

SOP-C-152

**Determination of  
Soil Nitrate/Nitrite as Nitrogen**

Revision 5

Approval:

  
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Laboratory Manager

4-13-23  
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Date

  
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Concurrence

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Initials: \_\_\_\_\_

*Texas Institute for Applied Environmental Research*

## Determination of Soil Nitrate/Nitrite as Nitrogen

- i. **Identification of the method**
  - a. Methods of Soil Analysis, Part 3
- ii. **Applicable matrix or matrices**
  - a. Soils and other solids (not TNI accredited)
- iii. **Limits of detection and quantitation**
  - a. Determined at time of analysis
- iv. **Scope and application, including parameters to be analyzed**
  - a. The purpose of the procedure is to provide a method for the determination of nitrate/nitrite as Nitrogen in soil. This procedure establishes routine guidelines for determination of  $\text{NO}_3\text{-N}$  and  $\text{NO}_2\text{-N}$  to obtain comparable results between analysts.
- v. **Summary of the method**
  - a. Spectrophotometric
- vi. **Definitions**
  - a. Inorganic nitrogen may be found in soils as nitrate nitrogen ( $\text{NO}_3\text{-N}$ ) and nitrite nitrogen ( $\text{NO}_2\text{-N}$ ).
  - b. DI (Deionized) water – Water that has passed through anion and cation exchange resin bed cylinders that remove most ions to achieve low specific conductance and meets Type II ASTM standards of less than  $1.0 \mu\text{mho/cm}$ .
  - c. Gravity filtration – used to remove solid impurities from a liquid or a solution without use of a vacuum.
  - d. Wash Solution (Potassium chloride (KCl)) – Used to wash out sample lines between samples to prevent carryover.
  - e. Extraction solution – may be varying molarities of KCl or DI water, depending on project requirements.
- vii. **Interferences**
  - a. Soil treatments prior to sampling. e.g. recent fertilizer application.
  - b. Cellulose filters may contain significant amounts of  $\text{NH}_4\text{-N}$ , removal by washing the filter with the KCl solution is recommended if the sample is tested for  $\text{NH}_4$ . Also glass fiber filters which do not need to be washed may be used in place of the cellulose filters. Incomplete extraction may occur if the sample is not thoroughly shaken for at least 1 hour.
- viii. **Safety**
  - a. All aspects of this procedure shall comply with QAM-S-101, "Laboratory Safety."

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- b. The analyst should read the SDS for each of the species prior to performing the extraction and analysis of nitrate and nitrite of soils.
  - c. The analyst should wear protective equipment such as eyewear, lab coat, and gloves while performing the analysis operations.
  - d. All waste should be placed into the proper waste receptacle and disposed of in accordance with QAM-W-101, "Disposal of Laboratory Waste."
- ix. Equipment and supplies**
- a. Orbital shaker
  - b. Lachat Quikchem 8000 autoanalyzer or equivalent, see QAM-I-102, "Operation and Calibration of the Autoanalyzer."
  - c. Funnel rack
  - d. Electronic balance with a sensitivity of ~0.01g.
  - e. Long stem funnels with about 60° angles.
  - f. Erlenmeyer flasks, 125 and 50 mL
  - g. Class A volumetric flasks
  - h. 1 mL mechanical pipettor or equivalent
  - i. 5 mL mechanical pipettor or equivalent
  - j. Disposable culture tubes for autosampler (if used).
  - k. Whatman grade 42 filter paper, 9 cm diameter
- x. Reagents and standards**
- a. Reagents (all chemicals are ACs grade or equivalent)
    - i. DI (deionized) water for DI extractant and to make reagents
    - ii. Potassium chloride (2 M KCl) solution – Dissolve 150 g KCl in 800 mL of DI and dilute to 1 liter in a volumetric flask.
  - b. Standards:
    - i. Prepare standards in the same extraction solution used for samples
    - ii. See SOP-C-105, "Determination of Nitrate/ Nitrite as Nitrogen," for preparation of stock solutions for nitrate and nitrite.
    - iii. Prepare calibration and check standards from nitrate and nitrite stock solutions. Prepare the following calibration standards: 0.05, 0.2, 0.5, 1.0 mg/L for NO<sub>2</sub>-N and 0.05 (LOQ), 0.2, 0.5, 1.0, 2.0, 3.0, 4.0 mg/L for NO<sub>3</sub>-N or

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$\text{NO}_2 + \text{NO}_3\text{-N}$ . Standards are good for 48 hours if not preserved.

- xi. Sample collection, preservation, shipment and storage**
- Samples are refrigerated to  $>0\text{-}\leq 6^\circ\text{C}$  after extraction.
  - Samples are not normally collected or shipped by the lab.
  - Holding Time is 180 days
- xii. Quality control**
- All aspects of this procedure comply with QAM-Q-101, "Laboratory Quality Control".
  - All data shall be documented and maintained in accordance with QAM-A-102, "Laboratory Document and Data Control."
  - The analyst should refer to QAM-I-102, "Operation and Calibration of the Autoanalyzer," or the instrument manual if problems occur during instrument operations.
  - The analyst shall be trained prior to performing this procedure on samples.
  - Spike levels and sources used for this procedure may be chosen at the analyst's discretion from calibration standards or stocks depending on sample concentrations. Spikes are analyzed for information only and do not constitute acceptance criteria for method performance.
  - Calibration verification standards should be prepared from a second source.
- xiii. Calibration and standardization**
- Refer to SOP-C-105, "Determination of Nitrate/Nitrite as Nitrogen" for instruction.
- xiv. Procedure**
- Weigh 2.0 g of air-dried, ground and sieved soil into a labeled 125 mL Erlenmeyer flask. Refer to SOP-C-131, "Preparation of Soil Samples" for details on how to prepare soil samples for analysis.
  - Spike with 0.1 mL of 100 ppm standard.

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- c. Place flask on the orbital shaker and shake for 1 hour at about 180 RPM at room temperature. Allow the suspension to settle for 15-30 minutes.
  - d. Set up filtration stands and funnels. Place a fluted Whatman 42 filter in each funnel (or a 0.45µm filter into a filter flask).
  - e. Filters should be washed with 20 mL of extraction solution and 20 mL of DI and air or oven (40-70°C) dried before use. Alternatively, 0.45 µm membrane filters may be used.
  - f. Filter samples into corresponding labeled sample containers (50 mL Erlenmeyer, or plastic bottles).
  - g. Analyze by SOP-C-105, "Determination of Nitrate/Nitrite as Nitrogen."
- xv. Data analysis and calculations;**
- a.  $\text{ppm (mg/Kg dry)} = \frac{\text{(mg/L sample * V)}}{\text{g of soil}}$   
  
V = volume of extractant used
  - b. Report results on a dry weight basis.
- xvi. Method performance**
- a. Refer to QAM-Q-101, "Laboratory Quality Control."
- xvii. Pollution prevention**
- a. Pollution prevention: refer to QAM-W-101, "Disposal of Laboratory Waste"
- xviii. Data assessment and acceptance criteria for quality control measures**
- a. Data assessment and acceptance: refer to QAM-Q-101, "Laboratory Quality Control"
- xix. Corrective actions for out-of-control data**
- a. Corrective action: refer to QAM-Q-105, "Corrective Actions"
- xx. Contingencies for handling out-of-control or unacceptable data**

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- a. Refer to QAM-Q-101, "Laboratory Quality Control" and QAM-Q-105, "Corrective Actions."

**xxi. Waste management**

- a. Waste management: refer to QAM-W-101, "Disposal of Laboratory Waste". No hazardous waste is expected to be generated from this procedure unless from samples.

**xxii. References**

- a. Methods of Soil Analysis, Part 3: Chemical Methods, Soil Science of America, ed. D.L. Sparks, et.al., 1996, pg. 1130-1131.
- b. The National Environmental Laboratory Accreditation Conference Institute (TNI) standard, 2016.

**xxiii. Any tables, diagrams, flowcharts and validation data**

- a. none

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