Test Methods for the Examination of Composting and Compost - Draft Version 1

4.2 Nitrogen

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Research & Education Foundation

Test Methods: Nitrogen	**Last Updated:** Jan/27/2026
4.2-A: A Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique	**Units:** % dry weight
4.2-B: Nitrate Nitrogen	**Units:** mg/kg dry weight
4.2-C: Ammonium Nitrogen	**Units:** mg/kg dry weight
4.2-D: Total Nitrogen by Automated Combustion (Dumas Method)	**Units:** % dry weight
4.2-E: Total Nitrogen by Wet Oxidation	**Units:** % dry weight

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Methods 4.2-A, 4.2-B (Ion Chromatography), 4.2-B (Colorimetry -Cd reduction), 4.2-C, 4.2-D -

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Methods 4.2-B (Colorimetry -Hydrazine reduction), 4.2-D: Total Nitrogen by Automated Combustion (Dumas Method), 4.2 Nitrogen: Quality Control (QC) -

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TMECC Method 4.2 Nitrogen - Summary of Revisions (2026)

The 2026 revision of TMECC Method 4.2 Nitrogen is a comprehensive technical and editorial update to modernize nitrogen determinations in compost and compost feedstocks, improve interlaboratory comparability, and eliminate bias arising from sample handling variability and method ambiguity.

Across all nitrogen determinations—Method 4.2-A: Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique; Method 4.2-B: Nitrate Nitrogen; Method 4.2-C: Ammonium Nitrogen; Method 4.2-D: Total Nitrogen by Automated Combustion (Dumas Method); and Method 4.2-E: Total Nitrogen by Wet Oxidation—sample preparation has been standardized to require analysis of as-received moist material, with samples refrigerated at ≤ 6 °C and air-drying, oven-drying, grinding, milling, and sieving explicitly prohibited due to documented ammonia volatilization and ongoing microbial nitrogen transformations. Dry-weight reporting has been harmonized to an oven-dry basis of 104 ± 1 °C, reflecting the guidance introduced in the 2015 revision of TMECC Method 03.09: Total Solids and Moisture, which added Method 03.09-B (105 °C) to accommodate regulated feedstocks (e.g., U.S. EPA biosolids requirements) and to improve laboratory efficiency by reducing drying time relative to the original 70 °C protocol.

Method 4.2-A: Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique has been revised to eliminate conditional sample storage and drying options that previously depended on presumed nitrogen volatility, which required prior knowledge of nitrogen speciation and introduced subjectivity among laboratories.

Method 4.2-B: Nitrate Nitrogen has been expanded to explicitly include hydrazine reduction colorimetry as an approved technique, in addition to ion chromatography and cadmium reduction methods, while removing instrument-specific prescriptions and requiring verification of nitrate-to-nitrite reduction efficiency when operating outside U.S. EPA Method 353.2 configurations.

Method 4.2-C: Ammonium Nitrogen has been corrected to require 2 M potassium chloride extraction and an updated ammonium-to-nitrogen conversion factor.

Total Nitrogen, Method 4.2-D is now restricted to dry combustion (Dumas Method), and has been renamed 4.2-D: Total Nitrogen by Automated Combustion (Dumas Method). Wet chemical oxidation procedures have been separated into Method 4.2-E (Total Nitrogen by Wet Oxidation).

Throughout Method 4.2, sample sizes, reagent volumes, and instrument-specific parameters are identified as “recommended” rather than prescriptive, allowing laboratories operating under formal TMECC agreements to apply internal standard operating procedures and instrumentation-specific practices, provided that the quality control acceptance criteria are met. In contrast, sample handling, storage, drying conditions, and preservation requirements are explicitly prescribed, as these factors directly affect nitrogen stability, analytical accuracy, and regulatory defensibility.

Quality control requirements have been substantially revised to define batch size, and use relative standard deviation (RSD) for quality control acceptance and relative median deviation (RMD) for performance context.

These revisions aim to align TMECC Method 4.2 - Nitrogen with current EPA, AOAC, APHA, ISO, and manure-based analytical guidance, while preserving laboratory flexibility and reflecting contemporary analytical instrumentation, individual laboratory standard operating procedures and best practices. We welcome feedback to info@compostfoundation.org .

Disclaimers

The methods in this manual (Test Methods for the Examination of Composting and Compost, TMECC) do not address all safety concerns. Users are responsible for establishing appropriate safety and health practices, reviewing current Safety Data Sheets, and determining regulatory applicability before use. Safety measures including personal protective equipment, ventilation, handling, storage, emergency response, and waste disposal must comply with current institutional and regulatory standards. Chemical hazards and requirements vary by substance and jurisdiction and may change over time. This manual does not provide comprehensive safety information; users must consult current regulations and manufacturer instructions. Failure to follow proper procedures may result in injury, environmental harm, or legal violations.

All methods and sampling protocols are subject to revision to correct errors or reflect advances in practice. Report omissions or errors to the TMECC editors. Process alternatives, trade names, or commercial products mentioned are provided as examples only and do not constitute endorsement.

Values stated in International System of Units (SI) are to be regarded as the standard. Values given in parentheses are provided for information only.

Scope

This section covers tests for determination of nitrogen in compost and compost feedstocks.

Method 4.2-A: Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique — Determination of organic nitrogen with a semi micro-Kjeldahl method to measure total nitrogen along with instructions for including or excluding nitrate in the analysis.

Method 4.2-B: Nitrate Nitrogen — Two techniques for the determination of nitrate nitrogen; *Ion Chromatography* and *Colorimetry.*

Method 4.2-C: Ammonium Nitrogen — Two techniques for the determination of ammonium nitrogen; *Ion-Selective Electrode* and *Colorimetry.*

Method 4.2-D: Total Nitrogen by Automated Combustion (Dumas Method) — Automated dry combustion (Dumas method), measuring overall nitrogen content without differentiation of individual nitrogen forms.

Method 4.2-E: Total Nitrogen by Wet Oxidation — Total Nitrogen using wet chemical techniques.

Significance and Use

Nitrogen is a macronutrient and is a key element required for plant growth. It is an essential part of proteins, which comprise most of the dry weight of plant and animal cells. In composting, it is a critical macronutrient required for microbial growth and organic matter decomposition, typically supplied by materials like food scraps, grass clippings, and manure. Nitrogen is available to plants primarily in two inorganic forms: ammonium and nitrate. Nitrogen is converted between different forms [organic nitrogen, ammonium (NH_4^+), ammonia (NH_3), nitrite, and nitrate (NO_3^-)] via biological processes including ammonification, nitrification, and denitrification. Low nitrate concentration in compost indicates immaturity, and may cause nitrogen deficiency in plants when the compost is applied to soil. Nitrate is a potential pollutant in ground and surface waters. Biological reduction of nitrate and nitrite to molecular nitrogen or nitrogen oxides results in loss of nitrogen from soil or compost to the atmosphere.

Method 4.2-A: Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique — Wet chemical technique that measures organic nitrogen and ammonium nitrogen. Nitrate nitrogen and nitrite nitrogen are not determined unless the *nitrate reduction* step is included prior to digestion, in which case the result represents total nitrogen (*Method 4.2-E: Total Nitrogen by Wet Oxidation).* This method is an alternative to *Method 4.2-D: Total Nitrogen by Automated Combustion (Dumas Method).*

Method 4.2-B: Nitrate Nitrogen — Wet chemical technique that measures nitrate nitrogen. Used for determining the ammonium to nitrate ratio to assess compost maturity. It is also required for total nitrogen determination using *Method 4.2-E: Total Nitrogen by Wet Oxidation* where no *nitrate reduction* step is used with *Method 4.2-A Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique.*

Method 4.2-C: Ammonium Nitrogen — Wet chemical technique that measures ammonium nitrogen, used for calculating the ammonium-to-nitrate ratio to assess compost maturity. This method is not suitable for total nitrogen determination because it excludes organic nitrogen, nitrate nitrogen, nitrite nitrogen, and other nitrogen forms.

Method 4.2-D: Total Nitrogen by Automated Combustion (Dumas Method) — Automated dry combustion technique measuring total nitrogen regardless of its oxidation state or chemical form. No separate nitrate determination is required for determining total N. Preferred for samples containing nitrate, NO_2^-, or other nitrogen forms not fully recovered by *Method 4.2-A: Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique.* As an alternative to the Kjeldahl method, Dumas-type nitrogen analyzers measure all forms of nitrogen by thermal combustion, converting nitrogen in the sample to nitrogen gas (N_2), which is then quantified. Provides faster results and eliminates the use of hazardous wet chemistry reagents compared to *Method 4.2-E: Total Nitrogen by Wet Oxidation*.

Method 4.2-E: Total Nitrogen by Wet Oxidation — Calculates total nitrogen from nitrogen measured in either i) *Method 4.2-A Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique* with *nitrate reduction* step included, or ii) the sum of nitrogen measured with *Method 4.2-A Total*

Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique and nitrate determined using *Method 4.2-B Nitrate Nitrogen.*

Terminology

Ammonia, NH_3 — Colorless, pungent gas produced during microbial decomposition of organic nitrogen compounds. Ammonia can volatilize under high pH and temperature conditions or be converted to ammonium, NH_4^+ in moist, acidic environments.

Ammonium, NH_4^+ — Formed by the protonation of ammonia, NH_3. Ammonium is a plant-available form of nitrogen that is retained in soil and compost through cation exchange with negatively charged particles, and serves as the substrate for microbial nitrification under aerobic conditions.

Ammonium nitrogen (NH_4^+-N) — An analytical reporting convention that expresses only the nitrogen portion of ammonium, excluding hydrogen mass. Ammonium nitrogen is used to standardize nitrogen measurements and compare the amount of nitrogen in ammonium to the amount of nitrogen in other nitrogen forms (e.g., NO_3^--N, total N).

Denitrification — Microbial process that reduces nitrate, NO_3^- and nitrite, NO_2^- to gaseous nitrogen compounds (primarily N_2, also nitric oxide, NO and nitrous oxide, N_2O) under anaerobic or oxygen-limited conditions. In composting, denitrification is a potential nitrogen loss pathway, most common in waterlogged or compacted zones with high carbon availability, where bacteria use nitrate as an alternative electron acceptor. This completes the nitrogen cycle by returning nitrogen to the atmosphere and can be minimized through proper aeration and moisture control.

Nitrate, NO_3^- — An anionic form of nitrogen that is highly soluble and readily available for plant uptake. Formed through microbial nitrification of ammonium and often a result of organic matter decomposition. Nitrate is prone to leaching under wet conditions, and can be lost via denitrification in anaerobic environments.

Nitrate nitrogen, NO_3^--N — An analytical reporting convention that expresses only the nitrogen portion of nitrate, excluding oxygen mass. Standardizes nitrogen measurements and allows comparison of the amount of nitrogen in nitrate to the amount of nitrogen in other nitrogen forms (e.g., NH_4^+-N, total N).

Nitrite, NO_2^- — An intermediate, anionic form of nitrogen produced during the first step of nitrification by the microbial oxidation of ammonia or ammonium. Nitrite is typically present at low concentrations in compost because nitrite is rapidly oxidized to nitrate under aerobic conditions or reduced during denitrification under oxygen-limited conditions. Accumulation of nitrite may indicate transient imbalances in aeration, moisture, or microbial activity.

Nitrification — A two-step aerobic microbial process in which ammonia or ammonium is oxidized to nitrite by ammonia-oxidizing bacteria or archaea, and then to nitrate by nitrite-oxidizing bacteria.

Organic nitrogen — Organic nitrogen is nitrogen bound within carbon-containing organic molecules. Organic nitrogen can be mineralized by microbes into ammonium nitrogen.

Total Kjeldahl Nitrogen, TKN — The sum of organic nitrogen and ammonium nitrogen measured using the Kjeldahl digestion method. This method does not include nitrate nitrogen or nitrite

nitrogen, which must be analyzed separately. Total Kjeldahl Nitrogen should not be used alone to calculate the carbon-to-nitrogen (C:N) ratio as it may underestimate total nitrogen content due to the exclusion of nitrate and nitrite.

Total Nitrogen, TN — The measurement of all nitrogen forms present in materials, typically calculated as the sum of Kjeldahl nitrogen (organic nitrogen + ammonium nitrogen), nitrate nitrogen, and nitrite nitrogen. Total nitrogen may also be measured using dry combustion analysis (Dumas Method). This total nitrogen value is used to determine carbon-to-nitrogen ratios of both feedstock mixtures and compost products, expressed as % organic carbon to % (total Kjeldahl nitrogen + nitrate nitrogen) or as % organic carbon to % total nitrogen.

Interference and Limitations

See also individual methods for method-specific interference and limitations and *Appendix 4.2 Nitrogen - Quality Control.*

Sample Handling

Sample handling requirements are from APHA (2012), Hicks et. al. (2022b) and U.S. EPA (2026). Store samples refrigerated at ≤6 °C (typically 4 °C) immediately upon receipt and analyze as soon as possible (less than 7 days is recommended) to minimize ammonia volatilization and microbial transformation of nitrogen species. Freezing of samples is not recommended for routine nitrogen analysis. If freezing is unavoidable due to holding-time constraints, frozen samples should be analyzed immediately after thawing to room temperature because temperature affects pH and the ammonia-nitrite-nitrate balance within minutes of microbial activity resuming.

Ammonia loss through volatilization can occur during the air-drying step of sample preparation, particularly in nitrogen-rich manure or other compost feedstocks. This volatilization may lead to significant underestimation of total nitrogen. Air-drying shall be avoided prior to nitrogen analysis to minimize nitrogen loss before analysis.

Samples may be stored in polyethylene, fluoropolymer (polytetrafluoroethylene; Teflon®, or other fluoropolymer), or glass following U.S. EPA (2026), however, glass containers for compost storage and transport are discouraged (Hicks et. al., 2022b) due to breakage and contamination risks (glass shards). Select a representative test portion that is large enough to capture the compost's heterogeneity (e.g., moisture, particle size, and visible components). Homogenize gently and only as needed to achieve representativeness. Sieving, milling, grinding, or mechanical particle-size reduction are not permitted for nitrogen analyses. Selective removal of coarse material can bias ammonium, nitrate, and total nitrogen measurements because nitrogen is heterogeneously distributed among particle-size fractions. Excessive mechanical processing can reduce particle size, lyse cells, and promote oxidation and microbial transformations and bias nitrogen analyses by altering nitrogen speciation (e.g., ammonium, nitrate) prior to measurement (Ditsworth et al., 1990; Hicks et. al., 2022b, Stemmer et al., 1990). Sample sizes for nitrogen analyses are usually around 100mL.

See Table 4.2-1 for sample handling summary.

Table 4.2-1. Sample handling requirements for nitrogen analyses in compost samples. H_2SO_4 = sulfuric acid; P = polyethylene; FP = fluoropolymer (polytetrafluoroethylene; Teflon®, or other fluoropolymer); G = glass .

	Method A - Total Kjeldahl Nitrogen	***Method B - Nitrate Nitrogen***	***Method C - Ammonium Nitrogen***	***Method D - Total Nitrogen by Automated Combustion (Dumas Method)***	***Method E - Total Nitrogen by Wet Oxidation***
Sample storage and handling	Containers: P, FP Store at ≤ 6 °C upon receipt or analyze immediately. Freezing discouraged; if unavoidable, store at -18 °C; analyze immediately after thawing.	Containers: P, FP Store at ≤ 6 °C upon receipt or analyze immediately. Freezing discouraged; if unavoidable, store at -18 °C; analyze immediately after thawing.	Sealed Containers: P, FP Store at ≤ 6 °C immediately upon receipt. Freezing discouraged; if unavoidable, store at -18 °C; analyze immediately after thawing.	Container: P, FP Store at ≤ 6 °C immediately upon receipt. Freezing discouraged; if unavoidable, store at -18 °C; analyze immediately after thawing.	Refer to A and B
Sample drying	No drying, use as-received	No drying, use as-received	No drying, use as-received	No drying, use as-received	No drying, use as-received
Sieving/ milling	No sieving. Gentle manual homogenization.	No sieving. Gentle manual homogenization.	No sieving. Gentle manual homogenization.	No sieving. Gentle manual homogenization.	No sieving. Gentle manual homogenization.

Extract Storage	N/A	Containers: P, FP Refrigerate at ≤ 6 °C, analyze within 48 hours. For longer storage, acidify to pH < 2 with H_2SO for up to 28 days.	Containers: P, FP Refrigerate at ≤ 6 °C, analyze within 48 hours. For longer storage, acidify to pH < 2 with H_2SO for up to 28 days. Re-neutralize prior to ammonium determination.	N/A	Refer to A and B

Quality Control

See *Appendix 4.2 Nitrogen - Quality Control* for available precision & bias data and method-specific quality control.

4.2-A: Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique

Summary of Method 4.2-A: Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique

The Kjeldahl method is used to determine total nitrogen in organic compounds and ammonium. This method applies to compost, soil, peat, plants, manure, foods, feed, and other biological material. The procedure outlined in Method 4.2-A is a semi-micro adaptation of the *Association of Official Analytical Collaboration (AOAC) International 976.06 method, Protein (Crude) in Animal Feed - Semi-automated Method*. This technique measures nitrogen that can be converted to ammonium when the sample is digested in hot sulfuric acid. The acid and high-temperature environment enhances the rate of conversion of organic nitrogen to ammonia and increases the recovery of convertible nitrogen compounds. Ammonium in the digest is quantified by semi-automated colorimetric analysis using a salicylate/phenate (Berthelot) reaction. Reagents, sample size, and digestion tube size are all reduced five-fold from the AOAC method, and selenium is substituted for mercury as the catalyst. While selenium poses less risk than mercury, it still generates hazardous waste. Proper disposal must follow hazardous waste regulations.

Significance and Use for Method 4.2-A: Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique

The forms of nitrogen measured are those that can be reduced to ammonium when the sample is digested in hot sulfuric acid. The method measures organic nitrogen and ammonium, but it does not detect nitrogen held in bonds between nitrogens or nitrogen and oxygen that are found in nitrate, nitrite, or other nitro compounds, because these do not convert to ammonium under standard Kjeldahl digestion conditions. To quantitatively include these forms in the Kjeldahl procedure for total nitrogen, a *nitrate reduction* step must be included prior to sample digestion. The *nitrate reduction* step does not impact the total nitrogen value when the sum of ammonium nitrogen and nitrite nitrogen is less than 500 mg/kg.

Interference and Limitations for Method 4.2-A: Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique

Some nitrate in untreated samples may still be reduced during digestion due to the reducing power of organic matter, which can lead to overestimation of Kjeldahl nitrogen. To minimize this, an optional hydrogen peroxide treatment is included in the procedure.

Drying, even at low temperatures, leads to volatilization of ammonia and should therefore be avoided.

Temperature strongly influences the amount of potassium sulfate needed for the Kjeldahl digest. Potassium sulfate is added to the digestion mixture to raise the boiling point of sulfuric acid to achieve higher digestion temperatures and improve breakdown of organic matter and conversion of nitrogen to ammonium. However, high concentrations of potassium sulfate might cause the digest to solidify upon cooling. Careful attention must be given to mixing the warm digest with water after the digestion process in order to dissolve any remaining salts and avoid precipitation.

Total Kjeldahl nitrogen does not include nitrate or nitrite forms of nitrogen, although some conversion of nitrate/nitrite may take place at very high concentrations during the digest. When the combined concentration of nitrate nitrogen and nitrite nitrogen exceeds 500 mg/kg, the *nitrate reduction* step must be included before the *digestion* step.

Sample Handling for Method 4.2-A: Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique

For sample handling requirements see Table 4.2-1. Samples shall be refrigerated promptly upon receipt at ≤ 6 °C and analyzed as soon as possible; analysis within 7 days is recommended. Samples shall be analyzed as-received. Air-drying, oven-drying, grinding, sieving, or other aggressive mechanical homogenization is not permitted prior to digestion, as these actions may bias total Kjeldahl nitrogen results. Gentle manual mixing to obtain a representative test portion is allowed. Total solids and moisture shall be determined on a parallel aliquot for dry-weight correction of reported results.

Recommended Apparatus for Method 4.2-A: Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique

Aluminum Digestion Block — 64 hole; able to fit 25 x 200 mm glass tubes.

Pyrex Glass Tubes for Digestions — 25 × 200 mm, with 50 mL graduation mark.

AutoAnalyzer — automated colorimetric titration apparatus.

Disposable beakers — 5 mL capacity

Air scrubber — containing 10 % sulfuric acid (volume per volume [v/v])

Recommended Reagents and Materials for Method 4.2-A: Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique

Sulfuric Acid (H_2SO_4) — Concentrated (95 % to 98 %).

Tecator Kjeltabs® — Tablets that consist of 1.5 g potassium sulfate (K_2SO_4) and 7.5 mg selenium (Se) (e.g., No. 13 159-C, Fisher Scientific).

Salicylic Acid ($HC_7H_5O_3$) — powder, ≥ 99.5 % purity.

Sodium Thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) — Granular crystals (≥ 99 % purity).

Stock Nitrogen Standard (1000 mg/L N) — Dissolve 2.3584 g dried ammonium sulfate [$(NH_4)_2SO_4$] in a 500 mL volumetric flask and dilute to volume with deionized water. Working standards are made up in a composite of digested blanks diluted to the 50 mL mark of the digestion tubes, at concentrations spanning the sample range (usually 1-200 mg/L).

Stock Sodium Hydroxide ($NaOH$) Solution (20 %) — Make the stock solution of 50 % (weight by volume [w/v]) NaOH solution by weighing 250 g of NaOH pellets into a 500 mL volumetric flask. Dilute to volume with deionized water. To 600 mL deionized water, add 400 mL 50 % w/v NaOH solution. Cool to ambient laboratory temperature and dilute to 1 L with deionized water.

Stock 20 % Solution of Sodium Potassium Tartrate ($NaKC_4H_4O_6.4H_2O$) — Dissolve 200 g $NaKC_4H_4O_6.4H_2O$ in 600 mL of deionized water and bring to volume in a 1 L volumetric flask, mixing thoroughly.

Stock Buffer Solution 0.5 molar (M) Sodium Phosphate (Na_2HPO_4) — Dissolve 134 g $Na_2HPO_4 \cdot 7H_2O$ (or 71 g Na_2HPO_4) in 800 mL deionized water. Add 40 g NaOH solution, 50 % w/v, dilute to 1 L, and mix thoroughly.

Working Buffer Solution — Combine the reagents in this order: add 250 mL stock 20 % $NaKC_4H_4O_6.4H_2O$ solution to 200 mL stock 0.5 M Na_2HPO_4 buffer solution while swirling. Continuing to swirl, add 250 mL 20 % NaOH solution. Dilute to 1 L with deionized water then add 1 mL Brij-35 (surfactant, 30 % solution) and mix thoroughly.

Sulfuric Acid/Sodium Chloride (H_2SO_4/NaCl) Solution — Dissolve 100 g NaCl in 600 mL of deionized water. Add 7.5 mL of H_2SO_4 and dilute to 1 L with deionized water. Add 1 mL of Brij-35 and mix thoroughly.

Sodium Salicylate/Sodium Nitroprusside Solution ($NaC_7H_5O_3$ / $Na_2[Fe(CN)_5NO] \cdot 2H_2O$) — Dissolve 150 g $NaC_7H_5O_3$ and 0.30 g $Na_2[Fe(CN)_5NO] \cdot 2H_2O$ in about 600 mL distilled water. Filter through fast filter paper into a 1 L volumetric flask and dilute to volume with distilled water. Add 1 mL Brij-35, mix thoroughly, and store in a light-resistant container.

Sodium Hypochlorite (NaOCl) Solution, 0.315 % — Dilute 6.0 mL NaOCl solution to 100 mL with distilled water. Add 0.1 mL of Brij-35 and mix thoroughly. Prepare fresh daily.

Sample Wash Solution — Triple deionized water without a wetting agent.

Certified reference material — National Institute of Standards and Technology (NIST) pine needles or similar.

Recommended Procedure for Method 4.2-A: Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique

a) *Prepare sample* — Use as-received moist material to prevent nitrogen loss through volatilization. Manually mix the as-received moist sample to achieve representativeness. Do not sieve, grind, or mill material intended for nitrogen analysis. Determine moisture content on a separate, parallel aliquot to enable conversion of analytical results to a dry-weight basis (*TMECC Method 3.9-A: Total Solids and Moisture at 70 °C)*. Avoid cross-contamination of volatile ammonia between samples by using clean tools and containers.

b) Weigh 150 to 250 mg of compost onto nitrogen-free paper. Fold the paper to fully enclose the sample.

c) *Optional nitrate reduction:* Begin by treating the compost sample using one of the following options. If the combined nitrate and nitrite nitrogen concentration exceeds 500 mg/kg, perform the *nitrate reduction* process (Option 1) before proceeding to *step d) Digestion.*

Either:

Option 1) Nitrate reduction process to include nitrate: Samples must be pretreated with salicylic acid and sodium thiosulfate to convert nitrate to amino compounds that can be measured by the Kjeldahl method (Dalal et al., 1984; Stalcup & Williams, 1955). In this pretreatment step, nitrates react with salicylic acid in an acidic environment to form nitro compounds, which are reduced to the corresponding amino compounds by heating the mixture with sodium thiosulfate.

i) Weigh 75 mg salicylic acid onto a separate piece of nitrogen-free paper and place it into the digestion tube along with the compost sample and 3.5 mL concentrated sulfuric acid.

ii) Let the tube stand for 1 hour, then swirl gently until the paper is dissolved and the mixture is uniform. Allow the sample to stand undisturbed at room temperature for 12 to 18 hours. After this period, weigh and add 500 mg sodium thiosulfate to the digestion tube. A funnel may be used to direct the powder to the bottom.

iii) Place the tubes in the digestion block and gradually increase the temperature to 320 °C. Heat the sample until frothing subsides. Once the reaction is complete, remove the tubes and allow them to cool to approximately 240 °C before proceeding to step d) Digestion.

Or:

Option 2) To exclude nitrate: hydrogen peroxide is added to oxidize organic matter, which prevents unwanted reduction of nitrate during digestion to exclude nitrate nitrogen (Bowman et al., 1988). See *Procedure for Method 4.2-E: Total Nitrogen by Wet Oxidation* for implications for Total Nitrogen calculations.

d) Digestion — Add 1 selenium-based catalyst tablet (e.g., Kjeltab®) to each tube. Record the container or lot number for each new batch of catalyst tablets used. Insert the tubes into a preheated aluminum digestion block set to 400 °C. Temperature of the digest must remain 360 °C to 410 °C. Digest the samples for approximately 30 minutes after the disappearance of black carbon from the solution. Municipal solid waste compost samples typically require 1 to 2 hours to fully digest. Each digestion batch may contain up to 64 tubes.

e) Dilution — After digestion, allow tubes to cool for approximately 10 minutes. Carefully add 10 to 20 mL of deionized water to the digestion tubes while they are still warm. Mix with a vortex mixer to dissolve any crystals that may have formed. Avoid adding water when the tubes are too hot to avoid spattering and possible sample loss and injury. Dilute to the 50 mL mark with deionized water and mix after covering with 5 mL disposable beakers.

f) Ammonium Determination — Remove reagents from the refrigerator and warm to ambient laboratory temperature. Prepare fresh sodium hypochlorite solution. Prepare the colorimeter for use.

i) Pour samples into 5 mL plastic sampler cups, rinsing each cup with a small amount of sample digest before filling.

ii) Attach air lines of the ammonium channel to an air scrubber containing 10 % sulfuric acid (v/v).

iii) Pump deionized water through the manifold tubing (lines) for 5 to 10 minutes. Place all reagent lines in the respective reagent bottles with the exception of the sodium salicylate line.

iv) When the reagents have been pumping for at least 5 minutes, place the salicylate line in its container and allow the system to equilibrate for 10 minutes.

v) After equilibration, place the sample uptake line in deionized water and allow a stable baseline to be established. Load the sampler with reagent blanks, calibration standards, quality control samples, and samples. Initiate the analytical run according to the instrument manufacturer's operating sequence.

vi) Analyze a reagent blank followed by calibration standards; verify acceptable calibration before analyzing samples. Record absorbance at the specified wavelength after steady-state color development is achieved.

Report for Method 4.2-A: Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique

Reporting Units — Total Kjeldahl Nitrogen (TKN, % dry weight basis) or Total Nitrogen (TN, % dry weight basis) where *nitrate reduction* is applied (calculated as % Nitrogen = (mass nitrogen/mass sample) x 100).

Report values to ± 0.1 %.

Lower Reporting Limit — 0.1 %, using a routine dilution factor of 333.3.

Quality control for Method 4.2-A: Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique

See *Appendix 4.2 Nitrogen - Quality Control* for available precision & bias data and method-specific quality control.

4.2-B: Nitrate Nitrogen

Significance and Use for Method 4.2-B: Nitrate Nitrogen

Method 4.2-B: Nitrate Nitrogen. Nitrate nitrogen is a water-soluble, plant-available form of nitrogen that readily leaches from soil and compost. The U.S. EPA considers nitrate to be a potential pollutant in groundwater, surface water, and streams.

Nitrate nitrogen is used for determining the ammonia to nitrate ratio, an indicator of compost stability maturity. Low nitrate levels in conjunction with high ammonium concentrations in compost indicate instability and potentially high microbial activity that may cause nitrogen deficiency in plants when applied to a nitrogen deficient soil.

Summary of Method 4.2-B: Nitrate Nitrogen

Nitrate nitrogen is determined by ion chromatography or colorimetry on compost:water extracts. It is based on Mulvaney (1996); U.S. EPA (1978, 1993, 2007) and uses a recommended 1:5 solid:liquid slurry following *TMECC Method 4.12-F 1:5 Extraction Solution Procedure* on moist, as received samples (40 to 60 % moisture, wet weight basis [ww]).

Nitrate by Ion Chromatography — Nitrate is separated by ion chromatography and detected by suppressed conductivity or ultra violet (UV) absorbance. Nitrate exhibits strong UV absorbance in the lower UV range [200 to 220 nanometer (nm)] when UV detection is used. Refer to instrument manual for specific detection method and wavelength setting.

Nitrate by Colorimetry — Nitrate in compost is determined by reducing nitrate to nitrite in a copperized cadmium reductor or hydrazine sulfate reductor. The nitrite ion then reacts with sulfanilamide under acidic conditions to form a diazo compound, which is an organic compound with two nitrogen atoms and a neutral charge ($–N_2$). This compound then couples with N-1-naphthyl ethylene diamine dihydrochloride to form a reddish-purple azo dye. The absorbance of nitrate–nitrogen is measured at 540 nm. This wavelength also measures the nitrite–nitrogen form.

Interference and Limitations for Method 4.2-B: Nitrate Nitrogen

Nitrate Nitrogen by Ion Chromatography — Chloride and sulfate at high concentrations can interfere with nitrate detection. Sample dilution reduces these interferences but may compromise detection limits. Measurements are sensitive to temperature fluctuations so temperature compensation is necessary.

Nitrate Nitrogen by Colorimetry — Sample extract must be clear for accurate measurements. Suspended particles cloud a sample and inflate nitrate readings. Samples can be filtered through a 0.45-micron filter to reduce turbidity. Colored sample extracts will inflate nitrate readings if proper adjustments are not made. If a sample is yellow, brown or anything other than clear, results will not be reliable. This may be addressed by using in-line dialysis (semi-permeable membrane module that continuously separates dissolved analytes from particulates and high-molecular-weight matrix components prior to color development). In-line dialysis also extends the life of cadmium columns, thereby reducing hazardous waste.

Sample Handling for Method 4.2-B: Nitrate Nitrogen

For sample handling requirements see Table 4.2-1. Nitrate is chemically unstable and undergoes rapid transformation through drying and microbial processes such as nitrification, ammonification, and gaseous losses such as denitrification after sample collection. Microbial activity is minimized when compost samples are refrigerated at temperatures ≤ 6 °C (typically 4 °C). Samples should be transported at ≤ 6 °C (typically 4 °C), and prepared for analysis at as-received moisture updon receipt. Analysis within 7 days is recommended. Freezing discouraged; if unavoidable, store at -18 °C; analyze immediately after thawing. Extracts shall be collected in plastic (P) or fluoropolymer (FP) containers. Extracts shall be refrigerated at ≤ 6 °C and analyzed within 48 hours of preparation. For longer storage, extracts may be preserved by acidification to pH < 2 with sulfuric acid (H_2SO_4) and stored for up to 28 days. The extract must be re-neutralized prior to subsequent ammonium analysis if *Method 4.2-C: Ammonium Nitrogen* will be performed on the same extract.

Total solids and moisture shall be determined on a parallel aliquot for dry-weight reporting.

Recommended Apparatus for Method 4.2-B: Nitrate Nitrogen

Extraction apparatus:

Extraction Flasks — 250 mL hard plastic or glass capped flasks or 1-L plastic zipper bags.

Shaker — Wrist or table, capable of operating at 180 reciprocations or excursions per minute.

Centrifuge Extraction Apparatus — 200 mL centrifuge tubes, capable of withstanding a relative centrifugal force (RCF) of 8000 x g. (i.e., 8000 times the force of gravity). See *TMECC Method 4.12-F: 1:5 Extraction Solution Procedure* for centrifugation procedure and RCF to revolutions per minute (RPM) calculations.

Detection Apparatus

Either:

Ion Chromatograph — With anion column.

Or

Colorimeter — *C*onfigured with nitrate channels fitted with 540 nm wavelength filter for nitrate/nitrite.

Colorimetric nitrate determination requires a *cadmium reduction system* integrated into a continuous-flow analyzer. Either a *granular cadmium reduction column* or a *tubular cadmium reactor* may be used, provided reduction efficiency requirements are met. *Granular cadmium columns* are commonly buffered using an *ammonium chloride–EDTA solution* consistent with U.S. EPA Method 353.2 and are used in many contemporary segmented-flow analyzers (e.g., systems manufactured by SEAL Analytical or Skalar). *Tubular cadmium reactors* have been used in some continuous-flow analyzer configurations with *imidazole-based buffers* (e.g., earlier Alpkem RFA systems). When buffer chemistry and reactor geometry differ from the EPA 353.2 configuration, nitrate-to-nitrite reduction efficiency shall be verified at the start of each analytical run and whenever reagents or reduction columns are changed

Recommended Reagents and Materials for Method 4.2-B: Nitrate Nitrogen

Option 1: Ion Chromatography

Extracting Solution (H_2O) — Deionized water, 17 MΩ·cm, ammonia-free.

Option 2a: Colorimetry -Cadmium Reduction, Segmented Continuous-Flow Analyzer

Reagents (All Colorimetric Configurations)

Potassium Chloride Extracting Solution (2 M KCl) — Dissolve 1491 g reagent grade KCl in 10 L of 17 MΩ deionized water.

Nitrate Calibration Solution Standard — 1000 mg/L nitrate (NO_3^-; 225.8 mg/L nitrate-nitrogen NO_3^-–N). Weigh 1.6305 g of oven-dry (105 °C) potassium nitrate (KNO_3) into a 1 L volumetric flask. Make up to volume with 2 M KCl extracting solution.

Working Standards: 5.0 to 200 mg/L NO_3^- (1.13 to 45.2 mg/L NO_3^-–N) in solution. For example, a sample with 50 % moisture content (ww) is equivalent to 12.4 to 497 mg/kg NO_3^-–N on a dry weight basis. Prepare fresh daily or store at 2–6 °C for no more than 48 hours.

Copper Sulfate Stock Solution (0.01 M $CuSO_4$) — Dissolve 2.497 g $CuSO_4 \cdot 5H_2O$ (pentahydrate) or 1.596 g $CuSO_4$ (anhydrous) in 800 mL deionized water. Add more water to make a final volume of 1 L.

Cadmium Reduction System Select one configuration, either (i) or (ii)

Either:

Configuration (i) — Granular Cadmium Reduction Column, Ammonium Chloride–EDTA Buffer

Ammonium Chloride–EDTA Buffer — Dissolve 100 g ammonium chloride (NH_4Cl) and 10 g EDTA (disodium salt) in approximately 800 mL deionized water. Adjust to pH 8.5 with ammonium hydroxide (NH_4OH) and dilute to 1.0 L.

Storage: Store at room temperature in a sealed plastic container for up to 1 month.

Cadmium Reduction Column (Granular) — Packed cadmium or copper–cadmium granules to the required column length (e.g., 18–20 cm), fitted with a glass wool plug at the outlet.

Column Conditioning - Condition the column using the selected buffer and dilute copper sulfate solution in accordance with the analyzer manufacturer's instructions.

Storage: When not in use, store the column fully wetted with buffer, sealed to prevent air entry, for up to 1 week unless otherwise specified by the manufacturer.

Or:

Configuration (ii) — Tubular Cadmium Reactor, Imidazole Buffer

Stock Imidazole Buffer Solution (0.1 M Imidazole) — Dissolve 6.808 g imidazole ($C_3H_4N_2$) in 800 mL deionized water. Adjust to pH 7.5 with concentrated hydrochloric acid (HCl) and dilute to 1 L.

Storage: Store at room temperature for up to 1 month.

Working Imidazole Buffer Solution (0.05 M Imidazole) — Add 625 µL of 0.01 M $CuSO_4$ stock solution to 125 mL of 0.1 M imidazole buffer, dilute to 250 mL with deionized water, and add 0.25 mL Brij-35 (30 %).

Storage: Prepare fresh daily.

Cadmium Reactor (Tubular) — Tubular or coil-type cadmium reactor installed according to the analyzer manifold configuration.

Reactor Conditioning — Activate the reactor with buffered copper sulfate solution following manufacturer guidance, avoiding air entrapment.

Storage: Store the reactor filled with imidazole buffer and sealed when not in use; recondition if performance degrades or after extended storage.

Option 2b: Colorimetry -Hydrazine reduction, Segmented Continuous-Flow Analyzer

Potassium Chloride Extracting Solution (2 M KCl) — Dissolve 1491 g reagent grade KCl in 10 L of 17 MΩ deionized water.

Nitrate Calibration Solution Standard — 1000 mg/L nitrate (NO_3^-; 225.8 mg/L nitrate-N, NO_3^-–N). Weigh 1.6305 g of oven-dry (105 °C) potassium nitrate (KNO_3) into a 1 L volumetric flask. Make up to volume with 2 M KCl extracting solution.

Working Standards: 5.0 to 200 mg/L NO_3^- (1.13 to 45.2 mg/L NO_3^-–N) in solution. For example, a sample with 50 % moisture content (wet weight) is equivalent to 12.4 to 497 mg/kg NO_3^-–N on a dry weight basis. *Storage:* Prepare fresh daily or store at 2–6 °C for no more than 48 hours.

Color Developing/Reducing Reagents — Dissolve 10 g sulfanilamide ($C_6H_8N_2O_2S$) in 500 mL distilled water containing 200 mL concentrated phosphoric acid (H_3PO_4). Add 0.8 g N-(1-naphthyl)ethylenediamine dihydrochloride ($C_{12}H_{14}N_2 \cdot 2HCl$). Dilute to 1 L with distilled water. Store in a dark bottle at 4 °C. Storage: Store in an amber or dark bottle at 4 °C for up to 1 month.

Copper Sulfate Stock Solution (0.01 M $CuSO_4$) — Dissolve 2.497 g $CuSO_4 \cdot 5H_2O$ (pentahydrate) or 1.596 g $CuSO_4$ (anhydrous) in 800 mL deionized water. Add more deionized water to make a final volume of 1 L.

Sodium Hydroxide Stock Solution (10 M NaOH) — Dissolve 400 g NaOH in 750 mL distilled water, cool, then dilute to 1 L.

Hydrazine Sulfate (0.21 M $N_2H_6SO_4$) Stock Solution — Dissolve 27.5 g hydrazine sulfate ($N_2H_6SO_4$) in 900 mL water, dilute to 1 L. Stable for approximately 6 months. CAUTION: hydrazine sulfate is a hazardous material; adhere to the chemical safety data sheet for appropriate precautions. Dilute stock to working strength immediately prior to use per manifold instructions.

Recommended Procedure for Method 4.2-B: Nitrate Nitrogen

Sample Preparation and Extraction

a) Determine total solids ratio and moisture content on a parallel aliquot of the test sample. Measure and record the as-received tare weight on a parallel aliquot. Determine the dry-weight–equivalent aliquot size in accordance with TMECC Method 3.9-B: Total Solids and Moisture at 105 °C.

b) Test sample: Transfer a representative dry-weight–equivalent aliquot of as-received moist compost into an appropriate sample container (e.g., 250 mL capped flask). Prepare a 1:5 solid-to-liquid slurry following TMECC Method 4.12-F: 1:5 Extraction Solution Procedure.

Example: Add 40 g compost (dry-weight equivalent) to the flask and bring to a total volume of 200 mL by adding the deficit volume of deionized water to the moist sample. Shake thoroughly and extract the free liquid fraction by centrifugation at 8000 × g as specified in Method 4.12-F.

For extract storage and preservation requirements, refer to Table 4.2-1.

Nitrate Determination:

Determine nitrate concentration in the centrifugate as nitrate (NO_3^-) or as nitrate nitrogen (NO_3^-–N) using Option 1 (Ion Chromatography) or Option 2 (Colorimetry):

Either:

Option 1 — Ion Chromatography

Follow instrument manufacturer instructions for system setup, calibration, and sample analysis. In summary, inject the sample extract into the chromatographic system, where nitrate is separated from other anions and detected by suppressed conductivity or UV absorbance (typically 200–220 nm), depending on instrument configuration. Determine concentrations by comparing sample peak areas or heights to calibration standards prepared from potassium nitrate solutions. Control temperature effects and ionic interferences through appropriate calibration and, if necessary, sample dilution. Calculate nitrate or nitrate-nitrogen concentrations from the calibration curve on a dry-weight basis, accounting for the 1:5 extraction ratio (see below *Calculations and Corrections for Method 4.2-B: Nitrate Nitrogen*).

Or:

Option 2 — Colorimetry

Colorimetric nitrate determination may be performed using either a batch spectrophotometer or a segmented continuous-flow analyzer, following instrument manufacturer instructions. In summary, the sample extract is passed through a cadmium reduction system, where nitrate is reduced to nitrite. The nitrite is diazotized with sulfanilamide under acidic conditions and coupled with N-(1-naphthyl)ethylenediamine dihydrochloride to form a reddish-purple azo dye. Absorbance is measured at 529–540 nm, depending on instrument configuration.

Select the appropriate configuration below. For both configurations, establish calibration using nitrate standards prepared in the appropriate extracting solution. Determine nitrate or nitrate-nitrogen concentrations in samples by comparison to the calibration curve.

Either:

Option 2a — Colorimetry (Cadmium Reduction, Segmented Continuous-Flow Analyzer)

Either:

Configuration (i) — Granular Cadmium Reduction Column (Ammonium Chloride–EDTA Buffer). Operate the segmented continuous-flow analyzer using a granular cadmium reduction column buffered with ammonium chloride–EDTA solution. Set up the analyzer manifold, reagents, and detector in accordance with manufacturer instructions and the reagent specifications provided for Configuration (i).

Or:

Configuration (ii) — Tubular Cadmium Reactor (Imidazole Buffer). Operate the segmented continuous-flow analyzer using a tubular cadmium reactor buffered with imidazole solution. Set up the analyzer

manifold, reagents, and detector in accordance with manufacturer instructions and the reagent specifications provided for Configuration (ii).

Or:

Option 2b — Colorimetry (Hydrazine Reduction)

a) Perform nitrate determination using hydrazine reduction according to instrument manufacturer instructions and the reagent specifications provided for Option 2b. In this method, nitrate is chemically reduced to nitrite using hydrazine sulfate under alkaline conditions, followed by color development using sulfanilamide and N-(1-naphthyl)ethylenediamine dihydrochloride.
b) Measure absorbance at the wavelength specified for the instrument configuration.
c) Determine nitrate or nitrate-nitrogen concentrations by comparison to a calibration curve prepared from nitrate standards.

Reduction Efficiency Verification (Colorimetric Methods)

Verify nitrate-to-nitrite reduction efficiency at the start of each analytical run and whenever a new reduction reagent, coil, or column is prepared. Analyze a nitrate standard and a nitrite standard at the same concentration. After reduction, their absorbances shall agree within ±10 %. If the nitrate standard yields a lower absorbance than the nitrite standard, the reduction system is not fully functional and shall be regenerated, reconditioned, or replaced before sample analysis resumes.

Calculations and Corrections for Method 4.2-B: Nitrate Nitrogen

Calculate the concentration of nitrate expressed as nitrate nitrogen (NO_3^-–N) in milligrams of nitrogen per kilogram of compost on a dry-weight basis. The appropriate equation depends on whether the instrument reports nitrate as the ion NO_3^- or directly as nitrate nitrogen (NO_3^-–N) in the 1:5 compost extract.

If the instrument reports nitrate as the full ion (NO_3^-, mg/L) in a 1:5 extract: Convert the measured nitrate (NO_3^-) to nitrate nitrogen (NO_3^-–N) by dividing by 4.429, then multiply by 5 to account for the 1:5 extraction ratio (Eqn. 4.2-B1). The result is the nitrate nitrogen concentration expressed as mg NO_3^-–N per kg compost (dry weight).

$$(NO_3^-\text{–N}) = ([NO_3^-]/4.429)*5 \qquad \textit{Equation 4.2-B1}$$

where

(NO_3^-–N) = mg of nitrate nitrogen per kg of compost (mg/kg),
[NO_3^-] = measured nitrate concentration (mg/L),
4.429 = factor converting nitrate (NO_3^-) to nitrate nitrogen (NO_3^-–N),
5 = conversion factor for 1:5 dilution.

If the instrument reports nitrate directly as (NO_3^-–N, mg/L) in a 1:5 solution extract: No conversion is necessary. Multiply the measured value by 5 to adjust for the 1:5 extraction ratio. The result is the nitrate nitrogen concentration expressed as mg NO_3^-–N per kg of dry compost.

(NO_3^-–N) = [NO_3^-–N] *5 *Equation 4.2-B2*

where:

(NO_3^-–N) = mg of nitrate nitrogen per kg of compost (mg/kg,
[NO_3^-–N] = measured concentration of nitrate nitrogen (mg/L),
5 = conversion factor for 1:5 dilution.

Report for Method 4.2-B: Nitrate Nitrogen

Reporting Units — mg NO_3^--N per kg of oven dried compost (105 °C).

Technique — Report technique used. Chromatography or Colorimetry -Cd reduction or Colorimetry -Hydrazine reduction.

Lower Reporting Limit — 2 mg kg^{-1} N in compost on an oven-dried basis (105 °C).

Quality control for Method 4.2-B: Nitrate Nitrogen

See *Appendix 4.2 Nitrogen - Quality Control* for available precision & bias data and method-specific quality control.

4.2-C: Ammonium Nitrogen

Significance and Use of Method 4.2-C: Ammonium Nitrogen

Ammonium nitrogen is a water-soluble, plant-available form of nitrogen that can accumulate in compost during active decomposition. Elevated ammonium levels are typically associated with immature or unstable composts and may indicate ongoing microbial activity and incomplete organic matter stabilization.

Ammonium nitrogen is used in combination with nitrate nitrogen to calculate the ammonium-to-nitrate ratio, an important indicator of compost maturity and stability. High ammonium nitrogen levels accompanied by low nitrate concentrations suggest instability and may signal the potential for nitrogen immobilization when compost is applied to soil, resulting in plant nitrogen deficiency.

Summary of Method 4.2-C: Ammonium Nitrogen

Ammonium nitrogen is determined on a 1:5 aqueous extract of compost using one of the following analytical techniques:

Option 1 — Ion-Selective Electrode (ISE)— Ammonium is determined by measuring ammonia gas (NH_3) released from ammonium ions (NH_4^+) under strongly alkaline conditions (pH ≥11) using an ammonia ion-selective electrode with hydrophobic, gas-permeable membrane. Ammonia gas diffuses through this membrane into an internal electrolyte solution, where it generates a voltage signal proportional to the ammonium concentration. This voltage is measured and converted to ammonium nitrogen concentration using a pH meter or ion-selective electrode meter.

Option 2 — Colorimetry (Salicylate Method) — This technique involves a reaction of ammonia with salicylate, nitroprusside and hypochlorite in a buffered alkaline medium. The absorbance of the resulting emerald green complex is measured at 660 nm. Ammonia salts are water soluble; ammonium is generally held by ion exchange and requires a high concentration of salt for its complete removal. Potassium chloride solution (2M KCl) is used to exchange the ammonium ion with the potassium ion displacing NH_4^+ from binding sites into solution where it can be measured by the colorimetric reagents.

Option 3—Proposed Additional Method

Ammonium nitrogen may also be determined by automated continuous-flow diffusion conductivity (e.g., Timberline ammonia method; Standard Methods 4500-NH_3 D), where ammonia diffuses across a membrane and is quantified conductimetrically.

Interference and Limitations of Method 4.2-C: Ammonium Nitrogen

Ion-Selective Electrode — Interferences may arise from substances that either mimic or affect ammonia availability. Volatile amines such as methylamine and ethylamine can diffuse across the electrode membrane and be detected similarly to ammonia, falsely elevating readings. Mercury and other metal ions may form complexes with ammonia under alkaline conditions. This reduces the free ammonia concentration, potentially leading to underestimation of ammonium content. High concentrations of salts or suspended solids may interfere with electrode response.

*Colorimetr*y — The sample extract must be clear and colorless for accurate measurements. Suspended particles cloud a sample and inflate nitrate readings. Colored sample extracts will also inflate nitrate readings. If a sample is yellow, brown or anything other than clear, results will not be reliable.

Sample Handling for Method 4.2-C: Ammonium Nitrogen

For sample handling requirements see Table 4.2-1. Ammonium nitrogen is particularly sensitive to loss through ammonia (NH_3) volatilization and microbial transformation during sample handling and storage. Samples shall be transported and stored in sealed containers to prevent cross-contamination and ammonia exchange with the surrounding environment. Air-drying or storage in open containers is not permitted, as ammonia may be lost through volatilization or

absorbed from adjacent samples, resulting in biased results. Samples shall be refrigerated promptly upon receipt at ≤ 6 °C and analyzed as soon as possible; analysis within 7 days is recommended. Samples shall be analyzed as-received; drying, grinding, sieving, or aggressive mechanical homogenization is not permitted prior to extraction. Gentle manual mixing to achieve representativeness is allowed.

Extract Handling: Extracts shall be collected in plastic (P) or fluoropolymer (FP) containers. Extracts shall be refrigerated at ≤ 6 °C and analyzed within 48 hours of preparation. For longer storage, extracts may be preserved by acidification to pH < 2 with sulfuric acid (H_2SO_4) and stored for up to 28 days. Preserved extracts shall be re-neutralized prior to ammonium determination

Total solids and moisture shall be determined on a parallel aliquot for dry-weight correction.

Recommended Apparatus for Method 4.2-C: Ammonium Nitrogen

Extraction apparatus:

Extraction Flasks — 250 mL hard plastic or glass capped flasks, 1 L plastic zipper bags, etc.

Shaker — Wrist or table, capable of operating at 180 reciprocations or excursions per minute.

Centrifuge Extraction Apparatus — 200 mL centrifuge tubes, capable of withstanding a relative centrifugal force (RCF) of 8000 x g (i.e., 8000 times the force of gravity). See *TMECC Method 4.12-F 1:5 Extraction Solution Procedure* for centrifugation procedure and RCF to revolutions per minute calculations.

Detection Apparatus:

Option 1: Ion-Selective Electrode System

a) *Ammonia Ion-Selective Electrode*
b) *Electrometer or pH Meter, Resolution: 0.1 mV, measurement range: –700 mV to +700 mV (or equivalent)*
c) *Magnetic Stirrer with TFE-coated stirring bar*

Option 2: Colorimeter System

Colorimeter — configured with an ammonia channel fitted with wavelength filter for ammonium, 660 nm.

Recommended Reagents and Materials for Method 4.2-C: Ammonium Nitrogen

Option 1: Ion-Selective Electrode System

Extracting Solution (H_2O) — Deionized water, 17 MΩ·cm, ammonia-free.

Option 2: Colorimeter System

Potassium Chloride Extracting Solution (2 M KCl) — Dissolve 1491 g reagent grade KCl in 10 L of 17 MΩ deionized water.

Sodium Hydroxide Stock Solution (5 M NaOH) — Dissolve 200 g NaOH in 700 mL deionized water. Cool and dilute to 1 L. WARNING: This dilution creates excess heat. Add the NaOH cautiously with frequent stirring.

Sodium Potassium Tartrate (0.7 M $KNaC_4H_4O_6 \cdot 4H_2O$) Stock Solution — Dissolve 200 g $KNaC_4H_4O_6 \cdot 4H_2O$ in 700 mL deionized water. Dilute to 1 L.

Sodium Phosphate–Sodium Hydroxide Stock Buffer Solution (0.94 M Na_2HPO_4–0.5 M NaOH) — Dissolve 134 g dibasic sodium phosphate (Na_2HPO_4) in approximately 500 mL deionized water in a 1 L volumetric flask. Add 100 mL stock *Sodium Hydroxide Stock Solution (5 M NaOH)* to the 0.94 M Na_2HPO_4 solution. Mix and dilute to 1 L with deionized water.

Working Buffer Solution — Under continuous stirring, add 62.5 mL *Sodium Potassium Tartrate (0.7 M $KNaC_4H_4O_6 \cdot 4H_2O$) Stock Solution* to 50 mL *Sodium Phosphate–Sodium Hydroxide Stock Buffer Solution* in a 250 mL volumetric flask. Add 7.5 mL *Sodium Hydroxide Stock Solution (5 M NaOH)* while stirring. Dilute to 250 mL with deionized water and mix well. Add 0.25 mL of Brij-35 (30 %) and mix very gently so as not to create foam. Prepare solution weekly.

Sodium Salicylate and Sodium Nitroferricyanide Solution ($NaC_7H_5O_3$ and $Na_2Fe(CN)_5NO \cdot 2H_2O$) — Dissolve 75 g of sodium salicylate and 0.15 g of sodium nitroferricyanide in 400 mL of deionized water, mix and dilute to 500 mL. Filter the solution through No. 2 filter paper into a 500 mL brown bottle.

Sodium Hypochlorite Solution (NaOCl) — Dilute 6 mL 5.25 % NaOCl solution to 100 mL with deionized water. Prepare weekly.

Ammonium Chloride Stock Standard Solution (200 mg/L NH_4^+) — Dissolve 296.9 mg dried (105 °C) NH_4Cl in 500 mL deionized water. This provides 200 mg/L NH_4^+ (156 mg/L NH_4^+–N).

Ammonium Chloride Working Standard Solutions (1.0–15 mg/L NH_4^+) — Dilute *Ammonium Chloride Stock Standard Solution* to prepare standards in the range of 1.0 to 15 mg/L NH_4^+ (0.78 to 11.7 mg/L NH_4^+–N). For samples with 50 % moisture content, this range is equivalent to 8.58 to 128.7 mg/kg NH_4^+–N on a dry weight basis.

Recommended Procedure for Method 4.2-C: Ammonium Nitrogen

Sample Preparation and Extraction

a) Determine total solids ratio and moisture content on a parallel aliquot of the test sample. Measure and record the as-received tare weight on a parallel aliquot. Determine the dry-weight–equivalent aliquot size in accordance with TMECC Method 3.9-B: Total Solids and Moisture at 105 °C.

b) Test sample: Transfer a representative dry-weight–equivalent aliquot of as-received moist compost into an appropriate sample container, e.g., 250 mL capped flask and create a 1:5 solid:liquid slurry using *TMECC Method 4.12-F: 1:5 Extraction Solution Procedure.* For example, add 40 g of compost (dry weight equivalent) to a flask. Fill the flask to a volume of 200 mL by adding the deficit volume of *Potassium Chloride Extracting Solution (2 M KCl)* to the as-received moist compost aliquot and extract the free liquid fraction of the shaken slurry using centrifugation as outlined in *Method 4.12-F: 1:5 Extraction Solution Procedure* using a relative centrifugal force (RCF) of 8000 x g. (i.e., 8000 times the force of gravity). For extract storage and preservation requirements, see Table 4.2-1.

Ammonium Nitrogen Determination:

Determine ammonium nitrogen concentration in the extract using:

Either:

Option 1 — Ion-Selective Electrode (ISE)

Analyze extracts using an ammonia ion-selective electrode system in accordance with the instrument manufacturer's instructions.

In summary:

a) Introduce the sample extract into the analytical system.

b) Adjust the sample to pH ≥ 11 to convert ammonium (NH_4^+) to ammonia (NH_3).

c) Measure electrode potential under constant stirring using an ammonia ion-selective electrode.

d) Relate the measured potential to ammonium concentration using a calibration curve prepared from ammonium standards. Dilute samples as necessary to remain within the calibration range.

e) Determine ammonium or ammonium-nitrogen concentration by comparison to the calibration curve and report results on a dry-weight basis, accounting for the 1:5 extraction ratio.

Or:

Option 2 — Colorimetry (Salicylate Method)

Analyze extracts using either a batch spectrophotometer or a segmented continuous-flow analyzer in accordance with instrument manufacturer instructions.

In summary:

a) Introduce the sample extract into the analytical system.

b) Convert ammonium (NH_4^+) to ammonia (NH_3) under alkaline conditions.

c) React ammonia with salicylate, nitroprusside, and hypochlorite to form a colored complex.

d) Measure absorbance at 660 nm after color development.

e) Relate the measured absorbance to ammonium concentration using a calibration curve prepared from ammonium chloride standards.

f) Determine ammonium or ammonium-nitrogen concentration by comparison to the calibration curve and report results on a dry-weight basis, accounting for the 1:5 extraction ratio. Dilute samples as necessary to remain within the calibration range.

Calculations and Corrections for Method 4.2-C: Ammonium Nitrogen

Calculate the concentration of ammonium expressed as ammonium nitrogen (NH_4^+–N) in milligrams of nitrogen per kilogram of compost on a dry-weight basis. The appropriate equation depends on whether the instrument reports ammonium as the ion NH_4^+ or directly as ammonium nitrogen (NH_4^+–N) in the 1:5 compost extract.

Either:

If the instrument reports ammonium as the full ion (NH_4^+, mg/L) in a 1:5 extract: convert the measured ammonium (NH_4^+) to ammonium nitrogen (NH_4^+–N) by dividing by 1.288, then multiply by 5 to account for the 1:5 extraction ratio (Eqn. 4.2-C3). The result is the ammonium nitrogen concentration expressed as mg NH_4^+–N per kg compost (dry weight).

$$(NH_4^+–N) = ([NH_4^+]/1.288) \times 5 \qquad \textit{Equation 4.2-C3}$$

Where:

(NH_4^+–N) = mg of ammonium nitrogen per kg of compost (mg/kg),
[NH_4^+] = measured ammonium concentration (mg/L),
1.288 = factor converting ammonium (NH_4^+) to ammonium nitrogen (NH_4^+–N),
5 = conversion factor for 1:5 dilution.

Or:
If the instrument reports ammonium directly as (NH_4^+–N, mg/L) in a 1:5 solution extract: no conversion is necessary. Multiply the measured value by 5 to adjust for the 1:5 extraction ratio. The result is the ammonium nitrogen concentration expressed as mg NH_4^+–N per kg of dry compost.

$$(NH_4^+–N) = [NH_4^+–N] \times 5 \qquad \textit{Equation 4.2-C4}$$

Where:

(NH_4^+–N) = mg of ammonium nitrogen per kg of compost (mg/kg),
[NH_4^+–N] = measured concentration of ammonium nitrogen (mg/L),
5 = conversion factor for 1:5 dilution.

Report for Method 4.2-C: Ammonium Nitrogen

Reporting Units — NH_4^+–N mg/kg compost on an oven dried basis (105 °C). Report values to ± 0.1 mg/kg.

Technique — Report methods selected for determining ammonium nitrogen, either Ion-Selective Electrode or Colorimetry.

Lower Reporting Limit — 2 mg/ kg N in compost on a dry weight basis (105 °C).

Quality control for Method 4.2-C: Ammonium Nitrogen

See *Appendix 4.2 Nitrogen - Quality Control* for available precision & bias data and method-specific quality control.

4.2-D: Total Nitrogen by Automated Combustion (Dumas Method)

Summary of Method 4.2-D: Total Nitrogen by Automated Combustion (Dumas Method)

The dry combustion method determines total nitrogen in a sample by oxidizing all nitrogen-containing compounds to elemental nitrogen gas (N_2 gas) using high-temperature combustion. It measures nitrogen in all forms: organic nitrogen, ammonium (NH_4^+), nitrate (NO_3^-), nitrite (NO_2^-), and other reduced or oxidized nitrogen species.

A sample is combusted at high temperature (> 600 °C) in an oxygen-rich atmosphere with the aid of a catalyst. All nitrogen compounds are converted to nitrogen oxides, which are then reduced over heated copper to elemental nitrogen (N_2). The resulting gases passed through absorbents that remove carbon dioxide and water, leaving purified nitrogen. Nitrogen quantification is done by thermal conductivity detection or another gas analysis system that quantifies nitrogen.

Modern automated systems integrate combustion, gas purification, and elemental nitrogen detection in a single instrument. The classical Dumas (1831) method used a nitrometer and volumetric measurement.

Method 4.2-D: Total Nitrogen by Automated Combustion (Dumas Method) is based on the Kellogg Soil Survey Laboratory *Method 4H2a2* (Soil Survey Staff, 2022) and Recommended Methods of Manure Analysis *Method M-3.1 Nitrogen, Total by Combustion* (Hicks et al. 2022a).

Significance and Use of Method 4.2-D: Total Nitrogen by Automated Combustion (Dumas Method)

Instruments designed for simultaneous carbon, nitrogen and/or sulfur analyses allow simultaneous quantification of CO_2, N_2, and SO_2 from a single combustion. This provides accurate determination of total nitrogen (TN) in organic materials, as well as the carbon and sulfur content. See also *TMECC Methods X.X: Total Carbon by Combustion* and X.X: *Total Sulfur by Combustion.*

Interference and Limitations of Method 4.2-D: Total Nitrogen by Automated Combustion (Dumas Method)

High-temperature combustion measures total nitrogen and does not distinguish between organic, ammonium, and oxidized nitrogen species.

Denitrification of nitrate in stored samples or ammonia loss through volatilization during air-drying may result in a significantly lower total nitrogen determination. Incomplete recovery

of fixed ammonium nitrogen may occur in some materials with high levels of fixed nitrogen but in most typical samples, the remaining unrecovered fraction is minimal and does not significantly affect total nitrogen determination.

The method detection limit (MDL) for combustion analysis is higher than the Kjeldahl method (Hicks et. al., 2022), but since compost nitrogen concentrations are typically well above the MDL threshold, this is not a limitation for compost analyses.

Sample Handling for Method 4.2-D: Total Nitrogen by Automated Combustion (Dumas Method)

Nitrogen in compost and manure is chemically and biologically labile and may be lost during storage through ammonia (NH_3) volatilization and microbial transformation. Soil combustion methods commonly allow air-drying or oven-drying at ≤60 °C and fine grinding prior to analysis (for example, Robertson & VanderWulp, 2023). However, drying manure has been shown to increase ammonium nitrogen loss (Li et al., 2020; Hicks et al., 2022b). Therefore, *TMECC Method 4.2-D* follows manure-based guidance rather than soil combustion protocols for sample handling. This approach limits ammonia loss and nitrogen transformations across feedstocks that may include manure.

Samples shall be refrigerated promptly upon receipt at ≤ 6 °C (typically 4 °C) and analyzed as soon as possible. Analysis within 7 days is recommended. Samples shall be analyzed as-received. Air-drying, oven-drying, grinding, sieving, or aggressive mechanical homogenization are not recommended prior to combustion, as these may cause ammonia loss and bias total nitrogen results. Gentle manual mixing sufficient to obtain a representative test portion is permitted. Freezing is not recommended; if unavoidable, samples shall be thawed under refrigerated conditions and analyzed immediately.

Total solids and moisture shall be determined on a parallel aliquot for dry-weight correction of reported total nitrogen results.

Recommended Apparatus for Method 4.2-D: Total Nitrogen by Automated Combustion (Dumas Method)

a) Analytical balance, 0.0001 g precision.
b) Sample delivery vessels: stainless-steel crucibles, ceramic boats, or tin capsules.
c) Elemental combustion analyzer.

Recommended Reagents and Materials for Method 4.2-D: Total Nitrogen by Automated Combustion (Dumas Method

a) Consult instrument-specific SOP for reagents (catalysts, carrier gases).
b) Certified Reference Materials e.g. acetanilide (10.36 % nitrogen), glutamic acid (9.52 % nitrogen), EDTA (9.59 % nitrogen).
c) Calibration standard e.g. Sulfanilic acid calibration standard (41.6 % C, 4.1 % H, 8.1 % N, 27.7 % O, 18.5 % S).
d) Column packing materials as required (Ascarite/Sicapent, copper oxide, copper wire, platinum, silver or quartz wool, tungsten, zinc, cerium dioxide (1–2 mm)or corundum balls (3–5 mm).

Recommended Procedure for Method 4.2-D: Total Nitrogen by Automated Combustion (Dumas Method)

a) Weigh the sample to the nearest 0.0001 g and place it in a crucible, boat, or capsule. For compost and woodchips, typical sample sizes are 0.1 - 0.5 g.
b) Record the weight and sample ID in the instrument software.
c) Follow the manufacturer's procedures for packing combustion and reduction columns (checks, calibration, preparing the instrument for analysis).
d) Analyze samples and collect data automatically using the instrument software. See *Quality control for Method 4.2-D: Total Nitrogen by Automated Combustion (Dumas Method)* for use of blanks and reference standards.

Report for Method 4.2-D: Total Nitrogen by Automated Combustion (Dumas Method)

Report apparatus used and methods selected for determinations of total nitrogen.
Record and report the moisture and drying temperature.
Reporting Units: % total nitrogen, calculated as % total nitrogen = (mass nitrogen / mass sample) x100).
Report values to ±0.01 %.

Quality control for Method 4.2-D: Total Nitrogen by Automated Combustion (Dumas Method)

See *Appendix 4.2 Nitrogen - Quality Control* for available precision & bias data and method-specific quality control.

4.2-E: Total Nitrogen by Wet Oxidation

Summary of Method 4.2-E: Total Nitrogen by Wet Oxidation

Wet oxidation technique for determining total nitrogen using data obtained in *Method 4.2-A: Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique*, with nitrate either accounted for by the *nitrate reduction* step in *Method 4.2-A,* or by *Method 4.2-B: Nitrate Nitrogen by Colorimetry*.

Significance and Use of Method 4.2-E: Total Nitrogen by Wet Oxidation

The wet oxidation technique avoids the specialized combustion equipment needed for *Method 4.2-D: Total Nitrogen by Automated Combustion (Dumas Method)*, but requires hazardous reagents and longer digestion steps.

Interference and Limitations of Method 4.2-E: Total Nitrogen by Wet Oxidation

See M*ethod 4.2-A: Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique* and *Method 4.2-B: Nitrate Nitrogen by Colorimetry*. Potential interferences include incomplete digestion of resistant organic matter. Complete recovery of fixed ammonium may not occur with some materials containing high levels of fixed nitrogen.

Procedure for Method 4.2-E: Total Nitrogen by Wet Oxidation

Either:

Option 1: Follow *Method 4.2-A: Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique* and include the *nitrate reduction* step outlined in *Procedure for Method 4.2-A: Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique* step b).

Or:

Option 2: Omit *nitrate reduction* step in the *Method 4.2-A: Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique* determination and add %N from *Method 4.2-B: Nitrate Nitrogen by Colorimetry* to the % nitrogen from *Method 4.2-A: Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique .*

Calculation for Method 4.2-E: Total Nitrogen by Wet Oxidation

$TN = \%N(TKN) + \%N(NO_3^-)$ *Equation 4.2-E1*

Where:

TN = total nitrogen (% dry weight basis),

%N(TKN) = percent nitrogen measured by Total Kjeldahl Nitrogen method (% dry weight basis),

$\%N(NO_3^-)$ = percent nitrogen measured as nitrate nitrogen by colorimetry (% dry weight basis).

Report for Method 4.2-E: Total Nitrogen by Wet Oxidation

Reporting Units — Total Nitrogen (% dry weight basis)

Report values to ± 0.1 %.

Quality control for 4.2-E: Total Nitrogen by Wet Oxidation

See *Appendix 4.2 Nitrogen - Quality Control* for available precision & bias data and method-specific quality control.

Appendix 4.2 Nitrogen - Quality Control (QC)

Scope and Applicability

The guidance provided is for laboratory users to ensure that samples they submit, or analyses they perform themselves, produce reliable, defensible data. It is the responsibility of the laboratory to ensure that users receive appropriate instruction in the correct use of each instrument, including any calibration steps required prior to analysis. In cases where user calibration is not appropriate, the laboratory should provide instruments that are already calibrated, verified, and ready for use. Some analyses may fall under regulatory frameworks and compliance obligations may differ by jurisdiction and application. Established regulatory guidance for those contexts should be followed when applicable.

Precision and Bias

Precision describes the variability in repeated measurements, either within a single laboratory (repeatability) or among different laboratories (reproducibility). It does not include variation caused by failure to provide a representative compost sample (Sullivan et al., 2018).

Variability depends partly on the magnitude of analysis values. Lower analysis values generally have less precision (more variability). This is seen in Table 4.2-Ax1, which summarizes typical analytical performance for nitrogen methods used in compost analysis.

Bias describes the systematic difference between the measured value and a known or certified reference value (trueness). Together, precision and bias define analytical accuracy (ISO 5725; ASTM E691). The limits given below are consistent with US EPA SW-846 (2014), and AOAC (2016).

Table 4.2-Ax1 . Method performance summary for nitrogen determinations (Sullivan et al., 2018). Median analysis values are presented with within-laboratory precision (relative median deviation, RMD; n=3) and between-laboratory precision (RMD) on a dry-weight basis.

Method	Name	Median analysis value	Within-lab RMD (n=3)	Number of labs	RMD Between-lab
4.2-A	Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl	1.2 %	11%	12	27%
4.2-B	Nitrate Nitrogen	458 mg/kg	7%	24	12%
4.2-C	Ammonium Nitrogen	287 mg/kg	41%	30	67%
4.2-D	Total Nitrogen by Automated Combustion (Dumas)	1.5%	3%	33	5%

Within-laboratory precision is used for QC acceptance criteria. Between-laboratory precision in Table 4.2-Ax1 is provided for informational background. NOTE: Relative standard deviation (RSD) is used for all QC acceptance criteria and is the conventional precision metric. Relative median deviation (RMD) is a non-parametric measure based on medians, used to summarize historical interlaboratory performance (Table 4.2-Ax1). It is not interchangeable with RSD for routine QC. Current interlaboratory reproducibility expressed as relative standard deviation (%RSD) is summarized in Table 4.2-Ax2 for performance context

Table 4.2-Ax2. Method performance summary for nitrogen determinations from the Compost Analysis Proficiency Program (2026). Data provided by Robert O. Miller, Compost analysis proficiency testing program coordinator, Windsor, CO. Reproducibility is expressed as interlaboratory relative standard deviation (%RSD), calculated from the interlaboratory standard deviation and the median analysis value.

Method	Name	Median analysis value	Intra-lab repeatability (%) (n=3)		Number of labs	Inter-lab reproducibility (%)	
			Repeatability (%RSD)	Stdev		Reproducibility (%RSD)	Stdev
4.2-A	Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl	1.3%	9.4%	3.5%	3	39%	13%
4.2-D	Total Nitrogen by Automated Combustion (Dumas)	1.2%	12.1%	4.2%	3	50%	16%

Acceptance limits

Duplicate Samples — Duplicate acceptance criteria are method-specific and reflect the heterogeneity and chemical instability of compost materials.

RPD = |A-B|×100÷C Eqn 4.2-Ax1

Where:

RPD = Relative Percent Difference,
A = first duplicate,
B = second duplicate,
C = average of duplicates.

Acceptance limits are based on within-laboratory duplicate precision for compost matrices (Table 4.2-Ax1) and the following recommendations are set at approximately two times the typical relative variability. Table 4.2-Ax2 is provided for interlaboratory performance context.

Duplicate samples are recommended to agree within the following relative percent difference (RPD):

Method 4.2-A, Total Kjeldahl Nitrogen (Semi-Micro Kjeldahl Technique): ≤ 20%;
Method 4.2-B, Nitrate Nitrogen (Ion Chromatography or Colorimetric Determination): ≤ 15%;
Method 4.2-C, Ammonium Nitrogen: ≤ 50%;
Method 4.2-D, Total Nitrogen by Automated Combustion (Dumas Method): ≤ 10%.

If variability exceeds the applicable limit, repeat duplication with another test portion from the same batch; lack of precision is generally due to inadequate subsampling or mixing. Note: RPD is concentration-dependent and may exceed the criteria near the limit of quantification (LOQ). When either duplicate result is ≤ ~3× LOQ (or at very low %N), apply laboratory historical acceptance limits by matrix and concentration range, or evaluate precision using an alternate statistic (e.g., RSD on ≥3 replicates)

Reference Sample — The warning limit is the 95 % confidence interval based on the periodically updated cumulative mean and standard deviation of the reference sample. The rejection limit is outside the 99 % confidence interval (± 2.58 SD). If the standard reference result is rejected, repeat the analysis with the next batch of samples, including 1–2 % of the unknowns from the affected batch.

NIST Reference Checks — The warning limit is the 95 % confidence interval published by the National Institute of Standards and Technology (NIST). If results fall outside this range, apply the confidence limits derived from the laboratory's historical performance.

Reagent blanks - The reading should be less than the Limit of Quantitation (LOQ) provided by the equipment manufacturer.

See the method-specific quality control below:

Quality Control for Method 4.2-A:Total Kjeldahl Nitrogen, Semi-Micro Kjeldahl Technique

Reagent Blank: 3 reagent blanks (weighing paper, sulfuric acid, and a catalyst tablet) per batch (batch ≤ 20 samples). See *4.2 Quality Control - Acceptance limits*.

Duplicate: 1 duplicate per batch (batch ≤ 20 samples) to assess precision. See *4.2 Quality Control - Acceptance limits*.

Certified Reference Material (CRM): One CRM (e.g., NIST pine needles or equivalent) per batch to verify accuracy. See *4.2 Quality Control - Acceptance limits*.

In-House Reference Material: When a new in-house reference is introduced, include it with each batch (batch ≤ 20 samples) until a consistent mean and acceptable variability are established. See *4.2 Quality Control - Acceptance limits*.

Precision Target: Typical within-laboratory repeatability is 10-15 % relative mean deviation (see Table 4.2-Ax1).

The editors are seeking bias data for this method. To contribute please email info@compostfoundation.org

Quality Control for Method 4.2-B: Nitrate-Nitrogen

Each analytical run shall include:

Calibration Verification: Analyze a mid-range calibration check standard at the beginning and end of each run; results must be within ± 10 % of the expected value. Verify instrument stability every 10 samples with a calibration standard.

Reagent Blank: Include one per batch (batch ≤ 20 samples). See *4.2 Quality Control - Acceptance limits*.

Duplicate: One duplicate per batch (batch ≤ 20 samples) to assess precision. See *4.2 Quality Control - Acceptance limits*.

Spike or Recovery Check: Include at least one matrix spike per batch, prepared at 3–5 × the method limit of quantitation (LOQ), to verify recovery (target recovery = 100 ± 15 %).

Precision Target: Typical within-laboratory repeatability is5-10% relative mean deviation (RMD, Table 4.2-Ax1).

The editors are seeking bias data for this method. To contribute please email info@compostfoundation.org

Quality Control for Method 4.2-C: Ammonium-Nitrogen

Each analytical run shall include:

Calibration Verification: Analyze a mid-range calibration check standard at the beginning and end of each run; results must be within ± 10 % of the expected value. Verify instrument stability every 10 samples with a calibration standard.

Reagent Blank: Include one per batch (batch ≤ 20 samples). See *4.2 Quality Control - Acceptance limits*.

Duplicate: 1 duplicate per batch (batch ≤ 20 samples) to assess precision. See *4.2 Quality Control - Acceptance limits*.
Spike or Recovery Check: Include at least one matrix spike per batch, prepared at 3–5 × the method limit of quantitation (LOQ), to verify recovery (target recovery = 100 ± 15 %).
Precision Target: Typical within-laboratory repeatability is 35-45 % RMD (see Table 4.2-Ax1), reflecting chemical and biological instability of ammonium in compost matrices.

The editors are seeking bias data for this method. To contribute please email info@compostfoundation.org

Quality Control for Method 4.2-D: Total Nitrogen by Automated Combustion (Dumas Method)

Each analytical run shall include:
Combustion Blank: Analyze an empty capsule to verify baseline stability. See *4.2 Quality Control - Acceptance limits*.
Duplicate: 1 duplicate per batch (batch ≤ 20 samples) to assess precision. See *4.2 Quality Control - Acceptance limits*.
Calibration Verification and Drift Check: Verify instrument calibration at the beginning and end of each batch using certified standards (e.g., acetanilide or EDTA). Calibration verification results must be within ± 10 % of the expected value.
Certified Reference Material (CRM): Include one CRM (e.g., NIST soil or plant tissue) per batch (batch ≤ 20 samples) to verify accuracy.
Precision Target: Typical within-laboratory reproducibility is 3-6 % RMD (see Table 4.2-Ax1).

The editors are seeking precision and bias data for this method. To contribute please email info@compostfoundation.org.

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