

SEQUENTIAL MEASUREMENTS OF SOIL $\text{NH}_4\text{-N}$
AND $\text{NO}_3\text{-N}$ FROM TWO LONG TERM FERTILITY
EXPERIMENTS WITH VARIABLE N RATES

By

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INTRODUCTION

A thorough amount of information is available relative to soil testing for $\text{NO}_3\text{-N}$ in soils as a method of improving N-recommendations. However, surface soil $\text{NH}_4\text{-N}$ has not been employed as a diagnostic tool in most private and public soil testing laboratories. The actual window for soil testing in winter wheat production systems of the Southern Great Plains generally takes place within the months of July through September (after harvest and before planting and fertilization). Soil sampling during this period has been shown to provide an indication of residual soil-N before fall fertilization recommendations are determined. This measurement has been found to be extremely reliable when surface soil $\text{NO}_3\text{-N}$ is used as the soil test index. Consequently there have been few studies that have established a nitrogen recommendation index with simultaneous measurements of $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$. The objective of this experiment was to monitor surface soil $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ for an entire cycle in two long-term experiments and to obtain estimates of their relationship with time, environmental variables and grain yield.

SEQUENTIAL MEASUREMENTS OF SOIL $\text{NH}_4\text{-N}$ AND $\text{NO}_3\text{-N}$ FROM TWO
LONG TERM FERTILITY EXPERIMENTS WITH VARIABLE N RATES

ABSTRACT

Surface soil sampling is one of the few methods available for use as a diagnostic tool in the assessment of N recommendations in grain crops. The use of applied concepts such as $\text{NH}_4\text{-N}$ in surface soil samples as a function of time, remain untested in terms of improving N recommendation strategies. The objective of this study was to observe the dynamics of seasonal variations in surface soil $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ in two-long term fertility experiments as affected by N rate and to assess their relationship with time. The experimental sites were Experiment #222 in Stillwater, OK on a Kirkland silt loam (fine-silty, mixed, thermic Udic Argiustoll) and Experiment #502 in Lahoma, OK on a Grant silt loam (fine, mixed, thermic Udertic Paleustoll). These experiments were located on the Agronomy Research Station in Stillwater, OK and the North Central Research Station near Lahoma, OK. Treatments selected included N rates (kg ha^{-1}) of 0, 44, 88, and 132 at Stillwater, OK and 0, 22, 44, 66, 88, and 112 at Lahoma (P and K rates were adequate and were held constant at both sites). A randomized complete block design with four replications was employed at both locations. Soil samples (0-15 cm) were collected from September, 1991 through March, 1992. Samples were dried and ground to pass a 20 mesh

screen and two subsamples of 2 grams each were then extracted with 20 ml of 2M potassium chloride (Bremner, 1966) and analyzed for $\text{NO}_3\text{-N}$ and $\text{NH}_4\text{-N}$ using the 'Lachat-Quickchem' automated flow injection analysis system. Nitrate-nitrogen was determined using the modified Gries-Ilosvay method (Barns and Folkard, 1951; Bremner, 1965). The soil extract, once injected through a 'Lachat' NO_3 manifold (Quickchem No. 12-107-04-1-b), is passed through a column of copperized cadmium which reduces the NO_3 to NO_2 (Henriksen & Selmer-Olsen, 1970; Jackson et al. 1975). The "reduced" sample is then diazotized with sulfanilamide followed by coupling with N-(1-Naphthyl) ethylenediamine dihydrochloride and absorbance is determined at 520 nm (Lachat Instruments, 1989). Analyses of variance were performed by sampling date and environmental information was used in the interpretation of results. Regardless of the nitrogen rate used for the previous wheat crop, no differences in either surface soil $\text{NH}_4\text{-N}$ or $\text{NO}_3\text{-N}$ were found when sampling was initiated prior to fall fertilization at Stillwater. Although no differences in surface soil $\text{NH}_4\text{-N}$ were detected among N rates at Lahoma, significant differences in $\text{NO}_3\text{-N}$ were noted prior to fertilization. Following fertilizer applications, $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ demonstrated a positive linear relationship with increasing N rate (25 and 11 days after fertilizers were applied at Stillwater and Lahoma respectively). This linear relationship was significant up to 134 days after fertilizers were applied for both $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ at the Stillwater location. Surface $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ were considerably different at the two locations sampled. This was possibly due to differences in soil texture or clay content. Surface soil $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$

levels at the Stillwater location did not decrease to near check plot (0 N) levels until 134 days following fertilizer applications. Ammonium-N levels increased 186 days after fertilization suggesting mineralization of easily labile N pools. At the Lahoma site, $\text{NO}_3\text{-N}$ levels in all treatments were not significantly different 76 days following fertilization. Eighty seven and 88 days after fertilizers were applied at the Stillwater and Lahoma locations respectively, a tendency for $\text{NH}_4\text{-N}$ to decrease with a corresponding increase in $\text{NO}_3\text{-N}$ was observed. It was assumed that fertilizer/soil-N nitrification was taking place at this time. The relationship of $\text{NH}_4\text{-N}$ with $\text{NO}_3\text{-N}$ as a function of time was markedly different when comparing both locations. Experimental errors from analyses of variance were small for both $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$, indicating that combined field and laboratory errors (used for detecting differences among treatments) were minimal when compared to the differences observed when changing N rate. The inclusion of surface soil $\text{NH}_4\text{-N}$ in addition to the commonly used $\text{NO}_3\text{-N}$ soil test index may aid in establishing more precise fertilizer recommendations. Surface response models of $\text{NH}_4\text{-N}$ versus $\text{NO}_3\text{-N}$ and time may further assist in defining optimum times for soil sampling if both indexes are to be included in the soil N test.

LITERATURE REVIEW

The common forms of N utilized by plants are NH_4^+ and NO_3^- , however NO_3^- is often the predominant source of N because it can be present at higher concentrations in soil solution when compared to NH_4^+ (Tisdale et al., 1985). However, work by Olsen, 1986 has discussed the importance of NH_4^+ nutrition especially during grain filling stages of growth in wheat and corn. Much of the N found in surface soil horizons is present in an organic form that may persist for long periods of time. Only a small portion of the organic N reservoir of the soil is mineralized in each growing season. The rate at which organic-N is converted to NH_4^+ and NO_3^- is termed the mineralization rate (Alexander, 1977). Organic compounds are mineralized in three sequential reactions: amminization, ammonification, and nitrification. The first two are carried out by heterotrophic microorganisms and the third mainly by autotrophic soil bacteria (Tisdale et al., 1985). If readily degradable carbonaceous materials are present in the soil environment, NH_4^+ is assimilated rapidly into newly forming microbial biomass (Schmidt, 1982). Under certain circumstances microbial development is limited by available C and most of the NH_4^+ is oxidized to NO_3^- as fast as is formed. This is commonly understood as nitrification (Schmidt, 1982). Studies conducted relative to nitrification of fertilizers in no till and plowed soils, found that ratios of NO_3^- to NH_4^+ were higher in plowed soils except immediately following fertilization, and tillage (plow versus no-till) did not consistently affect nitrification when soils of both treatmentw were maintained at the same water content (Rice and Smith, 1983). In these studies nitrification was limited

primarily by substrate (NH_4) supply and/or distribution in the soil profile. It was further noted that since water evaporated more rapidly from plowed soils, nitrification was sometimes more rapid in no-till soils.

The dynamics of soil microorganisms are affected by resource quality, climate and soil conditions (Lynch and Hobbie, 1988). Microbial growth in soils can be slow when periods of activity are followed by longer periods of inactivity. Microorganism inactivity is often associated with stress in the natural environment (Lynch and Hobbie, 1988). Higashida and Takao (1985) studied seasonal fluctuation patterns of microbial numbers in grasslands and found that the factor responsible for peaks of bacterial numbers was the supply of substrates from the vegetation, and that soil water status also controlled the appearance of these peaks. Bramley and White (1989) did not find a relationship in four soils studied, between soil pH optimums for nitrification, short term nitrification assay (SNA), soil moisture content, soil temperature and organic carbon. Microbial activity is influenced by changes in the availability of soil moisture (Orchard and Cook, 1983). Linear relationships were found between water potential and microbial activity when activity was not limited by substrate availability (Orchard and Cook, 1983). Work conducted by Lund and Goksoyr, 1980, demonstrated that the highest N mineralization rates occurred when 80 to 90% of the total pore space was filled with water and that with decreasing moisture, N mineralization continued to decline. However, water levels above optimum often reduced mineral N accumulations which was possibly due to denitrification (Lund and Goksoyr, 1980). Regarding microbial

activity and water fluctuations, it has been observed, that drying and rewetting periods have an overall effect of increasing the mineralization rate where easily available nutrients from dead microorganisms are used, resulting in mineralization of humic substances (Lund and Goksoyr, 1980). This helps explain why fluctuating water activity in the soil may result in more rapid decomposition and mineralization than under a stable moisture level under optimal conditions (Lund and Goksoyr, 1980). Aerobic microbial activity is enhanced with soil water content up to the point where water displaces air and restricts the diffusion and availability of oxygen (Bhaumik and Clark, 1948). Therefore, the amount of soil pore space filled with water appears to be closely related to soil microbial activity under different tillage regimes (Linn and Doran, 1984).

The ability of soil to supply N is influenced by the amount of mineralizable organic N, mineralization rate and intensity factors such as moisture and temperature (Campbell, 1981). Sabey et al., 1956 studied nitrification of ammonium sulfate as influenced by temperature in three soils under laboratory conditions and noted that nitrification decreased at lower soil temperatures. Complete inhibition was not attained until soil temperature approached the freezing point (Sabey et al., 1956). Only slight oxidation of ammonium was found to occur under field conditions in soils that were fertilized after soil temperature had decreased below 10°C (Sabey et al., 1956). Brady (1990) indicated that microbial oxidation of ammonium ions to nitrate ions occurs most readily at temperatures of 27-32°C, and that this effect was negligible when the

soil temperature was lowered to 10°C. Carbon and N mineralization appears to be enhanced in practically all soils by drying temperature as well as drying-rewetting cycles (Agarwal et al., 1971). Greater releases of N were observed when incubation occurred after drying compared to when this step was omitted (Agarwal et al., 1971).

Work by Dalal and Mayer (1987) studying nitrogen mineralization potential, and nitrogen mineralization rate in six major soils which had been used for cereal cropping for over 20 years, found that mineralizable N could be estimated by taking N_0 as 5% of the total N in soils having < 40% clay, and 15% of total N for soils of > 40% clay. The mineralization rate, K, was found to be 0.066 week⁻¹ at 40°, 0.054 week⁻¹ at 35°C and 0.027 week⁻¹ at 25°C (Dalal and Mayer, 1987). Based on the N mineralization potential (N_0) and the mineralization rate constant (K), estimates of N availability increased within the sequence; loamy sand, coarse sandy loam and loam (Herlihy, 1979). Nitrogen availability was found to be consistently higher in the high labile organic matter fractions from the soils studied which was possibly due to the effects of soil texture in the initial stages of decomposition of organic matter (Herlihy, 1979). The rate constants that describe C and N mineralization vary due to the changing complexity of the C compounds decomposed (Honeycutt, 1988). The concentration of potentially mineralized nitrogen was greater in surface than in subsurface soil and directly proportional to the total soil C (Campbell et al, 1981). When determining the influence of N fertilizer on N and C mineralization from corn residues with a low C:N ratio, it was found that residue N

mineralization rates were reduced when N fertilizer was applied to soils with a low pH (Clay and Clapp, 1990). Soils with a low pH were found to have reduced nitrification rates, which result in high NH_4^+ concentrations that may inhibit microbial populations (Clay and Clapp, 1990).

Net loss of N occurs in soil through a process called denitrification that includes reduction of NO_3^- and NO_2^- with the liberation of molecular N_2 and N_2O (Alexander, 1977). There are differences in the adaptation of microbial populations of denitrifiers in different soils. For example, denitrifiers in temperate soils reduce NO_3^- at lower temperatures than those in subtropical soils (Powlson et al., 1987). Denitrification occurred at 10°C in a temperate soil with a sharp increase occurring from 5 to 10°C . This suggested that denitrification could be a major cause of N loss in temperate areas during the spring when N fertilizer is applied (Powlson et al, 1987). Nitrate-N added to two soils maintained under anaerobiosis for 7 days at 1°C was found to remain in the soil and that increasing the temperature to 7°C produced a slow increase in denitrification (Jacobson and Alexander, 1980). In general, denitrification was found to be markedly inhibited at 5°C (Bailey and Beauchamp, 1973). Incubation temperatures of 5 , 10 , 15 and 30°C apparently did not influence denitrification potential, however, the level of available C was found to affect this process (Smid and Beauchamp, 1976).

Work by Stanford et al, 1973, evaluating soil N availability and uptake of labeled and unlabeled N by plants, indicated that average mineral-N recovery by whole plants before and during the cropping period was about 85 percent.

Stanford, 1973, stated that with good management, recovery of applied N in corn grain and stover is between 50 and 70%. At near optimum N rates, essentially all of the unrecovered fertilizer N is subject to immobilization during decomposition of plant residues. However at rates higher than the optimum, a significant portion of the NO_3^- remains mobile and susceptible to loss by leaching or denitrification (Stanford, 1973).

The fate of N in the soils is a function of various factors. Measurements of $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ in soils is affected by soil texture, temperature, moisture, organic matter and the interacting relationships between each of these over time. The objectives of this study were to evaluate seasonal changes in soil $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ in two long term fertility experiments and their relationship with time, measured climatic variables and grain yield.

MATERIALS AND METHODS

Two long-term wheat fertility experiments were selected that had a range of N rates applied. The sites where sampling was conducted included Experiment #222 which was initiated in 1969 on the Agronomy Research Station in Stillwater, Oklahoma and Experiment #502 which was established on the North Central Research Station near Lahoma, Oklahoma in 1971. Initial soil test characteristics at each location are listed in Table 1. The soil type at Stillwater, was a Kirkland silt loam (fine, mixed, thermic, Udertic Paleustoll) and a Grant silt loam (fine, mixed, thermic, Udic Argiustoll) at Lahoma. The main purpose of these experiments was to evaluate long-term wheat grain yield

response to applied N, P and K in areas where continuous wheat is commonly grown. The experimental design in both cases was a randomized complete block design with four replications. Plot size was 6.09 x 18.3 m at Stillwater, and 5.0 x 18.3 m at Lahoma, OK. Four treatments were sampled in Experiment #222 and six in Experiment #502 (Table 2). Fertilizer sources (N-P-K) used included, ammonium nitrate (NH_4NO_3 , 34-0-0), triple superphosphate (0-20-0) and potassium chloride (KCl, 0-0-50). Surface soil sampling was initiated before fertilizers were applied in the fall and prior to planting. Sixteen cores were taken in each plot at each sampling date, to a depth of 15 cm. Cores were mixed to obtain a complete composite that were dried at ambient temperature for 6-7 days depending on the initial soil moisture, and ground to pass a 20 mesh screen. Two sub-samples of two grams each were extracted with 2M potassium chloride (Bremner and Keeney, 1966). Soil extracts were analyzed for $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ using a Lachat-flow injection analyzer. Sampling dates at Stillwater were: 9/9/91, 10/4/91, 10/14/91, 11/11/91, 12/5/91, 1/25/92, 3/13/92 and at Lahoma: 8/28/91, 9/9/91, 9/23/91, 10/4/91, 10/10/91, 11/13/91, 11/25/91, 1/17/92 and 3/12/92 respectively. At both locations, the first sampling date was one day before fertilizers were applied. Amounts of $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ were determined by taking the mean from the two subsamples. Daily temperature and precipitation was recorded at Stillwater and the same information in addition to soil temperature was obtained at Lahoma. Analysis of variance for $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ in surface soil samples obtained at Stillwater and Lahoma was performed including orthogonal contrasts of N rate

(linear and quadratic). Surface response models were generated to establish the relationships of $\text{NH}_4\text{-N}$ with $\text{NO}_3\text{-N}$ and time. Other surface response models were evaluated that employed soil temperature and cumulative rainfall.

RESULTS AND DISCUSSION

In general, the Lahoma experiment has been conducted on a soil that has better drainage than the soil at Stillwater. Both soils fall within the 'Thermic' temperature regime, having a mean soil annual temperature of 15°C or higher, but lower than 22°C (Soil Conservation Service, 1975). Soil sample analyses of variance, by date, are reported for $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ in Tables 3 and 4 for Stillwater and Lahoma, respectively.

Stillwater, OK, Experiment #222

A significant linear response of $\text{NH}_4\text{-N}$ to N fertilization was found at all dates excluding September 9 and March 13 (Table 3 and Figure 1). Air temperature maximums and precipitation for this same time period are plotted accordingly in Figure 2. Surface soil $\text{NH}_4\text{-N}$ in the first sampling date (prior to fertilization) was found to be significantly affected by N rate (Table 3, 0.10 probability level). Following fertilization surface soil $\text{NH}_4\text{-N}$ showed corresponding increases with N rates and time up to 63 days (Figure 1). However, surface soil $\text{NH}_4\text{-N}$ in the check plot (0N) did not increase with time. A tendency for small decreases was noted for $\text{NH}_4\text{-N}$, 63 to 134 days after

fertilizers were applied. With the increased temperature and cumulative precipitation received (134 to 186 days after fertilizers were applied), a marked increase in $\text{NH}_4\text{-N}$ was noted for the last sampling date (Figures 1 and 2). Crop uptake and accumulation of $\text{NO}_3\text{-N}$ and $\text{NH}_4\text{-N}$ in vegetative tissue is known to be taking place during this time (Gardner and Jackson, 1976, Raun and Westerman, 1991). However, even with the crop variable included, the significant increase in $\text{NH}_4\text{-N}$ 134 to 186 days after fertilizers were applied suggests that ammonification was taking place.

Similar to results for $\text{NH}_4\text{-N}$ noted at this location, a significant linear response to N fertilization was found for $\text{NO}_3\text{-N}$ at all dates excluding September 9 and March 13 (Table 3 and Figure 1). Twenty five days after fertilizers were applied, $\text{NO}_3\text{-N}$ levels increased significantly and then declined at the 35 and 63 day sampling dates for all treatments. Given that these plots are representative of long-term applications, it was interesting to observe an increase in $\text{NO}_3\text{-N}$ in the check plot where no fertilizers have been applied for over 20 years. This suggests that fluctuations in mineral-N as a function of the total organic-N pool were still notable in plots receiving no N for over twenty years. Because temperature and precipitation were favorable for mineralization and more specifically, nitrification, between the first and second sampling date, an expected increase in both $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ was expected since N applications would narrow the C:N ratio. This was also noted in the check plot receiving no N fertilization. Nitrate-N subsequently increased between the 63 and 87 day sampling dates in all plots receiving N. This increase was

associated with a corresponding decrease in $\text{NH}_4\text{-N}$ at the same respective dates. Given the abnormally low (late October) followed by abnormally high ambient temperatures (mid November) in association with above average precipitation received during this time period, it is thought that nitrification was taking place (Figures 1 and 2). The markedly low temperatures noted at the beginning of November (-5 to 10°C) may have affected microbial oxidation of $\text{NH}_4^+\text{-N}$ to $\text{NO}_3^-\text{-N}$. According to Brady (1990), this process proceeds rapidly at soil temperatures of 27 to 32°C and is negligible below 10°C. Between the fourth and sixth sampling (63 to 134 days after fertilization), the mild temperature conditions (13 to 23°C) associated with short wetting and drying periods (precipitation) may have favored the nitrification process. Work by Lund and Goksoyr, 1980 has indicated that fluctuating water activity in the soil can result in more rapid decomposition and mineralization of the microbial biomass. Only very small changes in surface soil $\text{NO}_3\text{-N}$ were noted during the 134 to 186 day period (Figure 1). The nitrification rate may have been slower than that mentioned for ammonification, however, because $\text{NO}_3\text{-N}$ is mobile and is known to accumulate in wheat during this time period (134 to 186 days), it was not surprising to find small differences in this soil test variable. By the final sampling date, 186 days following fertilization, surface soil $\text{NO}_3\text{-N}$ levels were all less than 10 mg kg⁻¹ which was very similar to that noted when sampling was initiated. Because surface soil $\text{NO}_3\text{-N}$ would be subject to crop depletion, leaching, and possible immobilization and/or denitrification during the 186 day period, only limited amounts were expected in these surface samples following

the given time period.

Lahoma, OK, Experiment #502

In Experiment #502 near Lahoma, OK, analyses of variance for surface soil $\text{NH}_4\text{-N}$ indicated that the main effect of N-rate was significant in seven of the nine sampling dates (Table 4). Partitioning the main effect of N rate into orthogonal linear and quadratic single degree of freedom contrasts demonstrated that surface soil $\text{NH}_4\text{-N}$ was detectable as a linear function of N rate at all sampling dates excluding August 28 before fertilizers were applied. Following fertilization, $\text{NH}_4\text{-N}$ was significantly affected by N rate up to 196 days after fertilization. Surface soil $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ are represented as a function of time and N rate in Figure 3. Unlike results noted at the Stillwater location, $\text{NH}_4\text{-N}$ levels were below 10 mg kg^{-1} by 88 days after fertilizers were applied. However, similar to that noted at the Stillwater location, $\text{NH}_4\text{-N}$ showed positive linear correlation with N rate immediately following fertilization. Only limited fluctuations in $\text{NH}_4\text{-N}$ were observed 11 to 76 days after fertilizers were applied. From 76 to 88 days after fertilizers were applied, a sharp decrease in $\text{NH}_4\text{-N}$ was noted with a corresponding tendency for increased surface soil $\text{NO}_3\text{-N}$. By the last two sampling dates, 141 and 196 days after fertilization, $\text{NH}_4\text{-N}$ levels had declined significantly. It is important to note that even within this extremely small range ($0\text{-}10 \text{ mg kg}^{-1} \text{NH}_4^+\text{-N}$) a significant linear relationship was detected for N rate 88, 141 and 196 days after fertilization. This would appear to indicate that the combined experimental precision (random error) and associated

laboratory accuracy (bias error) was extremely sensitive. Because this was also noted at the Stillwater location, it would further suggest that the experimental precision needed to estimate $\text{NH}_4\text{-N}$ (given the procedures employed) within a long-term experiment, is more than adequate. If $\text{NH}_4\text{-N}$ were to be used as a soil test index variable for improving N recommendations, present laboratory, sampling and field experimental procedures appear to be minimizing random and bias errors since the controlled independent variable (N rate) was highly significant.

Surface soil $\text{NO}_3\text{-N}$ from the nine Lahoma sampling dates demonstrated linear increases with N rate both before fertilization and up to 76 days following the time when fertilizers were applied (significant N-Rate linear effect, Table 4). It was interesting to note a significant relationship between soil $\text{NO}_3\text{-N}$ and N rate (Aug 28, 1991 before fertilizers were applied in 1991) more than 1 year after fertilizers were applied in 1991 (August 2, 1990). However, prior to fertilization only the 88 and 112 kg N ha⁻¹ rates were significantly greater than the check indicating that residual amounts (following harvest) in the soil were minimal for the 22, 44, and 66 kg N ha⁻¹ rates (Table 4 and Figure 3). The marked decrease in soil $\text{NO}_3\text{-N}$ 36 to 42 days after fertilization is difficult to explain. During this time period, soils at this site were initially very wet and then subjected to abnormally high temperatures for over 20 days (Figure 4). Marginal decreases were then noted up until the last sampling date, 196 days after fertilization where small increases in $\text{NO}_3\text{-N}$ were found (Figure 4). Surface soil $\text{NO}_3\text{-N}$ found in the 141 and 196 day sampling dates was below that

measured before fertilization when sampling was initiated (Figure 4).

Surface Response Models

A quadratic surface response model of $\text{NH}_4\text{-N}$ versus $\text{NO}_3\text{-N}$ and time employing a linear interaction term ($\text{NH}_4\text{-N} * \text{Time}$) was generated for the combined data obtained at Stillwater (-1 to 186 days) and Lahoma (-1 to 196 days). Following the independent relationship noted between surface soil $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ as a function of time, surface response models allowed further observation of possible interactions. Individual regression equations, and significance levels are reported accordingly in Table 5. Graphic illustration of the surface response models are found in Figures 5 and 6 for the Stillwater and Lahoma experiments respectively. In both models, the main effects of $\text{NH}_4\text{-N}$ and $\text{NH}_4\text{-N}^2$ were highly significant in predicting surface soil $\text{NO}_3\text{-N}$ (Table 5). In addition, both models demonstrated significant independent effects of Time and $\text{NH}_4\text{-N} * \text{Time}$. It was expected that the difficulty in detecting surface soil $\text{NH}_4\text{-N}$ at Lahoma (narrow range observed at the end of the sampling period), could be associated with the lower cation exchange capacity present at this site. Surface soil nitrate-N at this location should also have been lower than that observed at Stillwater since these soils are well drained and would thus be subject to more rapid $\text{NO}_3\text{-N}$ leaching losses. In general, both surface response models mirrored independent observations of $\text{NH}_4\text{-N}$ and time and $\text{NO}_3\text{-N}$ with time. However, when combined, multiple regression equations were both significant at the 0.001 probability level and accounted for more than 50%

of the variability in surface soil $\text{NO}_3\text{-N}$. The utility of the surface response models discussed lies in the ability to detect surface soil $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ at levels that continue to reflect linear changes in N rate. In addition, once the sampling cycle is complete, these models may further assist in identifying optimum times for soil sampling if both $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ are to be included within the soil test index. Because a high level of precision was noted in the detection of surface soil $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ it is of utmost importance to identify the ideal concentration range (time), in order to improve the accuracy of ultimate fertilizer N recommendations.

CONCLUSIONS

Sequential surface soil samples (0-15 cm) were taken during the months of August 1991 through March, 1992 from two long-term soil fertility experiments with variable N rates (Experiment #222, Stillwater, OK and Experiment #502, Lahoma, OK). Samples were analyzed for $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ as defined by Bremner and Keeney, 1966. In general, it was difficult to detect differences in surface soil $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ prior to the time when fertilizers were applied (September). However, a linear response was observed between surface soil $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ and N rate 134 and 196 days after fertilization at Stillwater and Lahoma respectively. Although both data ranges for surface soil $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ were small ($<10 \text{ mg kg}^{-1}$) at the Lahoma location (>42 days after fertilization) experimental precision was still adequate in being able to detect significant differences as a function of N rate. Results from this study suggest that $\text{NH}_4\text{-N}$ may need to be included within the soil N test which at present establishes N recommendations based only on $\text{NO}_3\text{-N}$. Upon the completion of analysis from surface soil samples taken for the entire cycle (September through August), it may be possible to define the optimum time when sampling should take place that includes both $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$. Surface response models of $\text{NO}_3\text{-N}$ versus $\text{NH}_4\text{-N}$ and Time are expected to assist in this regard. Future work in refining N recommendations for crop and forage yields should include both $\text{NH}_4^+\text{-N}$ and $\text{NO}_3^-\text{-N}$ analyses.

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Table 1. Soil description and soil test characteristics.

Location	Soil Classification	pH mg kg ⁻¹	P mg kg ⁻¹	K	BI
Stillwater Experiment #222	fine-silty, mixed, thermic Udic Argiustoll	5.0	61	366	6.7
Lahoma Experiment #502	fine, mixed, thermic Udertic Paleustoll	5.3	146	822	6.7

pH 1:1 soil:water, P and K = Mehlich III.

Table 2. Treatments sampled at each location

Treatments	N	P	K
	-----kg ha ⁻¹ -----		
Stillwater, Experiment #222			
1	0	26	33
2	44	26	33
3	88	26	33
4	132	26	33
Lahoma, Experiment #502			
2	0	17	50
3	22	17	50
4	44	17	50
5	66	17	50
6	88	17	50
7	112	17	50

Table 3. Analyses of Variance for $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ from seven sequential sampling dates in Experiment #222, Stillwater, OK.

Days after Fertilization		-1	25	35	63	87	134	186
		Mean Squares $\text{NH}_4\text{-N}$						
Source of Variation	df	Sep 9	Oct 4	Oct 14	Nov 11	Dec 5	Jan 21	Mar 13
Rep	3	18.50	98.75	49.54	80.11	49.91	3.60	15.71
Trt	3	15.36	276.05**	527.85	1166.85**	1140.39**	121.85**	118.16
Error	9	10.95	36.78	157.24	54.84	56.82	8.30	86.37
N-Rate linear	1		**	*	**	**	**	
N-Rate quadratic	1	@				*	*	
Coefficient of Variation, %		32	38	62	35	45	38	41
		Mean Squares $\text{NO}_3\text{-N}$						
Source of Variation	df	Sep 9	Oct 4	Oct 14	Nov 11	Dec 5	Jan 21	Mar 13
Rep	3	5.54	138.61	4.55	6.48	8.64	3.28	1.97
Trt	3	5.35	614.26*	104.02	71.08**	410.66**	106.90**	7.15
Error	9	15.39	114.15	36.47	2.80	6.06	2.19	2.84
N-Rate linear	1		**	*	**	**	**	
N-Rate quadratic	1				**			
Coefficient of Variation, %		44	34	39	17	15	23	26

@, *, ** - significant at 0.10, 0.05 and 0.01 probability levels respectively. Significance levels for N Rate linear and quadratic were determined as the probability of obtaining values greater than absolute T.

Table 4. Analyses of Variance for $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ from nine sequential sampling dates in Experiment #502, Lahoma, OK.

Days after Fertilization		-1	11	25	36	42	76	88	141	196
		Mean Squares $\text{NH}_4\text{-N}$								
Source of Variation	df	Aug 28	Sep 9	Sep 23	Oct 4	Oct 10	Nov 13	Nov 25	Jan 17	Mar 12
Rep	3	5.26	93.33	45.61	20.37	4.26	15.68	1.80	2.35	6.53
Trt	5	4.25	306.04**	234.95**	247.70**	333.52**	285.52**	48.82**	14.32**	4.79
Error	15	2.47	49.83	11.00	10.77	19.01	12.46	2.61	1.71	2.92
N-Rate linear	1		**	**	**	**	**	**	**	*
N-Rate quadratic	1			**	**	**	**	**	**	
Coefficient of Variation, %		35	40	40	27	33	32	29	29	45
		Mean Squares $\text{NO}_3\text{-N}$								
Source of Variation	df	Aug 28	Sep 9	Sep 23	Oct 4	Oct 10	Nov 13	Nov 25	Jan 17	Mar 12
Rep	3	1.98	25.87	15.55	12.21	4.83	1.83	12.38	0.87*	0.57
Trt	5	101.81**	1038.63**	881.51**	1192.48**	169.40**	20.36**	58.23**	7.49**	2.70
Error	15	13.16	15.81	11.85	31.82	17.854	1.77	13.96	0.25	1.07
N-Rate linear	1	**	**	**	**	**	*	**	**	**
N-Rate quadratic	1								*	
Coefficient of Variation, %		25	12	10	13	26	22	67	21	27

@, *, ** - significant at 0.10, 0.05 and 0.01 probability levels respectively. Significance levels for N Rate linear and quadratic were determined as the probability of obtaining values greater than absolute T.

Table 5. Regression equations, significance and R^2 for $\text{NO}_3\text{-N}$ (0-15 cm soil depth) versus time and $\text{NH}_4\text{-N}$ concentration Experiment #222, Stillwater, and Experiment #502, Lahoma, OK.

Location	Model	R^2	Significance Prob >F
Stillwater	$Y = 1.205 + 0.636(T) + 1.5101(\text{NH}_4\text{-N})^{**}$ $- 0.004(T^2)^{\textcircled{a}} - 0.0032(\text{NH}_4\text{-N} \cdot T)^* - 0.0017$ $(\text{NH}_4\text{-N}^2)^{**}$	0.50	<0.001
Lahoma	$Y = 82.007^{**} - 1.240(T)^{**} + 3.011(\text{NH}_4\text{-N})^{**}$ $+ 0.00531(T^2)^* - 0.0169(\text{NH}_4\text{-N} \cdot T)^{**}$ $- 0.00419(\text{NH}_4\text{-N}^2)^{**}$	0.62	<0.001

@, *, ** - significant at 0.10, 0.05 and 0.01 probability levels respectively. Multiple regression coefficient significance levels determined as the probability of obtaining values greater than absolute T.

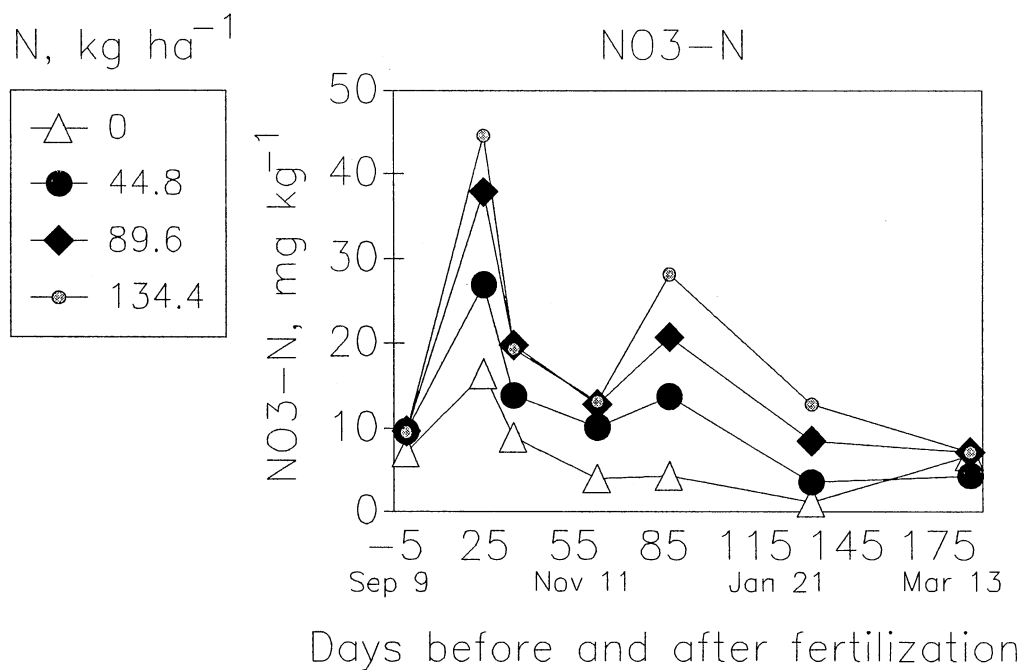
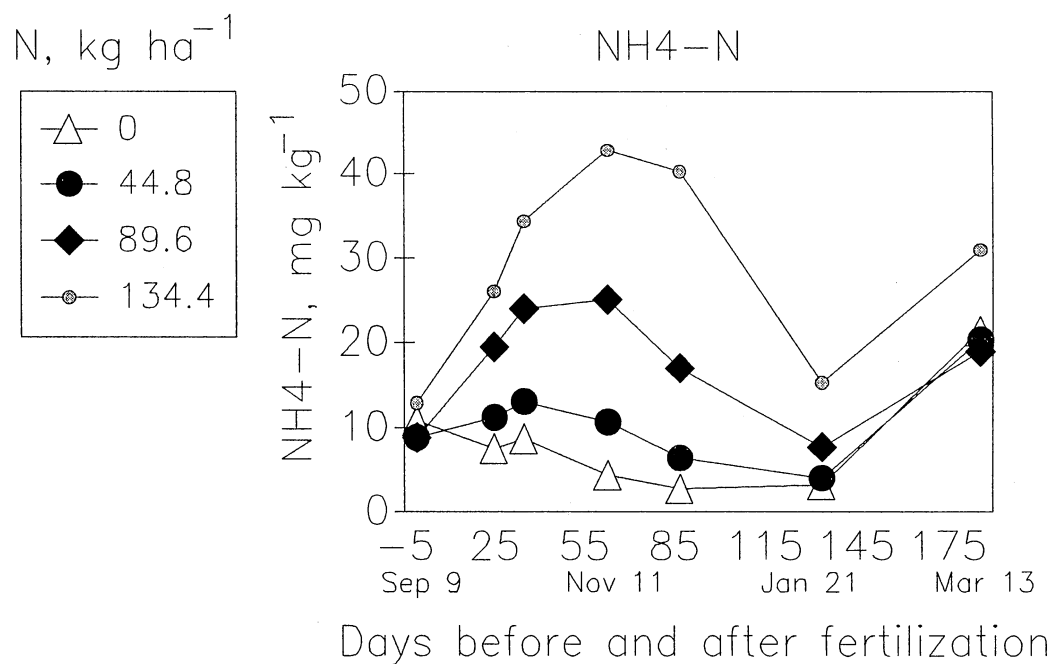


Figure 1. Surface soil (0-15 cm) NH₄-N, and NO₃-N with increasing N rates in Experiment #222 before and after fertilization, Stillwater, OK.

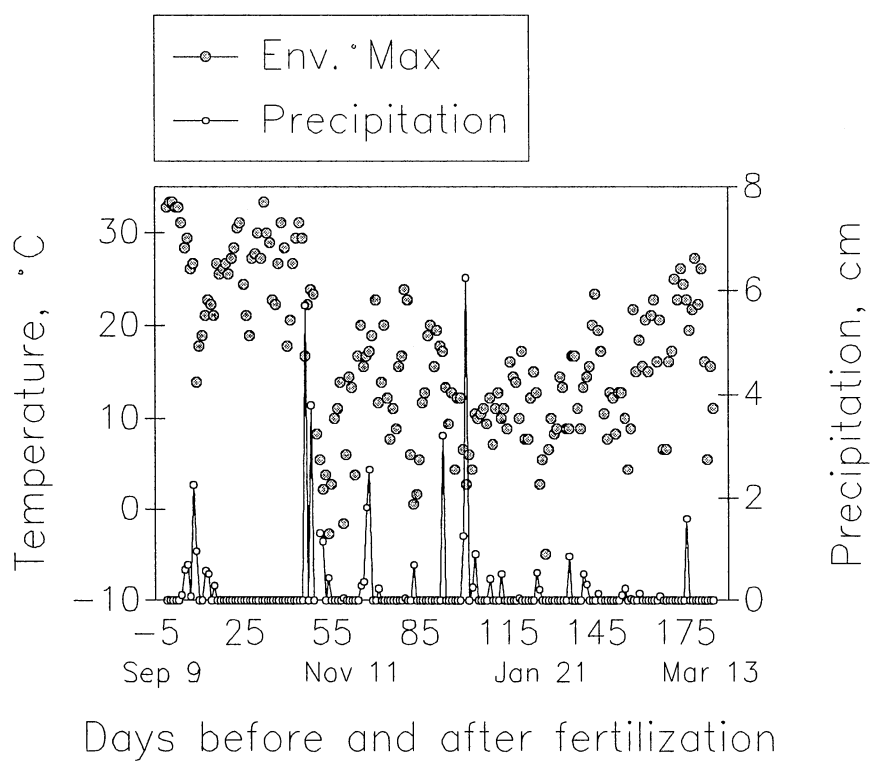


Figure 2. Maximum ambient temperatures and precipitation during the study period, Stillwater, OK.

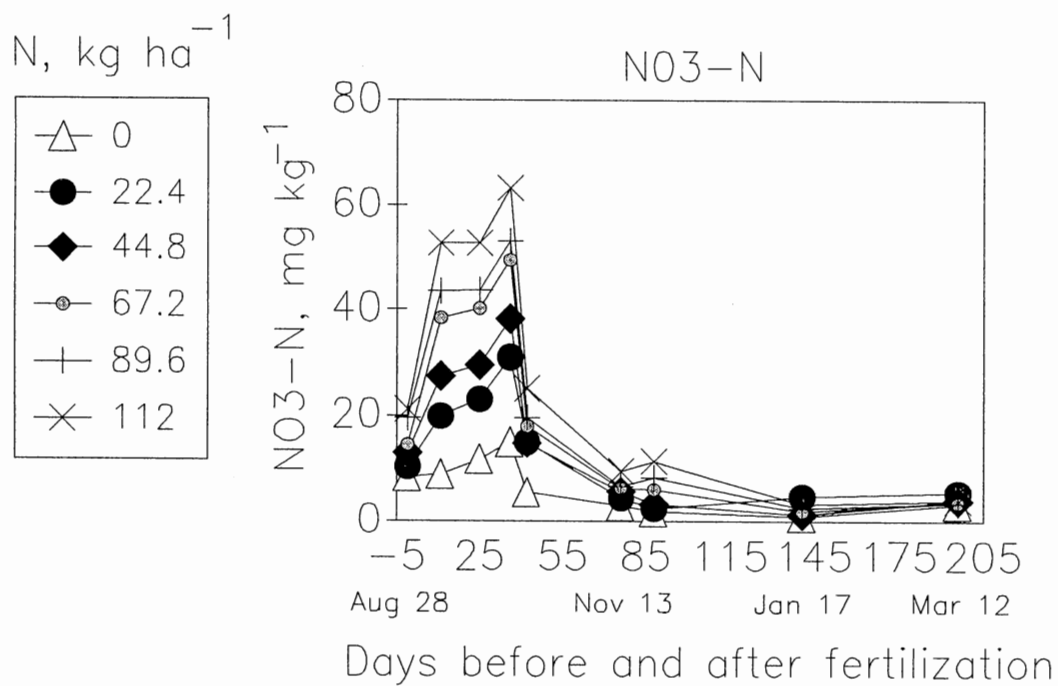
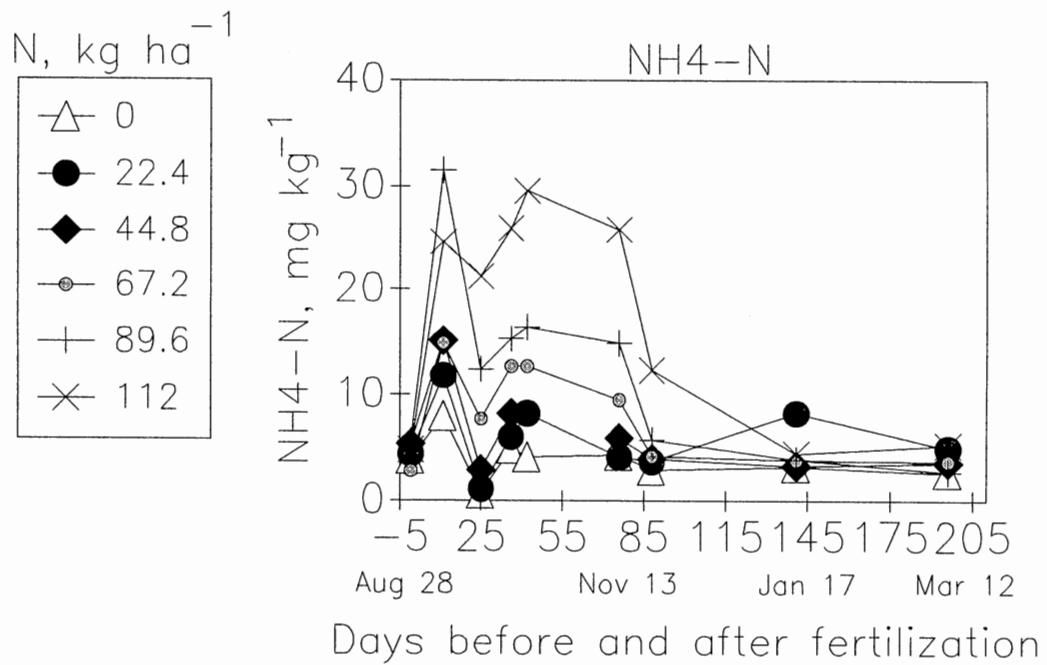


Figure 3. Surface soil (0-15 cm) NH₄-N and NO₃-N with increasing N rates in Experiment #502 before and after fertilization, Lahoma, OK.

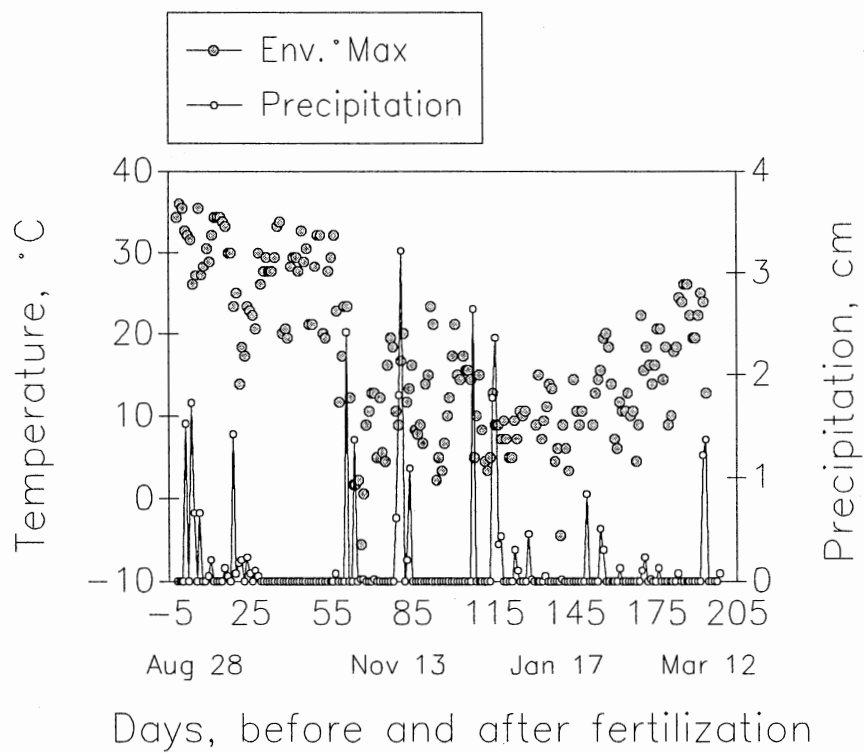


Figure 4. Maximum ambient temperatures, and precipitation during the study period, Lahoma, OK.

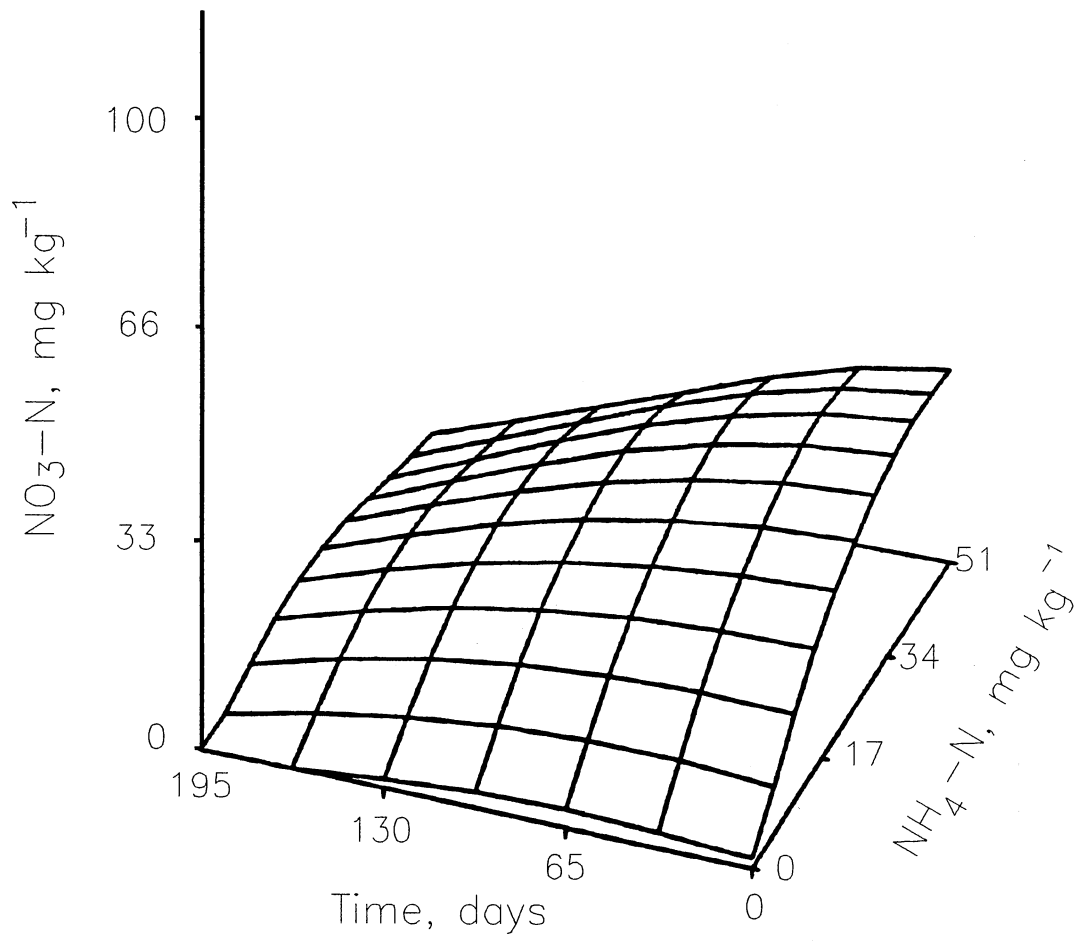


Figure 5. Surface response model of $\text{NO}_3\text{-N}$ versus $\text{NH}_4\text{-N}$ and time, Stillwater, OK.

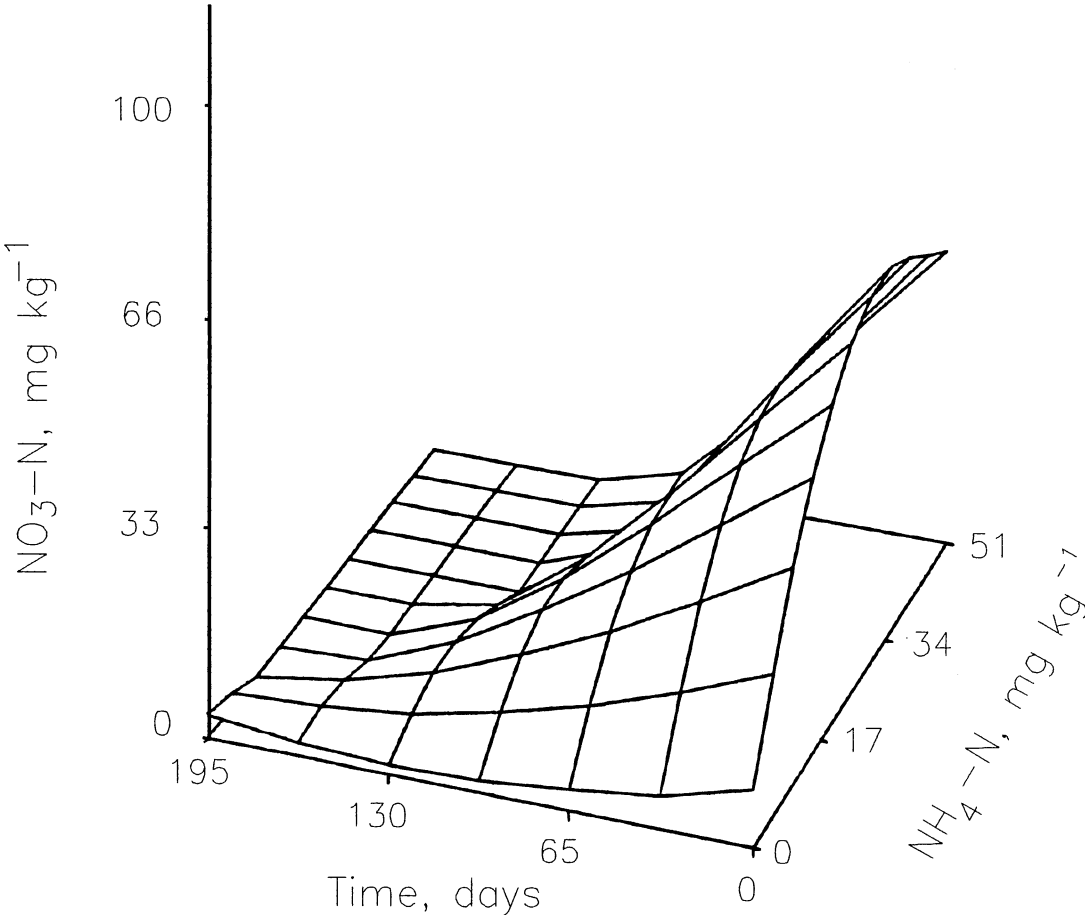


Figure 6. Surface response model of $\text{NO}_3\text{-N}$ versus $\text{NH}_4\text{-N}$ and time, Lahoma, OK.

APPENDICES

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APPENDIX A. Ammonium-N, (mg kg^{-1}) in the surface soil horizon (0-15 cm), Stillwater, Oklahoma.

Replication	Treatment	Days before and after fertilization(September 9/92) and $\text{NH}_4\text{-N}$.					
		-1	25	35	63	87	134
1	1	8.272	6.44	4.995	3.826	2.755	4.688
2	1	4.054	17.546	14.890	9.765	9.267	5.691
3	1	8.920	28.995	17.814	20.473	19.573	5.774
4	1	9.459	40.831	46.551	29.507	24.838	9.206
5	2	7.590	5.366	19.975	3.683	2.430	2.178
6	2	12.195	7.707	9.938	14.309	6.829	3.435
7	2	5.993	17.138	26.250	24.738	16.912	7.801
8	2	12.258	21.525	4.424	61.480	56.998	21.226
9	3	9.584	13.188	6.310	5.892	3.259	3.112
10	3	9.200	10.577	11.516	7.581	3.665	3.281
11	3	11.106	16.743	28.102	28.796	21.765	9.139
12	3	18.170	13.378	35.763	32.228	44.223	15.031
13	4	17.593	5.049	3.822	4.526	2.650	2.774
14	4	9.861	8.922	15.830	11.410	5.825	3.801
15	4	9.282	15.236	23.811	26.457	9.669	8.083
16	4	11.876.	28.149	50.580	48.488	34.987	15.742

APPENDIX B. Nitrate-N, (mg kg⁻¹) in the surface soil horizon (0-15 cm), Stillwater, Oklahoma.

Replication	Treatment	Days before and after fertilization (September 9/92) and NO ₃ -N.					
		-1	25	35	63	87	134
1	1	5.979	12.735	5.862	4.784	4.047	1.257
1	2	13.165	42.090	13.299	7.945	17.068	4.794
1	3	9.722	57.283	18.466	12.175	21.539	7.504
1	4	5.025	44.156	23.258	11.990	28.056	11.205
2	1	8.370	7.177	17.580	2.836	3.381	0.965
2	2	8.695	20.728	12.248	12.029	13.695	4.546
2	3	8.134	34.396	20.383	16.858	24.574	11.377
2	4	17.785	50.465	6.374	15.896	31.429	13.021
3	1	5.850	35.837	6.924	4.482	5.895	1.346
3	2	10.487	26.807	15.326	9.702	9.501	2.553
3	3	11.961	29.870	20.354	11.425	19.922	5.668
3	4	4.843	39.200	21.098	11.492	26.825	12.223
4	1	8.799	10.025	5.619	4.006	4.174	1.204
4	2	5.877	18.098	14.519	10.791	14.360	2.667
4	3	8.646	29.914	20.192	11.054	16.817	9.617
4	4	10.400	44.730	26.281	12.847	26.221	14.745

APPENDIX C. Ammonium-N, (mg kg^{-1}) in the surface soil horizon (0-15 cm), Lahoma, OK.

Replication	Treatment	Days before and after fertilization(August 28/92), and $\text{NH}_4\text{-N}$.							
		-1	11	25	36	42	76	88	142
1	2	5.314	7.290	.	4.995	4.624	5.142	3.515	0.2790
1	3	4.991	12.333	0.193	6.307	4.367	3.807	4.458	0.2637
1	4	3.990	29.676	0.683	7.721	6.128	3.992	2.958	0.3427
1	5	2.779	14.442	4.043	11.717	10.424	8.930	3.196	0.4316
1	6	7.559	49.819	8.182	12.930	17.471	9.586	5.470	0.4023
1	7	2.649	24.223	16.319	23.178	30.904	22.954	8.865	0.6625
2	2	3.060	11.124	.	5.005	3.369	3.223	2.164	0.2311
2	3	2.383	17.617	2.275	4.596	5.402	2.598	3.450	0.2357
2	4	3.062	15.010	4.172	8.391	7.734	5.898	4.581	0.2745
2	5	1.540	15.315	9.653	14.182	14.506	6.907	2.812	0.2735
2	6	5.302	22.188	22.054	15.262	23.104	16.644	6.069	0.4811
2	7	3.762	29.560	22.992	35.616	31.272	34.993	16.963	0.8880
3	2	3.291	5.613	0.344	3.892	4.407	3.406	2.633	0.2933
3	3	5.927	7.367	1.382	4.643	5.699	3.232	3.553	0.2084
3	4	6.461	5.717	4.970	9.664	11.173	7.151	4.379	0.4664
3	5	3.141	15.035	8.856	11.507	17.225	9.342	5.783	0.3112
3	6	4.126	34.791	12.265	14.313	16.287	18.003	6.179	0.5363
3	7	7.717	19.964	29.153	17.326	25.683	18.295	12.084	1.0496
4	2	3.940	8.006	1.087	5.816	4.153	5.788	3.372	0.4872
4	3	4.435	10.159	0.813	8.679	17.369	6.848	3.668	0.6047
4	4	8.450	10.078	1.937	7.254	6.227	6.868	3.817	0.4588
4	5	3.762	14.900	8.280	13.622	8.863	12.992	5.234	0.5369
4	6	5.486	19.278	7.239	19.015	8.864	15.543	5.197	0.3572
4	7	5.286	25.138	16.925	28.254	30.699	27.516	11.621	0.6967

APPENDIX D. Nitrate-N (mg kg^{-1}) in the surface soil horizon (0-15cm), Lahoma, Oklahoma.

Replication	Treatment	Days before and after fertilization(August 28/92), and $\text{NO}_3\text{-N}$.							
		-1	12	26	37	43	77	89	142
1	2	9.525	12.462	12.492	18.183	6.237	3.525	2.092	0.695
1	3	9.982	19.698	22.226	33.958	13.160	3.920	2.196	0.942
1	4	9.091	30.602	25.681	33.167	11.716	4.568	2.322	1.181
1	5	17.805	43.556	41.482	56.726	20.168	8.509	12.251	2.489
1	6	19.994	39.662	40.090	50.064	22.507	7.438	8.172	2.691
1	7	24.543	54.440	50.645	64.939	25.907	10.784	7.223	3.356
2	2	10.304	7.709	11.646	14.947	5.034	2.432	1.930	0.714
2	3	9.949	16.259	22.005	28.970	10.888	3.982	2.789	0.876
2	4	11.220	25.873	31.910	39.418	15.657	5.581	2.962	2.060
2	5	9.579	29.832	34.790	42.691	15.219	4.253	2.806	1.565
2	6	22.062	47.505	49.475	59.457	24.213	6.923	15.259	3.420
2	7	25.169	53.220	58.559	70.851	29.828	10.659	16.118	4.978
3	2	6.163	6.897	14.254	13.283	6.050	2.757	1.626	0.937
3	3	10.606	17.658	24.343	30.826	13.674	5.232	2.162	1.481
3	4	20.026	28.166	34.933	46.866	20.304	6.856	3.518	1.584
3	5	14.989	39.856	41.205	46.376	22.147	5.952	4.336	2.268
3	6	15.841	42.055	43.803	44.492	18.384	4.513	4.723	2.715
3	7	15.036	45.348	51.768	56.920	21.221	6.66	4.960	5.518
4	2	7.674	8.655	9.161	13.722	4.916	2.945	1.668	1.160
4	3	11.065	26.082	24.143	30.540	22.317	4.620	2.175	1.755
4	4	12.014	25.282	25.675	33.641	11.638	5.750	4.108	2.391
4	5	15.765	40.645	43.018	51.495	14.132	6.815	4.855	3.469
4	6	20.292	44.740	41.151	57.727	13.328	7.917	5.156	3.450
4	7	20.379	57.563	49.575	60.857	23.803	10.195	17.633	4.663

APPENDIX E. Maximum daily temperature(°F), Stillwater, Oklahoma

Day of Month	Sept	October	November	December	January
1	87	81	36	43	50
2	79	83	39	33	52
3	86	87	27	35	48
4	88	88	37	42	61
5	84	76	50	53	58
6	85	70	52	55	57
7	79	66	57	66	50
8	91	82	43	60	63
9	91	82	43	60	46
10	92	86	58	67	46
11	92	81	56	64	54
12	91	92	39	63	59
13	91	86	62	56	55
14	88	84	68	49	37
15	83	73	60	55	42
16	85	72	62	40	23
17	79	80	63	54	44
18	80	88	66	54	50
19	57	83	73	44	47
20	64	64	53	37	48
21	66	69	57	43	58
22	70	80	68	40	-
23	73	85	54	51	-
24	72	88	46	50	-
25	70	85	52	51	-
26	80	62	48	52	-
27	78	72	60	49	-
28	79	75	62	54	-
29	80	74	75	45	-
30	78	47	73	52	-
31		42		55	-

- Not included in the period of study

APPENDIX F. Precipitation data, Stillwater (inches), Oklahoma.

Day of Month	Period of study, September 9, 1991 through January 21, 1992				
	September	October	November	December	January
1	1.62	0	0.45	0	0.20
2	1.50	0	0	0.27	0
3	0	0	0.17	0	0
4	0	0	0	0	0
5	0	0	0	0	0
6	0	0	0	0	0
7	0.07	0	0	0	0.02
8	0.15	0	0.02	0	0
9	0	0	0	0	0
10	0	0	0	0	0
11	0	0	0	0	0
12	0	0	0	1.27	0
13	0	0	0	0	0.21
14	0.04	0	0.11	0	0.08
15	0.23	0	0.14	0	0
16	0.27	0	0.71	0	0
17	0.03	0	1.01	0	0
18	0.89	0	0	0	0
19	0.37	0	0	0.49	0
20	0	0	0.09	2.46	0
21	0	0	0	0	0
22	0.22	0	0	0.10	
23	0.2	0	0	0.35	
24	0	0	0	0	
25	0.11	0	0	0	
26	0	2.25	0	0	
27	0	0	0	0	
28	0	1.50	0	0.16	
29	0	0	0.02	0	
30	0	0	0	0	
31		0.51		0	

APPENDIX G. Maximum daily temperature (°F), Lahoma, Oklahoma

Day of Month	September	October	November	December	January
1	89	82	35	36	51
2	79	85	36	41	50
3	81	92	22	38	51
4	86	93	33	44	na
5	81	68	48	50	na
6	83	69	51	54	na
7	87	67	55	63	48
8	84	83	55	70	59
9	90	85	41	59	45
10	94	85	54	58	45
11	94	82	42	63	52
12	94	91	40	60	57
13	93	84	61	60	56
14	92	87	67	58	40
15	86	70	65	41	43
16	86	70	51	50	24
17	74	83	48	59	48
18	77	90	62	47	-
19	57	90	68	40	-
20	65	68	53	38	-
21	63	67	56	41	-
22	74	82	61	55	-
23	73	85	47	48	-
24	72	90	46	48	-
25	69	73	48	45	-
26	86	53	44	49	-
27	79	63	57	45	-
28	82	74	59	41	-
29	85	74	74	41	-
30	82	54	70	49	-
31		35		45	-

na Not available, - Not included in the period of study

APPENDIX H. Precipitation data (inches), Lahoma, Oklahoma.

Day of Month	Period of study, September 1, 1991 through January 21, 1992				
	September	October	November	December	January
1	0.68	0	0	0	0
2	0.26	0	0.01	0	0
3	0	0	0.01	0	0
4	0.26	0	0	0	0
5	0	0	0	0	0
6	0	0	0	0	0
7	0.02	0	0.01	0	0
8	0.08	0	0	0	0
9	0	0	0	0	0
10	0	0	0	0	0
11	0	0	0	0	0
12	0	0	0	1.04	1.04
13	0.05	0	0	0	0
14	0.02	0	0	0	0
15	0	0	0.24	0	0
16	0.56	0	0.71	0	0
17	0.03	0	1.27	0	0
18	0.07	0	0	0	0
19	0.08	0	0.08	0.70	0.70
20	0	0	0.43	0.93	0.93
21	0.09	0	0	0.14	0.14
22	0.03	0	0	0.17	
23	0	0	0	0	
24	0.04	0.03	0	0	
25	0.02	0	0	0	
26	0	0	0	0	
27	0	0	0	0.12	
28	0	0.95	0	0.04	
29	0	0	0	0	
30	0	0	0	0	
31		0.54		0	

VITA 

Edgar Noel Ascencio

Candidate for the Degree of
MASTER OF SCIENCE

Thesis: SEQUENTIAL MEASUREMENTS OF SOIL $\text{NH}_4\text{-N}$ AND $\text{NO}_3\text{-N}$ FROM
TWO LONG TERM FERTILITY EXPERIMENTS WITH VARIABLE N
RATES

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