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EVALUATION OF METHODS FOR SOIL INORGANIC NITROGEN ANALYSIS

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ABSTRACT: This study determined the effects of soil preservation methods on inorganic nitrogen (N) analysis and evaluated methods of soil inorganic N analysis. Soils were preserved by oven-drying at 55°C, air-drying at 27°C, and freezing at - 7°C. Inorganic N results were compared with initial N levels prior to imposing preservation treatments. Soil preservation effects on ammonium-nitrogen (NH_4^+-N) were not consistent across soil types. Soil nitrate-nitrogen (NO_3^--N) levels after air-drying and freezing compared most favorably with initial levels indicating that both are acceptable methods of soil inorganic-N preservation. Levels of NH_4^+-N averaged across soils were 3.9 mg/kg for steam distillation, 4.2 mg/kg for sodium salicylate-hypochlorite, and 3.7 mg/kg for indophenol blue. When compared with steam distillation averaged across soils, NO_3^--N for cadmium-copper (Cd-Cu) reduction was 4 mg/kg greater, followed by nitrate electrode at 3 mg/kg, and salicylic acid at 2 mg/kg. Recovery of added N ranged from 83.3 to 94.8% for the NH_4^+-N methods and from 74.8 to 112.4% for the NO_3^--N methods with the nitrate electrode averaging 98.3%.

INTRODUCTION

Accurate determination of fertilizer N requirements of a crop is essential to optimizing economic returns and minimizing environmental risks. Determination of residual soil NO_3^--N has improved the predictability of fertilizer N requirements for crop production (12,16).

Quantification of plant available forms of N is complicated by the possibility of changes in NH_4^+-N and NO_3^--N concentrations during the interim period between sampling and analysis due to microbial activity. As reviewed by Bremner (1), preservation of soil with toluene and other compounds has been ineffective and the preservatives can interfere with inorganic N determination. Differences in inorganic N concentration between methods of preserving soil; principally freezing, air-drying, oven-drying, and analysis at field-moisture levels have been found (20,23,26,). Piper (21) recommended that if field-moist soil cannot be analyzed for N immediately after sampling, the soil should be dried for 24 h at 55°C. There is evidence that air- or oven-drying of soil can significantly affect exchangeable NH_4^+-N and NO_3^--N concentrations (8,20). Nelson and Bremner (20) analyzed 10 different soils and found NH_4^+-N increased 4.2 mg/kg and NO_3^--N increased 1.7 mg/kg after air-drying at 22°C. Oven-drying at 55°C resulted in an average increase of 5.1 mg/kg for NH_4^+-N and 1.0 mg/kg for NO_3^--N as compared to immediate analysis of field moist samples. It has also been demonstrated that exchangeable NH_4^+ can significantly increase during storage of air-dried soil (26). Some studies have shown that soil can be preserved by storing frozen until analysis (7,20,23,27). Westfall et al. (27) noted that immediate oven-drying (30°C) or freezing (-7°C) resulted in minimal changes in NO_3^--N concentration, whereas storage at room temperature or refrigeration (4°C) was unacceptable.

Currently, the cotton-modeling program GOSSYM/COMAX is undergoing preliminary on-farm evaluation and requires residual N data at planting as an input (28). Weather conditions in Mississippi are warm and humid with the majority of rainfall occurring during winter and early spring. Meisinger (17) concluded that NO_3^--N carryover depends on climatic factors and soil characteristics. Farmers and crop consultants using a crop-modeling program, such as GOSSYM/COMAX, or the Pre-Sidedress Nitrate Test (15) must be aware of proper soil handling procedures to minimize changes in inorganic-N content.

Various analytical methods for N determination are available. Steam distillation (2) is time-consuming and tedious and is subject to interference from phosphate and alkali-labile organic N compounds, such as volatile amides (2,6), but is usually the method to which others are compared. Other methods include colorimetric (3,4,5,9,10,11,25 29), microdiffusion (14), and specific-ion electrode (18) analysis. Colorimetric analysis can be rapid and accurate, while microdiffusion methods are simple and require little equipment. Colorimetric methods are subject to interferences, but are capable of providing accurate and precise results. Analysis using ion-specific electrodes is simple, rapid, and inexpensive compared to steam distillation and colorimetric methods. The nitrate-electrode method suffers from interferences and requires continual standardization (14). Meyers and Paul (19) reported that with the nitrate-electrode procedure, interference can occur from concentrations of Cl^- greater than 177 mg/L and HCO_3^- greater than 305 mg/L, but SO_4^{2-} and PO_4^{3-} caused no interference. In an effort to avoid these interferences, Siegel (24) showed that NO_3^- conversion to NH_4^+ and subsequent measurement using an ammonia electrode to be acceptable for KCl extracts.

The present study was initiated to (i) determine soil preservation effects on inorganic N analysis, (ii) evaluate soil inorganic N procedures, and (iii) determine recovery of added NH_4^+ and NO_3^- as influenced by analytical procedure.

MATERIALS AND METHODS

Topsoil samples (0-15 cm) from a Dubbs (fine-silty, mixed, thermic, Typic Halpludalf), and Dundee (fine-silty, mixed, thermic, Aeric Ochraqualf) were collected in Yazoo County, MS. A Marietta (fine-loamy, mixed, thermic, siliceous, Aquic Fluventic Halplaquept) was collected from the Plant Science Research Center, Mississippi State, MS, and a Loring (fine-silty, mixed, thermic, Typic Fragiudalf) was collected from the North Mississippi Branch Experiment Station at Holly Springs, MS. Soils selected were representitive of major cotton producing areas of Mississippi. Each soil was hand crushed, mixed thoroughly, sieved through a 2.0-mm sieve, air-dried, and stored in covered polyethylene buckets.

Experiment I

Experiment I was conducted to investigate the effects of soil preservation methods on inorganic N levels. Initially, each soil was oven-dried for 24 hours at 55°C. A 1.0 kg quantity of each soil was then incubated at approximately 12% gravimetric water content and ambient laboratory temperature (approximately 23°C) in a plastic bag. Soils were mixed and aerated about every three days during a two week period. After incubation, each soil was separated into four equal portions to which the following treatments were imposed: i) immediate analysis at current moisture, ii) analysis after air-drying at 27°C for 48 hours, iii) analysis after oven-drying at 55°C for 24 hours, and iv) analysis after freezing at the initial moisture content for 72 hours at -7°C. Treatments were a factorial arrangement of handling procedures and soils in a completely randomized design. Five subsamples of each soil were analyzed for NH_4^+ and NO_3^- using steam distillation (14).

Experiment II

Experiment II was conducted to compare analysis procedures for soil inorganic N with differing soil types. Treatments (soil type x analytical method) were in a factorial arrangement in a completely randomized design with five subsamples of each treatment. Soil which was air-dried for Experiment I was used for Experiment II. Data are presented on an oven-dry basis (105°C for 24 h). Determination of NH_4^+-N was performed using steam distillation (14), indophenol blue (5), and sodium salicylate-hypochlorite (9). Procedures for analysis of NO_3^--N were steam distillation (14), nitrate electrode (13), salicylic acid (3), and Cd-Cu reduction (29). The results from each procedure were correlated with the results from steam distillation.

Experiment III

Experiment III was conducted to determine the recovery of added NH_4^+ and NO_3^- as influenced by analysis procedure. Treatments were a factorial arrangement of analytical procedures and soil types in a completely randomized design. Five subsamples were analyzed for each treatment. Data are presented on an oven-dry basis (105°C for 24 h). To determine NH_4^+-N recovery, 20 mL of 20 μg NH_4^+-N/mL was added to 20 g soil along with 200 mL M KCl-extracting solution. Each soil was then extracted using this solution according to the procedure of Keeney and Nelson (1982). The NH_4^+-N in the extracts was determined by either steam distillation (14) or indophenol blue (5). Also, 5 ml of 2 μg NH_4^+-N/mL was added to 2 g soil, and then extracted and analyzed for NH_4^+-N using the sodium salicylate- hypochlorite method (9). For determination of NO_3^--N recovery, 400 μg NO_3^- was added in solution to soil which was then extracted and analyzed for NO_3^--N either by steam distillation (14), salicylic acid (3), or nitrate electrode (13). For Cd-Cu reduction (29) determination of NO_3^--N, 5 mL of 2 μg NO_3^--N/mL was added to 2 g soil prior to extraction.

TABLE 1. Chemical Properties of the Soils used in this Study.

Soil properties†	Dubbs	Dundee	Marietta	Loring
pH	6.6	5.9	8.0	5.6
	- - - - - - - - - - -g/kg- - - - - - - - - - - -			
Organic matter	9.4	11.2	8.1	8.5
	- - - - - - - - - - -mg/kg - - - - - - - - - - - -			
P	59.8	69.5	70.9	37.9
K	225.8	282.8	167.6	193.9
Ca	1087.0	2440.0	4165.0	1761.1
Mg	322.1	592.6	110.4	296.8
Zn	2.9	1.8	1.9	1.2

† Analyses were performed by Mississippi State University Soil Testing Laboratory, Mississippi State, MS.

TABLE 2. Particle Size Distribution for Soils used in this Study.

Series name	Clay	Silt	Sand	Textural class
	- - - - - - % - - - - - - -			
Dubbs	10.8	33.2	56.0	sandy loam
Dundee	33.3	51.2	15.5	silty clay loam
Marietta	18.8	27.2	54.0	sandy loam
Loring	21.8	74.3	3.9	silt loam

RESULTS AND DISCUSSION

The soils selected for this study differed markedly in pH and organic matter (Table 1). The extractable P and K were high according to the Mississippi Soil Test Method. The soils also differed markedly in clay, silt, and sand contents (Table 2). The Dubbs and Dundee are representative soils from the Delta region. The Loring is representative of soils of loessial origin and the Marietta is found in the Blackland Prairie area.

Experiment I

The NH_4^+- and NO_3^--N levels of the soils were influenced by different handling procedures (Table 3 and 4). Different soil types had different initial levels of NH_4^+-N and ranged from 0.8 to 6.3 mg/kg. Increasing NH_4^+-N concentration appeared to be related to increasing clay content although an inconsistency existed between the Dubbs and Marietta soils. Concentration of NH_4^+-N decreased in the Dubbs after oven-drying and freezing and for all treatments with the Loring soil. All handling procedures resulted in an increase in NH_4^+-N for the Dundee soil compared to initial NH_4^+-N levels. Decreases in NH_4^+-N as a result of handling procedures ranged from 0.5 to 1.0 mg/kg for the Dubbs and 1.2 to 1.4 mg/kg for the Loring compared to initial levels. Increases in NH_4^+-N levels as a result of handling procedures ranged from 0.8 to 1.0 mg/kg in the Dundee compared to the initial level. Levels of NH_4^+-N in the Marietta showed an increase of 0.9 mg/kg when oven-dried and decreased 0.4 mg/kg when air-dried and 0.5 mg/kg when frozen compared to the initial levels. Nelson and Bremner (20) analyzed 10 different soils and concluded that air-drying at 22°C increased NH_4^+-N 4.2 mg/kg and oven-drying at 55°C increased NH_4^+-N 5.1 mg/kg. Although significant differences in NH_4^+-N levels occurred, the magnitude was small. All handling procedures appear suitable for routine testing of soils having low levels of NH_4^+-N.

Different soil types had different amounts of initial NO_3^--N levels and ranged from 6.5 to 23.3 mg/kg (Table 4). There was a trend for increasing NO_3^--N with decreasing clay content, except for the Marietta soil. Levels of NO_3^--N in the Dubbs showed an increase after air-drying, while the other soils were unaffected. Levels of NO_3^--N decreased for the Dundee and Loring after oven-drying. Nelson and Bremner (20) showed that air-drying at 22°C increased NO_3^--N 1.7 mg/kg and oven drying at 55°C increased NO_3^--N 1.0 mg/kg. They found storing the soil

TABLE 3. Effects of Soil Type and Handling Procedures on NH_4^+-N Levels.

	Initial	Air-dried	Oven-dried	Frozen
	- - - - - - - - - -	mg/kg	- - - - - - - - - -	
Dubbs	1.7± 0.4†	1.2± 0.3	1.0± 0.5	0.7± 0.2
Dundee	6.3± 0.4	7.1± 0.8	7.2± 0.3	7.3± 1.0
Marietta	0.8± 0.5	0.4± 0.1	1.7± 0.3	0.3± 0.1
Loring	2.3± 0.2	1.0± 0.3	1.1± 0.6	0.9± 0.4
$LSD_{(0.05)}$	0.6			
CV, %	17.6			

† Mean of five subsamples plus or minus one standard deviation.

TABLE 4. Effects of Soil Type and Handling Procedures on NO_3^--N Levels.

	Initial	Air-dried	Oven-dried	Frozen
	- - - - - - - - - -	mg/kg	- - - - - - - -	
Dubbs	23.3± 1.4†	26.9± 3.2	22.2± 3.6	24.8± 2.6
Dundee	12.9± 0.3	11.5± 2.7	10.1± 2.7	13.8± 0.7
Marietta	6.5± 1.2	5.2± 1.1	6.2± 0.4	5.8± 1.1
Loring	14.1± 1.5	13.1± 1.8	7.6± 1.3	13.1± 0.9
$LSD_{(0.05)}$	2.4			
CV, %	13.8			

†Mean of five subsamples plus or minus one standard deviation.

TABLE 5. Effects of Soil Type and Analytical Method on NH_4^+-N Levels.

	Steam distillation	SSH†	Indophenol blue
	mg/kg		
Dubbs	5.7± 0.5‡	5.0± 0.3	5.0± 0.4
Dundee	6.2± 0.3	7.1± 0.4	6.8± 0.4
Marietta	1.6± 0.4	2.2± 0.2	1.5± 0.5
Loring	2.1± 0.2	2.4± 0.1	1.5± 0.2
$LSD_{(0.05)}$	0.5		
CV, %	9.1		

† SSH - Sodium salicylate-hypochlorite.
‡Mean of five subsamples plus or minus one standard deviation.

frozen at -5°C did not result in a significant change in NH_4^+- or NO_3^--N levels compared to the initial levels. The results of this experiment show freezing did not change NO_3^--N levels from the original levels found, but changes in NO_3^--N concentrations were possible with oven-drying.

Experiment II

Levels of NH_4^+-N detected with each analytical method were dependent on soil type (Table 5). Levels of NH_4^+-N for all the soils ranged from 1.6 to 6.2 mg/kg for steam distillation. Sodium salicylate-hypochlorite and indophenol blue NH_4^+-N values were less than those determined by steam distillation for the Dubbs soil. Levels of NH_4^+-N detected in the Dundee soil were greatest when soils were analyzed using sodium salicylate-hypochlorite and indophenol blue methods compared to steam distillation. Sodium salicylate-hypochlorite analysis detected greater NH_4^+-N concentrations for the Marietta soil compared to steam distillation. Indophenol blue detected lower levels of NH_4^+-N in the Loring soil compared to steam distillation. The correlation coefficient values (r) for NH_4^+ when compared to steam distillation were 0.96 for indophenol blue and 0.94 for sodium salicylate-hypochlorite. Dorich and Nelson (5) analyzed soils using steam

TABLE 6. Effects of Soil Type and Analytical Method on NO_3^--N Levels.

	Steam distillation		CR †		NO_3^- electrode		Salicylic acid	
	-mg/kg-							
Dubbs	9.2±	0.5‡	9.9±	0.2	7.2±	0.9	12.5±	3.4
Dundee	17.0±	2.2	22.9±	0.5	20.1±	2.2	20.0±	2.0
Marietta	11.8±	1.1	17.6±	0.3	17.0±	0.2	13.4±	3.0
Loring	10.0±	1.5	15.3±	0.2	16.0±	0.4	11.7±	1.5
$LSD_{(0.05)}$	1.9							
CV, %	10.7							

† CR - Cd-Cu reduction.
‡Mean of five subsamples plus or minus one standard deviation.

TABLE 7. Effects of Soil Type and Analytical Method on Recovery of a Known Quantity of Added NH_4^+.

	Steam distillation	SSH †	Indophenol blue
	- - - - - - % recovery - - - - - -		
Dubbs	83.9± 4.3‡	78.6± 9.6	95.2± 4.6
Dundee	91.5± 2.6	80.9± 9.9	99.6± 4.8
Marietta	85.8± 1.7	81.9± 2.8	90.6± 6.3
Loring	87.8± 1.9	92.0± 2.4	93.2± 2.8
$LSD_{(0.05)}$	4.7		
CV, %	5.9		

† SSH - Sodium salicylate-hypochlorite.
‡Mean of five subsamples plus or minus one standard deviation.

distillation and indophenol blue and found no difference in the NH_4^+-N concentrations between the two procedures.

Levels of NO_3^--N were dependent on soil type and analytical method (Table 6). The NO_3^--N levels for the different soils ranged from 9.2 to 17.0 mg/kg as determined by steam distillation. Concentrations of NO_3^--N detected in the Dubbs soil were greater for salicylic acid and lower for nitrate electrode methods compared to steam distillation. Concentrations of NO_3^--N in the Dundee soil were greater when analyzed by Cd-Cu reduction, nitrate electrode, and salicylic acid methods compared to steam distillation. For the Marietta and Loring soils, the NO_3^--N levels were greater when analyzed by Cd-Cu reduction and nitrate-electrode procedures compared to steam distillation. The correlation coefficients (r) were 0.88 for Cd-Cu reduction, 0.71 for the nitrate electrode, and 0.82 for the salicylic acid methods when compared to steam distillation.

Experiment III

Recovery of added NH_4^+-N from each soil was dependent on the analytical procedure (Table 7). Recovery of added NH_4^+-N ranged from 78.6 to 99.6%. The greatest recovery of NH_4^+-N was found using the indophenol blue method, while sodium salicylate-hypochlorite tended to have the lowest recovery. Other researchers found similar results for recovery of added NH_4^+. Rice et al. (22) found 93 to 100% recovery of NH_4^+-N using the indophenol blue procedure. They also found 77 to 96% recovery using steam distillation and a lower coefficient of variation for indophenol blue than for steam distillation. They concluded that recovery of NH_4^+-N was dependent on the amount added to the soil. Dorich and Nelson (5) reported NH_4^+-N recovery of 99 to 102% from KCl extracts of soils using indophenol blue.

Recovery of added NO_3^--N for each soil was dependent on the analytical procedure (Table 8). Greatest recovery was found using salicylic acid, but for the Dundee, Marietta, and Loring soils, values were greater than 100%. The Cd-Cu reduction method resulted in the lowest recovery of added NO_3^--N and ranged from 59.7% for the Marietta soil to 88.2% for the Dubbs soil. Rice et al. (22) concluded that recovery of NO_3^--N was dependent on the amount added to the soil. Therefore, a direct comparison between Cd-Cu reduction and other methods may not be valid here since a much lower quantity of NO_3^--N was used with this method. Meyers and Paul (19) reported recoveries of 85 to 102% of NO_3^--N added to soils using the nitrate-electrode procedure which is similar to results found in this experiment.

CONCLUSIONS

Changes in NH_4^+-N levels occurred for all handling methods and soils when compared to the original levels. These differences were small and all handling procedures would be acceptable for determination of low soil concentrations of NH_4^+-N. In agronomic practice, a 1.0 mg/kg concentration of NH_4^+-N for 15 cm increments to a depth of 90 cm would be about 12 kg/ha of NH_4^+-N which would not greatly affect a fertilizer N recommendation. The results of this experiment showed soil NO_3^--N concentrations did not change while soil was stored frozen for a short period. Air-drying resulted in a slight increase for only one soil and would be the most workable practice for farmers or consultants. These recommendations are similar to those reported by Westfall et al. (27)

Inorganic N extracted from various soils differed between analytical procedures. This may be due to textural and chemical properties of the soils used. More research is needed to examine the methods more closely to determine the source(s) of deviation.

TABLE 8. Effects of Soil Type and Analytical Method on Recovery of a Known Quantity of Added NO_3^-.

	Dubbs		Dundee		Marietta		Loring	
	%		%		%		%	
Steam distillation	86.8±	7.4‡	82.1±	5.6	88.1±	8.5	89.4±	4.1
CR †	88.2±	9.1	74.3±	8.6	59.7±	7.5	76.1±	3.4
NO_3^- electrode	96.2 ±	6.2	93.5±	2.6	105.0±	4.0	98.3±	1.8
Salicylic acid	98.8 ±	9.7	127.0±	11.6	122.0±	8.3	120.5±	4.7
$LSD_{(0.05)}$	9.7							
CV, %	7.9							

† CR - Cd-Cu reduction.

‡Mean of five subsamples plus or minus one standard deviation.

Experimental error or interferences could have contributed to recovery values greater than 100% for some of the methods in Experiment III. In selecting a method for inorganic-N analysis by a soil testing laboratory, it is important to consider technician time required for the analysis and precision and accuracy of the method. Based on these considerations and the results of Experiments II and III, the indophenol blue method appears suitable for NH_4^+-N analysis, and the nitrate electrode for NO_3^--N analysis for routine soil testing.

REFERENCES:

1. Bremner, J. M. 1965 Inorganic forms of nitrogen, pp. 1179-1237. IN: C. A. Black, et al. (eds.) Methods of Soil Analysis, Part 2. Agronomy Monograph 9. American Society of Agronomy, Madison, WI.

2. Bremner, J. M. and D. R. Keeney. 1966. Determination and isotope-ratio analysis of different forms of nitrogen in soils: 3. Exchangeable ammonium, nitrate, and nitrite by extraction-distillation methods. Soil Sci. Soc. Amer. Proc. 30:577-582.

3. Cataldo, D.A., M. Haroon, L.E. Schrader, and V.L. Youngs. 1975. Rapid colorimetric determination of nitrate in plant tissue by nitration of salicylic acid. Commun. Soil Sci. Plant Anal. 6:71-80.

4. Crooke, W. M. and W. E. Simpson. 1971 Determination of ammonium in kjeldahl digests of crops by an automated procedure. J. Sci. Food Agric. 22:9-10.

5. Dorich, R. A. and D. W. Nelson. 1983. Direct colorimetric measurement of ammonium in potassium chloride extract of soils. Soil Sci. Soc. Amer. J. 47:833-836.

6. Freney, J. R. 1971. Phosphate interference in the Devarda's alloy reduction method for nitrate. Commun. Soil Sci. Plant Anal. 6:479-484.

7. Gasser, J. K. R. 1958. Use of deep-freezing in the preservation and preparation of fresh soil samples. Nature. 181:1334-1335.

8. Gasser, J. K. R. 1961. Effects of air-drying an airdry storage on the mineralizable nitrogen of soils. J. Sci. Food Agr. 12:778-784.

9. Gentry, C. E. and R. B. Willis. 1988. Improved method for automated determination of ammonium in soil extracts. Commun. Soil Sci. Plant Anal. 19:721-737.

10. Henzell, E. F., I. Vallis, and J. E. Lindguist. 1968. Automatic colorimetric methods for the determination of nitrogen in digests and extracts of soils, pp. 513-520. IN: J.W. Holmes (ed.) Ninth International Congress of Soil Science Transactions, volume 3. Elsevier Publishing Company, Inc., New York, NY.

11. Huffman, S. A. and K. A. Barbarick. 1981. Soil nitrate analysis by cadmium reduction. Commun. Soil Sci. Plant Anal. 12:79-89.

12. James, D. W., C. E. Nelson, and A. R. Halvorson. 1967. Soil Testing for Residual Nitrates as a Guide for Nitrogen Fertilization of Sugarbeets. Washington Agr. Exp. Sta. Cir. 480.

13. Johnson, G. V. 1992. Determination of nitrate-nitrogen by specific-ion electrode, pp. 25-27. IN: S. J. Donbohue (ed.) Reference Soil and Media Diagnostic Procedures for the Southern Region of the United States. Virginia Agric. Exp. Stn.-Southern Cooperative Series Bull. 374.

14. Keeney, D. R. and D. W. Nelson. 1982 Nitrogen: Inorganic forms, pp. 643-698. IN: A. L. Page et al. (eds.) Methods of Soil Analysis, 2nd edition. American Society of Agronomy, Madison, WI.

15. Magdoff, Frederick R. 1991. Field nitrogen dynamics implications for assessing N availability. Commun. Soil Sci. Plant Anal. 22:1507-1517.

16. Magdoff, F. R., D. Ross, and J. Amadon. 1984. A soil test for nitrogen availability to corn. Soil Sci. Soc. Amer. J. 48:1301-1304.

17. Meisinger, J.J. 1984. Evaluating plant-available nitrogen in soil-crop systems, pp. 391-416. IN: R.D. Hauck et al. (eds.) Nitrogen in Crop Production. American Society of Agronomy, Madison, WI.

18. Milham, P. J., A. S. Awad, R. E. Paull, and J. M. Bull. 1970. Analysis of plants, soil, and water for nitrate by an ion-selective electrode. Analyst 95:751-757.

19. Meyers, J. K. and E. A. Paul. 1968. Nitrate ion electrode method for soil nitrate nitrogen determination. Can. J. Soil Sci. 48:369-391.

20. Nelson, D. W. and J. M. Bremner. 1972. Preservation of soil samples for inorganic nitrogen analysis. Agron. J. 64:196-199.

21. Piper, C. S. 1944. Soil and Plant Analysis. Interscience Publisher. Inc., New York, NY.

22. Rice, C. W., M. S. Smith, and J. M. Crutchfield. 1984. Inorganic N analysis of soil extracts by automated and distillation procedures. Commun. Soil Sci. Plant Anal. 15:663-672.

23. Selmer-Olsen, A. R., A. Oien, R. Baerug, and I. Lungstad. 1971. Pretreatment and storage of soil samples prior to mineral nitrogen determination. Acta. Agr. Scand. 21:57-63.

24. Siegel, R. S. 1980. Determination of nitrate and exchangeable ammonium in soil extracts by an ammonia electrode. Soil Sci. Soc. Amer. J. 44:943-947.

25. Sims, J. R. and G. D. Jackson. 1971. Rapid analysis of soil nitrate with chromotropic acid. Soil Sci. Soc. Amer. Proc. 35:603-606.

26. Storrier, R. R. 1966. The pre-treatment and storage of soil samples for nitrogen analyses. J. Aust. Inst. Agr. Sci. 32:106-113.

27. Westfall, D. G., M. A. Henson, and E. P. Evans. 1978. The effect of soil sample handling between collection and drying on nitrate concentration. Commun. Soil Sci. Plant Anal. 9:169-185.

28. Whisler, F. D., B. Acock, D. N. Baker, R. E. Fye, H. F. Hodges, J. R. Lambert, H. E. Lemmon, J. M. McKinion, and V. R. Reddy. 1986. Crop Simulation models in agronomic systems. Adv. Agron. 40:141-208.

29. Willis, R. B. and C. E. Gentry. 1987. Automated method for determining nitrate and nitrite in water and soil extracts. Commun. Soil Sci. Plant Anal. 18:625-636.