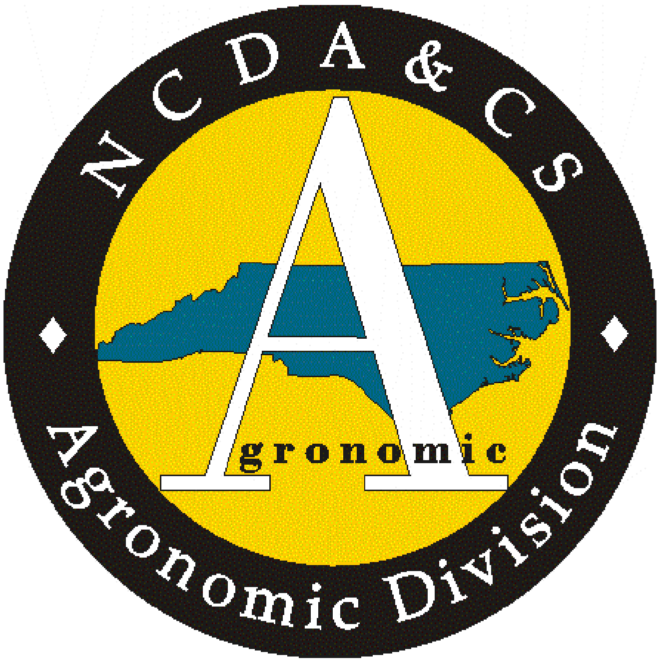
NCDA&CS
Waste and Compost Analytical Methods



North Carolina Department of Agriculture & Consumer Services

Agronomic Division

Waste and Compost Analysis Laboratory

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Introduction

Waste and compost analysis is used to measure 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 for the use of these materials in land application for agricultural production.

The NCDA&CS Waste Analysis Lab performs the following analyses based on standard industry methods:

- Nitrogen, total
- Elements: Phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg), sulfur (S), iron (Fe), manganese (Mn), zinc (Zn), copper (Cu), boron (B), sodium (Na), and molybdenum (Mo) and aluminum (Al)
- Nitrate-nitrogen (NO_3-N)
- Ammonium-nitrogen (NH_4-N)
- Carbon, total
- Heavy Metals: cadmium (Cd), nickel (Ni), lead (Pb), arsenic (As), chromium (Cr), and selenium (Se)
- Calcium carbonate equivalent (CCE)
- pH
- Electrical conductivity (EC)

Minimum Sample Masses and Volumes

Submitting an adequate sample size is essential for producing accurate and precise analytical data. NCDA&CS is not responsible for imprecise data associated with client submission of samples that are lower than the recommended limits of the method.

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^3) for solids and semi-solids. Where this is not possible, please note the minimum sample volume required for 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**
N, total and C, total	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_3-N, NH_4-N	1.0 g	KCl extraction; Flow Injection Analysis	EPA 353.2 EPA 350.1
pH	10 cm^3	pH meter	EPA 9045D
EC/SS	10 cm^3	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.

Liquid Samples Method Summary			
Sample Test	**Minimum Volume**	**Analytical Method**	**Reference**
Nitrogen, total	5 mL	Chemiluminescence	SM 4500-N
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_3-N, NH_4-N	15 mL	Filtered; Flow Injection Analysis	EPA 353.2 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

Sample Processing & Storage

Liquid samples are refrigerated at 4 °C upon receipt and homogenized immediately prior to analysis by manual shaking (EC, CCE, NO_3-N, NH_4-N) or with a roto-stator homogenizer (pH, total N, ICP-OES). All samples are analyzed on an as-received basis and are reported in mg L^{-1} and lb 1000 gal^{-1} for all elements.

Solid and semi-solid samples are refrigerated at 4 °C upon receipt. For total N, total C, NO_3-N, NH_4-N, pH, and EC analyses on samples where ammonia loss is a concern (e.g., manures and manure compost), the sample is homogenized prior to subsampling using a blade homogenizer (e.g., commercial food processor). A 50-100 cm^3 subsample is collected from this homogenized primary sample and stored at 4 °C until analyzed. For ICP-OES and CCE analysis on this type of sample, a 250 cm^3 subsample is weighed (Mettler Toledo ME4002TE; Mettler-Toledo, LLC; Columbus, OH) and dried for 48 hr at 80 ^oC in a forced air dryer (Horizontal Flow Oven; VWR International LLC, Radnor, PA).

For non-manure samples, a 250 cm^3 non-homogenized sample is weighed and dried for 48 hr at 80 ^oC in a forced air dryer. All dried samples are 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 Hicks et al. 2022d). Small volume (< 100 cm^3) 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 are 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. Percent solids, or dry matter percent is reported on the client report and is calculated as:

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

Where:

DM = dry matter
DW = oven-dry weight
AW = as-received weight

Analytical Methods

Nitrogen and Carbon--Solid and Semi-Solids

Total N (organic and inorganic) is measured on all solid and semi-solid samples. Total C 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).

Manure samples. Where ammonia loss is a concern, such as manures, 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_2 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^{-1} on an as-received basis as follows:

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

Non-manure samples. Samples other than manure, e.g., paper fibers, ash materials, crop residues, are analyzed on a dry basis. 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_2)-containing environment with subsequent quantification by thermal conductivity detector (AOAC 1990b; AOAC 2006; Hicks et al. 2022a; Hicks et al. 2022b). N and C results are measured as % and reported in mg kg^{-1} on a dry-weight basis as follows:

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

Estimate of Available Nutrients. In addition to reporting N in mg kg^{-1} on either an as-received basis or dry-weight basis, N is also reported as lb ton^{-1} on an as-received basis for all sample types. The coefficient used to determine the N availability in manure samples is specific to the Waste Code and Application Method reported by the client and careful consideration should be used in selecting the appropriate code to report.

For manure samples analyzed as-received, the Estimate of Available Nutrients is as follows:

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

Where:

NAC = Nitrogen Availability Coefficient

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

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

Where:

DM% = Dry matter %, a moisture correction factor

Carbon:Nitrogen Ratio (C:N) is calculated from this analysis and is reported in whole numbers as C mg kg^{-1} / N mg kg^{-1}. 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.

Nitrogen and Carbon Quality Control:
Method detection limits (MDL) are determined when a new instrument or method is put into use and verified annually.

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.

Nitrogen--Liquids

Total nitrogen is quantified in liquid waste samples by redox and chemiluminescence detection (APHA 2023). This method quantitatively determines total bound nitrogen (TNb) defined as the sum of inorganic nitrogen and organic nitrogen. The sample is oxidized by injection into a high temperature furnace (720°C) and quantified by a chemiluminescence detector. 40-50 mL of sample is homogenized by custom-built robot (Automation Techniques; Greensboro, NC) equipped with a roto-stator homogenizer (Omni International; Kennesaw, GA) for 8 seconds at a speed of 7500 rpm. 2 mL of sample is diluted up to 10 mL with deionized water and quantified using a chemiluminescence TOC/TNb analyzer (Trace Elemental Instruments; Houston, TX).

Nitrogen Quality Control
Method detection limits (MDL) are determined when a new instrument or method is put into use and verified annually.

Samples are quantified using a 6-point calibration. A method blank, reference material (Glycine), 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. An independent calibration verification solution is analyzed at the beginning and end of each run.

Inorganic nitrogen: NO_3-N and NH_4-N

For liquid samples, nitrate-nitrogen (NO_3-N) and ammonium-nitrogen (NH_4-N) are determined on a 10 mL sample which is shaken and then filtered through a pre-folded Advantec #2 filter paper (Folded Filter Paper, Albuquerque, NM). For solid and semi-solid samples, NO_3-N and NH_3-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 centrifuged and filtered through a 110 mm filter.

NO_3-N is determined on the filtrate or extractant by cadmium reduction, where nitrate is reduced to nitrite with copperized cadmium, under alkaline conditions. The NO_2-N concentration (that originally present plus reduced nitrate) is determined by diazotizing with sulfanilamide and coupling with N-(1-Naphthyl) ethylenediamine dihydrochloride to form a magenta-colored azo dye which is measured at 520 and 600 nm (Hicks 2022; USEPA 1993a; FIA NO3-W-1-1).

NH_4-N is determined on the filtrate or extractant based on a modified Berthelot reaction where hypochlorite and sodium salicylate react with ammonia in a two-step reaction, converting it to 5-aminosalicylate. The aminosalicylate is oxidized in the presence of sodium nitroferricyanide to form a blue-green colored complex, which is then measured at 660 nm (Miller 2022; USEPA 1993b; FIA NO3-W-1-2).

Both NO_3-N and NH_4-N are quantified by flow injection analysis (FIAlyzer-3000, FIA Lab). Nitrate-nitrogen (NO_3-N) and nitrite-nitrogen (NO_2-N) are reported as NO_3-N on the Waste Analysis Report. Ammonium-nitrogen (NH_3-N + NH_4-N) is reported as NH_4-N on the Waste Analysis Report. Results are expressed in mg kg^{-1} or mg L^{-1}.

Inorganic Nitrogen Quality Controls
Method detection limits (MDL) are determined when a new instrument or method is put into use and verified annually.

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_2-N) are analyzed to verify the completeness of the nitrate reduction reaction at the beginning and end of each run.

Elemental Analysis

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) (USEPA 2001; Hicks et al. 2022e), after closed-vessel nitric acid (HNO_3) digestion in a microwave digestion system (MARS 6 Microwaves; CEM Corp.; Matthews, NC) (Spargo and Miller 2022).

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_3. 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_3 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 Advantec #2 filter paper (Folded Filter Paper, Albuquerque, NM). 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)**
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^{-1} and in lb 1000 gal^{-1}.

Solid and semi-solid results are reported in mg kg^{-1} on a dry-weight basis and in lb ton^{-1} 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:

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

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^{-1}. P and K are reported as P_2O_5 and K_2O on an as-received basis in lb ton^{-1} as follows:

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

Where:

E2 = P or K
E3 = P_2O_5 or K_2O
NM = conversion factor for P (2.29) to P_2O_5 or K (1.20) to K_2O
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, and external reference are digested and analyzed with each batch. An interference blank is analyzed at the start and end of each analysis.

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.

pH

For raw solid and semi-solid samples, 40 mL of deionized water is added to 20 cm^3 of homogenized sample and stirred to create a 1:2 (v/v) slurry. For dried, ground solid and semi-solid samples, 10 mL of deionized water is added to 10 cm^3 of homogenized sample and stirred to create a 1:1 (v/v) slurry. All sample types are allowed to stand for 30 minutes after DI water has been added, and pH is measured with a Thermo Scientific Orion Versa Star Pro meter with a Green epoxy non-fillable pH electrode with BNC connection and an Automatic Temperature Compensation probe (USEPA 2004b; Spargo et al. 2022a).

The pH of liquid waste is determined directly on samples at 25 °C using a Thermo Scientific Orion Versa Star Pro meter with a Green epoxy non-fillable pH electrode with BNC connection and an Automatic Temperature Compensation probe (USEPA 2004a; Spargo et al. 2022a).

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

Electrical conductivity

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 Thermo Scientific Orion Versa Star Pro meter with an Orion EC probe and an Automatic Temperature Compensation probe (Spargo et al. 2022b).

For raw solid and semi-solid samples, 40 mL of deionized water is added to 20 cm^3 of homogenized sample and stirred to create a 1:2 (v/v) slurry. The sample is allowed to stand for 30 minutes. For dried, ground solid and semi-solid samples, 20 mL of deionized water is added to 10 cm^3 of homogenized sample and stirred to create a 1:2 (v/v) slurry. The sample is allowed to stand for 60 minutes. Electrical conductivity (EC) is measured on this 1:2 slurry using a Thermo Scientific Orion Versa Star Pro meter with an Orion EC probe and an Automatic Temperature Compensation probe (Spargo et al. 2022b).

EC is reported in units of $mS\ cm^{-1}$ and SS is expressed in units of $10^{-5}\ S\ cm^{-1}$.

EC Quality Control

The EC probe is checked daily with two independent calibration verification solutions.

Calcium carbonate equivalent

Calcium carbonate equivalent (CCE%) quantitatively determines the acid-neutralizing capacity of manure or other organic residuals (e.g., biosolids, food waste, compost) relative to pure calcium carbonate ($CaCO_3$) 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 for 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; Hicks et al. 2022c). 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 (% $CaCO_3$) 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^{-1}. For example, an ALE of 2.0 indicates that 2.0 tons of the waste material has the liming value of 1.0 ton of agricultural grade limestone.

CCE Quality Control

A reference material of laboratory grade (99.8%) $CaCO_{3.}$ is analyzed in each batch with an acceptance criteria of 95-105 % CCE.

References

Association of Official Analytical Chemists. (1990a). AOAC official method 955.01: neutralizing value for liming materials. In: Official methods of analysis. Volume 1. 15th ed. Arlington (VA): AOAC International. p 1–2.

Association of Official Analytical Chemists. (1990b). AOAC official method 972.43: microchemical determination of carbon, hydrogen, and nitrogen. In: Official methods of analysis. Volume 1. 15th ed. Arlington (VA): AOAC International. p 341.

Association of Official Analytical Chemists. (2006). AOAC Official Method 990.03. Protein (Crude) in Animal Feed, Combustion Method, in Official Methods of Analysis of AOAC International, 18th Edition. Revision 1, Chapter 4, pp. 30-31. AOAC International, Arlington, VA.

American Public Health Association. (2023). Standard Methods for the Examination of Water and Wastewater. 24th Edition. SM 4500-N: Total Nitrogen Determination by Redox and Chemiluminescence Detection. Washington (DC): Public Health Association, Water Environment Federation, and American Water Works Association.

Hicks, K. and R. O. Miller. (2022). Recommended Methods of Manure Analysis, 2nd ed. M-4.3 Nitrogen, Nitrate by Cadmium or Hydrazine Reduction; Automated Spectrophotometry. P. 55-60.

Hicks, K., R. Brimmer, J.T. Spargo. (2022a). Recommended Methods of Manure Analysis, 2nd ed. M-2.1 Carbon, Total by Combustion. P. 23-25.

Hicks, K., R. Brimmer, J.T. Spargo. (2022b). Recommended Methods of Manure Analysis, 2nd ed. M-3.1 Nitrogen, Total by Combustion. P. 27-30.

Hicks, K., T. Hoerner, and J. T. Spargo. (2022c). Recommended Methods of Manure Analysis, 2nd ed. M-7.1 Calcium Carbonate Equivalent (CCE). P. 87-90.

Hicks, K., T. Hoerner, and J. T. Spargo. (2022d). Recommended Methods of Manure Analysis, 2nd ed. S-2.0 Laboratory Sample Processing & Storage. P. 9-14.

Hicks, K., R. O. Miller, and J. T. Spargo. (2022e). Recommended Methods of Manure Analysis, 2nd ed. M-5.2 Phosphorous, Potassium, & Metals by Inductively Coupled Plasma—Optical Emission Spectroscopy. P. 71-78.

Miller, R.O., K. Hicks, J. Lessl, J. T. Spargo, and J. Mowrer. (2022). Recommended Methods of Manure Analysis, 2nd ed. M-4.2 Nitrogen, Ammonium (NH4-N) by Distillation, Spectrophotometry, and Diffusion-Conductivity. P. 47-53.

Spargo, J. T. and R. O. Miller. (2022). Recommended Methods of Manure Analysis, 2nd ed. M-5.1 Digestion & Dissolution for Elemental Analysis. P. 61-69.

Spargo, J. T., R. Brimmer, J. Lessl, B. Thayer, and K. Hicks. (2022a). Recommended Methods of Manure Analysis, 2nd ed. M-8.1 pH. P. 91-93.

Spargo, J. T., R. Brimmer, J. Lessl, B. Thayer, and K. Hicks. (2022)b. Recommended Methods of Manure Analysis, 2nd ed. M-9.1 Electrical Conductivity (EC). p. 95-97.

United States Composting Council. (2001).Test Methods for the Examination of Composting and Compost. Ed W.H. Thompson. Method 4.10-A. USDA and U.S. Composting Council.

United States Environmental Protection Agency. (1993a). Method **353.2**, Revision 2: Methods for Determination of Nitrate-Nitrite Nitrogen by Automated Colorimetry. https://www.epa.gov/sites/default/files/2015-08/documents/method_353-2_1993.pdf

United States Environmental Protection Agency. (1982). Method **120.1**. Methods for the Chemical Analysis of Water and Wastes. Conductance (specific conductance, µmhos/cm at 25 °C). Available at https://www.nemi.gov/methods/method_summary/5210/

United States Environmental Protection Agency. (1993b). Method **350.1**. Determination of Ammonia Nitrogen by Semi-Automated Colorimetry. Available at https://www.nemi.gov/methods/method_summary/5405/

United States Environmental Protection Agency. (1996a). Method **3052**. Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices. Available at: https://www.epa.gov/hw-sw846/sw-846-test-method-3052-microwave-assisted-acid-digestion-siliceous-and-organically-based .

United States Environmental Protection Agency. (2001). Method **200.7**. Trace elements in water, solids, and biosolids by inductively coupled plasma–atomic spectrometry, revision 4.4. Available at nepis.epa.gov/EPA/