

SOP-C-163

Determination of Ammonia Nitrogen in Soil

Revision 0

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Laboratory Manager

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JMI

Texas Institute for Applied Environmental Research

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- I. Identification of the method**
 - a. Soil Analysis of Ammonia Nitrogen (Seal Analytical. 2017. AQ2 method. AGR-210-A Rev. 1)

- II. Applicable matrix or matrices**
 - a. Soils and other solids (not TNI accredited)

- III. Range and limits of detection and quantitation**
 - a. Range 0.2 – 10.0 mg N/L
 - b. Estimate of Reporting Limit 0.2 mg N/L
 - c. By USEPA procedure MDL = 0.04 mg N/L

- IV. Scope and application, including parameters to be analyzed**
 - a. The purpose of the procedure is to provide a method for the determination of ammonia nitrogen in soil. This procedure establishes routine guidelines for the collection, preparation, and analysis of soil samples for ammonia nitrogen to obtain comparable results between analysts.

- V. Summary of the method**
 - a. Ammonia displacement from soil cation exchange sites and extraction from soil solution by 2 N KCl.
 - b. Spectrophotometric assessment of the reaction of ammonium with alkaline phenol in the presence of nitroferricyanide followed by a static incubation at 40°C results in the formation of a blue indophenol dye. The absorbance of this complex is measured photometrically at 660 nm.

- VI. Definitions**
 - a. Extraction – Use of a reagent that displaces the nutrient to be analyzed into the solution so that the nutrient can be analyzed.
 - b. Colorimetric analysis – a wet chemistry analysis procedure based on the reaction between chemicals in reagents and the nutrient of interest to produce a colored product that varies in value depending on the concentration of nutrient in the solution.
 - c. DI (Deionized) water – Water that has passed through anion and cation exchange resin bed cylinders that remove most ions to

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achieve low specific conductance and meets Type II ASTM standards of less than 1.0 $\mu\text{mho/cm}$.

d. Refer to QAM-Q-101, 'Laboratory Quality Control.'

VII. Interferences

- a. Dissolved calcium and magnesium may form precipitation under the alkaline reaction conditions to cause positive bias.
- b. Heavy-metal cations may precipitate similarly. Reagent recipes for this method include disodium EDTA to prevent precipitation.
- c. Bias from sample turbidity or color is correctable using sample blanking feature of AQ software.

VIII. Safety

- a. All aspects of this procedure shall comply with QAM-S-101, "Laboratory Safety".
- b. The analyst shall wear proper laboratory attire when conducting this analysis, including safety glasses, gloves, and laboratory coat.
- c. Ammonium chloride (NH_4Cl) is an irritant.
- d. EDTA disodium salt dihydrate ($\text{C}_{10}\text{H}_{14}\text{N}_2\text{Na}_2\text{O}_8 \cdot 2\text{H}_2\text{O}$) causes serious eye irritation, may be harmful if inhaled.
- e. Phenol ($\text{C}_6\text{H}_5\text{OH}$), Sodium hydroxide (NaOH), Sodium hypochlorite (NaClO), and Sulfuric acid (H_2SO_4) are corrosive.
- f. Sodium nitroferricyanide dihydrate ($\text{Na}_2[\text{Fe}(\text{CN})_5\text{NO}] \cdot 2\text{H}_2\text{O}$) and Phenol ($\text{C}_6\text{H}_5\text{OH}$) are toxic.
- g. Phenol ($\text{C}_6\text{H}_5\text{OH}$) poses a health hazard.
- h. Phenol ($\text{C}_6\text{H}_5\text{OH}$) and Sodium hypochlorite (NaClO) have aquatic toxicity.

IX. Equipment and supplies

- a. Ponar dredge
- b. AMS sediment sampler or hammer probe
- c. Plastic or glass bottles
- d. Balance capable of accurately weighing to the nearest 0.01 g.
- e. Weighing pans and spatulas
- f. 125 ml plastic bottles
- g. Shaking stand
- h. Funnels, filter paper, and filter stand
- i. Filtrate collection vials

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- j. Assorted glassware, Class A
- k. SEAL autoanalyzer
- l. Laptop computer with SEAL AQ2 software installed.
- m. SEAL sample cups
- n. SEAL reagent wedges
- o. SEAL reaction segments

X. Reagents and Standards

a. Reagents

- i. 2N KCl extraction solution: To a 1000 mL Erlenmeyer flask add 149.1 g potassium chloride (KCl) (CAS: 7447-40-70) and about 800 mL deionized (DI) water (precautions must be taken to ensure that the water remains ammonia free). Stir to dissolve. Transfer to a 1000 mL volumetric flask, fill to the mark with DI water, and mix.
- ii. EDTA buffer, 75 g/L EDTA, 2 g/L NaOH: To a 500 mL Erlenmeyer flask add 37.5 g disodium ethylenediaminetetraacetic acid (EDTA) dihydrate ($C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O$) (CAS 6381-92-6), 1.0 g sodium hydroxide (NaOH) (CAS 1310-73-2), and about 400 mL DI water. Stir with gentle warming to dissolve, adding DI water as needed. Transfer to a 500 mL volumetric flask, fill to the mark with DI water and mix.
- iii. Alkaline phenate: To a 500 mL Erlenmeyer flask add 10 g NaOH and about 150 mL DI water. Swirl to dissolve and cool to near room temperature. In the fume hood, add 25 g crystalline phenol (C_6H_5OH) (CAS 108-95-2) and swirl to dissolve. Transfer to a 250 mL volumetric flask, fill to the mark with DI water and mix. Allow to stand overnight. This reagent may be stored in the refrigerator at least 1 month or until its color becomes dark brown. Crystalline phenol is preferred to liquefied phenol, 90% w/w.
- iv. Sodium hypochlorite, 5.25% or 6.0% NaOCl (CAS 7681-52-9) (w/w). Use house-brand bleach that does not contain thickeners, fragrances, or additives that has been purchased within 6 months of use. (The concentration of the bleach used will be entered under the "Reagents" tab in the SEAL set up program.) Refrigerate this reagent.

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- v. Nitroferricyanide - Dissolve 1.0 g sodium nitroferricyanide dihydrate ($\text{Na}_2[\text{Fe}(\text{CN})_5\text{NO}] \cdot 2\text{H}_2\text{O}$) (CAS 13755-38-9) in 500 mL DI water. This solution can be stored at least 1 month in a dark bottle. Discard if the reagent becomes bluish in color.
- vi. Sulfuric acid (H_2SO_4) (CAS 7664-93-9), 5 N. Add approximately 60 mL DI water to a beaker. In a fume hood, add 27.2 mL of concentrated sulfuric acid to the beaker. Cool and bring to volume with DI water in a volumetric flask.

b. Standards

- i. Ammonia stock standard solution (1000 mg N/L): Dissolve 3.819 g ammonium chloride, anhydrous (NH_4Cl) (CAS: 12126-02-9) (dried at 105°C) in about 800 mL DI water in a 1 L volumetric flask. Up to 3.0 mL sulfuric acid (H_2SO_4) (CAS 7664-93-9), 5 N, may be added for preservation. Dilute to the mark with DI water. Prepare fresh monthly.
- ii. Ammonia standard solution (10mg N/L) - To a 500 mL volumetric flask, pipet 5.0 mL Ammonia-N stock standard solution (1000 mg N/L) and dilute to the mark with 2N KCl extraction solution and invert to mix. Store at 4°C . Prepare fresh weekly.

XI. **Sample collection, preservation, shipment and storage**

- a. Holding time for air dried soils—180 days
- b. Sediment collection from rivers and bayous will follow sampling methods as described in "Surface Water Quality Monitoring Procedures, Volume 1: Physical and Chemical Monitoring Methods", Chapter 6, "Collecting Sediment Samples."
- c. Collection of sediments from locations with deep water accessible only by a bridge, a Ponar dredge will be used. At other locations, where the stream width is narrow and stream depth is relatively shallow, sediment samples will be taken with an AMS® sediment sampler attached to hammer probe or with a Teflon or Teflon-coated scoop.
- d. Soil samples will be collected using an AMS® hammer probe. Soil samples may be collected at 2 depths: 0-15 cm and 15-30.
- e. Soil samples include replicate samples (usually 5 -7) that are collected into a clean bucket, mixed, and subsamples collected into labeled glass jars or plastic bottles.

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- f. Prior to soil extraction with 2N KCl, samples are dried following SOP-C-132, "Preparation of Soil Samples for Analysis on SEAL AQ300."

XII. **Quality control**

- a. All aspects of this procedure comply with QAM-Q-101, "Laboratory Quality Control".
- b. All data shall be documented and maintained in accordance with QAM-A-102, "Laboratory Document and Data Control".
- c. The analyst should refer to QAM-I-120, "Operation of the SEAL AQ300 Autoanalyzer" or the instrument manual if problems occur during instrument operations.
- d. The analyst shall be trained prior to performing this procedure on samples.

XIII. **Calibration and standardization**

- a. Use calibration standards to establish the standard curve ($R^2 > 0.98$)
- b. Analyze an ICV and IBV immediately after calibration, and a CCV and CCB following every 10 samples, and at the end of the run.

XIV. **Procedure**

- a. Extraction of ammonia nitrogen from soil
 - i. Prepare 2N KCl extraction solution (X.a.i.)
 - ii. Weigh 10.0 g of air-dried, ground, and sieved soil into appropriately labeled 125 mL plastic bottle. Refer to SOP-C-132, "Preparation of Soil Samples for Analysis on SEAL AQ300" for details on how to prepare soil samples of analysis.
 - iii. Add 100 mL of 2 M KCl extraction solution.
 - iv. Shake on a reciprocating shaker at 200 rpm for 60 minutes.
 - v. Filter using Whatman #1, #2, or # 42 filter paper.
 - vi. Store samples in a refrigerator for up to 60 days
- b. Colorimetric analyses of ammonia nitrogen in soil extracts.
 - i. Follow procedures describe in SEAL AQ2 method AGR-210-A Rev. 1 and QAM-I-120, "Operation and Calibration of the SEAL AQ300 Autoanalyzer".

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- ii. A standard curve will be developed automatically by the SEAL AQ300 instrument based on pre-programmed levels of diluting the "standard solution." This standard curve is produced at the beginning of each run.

XV. Data Analysis and calculations

- a. Review the graphic display of the output and identify, correct, or resolve any anomalies by examining any problems with the function of the SEAL AQ300 instrument and ensuring all reagents are properly prepared, not outdated, and installed in proper analysis wedges.
- b. Print a copy of the SEAL data sheet for placement in the laboratory logbook.
- c. All data are documented and maintained in accordance with QAM-A-102, "Document and Data Control".
- d. The baseline is within \pm the LOQ value for the method.
- e. All dilution procedures are described in detail.

XVI. Method performance

- a. Method performance: 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. Refer to QAM-Q-105, "Corrective Actions."

XX. Contingencies for handling out-of-control or unacceptable data

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

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XXI. Waste management

- a. All waste is placed into the proper waste receptacle and disposed of in accordance with QAM-W-101, "Disposal of Laboratory Waste".

XXII. References

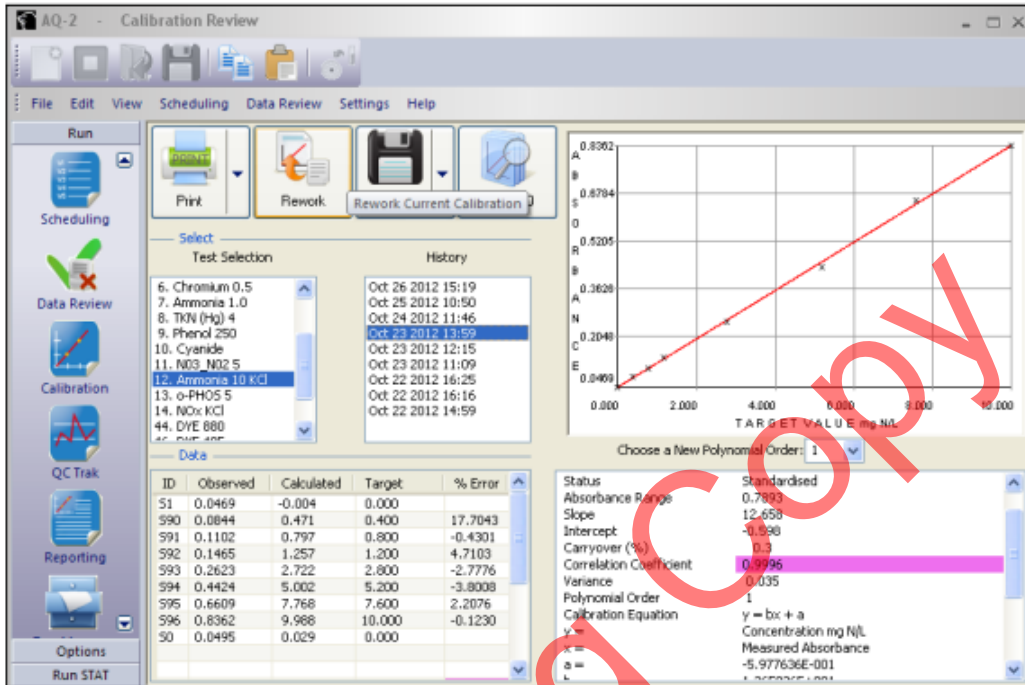
- a. Seal Analytical. 2017. AQ2 method. AGR-210-A Rev. 1
- b. TCEQ. 2020. Surface Water Quality Monitoring Procedures, Volume 1: Physical and Chemical Monitoring Methods. Texas Commission on Environmental Quality.
www.tceq.texas.gov/publications/rg/rg-415.
- c. USEPA. 1993. Methods for the Determination of Inorganic Substances in Environmental Samples, USEPA 600/R 93/100, August 1993: Method 350.1, Revision 2.0

XXIII. Tables, diagrams, flowcharts, and validation data

- a. Example Calibration Curve Table

Working Copy

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Attachment 1
Example of Calibration Curve Table



DEFINITIONS:

Abs. Range: Difference between the absorbance of the highest standard and the absorbance of blank

Variance: Calculated as follows:

$$\text{Variance} = \frac{\sum(\text{Deviations, i.e., errors})^2}{\sum(\text{Observed values})^2} \times 100$$

Carryover: Calculated as follows:

$$\text{Carryover (\%)} = \frac{(A-B)}{C} \times 100\%$$

Where, A = absorbance of carryover blank (S0)

B = absorbance of blank (S1)

C = calibration absorbance range

From: Seal Analytical. 2017. AQ2 method. AGR-210-A Rev. 1