

# RELATIONSHIP BETWEEN MINERALIZABLE N AND HOT KCl EXTRACTABLE NH<sub>4</sub>-N - VARIABILITY AT ONE SITE

F. Sellesl, C.A. Campbell, G. Wenl, D. Messerl and S. Brandt<sup>2</sup>

<sup>1</sup>Agriculture and Agri-Food Canada, Semiarid Prairie Agricultural Research Centre, Swift Current, SK S9H 3X2

<sup>2</sup>Agriculture and Agri-Food Canada, Experimental Farm, Scott, SK S0K 4A0

## ABSTRACT

To characterize the N supplying capacity of the soil at a site where a new long-term experiment was being initiated at Scott, Saskatchewan, we soil sampled the site in a systematic manner, taking 160 cores. We used these cores to measure mineralizable N in the 0-7.5 and 7.5-15 cm depths, by 24 wk laboratory incubation at 35°C and optimum moisture. We also extracted NH<sub>4</sub>-N from the soils by digesting with hot 2M KCl at 100°C for 4 hr, followed by distillation. Using a geostatistic procedure, and correlation and regression analysis, we demonstrated the existence of a close association between the N supplying power of the soil, estimated by biological vs chemical extraction techniques. Kriging was used to show that these variables were similarly distributed in space over the landscape of the experimental site, and was also closely associated with total soil organic N. We concluded that the hot KCl extracted NH<sub>4</sub>-N should prove to be a useful index for use in quantifying the N supplying capacity of soils. Further, it may allow more accurate estimates of fertilizer N requirements to be made by soil testing laboratories. As well, this new index may prove useful to scientists interested in researching precision farming because it offers a quick laboratory method of quantifying N supplying capacity of soils and its distribution in the field.

## INTRODUCTION

In spring 1994, a new long-term experiment (Fig. 1) was initiated on the Experimental Farm at Scott, Saskatchewan, to evaluate the impact of cropping diversity and input levels on various agronomic and soil quality characteristics. To assess future changes due to the treatments, it was necessary to characterize the soil characteristics. One of the soil characteristics we measured was its N supplying capacity. We conducted this analysis using a biological technique (long-term incubation in the laboratory) and a chemical extraction technique which would be much faster (Jalil et al. 1996) and thus may be appropriate for use in a soil testing laboratory.

We require a good understanding of the underlying distribution of soil properties that were likely to be affected by the treatments in each experimental unit. However, the cost of sampling individual plots with an appropriate number of samples to characterize the state of each plot and the dispersion of the data, precluded an exhaustive sampling of the site. Instead, an alternative sampling protocol was designed with a pseudo random distribution of samples in the field, that covered a range of distances among sampling points (Fig. 2). This sampling strategy allowed determination of the spatial structure of the variance in the field by means of geostatistical techniques. Kriging procedures could then be used to obtain mean and standard error estimates of the various properties for each parameter in each experimental plot.

The objective of this study was to determine (i) the N supplying power of the test site by biological and chemical techniques, (ii) to relate the results obtained by these two methods, (iii) to relate the N supplying capacity as measured by these methods to total organic matter in the soil, and (iv) to establish the statistical and spatial distribution of the parameters assessed in this study.

## MATERIALS AND METHODS

In June 1994 we sampled the experimental site at Scott using the experimental grid shown in Figure 2. We drilled 160 holes to provide samples of 0-7.5 and 7.5- 15 cm soil layers. When soil samples were taken, field operations, including fertilizing (urea banded) and seeding, had already been done. The soils were brought back to the lab. They were screened (< 2 mm), air-dried, labelled, and stored for analysis.

The soils were used to determine the potentially mineralizable N (No) and rate constant (k) by incubating at 35 °C for 24 wk with intermittent leaching of the mineral N formed, using dilute  $\text{CaCl}_2$  followed by a minus N nutrient solution (Campbell et al. 1993). A second sample of each soil was extracted with 2M KCl, heated to 100 °C for 4 hr, followed by steam distillation to collect extracted  $\text{NH}_4\text{-N}$  (Jalil et al. 1996). Total organic C and N were determined by grinding soil to < 153  $\mu\text{m}$  and measuring C and N with an automated combustion technique (Carlo Erba™, Milan, Italy).

### Theory of Geostatistics

The first step in the analysis of spatial variability is to determine the spatial dependence of the variance. The semivariogram is one of the major tools used for analysis of spatial variability of regionalized variables. A basic assumption for the use of semivariograms is that the regionalized variable  $\mathbf{Z}(\mathbf{x})$  must have second order stationarity (Clark 1979). That is, the expected value of the variable  $\mathbf{Z}(\mathbf{x})$  is independent of location  $\mathbf{x}$ , and the covariance for each pair of variables [ $\mathbf{Z}(\mathbf{x})$ ,  $\mathbf{Z}(\mathbf{x} + \mathbf{h})$ ] exists, and depends only on the separation  $\mathbf{h}$  between variables (Vieira et al. 1983).

A semivariogram allows us to split the variance into a nugget ( $C_0$ ) component and a spatial component ( $C_s$ ). The random component of the variance is made up of (i) spatial structures occurring at distances smaller than the sampling interval, (ii) micro heterogeneity, and (iii) experimental error. The spatial or structural component of the variance is the proportion of the variance that can be explained by the distance or lag between sampling points. The sum of  $C_0$  and  $C_s$  in semivariograms that achieve a steady value of  $C_s$  (Sill) equals the variance of the sample. Thus, in a purely random sample (no spatial structure) the nugget accounts for all the variance while in a spatially dependent variable, a large proportion of the variance will be explained by  $C_s$  with the remainder explained by  $C_0$ .

The second step was to estimate values for the variables at points where no samples exist. This is usually achieved by the use of Kriging. Kriging involves use of a weighted-moving-average interpolation procedure, where the weights assigned to samples minimize the estimation variance. The latter is computed as a function of the variogram model, and the locations of the samples relative to each other and to the point being estimated (Clark 1979). To determine the goodness of fit of the kriging procedure we used "jackknifing", which is a cross validation procedure where each sampling point is estimated based only on neighbouring points, and the estimated points are compared to their original values (Vieira et al. 1983).

[Note: Because urea was band applied just a few days prior to our sampling, the results for 0-7.5 cm depth were fairly "noisy" (Campbell et al. 1996b); consequently, we only analyze and show results for the 7.5-15 cm depth which was rarely affected by the fertilizer.]

## RESULTS AND DISCUSSION

The cumulative N mineralized in 24 wk was analyzed to estimate potentially mineralizable N (No), the rate constant (k) and the initial potential rate of mineralization (**N<sub>0</sub>k**) as described by Campbell et al. (1993). We then made a first estimate of the variability of NO, **N<sub>0</sub>k** and the hot KC1 **NH<sub>4</sub>-N** by conducting an analysis of variance using the 3 input levels and 4 blocks (Fig. 1) as the variables. The 3 input levels are randomized within the 4 blocks. This analysis indicated that these three variables were highly heterogeneous, showed significant effects of block, input levels, and block x input interaction (Table 1).

We developed relative semivariograms for the 3 variables. These showed that a large proportion of the variance was due to random variability (65 to 74%) and the rest due to spatial structure (Fig. 3). Interestingly, the semivariogram for **N<sub>0</sub>k** had a similar spatial structure to that obtained for Kjeldahl N (Selles et al. 1996). The relative semivariogram of **N<sub>0</sub>k** rose gradually from the nugget of 0.74 to reach the sill at lags of about 160 m (Fig. 3b). At lags beyond 300 to 350 mm there was a polynomial trend indicating a change of domain at these lags. This trend was also present in the semivariograms for **N<sub>0</sub>** and hot KC1 **NH<sub>4</sub>-N**, although they are not shown on Fig. 4 because the lags of interest for the latter two variables are much shorter than for **N<sub>0</sub>k**. It is possible that this polynomial trend is a reflection of a change in soil association.

Indeed, while the soil in the majority of the experimental area is a Scott loam, a substantial area in the southwest corner is an Elstow loam, and there are minor inclusions of Weyburn loam at the southeast end and in the northeast corner.

Because the proportion of the variance explained by spatial structure was small, the jackknifing procedure showed a low, though significant ( $P < 0.05$ )  $r^2$  value for NO and hot KC1 **NH<sub>4</sub>-N** ( $r^2 = 0.24$ ) and for **N<sub>0</sub>k** ( $r^2 = 0.37$ ). Nonetheless, when the kriged estimates were grouped by block and input level, as was done for data measured with core sampling, the means of the kriged estimates were closely correlated with the measured means (Fig. 4). Further, the coefficients of variability of the kriged estimates were much lower than those of the measured means (data not shown).

Field maps prepared using the kriged values of the three variables (NO, **N<sub>0</sub>k** and hot KC1 **NH<sub>4</sub>-N**) (Figs. 5 and 6), showed that these variables were similarly distributed throughout the landscape. For example, the lowest values were located in the northeast corner of the field and the highest values in the middle of the western border. Further, these three variables appeared to be closely associated with values of organic N at the same depth (Fig. 7).

Regression and correlation analysis showed that there was a close association between the measured NO and hot KC1 **NH<sub>4</sub>-N** ( $r^2 = 0.64$ , significant at  $P < 0.0001$ ), and **N<sub>0</sub>k** vs hot KC1 **NH<sub>4</sub>-N** ( $r^2 = 0.62$ , significant at  $P < 0.0001$ ). The relationship between the cumulative amount of N mineralized in 24 wk (y) and hot KC1 **NH<sub>4</sub>-N** (x) was even more precise ( $y = -35.1 + 8.42 x$ ;  $r^2 = 0.73$ , significant at  $P < 0.0001$ ).

Because these results show a close association between the chemical estimate of N supplying power (hot KC1 **NH<sub>4</sub>-N**) and the biological estimates (NO, **N<sub>0</sub>k**, cumulated **N<sub>min</sub>**), and because we have demonstrated a close association between grain yield and the N supplying power of soil measured as **N<sub>0</sub>k** (Campbell et al. 1996a), it appears that the hot KC1 **NH<sub>4</sub>-N** might be a very useful index for quantifying the potential of soils to mineralize N during a growing season. Further, as shown before (Jalil et al. 1996), it is possible to estimate NO from the hot KC1 **NH<sub>4</sub>-N**.

Thus, we should be able to use such relationships, together with historical weather data, to calculate probable amounts of N a soil will mineralize during a growing season, using the relationship:  $N_{\min} = N_0 (1 - \exp(-kt))$ , where  $k \approx 0.06 \text{ wk}^{-1}$  (Jalil et al. 1996) and  $t$  is time in weeks with  $k$  modified for moisture and temperature as discussed by Campbell et al. (1994).

Finally, the success of our spatial analysis suggest that the hot KCl  $\text{NH}_4\text{-N}$  might provide a powerful index for those interested in quantifying the N supplying power of soils in precision farming. Used with a  $\text{NO}_3\text{-N}$  test for initial fertility, this test should improve the accuracy with which we are able to estimate N provided by the soil and thus the amount that should be applied as fertilizer.

#### ACKNOWLEDGEMENTS

The authors wish to thank D. Hahn, R. Ljunggren and T. Campbell for technical assistance. This study was funded under the Canada-Saskatchewan Agriculture Green Plan Agreement.

#### REFERENCES

- Campbell, C.A., Ellert, B.H. and Jame, Y.W. 1993. Nitrogen mineralization in soils. p. 341-349 *In* M.R. Carter (Ed.). Can. Soc. Soil Sci. Soil Sampling and Analytical Methods. Lewis Pub., Boca Raton, U.S.A.
- Campbell, C.A., Jame, Y. W., Akinremi, O.O. and Beckie, H.J. 1994. Evaluating potential nitrogen mineralization for predicting fertilizer nitrogen requirements of long-term field experiments. p. 81-100 *In* J. Havlin, J. Jacobson and P. Fixen (eds.) Soil Testing: Prospects for improving nutrient recommendations, SSSA Special Pub. 40.
- Campbell, C.A., Lafond, G.P., Harapiak, J.T. and Selles, F. 1996(a). Relative cost to soil fertility of long-term crop production without fertilization. Can. J. Plant Sci. 76: 401-406.
- Campbell, C.A., Selles, F. and Messer, D. 1996(b). Estimating N mineralization of soil using the hot KCl extracted  $\text{NH}_4\text{-N}$  technique. p. 49-50. *In* B.L. Frick (ed.) Alternative crop production systems project workshop proceedings, Saskatoon, SK. April 22, 1996.
- Clark, I. 1979. Practical geostatistics. Elsevier Applied Science. London. 129 pp.
- Jalil, A., Campbell, C.A., Schoneau, J., Henry, J.L., Jame, Y.W. and Lafond, G.P. 1996. Assessment of two chemical extraction methods as indices of available N. Soil Sci. Soc. Am. J. 60: 1954-1960.
- Selles, F., Campbell, C.A., Moulin, A. and McConkey, B.G. 1996. Spatial evaluation of soil properties of experimental site. p. 35-45 *In* B.L. Frick (ed.) Alternative crop production systems project workshop proceedings, Saskatoon, SK. April 22, 1996.
- Vieira, S.R., Hatfield, J.L., Nielsen, D.R. and Biggar, J.W. 1983. Geostatistical theory and application to variability of some agronomic properties. Hilgardia 5 1: 1-75.

Table 1. Distribution of  $N_0$ ,  $N_0k$  and hot  $KCl NH_4-N$  in main plots

Variables	BLOCK											
	1			2			3			4		
	Input Level <sup>1</sup>											
	Organic			Reduced			High			Reduced		
	Ave	Max	Min	Ave	Max	Min	Ave	Max	Min	Ave	Max	Min
$N_0$	53.6	141	26	69.7	186	29	65.1	390	17	54.4	209	15
$N_0k$	5.8	10	4	5.4	8.6	3	7.7	55	2	3.5	7	2
$HKCl^2$	10.9	17.4	6.3	11	16.4	6.7	9	12.5	5.1	9.1	14.4	5.9
n	14			13			13			14		
	Reduced			High			Organic			High		
$N_0$	108.2	257	33	74.5	223	27	89.6	185	22	51.4	76	27
$N_0k$	9.4	15	5	7.5	17	3	7.3	13	3	6.3	9	3
$HKCl$	14.3	23.4	6.1	11.7	18.4	6.4	12.5	23.9	7.3	9.5	13.4	4.7
n	14			13			13			14		
	High			Organic			Reduced			Organic		
$N_0$	73.4	145	25	79	212	27	60.5	132	14	76.5	224	18
$N_0k$	7.9	15	4	8.4	19	3	6.8	13	2	8.6	20	3
$HKCl$	13.5	19.8	9.3	13.2	23.4	6.3	12	20.1	7.7	13.7	25.9	8.4
n	14			12			12			14		

1 Position of input level on table reflects position of treatment in the field

2  $HKCl$  = hot  $KCl NH_4-N$ .

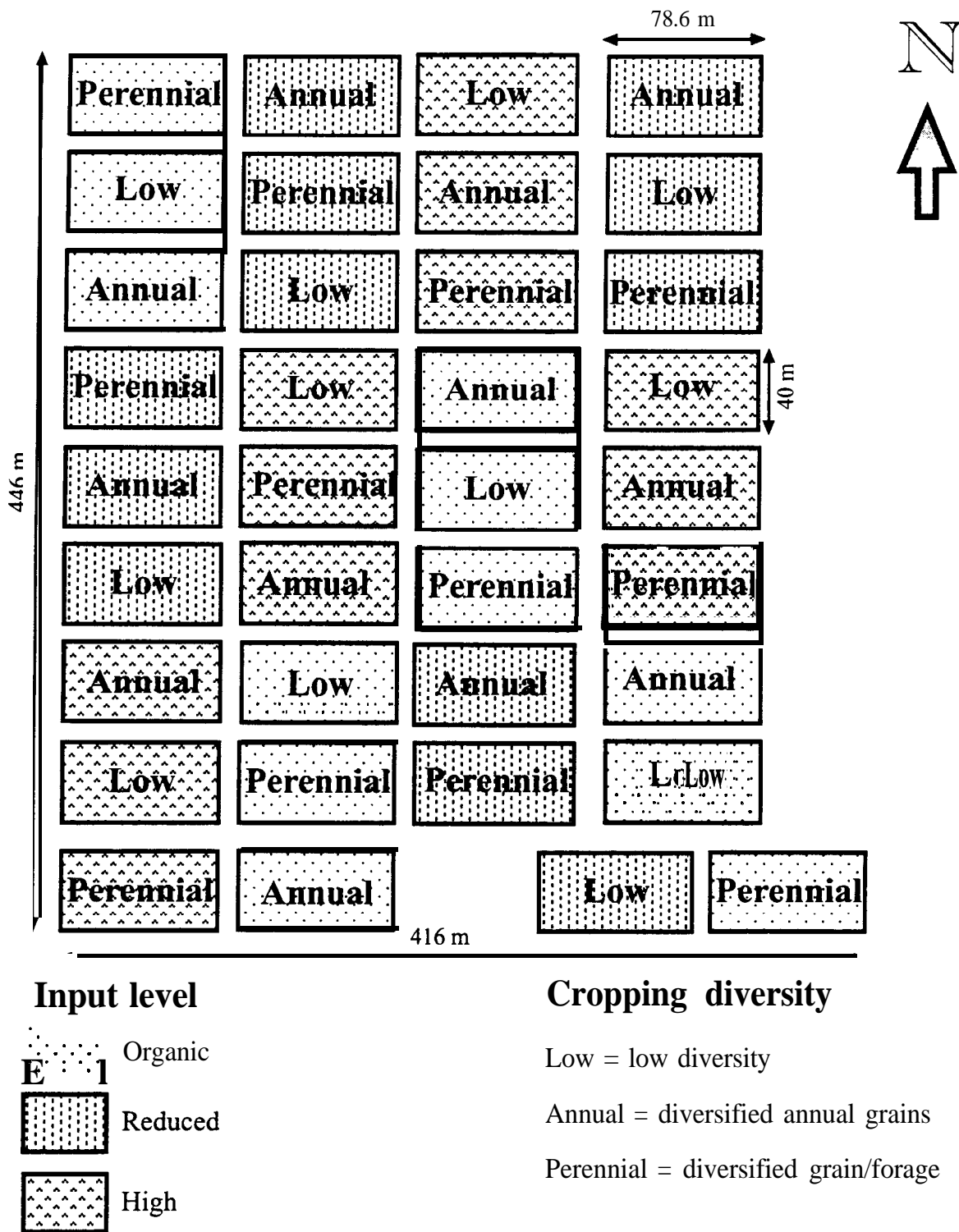
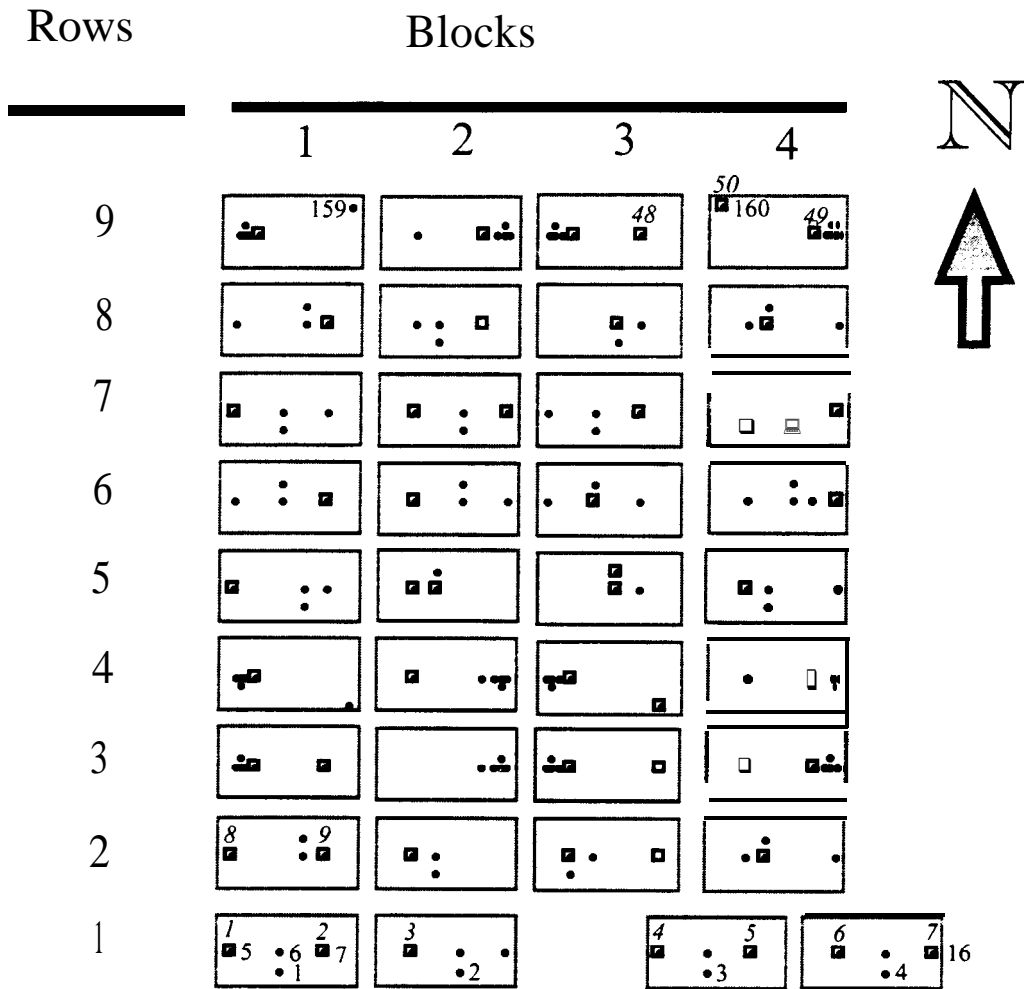


Figure 1. Experimental layout



- Circles indicate location of 0- 120 cm cores (numbers 1 to 160)  
Core number plain number beside symbol
- ◻ Squares indicate location of 0-240 cm cores (numbers 1 to 50)  
Core number in italics above symbol

**Figure 2.** Location of sampling points in the field

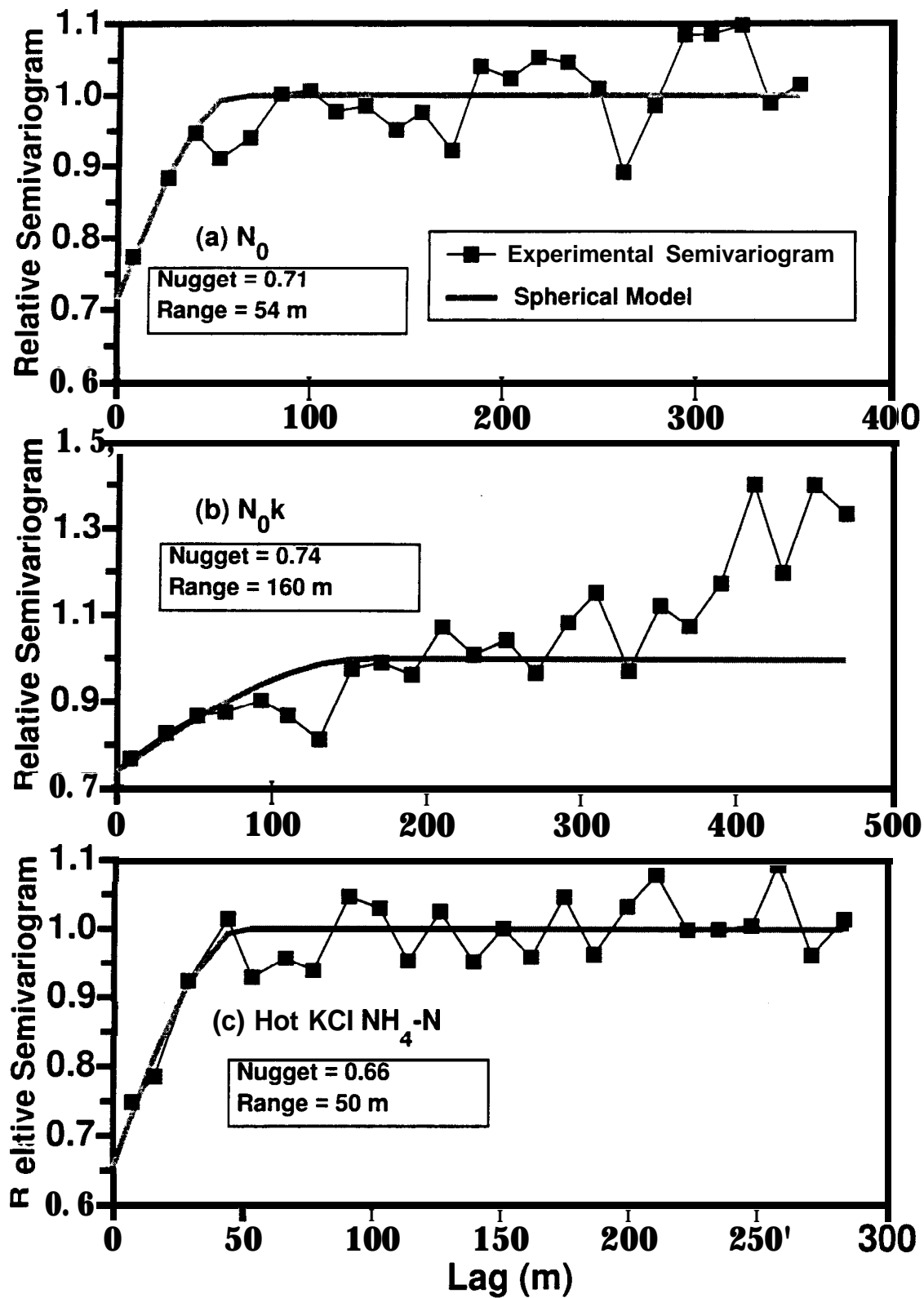
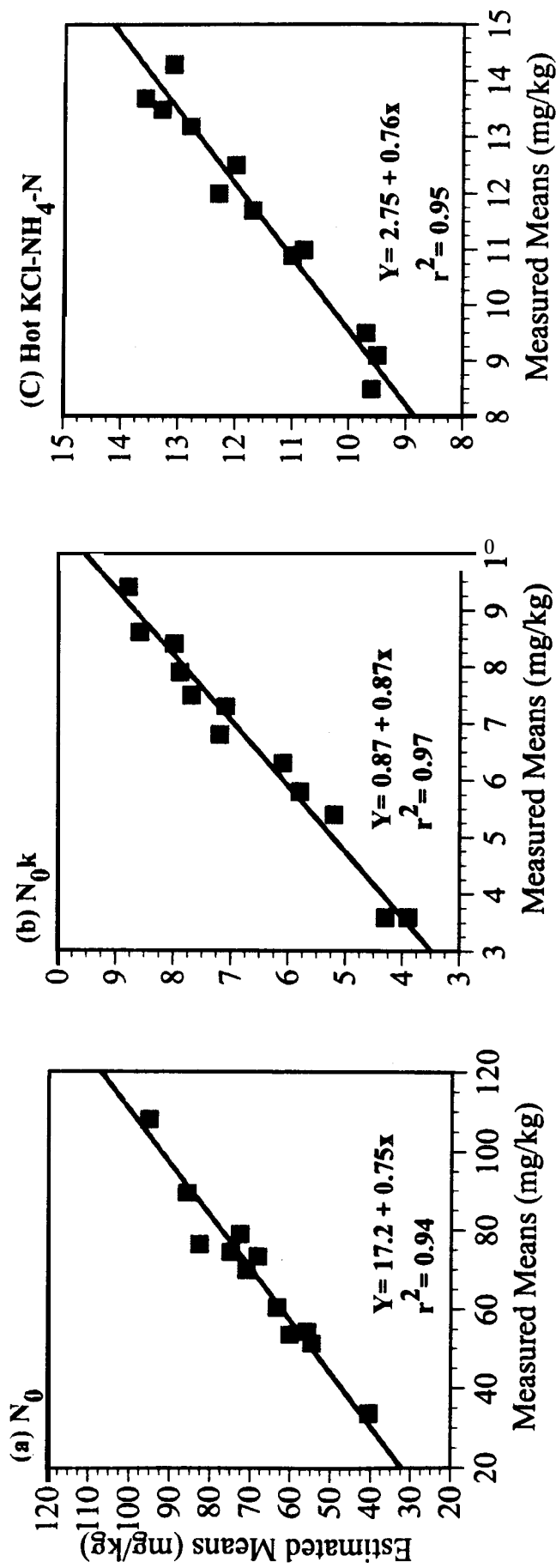
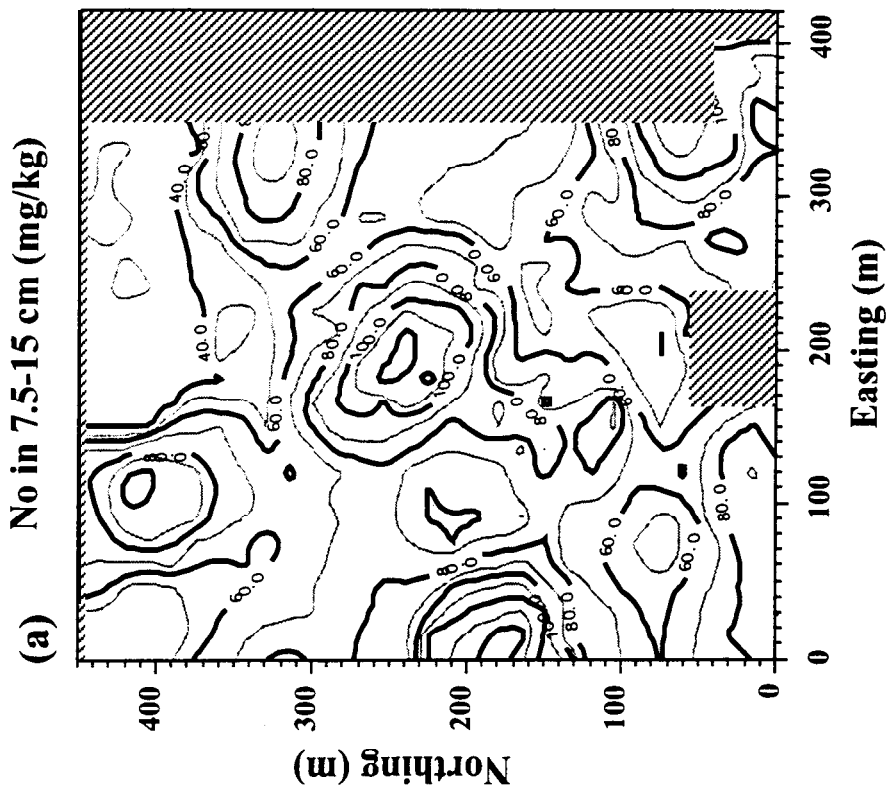
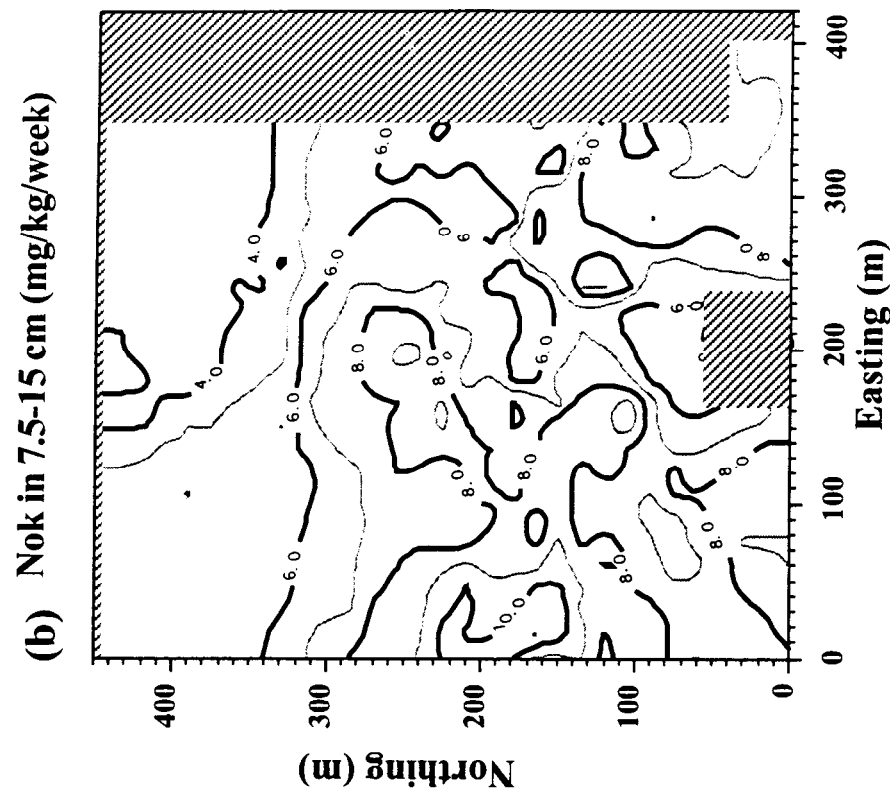


Figure 3. Relative semivariograms for  $N_0$  (a),  $N_0k$  (b), and hot  $KCl NH_4-N$  (c).

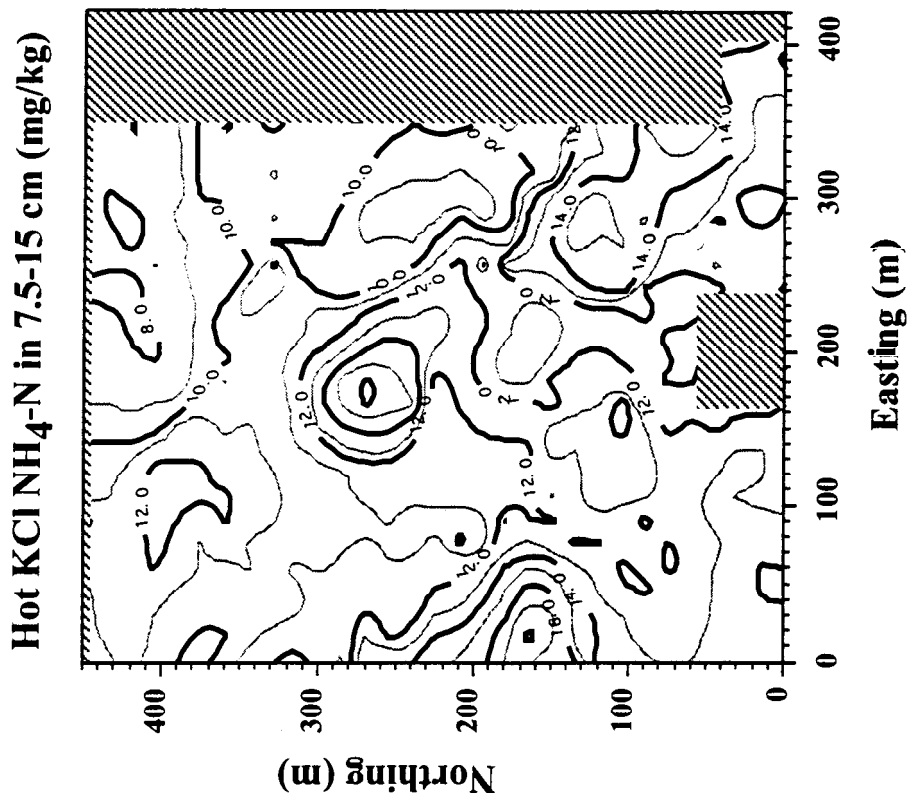




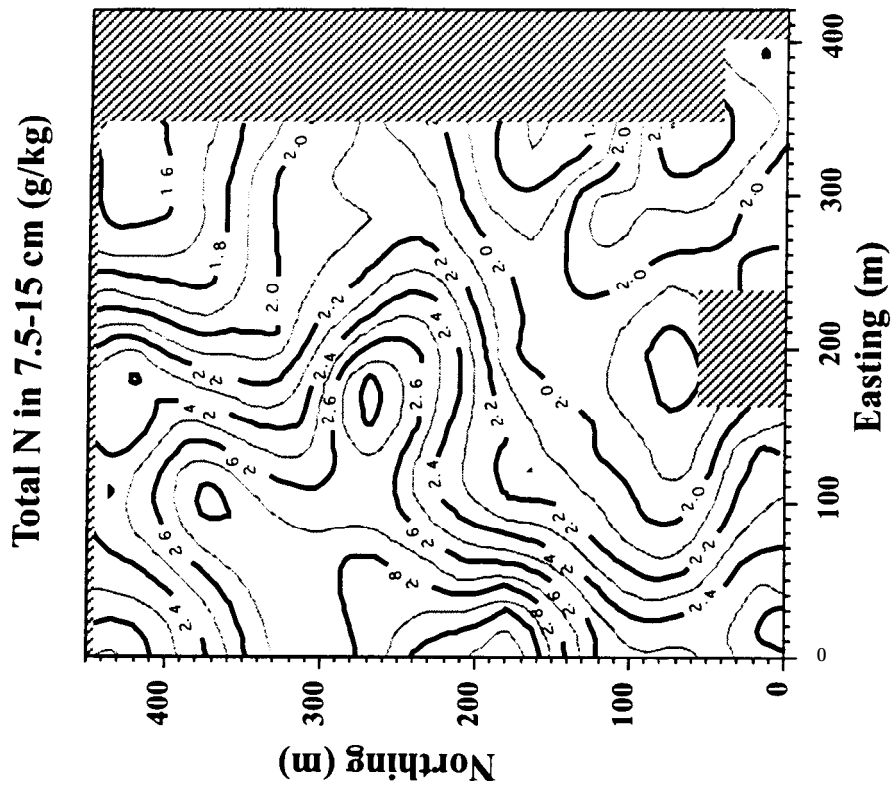
**Figure 4.** Correlations between measured means and means of kriged estimates for  $N_0$  (a),  $N_{0k}$  (b), and hot KCl  $NH_4-N$  (c) at the 7.5 -15 cm depth.



**Figure 5.** Contour map showing the distribution of No (a) and Nok (b) in the experimental site at the 7.5 - 15 cm depth (based on kriged estimates).



**Figure 6.** Contour map showing distribution of Hot KCl-NH<sub>4</sub>-N in the experimental site at the 7.5 - 15 cm depth (based on kriged estimates)



**Figure 7.** Contour map showing the distribution of kjeldahl N at the 7.5 - 15 cm depth (from Selles et al., 1996)