preparation to evaluate within-sample variability.

NOTE—A Reference Sample (in-house) of municipal solid waste compost was created with excess material from one of the original three locations. This material was first air-dried at 36°C, sieved through a 4-mm sieve and milled with a Stein mill (carbide-tipped blade). The milled material was oven-dried at 70°C to minimize enzymatic degradation, mixed in a tumble blender for 2 d, split with a sample splitter and stored in 2 L polyethylene bottles at room temperature (~28°C).

Phase II—Temporal Variability. Sampling continued on a monthly basis for one year at the previously mentioned sites. Five additional facilities were added for the duration of the first year's monthly sampling phase.

Phase III—Temporal Variability. Sampling frequency was decreased to a quarterly basis for seven facilities during the second year of sampling. One facility was lost to fire.

Test Parameters from MN-CUP

During the MN-CUP project, approximately 40 parameters were considered. The methods were modified and adapted from existing ASTM, ASA-SSSA, SW-846 and AOAC methods developed for other materials. Test parameters considered:

I. *Chemical Analyses*—using US EPA 3051 digest modified for compost's high organic matter and ICP-AES determinations for metals and salts; cold vapor for Hg; wet combustion determination for N (total Kjeldahl nitrogen, micro-digest technique); colorimetric NO₃ and NH₄; and cation exchange capacity (modified ammonium displacement technique on milled material).

II. *Physical Analyses*—for total solids and moisture (wet basis); ash (volatile solids); man-made inerts (plastics, metal, glass); bulk density; water-holding capacity; and air-capacity.

III. *Biological Analyses*—for stability (oxygen uptake); growth and germination (a direct seeding technique); and organic carbon using dry combustion.

IV. *Pathogens Analysis*—included fecal coliforms (determined at private laboratories outside of the University of MN system).

V. *Organics Analyses*—included volatile fatty acids and polychlorinated biphenyls (PCB's determined at private laboratories outside of the University of MN system).

Stage 3. Draft of Sampling and Analysis Protocols

A scientifically based catalog and laboratory manual of methods was drafted for use with feedstock and compost analysis to initiate the standardization process for regulatory and market requirements, and management of the composting process.

This work included formatting, enhancement and critical review of methods devised and modified at the University of Minnesota's Department of Soil, Water and Climate Research Analytical Laboratory, St. Paul,

Preface

by Robert Munter's group and private laboratories for the MN-CUP project. Other methods developed during parallel compost projects were added to complement the MN-CUP work, funded by the Composting Council Research and Education Foundation and The Procter & Gamble Company are included in the manual.

Goal—To provide a science-based manual of rigorous test methods specifically appropriate to feedstocks and finished compost, as distinct from soil, manure, and fertilizers, and applicable to regulatory and market requirements, and to augment these methods with a suite of *quick tests* for managing the composting process.

Stage 4. Introduction and Peer Review—December 1997

Goal—Introduce TMECC to the composting community and familiarize users with its intended purpose and content; solicit feedback to refine and expand manual content. A review draft of TMECC was provided to approximately 160 laboratories, compost production facilities, and academic institutions. Collaborating groups and individuals were requested to perform the following:

- 4.1 analyses of composts by methods provided in TMECC,
- 4.2 document commentary and critiques of existing methods, and
- 4.3 solicit for and add missing test methods.

Product—Revision of the First Draft of *Test Methods for the Examination of Composting and Compost*.

Stage 5. Collaborative Evaluation, On-Going

Goal—Develop consensus for test definitions:

5.1 provide replicated samples of composts from varying feedstock types and combinations; include a minimum of three laboratories per test to establish method precision with resulting data to be used in precision tables to identify and document sources of bias,

5.2 synthesize user feedback to identify and document consensus among participating laboratories for acceptance of at least one test method for each test parameter where appropriate,

5.3 remove antiquated methods, and

5.4 solicit for and add missing test methods.

Products—*Reference Editions of Test Methods for the Examination of Composting and Compost*.

Stage 6. USDA Greenhouse and Field Testing

Goal—Identify and document correlation among different test method values and calibrate tests to obtain interpretive information about using the compost.

Product—Test interpretation guidelines for compost application management.

ACKNOWLEDGMENTS

EDITOR-IN-CHIEF

Thompson, Wayne H.	Edaphos International Houston, TX
--------------------	--------------------------------------

CO-EDITORS

<i>Name</i>	<i>Affiliation</i>
Leege, Philip B.	The Philip B. Leege Company, Ltd. Cincinnati, OH
Millner, Patricia D.	Soil Microbial Systems Laboratory USDA-ARS-BARC Beltsville, MD
Watson, Maurice E.	Ohio Agri-Research and Development Center, and Ohio Compost and Manure Management Program Ohio State University Wooster, OH

REVIEW COORDINATORS

<i>Coordinators</i>	<i>Affiliation</i>	<i>Peer Review Subject Area</i>
Watson, Maurice E.	Ohio Agri-Research and Development Center, and Ohio Compost and Manure Management Program Ohio State University Wooster, OH	1 – Field Sample Collection
Shields, Frank	Soil Control Lab A Division of Control Laboratories, Inc. Watsonville, CA	2 – Laboratory Sample Preparation
Fitzpatrick, George E.	Fort Lauderdale Research and Education Center University of Florida Gainesville, FL	3 – Physical Parameters
Miller, Robert O.	Department of Soil and Crop Sciences Colorado State University Fort Collins, CO	4 – Analytical, Inorganic Chemistry
Das, Keshav C.	Department of Biological and Agricultural Engineering University of Georgia Athens, GA	5 – Physical Chemistry
Thompson, Wayne H.	Edaphos International Houston, TX	6 – Stability, Maturity and Biological Activity
Cole, Michael A.	Department of Natural Resources and Environmental Sciences University of Illinois Urbana-Champaign, IL	7 – Organic Chemistry
Switzenbaum, Michael S.	Department of Civil and Environmental Engineering University of Massachusetts Amherst, MA	8 – Physical Organic Chemistry
Michel, Frederick C. Jr.	Ohio Agri-Research and Development Center Department of Food, Agricultural and Biological Engineering Ohio State University Wooster, OH	9 – Microbiology and Pathogens Testing

Acknowledgments

PEER REVIEW PARTICIPATION

<i>Reviewer</i>	<i>Affiliation</i>	<i>Test Methods (listed by peer review subject area)</i>
Adler, Paul	USDA-ARS – Kearneysville, WV	3 – 03.01, 03.02, 03.04, 03.07, 03.09, 03.10 5 – 04.10, 04.11
Aiello, Kevin	Middlesex County Utilities Authority – Sayreville, NJ	5 – 04.09, 04.10, 04.11
Anderson, Kim A.	Analytical Sciences Laboratory Department of Food Sciences and Toxicology Holm Research Center, University of Idaho – Moscow, ID	2 – 02.02, 03.06-A, 03.08-A
Asdal, Smund	The Norwegian Crop Research Institute Apelsvoll Research Centre, Division Landvik Apelsvoll – NORWAY	6 – 05.05
Barkman, Joel E.	P.E. Wilbark Consultants, Inc. – Fort St. John, BC CANADA	3 – 03.01
Beffä, Trello	Laboratoire de Microbiologie Universite Neuchatel – Neuchatel, SWITZERLAND	9 – 07.01, 07.02, 07.03
Beharee, Vihita	University of Natal – Natal, SOUTH AFRICA	5 – 04.09, 04.10, 04.11
Bess, Vicki	BBC Laboratories, Inc. – Tempe, AZ	5 – 04.09, 04.10, 04.11 6 – 05.05, 05.08
Bidlingmaier, Werner	Professur Abfallwirtschaft, Fakultat Bauingenieurwesen Bauhaus-Universität, Weimar – GERMANY	2 – 02.02, 03.06-A, 03.08-A 5 – 04.09, 04.10, 04.11
Bloom, Paul R.	Department of Soil, Water, and Climate University of Minnesota – St. Paul, MN	4 – 04.01, 04.07 6 – 05.05, 05.07, 05.08
Bouwkamp, John	Department of Horticulture University of Maryland – College Park, MD	6 – 05.02
Breitenbeck, Gary	Department of Agronomy Louisiana State University – Baton Rouge, LA	1 – 02.01 2 – 02.02, 03.06-A, 03.08-A
Briceno, Jorge A.	Centro de Investigaciones Agronomicas Universidad de Costa Rica – COSTA RICA	3 – 03.01, 03.02, 03.09
Brinton, William F., Jr.	Woods End Research Laboratory – Mount Vernon, ME	2 – 02.01-A, 02.01-B 3 – 03.01, 05.01 6 – 05.02-G, 05.03, 05.06, 05.07-B, 05.08, 05.09 8 – 05.04-B
Buchanan, Marcus	Buchanan Associates – Scotts Valley, CA	6 – 05.02-G
Butler, Tracy	Soil Microbial Systems Laboratory USDA-ARS-BARC – Beltsville, MD	6 – 05.05-A
Chaney, Rufus L.	Environmental Chemistry Laboratory USDA-ASR-BARC – Beltsville, MD	4 – 04.12-E 6 – 05.02-E
Chu, Paul	A&L Eastern Lab – Richmond, VA	4 – 04.01, 04.02, 04.03, 04.04, 04.05, 04.06, 04.07, 04.08, 04.12, 04.13
Cole, Michael	Natural Resources and Environmental Sciences University of Illinois – Urbana-Champaign, IL	2 – 02.02, 03.06-A, 03.08-A
Cook, Bruce	Department of Soil, Water, and Climate University of Minnesota – St. Paul, MN	6 – 05.08-B
Crenshaw, Teresa A.	Delaware Department of Agriculture – Dover, DE	1 – 02.01
Croteau, Gerald A.	E&A Environmental Consultants, Inc. – Bothel, WA	6 – 05.03, 05.06-A
Dancer, William E.	Research Analytical Laboratory Department of Soil, Water, and Climate University of Minnesota – St. Paul, MN	4 – 04.06-Hg, 04.12-A, 04.13, 04.14
Dellavalle, Nat	Dellavalle Laboratory - Fresno, CA	4 – 04.02
Eliason, Roger	Research Analytical Laboratory Department of Soil, Water, and Climate University of Minnesota – St. Paul, MN	3 – 03.01, 05.01 4 – 04.12-A, 04.13, 04.14

PEER REVIEW PARTICIPATION, *continued*

<i>Reviewer</i>	<i>Affiliation</i>	<i>Test Methods (listed by peer review subject area)</i>
Elwell, David	Ohio Agri-Research and Development Center Department of Agricultural Engineering Ohio State University – Wooster, OH	6 – 05.08-A
Epstein, Eliot	E&A Environmental Consulting – Stoughton, MA	6 – 05.02
Ernst, Joann	Environmental Education and Testing Services Inc. Bellingham, WA	5 – 04.10, 04.11 7 – 06.03, 06.04-A 9 – 07.02-A
Evanylo, Greg	Virginia Polytech and State University – Blacksburg, VA	5 – 04.09, 04.10, 04.11
Farrell, Brenda F.	The Keck Center for Computational Biology Department of Biochemistry and Cell Biology Rice University – Houston, TX	3 – 03.04 8 – 05.04, 05.10-A
Flock, Mark A.	Brookside Laboratory, Inc. – New Knoxville, OH	2 – 02.02, 03.06-A, 03.08-A 4 – 04.01, 04.02, 04.03, 04.04, 04.05, 04.06, 04.07, 04.08, 04.12, 04.13
Gouin, Frank R.	Department of Horticulture (Professor Emeritus) University of Maryland – Baltimore, MD	3 – 03.01, 03.02, 03.04, 03.07, 03.09, 03.10, 05.01 6 – 05.05-B, 05.02-B
Grebus, Marcella E.	Department of Plant Pathology University of California – Riverside, CA	6 – 05.05-A, 05.08-A
Guttererres, Fancisco	California Integrated Waste Management Board Sacramento, CA	1 – 02.01 2 – 02.02, 03.06-A, 03.08-A 3 – 03.01, 03.02
Halbach, Tom	Department of Soil, Water, and Climate University of Minnesota – St. Paul, MN	3 – 03.01-C, 03.09, 03.10-C, 03.10-E 6 – 05.05-C, 05.07-A
Hampton, Monica-Ozores	Southwest Florida Research and Education Center University of Florida – Immokalee, FL	5 – 04.10, 04.11
Hanninen, Kari	Department of Biology and Environmental Sciences University of Jyuaskyla – Jyuaskyla, FINLAND	1 – 02.01
Hanson, Dean	Central Analytical Laboratory Oregon State University – Corvallis, OR	2 – 02.02, 03.06-A, 03.08-A
Harrison, Robert B.	College of Forest Resources University of Washington – Seattle, WA	2 – 02.02-D
Hernandez, T.	Centro de Edafología y Biología Aplicada del Segura (CEBAS), dependiente del Consejo Superior de Investigaciones Científicas (C.S.I.C) – Murcia, SPAIN	6 – 05.04
Hoitink, Henricus A.	Ohio Agri-Research and Development Center Department of Plant Pathology Ohio State University – Wooster, OH	6 – 05.08-A
Ingram, David	Soil Microbial Systems Laboratory USDA-ARS-BARC – Beltsville, MD	9 – 07.00, 07.01, 07.02, 07.03
King, Mark	Maine Department of Environmental Protection Augusta, ME	2 – 02.02, 03.06-A, 03.08-A
Kotuby-Amacher, Janice	Utah State University – Logan, UT	4 – 04.01, 04.02, 04.03, 04.04, 04.05, 04.06, 04.07, 04.08, 04.12, 04.13
Li, Yuncong	Department Soil and Water Science Tropical Research and Education Center University of Florida – Gainesville, FL	2 – 02.02, 03.06-A, 03.08-A 4 – 04.09, 04.10, 04.11
Loane, John	California Integrated Waste Management Board Sacramento, CA	2 – 02.02, 03.06-A, 03.08-A
Logan, Tina	Thornton Laboratory – Tampa, FL	2 – 02.02, 03.06-A, 03.08-A
Low, Clifford B.	Perry Labs – Watsonville, CA	2 – 02.02, 03.06-A, 03.08-A
MacLeod, John A.	Agriculture and Agricultural Food Canada Research Center – Charlottetown, PEI CANADA	2 – 02.02, 03.06-A, 03.08-A
Mamo, Martha	Department of Agronomy University of Nebraska – Lincoln, NE	4 – 04.01, 04.02 6 – 05.07

Acknowledgments

PEER REVIEW PARTICIPATION, *continued*

<i>Reviewer</i>	<i>Affiliation</i>	<i>Test Methods (listed by peer review subject area)</i>
McGinley, Charles M.	St. Croix Sensory, Inc. – St. Croix, WI	6 – 05.06
Meckes, Mark	Biohazard Assessment Research Branch National Exposure Research Laboratory U.S. Environmental Protection Agency – Cincinnati, OH	9 – 07.00
Michel, Frederick C. Jr.	Ohio Agri-Research and Development Center Department of Food, Agricultural and Biological Engineering Ohio State University – Wooster, OH	6 – 05.08-A 8 – 05.04, 05.10-A
Miller, Melinda	Whatcom County Health and Human Services, Environmental Health – Bellingham, WA	3 – 03.01-A, 03.01-B
Miller, Robert O.	Department of Soil and Crop Sciences Colorado State University – Fort Collins, CO	1 – 02.01 5 – 04.10, 04.11
Munter, Robert C.	Research Analytical Laboratory (Retired) Department of Soil, Water, and Climate University of Minnesota – St. Paul, MN	3 – 03.02, 03.09, 03.10-D 4 – 04.01, 04.02, 04.03, 04.04, 04.05, 04.06, 04.07, 04.12, 04.13 5 – 04.09, 04.10, 04.11 6 – 05.05-A, 05.08-A
Normandin, Vicki	IAS Labs – Phoenix, AZ	4 – 04.01, 04.02, 04.03, 04.04, 04.05, 04.06, 04.07, 04.08, 04.12, 04.13
Overcash, Michael R.	Department of Chemical Engineering North Carolina State University – Raleigh, NC	7 – 06.01, 06.02, 06.03, 06.04, 06.05, 06.06, 06.07
Parker, Lois	A&L Great Lakes Laboratories, Inc. – Fort Wayne, IN	2 – 02.02-B, 02.02-C 4 – 04.02-D, 04.12 5 – 04.10, 04.11 6 – 05.05-A, 05.07-A, 05.08-B
Pettygrove, Stuart	University of California – Davis, CA	5 – 04.10, 04.11
Reddy, C.A.	Department of Microbiology Michigan State University – East Lansing, MI	9 – 07.00, 07.01, 07.02, 07.03
Schaefer, Frank	Biohazard Assessment Research Branch National Exposure Research Laboratory U.S. Environmental Protection Agency – Cincinnati, OH	9 – 07.04
Schnug, Ewald	Institute of Plant Nutrition and Soil Science Federal Agricultural Research Center Braunschweig – GERMANY	4 – 04.01, 04.02, 04.03, 04.04, 04.05, 04.06, 04.07, 04.12, 04.13 5 – 04.10, 04.11 6 – 05.07
Shields, Frank	Soil Control Lab, A Division of Control Laboratories Inc. Watsonville, CA	4 – 04.01-A, 04.02, 04.03, 04.04, 04.05, 04.08 5 – 04.10, 04.11 6 – 05.02-F, 05.02-G, 05.05, 05.07, 05.09
Shiralipour, Aziz	Center for Biomass Programs Institute of Food and Agricultural Sciences University of Florida – Gainesville, FL	6 – 05.05-B, 05.09 8 – 05.10
Switzenbaum, Michael S.	Department of Civil and Environmental Engineering University of Massachusetts – Amherst, MA	6 – 05.02-G, 05.08 8 – 05.04, 05.10
Szmidt, Robin	The Scottish Agricultural College Aberdeen, Scotland – UNITED KINGDOM	5 – 04.10, 04.11
Torleiv Næss Ugland	The Norwegian Crop Research Institute Apelsvoll Research Centre, Division Landvik Apelsvoll – NORWAY	6 – 05.05
van de Kamp, Maartin	University of Massachusetts – Amherst, MA	2 – 02.02, 03.06-A, 03.08-A, 03.12
West, James	Soil and Plant Laboratory – Santa Clara, CA	2 – 02.02, 03.06-A, 03.08-A
Wolf, Ann M.	Ag Analytical Services Laboratory Pennsylvania State University – University Park, PA	5 – 04.10, 04.11

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ABBREVIATIONS

Oac	-acetate	μm	micrometer(s)
ACS	America Chemical Society	meq	milli equivalent(s)
ASTM	American Society for Testing Materials	mL	milliliter(s)
ASA	American Society of Agronomy	mm	millimeter(s)
Å	Ångström units (10 ⁻⁸ cm, or 0.1 nm)	mMhos	milliMhos, equal to mS
AshW	ash weight determined at 550°C	ms	millisecond(s)
AOAC	Association of Official Analytical Chemists	min	minute(s), time
Atm	Atmosphere(s), 1 atm = 101,325 Pa	M	mega
AA	atomic absorption	MSW	mixed municipal solid waste
cm	centimeter(s)	MMW	mixed municipal waste
cmol	centi mol(s), cmol kg ⁻¹ ≡ meq 100 g ⁻¹	M	molar
CCFREF	Composting Council Research and Education Foundation	MW	molecular weight
C	coulomb	MSW	municipal solid waste
d	day(s), time	ng	nanogram(s)
Δ	delta, change, or difference	nm	nanometer(s)
°C	degrees Celsius	USCC	US Composting Council, The
°F	degrees Fahrenheit °C = 5 ÷ 9 × (°F - 32)	NIH	National Institute of Health, US
dw	dry weight basis, equal to TS basis	N	normal concentration
÷	divided by, division symbol	No.	number, #
dS	deci-Seiman, equal to dMhos	OM	organic matter
EC	Enzyme Commission	Ω	ohm, unit of resistance (1 Mhos ⁻¹)
=	equal to	oz	ounce(s) US fluid (0.02957 L)
≡	equivalent to	o.d.	outer diameter (dimension)
EtOH	ethanol	dw	oven-dry weight basis determined at 70±5°C
Fig	figure, illustration, chart, drawing, diagram	ODW	oven-dry weight basis determined at 70±5°C
ft	foot (feet) (30.480061 cm)	Pa	pascal(s)
e.g.	for example	ppb	parts per billion (1 × 10 ⁹), e.g., μg kg ⁻¹
gal	gallon(s), US liquid (3.7853 L)	ppm	parts per million (1 × 10 ⁶), e.g., mg kg ⁻¹
GC	gas chromatography	%	percent (parts per 100); percentage
g	gram(s)	pt	pint(s), US liquid (0.4732 L)
g	gravitational force, cm sec ⁻² , ft sec ⁻²	TD	pipette volume to deliver
>	greater than, more than, exceeds	lb	pound(s) (453.6 g)
h	hour(s), time	psi	pounds per square inch (0.06805 atm)
in.	inch(es) (2.54 cm)	PRS	process to reduce sharps
ICP-	inductively coupled plasma - atomic emission	qt	quart(s), US liquid (0.9463 L)
AES/M	spectroscopy/mass spectroscopy	s	second(s), time
i.d.	inner diameter (dimension)	S	Seiman, equal to Mhos
ISO	International Organization for Standardization	rpm	revolutions per minute
kg	kilogram(s)	SSSA	Soil Science Society of America
<	less than, under, below	STP	standard temperature (25°C) and pressure (101,325 Pa)
L	liter(s), liquid	t	time
mhos	unit of conductance (Sieman's unit, Ω ⁻¹)	×	times, multiplication symbol
MS	mass spectrometry	USDA	United States Department of Agriculture
MΩ	megohm(s)	US EPA	United States Environmental Protection Agency
m	meter(s)	W	watts
μg	microgram(s)	yd	yard(s) (0.9144 m)
μL	microliter(s)	SM	Standard Methods for the Examination of Water and Wastewaters

MANUAL FORMAT

1. Test Method Categories

1.1 The test methods presented in TMECC are separated into seven [7] chapters categorized by sample collection and preservation (02.00), physical attribute tests (03.00), chemical analysis (04.00), and organic and biological determinations (05.00), with references to pertinent synthetic organic chemicals determination methods (06.00) and pathogen testing procedures (07.00). Each test method is designed for analyzing compost materials at one or more of the six [6] composting process steps described in chapter 01.00, and to document compost safety standards or market attributes.

2. Test Method Coding System

2.1 *Alpha-Numeric Test Method Codes*—Each test method code contains two integers and one hyphenated letter. The first integer identifies the chapter and the second integer represents the test parameter, while the hyphenated letter represents one of various possible test methods that may be used for the measurement, or determination of a test parameter.

EXAMPLE 1—the code “05.08-D” represents test method “D” of test parameter eight [8] in chapter five [05].

2.2 *Referenced Methods*—Test methods of interest may not be included in TMECC because:

2.2.1 the method is proprietary;

2.2.2 methods are well documented in other manuals; or

2.2.3 the method has not yet been adequately optimized for use with composting materials.

2.3 *Page Numbering*—Page numbers are located on the outside lower corner of each page. The page number is preceded by the hyphenated chapter number and section number.

EXAMPLE—“02.01-8” represents page eight of section one [1] in chapter two [2].

2.4 *Figures and Tables*—The alpha-numeric code for test methods is expanded to include an additional number following the hyphenated letter. Both figures and tables are numbered from one for each test method.

The first number indicates the chapter, the second number indicates the test parameter, and the letter corresponds with the test method, while the last number indicates the figure or table within a method.

EXAMPLE—Fig 04.02-A1 Conceptual example of a standard addition plot.

EXAMPLE—Table 04.04-A1 General interpretation guidelines for greenhouse growth media analyzed by the Saturated Media Extract method (dS m⁻¹).

3. Test Method Page Format

3.1 A test method applications guide is provided as the header for each test method to indicate which methods are appropriate for each of the six composting process steps. Test methods are represented by alpha-numeric code by column under each process step.

3.2 Each test parameter is presented in three parts:

3.2.1 parameter introduction and background;

3.2.2 procedural outlines where more than one procedure may be presented for a parameter; and

3.2.3 method summaries.

3.3 An abbreviated test method application guide for each test method is provided on the first page of each method.

4. Method Guide Format

4.1 The application guide headings provide the following test method information (Fig 00.01-1).

4.1.1 *Test Parameter*—product attribute, such as pH, total solids, etc.

4.1.2 *Test Method*—analytical procedure or quick test for measuring the parameter.

4.1.3 *Reporting Units*—reporting units and moisture basis, such as mg kg⁻¹ dw, g g⁻¹ % wet basis, g cm⁻³ dw, etc. Refer to the list of abbreviations presented in this preface for a description of each abbreviation used in this manual.

4.1.4 *Test Method Applications*—Test method codes are inserted where analysis is appropriate for the indicated process management steps, or safety and market attributes (detailed in chapter one).

Test Method: <i>Parameter (see 4.1.1). Test method (see 4.1.2)</i>							Units: <i>(see 4.1.3)</i>	
Test Method Applications								
Process Management						Product Attributes		
<i>Step 1:</i> Feedstock Recovery	<i>Step 2:</i> Feedstock Preparation	<i>Step 3:</i> Composting	<i>Step 4:</i> Odor Treatment	<i>Step 5:</i> Compost Curing	<i>Step 6:</i> Compost Screening and Refining	<i>Step 7:</i> Compost Storing and Packaging	<i>Safety Standards</i>	<i>Market Attributes</i>
<i>(see 4.1.4)</i>								

Fig 00.01-1 Test method applications guide.

SAMPLE FATE CHART

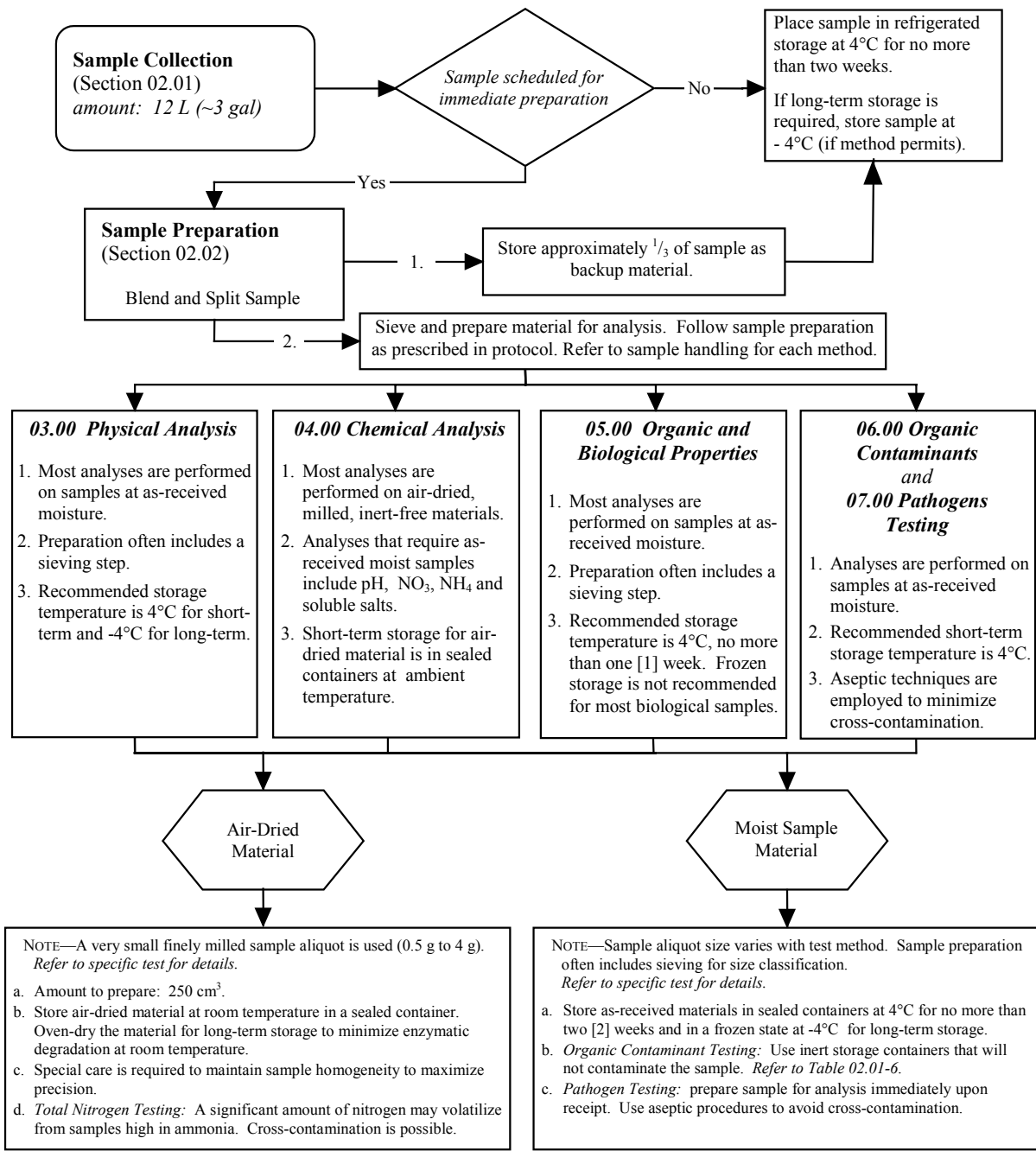


Fig 00.01-2 Fate chart of sample flow from collection through laboratory preparation and analysis.

