

**Evaluation of potassium soil tests and methods for mapping soil fertility properties in
Iowa corn and soybean fields**

by

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This is to certify that the doctoral dissertation of

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has met the dissertation requirements of Iowa State University

Major Professor

For the Major Program

To:

My wife Marina

My son Ignacio and daughter Maite

TABLE OF CONTENTS

CHAPTER 1. GENERAL INTRODUCTION	1
Introduction	1
Dissertation Organization	4
 CHAPTER 2. IMPACT OF SAMPLE DRYING ON SOIL POTASSIUM EXTRACTION AND FIELD CALIBRATION OF A TEST BASED ON FIELD- MOIST SOIL SAMPLES FOR CORN AND SOYBEAN	 5
Abstract	5
Introduction	6
Materials and Methods	9
Results and Discussion	14
Conclusions	22
References	23
 CHAPTER 3. FIELD CALIBRATION OF AMMONIUM ACETATE, MEHLICH-3, AND SODIUM TETRAPHENYL-BORON SOIL POTASSIUM TESTS FOR CORN AND SOYBEAN	 44
Abstract	44
Introduction	45
Materials and Methods	49
Results and Discussion	53
Conclusions	60
References	62
 CHAPTER 4. INTEGRATING GEOSTATISTICS AND GIS TO STUDY SPATIAL VARIABILITY AND MAP SOIL FERTILITY PROPERTIES IN IOWA FIELDS	 79
Abstract	79
Introduction	80
Materials and Methods	83
Results and Discussion	86
Conclusions	90
References	91
 CHAPTER 5. GENERAL CONCLUSIONS	 108
 ACKNOWLEDGMENTS	 111

CHAPTER 1. GENERAL INTRODUCTION

INTRODUCTION

Potassium is an essential nutrient for plant growth, and one of the three main macronutrients together with N and P. Potassium is present in the soil in water-soluble, exchangeable, nonexchangeable, and mineral forms. These four forms of K give a general representation of the potential sources for plant-available K, but no distinct boundaries exist among them. Plants take up K from soil solution, which is readily replenished by soil exchangeable K. Some nonexchangeable K can become exchangeable when solution and exchangeable K are depleted by plant removal, leaching, or exchange reactions with other cations. However mineral K, which is the major proportion of total K in soils, can become available only very slowly through long-term soil weathering. Distribution of K among these forms also occurs as K is added to soil as fertilizer, manure, or crop residues. It has long been recognized that wetting-drying and freezing-thawing cycles influence transformations of K between nonexchangeable, exchangeable, and solution phases. Soils initially high in exchangeable K tend to fix K upon drying while those with initially low exchangeable K levels release K upon drying. Freezing of moist soils has the same effect that drying the soil.

Soil testing is the most commonly used diagnostic tool in production agriculture to assess plant K availability in soils. Predicting plant available soil K has proven to be a difficult task due to the complexity of the dynamic equilibrium among the various forms of soil K. The estimate of soil exchangeable K with the ammonium-acetate extractant from air-dried or oven-dried soil samples is the most widely used method to predict plant-available K. Iowa research in the 1960s showed that soil K extracted with the ammonium-acetate extractant from field-moist samples was better correlated with crop K uptake than K extracted from dried soil samples. Therefore, a field-moist based K test was field calibrated and implemented at that time. However, no other private or University laboratory adopted

this test because it was a complicated laboratory procedure. In 1988 Iowa State University decided to abandon the field-moist based test and to support the commonly used test based on dried samples. A correction factor based on the average additional amount of K extracted from dried Iowa soil samples compared with moist samples was used to adjust the field calibrations. Observations of K deficiency symptoms in crops and unpublished research since the early 1990s has suggested that this test provides a poorer prediction of crop response to fertilization than the field-moist test, and that the adjusted interpretations needed to be improved. The Mehlich-3 extractant began to be rapidly adopted by soil testing laboratories in recent years because it can also be used to extract other nutrients from the soil. However, this test seems to provide similarly poor predictions of crop response to applied K because it relies on a similar extraction mechanism. New field calibration research at many locations could provide improved interpretations for these tests based on dried samples or, alternatively, the field-based information needed to demonstrate that a test based on field-moist samples should be implemented.

Drying soil samples is a factor that can influence soil exchangeable K measurements and partly explain the poor performance of currently used ammonium-acetate and Mehlich-3 tests for K. However, avoidance of soil sample drying still may not result in a better index of K availability for crops when significant quantities of nonexchangeable K forms become available. For these soils or conditions extractants that measure a fraction of the nonexchangeable K forms, such as the sodium tetraphenylboron test, have demonstrated a potential to be better predictors of plant-available K. Therefore, field studies of crop response to K fertilization are needed to update the soil-test calibration data supporting fertilizer recommendations and to find soil-test K methods that predict better plant-available K.

The recent advent of precision agriculture technologies, such as global positioning systems (GPS), yield monitors, geographic information systems (GIS), and variable-rate

applicators among others has facilitated the collection of large amounts of spatially referenced data from producer's fields. These technologies are being adopted by producers and are relatively easy to implement. The use of these technologies, in combination with dense grid soil-sampling methods, has confirmed the assumptions about the lack of uniform soil test values, yields, and yield responses to nutrient inputs within fields. A more rational use of fertilizer is desirable to optimize economic and environmental benefits. Although variable-rate fertilization has good potential to improve fertilizer use, its effectiveness has been limited because nutrient levels in the soil generally show a high within-field variation at scales that changes across fields. This spatial variability arises through complex interactions between soil-forming and management factors. Management practices such as tillage, fertilization, manure application, and others can affect variability patterns of soil fertility properties. A successful use of precision agriculture technologies relies on the ability of soil sampling to identify areas that will likely respond to added fertilizer and areas that will not respond. The first step to successfully implement variable-rate fertilizer application is to accurately and reliably assess spatial variability in soil fertility. The development of computer-controlled fertilizer application equipment and GPS has renewed challenges to develop cost-effective sampling and mapping procedures that accurately describe the within-field spatial variability. A very important issue is to find appropriate methods to derive reliable soil-test maps through interpolation of data from an affordable number of soil samples.

This research involved two different studies to collect information needed to improve the use of soil testing in production agriculture. One study involved K response trials conducted over several years and sites and using conventional small-plot methodology and field-scale strip trials to correlate various soil test methods for corn and soybean crops. Specific objectives were to correlate ammonium-acetate K tests based on field-moist and oven-dry soil samples; to compare soil K extraction by ammonium-acetate, Mehlich-3, and

sodium tetraphenylboron (NaBPh₄) soil K extractants from dried soil samples; and to correlate the NaBPh₄ K extractant. The second study used a dense grid sampling method, classic statistics, and geostatistics to assess the spatial variability of soil fertility properties in two Iowa fields and evaluate data interpolation methods to improve soil-test mapping.

DISSERTATION ORGANIZATION

This dissertation is presented as three papers suitable for publication in scientific journals of the American Society of Agronomy. The title of the first paper is “Impact of sample drying on soil potassium extraction and field calibration of a test based on field-moist soil samples for corn and soybean”. The title of the second paper is “Field calibration of ammonium acetate, Mehlich-3, and sodium tetraphenylboron soil potassium tests for corn and soybean”. The third paper is entitled “Integrating geostatistics and GIS to study spatial variability and map soil fertility properties in Iowa fields”. Each paper is divided in sections that include abstract, introduction, materials and methods, results and discussion, conclusions, references, tables, and figures. The papers are preceded by a general introduction and are followed by a general conclusion.

**CHAPTER 2. IMPACT OF SAMPLE DRYING ON SOIL POTASSIUM
EXTRACTION AND FIELD CALIBRATION OF A TEST BASED ON FIELD-
MOIST SOIL SAMPLES FOR CORN AND SOYBEAN**

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ABSTRACT

Soil K extraction with neutral 1 M ammonium-acetate (NH₄OAc) based on air-dried or oven-dried samples is the most widely used soil test for K. It has long been recognized that sample drying often increases K extracted by this test. An NH₄OAc test based on field-moist samples (MK) was used until 1988 by the Iowa public laboratory but was abandoned because no other laboratory adopted it, and a factor of 1.25 was used to adjust upwards soil-test interpretation classes. The objectives of this study were to assess the impact of sample drying on soil K extraction from several Iowa soil series, conduct field correlations of NH₄OAc tests based on oven-dried samples (DK) and a modified version of the MK test for corn (*Zea mays* L.) and soybean [*Glycine max* (L.) Merr.]. Single-yr and multi-yr K response trials were conducted at 54 locations from 2001 to 2004. Soil K extracted with the DK test was higher than for the MK test and the difference increased with increasing drying temperature (air dried to 50 °C). However, no single factor can be used to relate DK and MK test results because the difference was inversely related to the soil K level and the drying effect varied greatly between soil series. The MK test correlated better with yield response and showed a better defined critical K concentration compared with DK. Results showed that different calibrations are needed for different soils and (or) growing conditions for DK but not for MK. Critical concentration ranges defined by Cate-Nelson (CN) and linear-plateau (LP) models across all soils for corn were (mg K kg⁻¹, 15 cm sampling depth) 144 to

201 for DK and 62 to 76 for MK, while critical concentrations ranges for soybean were 121 to 214 for DK and 52 to 90 for MK. An NH_4OAc K test based on field-moist samples predicts crop response to K fertilizer significantly better than the commonly used test based on dried samples, and the degree of the improvement may justify more laborious laboratory procedures for the field-moist test.

Abbreviations: Ammonium-acetate, NH_4OAc ; field-moist K test, MK; oven-dried K test, DK; Cate-Nelson, CN; linear-plateau, LP.

INTRODUCTION

Soil testing is an important diagnostic tool of crop production systems to assess plant K availability. An estimation of exchangeable soil K by extracting soil with neutral 1.0 M NH_4OAc from air-dried or oven-dried soil samples is the most widely used soil test for K and provides the basis for K fertilizer recommendations. The Mehlich-3 extractant, for which adoption by soil-testing laboratories is increasing, includes NH_4OAc in its extracting solution. These two tests are the suggested soil K tests for soils of the north-central region of the USA by the North-Central Regional Committee for Soil Testing and Plant Analysis (Warncke and Brown, 1998).

It has been stated that a soil should be tested without disturbing or altering it chemically or mechanically in the process of sample preparation (Gelderman and Mallarino, 1998). However testing *in situ* is not technically feasible yet, and soil samples are usually dried and crushed to simplify sample handling and provide a homogenous mix for subsampling. It has long been recognized that drying soil samples can influence the amount of K extracted with the NH_4OAc test. Many researchers have found an increase on extractable K upon drying (Steenkamp, 1928; Atoe, 1947; Luebs et al., 1956; Hanway and Scott, 1959) while others reported that soils high in exchangeable K tend to fix K and soils

low in exchangeable K tend to release K upon sample drying (Cook and Hutcheson, 1960; Hanway et al., 1962; Haby et al., 1988).

The impact of sample drying on changes in soil exchangeable K depends on the equilibrium concentration of K, the deviation from the equilibrium concentration at sampling time, and soil mineralogy (Haby et al., 1990). Cook and Hutcheson (1960) postulated an equilibrium level of 196 mg K kg⁻¹ for Kentucky soils and indicated that K is fixed upon drying when exchangeable K is higher than that level for field-moist samples and released when the K level is lower. Dowdy and Hutcheson (1963), also working with selected Kentucky soils, indicated that the field-moist K equilibrium value of these soils was 175 mg kg⁻¹. However, Haby et al. (1988) found that the field-moist K equilibrium value of 18 Montana soils was 420 mg kg⁻¹. Dowdy and Hutcheson (1963) stated that clay mineralogy of the soils was closely related with K release or fixation observed upon soil sample drying. They found that illite appeared to be the source of K released by drying, while vermiculite and montmorillonite were associated with K fixation. McLean and Watson (1985) stated that, if the soil is relatively low in exchangeable K, soil sample drying causes scrolling of layers of the micaceous clay that releases nonexchangeable K. However, if the soil is relatively high in exchangeable K or has had K added to it recently, drying usually drives water out from between the layers, causing them to collapse and trap K in nonexchangeable form (McLean and Watson, 1985). Depending on the relative magnitude of these two mechanisms a net release, fixation, or no change in exchangeable K could be observed.

Luebs et al. (1956) in a greenhouse study with 13 Iowa soils showed that K extracted from field-moist samples was better correlated with corn plant uptake than K extracted from air-dried soil. Superior correlations with field-moist samples were also found in K studies conducted in the north central region in greenhouse with millet (Barber et al., 1961), in the field with alfalfa (Hanway et al., 1961), and with corn (Hanway et al., 1962). The improved

relationship resulting from undried samples can be attributed generally to variable increase in exchangeable K when soils were dried (Grimes and Hanway, 1967).

Because of the effect of sample drying on K test results and the allegedly better correlation with plant uptake found for the moist K test, a method for testing field-moist soil samples based on a slurry was developed and implemented in Iowa until 1988 and was among tests suggested by the North-Central Region NCR-13 soil testing committee (Brown and Warncke, 1988; Eik and Gelderman, 1988). Field correlations for corn and soybean for this slurry K test from long-term Iowa experiments were published by Mallarino et al. (1991a, 1991b). However the Iowa State University Soil and Plant Analysis Laboratory discontinued analyzing samples with the slurry K test in 1988 because no private laboratory operating in Iowa adopted it due to impractical laboratory procedures (mainly soil moisture determination and slurry preparation). Therefore, based on comparisons of amounts of soil K extracted using dried (35 to 40 °C) or moist soil samples (not field calibrations) the soil-test interpretation categories for the slurry NH_4OAc K test were increased by a factor of 1.25 for Iowa recommendations published in 1988 (Voss and Killorn, 1988) and 1996 (Voss et al., 1996). The old database for the slurry K test and a 1.25 factor continued to be used for the NH_4OAc K test and the Mehlich-3 K test (for the first time, although interpretations were similar for both tests) for updated recommendations published in 1999 (Voss et al., 1999).

Increasing frequency of K deficiency symptoms in corn and soybean was observed across Iowa in the 1990's. These symptoms occurred in low-testing soils, but also in soils that tested medium or optimum according to the soil-test K interpretations at the time (Voss et al., 1996, 1999). Therefore, new field calibration research results for corn and soybean for NH_4OAc and the Mehlich-3 K tests based on oven-dried samples (Mallarino et al., 2002) were used to update soil-test K interpretations (Sawyer et al., 2002). However large response variation across soils with similar soil-test K levels was still observed for both corn and soybean crops when oven-dried K tests were used (Mallarino and Blackmer, 1994; Mallarino

et al., 2002, 2003), which indicated a need for continuing research to improve soil testing methods and interpretations for K in Iowa soils. Voss (1998) based on a national survey of recommendations by land grant universities strongly suggested a need to update the soil-test calibration data supporting recommendations for many regions, although soil-test calibration research has become difficult due to unwillingness of traditional agricultural research funding sources to fund this kind of research (Beegle, 2005). Therefore, the objectives of this study were to assess the effect of drying temperature on soil K extracted with the NH_4OAc extractant for several Iowa soil series and the effectiveness of the NH_4OAc tests based on oven-dried (DK) and field-moist (MK) samples by conducting field correlations for corn and soybean crops.

MATERIALS AND METHODS

Corn and soybean grain yields and soil samples were collected from K response trials conducted from 2001 to 2004 at 54 locations that included 20 Iowa counties. There were 20 single-year trials and 34 trials that were evaluated 2, 3, or 4 years by re-applying treatments for successive crops, which resulted in 121 site-years of data (64 with corn and 57 with soybean). The soils included 31 typical Iowa soil series in which row-crop production predominates (Table 1). Nitrogen and P fertilizers were applied uniformly to plots of all trials following Iowa State University recommendations for these nutrients (Sawyer et al., 2002; Blackmer et al., 1997). Other crop management practices, such as tillage, corn hybrids, soybean cultivars, seeding dates and rates, and weed control, were those normally used by the farmers.

Twenty-six single-year or multi-year trials for corn-soybean rotations involved small-plot methodology, a plot size ranging from 54 to 108 m², randomized complete-block or split-plot designs (when any additional treatment was also evaluated) with three to six replications, and hand application of K treatments (commercial KCl fertilizer). Twelve trials

evaluated five K fertilization rates (0 to 168 kg K ha⁻¹) for crops managed with chisel-plow/disk tillage. Ten long-term trials established in 1994 evaluated three annual K fertilization rates (0 to 65 kg K ha⁻¹) broadcast or banded with the planter for crops managed with no-till or chisel-plow/disk tillage. Two long-term trials established in 1979 evaluated the factorial combinations of 0, 22, and 44 kg P ha⁻¹ and 0, 67, and 134 kg K ha⁻¹ broadcast annually for crops managed with chisel-plow/disk tillage. Plots that received the highest P rate and either no K or the highest K rate were used for this study. Two other long-term K experiments established in 1976 evaluated effects of several initial contrasting soil-test K levels and four broadcast annual K rates (0 to 100 kg K ha⁻¹) superimposed to the initial-K treatments for crops managed with chisel-plow/disk tillage. Partial yield results for some treatments and details of the long-term trials were published before (Mallarino et al., 1991a and 1991b; Bordoli and Mallarino, 1998; Borges and Mallarino, 2000), but results since 2001 used for this study have not been published.

Twenty-eight field-scale strip trials evaluated two K fertilization rates (0 and 100 to 186 kg K ha⁻¹, depending on the field) broadcast to strips measuring 18.3 m in width and 250 to 450 m in length depending on the field. The K treatments (commercial KCl fertilizer) were applied with commercial fertilizer spreaders and were arranged in randomized complete-block designs with three or four replications.

Composite soil samples (12 cores, 0-15 cm depth) were collected in the fall after harvest of the previous crop and before applying K treatments. For single-year or first-year of multi-year small-plot trials, the samples were collected from each replication and averaged to provide one soil-test K value for the experimental area. For following years of multi-year trials, samples were collected from plots receiving no K fertilizer. For the strip trials, soil samples were collected using a systematic, grid-point sampling method (Wollenhaupt et al., 1994) adjusted to the field design. The width of the grid cells across the strips coincided with the width of a replication (36.6 m) and the length was also 36.6 m, which resulted in 0.134 ha

cells. The soil cores for each composite sample were collected following a random pattern from areas approximately 100-m² in size at the center of each grid cell.

The soil samples were stored at 5°C from 2 to 10 weeks after sampling, sieved through a 5-mm mesh, mixed thoroughly, and divided in two sub-samples. One sub-sample was analyzed with the procedure suggested for the North-Central Region by the NCR-13 committee (Warncke and Brown, 1998). All soil analyses described in this section were done on duplicate soil samples. Samples were dried at 40 °C, crushed to pass through a 2-mm sieve, 1 g of soil was extracted with 10 mL neutral 1 M NH₄OAc, shaking at 200 oscillations min⁻¹ for 5 min, and measuring K by atomic absorption spectroscopy. The other sub-sample was not dried and was used for an MK analysis. Soil moisture content was determined by calculating the difference between weight of field-moist and samples dried to constant weight at 40°C to constant weight. To minimize sub-sampling error, the sample size for the analysis was increased to 2 g equivalent dry weight but the soil/solution ratio was maintained by using 20 mL of extractive solution. Otherwise, the procedure was similar to that used for oven-dried samples.

Effects of soil drying temperature on DK test results were studied on 739 soil samples selected from trials conducted at 11 locations in three years (2001, 2002, and 2003) that encompassed 11 of the most typical Iowa soil series and approximately represented soil moisture and soil conditions of the trials in the study. Soil K was measured with the DK procedure described above (by drying samples at 40 °C), by air-drying samples at room conditions (approximately 23°C and 65% relative humidity) using forced air for 7 d, and by drying samples at 50 °C.

The sample handling procedure used for the MK test in this study differed from the procedure used until 1988 by the Iowa State University Soil Testing Laboratory (Eik and Gelderman, 1988) in that a soil/water slurry was not prepared. Therefore, the two procedures were compared by analyzing 113 soil samples selected from trials conducted at 25 sites in

three different years (2001, 2002, and 2004) to approximately represent the soils, soil moisture, and soil conditions of the trials in the study. For the slurry procedure, an amount of field-moist soil equivalent to 50 g of oven-dry soil was mixed with 100 mL deionized water for 1 min using an electrical stirrer. A 6 mL aliquote of the slurry was extracted with 6 mL of 1.67 M NH_4OAc solution so that the equivalent dry soil/solution ratio was maintained similar to that of the DK procedure. Otherwise, the procedure was similar to that used for the DK test.

The cation exchange capacity (CEC) of the soils was estimated by summation of exchangeable Ca, Mg, K, Na, and neutralizable soil acidity (Warncke and Brown, 1998). Soil pH was measured using 1:1 soil/water ratio and ranged from 5.4 to 8.1 across sites. Soil organic C was measured following the combustion method described by Wang and Anderson (1998) using a LECO CHN-2000 analyzer (LECO Corp., St. Joseph, MI). Levels of soil organic matter (SOM) across sites ranged from 27 to 69 g kg^{-1} .

Corn and soybean grain from small-plot trials was harvested with plot combines (15-m length of three or four rows) or by hand (7.5-m length of two rows) from the center of each plot. Grain yield from strip trials was measured using combines equipped with commercial impact flow-rate yield monitors, moisture sensors, and global positioning system (GPS) receivers following a methodology used and described before in detail for evaluating various treatments (Bermudez and Mallarino, 2002; Wittry and Mallarino, 2004). Briefly, grain yield monitor data recorded every 1 s from experimental areas at least 40 m away from any field border were imported into ArcView GIS (Environmental Systems Research Institute, Redlands, CA). Any data point affected by common yield monitor errors (such as effects of waterways and unexpected combine stops) was deleted. Also, only data from combine passes from the center of each treatment strip (unaffected by treatment borders) were kept (for corn two passes 4.5 or 6 m wide each and for soybean either two passes 4.5 to 7.5 m wide each or one pass 9 m wide). Data from yield monitor points were averaged for small

areas defined by the width of each treatment strip and the separation distance of grid soil sampling lines along strips (0.067 ha). Therefore, the small field area represented by the two K treatments match the field area represented by each grid soil-sampling cell (0.134 ha). ArcView GIS was used to average these grain yield means and the corresponding soil-test K values for polygons representing the soil series present in the experimental areas from digitized (scale 1:12000) soil survey maps (Iowa Cooperative Soil Survey, 2003).

The grain yield data used in this study is expressed as relative responses to K. Relative response was calculated for each site-year by dividing the mean yield of non-fertilized plots by the mean of the highest K fertilization treatment and multiplying the result by 100. In small-plot trials that evaluated K placement methods, means of the highest K rate were calculated across all methods because yield response was seldom affected by the K placement method. For small-plot trials, a data pair (relative yield and soil-test K) is represented in figures by one point and represents one site-year. The soil-test K value represents the entire experimental area for 1-yr trials and the mean of the non-fertilized plots for trials evaluated more than 1 yr. For strip trials, a data point represents a pair of data (relative yield and soil-test K) for each dominant soil series present in the experimental areas. A dominant soil series was defined as the series that at least occupied the entire area of three 0.134 grid-sampling cells.

Soil test critical concentrations were calculated with the statistical Cate-Nelson (CN) method (Cate and Nelson, 1971) and with the linear-plateau (LP) and quadratic-plateau (QP) segmented models (Waugh et al., 1973). The critical concentration defined by the CN method was determined with the General Linear Models (GLM) procedure of SAS (SAS Institute, 2000) as the value that split the yield response data into the two groups that accounted for the largest proportion of the total variability (R^2). Critical concentrations defined by the segmented models were determined with the Nonlinear Model (NLIN)

procedure of SAS, and represent the soil-test values at which the two portions of each model joined.

RESULTS AND DISCUSSION

Amount of K extracted by the soil tests

Soil-test K levels ranged across all sites from 56 to 388 mg K kg⁻¹ for DK and from 30 to 356 mg kg⁻¹ for MK. The DK test results were 0 to 350% higher than the MOIST K test results (140% higher on average across all soils and sites) and differences were larger at low STK values, which explain a poor correlation across all soils (Fig. 1). Our results show that no single factor can be used to express results of one test based on results for the other. Furthermore, the difference between DK and MK tests varied significantly among soil series. Figure 2 shows changes of K extracted after drying soil samples at different temperatures for some typical Iowa soil series included in this study. The effects of drying on changes in measured K found across these soils agree with results reported by several authors (Luebs et al., 1956; Hanway and Scott, 1959). The increase in the amount of K extracted depended upon the drying temperature. The air-dried values for almost all soil series were somewhere in-between values of field-moist samples and samples dried at 40 °C, whereas values were always highest when samples were dried at 50 °C. Others have shown that increasing the drying temperature increases extractable K (Burns and Barber, 1961; Hanway et al., 1961; Haby et al., 1988). For example, Haby et al. (1988) found that oven-drying soil samples at 60 °C resulted in much higher extracted K than air-drying the soils.

Data in Fig. 3 show that the relative difference between DK and MK tests (their ratio) and the absolute difference decreased with increasing MK values. However, the strength of the relationship was weaker for the absolute difference (Fig. 3b). Haby et al. (1988) also found that the increase in exchangeable K upon sample drying becomes proportionally less as the K level in the field-moist samples increased. Moreover, they reported that at certain

intermediate soil K content, K extracted from oven-dried soils equaled the K extracted from field-moist samples (equilibrium concentration of K), but at higher K levels drying decreased extracted K compared with field-moist samples.

The additional K measured by DK compared with MK was positively and linearly related with soil CEC and total bases when it was expressed either as relative (DK/MK extraction ratio) or absolute terms (Figs. 4 and 5). The strength of the relationships was weak, however. Similar trends were observed for relationships between SOM and the relative or absolute difference between tests (not shown). No significant ($P \leq 0.05$) relationship was found between soil pH and the difference between DK and MK. Interpretations of cause and effect concerning the weak relationships between tests differences and CEC, total bases, or SOM are difficult and speculation is risky because these soil properties normally are related to soil texture (higher as the texture becomes finer) and soil mineralogy. Barber et al. (1961) found that soil texture and the level of exchangeable K in the soil were the primary factors influencing the change in exchangeable K measured upon sample drying. Our results (Fig. 6) suggest that not only the extractable K level but also the relationship between Ca and Mg to K (Ca + Mg / K ratio) would be affecting the increase in exchangeable K observed on drying.

The history of K fertilization seems to have an effect on the amount of K released upon sample drying. Observed differences between DK and MK were greater in samples from non-fertilized plots than in samples from plots receiving K fertilizer treatments. Calculations across plots of multi-year trials in this study (not shown) indicated that on average DK was 3.5 times higher than MK for non-fertilized plots but was only 1.9 times higher for plots that received K for many years. Grava et al. (1961) reported a similar effect of the K fertilization history on extractable K for a Nicollet clay-loam soil in Minnesota.

The soil moisture content at the time of collecting the samples was not related with the differences between DK and MK across sites (not shown). Soil moisture overall sites

ranged from 60 to 310 g kg⁻¹ with an average of 210 g kg⁻¹. These results are in agreement with Luebs et al. (1956), who showed little increase in exchangeable K measured on field-moist samples until the soil moisture level was about 50 g kg⁻¹ or below.

An additional important factor that could be affecting K dynamic in soils is drainage regime of the soils. The wetter the soil is, the longer the redox potential of the soil solution is likely to remain low enough to reduce Fe. The more reduced Fe in the structure of clay minerals, the greater will be the net negative charge, and thus the stronger will be the "hold" on positively charged K ions in interlayer positions. There is laboratory evidence that this happens in soil clays (Khaled and Stucki, 1991; Shen and Stucki, 1994), but in actual soil with much SOM this effect has not been documented (Schindler et al., 2003). Similarly, high SOM also has the potential to block access of K ions to interlayer sites, favoring retention of K at less energetic exchange sites. It is also worth noting that soils that are poorly drained, in which the redox effect described above might occur, also tend to be fine textured soils with higher CEC and SOM contents, so the redox effect might never be measurable.

We believe that field moisture relations (associated to physical soil properties, internal soil drainage, and/or landscape position) and soil sample drying in the laboratory would be important factors explaining the observed variation of NH₄OAc-extractable K upon sample drying. Our results suggest that the effect of sample drying (and of the temperature used) on extracted soil K varies greatly across soil series, with soil characteristics like texture, CEC, total bases, SOM, and Ca and Mg to K ratios and with soil-test K level.

Relationship between field-moist based K test methods

The amount of soil K extracted by the NH₄OAc extractant from field-moist samples by the slurry method used until the late 1980s by the Iowa State University Soil Testing Laboratory (Brown and Warncke, 1988; Eik and Gelderman, 1988) was positively and linearly related ($r = 0.99$) with the MK method used in this study (Fig. 7). However, 17% more K was measured with the slurry method compared with the MK method. The most

likely reason for this is the more aggressive extraction of K when soil and water were stirred for 1 min prior to the 5 min extraction period in the slurry method. Observation of sediment in the extraction flasks after the extraction sometimes indicated the presence of small soil aggregates after shaking soil for the MK method but very little after shaking the slurry. These visual observations and the high linear correlations between methods indicated that this effect was not clearly different for the soils included in this correlation, which varied greatly in initial moisture and texture. However, these results suggest that a slurry method may provide a less variable K measurement than the quicker and simpler sieving procedure used for MK in this study.

Field Correlation of yield response to K fertilization

Field response trials that encompass a wide range of soil-test levels and crop production conditions offer an appropriate basis for soil-test correlation and calibration. The wide range of growing conditions evaluated in this study resulted in grain yields for K fertilized plots that varied from 5.0 to 14.4 Mg ha⁻¹ for corn and from 1.1 to 5.0 Mg ha⁻¹ for soybean, and averaged 10.8 and 3.2 Mg ha⁻¹ for corn and soybean, respectively. Analyses of variance of K effects on yield for individual sites indicated a significant yield increase ($P \leq 0.05$) in 41% of the site-years with corn and 35% of the site-years with soybean (not shown).

Relationships between relative corn and soybean yield and soil K measured with the DK test are shown in Figs. 8 and 9. These data show the classic relationship between yield response and soil-test values. However, although the highest portion of the curve indicates relative yield values within about 5% of a 100% yield level, there is very high variation at medium to low soil-test K levels. These results indicate a poor capacity of the DK test to predict a crop response to K fertilization, particularly in soybean. This variability did not originate from experimental error because we included three to six replications in the trials, the laboratory analyses were done in duplicate, and similar variation was observed for on-farm strip trials and for conventional small-plot trials at research farms where sources of

error could have been controlled better. Only in a few instances the variation in the relationship could be explained by yield levels (for example, yields limited by low rainfall in some site-years). Therefore, we conclude that the soil-test itself is the cause of the variation and of poor prediction of response when soil-test levels are low to medium. Beegle and Oravec (1990) and Mallarino and Blackmer (1994) also showed poor relationships between corn relative yield and soil K extracted with NH_4OAc from dry samples.

The distribution of the data points in Fig. 8 suggests different relationships for two groups of soil series. The white data points represent results for soils in which DK values ranging from approximately 130 to 145 mg K kg^{-1} produced more than 95% corn relative yield. The black data points represent results for soils in which DK values ranging from approximately 170 to 180 mg K kg^{-1} produced more than 95% corn relative yield. The soils represented by the black points were Nicollet, Webster, and Canisteo soils developed on glacial till materials, which predominate in central and north-central Iowa. The apparently different relationship for the two groups of soils was not as clear in soybean as it was in corn (Fig. 8). All the soils represented by black data points have in common deep profiles, somewhat poor to very poor drainage, moderate to poor permeability, slope from 0 to 4%, texture from loam (Nicollet) to clay loam (Webster and Canisteo) in the top 6 inches, and high CEC, exchangeable Ca, and SOM compared with other Iowa soils. A few of these soils (such as Canisteo) have high pH due to elevated calcium carbonate concentration. Although data in Figure 8 suggest different STK requirements for different soils, because of the wide data spread below a STK value of about 180 mg K kg^{-1} , particularly in soybean, no adjustments for soil series can be reasonably supported. Figure 9 shows the same relationship but with site-yr identified by the texture of the surface horizon (from ISPAID, 2004). Apparently sites with loam and silty-clay-loam surface texture, represented by white and grey dots, had lower K requirements for crop production than sites with clay-loam surface texture, but again this is not as clear for soybean as it is for corn.

Subsoil K levels are considered for routine soil-test K interpretations in Iowa. The recommended soil-test K levels for production of corn and soybean (Sawyer et al., 2002) are higher for soil series with DK levels lower than 50 mg K kg⁻¹. In Table 1 we classified the sites of this study following the more detailed subsoil K classification used by the Iowa Soil Properties and Interpretations Database (ISPAID, 2004). The distribution of points in Fig. 10 indicate that approximately one-half of site-years classified as having the lowest subsoil K levels (VL-, <25 mg K kg⁻¹) showed the highest corn or soybean response to K fertilization. However, the rest of the site-years, with lower response to K, showed an indistinct distribution concerning subsoil K levels and high variability.

Several reasons could explain different soil K requirements across soils and large response variation across soils with similar DK levels, but the most important one seemed to be the variable increase in extractable K upon sample drying across soils. Figures 11 and 12 show the relationships between relative corn and soybean yields and soil K measured with the MK test. The results show that the MK test has superior capacity to predict corn and soybean response to K than the DK test. Previous research in Iowa and in the north-central US showed that soil-test K determined on field-moist samples was a better predictor of plant availability than air-dry or oven-dry K tests (Luebs et al., 1956; Barber et al., 1961; Hanway et al., 1961; Hanway et al., 1962). Results showed that MK extracts proportionally less K than DK from soils represented by black points in which the DK test suggests that higher soil K is needed to produce certain relative yield level. Soils series represented by white and black data points seems to blend into the same trend for corn and soybean (Fig. 11). Yet, the variability in the relationship was higher for soybean than for corn and soil series represented by white points at intermediate MK values seems to show higher variation compared to other soils. Sites with different surface soil textures represented by white, gray, and black data points also blend into the same relationship for corn and soybean (Fig. 12) and the same occurred with the different subsoil K classifications (not shown). It must be noted that the

soil textures used for Fig. 12 are from the Iowa soil database and that budget limitations did not allow for measurement of soil texture for the plots of this study.

Critical soil-test K concentrations calculated by the CN, LP, and QP models are shown in Table 2. Linear-plateau model fits to relationships between relative corn or soybean yield and DK or MK tests are shown in Fig. 13. Critical concentrations were approximately similar for corn and soybeans, but varied markedly depending on the soil-test method and model used. It is well known that soil-test critical concentrations determined by various models can differ significantly (Dahnke and Olson, 1990; Mallarino and Blackmer, 1992; Mallarino and Blackmer, 1994). The critical concentrations determined for the MK test were considerably lower than those determined for the DK test, as would be expected from differences in measured K.

Selection of an appropriate critical value may be as important as selection of a soil test method, particularly in regions dominated by high-testing soils (Mallarino and Blackmer, 1992; Mallarino and Blackmer, 1994). The ability to identify appropriate critical values is one important requisite for distinguishing between reliable and unreliable soil tests. There is no clearly superior or widely accepted method for establishing economically optimum critical concentration ranges (Dahnke and Olson, 1990; Mallarino and Blackmer, 1992). Previous research (Mallarino and Blackmer, 1992; Mallarino and Blackmer, 1994) showed that short-term economically optimum soil P critical concentrations usually are within a range of critical concentrations defined by the CN and LP models. They demonstrated that critical concentrations defined by the QP model usually are much higher than those defined by the CN and LP models, and their use resulted in smaller returns to fertilization across many fields. Critical soil-test K concentration ranges defined by the CN and LP models in this study for DK were 144 to 201 mg kg⁻¹ for corn and 121 to 214 mg kg⁻¹ for soybean. Critical soil-test K concentration ranges defined by these two models for the MK test were 62 to 76 mg kg⁻¹ for corn and 52 to 90 mg kg⁻¹ for soybean.

The critical concentration ranges defined by the CN and LP models for the DK test are slightly higher and much wider compared with the optimum range of current Iowa interpretations for corn and soybean for most soils of the state (131-170 mg K kg⁻¹ for both crops, Sawyer et al., 2002). Only K fertilization to maintain soil-test K based on prevailing crop K removal is recommended within this category. The current Iowa interpretations recommend higher soil-test K levels for corn and soybean than those recommended for soils in the neighboring states of Minnesota, Nebraska, and South Dakota (Gerwing and Gelderman, 1998; Rehm et al., 2001; Shapiro et al., 2001) but not in Illinois (Hoefl and Peck, 2001). Applying the current optimum interpretation range (131-170 mg K kg⁻¹) used in Iowa for the DK test to corn and soybean responses in this study corresponds to mean relative response values of 93 and 95%, respectively. However, the range identified by the CN and LP models for DK and MK tests corresponds to a slightly higher mean yield response (96 and 95% for corn and soybean averaged). The range identified by the CN and LP models for MK and DK tests for soybean is considerably wider compared with corn, probably because of less well-defined distinction between responsive and not responsive sites for this crop.

It is of interest to compare the critical concentration ranges identified in this study for the MK test with critical concentration ranges suggested by past research for the slurry version of the field-moist test that was used in Iowa until the late 1980s. Because the K method used in this study extracted less K than the slurry method but the tests were well correlated (Fig. 7), we used the equation in Fig. 7 to estimate the critical concentration ranges for the slurry test from the ranges determined for the DK test. The critical concentration ranges for the slurry test would be 79 to 94 mg kg⁻¹ for corn and 67 to 110 mg kg⁻¹ for soybean. Published field correlations of the slurry K (Mallarino et al., 1991a and 1991b) showed that K fertilization seldom increased corn and soybean yield below a range of 68 to 100 mg K kg⁻¹ and did not increase yield at higher levels. This soil-test K range corresponds to the Optimum interpretation class (formerly denominated Medium) of Iowa State

University recommendations for the slurry test during the 1980s (Voss, 1982). Therefore, although the determined critical concentration range in this study was wider for soybean than for corn, we conclude that our results match very well interpretations used 20 years ago for the slurry K test.

CONCLUSIONS

The amount of soil K measured with the DK test, the K test being used in Iowa and most of the world, was significantly higher than amounts measured by the MK test. The additional amount of K extracted by the DK test increased with the drying temperature, decreased with increasing soil K measured by tests based on oven-dried or field-moist samples, and differed significantly between many soil series included in the study.

The results showed that MK test has superior capacity to predict corn and soybean response to K fertilization compared with the commonly used DK test. The MK test blends soil-test K values and relative yield responses into the same general relationship, which is not the case for the DK test. The improvement in this relationship by the MK test is mainly explained by proportionally less K extracted by this test than the DK test from soils in which the latter indicates higher soil K levels are needed to produce a certain corn or soybean relative yield level compared with other soils.

Critical soil-test K concentration ranges defined by the CN and LP models in this study for the DK test were 144 to 201 mg kg⁻¹ for corn and 121 to 214 mg kg⁻¹ for soybean. Critical soil-test K concentration ranges defined by these two models for the MK test were 62 to 76 mg kg⁻¹ for corn and 52 to 90 mg kg⁻¹ for soybean. Estimates of the critical concentrations that would have been determined for the slurry K test based on correlations with the MK test for 113 representative samples indicated that interpretations based on the new dataset would have been similar to interpretations used in Iowa for the slurry K test in the 1980s.

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Table 1. Information on soils for trials evaluating the response of corn and soybean to K fertilization.

Series	Subgroup	Family	Subsoil K [†]	Texture [‡]
Aredale	Typic Hapludolls	Fine-loamy, mixed, mesic	L	L
Calco	Cumulic Endoaquolls	Fine-silty, mixed (calcareous), mesic	VL+	SICL
Canisteo	Typic Endoaquolls	Fine-loamy, mixed (calcareous), mesic	VL-	CL
Clarion	Typic Hapludolls	Fine-loamy, mixed, mesic	VL+	L
Clyde-Floyd	Typic Endoaquolls	Fine-loamy, mixed, mesic	VL-	CL
Colo	Cumulic Endoaquolls	Fine-silty, mixed, mesic	VL+	SICL
Colo-Ely	Cumulic Endoaquolls	Fine-silty, mixed, mesic	VL-	SICL
Crippin	Aquic Hapludolls	Fine-loamy, mixed, mesic	VL-	L
Dinsdale	Typic Argiudolls	Fine-silty, mixed, mesic	VL+	SICL
Donnan	Aquollic Hapludalfs	Fine-loamy over clayey, mixed, mesic	VL+	L
Galva	Typic Hapludolls	Fine-silty, mixed, mesic	VL+	SICL
Kenyon	Typic Hapludolls	Fine-loamy, mixed, superactive, mesic	VL-	L
Killduff	Dystric Eutrudepts	Fine-silty, mixed, mesic	VL+	SICL
Klinger	Aquic Hapludolls	Fine-silty, mixed, mesic	VL+	SICL
Klinger-Maxfield	Aquic Hapludolls	Fine-silty, mixed, mesic	VL+	SICL
Lawler	Aquic Hapludolls	Fine-loamy over sandy or sandy-skeletal, mixed, mesic	VL+	L
Lilah	Psammentic Hapludalfs	Sandy, mixed, mesic	VL+	SL
Mahaska	Aquertic Argiudolls	Fine-montmorillonitic, mesic	L	SICL
Marshall	Typic Hapludolls	Fine-silty, mixed, mesic	L	SICL
Muscatine	Aquic Hapludolls	Fine-silty, mixed, mesic	VL+	SICL
Nevin	Aquic Argiudolls	Fine-silty, mixed, mesic	L	SICL
Nicollet	Aquic Hapludolls	Fine-loamy, mixed, mesic	VL+	L
Olin	Typic Hapludolls	Coarse-loamy, mixed, mesic	VL-	FSL
Otley	Oxyaquic Argiudolls	Fine-montmorillonitic, mesic	VL+	SICL
Readlyn	Aquic Hapludolls	Fine-loamy, mixed, mesic	VL+	L
Saude	Typic Hapludolls	Coarse-loamy over sandy or sandy-skeletal mixed, mesic	VL+	L
Spillville	Cumulic Hapludolls	Fine-loamy, mixed, mesic	VL+	L
Taintor	Vertic Argiaquolls	Fine-montmorillonitic, mesic	VL+	SICL
Tama	Typic Argiudolls	Fine-silty, mixed, mesic	VL+	SICL
Webster	Typic Endoaquolls	Fine-loamy, mixed, mesic	VL-	CL
Wiota	Typic Argiudolls	Fine-silty, mixed, mesic	VL+	SIL

[†] Subsoil K (30-60 cm depth). Very Low minus (VL-) <25 mg K kg⁻¹, Very Low plus (VL+) 25-50 mg K kg⁻¹, Low (L) 50-79 mg K kg⁻¹ (ISPAID, 2004).

[‡] Texture of the surface horizon, CL= Clay loam, FSL= Fine sandy loam, L= Loam, SICL= Silty clay loam, SIL= Silty loam, SL= Sandy loam (ISPAID, 2004).

Table 2. Critical concentrations of soil-test K for corn and soybean estimated by NH₄OAc extractant on dried and field-moist soil samples and three models.

Crop	Soil test	Model [†]	Equation	R ²	CC [‡]
					mg K kg ⁻¹
Corn	Dried	CN	na [§]	0.26	144
		LP	$Y = 79.3 + 0.10X$	0.24	201
		QP	$Y = 75.3 + 0.16X - 0.0003X^2$	0.23	304
	Moist	CN	na	0.38	62
		LP	$Y = 73.5 + 0.33X$	0.47	76
		QP	$Y = 65.2 + 0.67X - 0.0033X^2$	0.48	101
Soybeans	Dried	CN	na	0.25	121
		LP	$Y = 82.7 + 0.08X$	0.25	214
		QP	$Y = 77.9 + 0.15X - 0.0003X^2$	0.26	300
	Moist	CN	na	0.30	52
		LP	$Y = 83.0 + 0.18X$	0.37	90
		QP	$Y = 75.7 + 0.44X - 0.0021X^2$	0.38	107

[†] CN, Cate-Nelson; LP, linear-plateau; and QP, quadratic-plateau. The LP and QP models apply for X less than or equal to the critical concentration, which is the value at which the plateau portion of each model starts. The statistical significance of the relationships always was $P \leq 0.001$.

[‡] CC, critical concentration. For LP and QP models CC is the concentration at which the linear or quadratic portions of the models join with the predicted plateau yield.

[§] Not applicable.

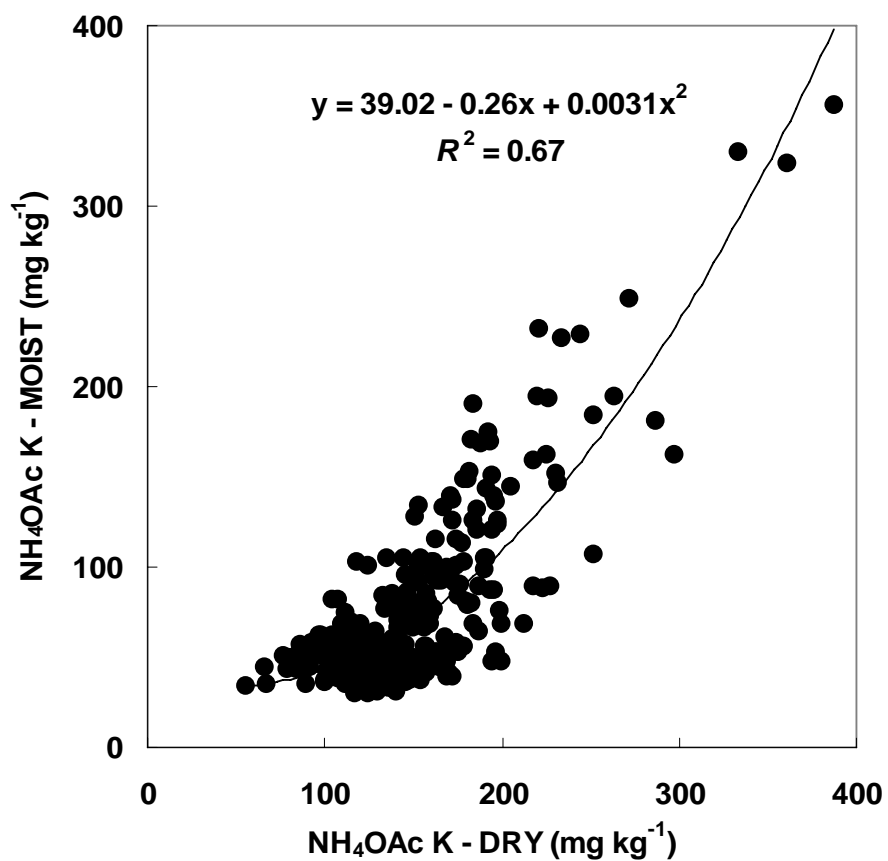


Figure 1. Relationship between exchangeable K measured with ammonium acetate (NH₄OAc) based on field-moist (MOIST) and oven-dry (DRY) soil samples.

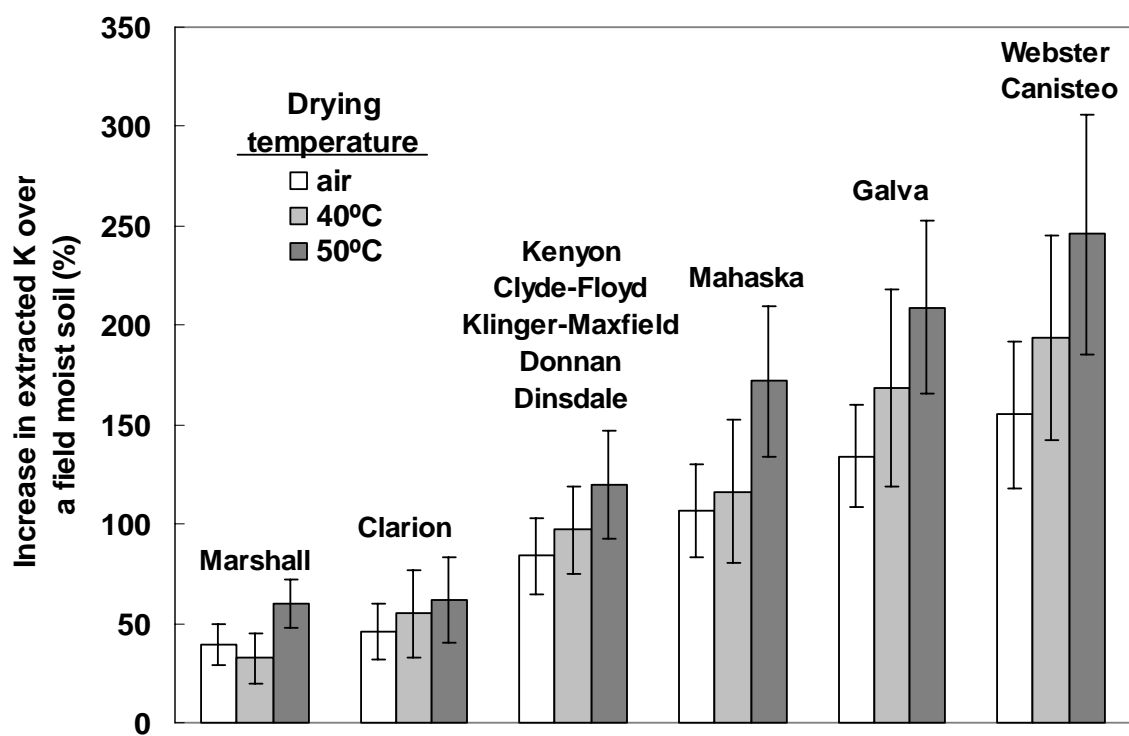


Figure 2. Effect of sample drying temperatures on ammonium acetate (NH_4OAc) extracted K relative to K measured on field-moist samples for typical Iowa soil series. Vertical bars represent one standard deviation.

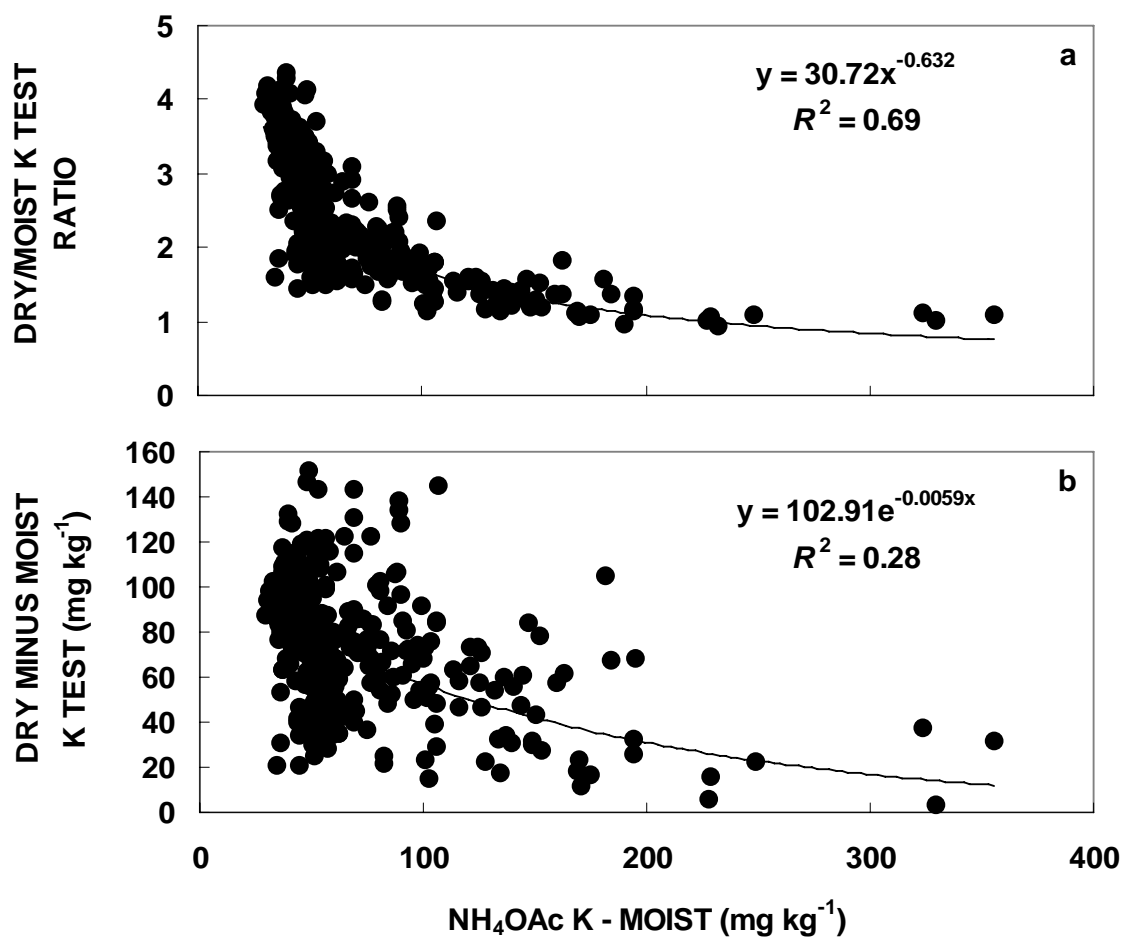


Figure 3. Relationships between exchangeable K measured with ammonium acetate (NH₄OAc) based on field-moist soil samples (MOIST) and the relative (a) or absolute (b) difference from soil K measured by the same extractant but with determination of exchangeable K based on oven-dry soil samples (DRY).

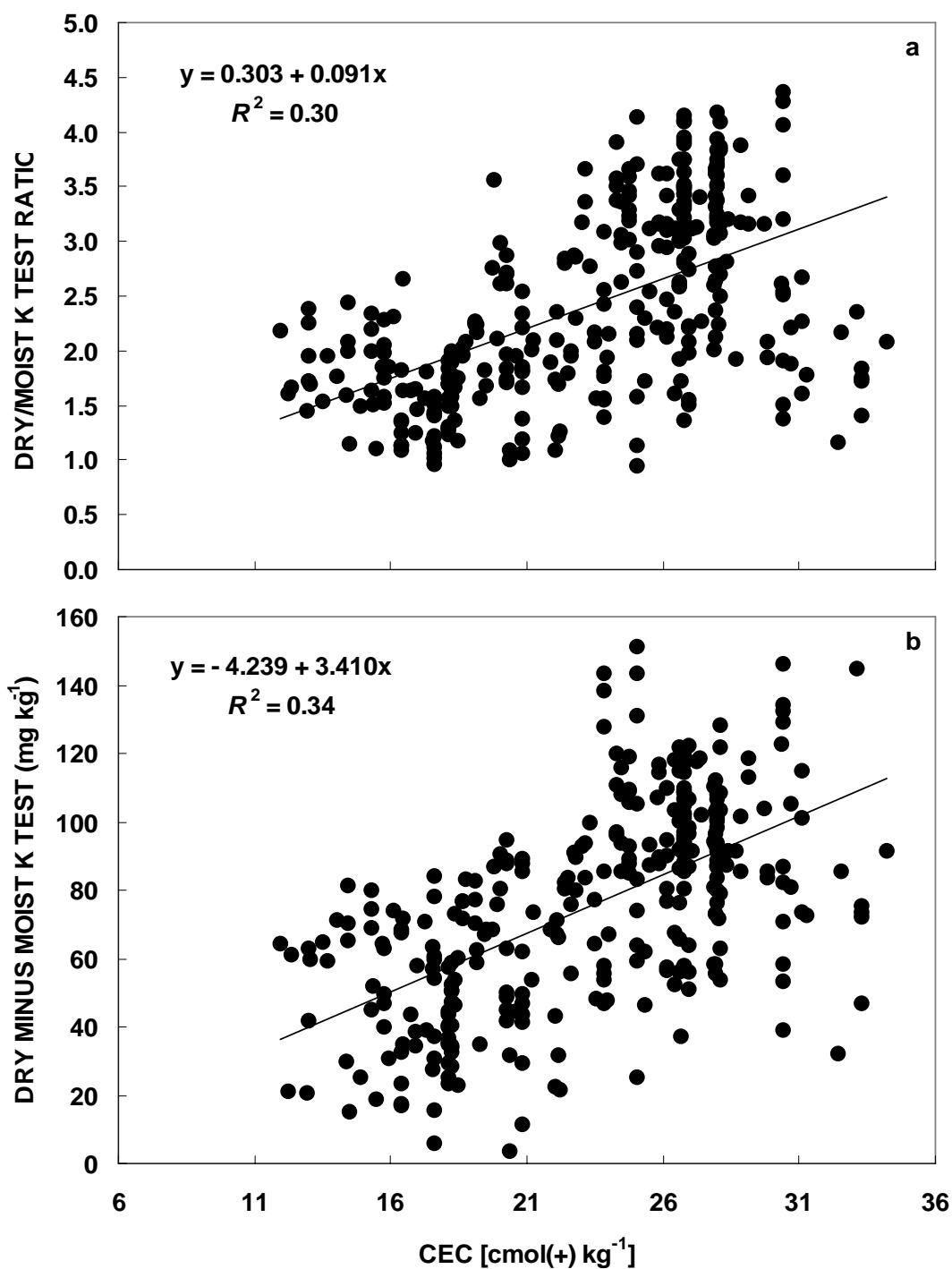


Figure 4. Relationships between cation exchange capacity (CEC) and the relative (a) or absolute (b) difference in soil K measured with ammonium acetate (NH₄OAc) based on field-moist soil samples (MOIST) or on oven-dry soil samples (DRY).

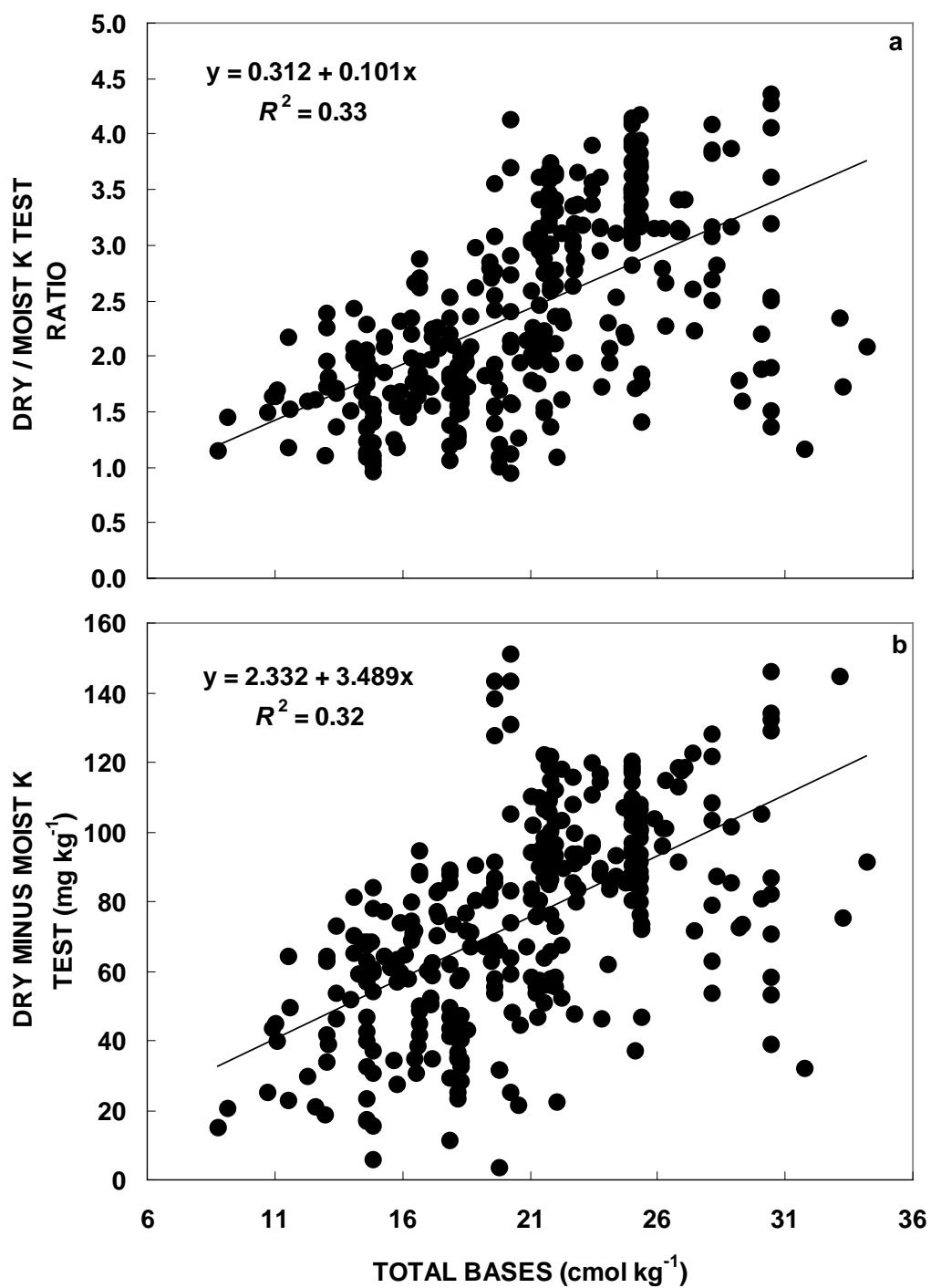


Figure 5. Relationships between total bases and the relative (a) or absolute (b) difference in soil K measured with ammonium acetate based on field-moist soil samples (MOIST) or on oven-dry soil samples (DRY).

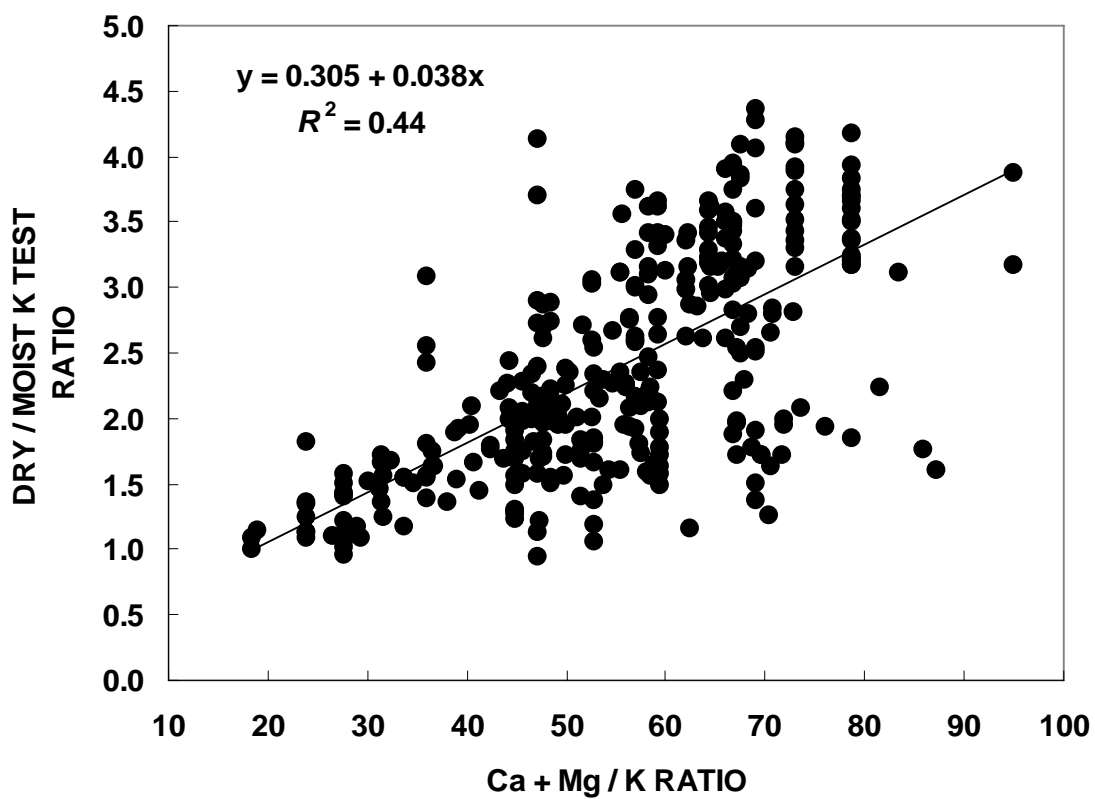


Figure 6. Relationship between Ca + Mg / K ratio and the relative difference in soil K measured with ammonium acetate based on field-moist soil samples (MOIST) or on oven-dry soil samples (DRY).

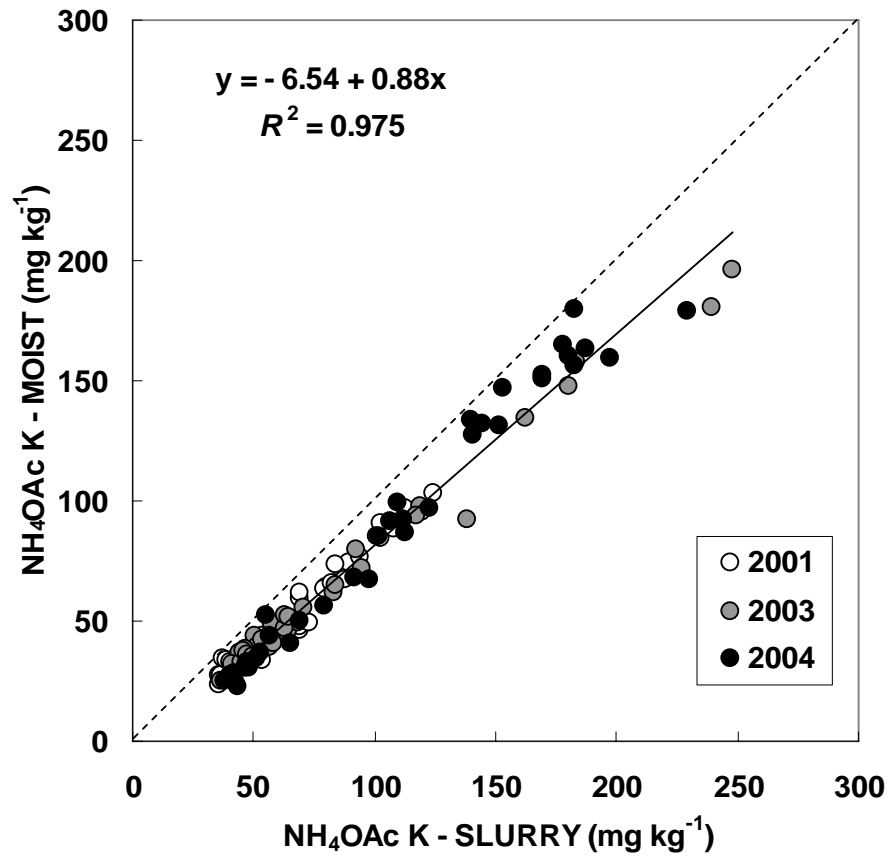


Figure 7. Correlation between exchangeable K measured with ammonium acetate (NH₄OAc) based on field-moist samples with the formerly used “slurry” methodology (SLURRY) and with the newly developed MOIST methodology.

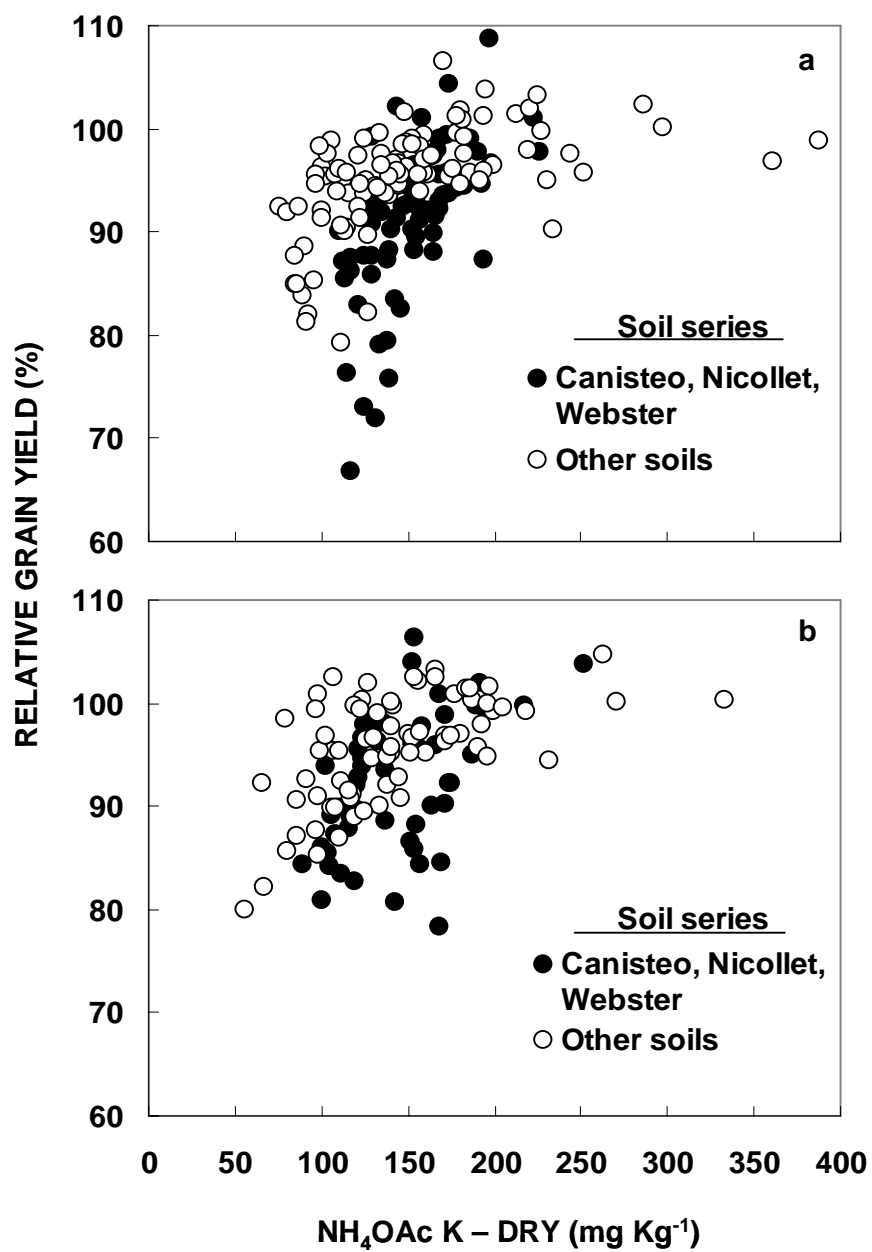


Figure 8. Relationships between relative corn (a) and soybean (b) yield and exchangeable K measured with ammonium acetate (NH_4OAc) based on oven-dry (DRY) soil samples.

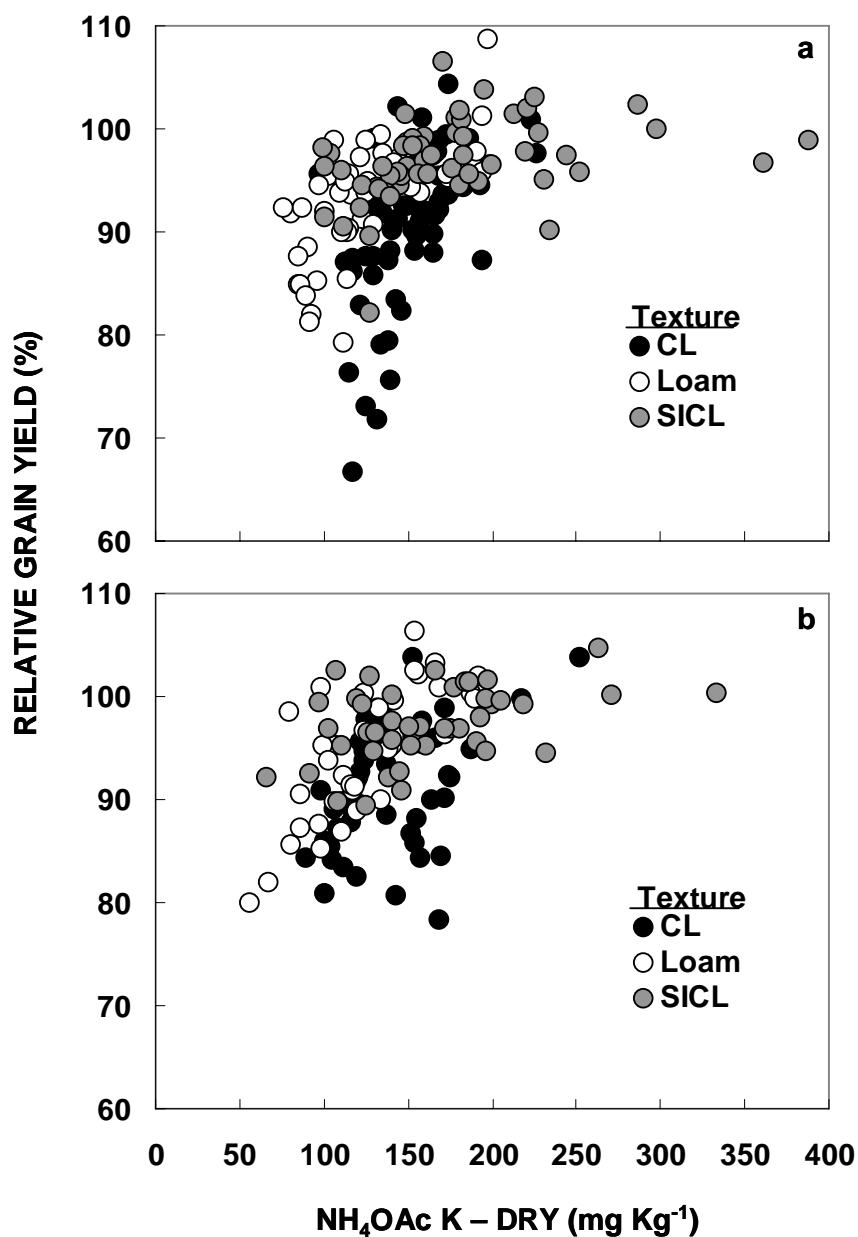


Figure 9. Relationships between relative corn (a) and soybean (b) yield and exchangeable K measured with ammonium acetate (NH₄OAc) based on oven-dry (DRY) soil samples. Data-points are classified by texture of the surface horizon according to ISPAID (2004): CL = Clay-loam, L = loam, and SICL = silty-clay-loam.

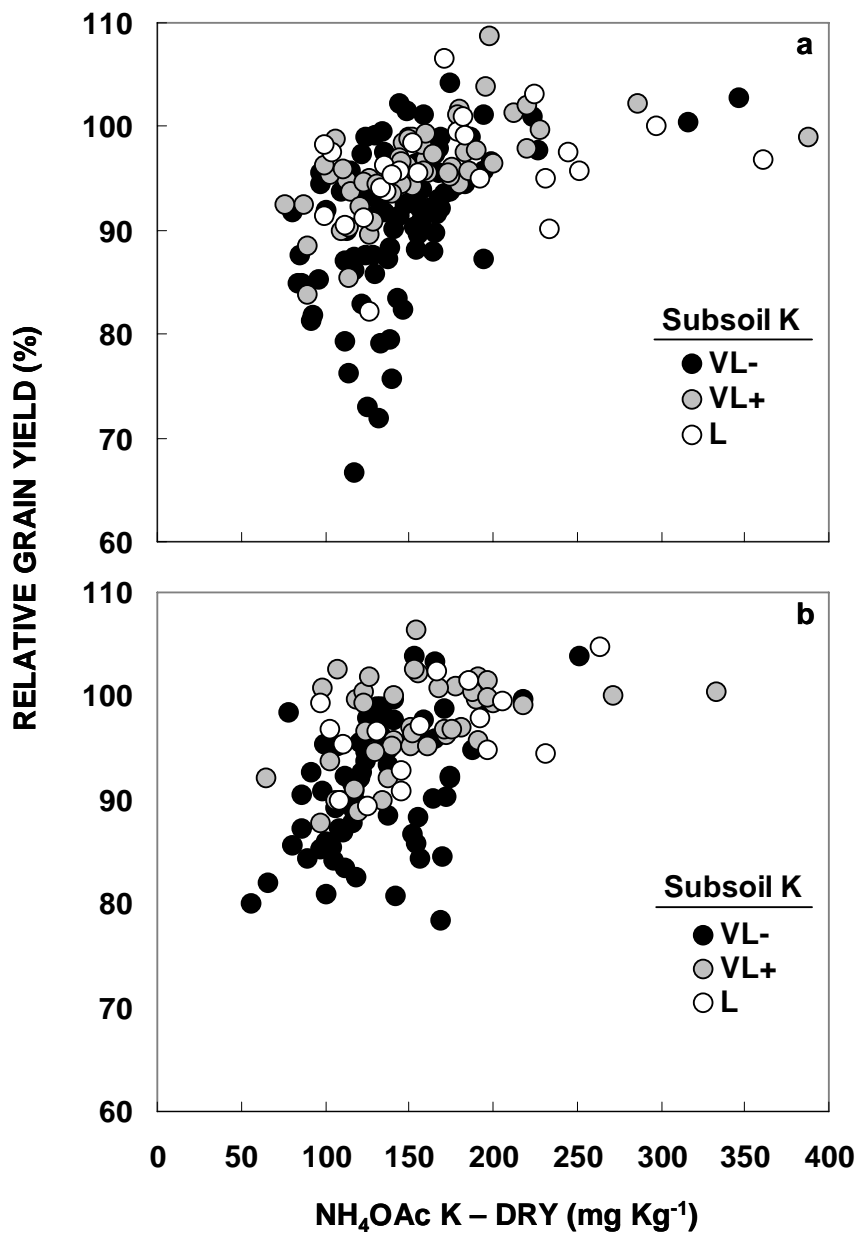


Figure 10. Relationships between relative corn (a) and soybean (b) yield and exchangeable K measured with ammonium acetate (NH₄OAc) based on oven-dry (DRY) soil samples. Data-points are classified by subsoil K level according to ISPAID (2004): VL- (<25 mg K kg⁻¹), VL+ (25 to 50 mg K kg⁻¹), and L (50 to 79 mg K kg⁻¹).

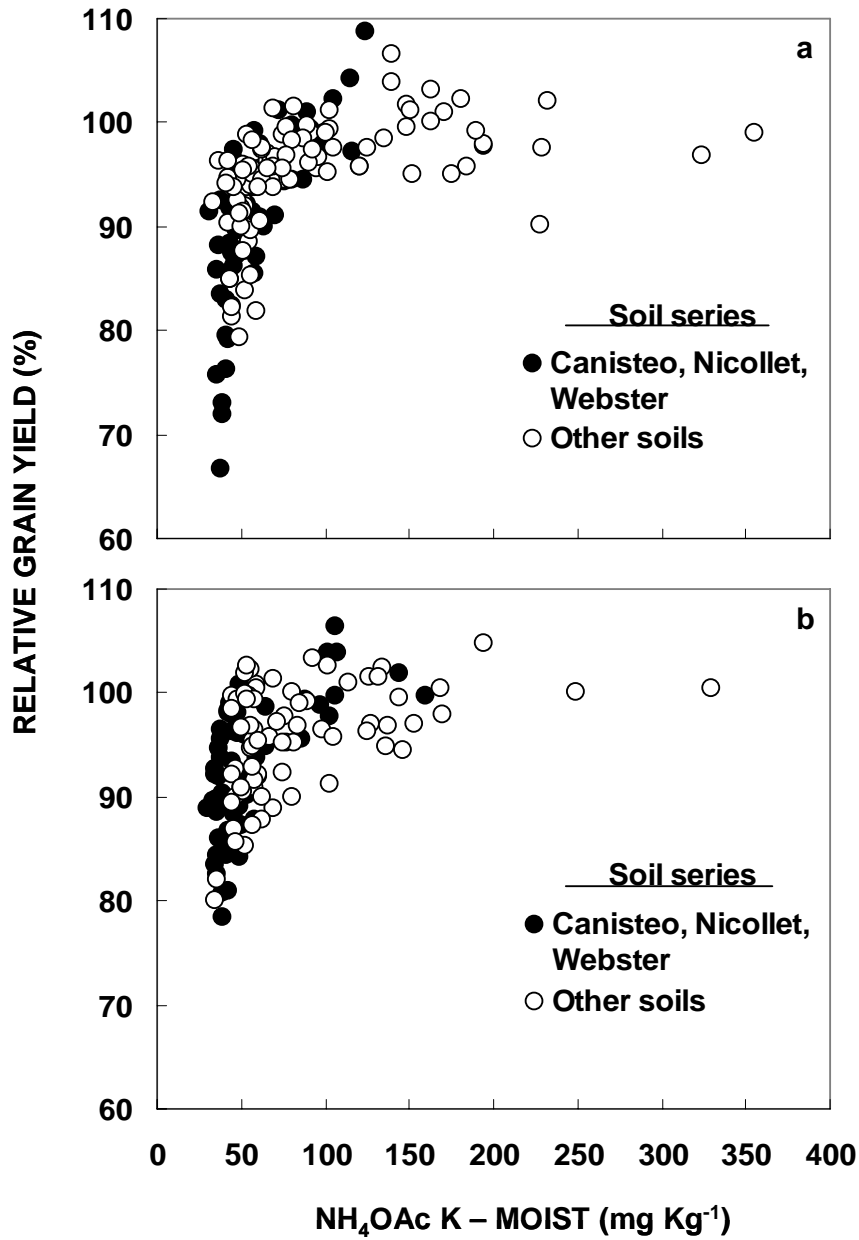


Figure 11. Relationships between relative corn (a) and soybean (b) yield and exchangeable K measured with ammonium acetate (NH_4OAc) based on field-moist soil samples (MOIST).

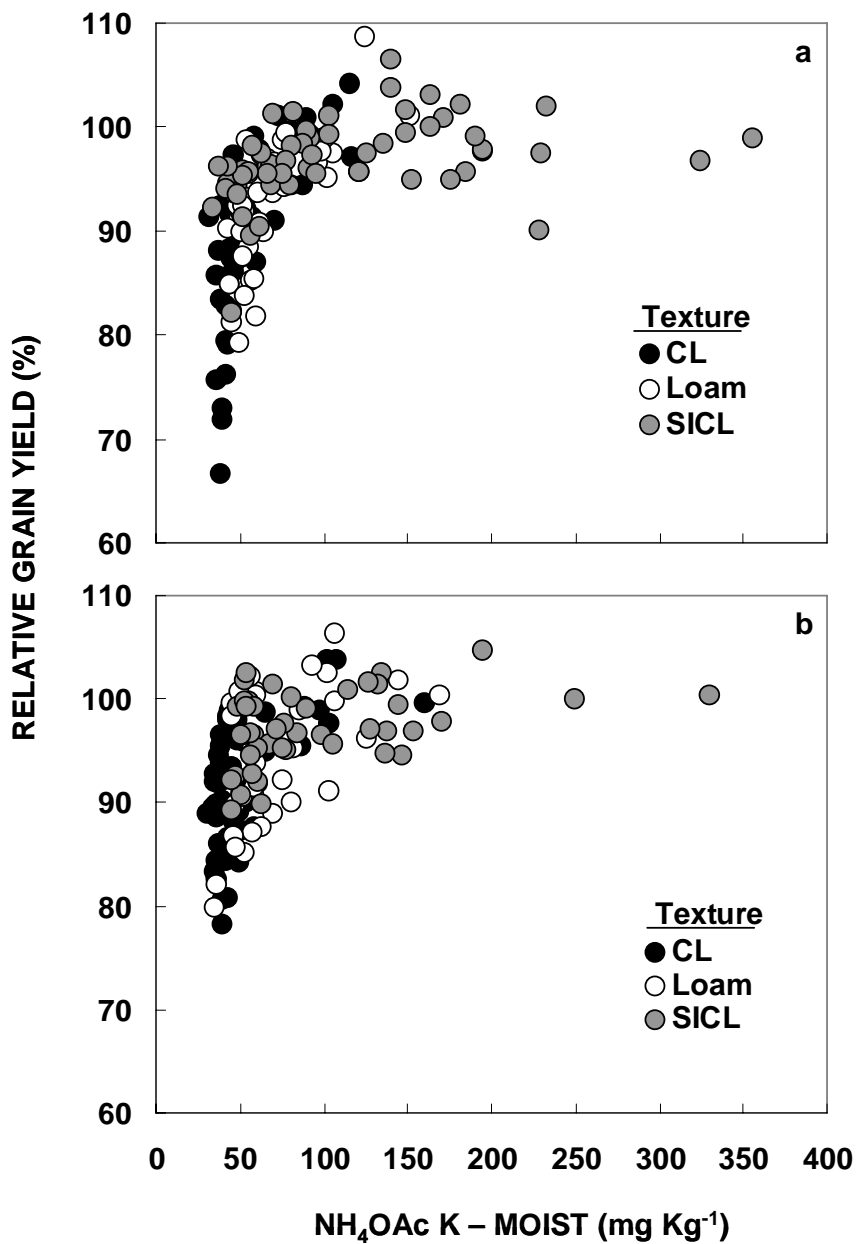


Figure 12. Relationships between relative corn (a) and soybean (b) yield and exchangeable K measured with ammonium acetate (NH_4OAc) based on field-moist soil samples (MOIST). Data-points are classified by texture of the surface horizon according to ISPAID (2004): CL = Clay-loam, L = loam, and SICL = silty-clay-loam.

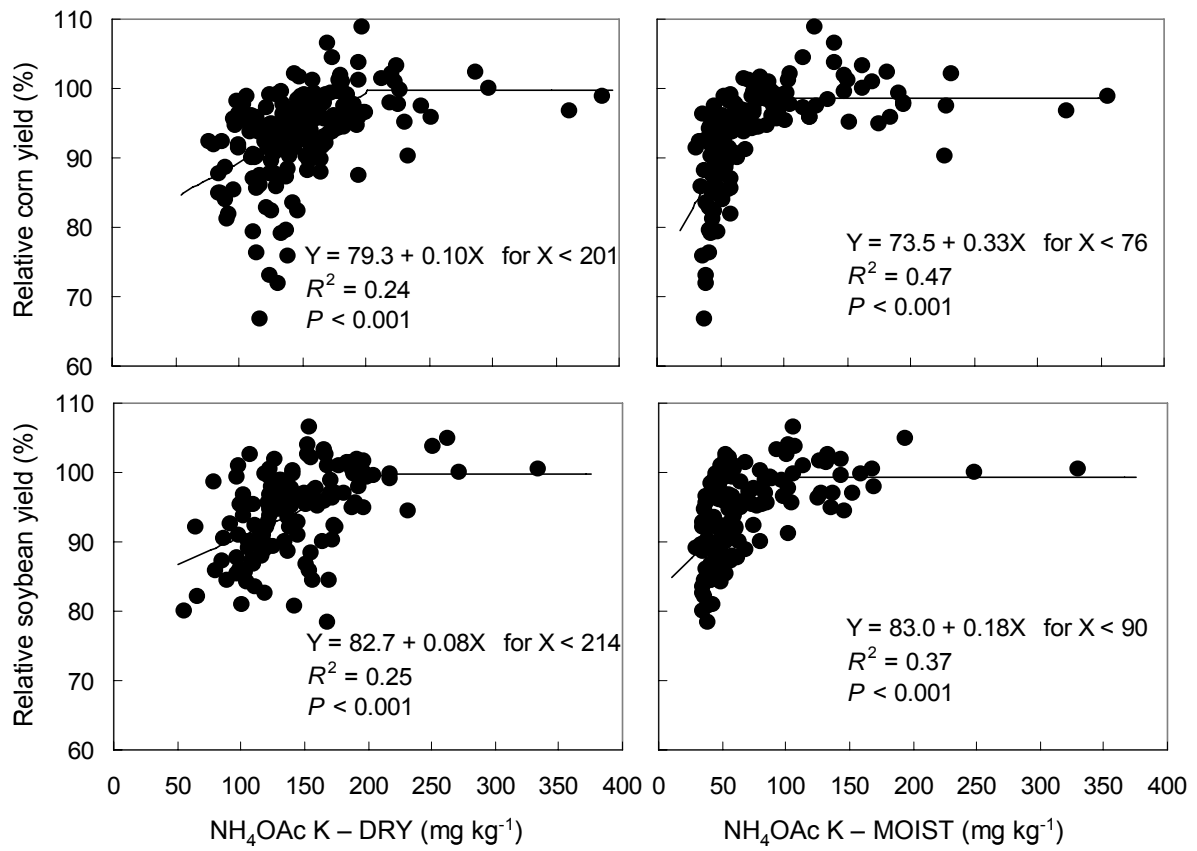


Figure 13. Relationships between relative corn and soybean yield and exchangeable K measured with ammonium acetate (NH₄OAc) based on oven-dry (DRY) and field moist (MOIST) soil samples, with linear-plateau model fitted to the data points.

**CHAPTER 3. FIELD CALIBRATION OF AMMONIUM ACETATE, MEHLICH-3,
AND SODIUM TETRAPHENYLBORON SOIL POTASSIUM TESTS FOR CORN
AND SOYBEAN**

A paper to be submitted to Soil Science Society of America Journal

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ABSTRACT

The ammonium-acetate (NH_4OAc) and Mehlich-3 (M3) extractants are the most widely used soil tests for K and estimate exchangeable K. Recent research has shown that these tests predict crop response to fertilization poorly, and laboratory studies have suggested that a test partially measuring nonexchangeable K could predict response better. The objectives of this study were to assess and compare the efficacy of NH_4OAc , M3, and sodium tetraphenylboron (NaBPh_4) soil K extractants in determining plant-availability of K by conducting field correlation and calibration studies with corn (*Zea mays* L.) and soybean [*Glycine max* (L.) Merr.] across several Iowa soils. Potassium response trials were conducted at 54 locations from 1999 to 2004 for a total of 63 site-years with corn and 54 site-years with soybean. Approximately similar soil K extraction and very high correlation between M3 and NH_4OAc tests indicated that these methods measure the same soil pool of soil K. The amount of NaBPh_4 -extractable K was significantly higher than amounts measured by the NH_4OAc or M3 tests, and the difference increased with increasing soil K and decreased as the Ca and Mg to K ratio increased. Ammonium-acetate and M3 tests showed similarly poor capacity to predict corn and soybean response to K fertilization. The NaBPh_4 test did not show a consistently superior capacity to predict corn and soybean response to K. Critical soil-test K concentration ranges defined by the CN and LP models across all soils for the NH_4OAc test were 133 to 216 mg K kg^{-1} for corn and 122 to 191 mg K

kg⁻¹ for soybean. Critical soil-test K concentration ranges for the M3 test were 128 to 199 mg K kg⁻¹ for corn and 114 to 185 mg K kg⁻¹ for soybean. These ranges encompass the current Optimum interpretation class used in Iowa for these tests. Critical concentration ranges for the NaBPh₄ test were 421 to 641 mg K kg⁻¹ for corn and 473 to 556 mg K kg⁻¹ for soybean. The results do not support adoption of the NaBPh₄ K test in production agriculture because its correlation with crop response is not consistently better than for currently used tests and laboratory procedures are much more laborious and expensive.

Abbreviations: Ammonium-acetate, NH₄OAc; Mehlich-3, M3; Sodium tetraphenylboron, NaBPh₄; Soil-test K, STK; Soil test critical concentrations, CC; Cate-Nelson, CN; linear-plateau, LP.

INTRODUCTION

Potassium is an essential nutrient for plant growth, and one of the three main macronutrients together with N and P. Potassium is present in the soil in water-soluble, exchangeable, nonexchangeable, and mineral forms (Martin and Sparks, 1985). These four forms of K give a general representation of soil K availability, but no distinct boundaries among them exist. Plants take up the water-soluble form of K, which is readily replenished by soil exchangeable K. Some nonexchangeable K can become exchangeable when solution and exchangeable K are depleted by plant removal, leaching, or exchange reactions with other cations. However mineral K, which is the major proportion of total K in soils, can become available only very slowly through soil weathering of mineral K (Martin and Sparks, 1985). Redistribution of K among these forms also occurs as K is added to soil as fertilizer, manure, or crop residues.

Soil testing is an important diagnostic tool to assess plant K availability to crops. Even when the importance of nonexchangeable K to plant nutrition is recognized over a long term, exchangeable K is considered to be one of the best indices of plant available K

(McLean and Watson, 1985). The estimate of exchangeable K with the neutral 1 M NH₄OAc extractant is the most widely used soil test for K and provides the basis for K fertilizer recommendations (Haby et al., 1990; Allen et al., 1994). Currently this is the recommended soil test for soils of the north-central region of the USA by the North-Central Regional Committee for Soil Testing and Plant Analysis (Brown, 1998) along with the Mehlich-3 (M3) method. Adoption of the M3 test (Mehlich, 1984) by soil testing laboratories is gaining in popularity in lieu of the traditional NH₄OAc (Haby et al., 1990; Allen et al., 1994; Eckert, 1994). The main reason is that M3 allows simultaneous extraction and determination of several elements in one procedure. Several authors (Beegle and Oravec, 1990; Eckert, 1994; Gartley et al., 2002; Wang et al., 2004) showed that these two extractants remove almost the same amount of K from soil. Therefore, regardless of improvements in laboratory efficiency and probable cost savings, simply changing from NH₄OAc test to M3 appears unlikely to improve predictability of fertilizer responses (Eckert, 1994).

However, some evidence indicates that the measurement of exchangeable K may not be the most reliable index of plant-available K for many temperate region soils (Bertsch and Thomas, 1985), particularly in those soils that contain substantial amounts of K fixing phyllosilicate clay minerals. The reliability of the NH₄OAc soil test method is influenced by the interaction between soil properties and environmental conditions, such as wet-dry and freeze-thaw cycles that influence K fixation and release to or from nonexchangeable forms (Cox et al., 1999). Part of the nonexchangeable K held in the interlayers of expandable clay minerals such as illite and vermiculite can be released relatively easily to supply a fraction of the K taken up by plants (Singh et al., 1983; Meyer and Jungk, 1993; Mengel and Uhlenbecker, 1993). Cox and Joern (1996) showed that the NH₄OAc method predicted plant-available K poorly in soils where nonexchangeable K contributed significantly to K nutrition in winter wheat (*Triticum aestivum* L.). Furthermore, NH₄OAc-extractable K underestimated plant available K in two soils with the greatest K fixing capacity (Cox et al.,

1999). Therefore an ideal soil-test for K should quantify the proportion of nonexchangeable K that may potentially become available to plants during the growing season.

The sodium tetraphenylboron (NaBPh_4) K extraction method, developed by Scott et al. (1960), has received attention as a potential method to estimate plant-available K due to its ability to extract exchangeable K and also part of the nonexchangeable forms of K in soils. Extraction of K by NaBPh_4 is a less drastic treatment of the mineral structures than extraction with boiling 1 M HNO_3 , which is the classic method used to measure nonexchangeable K in soils (McLean and Watson, 1985). Moreover, Scott and Reed (1960) demonstrated that the NaBPh_4 is an effective extractant for nonexchangeable K from mica structures. The BPh_4^- anion facilitates the release of nonexchangeable K by combining with K in solution and precipitating it as KBPh_4 , while Na exchanges with interlayer K (Scott and Reed, 1962). As a result, this method mimics the action of K uptake by plant roots by depleting soil solution K and promoting further release of exchangeable and nonexchangeable K. This mode of extraction may improve the ability of the NaBPh_4 test to predict plant K uptake, particularly when plants obtain considerable amounts of K from nonexchangeable forms and for a wide range of soil types and environmental conditions (Cox et al., 1999). Cox et al. (1996) modified the method described by Smith and Scott (1966) by using Cu^{2+} instead of Hg^{2+} to destroy the BPh_4^- anion and recover precipitated K. Cox et al. (1999) refined the method further by decreasing extraction time to facilitate its potential use as a soil-test method for K.

Wentworth and Rossi (1972) showed that NaBPh_4 -extractable K (1-h extraction) was highly correlated with K taken up by barley (*Hordeum vulgare* L.) from five layer silicate minerals. Cox et al. (1999) also showed that K measured by the modified NaBPh_4 test was a better predictor of K uptake by winter wheat than NH_4OAc because of the inability of the latter to measure plant-available nonexchangeable K. However, most published studies evaluating NaBPh_4 as a soil test for K have been conducted in the greenhouse under

controlled conditions. Schindler et al. (2002), working in South Dakota under field conditions, found no advantage of using NaBPh₄ over the common NH₄OAc to predict plant K concentration in corn. The reason NaBPh₄ was not a better test was that the clay fraction mineralogy of the soils studied was montmorillonite, which would not be expected to contribute much plant-available nonexchangeable K.

There is a lack of adequate field correlation and calibration data for the NaBPh₄ K test and commonly used soil tests for K. Voss (1998), based on a national survey of land grant universities in charge of conducting the majority of the research that supports fertilizer recommendations, strongly suggested a need to update the soil test calibration data supporting recommendations for many regions. Difficulties of getting funds for soil-test calibration research and publish this kind of research are among factors that explain a lack of extensive recent soil-test calibration data for many crops and different regions (Beegle, 2005). Relationships between soil tests and yield responses are, in general, relatively weak. Therefore, estimating the correlation of crop response with a new soil test through correlations between amounts of nutrient measured by the new test and an already calibrated test is a questionable methodology. Several studies have focused on correlations of amounts of K extracted by various soil tests (Hanlon and Johnson, 1984; Michaelson et al., 1987; Gartley et al., 2002; Wang et al., 2004), but few have used field calibration studies to compare common soil tests for K such as M3 and NH₄OAc (Beegle and Oravec, 1990; Mallarino and Blackmer, 1994), and even fewer have compared field calibrations for these tests and the NaBPh₄ test (Schindler et al., 2002). Furthermore, no field calibrations of soil-test K for soybean have been published in or around the north central region of United States during the last 20 years.

The objectives of this study were to assess and compare the efficacy of NH₄OAc, M3, and NaBPh₄ soil test extraction methods to measure plant-available K in soils by conducting

field correlation studies with corn and soybean crops across several Iowa soils, and to establish critical concentration ranges for the tests.

MATERIALS AND METHODS

Corn and soybean grain yields and soil samples for this study were collected from K response trials conducted from 1999 to 2004 at 54 Iowa locations that included 20 counties. There were 19 single-year trials and 35 trials evaluated 2, 3, or 4 years by re-applying treatments for successive crops, which resulted in 117 site-years of data (63 with corn and 54 with soybean). The soils included 30 typical Iowa soil series in which row-crop production predominates (Table 1). Nitrogen and P fertilizers were applied uniformly to plots of all trials following Iowa State University recommendations for these nutrients (Sawyer et al., 2002; Blackmer et al., 1997). Other crop management practices, such as tillage, corn hybrids, soybean cultivars, seeding dates and rates, and weed control, were those normally used by the farmers.

Twenty-six single-year or multi-year trials for corn-soybean rotations involved a small-plot methodology, plot size ranging from 54 to 108 m², randomized complete-block or split-plot designs (when any additional treatment was also evaluated) with three to six replications, and hand application of K treatments (commercial KCl fertilizer). Twelve trials evaluated five K fertilization rates (0 to 168 kg K ha⁻¹) for crops managed with chisel-plow/disk tillage. Ten long-term trials established in 1994 evaluated three annual K fertilization rates (0 to 65 kg K ha⁻¹) broadcast or banded with the planter for crops managed with no-till or chisel-plow/disk tillage. Two long-term trials established in 1979 evaluated the factorial combinations of 0, 22, and 44 kg P ha⁻¹ and 0, 67, and 134 kg K ha⁻¹ broadcast annually for crops managed with chisel-plow/disk tillage. Plots that received the highest P rate and either no K or the highest K rate were used for this study. Two other long-term K experiments established in 1976 evaluated effects of several initial contrasting soil-test K

levels and four broadcast annual K rates (0 to 100 kg K ha⁻¹) superimposed to the initial-K treatments for crops managed with chisel-plow/disk tillage. Partial yield results for some treatments and details of the long-term trials were published before (Mallarino et al., 1991a and 1991b; Bordoli and Mallarino, 1998; Mallarino et al., 1999; Borges and Mallarino, 2000), but results since 1999 used for this study have not been published.

Twenty-eight field-scale strip trials evaluated two K fertilization rates (0 and 100 to 186 kg K ha⁻¹, depending on the field) broadcast to strips measuring 18.3 m in width and 250 to 450 m in length depending on the field. The K treatments (commercial KCl fertilizer) were applied with commercial fertilizer spreaders and were arranged in randomized complete-block designs with three or four replications.

Composite soil samples (12 cores, 0-15 cm depth) were collected in the fall after harvest of the previous crop and before applying K treatments. For single-year or first-year of multi-year small-plot trials, the samples were collected from each replication and averaged to provide one soil-test K value for the experimental area. For following years of multi-year trials, samples were collected from plots receiving no K fertilizer. For the strip trials, soil samples were collected using a systematic, grid-point sampling method (Wollenhaupt et al., 1994) adjusted to the field design. The width of the grid cells across the strips coincided with the width of a replication (36.6 m) and the length was also 36.6 m, which resulted in 0.134 ha cells. The soil cores for each composite sample were collected following a random pattern from areas approximately 100-m² in size at the center of each grid cell.

The soil samples were stored in sealed plastic-lined bags at 5°C from 2 to 10 weeks after sampling. Soil moisture content was determined by the difference between weight of field-moist and dried subsamples at 40°C to constant weight. The samples were dried at 40°C, crushed to pass through a 2-mm sieve, mixed thoroughly, and stored in plastic-lined bags. Soil K was extracted with the NH₄OAc, M3, and NaBPh₄ tests. All soil analyses described in this section were conducted on duplicate soil samples. Briefly, the NH₄OAc

method (Warncke and Brown, 1998) involves extracting soil K by using a 1M NH₄OAc solution buffered at pH 7.0, a soil/solution ratio of 1:10 (w/v), and shaking at 200 oscillations min⁻¹ for 5 min. The Mehlich-3 method (Mehlich, 1984) involves extracting K by using a 0.2 N CH₃COOH, 0.25 N NH₄NO₃, 0.015 N NH₄F, 0.013 N HNO₃, and 0.001M EDTA solution, a soil/solution ratio of 1:10 (w/v) and an extraction time of 5 min, and measuring K by atomic absorption spectroscopy. The NaBPh₄ extractable K was determined with a procedure similar to that used by Cox et al. (1999). Samples of 1 g soil were weighed into Folin Wu tubes and 3 mL of extracting solution (0.167 M NaBPh₄ + 1.7 M NaCl + 0.01 M EDTA) was added. The incubation time was 5 min and after that 25 mL of quenching solution (0.5 M NH₄Cl + 0.11 M CuCl₂) was added to the tubes to stop K extraction. The tubes were placed in a digestion block on a hot plate at 150°C until the precipitate dissolved completely (40 min). The suspension in the tubes was diluted to 100 mL with deionized water, mixed, and a 40-mL aliquot of the supernatant was filtered (filter paper Whatman 42) into 50-mL tubes containing 4-5 drops of 6 M HCl. The acidification of the extract helps prevent precipitation of Cu²⁺ and the breakdown products of NaBPh₄, if extracts need to be stored for 1 d. The extract was diluted (1:10) with deionized water and K was determined with an atomic absorption spectrophotometer.

The cation exchange capacity (CEC) of the soils was estimated by summation of exchangeable Ca, Mg, K, Na, and neutralizable soil acidity (Warncke and Brown, 1998). Soil pH (based on a 1:1 soil/water ratio) ranged from 5.4 to 8.1 across sites, averaging 6.4. Soil organic C was measured following the combustion method described by Wang and Anderson (1998) using a LECO CHN-2000 analyzer (LECO Corp., St. Joseph, MI). Levels of soil organic matter (SOM) averaged 46 g kg⁻¹ across sites and ranged from 26 to 82 g kg⁻¹.

Corn and soybean grain from small-plot trials was harvested with plot combines (15-m length of three or four rows) or by hand (7.5-m length of two rows) from the center of each plot. Grain yield from strip trials was measured using combines equipped with commercial

impact flow-rate yield monitors, moisture sensors, and global positioning system (GPS) receivers following a methodology used and described before in detail for evaluating various treatments (Bermudez and Mallarino, 2002; Wittry and Mallarino, 2004). Briefly, grain yield monitor data recorded every 1 s from experimental areas at least 40 m away from any field border were imported into ArcView GIS (Environmental Systems Research Institute, Redlands, CA). Any data point affected by common yield monitor errors (such as effects of waterways and unexpected combine stops) was deleted. Also, only data from combine passes from the center of each treatment strip (unaffected by treatment borders) were kept (for corn two passes 4.5 or 6 m wide each and for soybean either two passes 4.5 to 7.5 m wide each or one pass 9 m wide. Yields were always corrected to 155 g kg⁻¹ moisture for corn and to 130 g kg⁻¹ moisture for soybean. Data from yield monitor points were averaged for small areas defined by the width of each treatment strip and the separation distance of grid soil sampling lines along strips (0.067 ha). Therefore, the small field area represented by the two K treatments match the field area represented by each grid soil-sampling cell (0.134 ha). Afterward data pairs were averaged by site and soil series, considering only soil series present in at least three cells within a field. The data inputs for these analyses were calculated using ArcView GIS by overlaying yield maps, initial soil-test K (STK) maps, experimental layouts, and digitized (scale 1:12000) soil survey maps (Iowa Cooperative Soil Survey, 2003).

The grain yield data used in this study is expressed as relative responses to K. Relative response was calculated for each site-year by dividing the mean yield of non-fertilized plots by the mean of the highest K fertilization treatment and multiplying the result by 100. In small-plot trials that evaluated K placement methods, means of the highest K rate were calculated across all methods because yield response was seldom affected by the K placement method. For small-plot trials, a data pair (relative yield and soil-test K) is represented in figures by one point and represents one site-year. The soil-test K value

represents the entire experimental area for 1-yr trials and the mean of the non-fertilized plots for trials evaluated more than 1 yr. For strip trials, a data point represents a pair of data (relative yield and soil-test K) for each dominant soil series present in the experimental areas. A dominant soil series was defined as the series that occupied the entire area of three 0.134 grid-sampling cells.

Soil test critical concentrations (CC) were calculated with the statistical Cate-Nelson (CN) method (Cate and Nelson, 1971) and with the linear-plateau (LP) and quadratic-plateau (QP) segmented models (Waugh et al., 1973). The critical concentration defined by the CN method was determined with the General Linear Models (GLM) procedure of SAS (SAS Institute, 2000) as the value that split the yield response data into the two groups that accounted for the largest proportion of the total variability (R^2). Critical concentrations defined by the segmented models were determined with the Nonlinear Model (NLIN) procedure of SAS, and represent the soil-test values at which the two portions of each model joined.

RESULTS AND DISCUSSION

Amounts of Potassium Extracted by the Soil Tests

Soil-test K levels across sites ranged from 56 to 347 mg K kg⁻¹ for NH₄OAc, from 50 to 328 mg K kg⁻¹ for M3, and from 186 to 1240 mg K kg⁻¹ for NaBPh₄. There was a high correlation between amounts of K measured by the NH₄OAc and the M3 tests (Fig. 1), a result that should be expected because both extractants have NH₄ ions in its composition and primarily measure concentrations of exchangeable K in soil. Comparable relationships between these two tests have been reported by others (Michaelson et al., 1987; Alva, 1993; Gartley et al., 2002; Wang et al., 2004). Beegle and Oravec (1990), working in field calibration studies conducted at 67 sites in Pennsylvania, also reported a significant relationship between NH₄OAc and M3 extractable K. Correlations between the amounts of

K extracted by NH_4OAc or M3 and the NaBPh_4 test were not as high (Fig. 2), a finding to be expected because different soils have variable amounts of potentially extractable nonexchangeable K. Schindler et al. (2002), working with six soils from east-central South Dakota, found a higher correlation ($r = 0.95$) between K measured by NH_4OAc and NaBPh_4 (5-min incubation). The amounts of K measured using NaBPh_4 test in our study were 45 to 518% higher than K measured by the NH_4OAc test, and 50 to 515% higher than K measured by M3 test, averaging 239 and 254% higher, respectively across all soils and sites. In general, differences between K measured by NH_4OAc or M3 tests and NaBPh_4 were larger at high STK values, which explain a poor correlation across all soils (Fig. 2). The results show that no single factor may possibly be used to express results of one test based on the other. Data in Fig. 3 show that the relative difference between K measured by NaBPh_4 and NH_4OAc or M3 tests (their ratio) and the absolute difference increased with increasing soil K (NaBPh_4). However, the strength of the relationship was weaker for the relative difference (Fig. 3b) compared with the absolute difference.

The additional K measured with the NaBPh_4 test compared with NH_4OAc or M3 tests was negatively related with Ca and Mg to K ($\text{Ca} + \text{Mg} / \text{K}$) ratio (Fig. 4). McLean and Watson (1985) proposed that when exchangeable K is depleted to its critical level (low K saturation in the exchange complex or high Ca and Mg to K ratio), and there is a low concentration of K ions in solution (due to crop removal, for example), K release from nonexchangeable pools is favored to allow further K uptake, which would reduce the amount of nonexchangeable K potentially extractable by NaBPh_4 . Similar, but much weaker, trends were observed for relationships between CEC, total bases, and SOM and the relative or absolute difference in K measured with NaBPh_4 and the NH_4OAc or M3 tests (not shown). Interpretations of cause and effect are difficult and speculation is risky, however, because CEC, total bases, and SOM are normally related with soil texture (the finer the soil texture the higher the CEC, total bases, and SOM). No significant ($P \leq 0.05$) relationship was found

between soil pH and the relative or absolute difference between NaBPh₄ test and the NH₄OAc or M3 tests (not shown).

Cox et al. (1999) stated that NaBPh₄ test may provide reliable estimates of K supply for a wide range of soil types and environmental conditions because it is less sensitive to sample drying. The differences between amounts of K extracted by NaBPh₄ test and the NH₄OAc or M3 tests across sites in our study were not related with the soil moisture content at the time of collecting the samples (not shown).

Field Correlation of Yield Response to Potassium

Field response trials that consider crop production conditions and include wide STK ranges offer an appropriate basis for comparing soil tests and for soil-test correlation and calibration. The wide range of growing conditions evaluated in this study resulted in grain yields of fertilized plots ranging from 5.0 to 14.4 Mg ha⁻¹ for corn and from 1.1 to 5.0 Mg ha⁻¹ for soybean, and averaged 10.9 and 3.3 Mg ha⁻¹, respectively (means of the treatment that received the maximum K rate at each site). Analysis of variance of K effects on yields for individual sites indicated a significant yield increase ($P \leq 0.05$) in 40% of the site-years with corn and 33% of the site-years with soybean (not shown).

Relationships between relative corn and soybean yield and soil K measured with the NH₄OAc and M3 tests are shown in Fig. 5. These data show the classic relationship between yield response and soil-test values. However, although the highest portion of the curve indicates relative yield values within about 5% of a 100% yield level, there is very high variation at medium to low soil-test K levels. These results indicate a poor capacity of the two tests to predict crop response to K fertilization, particularly for soybean. The high variability observed likely did not originate from experimental error because we included three to six replications in the trials, the laboratory analyses were done in duplicate, and similar variation was observed for on-farm strip trials and for conventional small-plot trials at research farms where sources of error could have been controlled better. Only in a few

instances the lack of crop response at low soil K levels, and the large variation in the relationship at these levels, could be explained by low yield levels (for example, yields limited by low rainfall in some site-years). Therefore, we conclude that the soil-tests are the cause of the variation and poor prediction of crop response when soil-test levels are low to medium. Beegle and Oravec (1990) and Mallarino and Blackmer (1994) also showed poor relationships between corn relative yield and soil K extracted with NH_4OAc or M3 tests.

The distribution of the data points in Fig. 5 suggests different relationships for two groups of soil series. The white data points represent results for soils in which STK levels ranging from approximately 130 to 150 mg K kg^{-1} produced more than 95% corn relative yield. This STK range is suggested by data in the figures. The black data points represent results for soil series for which the critical concentration range is higher than for other soil series and could not be determined with certainty but seems to be around 170 to 180 mg K kg^{-1} for NH_4OAc and M3 tests. These soil series are Nicollet, Webster, and Canisteo soils developed on glacial till materials, which predominate in central and north-central Iowa but sometimes include other Iowa soil series. The apparently different relationship for the two groups of soils was less clear in soybean than in corn. All the soils represented by black data points have in common deep profiles, somewhat poor to very poor drainage, moderate to poor permeability, slope from 0 to 4%, texture from loam (Nicollet) to clay loam (Webster and Canisteo) in the top 6 inches, and high CEC, exchangeable Ca, and SOM compared with other Iowa soils. A few of these soils, such as Canisteo, have high pH due to elevated calcium carbonate concentration. Although data in Fig. 5 suggest different STK requirements for different soils when K was extracted by NH_4OAc and M3 tests, because of the wide data spread below a STK value of about 180 mg K kg^{-1} , especially in soybean, no adjustments for soil series can be reasonably supported.

Figure 6 shows the same relationships shown in Fig. 5 but with data points classified according to the texture of the surface horizon (from ISPAID, 2004). Apparently sites with

loam and silty-clay-loam surface texture, represented by white and grey dots, had lower K requirements for crop production than sites with clay-loam surface texture. However this is not as clear for soybean as it is for corn. It must be noted that the soil textures used are from the Iowa soil database and that budget limitations did not allow for measurement of soil texture for the plots of this study. Vitosh et al. (1995) stated that some soils, especially high clay soils from Ohio, require higher K levels to support optimum crop production than other lower clay content soils. They incorporated this information into the Indiana, Michigan and Ohio K fertilizer recommendations by increasing the critical level for K as the CEC increases. Hoefl and Peck (2001) also classified Illinois soils based on CEC into two general regions having high or low CEC, with a threshold level of 12 cmol kg^{-1} . In our study, however, classifying data points in Figs 5 and 6 according to CEC using different thresholds (not shown) did not result in a more clear discrimination of responsive sites than that shown for texture.

Subsoil K levels are considered for soil-test K interpretations in Iowa. The recommended soil-test K levels for production of corn and soybean (Sawyer et al., 2002) are higher for soil series with NH_4OAc or M3 K levels lower than 50 mg K kg^{-1} . In Table 1 we classified the sites of this study following the more detailed subsoil K classification used by the Iowa Soil Properties and Interpretations Database (ISPAID, 2004). The distribution of points in Fig. 7 indicate that approximately one-half of site-years classified as having the lowest subsoil K levels (VL-, $<25 \text{ mg K kg}^{-1}$) showed the highest corn or soybean response to K fertilization. However, the K response variability and apparent STK needed in surface soil of site-years having VL- subsoil K levels was high. The site-years that encompassed soils with L and VL+ subsoil K levels (25 to 79 mg K kg^{-1}) showed lower response to K and apparently less variability.

Several reasons could explain different soil K requirements across soils and large response variation across soils with similar NH_4OAc and M3 K levels. Barbagelata and

Mallarino (Chapter 2 of this dissertation) showed that one likely reason is a variable increase in NH_4OAc -extractable K when soil samples are dried before the analysis. Drying the soil samples would likely have the same effect on M3-extractable K, since this test measures soil K from the same pool than NH_4OAc test. Previous research in Iowa and in the north-central US also showed that extracting soil K with the NH_4OAc test from field-moist samples was a better predictor of plant availability of K than air-dry or oven-dry K tests (Luebs et al., 1956; Hanway et al., 1962). Cox et al. (1996) showed that the NaBPh_4 test could improve estimate of plant-available K because it partially extracts nonexchangeable K and is less sensitive to sample drying.

Figure 8 shows the relationships between relative corn and soybean yields and soil K measured by the NaBPh_4 test. The results show that the NaBPh_4 test has superior capacity to predict soybean response to K than the NH_4OAc or M3 tests. However NaBPh_4 did not clearly improved the predictability of corn response to K compared with the other tests. The NaBPh_4 test did extract proportionally less K than NH_4OAc and M3 extractants from soils represented by black points in Fig. 8, for which the NH_4OAc and M3 tests suggest that higher soil K is needed to produce a certain corn or soybean relative yield level. Soils series represented by white and black data points seem to blend into the same trend for both corn and soybean (Fig. 8). However, this difference between tests clearly improved the field correlation for soybean but did not help much for corn. In corn, soil series represented by white points at intermediate NaBPh_4 values seems to show higher variation compared to other soils. Sites with different surface soil textures represented by white, gray, and black data points also blend into the same relationship when STK was measured by NaBPh_4 for both crops. The same occurred with the different subsoil K classifications (Fig. 8).

The difference between crops could not be explained with the methods used in this study, although it may have been related to variation in soil conditions between sites planted to corn and soybean. Schindler et al. (2002) did not find an advantage of using NaBPh_4

over the NH_4OAc test to estimate plant K concentration in corn. They explained this lack of benefit of using NaBPh_4 because the clay fraction mineralogy of the soils studied was montmorillonite, which would not be expected to contribute much plant-available nonexchangeable K. The clays of most Iowa soils included in this study are dominantly smectites (montmorillonite) with appreciable amounts of mica (illite) and small amounts of other clay minerals (Ruhe, 1984; Khan, 1985).

Table 2 shows critical soil-test K concentrations for NH_4OAc , M3, and NaBPh_4 K tests across all sites calculated by the CN, LP, and QP models for relationships shown in Figs. 5 and 8. Critical concentrations for each of the three soil tests were approximately similar for corn and soybeans, but varied markedly depending on the model used. Others also showed large differences in soil critical concentrations depending on the model used (Dahnke and Olson, 1990; Mallarino and Blackmer, 1992; Mallarino and Blackmer, 1994). The STK critical concentrations for NH_4OAc and M3 tests were approximately similar. There is no clearly superior or widely accepted method for establishing economically optimum critical concentration ranges (Dahnke and Olson, 1990; Mallarino and Blackmer, 1992). Previous research (Mallarino and Blackmer, 1992; Mallarino and Blackmer, 1994) showed that short-term economically optimum soil critical concentrations usually are within a range of critical concentrations defined by the CN and LP models. These authors demonstrated that critical concentrations defined by the QP model usually are much higher than those defined by the CN and LP models, and their use resulted in smaller returns to fertilization across many fields. Critical soil-test K concentration ranges defined by the CN and LP models in this study for NH_4OAc test were 133 to 216 mg kg^{-1} for corn and 122 to 191 mg kg^{-1} for soybean. Critical soil-test K concentration ranges defined by these two models for the M3 test were 128 to 199 mg kg^{-1} for corn and 114 to 185 mg kg^{-1} for soybean. As expected, the critical concentrations determined for the NaBPh_4 test were considerably higher than those determined for the NH_4OAc and M3 tests for corn and soybean, since the

former method measures exchangeable and part of the nonexchangeable forms of K in soil. The critical concentration ranges defined by CN and LP models for the NaBPh₄ test were 421 to 641 mg kg⁻¹ for corn and 473 to 556 mg kg⁻¹ for soybean.

The critical concentration ranges defined by the CN and LP models for the NH₄OAc and M3 tests encompass (are somewhat wider) the Optimum class of current Iowa STK interpretations for corn and soybean for most soils of the state (131-170 mg K kg⁻¹ for both soil-tests and crops, Sawyer et al., 2002). Only K fertilization to maintain STK based on expected crop K removal is recommended within this category. The current Iowa interpretations recommend higher soil-test K levels for corn and soybean than those recommended for soils in the neighboring states of Minnesota, Nebraska, and South Dakota (Gerwing and Gelderman, 1998; Rehm et al., 2001; Shapiro et al., 2001) but not in Illinois (Hoeft and Peck, 2001). Applying the current optimum interpretation range (131-170 mg K kg⁻¹) used in Iowa for the NH₄OAc and M3 tests to corn and soybean responses in this study corresponds to mean relative response values of 94 to 95%, respectively. The ranges identified by the CN and LP models for the NaBPh₄ test correspond to mean relative response of 95 and 98 for corn and soybean, respectively. The range identified by the CN and LP models for the NaBPh₄ test for corn was noticeably (265%) wider than for soybean, most likely because of a better-defined distinction between responsive and nonresponsive sites for soybean with this test.

CONCLUSIONS

Amounts of soil K measured with the NH₄OAc and M3 K tests being used in Iowa and other regions were approximately similar and very highly correlated, indicating that the two methods measure soil K from the same pool of plant-available K. The amount of NaBPH₄-extractable K was significantly higher than amounts measured by the NH₄OAc or M3 tests. The additional amount of K extracted by the NaBPH₄ test increased with

increasing soil K measured by NH_4OAc or M3 tests and decreased as the Ca and Mg to K ratio increased.

The results showed that NH_4OAc and M3 tests have similarly poor capacity to predict corn and soybean response to K fertilization. The amount of K extracted by these tests had a different meaning in terms of crop response to K fertilization for different soil series. However, this difference was not consistent across trials established at sites with similar soils, different critical concentrations could not be clearly established, and the differences could not be fully explained by measured soil properties (such as texture, CEC, cation ratios, or pH). Critical soil-test K concentration ranges defined by the CN and LP models across all sites for NH_4OAc test were 133 to 216 mg K kg^{-1} for corn and 122 to 191 mg K kg^{-1} for soybean. Critical soil-test K concentration ranges for the M3 test were 128 to 199 mg K kg^{-1} for corn and 114 to 185 mg K kg^{-1} for soybean. These ranges encompass (are somewhat wider) the current Optimum STK interpretation class used in Iowa for these tests, for which only maintenance K fertilization is recommended.

The NaBPH_4 test showed a superior capacity to predict soybean response to K compared with NH_4OAc or M3 tests. The NaBPH_4 test results did not clearly show the soil series differences observed for the other tests, and blended extracted K values and relative yield responses into the same general relationship. The improvement in this relationship by the NaBPH_4 test was mainly explained by proportionally less K extracted by this test from soils in which the NH_4OAc or M3 tests indicated that higher soil K levels were needed to produce a certain corn or soybean relative yield level compared with other soils. However relationships between STK and relative corn yield responses were not improved by the NaBPH_4 test. This result could not be explained with the methods used in this study. No true crop effect can reasonably explain the differences between crops, and results probably are explained by different soil and growing conditions between corn and soybean trials.

Critical concentration ranges defined by CN and LP models for the NaBPh₄ test were 421 to 641 mg kg⁻¹ for corn and 473 to 556 mg kg⁻¹ for soybean.

Overall, the results provided no strong support for adopting the NaBPh₄ K test in production agriculture for two main reasons. First, results showed that its correlation with crop response is not consistently better than those for the currently used tests. Second, although modifications of the original NaBPh₄ test reduced the time needed for the analysis, the laboratory procedures still are much more laborious and expensive than procedures for commonly used tests and its adoption by routine soil testing laboratories is very unlikely.

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Table 1. Information on soils for trials evaluating the response of corn and soybean to K fertilization

Series	Subgroup	Family	Subsoil K [†]	Texture‡
Aredale	Typic Hapludolls	Fine-loamy, mixed, mesic	L	L
Calco	Cumulic Endoaquolls	Fine-silty, mixed (calcareous), mesic	VL+	SICL
Canisteo	Typic Endoaquolls	Fine-loamy, mixed (calcareous), mesic	VL-	CL
Clarion	Typic Hapludolls	Fine-loamy, mixed, mesic	VL+	L
Clyde-Floyd	Typic Endoaquolls	Fine-loamy, mixed, mesic	VL-	CL
Colo	Cumulic Endoaquolls	Fine-silty, mixed, mesic	VL+	SICL
Colo-Ely	Cumulic Endoaquolls	Fine-silty, mixed, mesic	VL+	SICL
Crippin	Aquic Hapludolls	Fine-loamy, mixed, mesic	VL-	L
Dinsdale	Typic Argiudolls	Fine-silty, mixed, mesic	VL+	SICL
Donnan	Aquollic HapludalFs	Fine-loamy over clayey, mixed, mesic	VL+	L
Galva	Typic Hapludolls	Fine-silty, mixed, mesic	VL+	SICL
Kenyon	Typic Hapludolls	Fine-loamy, mixed, superactive, mesic	VL-	L
Killduff	Dystric Eutrudepts	Fine-silty, mixed, mesic	VL+	SICL
Klinger	Aquic Hapludolls	Fine-silty, mixed, mesic	VL+	SICL
Klinger-Maxfield	Aquic Hapludolls	Fine-silty, mixed, mesic	VL+	SICL
Lawler	Aquic Hapludolls	Fine-loamy over sandy or sandy-skeletal, mixed, mesic	VL+	L
Mahaska	Aquertic Argiudolls	Fine-montmorillonitic, mesic	L	SICL
Marshall	Typic Hapludolls	Fine-silty, mixed, mesic	L	SICL
Muscatine	Aquic Hapludolls	Fine-silty, mixed, mesic	VL+	SICL
Nevin	Aquic Argiudolls	Fine-silty, mixed, mesic	L	SICL
Nicollet	Aquic Hapludolls	Fine-loamy, mixed, mesic	VL+	L
Olin	Typic Hapludolls	Coarse-loamy, mixed, mesic	VL-	FSL
Otley	Oxyaquic Argiudolls	Fine-montmorillonitic, mesic	VL+	SICL
Readlyn	Aquic Hapludolls	Fine-loamy, mixed, mesic	VL+	L
Saude	Typic Hapludolls	Coarse-loamy over sandy or sandy-skeletal mixed, mesic	VL+	L
Spillville	Cumulic Hapludolls	Fine-loamy, mixed, mesic	VL+	L
Taintor	Vertic Argiaquolls	Fine-montmorillonitic, mesic	VL+	SICL
Tama	Typic Argiudolls	Fine-silty, mixed, mesic	VL+	SICL
Webster	Typic Endoaquolls	Fine-loamy, mixed, mesic	VL-	CL
Wiota	Typic Argiudolls	Fine-silty, mixed, mesic	VL+	SIL

[†]Subsoil K (30-60 cm depth), Very Low minus (VL-) <25 mg K kg⁻¹, Very Low plus (VL+) 25-50 mg K kg⁻¹, Low (L) 50-79 mg K kg⁻¹. (ISPAID, 2004).

[‡]Texture of the surface horizon, CL= Clay loam, FSL= Fine sandy loam, L= Loam, SICL= Silty clay loam, SIL= Silty loam (ISPAID, 2004).

Table 2. Critical concentrations of soil-test K for corn and soybean estimated by ammonium acetate (NH₄OAc), Mehlich-3, and sodium tetraphenylboron (NaBPh₄) tests and three models.

Crop	Soil test	Model†	Equation	R ²	CC‡	
					mg K kg ⁻¹	
Corn	NH ₄ OAc	CN	na§	0.24	133	
		LP	$Y = 78.1 + 0.105X$	0.28	216	
		QP	$Y = 74.5 + 0.17X - 0.00026X^2$	0.27	325	
	Mehlich-3	CN	na	0.25	128	
		LP	$Y = 76.7 + 0.121X$	0.33	199	
		QP	$Y = 72.2 + 0.20X - 0.00034X^2$	0.31	294	
	NaBPh ₄	CN	na	0.32	421	
		LP	$Y = 78.0 + 0.03X$	0.31	641	
		QP	$Y = 74.8 + 0.05X - 0.00003X^2$	0.29	972	
	Soybeans	NH ₄ OAc	CN	na	0.18	122
			LP	$Y = 81.7 + 0.085X$	0.24	191
			QP	$Y = 75.0 + 0.19X - 0.00039X^2$	0.23	243
Mehlich-3		CN	na	0.21	114	
		LP	$Y = 82.0 + 0.09X$	0.24	185	
		QP	$Y = 74.3 + 0.21X - 0.0005X^2$	0.23	227	
NaBPh ₄	CN	na	0.44	473		
	LP	$Y = 75.6 + 0.04X$	0.44	556		
	QP	$Y = 67.09 + 0.09X - 0.0001X^2$	0.42	738		

† CN, Cate-Nelson; LP, linear-plateau; and QP, quadratic-plateau. The LP and QP models apply for X less than or equal to the critical concentration, which is the value at which the plateau portion of each model starts. The statistical significance of the relationships always was $P \leq 0.01$.

‡ CC, critical concentration. For LP and QP models CC is the concentration at which the linear or quadratic portions of the models join with the predicted plateau yield.

§ Not applicable.

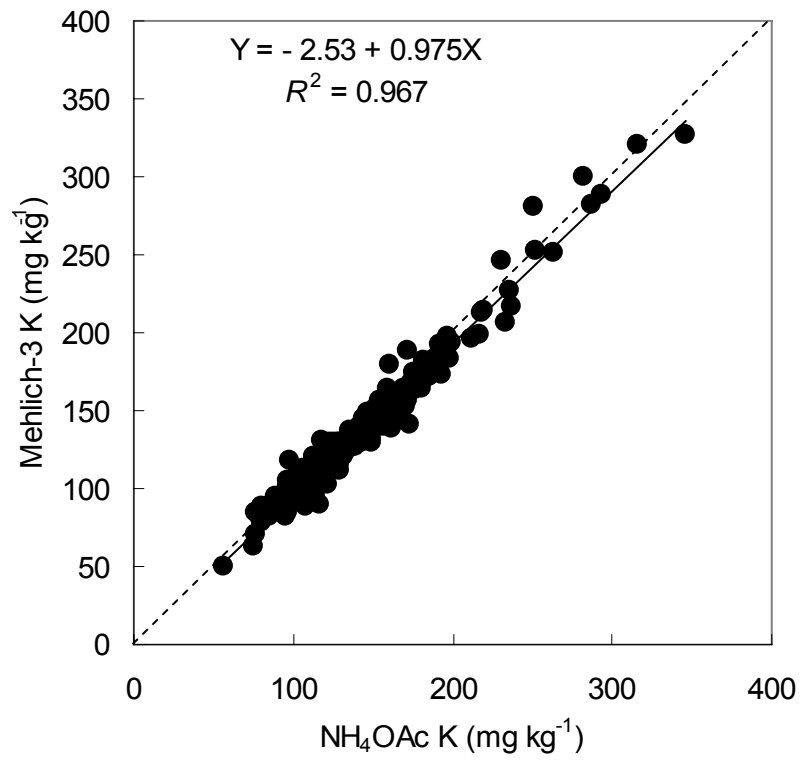


Figure 1. Correlation between soil K extracted with ammonium-acetate (NH₄OAc) and Mehlich-3 tests.

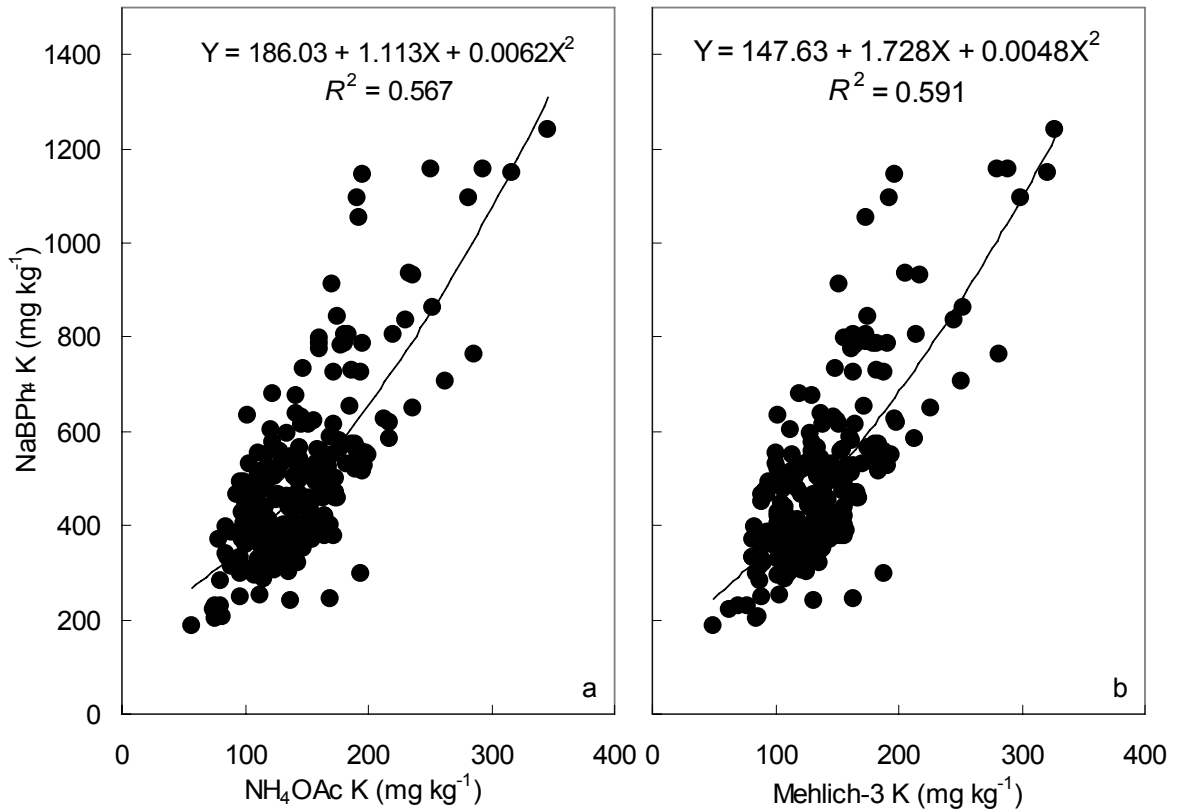


Figure 2. Correlation between soil K measured with sodium tetraphenylboron (NaBPh₄) and with the ammonium-acetate (NH₄OAc) test (a), or with the Mehlich-3 (b) test.

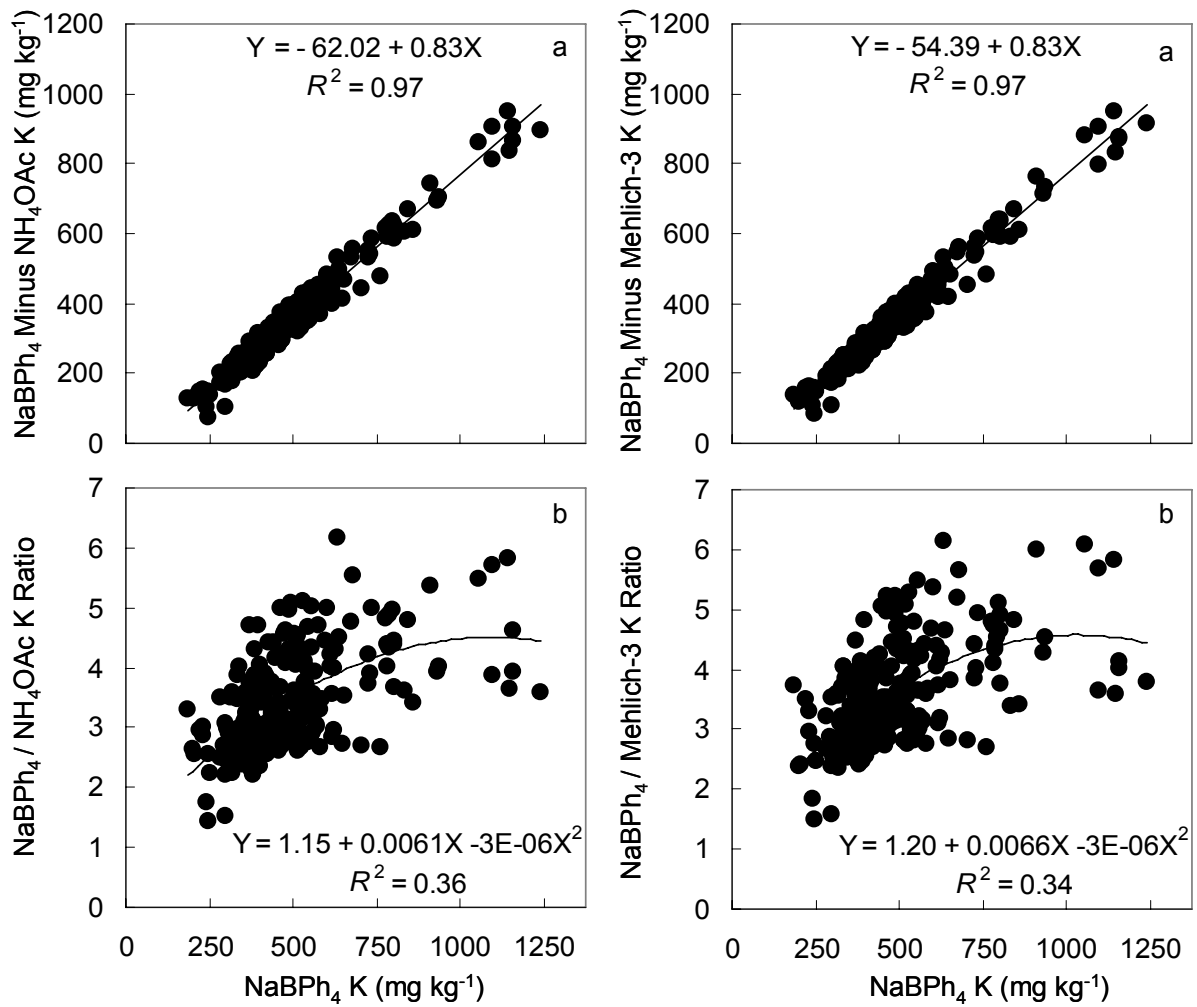


Figure 3. Relationships between K measured by sodium tetraphenylboron (NaBPh₄) and the absolute (a) or relative (b) difference with soil K measured by the ammonium-acetate (NH₄OAc) or Mehlich-3 tests.

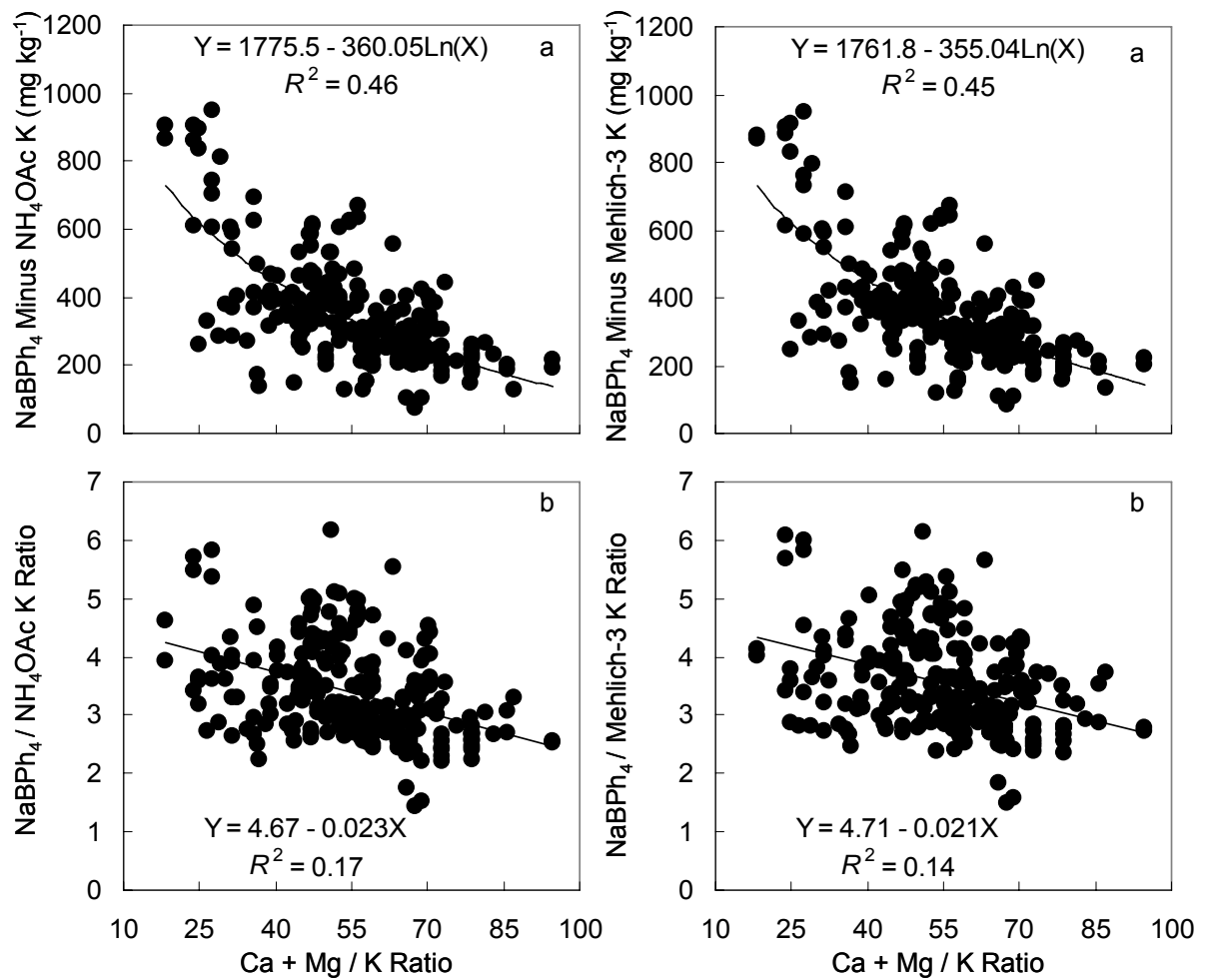


Figure 4. Relationships between Ca + Mg / K ratio and the absolute (a) or relative (b) difference in soil K measured with sodium tetraphenylboron (NaBPh₄) and ammonium-acetate (NH₄OAc) or Mehlich-3 tests.

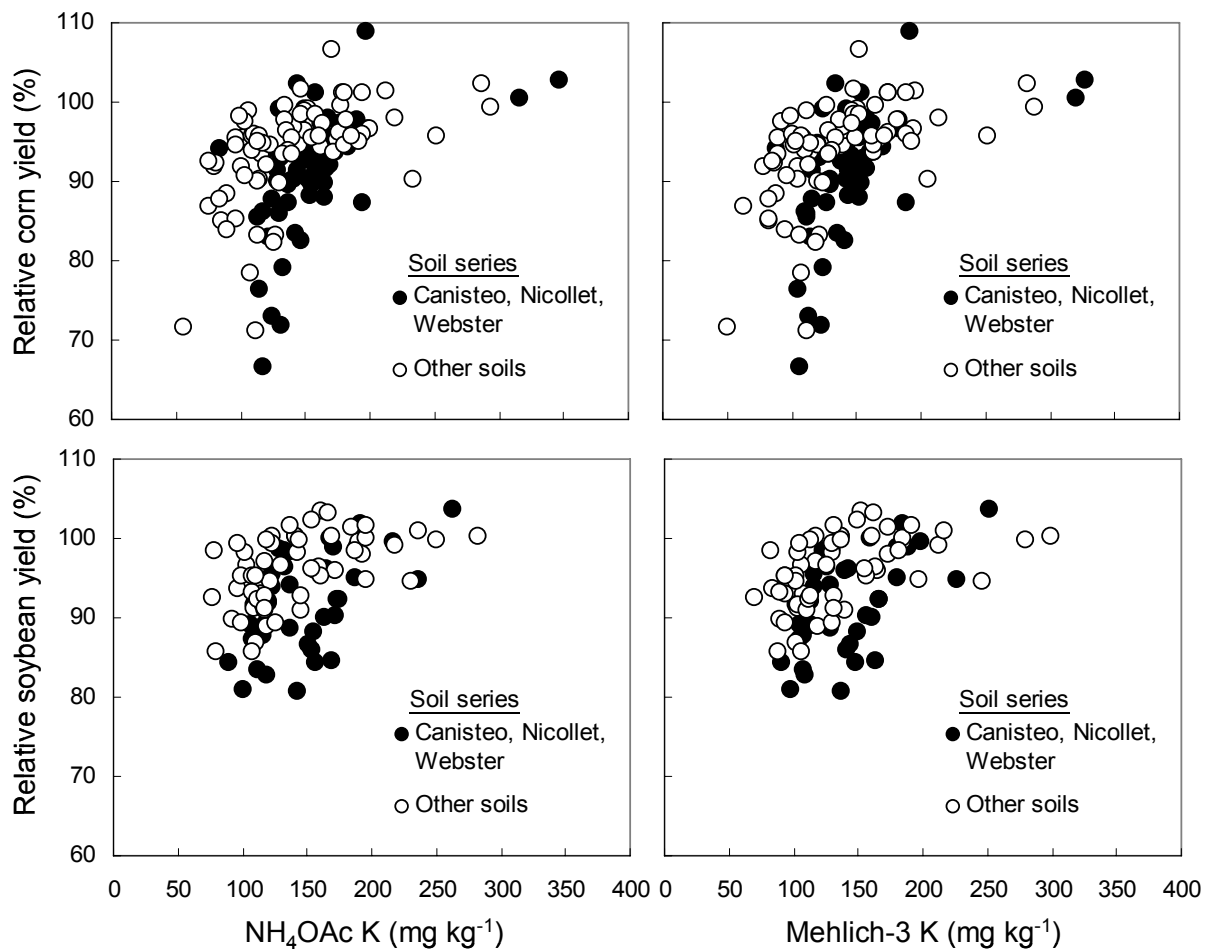


Figure 5. Relationships between relative corn and soybean yield and soil K measured with ammonium-acetate (NH₄OAc) and Mehlich-3 tests.

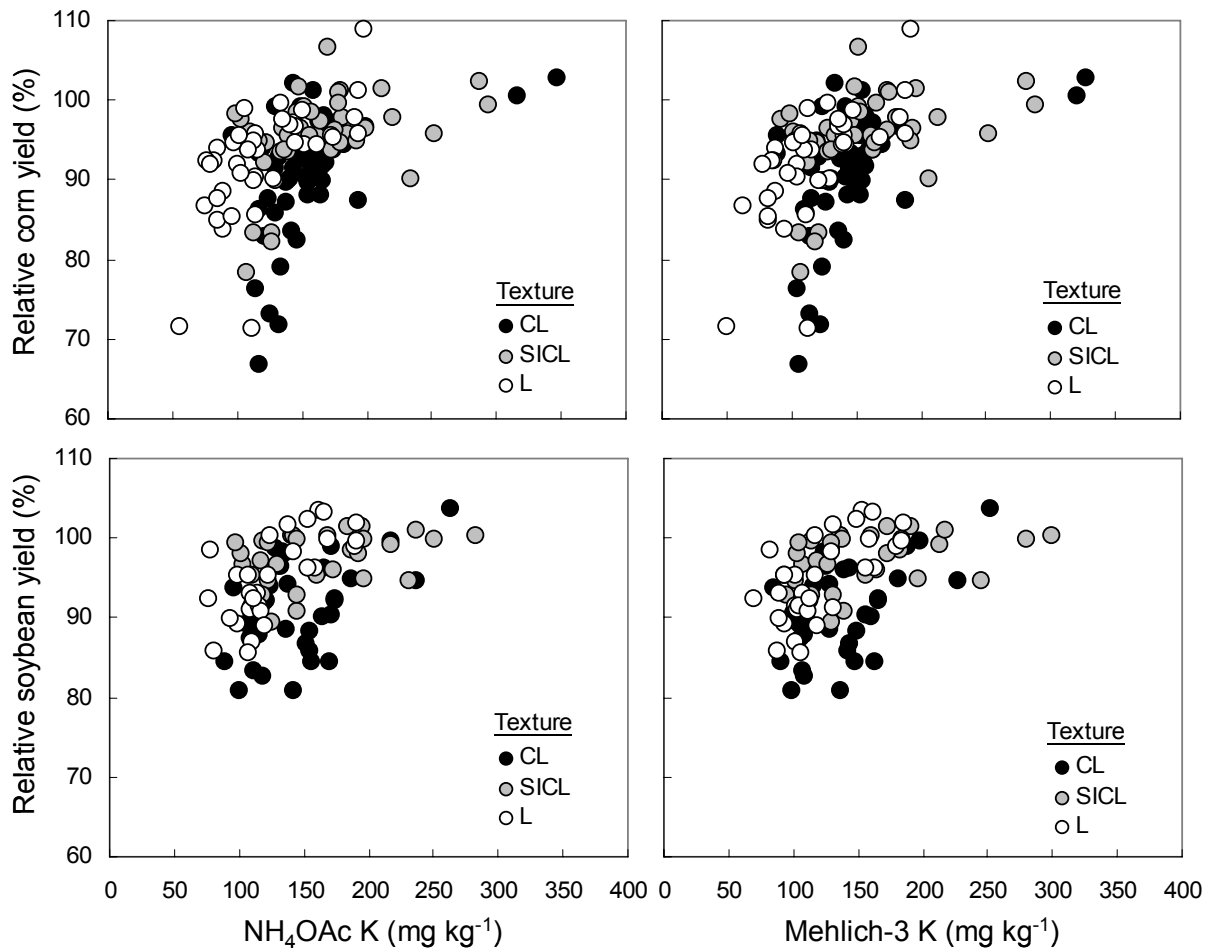


Figure 6. Relationships between relative corn and soybean yield and soil K measured with ammonium-acetate (NH₄OAc) and Mehlich-3 tests. Data-points are classified by texture of the surface horizon according to ISPAID (2004): CL = Clay-loam, SICL = silty-clay-loam, and L = loam.

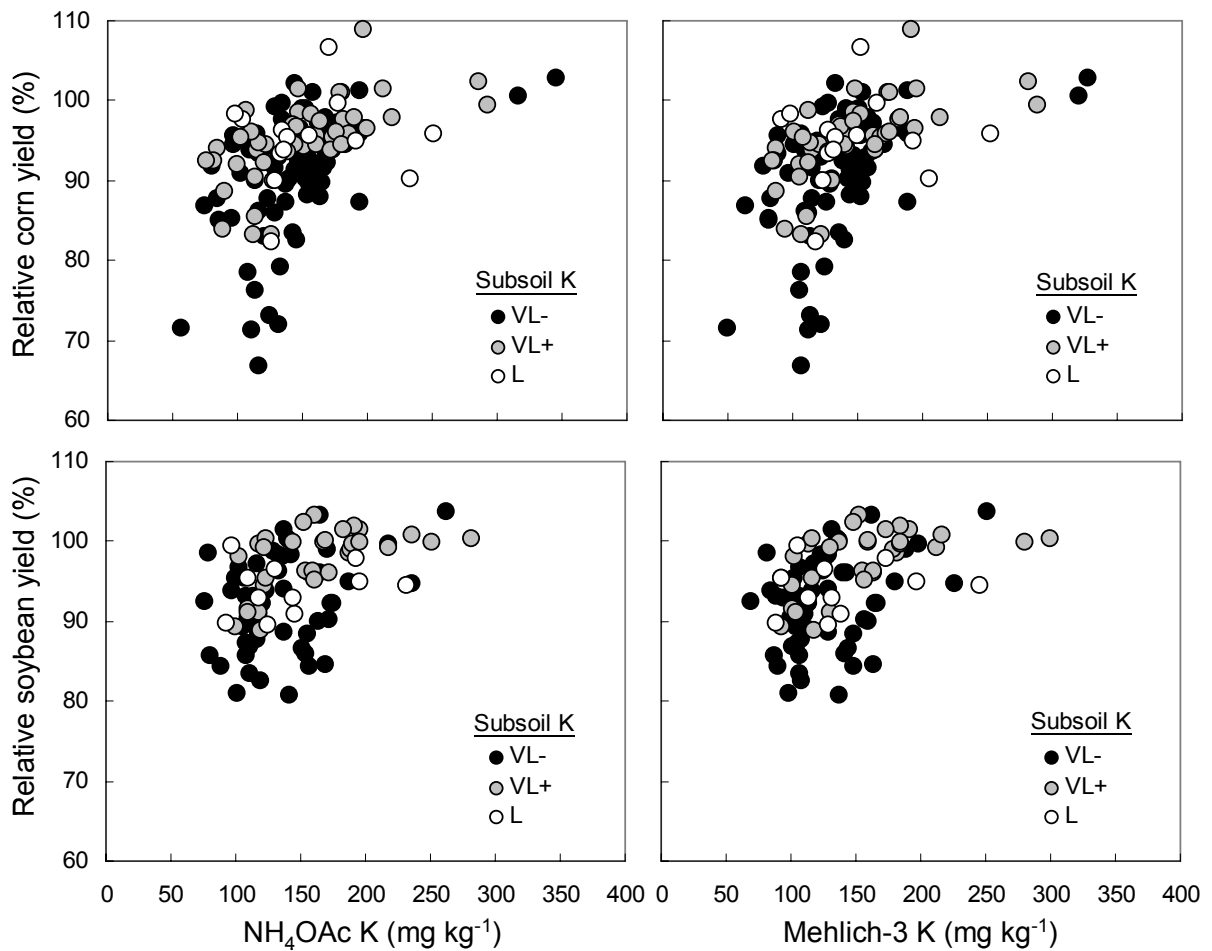


Figure 7. Relationships between relative corn and soybean yield and soil K measured with ammonium-acetate (NH₄OAc) and Mehlich-3 tests. Data-points are classified by subsoil K level according to ISPAID (2004): VL- (<25 mg K kg⁻¹), VL+ (25 to 50 mg K kg⁻¹), and L (50 to 79 mg K kg⁻¹).

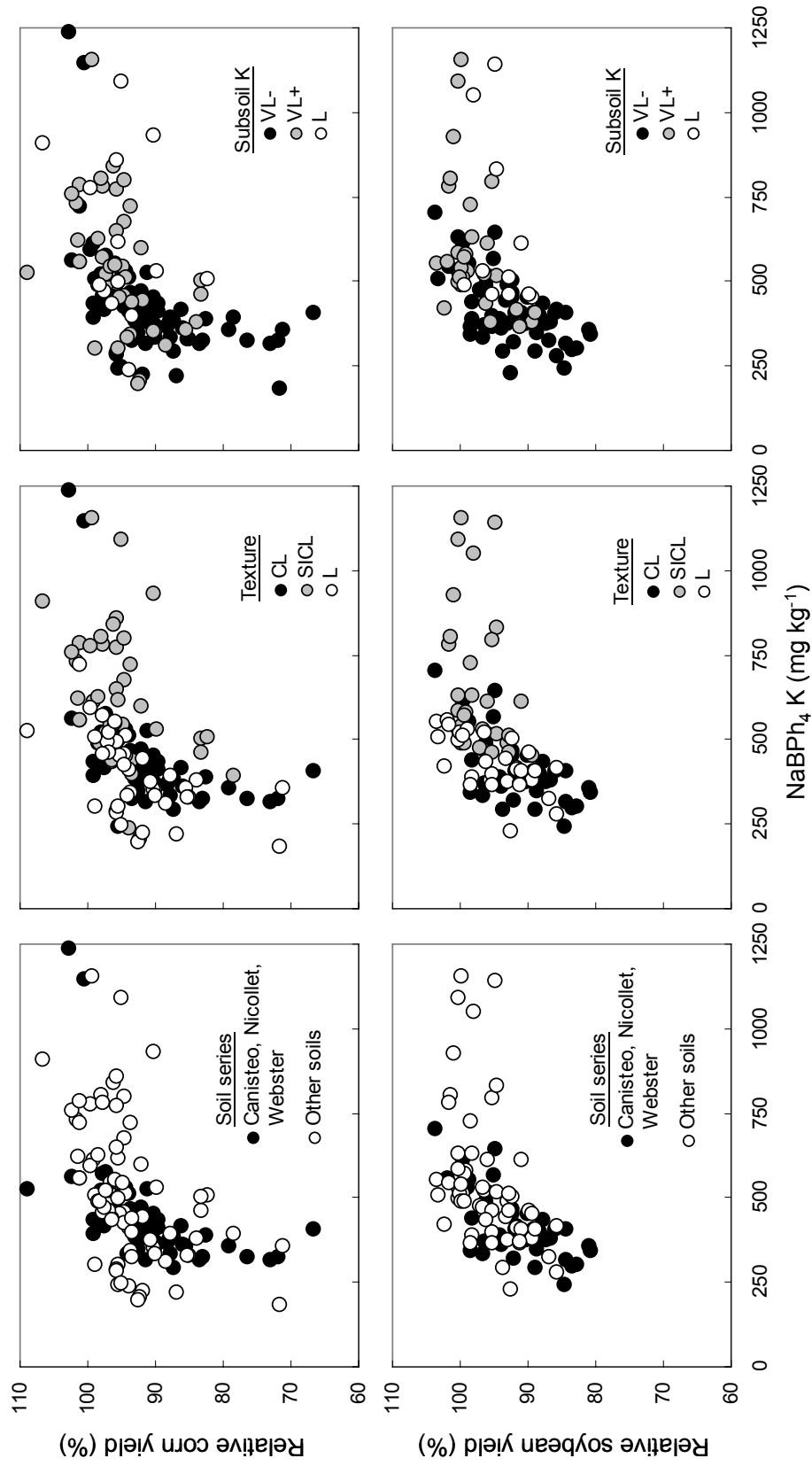


Figure 8. Relationships between relative corn and soybean yield and soil K measured with NaBPh₄. Data-points are classified by soil series, texture, or subsoil K level according to ISPAID (2004). Textures: CL = Clay-loam, SICL = silty-clay-loam, and L = loam. Subsoil K levels: VL- (<25 mg K kg⁻¹), VL+ (25 to 50 mg K kg⁻¹), and L (50 to 79 mg K kg⁻¹).

CHAPTER 4. INTEGRATING GEOSTATISTICS AND GIS TO STUDY SPATIAL VARIABILITY AND MAP SOIL FERTILITY PROPERTIES IN IOWA FIELDS

A paper to be submitted to Precision Agriculture

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ABSTRACT

One of the primary components of precision agriculture is the database that provides information needed to develop an appropriate input response to various site-specific conditions. Assessing soil fertility is a primary need to develop databases for precision agriculture. The objectives of this study were to examine and compare the spatial variability of selected soil properties, to define spatial classes of variables based on interpretation of geostatistical parameters, and to compare inverse distance weighting (IDW), ordinary kriging (OK), and universal kriging (UK) for mapping of soil P, K, and pH. Two 12 ha segments of adjacent fields located in Linn County (Iowa) that had similar soil types and long term management histories were sampled following an unaligned grid-point sampling scheme for this study. Cell size was 0.2 ha, and smaller sampling points (100 m²) were randomly selected within each cell using a geographic information system (GIS) software. There were 60 sampling positions for each field. Composite soil samples (15-20 cores, 0-15 cm depth) were collected from each sampling point. Soil variables measured included soil organic matter, soil pH, exchangeable Ca and Mg, cation exchange capacity (CEC), soil-test P and K. Spatial variability was investigated using traditional summary statistics and semivariograms. Differences in nugget, total semivariance (sill), and range for the soil variables were examined. Results suggest that the sampling density required for effective use of variable-rate technology should be different for different nutrients and fields. Median polish kriging was the best approach to obtain a reliable map of Mg (a soil property showing a clear trend)

compared to UK for trend removal evaluated using cross-validation. The UK interpolation method with a first-degree polynomial for trend removal was the least accurate of the interpolation methods used to map soil P, K, and pH compared to IDW and OK, even worse than a field average approach. Overall, the results indicated that IDW and OK are a relatively safe choice for all data sets similar to the ones examined in this study.

Abbreviations: Variable rate technology, VRT; Inverse distance weighting, IDW; Ordinary kriging, OK; Universal kriging, UK; Mean squared prediction error, MSPE; Goodness of prediction criterion, *G*.

INTRODUCTION

Historically, for the majority of agriculture fields, the predominant practice has been to apply crop production inputs as uniformly as possible and adjust rates only between fields. Today, however, a number of fields do receive spot spreading of constant rates or by using variable rate technology (VRT). With VRT, an input rate is changed within fields in response to spatially variable factors that affect the optimum application rate.

The premise of VRT is that a uniform input application within a field does not maximize input efficiency or profitability. Sawyer (1994) stated that the assumptions for VRT are that: (i) within-field spatial variation of important factors that affect crop yield exists; (ii) variation does influence crop yield; (iii) variation can be identified, measured and delineated (mapped); (iv) precise crop response models are available to determine appropriate variable input rates; and (v) data processing procedures and application equipment are available that can effectively manage and variably apply crop production inputs. Application equipment with real-time mechanisms for controlling nutrient, pesticide, seed, water, or any other crop production inputs is available. However, questions about the rest of the VRT concept remain.

The first step to implement VRT is to assess accurately and reliably spatial variability in soil fertility (Mueller et al., 2001). This is the basis for VRT and is a prerequisite for its success. Addressing soil heterogeneity is one of the oldest challenges facing farmers and agricultural researchers (Franzen and Peck, 1995). These authors also acknowledged that extensive soil sampling has long been recognized as a basis for site-specific fertilizer application. However as fertilizers became less expensive, it was simpler to treat entire fields than to complicate the process with spot application. Consequently, a soil sampling protocol was usually based on finding a central tendency for a field or field segment. The development of computer-controlled fertilizer application equipment and global positioning systems (GPS) have renewed challenges to develop efficient sampling and mapping procedures that accurately define spatial variability (Wollenhaupt et al., 1994; Franzen and Peck, 1995; Pierce et al., 1995).

Sampling density required for VRT is not clearly defined and may be different for different soil tests, fields, and regions. Sampling based on square 1-ha grids is often used in the Midwest to identify variation in soil test pH, P, and K (Sawyer, 1994). However, studies conducted to determine appropriate soil-test sampling protocols for precision agriculture and VRT have suggested that grid points should not be spaced more than 66 to 100 m (Wollenhaupt et al., 1994; Franzen and Peck, 1995; Pierce et al., 1995).

Since the adoption of geostatistics by soil scientists there has been much research into describing and quantifying the spatial variability of soil properties. One aspect of geostatistics consistently found to be useful in describing soil properties variability is the variogram (McBratney and Pringle, 1999). The variogram makes possible to optimize the sampling interval to estimate both the regional mean and local values for mapping. For estimation by kriging, or any other method of interpolation, the distances between neighboring sampling points should be well within the correlation range (Webster and

Oliver, 2001). Kerry and Oliver (2003) suggested that sampling interval should be less than half the range of the variograms to develop appropriate sampling schemes of soil properties.

The interpolation methods commonly used in agriculture include inverse distance weighting (IDW) and kriging (Franzen and Peck, 1995; Kravchenko and Bullock, 1999). Both methods estimate values at unsampled locations based on the measurements from the surrounding locations with certain weights assigned to each measurement. Inverse distance weighing is easier to implement, while kriging is more time-consuming and cumbersome. However, kriging often provides a more accurate description of the data spatial structure and produces valuable information about estimation of error distributions.

Wollenhaupt et al. (1994) compared IDW and kriging for mapping soil P and K levels and found IDW to be relatively more accurate, but concluded that the number of soil samples collected is more important than the technique used to map the data. Weber and Englung (1994) indicated that the accuracy of kriging and IDW depends on the nature of the data as reflected in variance, skewness, kurtosis, anisotropy, data clustering, and other spatial data characteristics. They suggested that for certain data sets, kriging will be better choice; but IDW may be preferred for other data sets. For mapping soil properties important for VRT, the choice between IDW or kriging methods remains unclear.

The objectives of this study were i) to examine and compare the spatial variability of several soil properties, ii) to define spatial classes of variables based on interpretation of geostatistical parameters, iii) to evaluate two methods for kriging nonstationary data (median polish kriging and universal kriging (UK)) and iv) to compare IDW, ordinary kriging (OK), and UK for mapping of soil P, K, and pH using cross-validation.

MATERIALS AND METHODS

Site Description and Soil Data

The study was conducted in two Iowa fields located in Linn County. The two fields were managed with 2-year corn-soybean crop rotations and with no-till management. The rest of the management practices were those selected by the farmer. Two field areas 12 ha in size located at least 40 m away from border areas were selected in each of the two adjacent fields (Fig. 1). Soil samples were collected in spring before soybean planting following an unaligned grid-point sampling scheme (Wollenhaupt et al., 1994). Cell size was 0.2 ha (45 by 45m), and smaller sampling points (100 m²) were randomly selected within each cell using geographic information system, ArcView GIS (Environmental Systems Research Institute, Redlands, CA) software and located in the fields with a hand-held GPS unit. There were 60 sampling positions for each field. Composite soil samples (15-20 cores, 0-15 cm depth) were collected from each sampling point.

Soil series present in the experimental areas were approximately the same for the two fields and were representative of major agricultural soils of the region (Table 1). Soil series map units for each field were identified using digitized soil survey maps on a 1:12000 scale (Iowa Cooperative Soil Survey, 2003).

Soil variables measured included: soil organic matter (SOM) by the Walkley-Black test, soil pH, exchangeable Ca and Mg, cation exchange capacity (CEC), soil-test P by Olsen test (P), and soil-test K by 1 M ammonium acetate test (K). The testing procedures used were those suggested for soils of the north-central region of the USA by the North-Central Regional Committee for Soil Testing and Plant Analysis (Brown, 1998). Soil-test P and K data were grouped into soil test categories (Table 2) as defined for field corn and soybean production on Iowa soils (Sawyer et al., 2002).

Statistical and Geostatistical Analysis

Data analysis for each of the two field areas was conducted in three stages: (i) frequency distributions were examined to check for normality; (ii) the distributions were described using traditional summary statistics such as mean, median, standard deviation (SD), and coefficient of variation (CV); and (iii) semivariograms were defined and differences in nugget and total semivariance and range examined for the variables.

Non-normal data were log-transformed to stabilize the variance prior to calculation of the semivariance. Statistical software R (Ihaka and Gentleman, 1996) and geoR (Ribeiro and Diggle, 2001) were used to analyze the spatial structure of the data and to define the semivariograms. Maximum lag distance was 230 m, which was divided in 9 to 13 lag distance classes separated by an average of 18 m. Each lag distance class contained at least 30 pairs of points for the calculation of the semivariance. Selection of theoretical models for semivariograms was made based on how well these models fit the empirical variograms using weighted least squares (WLS), where the weights were given by the number of pairs of points in each bin. Directional sample variograms were calculated in four directions (0°, 45°, 90°, and 135°) and then inspected visually. Since there was no an underlying physical phenomenon to cause the data to be anisotropic, and there was no apparent anisotropy in directional variograms, only omnidirectional variograms were used in further analysis (Goovaerts, 1997; Kravchenko and Bullock, 1999). Therefore, isotropic models for the semivariograms were fitted using WLS. The nugget variance expressed as a percentage of the total semivariance was used to classify spatial dependence. A ratio <25% indicated strong spatial dependence, between 25 and 75% indicated moderate spatial dependence, and >75% indicated weak spatial dependence (Cambardella et al., 1994; Cambardella and Karlen, 1999; Kravchenko, 2003).

The two methods evaluated for kriging nonstationary data were median polish kriging (Cressie, 1986) and UK. The soil property selected, based on the presence of trend, was Mg.

Before running median polish for trend removal we transformed the original irregular lattice (Fig. 1) into a regular one, because for this method data need to be arranged in rows and columns. The originally irregularly located values of Mg within each grid-cell were assigned to the center of the grid. For universal kriging we used a first-degree polynomial to remove the trend.

For both IDW and kriging interpolation methods, the value of variable Z at unsampled locations x_0 , $Z^*(x_0)$ is estimated based on the data from the surrounding locations, $Z(x_i)$, as:

$$Z^*(x_0) = \sum^n w_i Z(x_i) \quad [\text{Eq. 1}]$$

where w_i are the weights assigned to each $Z(x_i)$ value and n is the number of the closest neighboring sampled data points used for estimation. The weights for IDW method are:

$$W_i = 1/d_i^p / \sum^n 1/d_i^p \quad [\text{Eq. 2}]$$

where d_i is the distance between the location x_0 and the sample point x_i , and p is an exponent parameter. It has been shown that the choice of the exponent value can significantly affect the estimation quality (Gotway et al., 1996). In our study we used the option ‘‘Optimize Power Value’’ within Geostatistical Analyst extension of the ArcGIS software (ESRI, Redlands, CA). The optimal power value is determined by minimizing the root mean squared prediction error (MSPE) calculated from cross-validation.

The other factor affecting the precision of IDW and kriging is the number of the nearest samples used for estimation. In this study, we decided to use the six nearest samples within a circular search neighborhood after evaluating results with varying number of neighboring samples (from 4 to 12). For UK, a first-order polynomial was chosen to remove trend based on minimization of MSPE criterion.

The two criteria used to check and compare map accuracy were cross-validation MSPE and goodness of prediction criterion G (Gotway et al., 1996). Mean squared prediction error was calculated as:

$$\sigma_{cv}^2 = 1/n \sum d_i^2, \quad [\text{Eq. 3}]$$

where d_i = predicted - observed value at s_i

The G criterion was calculated as:

$$G = (1 - \text{MSPE}/\text{MSPE}_{\text{average}}) \times 100\% \quad [\text{Eq. 4}]$$

where $\text{MSPE}_{\text{average}}$ is the mean squared error obtained from using field average values as estimates for all test data. Positive G values indicate that the map obtained by interpolating data from grid samples is more accurate than a field average. Negative and close to zero G values indicate that the field average predicts the values at unsampled locations as accurately as or even better than grid sampling estimates (Kravchenko, 2003).

RESULTS AND DISCUSSION

Spatial variability of soil properties

Three of the five soil properties measured in each field (P, K, and SOM) were highly skewed and non-normally distributed. Log transformations normalized the properties and reduced skewness. Frequency plots of K and P from both fields before (Fig. 2a and 3a) and after (Fig. 2b and 3b) log-transformation showed reduced skewness.

The mean and median were used as the primary estimates of central tendency, and the SD and CV were used to describe the variability (Table 3). Despite the skewness of the distributions, the mean and median values of the soil properties analyzed were similar, with the medians having slightly smaller values than the means. This indicates that the measures of central tendency are not dominated by the outliers in the distributions. A similarity of means and medians for different soil properties was also reported by Cambardella et al. (1994). Mean and median values of SOM and K were more than 44% higher at North field

compared with South field. Mean and median values of the rest of the properties analyzed were similar for the two field sites. The ranking of the variability for the five soil properties evaluated was similar in the two fields (Table 3). Soil-test P showed the highest variability and soil pH the lowest (average CV 35 and 3.5%, respectively). In general, variability for all soil properties, except for CEC, was more than 19% higher at South field.

Spherical models were defined for most soil variables measured at both sites (Tables 4 and 5 and Fig. 4). In a few instances where differences in WLS were small between spherical and alternative models (exponential, Gaussian, and others), the spherical model was used to allow direct comparison of the nugget, sill, and range values among different soil properties. In these cases, the alternative models generated estimates of nugget and total semivariance that were similar to the spherical model.

Semivariograms indicated strong spatial dependence for SOM, K, and CEC for South field (Table 5). It has been hypothesized that strong spatial dependency for these soil properties may be controlled by intrinsic variations in soil characteristics, such as texture and mineralogy (Cambardella et al., 1994). The stronger the spatial correlation, the more accurate the soil property map that could be obtained using kriging (Kravchenko, 2003). Soil properties with strong and medium spatial structure, regardless of the overall variability, can be mapped relatively accurately even with a small number of sample locations. To achieve accurate maps of soil properties with weak spatial structures, very intensive sampling schemes should be performed (Kravchenko, 2003).

Total semivariance estimates (sill) for soil properties at North field were generally lower than those at South field (Tables 4 and 5), with the exception of CEC. Variability assessed by parametric statistics was also higher for South field properties compared with North field properties (Table 3), as discussed before. Extrinsic management effects probably contributed to the observed differences in total variability. The range values, which indicate the limit of spatial dependence, showed considerable variability among the properties

measured in each field (Tables 4 and 5). Soil organic matter and CEC showed similar range values in North and South fields. However P and K range values varied considerably between fields, being 72 and 80 m and 120 and 150 m for P and K at North and South fields, respectively.

McBratney and Pringle (1999) stated that average or proportional variograms developed based on published experimental variograms are useful to provide approximate estimates of spatial structure for different soil properties. The accuracy of this approach would be limited since our results showed that spatial structure and correlation range of soil properties varied widely from field to field, and no generalization should be made. Large variability in soil fertility properties among fields in Iowa was reported before (Mallarino, 1996; Mallarino and Wittry, 2004). Today, most commercial soil sampling for VRT is conducted on a 1-ha grid basis. However researchers have proposed that soil sampling on 60-m grids (Hammond, 1993; Wollenhaupt et al., 1994) might be needed for developing soil property maps of acceptable accuracy. Sampling interval used in our study was based on 45-m grids and we found large differences in spatial structure and correlation ranges in soil fertility properties evaluated between two fields with similar characteristics such as management practices, slopes, and soils. This suggests that sampling density required for effective use of variable rate technology should be different for different nutrients and fields.

Comparison of methods for Kriging nonstationary data

For kriging nonstationary data, median polish and UK, with a first-degree polynomial, were compared as plausible methods for trend removal. Directional variograms after removing the trend with both methods showed a considerable improvement compared to the directional variograms for the original data (Fig. 5).

We ran cross-validation using the leave-one-out strategy to assess map quality and compare between the two methods. In this strategy data points are removed one by one and predicted by kriging using the remaining data. Figure 6 shows the measured versus the

predicted Mg content by cross-validation for median polish kriging and UK and the MSPE and G values for each method. These plots show considerable deviation from the 1:1 line and low G values ($\leq 30\%$). A comparison of the two methods indicates that median polish kriging did a better job than UK in reducing the mean squared prediction error for this variable (MSPE 27% lower for median polish compared with UK).

Comparison of methods for mapping soil properties

The comparison criteria (MSPE and G) described before were calculated for North and South fields, three soil fertility properties (P, K, and pH), and three interpolation methods (IDW, OK, and UK) (Table 6). These results indicate that the choice of the most accurate interpolation and mapping approach from those considered in this study depends on the soil property being mapped and the specific field where the property is mapped.

In order to use OK one important condition must be met. This is the intrinsic hypothesis also referred as stationarity, which means constant mean and variogram as function of displacement. Cressie (1986) stated that it is highly unrealistic to assume constant mean and that data almost never exhibit stationarity. Therefore this is one of the most often violated assumptions. Mulla and McBratney (2000) affirmed that nonstationarity can result from three types of problems: a long-range systematic change in the mean with location (trend), a short-range stochastic change in the mean with location (drift), and a change in variance with location. They also stated that UK can be used to remove these problems. In our study the UK interpolation method with a first-degree polynomial for trend removal was, by far, the least accurate of the interpolation methods analyzed, even worse than the field average approach. Predictors with a negative G value should not be used since it indicates that using the sample mean (which is much easier to compute) is more accurate than the tested predictor (Gotway et al., 1996). The poor performance of UK found here can be explained for the lack of trend in the data sets studied; or for the robustness of the OK method to the type of data, if some kind of trend was in fact present.

For the three soil fertility properties studied, the accuracy of IDW and OK was almost the same. For P, a soil property with very high variability, the accuracy of both methods was poor compared with the field average approach, and OK was slightly better than IDW. Prediction maps for P from North and South fields and the interpolation methods evaluated are presented in Fig. 7. Inverse distance weighing and OK maps appear smoother than the maps produced by UK. For soil test K, with intermediate variability between P and pH, the accuracy of IDW and OK was relatively good, with IDW slightly better than OK. For the least variable soil property analyzed, pH, the accuracy was also good, especially in the South field, and IDW was moderately more accurate than OK in North field and was the opposite in South field.

CONCLUSIONS

Geostatistical techniques offer alternative methods to conventional statistics to study the spatial distribution of soil properties, their associated variability, and interpolation of data. Semivariance analysis demonstrated that there were some similarities in the patterns of spatial variability for SOM and CEC but not much for the other soil properties studied. This may suggest that spatial relationships derived from one set of measurements for one field may not have applicability at other field sites within the same region with similar soil types and landscapes. The results obtained here may confirm what have been suggested previously in the literature that the spatial structure of soil test variability is site and nutrient specific and no generalizations can be made.

The accuracy of spatial maps depends on many factors such as sample density, sample configuration, and interpolation-prediction method. Median polish kriging reduced the mean squared prediction error by 27%, compared with UK, indicating that median polish kriging was the best approach to obtain a reliable map of Mg, a soil property showing a clear trend.

The results of the comparison of the interpolation methods for P, K, and pH showed that map accuracy attained with UK method was noticeably inferior compared to IDW and OK. Overall, the results indicated that IDW and OK are a relatively safe choice for all data sets similar to the ones tested here. Inverse distance weighting is a fast (in terms of computation), exact, deterministic interpolator with few parameters to set and no assumptions are made about the data. The main disadvantages of this method is that it tends to produce unrealistic “bull’s-eyes” patterns around the observed data points, and it does not assess prediction error. The major disadvantages of kriging is the amount of modeling decisions that need to be made (transformations, trends, models parameters, and neighborhoods) and the assumption that the data are normally distributed and come from a stationary process. On the other hand kriging allows a measure of error in each predicted (interpolated) value.

All maps constructed from interpolation are subject to some degree of uncertainty. Uncertainty in the maps of the soil properties will translate into uncertainty in the amount of fertilizer recommended at any particular field. Measuring this uncertainty and incorporating it into the recommendations for a field may be important for developing efficient soil sampling methods and effective use of VRT.

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Table 1. Soil series present and percentage area occupied in the experimental areas.

South field			North field		
Series	Subgroup†	Area	Series	Subgroup†	Area
		%			%
Kenyon	T. Hapludolls	36.7	Clyde-Floyd	T. Endoaquolls	31.7
Klinger	A. Argiudolls	32.5	Kenyon	T. Hapludolls	25.0
Dinsdale	T. Argiudolls	26.5	Dinsdale	T. Argiudolls	25.0
Donnan	Aq. Hapludalfs	3.3	Klinger	A. Argiudolls	15.8
Aredale	T. Hapludolls	1.0	Aredale	T. Hapludolls	2.5

† A., Aguic; Aq., Aquollic; T., Typic.

Table 2. Phosphorus and potassium soil test categories for corn and soybean production in Iowa soils.

Nutrient (method)	Soil test category [†]				
	Very Low	Low	Optimum	High	Very High
	-----mg kg ⁻¹ -----				
Phosphorus (Olsen)	0-5	6-10	11-14	15-20	>21
Potassium (Amonium acetate)	0-90	91-130	131-170	171-200	>201

[†] From Sawyer et al., (2002).

Table 3. Descriptive statistics for soil properties for North and South fields.

Soil property†	North Field				South Field			
	Mean	Median	SD	CV‡	Mean	Median	SD	CV‡
SOM (g kg ⁻¹)	46.5	45.5	10.0	21.5	32.1	30.6	8.4	26.1
pH	6.8	6.8	0.2	2.8	6.8	6.7	0.3	4.2
P (mg kg ⁻¹)	19.8	18.9	6.4	32.4	19.0	18.0	7.3	38.3
K (mg kg ⁻¹)	164.7	159.3	22.0	13.3	113.9	114.0	18.1	15.9
CEC (cmol kg ⁻¹)	11.0	10.5	2.1	19.4	10.3	10.0	1.9	18.5

† SOM = soil organic mater; CEC = cation exchange capacity.

‡ SD = standard deviation, CV = coefficient of variation (%).

Table 4. Parameters for semivariogram models for North field.

Soil property [†]	Model [‡]	Nugget	Sill	Range	N/S ratio [§]	Spatial [¶] Class
SOM (g kg ⁻¹)	Spherical	0.013	0.048	160.0	27.8	M
pH	Spherical	0.026	0.044	150.0	60.2	M
P (mg kg ⁻¹)	Spherical	0.050	0.093	72.4	54.0	M
K (mg kg ⁻¹)	Spherical	0.005	0.014	80.2	35.0	M
CEC (cmol kg ⁻¹)	Spherical	1.90	4.38	115.4	43.4	M

[†] SOM = soil organic mater; CEC = cation exchange capacity.

[‡] Models are all isotropic.

[§] N/S ratio = (nugget semivariance/total semivariance) * 100

[¶] Spatial Class: S = strong spatial dependence (N/S ratio<25); M = moderate spatial dependence (N/S ratio between 25 and 75); W = Weak spatial dependence (N/S ratio>75).

Table 5. Parameters for semivariogram models for South field.

Soil property [†]	Model [‡]	Nugget	Sill	Range	N/S ratio [§]	Spatial [¶] Class
SOM (g kg ⁻¹)	Spherical	0.000	0.066	168.4	0.0	S
pH	Linear	0.037	>.095	>230.0	<38.0	M
P (mg kg ⁻¹)	Spherical	0.050	0.148	119.6	33.8	M
K (mg kg ⁻¹)	Spherical	0.005	0.023	150.0	22.2	S
CEC (cmol kg ⁻¹)	Spherical	0.400	3.872	129.6	10.3	S

[†] SOM = soil organic mater; CEC = cation exchange capacity.

[‡] Models are all isotropic.

[§] N/S ratio = (nugget semivariance/total semivariance) * 100

[¶] Spatial Class: S = strong spatial dependence (N/S ratio<25); M = moderate spatial dependence (N/S ratio between 25 and 75); W = Weak spatial dependence (N/S ratio>75).

Table 6. MSPE for IDW, OK, and UK interpolation methods for soil test P, K, and pH in North and South fields.

Interp. Methods [†]	Measure [‡]	P		K		pH	
		North	South	North	South	North	South
IDW	MSPE	6.4	7.5	19.3	17.1	0.176	0.227
	G	0.3%	-3.5%	12.3%	5.6%	8.7%	20.3%
OK	MSPE	6.4	7.5	19.5	17.4	0.179	0.224
	G	0.5%	-3.3%	11.4%	4.2%	7.2%	21.3%
UK	MSPE	7.7	8.8	22.0	19.5	0.219	0.298
	G	-20.4%	-20.6%	0.0%	-7.5%	-13.3%	-4.8%

[†] Interpolation methods, IDW= Inverse distance weighting, OK= Ordinary Kriging, UK= Universal kriging.

[‡] MSPE= Mean squared prediction error, G= goodness-of-prediction measure.

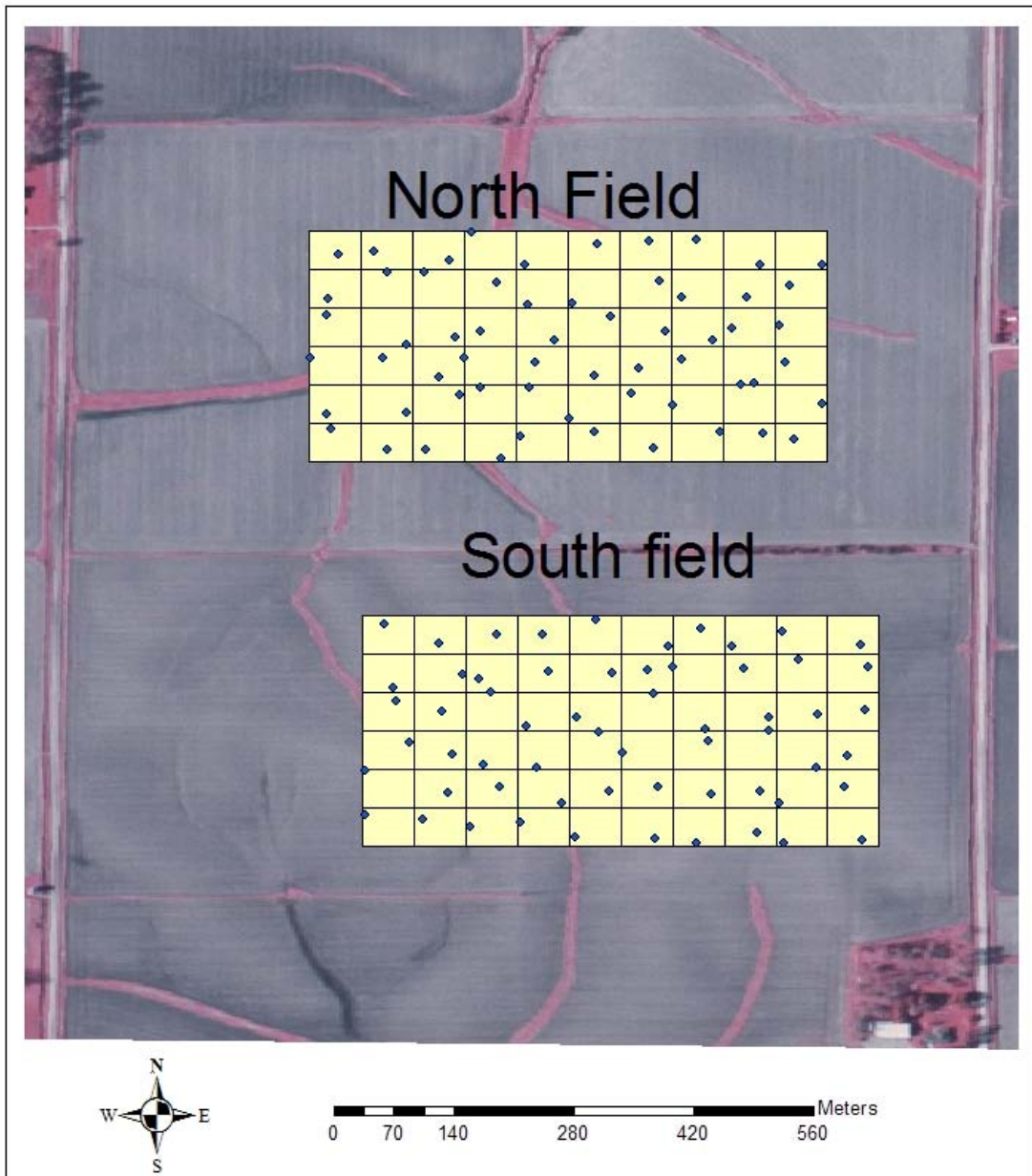


Figure 1. North and South field with the layout of 45m grid cells and location where soil samples were collected (total of 60 samples in each field) on an infrared aerial photograph (downloaded from <http://ortho.gis.iastate.edu/>).

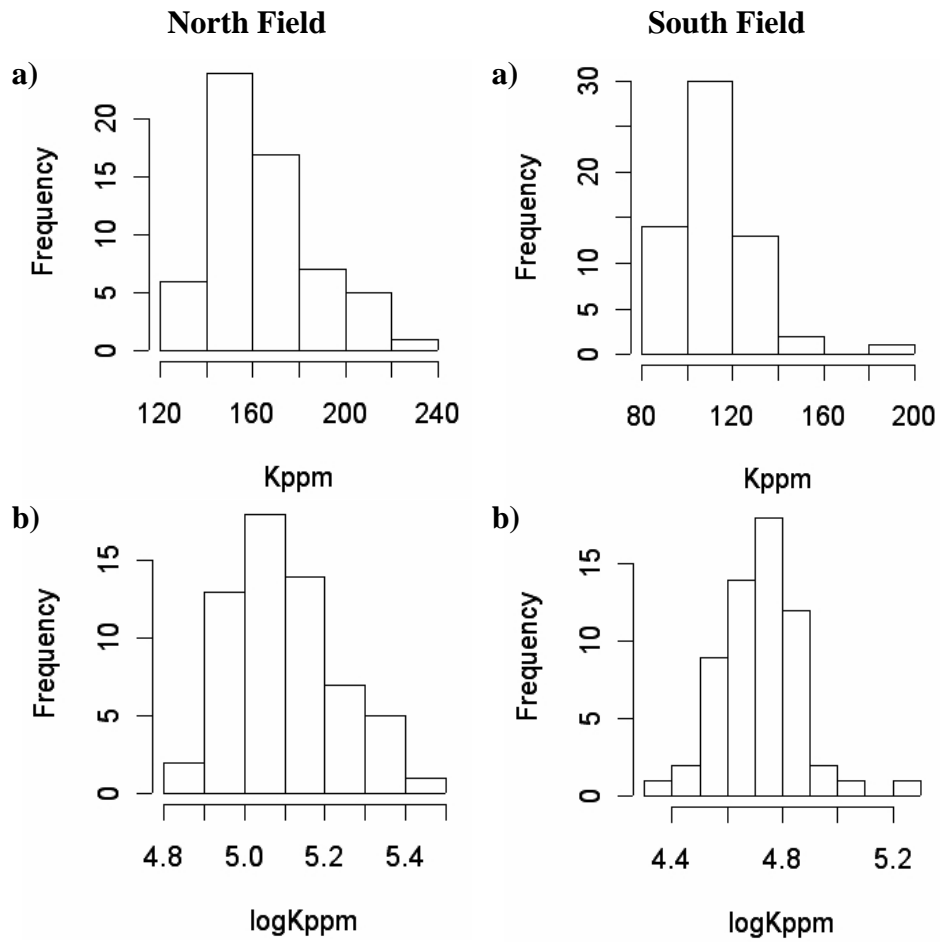


Figure 2. Frequency distribution of (a) K and (b) log-transformed K for North and South field.

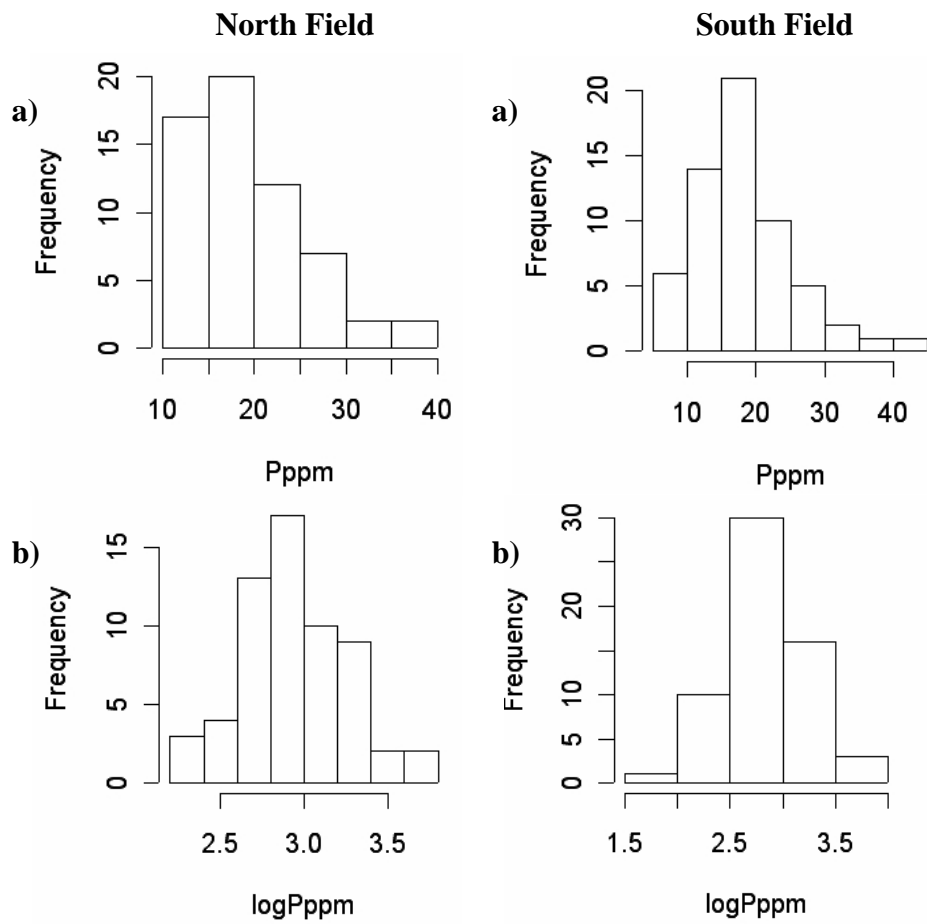
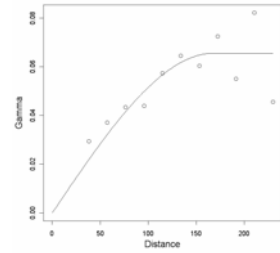
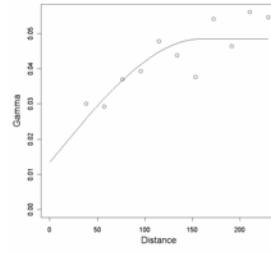


Figure 3. Frequency distribution of (a) P and (b) log-transformed P at North and South fields.

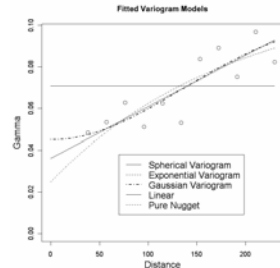
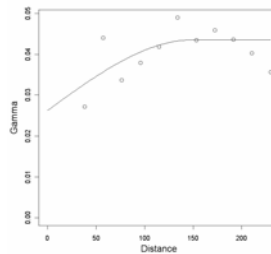
North field

South field

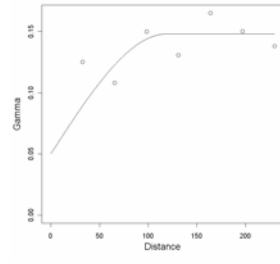
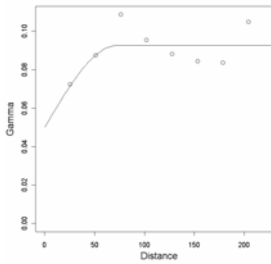
SOM



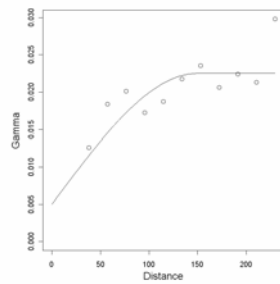
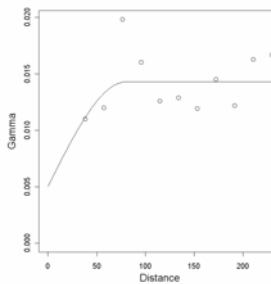
pH



P



K



CEC

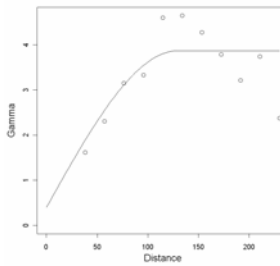
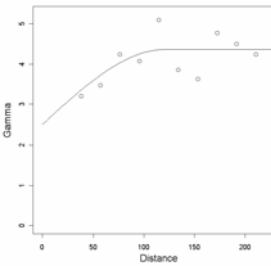


Figure 4. Omnidirectional empirical semivariograms and fitted spherical semivariograms models for SOM, pH, P, K, and CEC at the two fields.

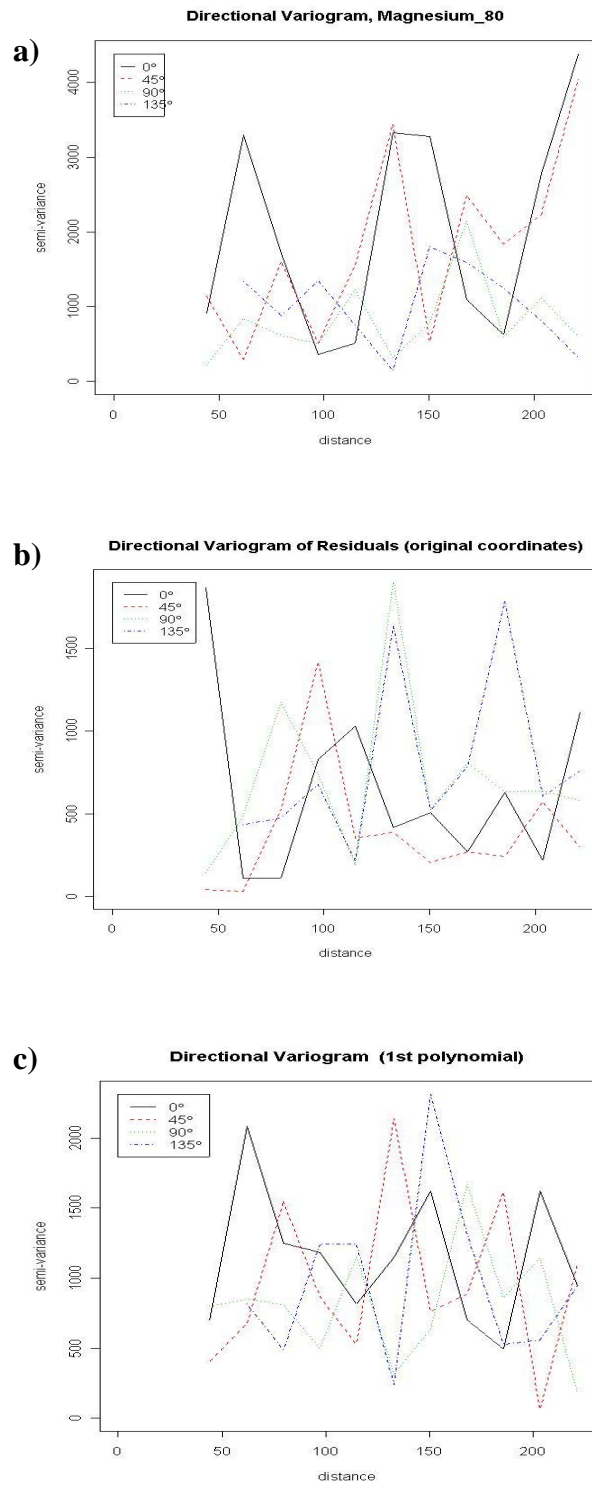


Figure 5. Directional variograms for a) the original data, b) after removing the trend with median polish, and c) after removing trend with a first-degree polynomial.

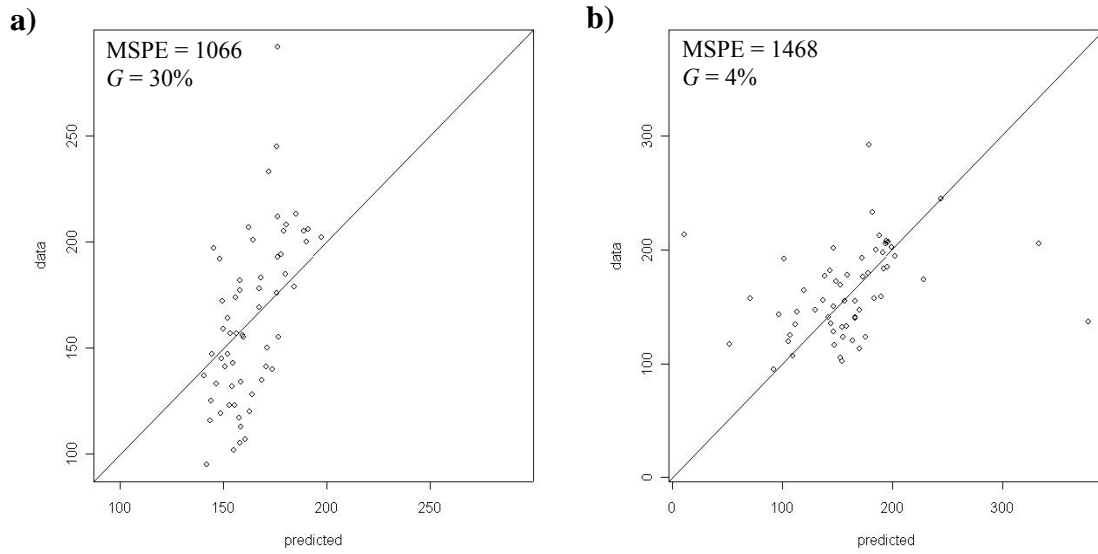


Figure 6. Predicted vs. measured Mg by cross-validation with: a) Median Polish, and b) Universal kriging. MSPE= Mean square prediction error; G = Goodness of prediction.

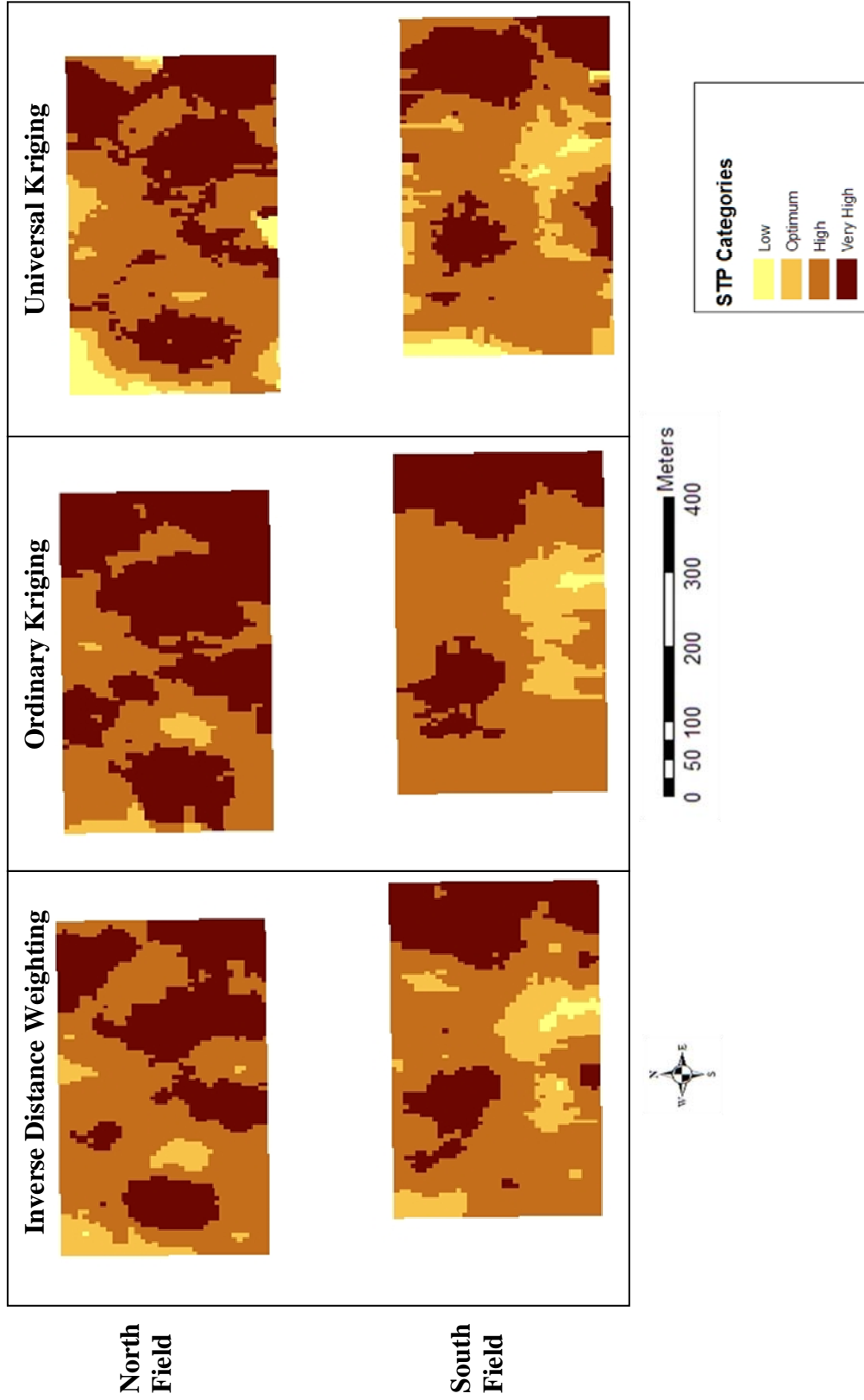


Figure 7. Soil test Phosphorus maps from North and South fields constructed using IDW, OK, and UK interpolators.

CHAPTER 5. GENERAL CONCLUSIONS

The overall goal of this research was to collect information needed to improve the use of soil testing in production agriculture. The field and laboratory work as well as interpretation and summarization of results were developed to focus into three main areas of study. Specific objectives of one study were to assess the impact of sample drying on soil K extraction from several Iowa soil series and to conduct field correlations for corn and soybean of an ammonium-acetate K test based on field-moist soil samples (MK). Data from K response trials involving 64 site-years with corn and 57 site-years with soybean conducted from 2001 to 2004 were used. The objective of the second study was to compare the efficacy of ammonium-acetate, Mehlich-3, and sodium tetraphenylboron soil K extractants based on the commonly used sample drying procedure in determining plant-availability of K by conducting field correlation and calibration studies with corn and soybean across several Iowa soils. Data from K response trials involving 63 site-years with corn and 54 site-years with soybean conducted from 1999 to 2004 were used. The objectives of the third study were (1) to examine and compare the spatial variability of various soil fertility properties, (2) to define spatial classes of variables based on interpretation of geostatistical parameters, and (3) to compare inverse distance weighting, ordinary kriging, and universal kriging for mapping of soil fertility properties. Soil-test data from samples collected using a dense grid-sampling method were used.

Results from the first study showed that K extracted with the commonly used ammonium-acetate test from dried soil samples was greater than K extracted from field-moist samples, and the difference increased with higher drying temperature (from air dried to 50 °C). However, no single factor can be used to relate these tests because the difference was inversely related to the soil K level and the sample drying effect varied greatly between soil series. The moist-based K test correlated better with yield response to fertilization and

showed a better defined critical K concentration compared with the commonly used test based on dried soil samples. Results showed that different calibrations are needed for different soils and (or) growing conditions for the dry-based test but not for the field-moist based test. Critical concentration ranges defined by Cate-Nelson and linear-plateau models across all soils for corn were (15 cm sampling depth) 144 to 201 mg K kg⁻¹ for the dry test and 62 to 76 mg K kg⁻¹ for the moist test, while critical concentrations ranges for soybean were 121 to 214 mg K kg⁻¹ for the dry test and 52 to 90 mg K kg⁻¹ for the moist test. It was concluded that an ammonium-acetate K test based on field-moist samples predicts crop response to K fertilizer significantly better than the commonly used test based on dried samples, and the degree of the improvement may justify more laborious laboratory procedures for the field-moist test.

Results from the second study showed that the amounts of soil K extracted between Mehlich-3 and the ammonium-acetate tests were similar and very highly correlated, indicating that these methods measure the same soil pool of soil K. The amount of sodium tetraphenylboron-extractable K was significantly higher than amounts measured by the ammonium-acetate or Mehlich-3 tests and the difference increased with increasing soil K. The additional K extracted by the sodium tetraphenylboron test decreased as the Ca and Mg to K ratio increased, but the relationship was poor. Ammonium-acetate and Mehlich-3 tests showed similarly poor capacity to predict corn and soybean response to K fertilization. The sodium tetraphenylboron test did not show a consistently superior capacity to predict corn and soybean response to K. Critical soil-test K concentration ranges defined across all soils for the ammonium-acetate test were 133 to 216 mg K kg⁻¹ for corn and 122 to 191 mg K kg⁻¹ for soybean. Critical soil-test K concentration ranges for the Mehlich-3 test were 128 to 199 mg K kg⁻¹ for corn and 114 to 185 mg K kg⁻¹ for soybean. These ranges encompass the current Optimum interpretation class used in Iowa for these tests. Critical concentration ranges for the sodium tetraphenylboron test were 421 to 641 mg K kg⁻¹ for corn and 473 to

556 mg K kg⁻¹ for soybean. It was concluded that the results do not support adoption of the sodium tetraphenylboron K test in production agriculture because the correlation with crop response is not consistently better than for currently used tests and laboratory procedures are much more laborious and expensive.

Results of the third study showed that the amount of within-field soil-test variability and its spatial structure vary greatly across different nutrients and fields. Median polish kriging was the best approach to obtain a reliable map of a soil property showing a clear trend (Mg) compared to Universal Kriging for trend removal evaluated using cross-validation. The Universal Kriging interpolation method with a first-degree polynomial for trend removal was the least accurate of the interpolation methods used to map soil P, K, and pH compared to Inverse Distance Weighing and Ordinary Kriging, even worse than a field average approach. In general, the results indicated that Inverse Distance Weighing and Ordinary Kriging are relatively safe choices for data sets similar to the ones examined in this study, and that the sampling density required for effective use of variable-rate technology likely is different across fields.

Overall, the results showed that further research is needed to better understand the complex equilibrium among the various forms of soil K, to find soil-test K methods that improve the prediction of plant-available K, and that the soil sampling density required to appropriately describe within-field soil-test variation is limited by economic considerations and will vary for different nutrients, fields, and intended uses.

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