

Scope and Application

This method involves the semiquantitative extraction of ammonium ($\text{NH}_4\text{-N}$) from soils using 2.0N KCl. Ammonium is determined by spectrophotometric, diffusion-conductivity instruments or distillation techniques. The method doesn't quantitatively extract ammonium from mineral structures (i.e. nonexchangeable $\text{NH}_4\text{-N}$) or bound to organic compounds. The method is readily adapted to manual or automated techniques. The procedure outlined follows that outlined by Keeney and Nelson (1982) for determining nitrate nitrogen with a modification in which 25 mL of KCl and 5.0 g of soil are used instead of 100 mL and 10 g soil. Care must be taken to avoid contamination from filter paper and operator handling. Soil ammonium concentrations are generally low in mineral soils ($< 10 \text{ mg kg}^{-1}$). The method detection limit is approximately 0.2 mg kg^{-1} (on a dry soil basis) and is generally reproducible $\pm 7\%$.

Equipment

1. Analytical balance: 100.0 g capacity, resolution $\pm 0.01 \text{ g}$.
2. Repipette dispenser, calibrated to $25.0 \pm 0.2 \text{ mL}$.
3. Reciprocating horizontal mechanical shaker, capable of 180 oscillations per minute (opm).
4. Extraction vessels and associated filtration vessel.
5. Whatman No. 42 or equivalent highly retentive filter paper.
6. Spectrophotometer, or flow injection analyzer (FIA), or distillation instruments.

Reagents

1. Deionized water, ASTM Type I grade.
2. Potassium chloride extracting solution, 2.0N KCl: Dissolve 150 g of reagent grade KCl in 500 mL deionized water and dilute to a 1000 mL (See Comment #1).
3. Standard calibration solutions of $\text{NH}_4\text{-N}$. Prepare six calibration standards ranging from 0.1 to 20.0 mg L^{-1} concentration, diluted in 2.0N KCl extraction solution prepared from $1000 \text{ mg L}^{-1} \text{ NO}_3\text{-N}$ standard solution.

Procedure

1. Weigh $5.0 \pm 0.05 \text{ g}$ of air-dried soil pulverized to pass 10 mesh sieve ($< 2.0 \text{ mm}$) into extraction vessel. Add 25.0 mL of 2.0N KCl extraction reagent using repipette dispenser (See Comment #2). Include a method blank.
2. Place extraction vessel(s) on reciprocating mechanical shaker for thirty (30) minutes.
3. Filter extract (See Comment #3), refilter if filtrate is cloudy (comment #4).
4. Ammonium-N content of the extract is determined using a spectrophotometer, diffusion-conductivity instruments or distillation techniques using standard calibration solutions (See Comment #4 and #5). The ammonium nitrogen content of the digest solution can be determined with a rapid flow analyzer (Technicon Method No. 334-74A/A) or a flow injection analyzer (FIA). This determination can also be made using the Kjeldahl distillation method. Adjust and operate instruments in accordance with manufacturer's instructions. Determine ammonium concentration of a method blank and unknown samples.

Calculation

$$\text{NH}_4\text{-N mg kg}^{-1} \text{ in soil} = (\text{NH}_4\text{-N mg L}^{-1} \text{ in filtrate} - \text{method blank}) \times 5$$

Report soil ammonium concentration to the nearest 0.1 mg kg⁻¹ (See Comment #6)

Comments

1. Soils may be extracted with 2.0 N KCl for the simultaneous determination of nitrate (Method 3.10).
2. Check repipette dispensing volume calibration using an analytical balance.
3. Check filter paper supply for possible contamination of and NH₄-N. If contamination is greater than 0.2 mg L⁻¹ on a solution basis, rinse filter paper with 2.0 N KCl.
4. It is recommended that soils extracted for ammonium be analyzed within two (2) hours after extraction.
5. Samples having ammonium concentrations exceeding the highest standard will require dilution and reanalysis.
6. Ammonium-nitrogen (NH₄-N) results can be expressed on a volume basis. Assuming the sample represents a 0-6 inch (0-15 cm) depth of the soil, then: NH₄-N mg kg⁻¹ × 2.0 ≅ NH₄-N lbs ac⁻¹

Literature

Bremner, J.M. and D.R. Keeney. 1965. Determination and isotopic ratio analysis of different forms of nitrogen in soils: I. Apparatus and procedure for distillation for and determination of ammonium. *Soil Sci. Soc. Am. Proc.* 29:504-507.

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Keeney, D.R. and D.W. Nelson. 1982. Nitrogen-inorganic forms. *In* A.L. Page (eds.) *Methods of soil analysis, part 2*. Agron. Monogr. 9, 2nd ed. ASA and SSSA, Madison, WI. p. 643-698.