

# **NCDA&CS**

## **Waste and Compost**

### **Analytical Methods**



North Carolina Department of Agriculture & Consumer Services

Agronomic Division

Waste and Compost Analysis Laboratory

Prepared by:

Kristin Hicks

Kyle Crump

Lindsay Dietrich

## Waste and Compost Analysis

Waste and compost analysis is used to test total nutrient concentrations (organic + inorganic) and other parameters relevant to nutrient management in animal manure, compost, compost feedstocks, and industrial by-products and to provide guidance to clients for the use of these materials in land application for agricultural production. The NCDA&CS Agronomic Division does not perform any testing for microbial agents (e.g., pathogens), organic contaminants (e.g., pesticides, herbicides, petroleum products) or concentrated inorganic fertilizers.

The standard waste analysis includes measurement of: nitrogen (N), phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg), sulfur (S), iron (Fe), manganese (Mn), zinc (Zn), copper (Cu), boron (B), sodium (Na), and aluminum (Al). For solid and semi-solid samples, N is reported as total N in mg kg<sup>-1</sup> and in lb ton<sup>-1</sup>. For liquid samples, N is reported as TKN (organic N + NH<sub>4</sub>-N) in mg L<sup>-1</sup> and in lb 1000 gal<sup>-1</sup>.

For some waste types (e.g., compost), measurements of total carbon (C), pH, and electrical conductivity (EC) are also provided at no charge with the standard analysis. Clients can request pH, EC or C on any suitable sample at no additional charge. *Note that pH and EC analysis is not suitable for very coarse waste materials such as compost feedstocks (e.g., wood chips, straw, etc.) and C is not suitable for liquid samples ≤ 10% solids.*

Standard waste analysis for <b>N.C. residents</b>	\$8.00
Standard waste analysis for <b>non-N.C. residents</b>	\$25.00
‡Standard waste analysis for <b>N.C. researchers</b>	\$12.00
‡Standard waste analysis for <b>non-N.C. researchers</b>	\$25.00

‡ A completed NCDA&CS Research Project Agreement is **required prior to submission** of research samples. *Please contact Dr. Kristin Hicks at [Kristin.Hicks@ncagr.gov](mailto:Kristin.Hicks@ncagr.gov) to set up a Research Project Agreement. The NCDA&CS Cooperative Research Agreement can be found here: [http://www.ncagr.gov/agronomi/documents/Research\\_Project\\_Agreement\\_PWSM.pdf](http://www.ncagr.gov/agronomi/documents/Research_Project_Agreement_PWSM.pdf)*

In addition to the standard analysis, certain tests are available by request for an additional fee per sample.

### Additional tests available by request:

- Inorganic nitrogen (NO<sub>3</sub>-N, NH<sub>4</sub>-N) + \$10
- Heavy Metals: cadmium, nickel, lead, arsenic, chromium, selenium + \$20
- Calcium carbonate equivalent (CCE) + \$10
- Molybdenum (Mo) + \$2

See the NCDA&CS Waste and Compost Analysis Guide for detailed information:  
<http://www.ncagr.gov/agronomi/pdffiles/wasteguide.pdf>

Sampling instructions can be found here:  
[http://www.ncagr.gov/agronomi/pdffiles/Waste\\_Sampling\\_Quick\\_Guide.pdf](http://www.ncagr.gov/agronomi/pdffiles/Waste_Sampling_Quick_Guide.pdf)

The sample submission form for growers can be found here:  
<https://www.ncagr.gov/agronomi/pdffiles/WasteSampleForm2019Fillable.pdf>

The sample submission form for researchers can be found here:  
[http://www.ncagr.gov/agronomi/pdffiles/Waste\\_Sample\\_Submission\\_Form\\_Researchers.pdf](http://www.ncagr.gov/agronomi/pdffiles/Waste_Sample_Submission_Form_Researchers.pdf)

## Minimum Sample Masses and Volumes

To obtain a representative sample, NCDA&CS strongly recommends a sample volume of 12-16 oz (~500 mL) for liquids and 1 pint (~500 cm<sup>3</sup>) for solids and semi-solids. Where this is not possible, please note the minimum sample volume required to perform each analysis (Tables 1 and 2).

Table 1. Solid and semi-solid (> 4% solids) waste methods summary with minimum mass or volume to perform the analysis. Minimum masses are on a dry-weight basis for ICP-OES and CCE.

Solid and Semi-Solid Samples Method Summary			
Sample Test	Minimum Amount	Analytical Method	Reference
Total N and Total C	200 mg	Oxygen combustion (Dumas method)	AOAC 972.43 AOAC 990.03
P, K, Ca, Mg, S, Fe, Mn, Zn, Cu, B, Na, Al, Mo, As, Cd, Cr, Ni, Pb, Se	0.25 g	Acid digestion; ICP-OES	EPA 200.7
Mo, As, Cd, Cr, Ni, Pb, Se	0.5 g	Acid digestion; ICP-OES	EPA 200.7
NO <sub>3</sub> -N, NH <sub>4</sub> -N	1.0 g	KCl extraction; Continuous Flow Analysis	EPA 353.1 EPA 350.1
pH	10 cm <sup>3</sup>	As received; pH meter	EPA 9045D
EC/SS	10 cm <sup>3</sup>	As received; EC meter	EPA 9050A; TMECC 4.10-A
CCE	1.0 g	Potentiometric titration	AOAC 955.01

Table 2. Liquid (< 4%) waste methods summary with minimum volume to perform the analysis.

<b>Liquid Samples Method Summary</b>			
<b>Sample Test</b>	<b>Minimum Volume</b>	<b>Analytical Method</b>	<b>Reference</b>
Total Kjeldahl Nitrogen (TKN)	5 mL	Acid digestion; Continuous Flow Analysis	EPA 351.2
P, K, Ca, Mg, S, Fe, Mn, Zn, Cu, B, Na, Al, Mo, As, Cd, Cr, Ni, Pb, Se	5 mL	Acid digestion; ICP-OES	EPA 200.7
NO <sub>3</sub> -N, NH <sub>4</sub> -N	15 mL	Filtered; Continuous Flow Analysis	EPA 353.1 EPA 350.1
pH	10 mL	As received; pH meter	EPA 9040C
EC/SS		As received; EC meter	EPA 120.1
CCE	10 mL	Potentiometric titration	AOAC 955.01

## **Analytical Methods**

### **Sample Processing & Storage**

Liquid waste samples are refrigerated at 4 °C upon receipt and homogenized immediately prior to analysis by manual shaking or with a roto-stator homogenizer. All samples are analyzed on an as-received basis and are reported in mg L<sup>-1</sup> and lb 1000 gal<sup>-1</sup> for all elements.

Solid and semi-solid samples are refrigerated at 4 °C upon receipt and homogenized prior to subsampling using a blade homogenizer (e.g., commercial food processor). For total N, total C, inorganic N (NO<sub>3</sub>-N, NH<sub>4</sub>-N), pH and EC analyses on manure and compost, a 50-100 cm<sup>3</sup> subsample is collected from the homogenized primary sample and stored at 4 °C until analyzed. For industrial wastes, a 250 cm<sup>3</sup> subsample is weighed (Mettler Toledo ME4002TE; Mettler-Toledo, LLC; Columbus, OH) and dried for 24-48 hr at 80 °C in a forced air dryer (Horizontal Flow Oven; VWR International LLC, Radnor, PA).

For ICP-OES and CCE analysis, a 250 cm<sup>3</sup> subsample is weighed (Mettler Toledo ME4002TE; Mettler-Toledo, LLC; Columbus, OH) and dried for 24-48 hr at 80 °C in a forced air dryer (Horizontal Flow Oven; VWR International LLC, Radnor, PA). The dried sample is weighed again and ground with a stainless-steel grinder (SM 300 cutting mill; Retsch, Haan, Germany) to pass through a 20-mesh (1 mm) screen (adapted from Hoskins et al. 2003). Small volume (< 100 cm<sup>3</sup>) samples are ground on a tube mill without a screen (IKA Tube Mill 100; IKA Works, Inc.; Wilmington, NC). Ash and powdered lime samples do not typically require grinding but may be sieved through a 1 mm #18 USA Standard Testing Sieve, as needed.

Because client results are reported on an as-received basis, ICP and CCE analysis on dried material require a correction for the moisture lost during drying. *Please note that this test is not the same as Total Solids (TS) which is not an available test at the NCDA&CS Waste Lab.* Percent solids, or dry matter percent is reported on the client report and is calculated as:

$$DM, \% = \left( \frac{DW}{AW} \right) \times 100$$

Where:

DM = dry matter

DW = oven-dry weight

AW = as-received weight

### **Total nitrogen (N) and carbon (C)**

Total N (organic and inorganic) concentration is measured on all solid and semi-solid waste samples. Total C concentration is measured on solid and semi-solid composts, non-composted raw materials (except lime), combustion/thermal by-products (e.g. ash), and solid waste treatment by-products (e.g. biosolids).

Where ammonia loss is a concern, such as manures, waste samples are analyzed as-received for total N and C. A 200-750 mg aliquot of the homogenized sample is introduced into a high temperature (> 900 °C) furnace where the material is combusted in the presence of O<sub>2</sub> based on the Dumas method (1831) (Vario MAX Cube; Elementar Americas, Inc.; Ronkonkoma, NY). N and C results are measured by the instrument as % and reported in mg kg<sup>-1</sup> on an as-received basis as follows:

$$N \text{ or } C, \text{ mg/kg} = N \text{ or } C \text{ results, \%} \times 10,000$$

While most samples are analyzed as-received, paper fibers, ash materials, and very coarse samples such as crop residues, are not suitable for this processing method and are dried and ground instead. Dried samples are analyzed for Total N (ThermoScientific FlashSmart N/Protein; CE Elantech Instruments; Lakewood, NJ) or Total N and Total C (ThermoFinnigan Flash EA1112; CE Elantech Instruments; Lakewood, NJ) by combustion in an oxygen (O<sub>2</sub>)-containing environment with subsequent quantification by thermal conductivity detector (Hoskins et al. 2003; AOAC 1990b; AOAC 2005). N and C results are measured as % and reported in mg kg<sup>-1</sup> on a dry-weight basis as follows:

$$N \text{ or } C, \text{ mg/kg} = N \text{ or } C \text{ results, \%} \times 10,000$$

In addition to reporting N in mg kg<sup>-1</sup> on either an as-received basis or dry-weight basis, N is also converted to and reported as lb ton<sup>-1</sup> based on the Estimate of Available Nutrients. The coefficient used to determine the availability is specific to the waste code reported by the client and careful consideration should be used in selecting the appropriate code to report. For samples analyzed as-received, the Estimate of Available Nutrients is as follows:

$$\text{Available N, } \frac{\text{lb}}{\text{ton}} = \text{N, ppm} \times 0.002 \times \text{NAC}$$

Where:

NAC = Nitrogen Availability Coefficient

For samples analyzed on a dry-weight basis, the Estimate of Available Nutrients is as follows:

$$\text{Available N, } \frac{\text{lb}}{\text{ton}} = \text{N, ppm} \times 0.002 \times \text{NAC} \times \frac{\text{DM}\%}{100}$$

Where:

NAC = Nitrogen Availability Coefficient

DM% = Dry matter %, a moisture correction factor

Carbon:Nitrogen (C:N) is calculated from this analysis and C:N ratio is reported in whole numbers as C mg kg<sup>-1</sup> / N mg kg<sup>-1</sup>. The C:N ratio is an important predictor of N mineralization in manures and compost stability and is used to determine optimum mixing rates of compost feed stocks. High C:N ratios slow the composting and mineralization processes because microbial decomposition of carbohydrates is limited by metabolic nitrogen requirements. When C:N ratio is low (< 15:1), oxygen is rapidly depleted and the aerobic carbohydrate metabolism necessary for the production of quality compost shifts to anaerobic metabolic pathways.

#### *Nitrogen and Carbon Quality Control:*

Method detection limits (MDL) are determined when a new instrument or method is put into use and verified annually. **The MDL for N on dried lime by-products is 0.056%. The MDLs for N/C on dried industrial wastes are 0.073% N and 0.387% C. The MDLs for N and C on as-received samples are 0.014% N and 0.097% C.**

As-received samples are quantified using a factory calibration, which is checked each day with the analysis and calculation of a daily factor. A method blank, duplicate, and matrix spike are analyzed with each batch. Dried samples are quantified using five (N) or six (C) calibration standards. For both dried and as-received samples, four internal and external reference materials are also analyzed at the start of the batch. An internal reference material is analyzed every 12 samples and at the end of the batch for continuing calibration verification.

### **Total Kjeldahl Nitrogen (TKN)**

Nitrogen is quantified in liquid waste samples as total Kjeldahl nitrogen. TKN is defined as the sum of organic nitrogen, which is converted to NH<sub>4</sub>-N during digestion, and free NH<sub>4</sub>-N, both of which are then quantified as NH<sub>4</sub>-N by continuous flow analysis. While the TKN method does not measure nitrate or nitrite, these nitrogen forms are assumed to be relatively negligible in liquid manure, and the TKN method is assumed to be equivalent to the total nitrogen method.

Briefly, a 10 mL aliquot of homogenized sample is digested in 10 mL concentrated sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) on a 350 °C digestion block for 1-2 hours. After cooling, the digest is brought to 100 mL with deionized water and then quantified using an auto-flow spectrophotometric analyzer (San<sup>++</sup> Segmented Flow Auto-Analyzer; Skalar Instruments; Breda, The Netherlands) (Skalar Analytical 1998; USEPA 1993b). Results are expressed in mg L<sup>-1</sup> and in lb 1000 gal<sup>-1</sup>.

#### *TKN Quality Control*

Method detection limits (MDL) are determined when a new instrument or method is put into use and verified annually. **The MDL for TKN is 19.7 mg L<sup>-1</sup>.**

Samples are quantified using a six-point calibration. A method blank, reference material (L-Lysine Monohydrochloride), and matrix spike sample are analyzed with each batch. A duplicate sample from the same day is analyzed with each batch and every 25 samples. A duplicate from the previous day and from the previous week are analyzed with each batch. A calibration verification solution and calibration blank are analyzed at the beginning and end of each batch and after every 10 samples. Two independent calibration verification solutions are analyzed at the beginning and end of each run. Drift checks are analyzed at the beginning and end of each run and every 20 samples.

#### **Inorganic nitrogen: NO<sub>3</sub>-N and NH<sub>4</sub>-N**

For liquid samples, nitrate-nitrogen (NO<sub>3</sub>-N) and ammonium-nitrogen (NH<sub>4</sub>-N) are determined on a 10 mL sample which is shaken and then filtered through a pre-folded Whatman #2 filter paper (Texas Scientific Products, Argyle, TX). For solid and semi-solid samples, NO<sub>3</sub>-N and NH<sub>3</sub>-N are extracted from the waste sample with a 1N KCl plus 2% acetic acid solution. Briefly, 20 mL KCl are added to a 2.0 g sample of raw homogenized material, the mixture is agitated on a reciprocating shaker (Wrist Action Model 75; Burrell Corp. Pittsburgh, PA) for 30 minutes and then filtered through a 110 mm filter.

NO<sub>3</sub>-N is determined on the filtrate or extractant by the hydrazine reduction method, where nitrate is reduced to nitrite with hydrazinium sulfate catalyzed by Cu<sup>2+</sup>, under alkaline conditions and at elevated temperature (Kempers and Luft 1988). The NO<sub>2</sub>-N concentration (that originally present plus reduced nitrate) is determined by diazotizing with sulfanilamide and coupling with α-naphthyl-ethylenediamine dihydrochloride to form a highly-colored azo dye which is measured at 540 nm (modified Griess reaction) (USEPA 1978b; Kempers and Luft 1988; Skalar Analytical 2018c). NH<sub>4</sub>-N is determined on the filtrate or extractant based on a modified Berthelot reaction where, after oxidation and oxidative coupling, a green-colored complex is formed, which is then measured at 660 nm (Krom 1980; Skalar Analytical 2018a; USEPA 1993).

Both NO<sub>3</sub>-N and NH<sub>4</sub>-N are quantified by continuous flow analysis using an auto-flow spectrophotometric analyzer (San<sup>++</sup> Segmented Flow Auto-Analyzer, Skalar Instruments; Breda, The Netherlands). Nitrate-nitrogen (NO<sub>3</sub>-N) and nitrite-nitrogen (NO<sub>2</sub>-N) are reported as NO<sub>3</sub>-N on the Waste Analysis Report. Ammonium-nitrogen (NH<sub>3</sub>-N + NH<sub>4</sub>-N) is reported as NH<sub>4</sub>-N on the Waste Analysis Report. Results are expressed in mg L<sup>-1</sup>.

### *Inorganic Nitrogen Quality Controls*

Method detection limits (MDL) are determined when a new instrument or method is put into use and verified annually. **In liquid samples, the MDLs for NH<sub>4</sub>-N and NO<sub>3</sub>-N are 0.87 mg L<sup>-1</sup> and 0.45 mg L<sup>-1</sup>, respectively. In solid and semi-solid samples, the MDLs for NH<sub>4</sub>-N and NO<sub>3</sub>-N are 13.02 mg kg<sup>-1</sup> and 9.10 mg kg<sup>-1</sup>, respectively.**

Samples are quantified using nine calibration standards. A method blank and duplicate spike sample are analyzed with each batch. A duplicate sample is analyzed with every KCl extraction batch. A calibration verification solution and calibration blank are analyzed at the beginning and end of each batch and after every 10 samples. Four independent calibration verification solutions are analyzed at the beginning and end of each run. Drift checks are analyzed at the beginning and end of each run and every 20 samples. Two nitrite checks (NO<sub>2</sub>-N) are analyzed to verify the completeness of the nitrate reduction reaction at the beginning and end of each run.

### **Phosphorus (P), potassium (K), calcium (Ca), sulfur (S), magnesium (Mg), boron (B), copper (Cu), iron (Fe), manganese (Mn), zinc (Zn), sodium (Na), aluminum (Al), nickel (Ni), cadmium (Cd), lead (Pb), arsenic (As), chromium (Cr), selenium (Se), and molybdenum (Mo)**

Total concentrations of P, K, Ca, Mg, S, Fe, Mn, Zn, Cu, B, Na, Al, Ni, Cd, Pb, As, Cr, Se, and Mo are determined with Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) (Spectro Arcos EOP and Arcos II EOP, Spectro Analytical: A Division of Ametek; Mahwah, NJ) (Donohue and Aho 1992; adapted USEPA 2001), after closed-vessel nitric acid (HNO<sub>3</sub>) digestion in a microwave digestion system (MARS 6 Microwaves; CEM Corp.; Matthews, NC) (Wolf N. 2003c).

For most analytes, a 5.0 mL aliquot of liquid waste or a 0.5 g aliquot of dried/ground solid or semi-solid waste sample is digested in 10 mL 15.6N HNO<sub>3</sub>. When heavy metals (Ni, Cd, Pb, As, Cr, Se) or Mo are requested on a solid or semi-solid, a 1.0 g aliquot of dried/ground sample is digested in 15 mL or 10 mL HNO<sub>3</sub> respectively. Liquid waste is digested for 20 minutes at 180 °C and solid waste is digested for 30 minutes at 200 °C in a microwave (modified EPA 3052 or CEM Plant Materials method). The digested sample volume is brought to 50 mL with deionized water and then filtered through pre-folded Whatman #2 filter paper (Texas Scientific Products, Argyle, TX). Elements are measured at the wavelengths listed in Table 3.

Table 3. Wavelengths used to quantify total elemental concentrations in waste materials by ICP-OES.

Element	Wavelength (nm)
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Aluminum (Al)	396.152
Arsenic (As)	189.042
Boron (B)	208.959
Cadmium (Cd)	214.438
Calcium (Ca)	183.801, 315.887, 318.128
Chromium (Cr)	267.716, 357.869
Copper (Cu)	324.754
Iron (Fe)	259.941
Lead (Pb)	220.353, 405.778
Magnesium (Mg)	279.079
Manganese (Mn)	257.611
Molybdenum (Mo)	202.095
Nickel (Ni)	341.476
Phosphorus (P)	178.287
Potassium (K)	404.721, 766.491
Selenium (Se)	196.090
Sodium (Na)	330.237, 589.592
Sulfur (S)	182.034
Zinc (Zn)	213.856

Liquid waste results are reported on an as-received basis in mg L<sup>-1</sup> and in lb 1000 gal<sup>-1</sup>.

Solid and semi-solid results are reported in mg kg<sup>-1</sup> on a dry-weight basis and in lb ton<sup>-1</sup> on an as-received basis as follows. Dry-weight concentrations are back-calculated to an as-received basis for all elements except P and K, as follows:

$$E, \frac{lb}{ton} = E, mg/kg \times 0.002 \times \frac{DM\%}{100}$$

Where:

E = Any element except P or K

DM% = Dry matter %, a moisture correction factor

P and K in solid and semi-solid samples are reported as P and K on a dry-weight basis in mg kg<sup>-1</sup>.

P and K are reported as P<sub>2</sub>O<sub>5</sub> and K<sub>2</sub>O on an as-received basis in lb ton<sup>-1</sup> as follows:

$$E3, \frac{lb}{ton} = E2, mg/kg \times 0.002 \times NM \times \frac{DM\%}{100}$$

Where:

E2 = P or K

E3 = P<sub>2</sub>O<sub>5</sub> or K<sub>2</sub>O

NM = conversion factor for P (2.29) to P<sub>2</sub>O<sub>5</sub> or K (1.20) to K<sub>2</sub>O  
 DM% = Dry matter %, a moisture correction factor

*ICP-OES Quality Controls*

Elements are measured using a curve with at least five calibration points. For liquid waste samples, a method blank, matrix spike, duplicate from the previous day, and duplicate from the previous week are digested and analyzed with each batch. A duplicate sample from the same day is also digested and analyzed with each batch and every 25 samples. For solid waste samples, a method blank and internal reference sample are digested and analyzed with each batch. A second internal reference sample is digested and analyzed once per day. For heavy metals on solid samples, a method blank, matrix spike, external reference, and interference blank are digested and analyzed with each batch.

A calibration verification solution and calibration blank are run after the daily calibration, after every 10 samples and at the end of each run. An independent calibration verification solution is analyzed at the beginning and end of each run. The method detection limits (MDLs) for each analyte are listed in Table 4.

Table 4. Method detection limits (MDL) of total elemental concentrations in liquid and solid/semi-solid waste by ICP-OES.

Element	MDL Liquid mg L <sup>-1</sup>	MDL Solid mg kg <sup>-1</sup>
Aluminum (Al)	0.25	1.26
Arsenic (As)	0.030	0.150
Boron (B)	0.25	1.24
Cadmium (Cd)	0.020	0.100
Calcium (Ca)	23.62	118.08
Chromium (Cr)	0.040	0.200
Copper (Cu)	0.06	0.31
Iron (Fe)	0.47	2.35
Lead (Pb)	0.060	0.300
Magnesium (Mg)	10.59	52.96
Manganese (Mn)	0.03	0.15
Molybdenum (Mo)	0.01	0.06
Nickel (Ni)	0.040	0.200
Phosphorus (P)	0.22	1.10
Potassium (K)	1.93	9.66
Selenium (Se)	0.040	0.200
Sodium (Na)	6.57	32.85
Sulfur (S)	0.48	2.41
Zinc (Zn)	0.10	0.50

**pH**

For solid and semi-solid samples, 40 mL of deionized water is added to 20 cm<sup>3</sup> of homogenized sample and stirred to create a 1:2 (v/v) slurry. The sample is allowed to stand for 30 minutes and pH is measured with a Thermo Scientific Orion Versa Star Pro pH meter with a Green epoxy

non-fillable pH electrode with BNC connection (USEPA 2004b; Wolf 2003a). Some sample types that are very dry upon receipt (e.g., paper fibers, chicken and turkey litters, some composts) may require more than 20 mL DI water to achieve the appropriate slurry consistency.

The pH of liquid waste is determined directly on samples at 25 °C using a Thermo Scientific Orion Versa Star Pro pH meter with a Green epoxy non-fillable pH electrode with BNC connection (USEPA 2004a; Wolf 2003a).

#### *pH Quality Control*

A three-buffer calibration is performed daily with a slope maintained between 98% and 102%.

### **Electrical conductivity (EC)**

Electrical conductivity (EC) is a measure of soluble salts (SS) and of the ability of an aqueous solution to carry a current. The EC of an aqueous solution depends on the total concentration, mobility, and valence of ions and on the temperature of the sample.

For liquid waste samples, electrical conductivity (EC) is measured directly on homogenized samples at 25 °C using a conductivity meter and probe (SevenMulti; Mettler-Toledo, LLC; Columbus, OH) (Wolf 2003b).

For solid and semi-solid samples, 40 mL of deionized water is added to 20 cm<sup>3</sup> of homogenized sample and stirred to create a 1:2 (v/v) slurry. The sample is allowed to stand for 30 minutes. Electrical conductivity (EC) is measured on this 1:2 slurry using a conductivity meter and probe (SevenMulti; Mettler-Toledo, LLC; Columbus, OH) (Wolf 2003b).

EC is reported in units of mS cm<sup>-1</sup> and SS is expressed in units of 10<sup>-5</sup> S cm<sup>-1</sup>.

#### *EC Quality Control*

The EC meter is calibrated daily with a 1000 µS conductivity standard and an independent calibration verification using a second source standard is analyzed weekly. A duplicate solution sample is analyzed daily as a quality control sample with an acceptance criteria of < 2% relative standard deviation.

### **Calcium carbonate equivalent (CCE%)**

This method quantitatively determines the acid-neutralizing capacity of manure or other organic residuals (e.g., biosolids, food waste, compost) relative to pure calcium carbonate (CaCO<sub>3</sub>) by reacting with acid at elevated temperature and titrating with base to a potentiometric end point. The test provides useful information for any material that may have significant CCE such as lime-stabilized biosolids, poultry litter, paper mill waste, and ash by-products. The method is suitable to both solid and liquid waste materials but is more frequently applicable to solid samples. Land application rates should not exceed calcium carbonate equivalent (CCE) loading rates necessary to optimize soil pH, as determined by soil testing.

For liquid samples, 50 mL 0.5N hydrochloric acid (HCl) is added to 10 mL of homogenized sample and for solid and semi-solid samples, 50 mL 0.5N HCl is added to 1.0 g of dried and ground sample. The sample is boiled gently for five minutes, allowed to cool and then back-titrated to pH 7.0 with 0.25N of sodium hydroxide (NaOH), according to the potentiometric titration method (AOAC, 1990a). The volume of NaOH required to attain the end point is inversely related to the CCE.

Results are reported as percentage of pure calcium carbonate (% CaCO<sub>3</sub>) on a dry weight basis for solid and semi-solid samples and on an as-received basis for liquid samples. The NCDA&CS Waste report also includes a calculation based on the % CCE that indicates the amount of the waste material on an as-received basis that provides a liming value equivalent to one ton of agricultural grade limestone (assuming 90% calcium carbonate). This value is called the Agricultural Liming Equivalent (ALE) and is expressed in ton ton<sup>-1</sup>. For example, an ALE of 2.0 indicates that 2.0 tons of the waste material has the liming value of 1.0 tons of agricultural grade limestone.

#### *CCE Quality Control*

A reference material of laboratory grade (99.8%) CaCO<sub>3</sub> is analyzed in each batch with an acceptance criteria of 97-103 % CCE.

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