RECALIBRATION OF SOIL POTASSIUM TEST FOR CORN IN NORTH DAKOTA

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ABSTRACT

Corn (*Zea mays* L.) production has greatly increased in North Dakota since the 1990s; yet, potassium (K) fertilizer recommendations for corn in North Dakota still date from the late 1970s and early 1980s when corn grain yields were comparatively lower and native soil K fertility was sufficient for most crop K requirements. To update K fertilizer recommendations for modern corn production and lower soil K levels, corn grain yield response to K fertilization, various soil K testing methods, and seasonal soil K variation were investigated in a two-year (2015 and 2016) study. The standard soil K testing method of NH₄OAC extraction on dried soil remained the best predictor of yield response, although corn on some soils did not respond in accordance with soil test K level. Soil test K was highest in spring and lowest in late summer, indicating that soil test K interpretation should account for seasonal variation.

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DEDICATION

To my paternal grandparents, Melvin and Lois Breker, who trained my novice hands in soil and

nurtured a budding love for plants.

ABSTRACT	iii
ACKNOWLEDGEMENTS	iv
DEDICATION	v
LIST OF TABLES	ix
LIST OF FIGURES	xi
LIST OF ABBREVIATIONS	xiii
LIST OF APPENDIX TABLES	xiv
GENERAL INTRODUCTION	
LITERATURE REVIEW	
Corn	
Corn Origin and Crop	
Global and U.S. Corn Production	7
North Dakota Corn Production	
Corn Fertility	9
General Corn Fertility	
Potassium in Corn Production	
Soil Potassium	
Potassium Forms in Soil	
Potassium-bearing Minerals	
Soil Clay Minerals	
Potassium Mineralogy of Northern Plains Soils	
Soil Potassium Dynamics	
Ion Selectivity	
Potassium Fixation and Release	

TABLE OF CONTENTS

Plant Availability of Nonexchangeable Potassium	
Long-term Effects of Cropping on Potassium Mineralogy	
Effect of Soil Water Content and Sample Drying on Exchangeable Potassium	ı 35
Temporal Variation in Exchangeable Potassium	
Potassium Fertilizer Application	
Potassium Fertilizer Sources	
Potassium Fertilizer Placement	40
Potassium Fertility Recommendations	
Soil Testing: Calibration and Interpretation	
Soil Testing: Soil Potassium Testing Methods	
Soil Testing: Sample Handling	
RECALIBRATION OF SOIL POTASSIUM TEST FOR CORN IN NORTH DAT	КОТА 51
Abstract	
Introduction	
Methods and Materials	
Field Study	
Soil and Tissue Analysis	56
Data Analysis	58
Results and Discussion	59
Comparison of Soil Potassium Testing Methods	59
Effect of Soil Sample Drying on Exchangeable Potassium	61
Corn Grain Yield Response to Fertilization	65
Corn Plant Tissue Potassium Response to Fertilization	
Role of Soil Mineralogy in Grain Yield Response	80
Summary and Conclusions	

SEASONAL VARIATION IN SOIL TEST POTASSIUM LEVELS
Abstract
Introduction
Methods and Materials
Field Study
Soil Analysis
Data Analysis
Results and Discussion
Effect of Sampling Date on Soil Test Potassium91
Modeling of Soil Test Potassium over Time
Summary and Conclusions
GENERAL CONCLUSIONS
REFERENCES
APPENDIX. SUPPLEMENTARY TABLES OF SOIL TEST POTASSIUM VALUES 147

LIST OF TABLES

<u>Table</u>		Page 1
1.	Corn production in 2014 by country	7
2.	Nutrient composition of corn aboveground biomass	9
3.	Effect of soil sample drying on exchangeable K and inclusion of subsoil K on degree of correlation for multiple regression models predicting corn grain yield and yield increase to K in the North Central regional K studies on corn	50
4.	Locations, soil types, and soil characterization for nineteen field trials	55
5.	Agronomic information for nineteen field trials	56
6.	Soil test K values at nineteen trials sampled in late May by various soil test methods.	60
7.	Correlation of various soil test K methods.	61
8.	Regression equations for NH ₄ OAc extractable K from air-dry soil and its absolute difference and ratio with NH ₄ OAc extractable K from field-moist soil	64
9.	Corn grain yield response to K fertilization and initial soil test K.	66
10.	Models fitted to relationships between relative yields of unfertilized corn and plant available K evaluated by various soil test K methods.	71
11.	Critical values of soil test K for various soil test methods as determined by various models.	72
12.	Plant tissue K analysis for 2016 fertilizer K trials.	75
13.	Regression models fitted to relationships between tissue K concentration and soil test K.	77
14.	Critical values of soil test K for whole plant tissue K at growth stage V5 as determined by various models.	78
15.	Soil mineral composition for K-bearing and select minerals	81
16.	Principal component analysis between relative yield, NH ₄ OAC extractable K, and content of selected minerals.	82
17.	Classification of K trials by smectite/illite ratio.	85

18.	Regression models fitted to relationships between relative yield of unfertilized corn and NH4OAC extractable K from air-dry soil for high and low smectite/illite soils.	86
19.	Analysis of variance (ANOVA) for sampling time on cropped and fallow treatments for nineteen sites	93
20.	Cubic regression models of NH4OAc extractable K from air-dry soil over time on cropped plots.	98
21.	Cubic regression models of NH4OAc extractable K from field-moist soil over time on cropped plots	99
22.	Cubic regression equations for soil water content over time for cropped and fallow plots	103
23.	Cubic regression models of NH4OAc extractable K from air-dry soil over time on fallow plots	104
24.	Cubic regression models of NH ₄ OAc extractable K from field-moist soil over time on fallow plots.	105

LIST OF FIGURES

<u>Figure</u>		Page
1.	Average corn grain yield for the Northern Plains states of North Dakota, South Dakota, and Minnesota.	4
2.	Harvested acreage of principal crops grown in North Dakota	4
3.	Total corn grain production in North Dakota from 1980 to 2016	8
4.	Relationship between NH ₄ OAc extractable K from air-dry and field-moist soil across all trials	62
5.	Relationship between the difference and ratio of NH ₄ OAc extractable K from air- dry and field-moist soil and NH ₄ OAc extractable K from field-moist soil across all trials	63
6.	Relationship between relative yield of unfertilized corn and various soil test K methods: NH ₄ OAc extractable K on air-dry and field-moist soil, respectively; tetraphenylboron extractable K for 5 min and 168 h; resin extractable K; and K saturation.	70
7.	Relationship between tissue K concentration at growth stage V5 or VT and NH ₄ OAc extractable K on air-dry and field-moist soil.	76
8.	Relationship between tissue K concentration at growth stage V5 or VT and grain yield and relative grain yield.	79
9.	Cluster tree of K trials by smectite/illite ratio using Ward's minimum variance cluster analysis.	84
10.	Relationship between relative yield of unfertilized corn and NH4OAc extractable K on air-dry soil for high smectite/illite group 1 soils and low smectite/illite group 2 soils.	85
11.	Soil test K from air-dry and field-moist soil from cropped and fallow plots over time for seven of thirteen 2015 sites	94
12.	Soil test K from air-dry and field-moist soil from cropped and fallow plots over time for six of thirteen 2015 sites.	95
13.	Soil test K from air-dry and field-moist soil from cropped and fallow plots over time for 2016 sites	96
14.	Soil test K from air-dry and field-moist soil over time for cropped plots of combined sites from 2015 and 2016.	100

15.	Soil water content to air-dryness and its standardized Z-score within a site over	
	time for 2015 and 2016 sites	102

LIST OF ABBREVIATIONS

DK	Ammonium acetate (NH ₄ OAc) extractable K from air-dry soil
DOY	.Day of year
ECEC	Estimated cation exchange capacity by summation of extractable cations.
K sat	.K saturation of estimated cation exchange capacity
MK	Ammonium acetate (NH4OAc) extractable K from field-moist soil
RK	.Resin extractable K
ТВК	.Sodium tetraphenylboron (NaBPh4) extractable K

LIST OF APPENDIX TABLES

<u>Table</u>	Ī	Page
A1.	Soil test K over time of cropped and fallow surfaces at site AB15	147
A2.	Soil test K over time of cropped and fallow surfaces at site AR15	147
A3.	Soil test K over time of cropped and fallow surfaces at site B15	148
A4.	Soil test K over time of cropped and fallow surfaces at site C15	148
A5.	Soil test K over time of cropped and fallow surfaces at site D15	149
A6.	Soil test K over time of cropped and fallow surfaces at site F115	149
A7.	Soil test K over time of cropped and fallow surfaces at site F215	150
A8.	Soil test K over time of cropped and fallow surfaces at site LN15	150
A9.	Soil test K over time of cropped and fallow surfaces at site LS15	151
A10.	Soil test K over time of cropped and fallow surfaces at site M15	151
A11.	Soil test K over time of cropped and fallow surfaces at site P15	152
A12.	Soil test K over time of cropped and fallow surfaces at site V15	152
A13.	Soil test K over time of cropped and fallow surfaces at site W15	153
A14.	Soil test K over time of cropped and fallow surfaces at site AK16	153
A15.	Soil test K over time of cropped and fallow surfaces at site CF16	154
A16.	Soil test K over time of cropped and fallow surfaces at site GD16	154
A17.	Soil test K over time of cropped and fallow surfaces at site LB16	155
A18.	Soil test K over time of cropped and fallow surfaces at site MO16	155
A19.	Soil test K over time of cropped and fallow surfaces at site VC16.	156

GENERAL INTRODUCTION

Soil fertility research in North Dakota has been ongoing since 1908 when the first longterm fertilizer plots were established at the North Dakota Agricultural Experiment Station in Fargo (Zubriski and Moraghan, 1983). This early research recognized the importance of N and P for crop production in North Dakota. Some small yield increases to K_2SO_4 in corn (Zea mays L. ssp. mays), potato (Solanum tubersoum L.), and wheat (Triticum aestivum L.) were also recorded at the Fargo station (Volkerding and Stoa, 1947). In the 1950s, potash fertilizer application (KCl, 500 g K kg⁻ ¹) on corn was evaluated in southeast North Dakota, but no significant yield increases to K were observed (Bauer et al., 1956). In the 1960s, investigations of high protein content in malting barley (Hordeum vulgare L.) showed that KCl application could provide a modest increase in barley yield and kernel plumpness even though soil K levels were high (Bauer and Vasey, 1964; Zubriski et al., 1970). Soil testing was first offered to farmers as a service by North Dakota State University in fall 1953 (Dahnke et al., 1983). The North Dakota State University Soil Testing Laboratory provided NaHCO₃ extractable P, electrical conductivity, and soil pH tests. The North Dakota fertilizer recommendations from 1966 included adjustments for P rate based on soil test P level; however, the K fertilizer recommendations were simply based on soil texture (Weiser and Vasey, 1966).

The North Dakota State University Soil Testing Laboratory adopted a soil K test in 1968 in response to positive effects of K on barley yield and quality and other observations that K fertilization produced small yield increases in corn and potato on some soils (Zubriski and Moraghan, 1983). In Montana, wheat and barley grain yield and quality responses to KCl were also observed on high K soils (Skogley and Haby, 1981). Later research in North Dakota (Timm et al., 1986), South Dakota (Fixen et al., 1986), and Montana (Engel et al., 1994) identified that positive yield and quality responses to KCl in small grains on high K soils were not from correction of K deficiency but instead from correction of Cl deficiency and/or disease suppression by the added Cl. By this time, the soil K test had become established as part of the routine soil testing program in North Dakota. The K supplying power of major North Dakota soils had been assessed in greenhouse studies using millet (*Setaria italica* (L.) P. Beauv.) and sudangrass (*Sorghum bicolor* (L.) Moench ssp. *drummondii* (Nees ex Steud.) de Wet and Harlan), showing that cumulative plant K uptake was well correlated ($r^2 = 0.76$) with NH₄OAc extractable K (Sudjadi, 1969). These greenhouse experiments provided some support for the 1 M ammonium acetate extractant K test in North Dakota (Warnacke and Brown, 2012); however, extensive field validation was lacking. Some investigations into K fertilizer application for irrigated alfalfa were documented (Bauer and Bauder, 1978; Thompson et al., 1987).

With the exception of potato production, historical application of K fertilizers in North Dakota has been minimal and infrequent because soil K levels were generally high through the 1990s. In 1980, 3% of soil tests in North Dakota indicated medium or lower exchangeable K levels (<130 mg K kg⁻¹) (Nelson, 1979). Grain K export has far exceeded fertilizer K added to North Dakota soils for decades (Cassel et al., 1973; Dahnke et al., 1986; IPNI, 2012). In eastern North Dakota, the recent cropping system change to corn- and soybean-intensive crop rotations from historical wheat-dominated rotations (Figure 2) has exacerbated grain K export (IPNI, 2012). Increased grain K export without maintenance K fertilizer applications has resulted in accelerated soil K removal and a greater number of low testing exchangeable K soils are being reported. In 2010, 17% of soil tests in North Dakota had exchangeable K levels below the current 160 mg K kg⁻¹ critical level (Fixen et al., 2010). In 2015, 16% of soil tests were below the critical level (IPNI, 2016).

The present K fertilizer recommendations for corn in North Dakota have their origins in the late 1970s and early 1980s. The recommendations introduced by Wagner et al. (1977) and Dahnke and Vasey (1981) employed tables to calculate K fertilizer application rates based on corn yield goal and exchangeable K levels less than 150 mg K kg⁻¹. During this time, exchangeable K levels much greater than 200 mg K kg⁻¹ were nearly ubiquitous across the state (Dahnke et al., 1982, 1992b), and average state corn yields ranged from 3 to 6 Mg ha⁻¹ (Figure 1), which are modest yields compared to present yields achieved by state growers. The native soil K fertility was sufficient for most crop K requirements through the 1990s with the exception of particularly sandy soils, such as those on beach ridges of glacial Lake Agassiz on the western side of the Red River Valley or for high K requiring crops such as potato (Zubriski and Moraghan, 1983).

Dahnke et al. (1992a) updated the K recommendation tables for higher corn grain yields, increased the soil test K critical level to 160 mg K kg⁻¹, and introduced an equation for determining K fertilizer rate:

K fertilizer rate =
$$(1.1660 - 0.0073 \text{ STK}) \text{ YG}$$
 (Eq. 1)

where K fertilizer rate (lb K₂O acre⁻¹); STK, exchangeable K (mg K kg⁻¹) for 0-15 cm soil depth; and YG, yield goal (bushel acre⁻¹). This equation (Eq. 1) remained part of the North Dakota fertilizer recommendations for corn until Franzen (2014) abandoned the yield goal concept in favor of economically optimum N rates, which simplified the soil test K recommendation tables. The present K recommendations are still based on the 160 mg K kg⁻¹ soil test K critical level introduced by Dahnke et al. (1992a). Throughout this time, corn yields and acreage increased greatly (Figure 1; Figure 2); however, the body of soil K research in North Dakota had remained scant until reinvestigation of current K recommendations for corn by Rakkar et al. (2015), who found that current soil testing approaches and the 160 mg K kg⁻¹ critical level did not accurately predict corn grain yield response to K fertilization.



Figure 1. Average corn grain yield for the Northern Plains states of North Dakota, South Dakota, and Minnesota (adapted from USDA-NASS, 2017).



Figure 2. Harvested acreage of principal crops grown in North Dakota (adapted from USDA-NASS, 2017).

The remainder of this thesis is organized as follows: chapter LITERATURE REVIEW provides an extensive review of corn K requirements, soil K mineralogy and dynamics, and soil K testing methods and interpretation; chapter RECALIBRATION OF SOIL POTASSIUM TEST FOR CORN IN NORTH DAKOTA includes an Introduction section, which summarizes literature concerning the evaluation of corn response to K fertilization and soil K testing methods, a Methods and Materials section, which details the site characteristics, experiment design, field procedures, laboratory methods, and statistical analysis methods, a Results and Discussion section, which presents and discusses experiment results for soil test methods, corn grain yield, corn tissue K, and soil mineralogy, and a Summary and Conclusions section to synthesize the study on soil K test recalibration for corn; chapter SEASONAL VARIATION IN SOIL TEST POTASSIUM LEVELS includes an Introduction section, which summarizes literature concerning seasonal variation in soil test K, a Methods and Materials section, which details the experiment design, field procedures, laboratory methods, and statistical analysis methods, and a Results and Discussion section, which presents and discusses experiment results for soil test K and soil water content over time, and a Summary and Conclusions section to synthesize the study on seasonal variation in soil test K; and chapter GENERAL CONCLUSIONS to synthesize the results of both studies.

LITERATURE REVIEW

Corn

Corn Origin and Crop

Corn (*Zea mays* L. ssp. *mays*), also referred to as maize, is the third most important cereal crop in the world after wheat (*Triticum aestivum* L.) and rice (*Oryza sativa* L.) (Duncan, 1975). Between 7,000 and 10,000 years ago, corn was domesticated from teosinte (*Zea mays* L. ssp. *parviglumis* Iltis & Doebley), a wild grass from Mexico (Iltis and Doebley, 1980; Ranum et al., 2014). The early domesticators selected for traits that resulted in significant changes in plant and inflorescence structure, ear size and shape, and seed color and casing (Doebley et al., 2006; Gross and Olsen, 2010). During the Columbian Exchange, corn was brought to the Old World, where it quickly became an important food crop throughout Europe by the 16th Century and spread to the Middle East, Africa, and Asia (Hehn, 1888). Historically, corn had been open-pollinated until plant breeders developed hybrid corn in the 1930s by developing inbred lines and then crossing them (Troyer, 1990). Genetic improvement through hybridization, along with improved agronomic practices (e.g., fertilizer, pesticides), greatly improved corn yields during the latter half of the 20th Century (Watson, 1988).

Corn grain contains about 72% starch, 10% protein, and 4% fat (Ranum et al., 2014). Of the numerous races of corn that have been developed in the Americas (Goodman and Brown, 1988), most grain corn produced can be classified as dent, flint, soft, waxy, sweet, or popcorn based on kernel composition, characteristics, and usage (Duncan, 1975); dent corn is the most widely grown grain corn in the United States. Grain corn is utilized for direct human consumption, animal feed, seed, ethanol production, and industrial uses (Ranum et al., 2014; Watson, 1988). Silage corn is grown for its high biomass yield rather than grain yield and is harvested as a vegetative animal feed (Allen et al., 2003).

Corn grain yield is a function of the kernel number established at pollination and the quantity of photosynthate available for grain filling (Duncan, 1975). The individual yield components of corn grain are plants per unit area (plant population), stalks per plant, ears per stalk, kernels per ear, and kernel weight.

Global and U.S. Corn Production

Corn is produced throughout the world; however, the majority of corn is grown between North or South latitudes 30° and 55° in regions with adequate summer rainfall or stored soil water for seasonal corn water use (30 to 84 cm), growing season temperatures between 21 and 27 °C, and 120 to 180 frost-free days (Shaw, 1988). Major corn producing countries or regions are the United States, China, South America, and the Black Sea Region (Table 1). In the 1950s and 1960s, the United States produced 60 to 70% of the total world corn crop, but expansion of corn production throughout the world had decreased the United States' share to 44 to 48% by the 1980s (Watson, 1988). In 2014, global corn production was estimated at 1,038 million Mg with the United States, China, and Brazil being responsible for 35, 21, and 8%, respectively (FAO, 2017). In 2016, the United States produced a record 386 million Mg of corn (USDA-NASS, 2017).

Table 1.Corn production in 2014 by country†.

Country Corn production		
	million Mg	
United States	361	
China	216	
Brazil	80	
Argentina	33	
Ukraine	28	
World	1038	
† Adapted from FAO (2017).		

North Dakota Corn Production

North Dakota has traditionally been on the northwestern fringe of United States Corn Belt with the main limitations being a short growing season and yield limiting rainfall (Ransom et al., 2004). Nevertheless, corn production in North Dakota has increased greatly since the early 1990s (Figure 3). The expansion of corn acreage in North Dakota (Figure 2) has been attributed largely to genetic improvement of early maturing corn hybrids and higher than average rainfall, particularly in the eastern third of North Dakota (Ransom et al., 2004). Corn yields in North Dakota tend to be lower than South Dakota or Minnesota, whose growing seasons and rainfall amounts and distribution are more accommodating (Figure 1). In 2016, North Dakota produced a record 13.2 million Mg, surpassing the previous record of 10.7 million Mg from 2012, which ranked North Dakota as 12th in corn production among the United States (USDA-NASS, 2017).



Figure 3. Total corn grain production in North Dakota from 1980 to 2016 (adapted from USDA-NASS, 2017).

Corn Fertility

General Corn Fertility

Seventeen elements are essential for plant growth and reproduction, i.e., seed or grain production (Havlin et al., 2005). Of these essential elements, 14 mineral elements are obtained from soil: N, P, K, Ca, Mg, S, Cl, Fe, B, Mn, Zn, Cu, Mo, and Ni. The average nutrient composition of corn, analyzed by individual study authors, is listed in Table 2. Nitrogen availability has been identified as a major limiting factor in high-yielding corn production (Franzen, 2017). Corn yield responses to P and K are widely recognized, and S deficiencies are becoming increasingly more common (Franzen, 2017). Corn may be responsive to B, Fe, Mn, and Zn in some regions (Franzen, 2017).

	Dry matter and concentration [†]			
	Hanway	Karlen et al.	Bender et al.	Ciampitti and
Nutrient	(1962)	(1988)‡	(2013)	Vyn (2014)
			Mg ha ⁻¹	
Dry matter	17.5	31.8	23.0	21.2 - 23.2
			- g kg ⁻¹	
Ν	11.5	12.1	12.4	11.2 - 12.0
Р	1.9	2.2	2.2	2.3 - 2.6
Κ	6.1	11.6	7.1	8.7 - 9.3
Ca		1.9		
Mg		1.4	2.6	1.5 - 1.7
S		1.3	1.1	0.9 - 1.0
		mg kg ⁻¹		
В		4.1	3.6	
Cu		4.4	6.1	
Fe		59.7	59.8	
Mn		28.3	23.6	
Zn		25.2	21.7	15 - 17

 Table 2.
 Nutrient composition of corn aboveground biomass.

[†] Based on total accumulation in biomass (grain and stover) at maturity, given optimal fertility.

‡ Irrigated.

Potassium in Corn Production

Potassium Function in the Plant

Potassium is absorbed through plant roots as K^+ from the soil solution (Mengel and Kirkby, 2001). Unlike other macronutrients, K is not assimilated into organic compounds but remains as a free K^+ in plant cell cytosol, vacuoles, and vascular tissues (Mengel and Kirkby, 2001). Potassium is involved in meristematic growth, cellular water regulation, photosynthesis, photosynthate translocation, and enzyme activation (Mengel and Kirkby, 2001; Welch and Flannery, 1985). Corn plants without adequate K exhibit decreased photosynthesis, particularly in lower leaves where leaf K concentration is lowest (Moss and Peaslee, 1965). Photosynthesis may be reduced in corn leaves with less than 2.0 g K kg⁻¹ on a fresh weight basis (Peaslee and Moss, 1966). As corn grows and total biomass increases, total plant K concentration decreases (Ciampitti et al., 2013), which is caused by a dilution effect from dry matter accumulation (Mengel and Kirkby, 2001; Munson and Nelson, 1990).

Corn plants with adequate K have larger stomatal openings, which allow more rapid diffusion of CO_2 into the leaf, thus fostering more photosynthesis (Welch and Flannery, 1985). Larger stomatal openings also increase the loss of water from leaves via transpiration. In spite of this, the relative increase in photosynthesis is greater than water loss through transpiration, resulting in a net increase in water use efficiency of corn (Welch and Flannery, 1985).

Potassium-stressed corn plants may have weaker stalks that are more susceptible to stalk lodging and breakage and root and stalk diseases (Welch and Flannery, 1985). Under these conditions, K fertilization has reduced stalk lodging (Boswell and Parks, 1957; Josephson, 1962; Krantz and Chandler, 1951; Wittels and Seatz, 1953) and *Gibberella* stalk rot incidence (Josephson, 1962; Otto and Everett, 1956). There was speculation that Cl in commonly used KCl fertilizer was the cause of reduced stalk lodging; however, evaluation of NH₄Cl and KCl fertilizer sources confirmed that reduced lodging was caused by adequate K nutrition (Liebhardt and Munson, 1976).

Potassium is mobile within the plant, and it is frequently cycled internally to facilitate photosynthate translocation in phloem (Mengel and Kirkby, 2001). If K is deficient in the plant, K will be translocated from older to younger leaves, where plant growth and photosynthesis is most active (Welch and Flannery, 1985). Potassium deficiency symptoms in corn are visually manifested as chlorosis and necrosis that begins at leaf tip and outer leaf margin and progresses from the leaf edges towards the mid-vein. Once plant tissue has turned to a tan color, the deficiency symptom within the tissue cannot be reversed with the application of K fertilizer because the tissue has been irreversibly senesced (Welch and Flannery, 1985), although newer tissue without deficiency necrosis can benefit. Although K-stressed plants may not exhibit visual K deficiency symptoms, photosynthesis, plant growth, and grain yield may be reduced; this has been termed hidden hunger (Havlin et al., 2005; Welch and Flannery, 1985).

Potassium Requirements and Response

As a high total biomass crop, corn requires K in high amount for adequate growth and grain yield (Table 2). Corn K uptake requirement is commonly estimated at 21 kg K Mg⁻¹ grain (IPNI, 2014). Corn rapidly accumulates K in early growth stages with 63% (Bender et al., 2013) to 100% (Ciampitti et al., 2013) of total plant K accumulated by growth stage R1. During periods of rapid growth (V10 to V14 growth stages), corn K accumulation rate may be as high as 4.8 kg K ha⁻¹ d⁻¹ (Bender et al., 2013). Corn grain K concentration is commonly estimated to be 3.7 kg K Mg⁻¹ grain (IPNI, 2014); however, grain K concentration may range from 1.8 to 4.6 kg K Mg⁻¹ grain with higher yielding corn containing more K in its grain (Ciampitti and Vyn, 2014). The addition of K

fertilizer seldom increases corn grain K concentration within a field (Clover and Mallarino, 2013; Mallarino and Higashi, 2009).

Plant analysis has frequently been employed to assess the nutritional status of crop plants. Corn tissue K concentration varies by plant part and growth stage (Karlen et al., 1988; Mallarino and Higashi, 2009; Walker and Peck, 1975); therefore, tissue K critical concentrations designating sufficient K status must be calibrated for specific plant parts and growth stages (Walker and Peck, 1975). Within single fields, corn grain yield and tissue K may correlate well (Tyner, 1947; Walker and Peck, 1975); however, attempts to correlate tissue K to grain yield response to K fertilization across sites have been less successful (Mallarino and Higashi, 2009). According to Havlin et al. (2005), sufficient corn tissue K concentrations are 30 to 50 g K kg⁻¹ for whole plants less than 30 cm in height, 20 to 35 g K kg⁻¹ for the third leaf of plants greater than 30 cm in height, and 20 to 30 g K kg⁻¹ for ear leaves at R1 growth stage. In general, tissue K critical concentration decreases for older plant parts. Clover and Mallarino (2013) determined that the optimum tissue K concentration to maximize corn grain yield in Iowa was 20.2 to 25.1 g K kg⁻¹ for whole plants at V5-V6 growth stage and 10.2 to 11.0 g K kg⁻¹ for ear leaves at R1 growth stage. Corn hybrids vary in their K uptake and utilization efficiency (Pettigrew, 2008); therefore, tissue K critical concentrations are not universal or permanent. At two long-term trials in south central Minnesota, ear leaf K concentration at R1 growth stage for optimal corn grain yield varied from 11.4 to 22.9 g K kg⁻¹ depending on hybrid and year (Randall et al., 1997a). Randall et al. (1997a) noted that optimal leaf K concentrations had decreased over time; this was attributed to hybrid improvement during the study period.

Corn is known to accumulate K beyond what is required for maximum dry matter or grain yield (Bartholomew and Janssen, 1929); this has been termed luxury consumption. Tissue K is

often increased with K fertilization; however, grain yield response may be less frequent (Mallarino and Higashi, 2009). At two long-term sites in south central Minnesota, K fertilization increased corn K leaf concentration consistently on medium (~110 mg K kg⁻¹) and high (~160 mg K kg⁻¹) exchangeable K soils alike, but corn grain yield was only increased in three of 14 years on the medium K soil and never on the high K soil (Randall et al., 1997a; 1997b).

Crop response to fertilization is dependent on the plant available nutrient supply from soil and fertilizer. For responsive sites in Iowa, corn grain yield was maximized between 106 to 127 kg K ha⁻¹ and often decreased at higher rates up to 168 kg K ha⁻¹ (Clover and Mallarino, 2013). In Wisconsin, corn grain and biomass yield was maximized at 112 kg K ha⁻¹ and decreased at 149 kg K ha⁻¹ (Laboski and Andraski, 2016). The availability of applied fertilizer K may be reduced by fixation reactions (to be discussed later). Potassium fixation is not generally a factor in limiting crop response to K fertilizer (Havlin et al., 2005); however, soils with high K fixation capacity may have serious production limitations. For example, a high K fixing soil from Croatia required a seasonal application of 1580 kg K ha⁻¹ to maximize corn grain yield (Kovačević and Vukadinović, 1992).

Soil Potassium

Potassium Forms in Soil

Earth's lithosphere contains approximately 23 (Mengel and Kirkby, 2001) to 26 g K kg⁻¹ (Lindsay, 1979; Rich, 1968) with an average content of 8.3 g K kg⁻¹ for soils (Lindsay, 1979) ranging from 0.4 to 26 g K kg⁻¹ (Sparks and Huang, 1985). The difference between lithosphere and soil K content reflects the removal of K through weathering and leaching during pedogenesis (Lindsay, 1979). Although the total K content of soils is relatively high compared to other nutrient elements (Sparks and Huang, 1985), the amount of soil K readily available to plants is much lower.

For edaphic purposes, soil potassium has been classified into four groups based on their relative availability to plants, in order of decreasing availability: solution K, exchangeable K, nonexchangeable K, and structural K (Sparks and Huang, 1985). Soil organisms have not been widely considered in soil K dynamics. In Ohio, microbial K biomass ranged from 68 to 179 mg K kg⁻¹ under conventional till and no-till corn production (Lorenz et al., 2010); this microbial K pool is not insignificant and requires more attention.

Solution K is comprised of K⁺ dissolved in the soil solution; this pool is directly available for plant uptake. As solution K is depleted through plant uptake, it is replenished by exchangeable K, nonexchangeable K, and structural K. Exchangeable K comprises K⁺ held by electrostatic attraction to negatively-charged exchange sites on clay minerals and soil organic matter (Sparks and Huang, 1985). Exchangeable K readily participates in cation exchange reactions with the soil solution and equilibrates rapidly. Merwin and Peech (1951) observed that 40 to 80% of exchangeable K was obtained from the clay fraction, 15 to 50% from the silt fraction, and a negligible amount from the sand fraction of four New York soils. Similarly, exchangeable K distribution for four Ohio soils was 30 to 74% from clay, 24 to 56% from silt, and 3 to 21% from sand (Munn et al., 1976).

At similar exchangeable K concentrations, high CEC soils have lower solution K concentrations and activity than low CEC soils (Bell et al., 2009); this suggests that high CEC soils may require greater exchangeable K levels to provide sufficient solution K concentrations for plant K uptake. This relationship between solution K and exchangeable K is consistent within soil mineralogical class. Generally, kaolinitic soils will exhibit higher solution K concentrations relative to exchangeable K than smectitic soils (Sharpley, 1989). Although the total K content of surface horizons decreases during pedogenesis through weathering and leaching, exchangeable K

is replenished and enriched in surface horizons relative to subsoil through nutrient uplift and cycling by plants (Barré et al., 2007a).

Nonexchangeable K comprises K⁺ held between adjacent tetrahedral layers of 2:1 phyllosilicates but not covalently bonded within the crystal structure (Martin and Sparks, 1985). As the name implies, nonexchangeable K does not readily participate in cation exchange reactions; however, its release is indeed a slow, diffusion-controlled exchange reaction (Huang, 2005) that can provide significant plant available K during a growing season. The definition for nonexchangeable K encompasses two other definitions commonly used in the literature: (i) fixed K, which describes K⁺ that has been added to the soil system and rendered nonexchangeable, often in frayed edges and wedge sites of 2:1 phyllosilicates and (ii) interlayer K, which more broadly describes K⁺ held in the collapsed interlayer space of 2:1 phyllosilicates. Structural K refers to K⁺ that is integral to mineral structure (Sparks and Huang, 1985). For K-feldspars, K⁺ is covalently bonded within their tectosilicate structure. This definition also applies to interlayer K that binds the 2:1 layers of micas.

Potassium-bearing Minerals

The principal source of K in soils are the primary minerals of the feldspar and mica groups (Sparks and Huang, 1985). These minerals encompass the vast majority of total K in soils. Potassium-tarankaite $[K_3Al_5H_6(PO_4)_8 \cdot 18H_2O]$ and K-alunite $[KAl_3(SO_4)_2(OH)_6]$ occur occasionally in soils with low pH and high activity of their ionic constituents, particularly those of volcanic origin (Huang, 2005). The soluble K complexes of sulfate, chloride, bicarbonate, hydroxide, and carbonate are of minor importance in well-drained soils (Lindsay, 1979); therefore, our attention will focus on silicate minerals.

Feldspars are tectosilicates with three-dimensional framework structure of linked Si-O and Al-O tetrahedra (Huang, 1989). The K-bearing feldspars include K⁺ in their structure to maintain electroneutrality. The K-bearing feldspars are polymorphs of identical chemical composition (KAl₂Si₃O₈): sanidine, orthoclase, and microcline, and adularia (Huang, 1989). Orthoclase and microcline are formed in igneous and metamorphic environments. Micocline, the low temperature polymorph, is more stable than orthoclase, thus less vulnerable to weathering in soils (Sparks and Huang, 1985). Sanidine is found in K-rich volcanic rocks, and adularia is formed in hydrothermal veins (Huang, 1989). The release of K from feldspars is facilitated by H⁺ attack more than low solution K concentration (Song and Huang, 1988).

Micas are 2:1 phyllosilicate minerals with nonhydrated interlayer cations tightly held in the interlayer space to balance high layer charge (Fanning et al., 1989). Mica minerals have dioctahedral or trioctahedral structure, which refers to cation occupation of the octahedral layer (Fanning et al., 1989). The octahedral cations are usually Al^{3+} for dioctahedral micas and Fe^{2+} or Mg^{2+} for trioctahedral micas (Fanning et al., 1989). The common K-bearing micas of igneous and metamorphic origin are biotite (trioctahedral) and muscovite (dioctahedral) (Sparks and Huang, 1985). Phlogopite is a trioctahedral mica found in serpentinitic rocks and metamorphosed Mg-rich rocks (Sparks and Huang, 1985).

Within the 2:1 mica structure, a hydroxyl group from the Al-O octahedral sheet is positioned within the siloxane cavity in the basal plane of the Si-O tetrahedral sheet. The hydroxyl groups within the siloxane cavities of trioctahedral micas are positioned perpendicularly to the cleavage plane, whereas the hydroxyl groups within the siloxane cavities of dioctahedral micas are positioned obliquely to the plane (Martin and Sparks, 1985). The oblique position of hydroxyl groups within the siloxane cavity of dioctahedral micas allows K⁺ to be held closer to negatively charged O atoms, providing a stronger electrostatic charge and tighter collapsed layer (Martin and Sparks, 1985). The interlayer K release rate from muscovite is 5 and 15% of that from biotite and phlogopite, respectively (Feigenbaum et al., 1981).

The removal of interlayer K from micas causes layer expansion and formation of frayed edges or mixed-layer structures (Scott and Reed, 1964). Frayed edges and partially expanded interlayers provide wedge sites with high selectivity for weakly hydrated cations such as K⁺ (Sawhney, 1972; Sparks and Huang, 1985). Micas serve as the precursor for expansible 2:1 clays such as smectite and vermiculite (Fanning et al., 1989). Interstratified mica-derived minerals with structures more similar to muscovite will release K more slowly (Ross and Kodama, 1970).

Soil Clay Minerals

Illite is a nonexpanding, muscovite-like 2:1 phyllosilicate in the clay-size fraction (Fanning et al., 1989; Środoń and Eberl, 1984); it is sometimes called hydrous mica or hydromica. The mineralogical definition of illite is not universally agreed upon, but illite is generally considered to have poorer crystallinity, lower K content, and higher H₂O content than muscovite (Fanning et al., 1989). The formation of illite is classically explained by removal of K from mica; however, illite may also form by rock–water interaction in K-feldspar (Velde and Meunier, 2008). Like micas, illite has K selective frayed edges and wedge sites capable of fixing K (Sparks and Huang, 1985).

In most cases, soil illite exhibits two X-ray diffraction peaks constituted of poorly- and well-crystalize illite (Velde and Meunier, 2008). Poorly-crystalline illite contains K that participates more readily in exchange reactions than the well-crystalline form (Velde and Meunier, 2008). Velde (2001), who surveyed 86 soils of the central United States, found that mixed-layer smectite–illite was the dominant clay mineral throughout the central United States. Mixed-layer

smectite–illite intergrades may have random or ordered arrangements of illitic material (Środoń and Eberl, 1984). The expandability of smectite–illite decreases with increasing amount of illitic material, where low interlayer K content has a random arrangement and high interlayer K content produces an ordered arrangement with thicker illitic stacks that contain nearly double the amount of K per (OH)₂ group (Środoń and Eberl, 1984).

Vermiculite is an expansible 2:1 clay-sized mineral with dioctahedral or trioctahedral structure with high layer charge originating from the tetrahedral layer (Douglas, 1989). Potassium can be fixed between vermiculite interlayers because K⁺ has low hydration energy, allowing interlayer collapse upon dehydration (Douglas, 1989). Collapsed vermiculite exhibits layer arrangements with K-selective wedge sites (Douglas, 1989). Smectites are a group of expansible 2:1 clay-sized minerals with dioctahedral structure and layer charge originating from either tetrahedral or octahedral layer (Borchardt, 1989). Montmorillonite and beidellite are the two important smectite species in soils (Borchardt, 1989). By most definitions, smectites generally do not fix K (Borchardt, 1989). Both vermiculite and smectite contribute greatly to soil CEC upon which exchangeable K may be held (Borchardt, 1989; Douglas, 1989).

Beidellite is a high layer charge smectite with tetrahedral substitution (Borchardt, 1989) believed to be derived from illite weathering (Środoń and Eberl, 1984). Beidellite can fix added K, which results in decreased CEC upon layer contraction (Ross and Mortland, 1966). The K fixation potential of beidellite is more similar to illite than montmorillonite (Foster, 1953). Foster (1953) expressed concern that common conceptual distinctions between montmorillonite and illite had been oversimplified and failed to address differences between tetrahedral-octahedral charge relationships relevant to K fixation (i.e., beidellite). The role of tetrahedral charge in vermiculite is widely recognized in K fixation; however, the recognition of high charge smectites and their K

fixation capacity remains neglected in introductory soil science (Brady and Weil, 2008) and soil fertility (Havlin et al., 2014) textbooks.

Potassium Mineralogy of Northern Plains Soils

The total K content of North American surface soils is strongly correlated ($r^2 = 0.83$) to their K-feldspar and illite content (Eberl and Smith, 2009). Across North America, the K-feldspar ($r^2 = 0.75$) and total K ($r^2 = 0.92$) content of soils is strongly correlated with higher latitudes (Eberl and Smith, 2009). In three Mollisols from eastern North Dakota, the K-feldspar content of the sand fraction ranged from 5 to 10% (Redmond and Whiteside, 1967). The total K content of soil parent material in the northern Red River Valley of North Dakota is between 8 to 17 g K kg⁻¹, with a higher K content in the clay fraction than the silt fraction (Klassen, 2009).

Soils formed on parent materials of glacial origin in eastern North Dakota (e.g., glacial till, glacial outwash, lacustrine sediments, and their local alluvium) are generally described as having mixed or smectitic mineralogy (USDA-NRCS, 2006). The specific mineralogical nature of the smectites in North Dakota has not been extensively investigated, however. Early mineralogical characterization of three eastern North Dakota Mollisols indicated that montmorillonite was the dominant clay mineral with minor amounts of illite in the A and B horizons (Redmond and Whiteside, 1967). In northwestern Minnesota, Pluth et al. (1970) characterized the mineralogy of the Fargo series (fine, smectitic, frigid Typic Epiaquerts) as being dominated by montmorillonite. Borchardt (1989) remarked that most soil smectites have been called montmorillonite even though they may have significant tetrahedral charge; the diagnostic procedures of Redmond and Whiteside (1967) and Pluth et al. (1970) did not distinguish between montmorillonite and beidellite smectite species (Borchardt, 1989). Badraoui et al. (1987), using a Li-saturation method, later identified beidellite as the dominant smectite species in the Fargo series. The extent or prevalence of

montmorillonite or beidellite is unknown in North Dakota as no comprehensive survey of smectite speciation had been conducted in North Dakota as of this review.

Velde (2001) estimated that mixed-layer smectite–illite clays in the central United States contained up to 20% high layer charge smectite. In east-central South Dakota, four smectitic soils were identified as montmorillonite dominant based on 17% of the total CEC attributed to the tetrahedral layer (Schindler et al., 2003); this amount of tetrahedral charge is similar to that in the mixed-layer smectite–illite observed by Velde (2001). In central Manitoba, vermiculite, illite, and kaolinite were identified as the dominant clays in soils derived from glacial Lake Agassiz sediments (Aide et al., 1999); the abundance of vermiculite and mixed-layer clays is greater in northern and central Manitoba than southern Manitoba (Klassen, 2009). Lake Agassiz sediments (Klassen, 2009).

Soil Potassium Dynamics

Ion Selectivity

Ion selectivity refers to the preferential sorption of certain cations in exchange reactions. Exchange reactions on smectites involving only strongly hydrated cations (e.g., Ca^{2+} , Mg^{2+} , Na^{+}) are non-selective and express no hysteresis; however, reactions involving at least one weakly hydrated cation (e.g., K^+ , NH_4^+) are non-ideal and often express hysteresis (Bourg and Sposito, 2011). Selectivity for K in soils has been well documented (Feigenbaum et al., 1991; Goulding and Talibudeen, 1979, 1984a; 1984b; Jardine and Sparks, 1984a; Wada and Odahara, 1993; Wada and Weerasooriya, 1990). The ionicity index derived by Marchuk and Rengasamy (2011), based on ionization potential and charge, predicts that Ca^{2+} and Mg^{2+} should be preferred over K⁺ by negative exchange sites. However, frayed edges and wedge sites on 2:1 phyllosilicates provide a

steric effect that results in selective preference for K (Martin and Sparks, 1985). This selective preference in soils is enthalpy driven by the binding energy to these K specific sites (Sparks and Jardine, 1981).

Selectivity for K is largely a function of the clay mineralogy. Solution K activity of different clay suspensions was, in decreasing order: kaolinite > montmorillonite >> illite > beidellite (Marshall and McLean, 1948). Drying decreased solution K activity for montmorillonite and beidellite clay suspensions (Marshall and McLean, 1948), indicating some fixation upon drying in smectites.

Since frayed edges and wedge sites in micas and illite exhibit selectivity for weakly hydrated cations, NH_4^+ is able to exchange with K^+ in wedge sites, but large strongly hydrated ions such as Ca^{2+} or Mg^{2+} are not (Bolt et al., 1963; Rich, 1964). As interlayer K is removed from illite, the selectivity for K increases as the frayed edges become larger (Sawhney, 1970). When interlayer K is fully depleted from mica leading to full expansion and absence of wedge sites, K selectivity should decrease, however (Sawhney, 1972). If hydroxy-Al or -Fe is present in clay interlayers to prevent layer collapse, Ca^{2+} or Mg^{2+} may also exchange for K⁺ (Rich and Black, 1964). Removal of Al³⁺ from illite by F⁻ was shown to increase K fixation (Stanford, 1948).

Heterogeneous soil systems exhibit multireactive exchange selectivity with preference for K at low solution K concentrations and preference for Ca at higher solution K concentrations (Jardine and Sparks, 1984a). At low K concentrations, micaceous minerals such as illite and biotite have greater K selectivity than vermiculite or montmorillonite, which is attributed to frayed edges and greater tetrahedral layer charge (Sawhney, 1970). As solution K concentration increases, vermiculite will have greater selectivity than micas (Sawhney, 1970), which can be attributed to

satisfaction of highly selective wedges sites in micas, whereas vermiculite has a greater surface area to satisfy.

Exchange reactions at selective sites are slower than non-selective sites (Beckett and Nafady, 1967), and those reactions at frayed edges are non-equilibrium process that are diffusion controlled (Martin and Sparks, 1985). In an Evesboro soil (mesic, coated Lamellic Quartzipsamments) from Delaware, rapid K adsorption saturated readily accessible exchange sites (35% of total CEC) in 8 min, whereas difficultly accessible exchange sites (65% of total CEC) required many hours (Jardine and Sparks, 1984b); the slower kinetics were attributed to vermiculite. First-order kinetics for K adsorption are rapid for kaolinite, intermediate for montmorillonite (saturated after 120 min), and slow for vermiculite (not equilibrated after 240 min) (Jardine and Sparks, 1984b). Desorption of K tends to be slower than adsorption (Sparks and Jardine, 1981).

Potassium selectivity is not limited to micas, illites, and vermiculites. Selectivity has also been observed in kaolinite and kaolinitic soils (Levy et al., 1988; Udo, 1978) and montmorillonite and montmorillonitic soils (Carson and Dixon, 1972; Feigenbaum et al., 1991; Knibbe and Thomas, 1972; Wada and Weerasooriya, 1990). In kaolinite and montmorillonite, preference for K is caused by heterogeneous charge sites (Goulding and Talibudeen, 1980; Talibudeen and Goulding, 1983a; 1983b). Mica impurities in kaolinite (Lee et al., 1975) and montmorillonite (Carson and Dixon, 1972; Knibbe and Thomas, 1972) are recognized as the source of charge heterogeneity. Selectivity for weakly hydrated cations on smectite increases with surface charge density (Shainberg et al., 1987). Beidellite (McLean, 1951) and the smectite–vermiculite component of mixed-layer minerals (Goulding and Talibudeen, 1979) exhibit greater K selectivity.
Even for relatively simple 2:1 clays like smectite, Bourg and Sposito (2011) recognized that ion selectivity was determined by the stacking arrangement of smectite lamellae, which is influenced by interlayer hydration level, magnitude and degree of layer charge, adsorbed cations, and thermodynamic activity of water. The heterogeneous nature of mixed-layer smectite–illite makes modeling its selectivity difficult. Tournassat et al. (2009) were able to develop a simple combination of smectite and illite exchange models to estimate selectivity coefficients for mixed layer smectite–illite minerals.

Potassium Fixation and Release

The irreversible fixation of K was observed when soil samples were dried and added K could not be fully recovered as exchangeable K (Joffe and Kolodny, 1937). Joffe and Kolodny (1937) proposed that K was being fixed in insoluble P complexes. Bray and DeTurk (1939) offered that fixation and release of K were exchange reactions that may lead to formation of a secondary mica like illite; they noted that fixation and release rates were too rapid to be explained by insoluble complexes that could persist under weathering in soils [Lindsay (1979) stated that ionic K complexes are of minor importance in well-drained soils]. Potassium fixation also decreased CEC, suggesting that K fixation was related to the exchange complex (Joffe and Kolodny, 1939; Joffe and Levine, 1940). Attoe (1947) demonstrated that soils did not have to be dried for fixation to occur as K could be fixed in moist and dried soils alike.

Potassium fixation occurs when interlayer binding forces to a 2:1 phyllosilicate mineral exceed ion hydration forces, causing ion dehydration, partial layer collapse, and physical restriction of K⁺ (Martin and Sparks, 1985). In smectite and vermiculite, K fixation is correlated with the tetrahedral-layer CEC (64%) and octahedral-layer CEC (36%) (Bouabid et al., 1991). Having greater tetrahedral charge, vermiculites fix more K than smectites, which generally do not

fix K (Borchardt, 1989; Douglas, 1989). The low hydration energy of K^+ , in contrast to strongly hydrated Ca^{2+} or Mg^{2+} (Huang, 2005), allows dehydrated K^+ to fit into collapsible interlayers or wedge sites more easily. Dennis and Ellis (1962) demonstrated that dehydration with alcohol exacerbated K fixation by vermiculite.

Release of nonexchangeable K requires entrance of an unhydrated ion into the interlayer space and its subsequent hydration to expand the interlayer to allow hydration of K⁺ (Martin and Sparks, 1985). Ellis and Mortland (1959a) proposed that release of fixed K was diffusion controlled. The physical entrapment of K makes diffusion the rate-limiting step of K release (Martin and Sparks, 1985). Potassium release is faster for K⁺ held on planar and peripheral sites of 2:1 phyllosilicates than deeper interlayer K (Goulding, 1981). Fixed K can be released through leaching of exchangeable K (Wood and DeTurk, 1943), and low solution K concentration provides the driving force for K diffusion from the interlayer (Martin and Sparks, 1985). The solution K critical concentration for nonexchangeable K release is 4 μ g mL⁻¹ for whole soils, 2.3 to 18.8 μ g mL⁻¹ for trioctahedral micas, and <0.1 μ g mL⁻¹ for muscovite and illite (Martin and Sparks, 1985). The release of fixed K from previously weathered biotite was faster than from unweathered biotite (Ellis and Mortland, 1959b), which suggests some loss of charge or layer configuration occurred during weathering or refixation.

Ammonium is fixed in 2:1 phyllosilicates by the same mechanism as K^+ (Stanford and Pierre, 1947) and the magnitude of fixation is linearly correlated (Rich and Lutz, 1965). The presence of K^+ and NH_4^+ in solution inhibits the release of the other fixed ion into solution by inhibiting layer expansion (Hanway et al., 1957; Welch and Scott, 1960). Co-application of anhydrous ammonia and KCl in fertilizer bands reduces K fixation and increases exchangeable K within the fertilizer band (Stehouwer and Johnson, 1991). This has been attributed to competition

for fixation sites (Stehouwer and Johnson, 1991) and increase in pH-dependent CEC on soil organic matter that exhibits greater Ca^{2+} selectivity and lower K⁺ selectivity (Stehouwer et al., 1993).

Potassium fixation is greater in soils dominated by 2:1 clays than 1:1 clays (Raney and Hoover, 1947; Seatz and Winters, 1944) because 1:1 clays generally lack expansible interlayer spaces or wedge sites. Sharpley (1990), who analyzed the fractionation of added K on 102 soils from the continental U.S. and Puerto Rico, found that resultant solution K was greater in kaolinitic soils, whereas smectitic soils had greater exchangeable K, fixed K, and K buffer capacity. The proportion of exchangeable K relative to HNO₃ extractable K was also much higher for kaolinitic soils than smectitic soils (Sharpley, 1989), indicating that kaolinitic soils have low interlayer K content and K fixation potential. Most K fixation research has focused on the clay-sized fraction; however, granitic alluvial soils of California exhibit greatest K fixation in the silt- and sand-fraction containing vermiculite and hydrobiotite (Murashkina et al., 2007a).

Role of Wetting and Drying Cycles

Potassium fixation has been shown to increase with wetting and drying cycles in pure vermiculite (Olk et al., 1995) and in vermiculitic and montmorillonitic soils (Zeng and Brown, 2000). Successive wetting and drying cycles leads to an overall decrease in solution K and exchangeable K (Zeng and Brown, 2000). In addition to potential water stress, wetting and drying cycle-induced K fixation decreased the tissue K concentration and K uptake of root, shoot, and total biomass of corn (Zeng and Brown, 2000).

Smectite is generally not considered a K fixing clay (Borchardt, 1989); however, highcharge smectite (e.g., beidellite) can fix added K following wetting and drying cycles, thus converting smectite to illite (Eberl et al., 1986; Šucha and Širáńová, 1991). Wetting and drying induces redistribution of interlayer K⁺ allowing lamellae to restructure, thus providing a mechanism for hydrated K⁺ to dehydrate for layer collapse (Gaultier and Mamy, 1979). The complete or partial dehydration of smectite layers may produce irreversibly fixed K (Środoń and Eberl, 1984). Even without wetting and drying cycles, K-saturated smectite forms mixed-layer structures with collapsed, partially expanded, and fully expanded interlayers (Čičel and Machajdík, 1981). Wetting and drying cycles may explain the formation of illite in surface soils where temperatures are much lower than those in diagenetic or hydrothermal environments (Środoń and Eberl, 1984). Eberl et al. (1986) suggested that K fixation by smectite during wetting and drying cycles may also explain accelerated K dissolution from K-feldspars in soils.

Role of Soil Organic Matter

Soil organic matter does not exhibit the same K selectivity that soil clays do. The removal of natural organic matter increases K exchange rate and K fixation, suggesting that adsorbed organic matter blocks some fixation sites (Ross, 1971). Apparent blocking of K fixation sites by organic residues has been observed following long-term farmyard manure application (Blake et al., 1999; Goulding and Talibudeen, 1984a). Higher soil organic matter under no-till compared to conventional tillage systems has also been attributed to decreasing K selectivity (Evangelou et al., 1986).

Potassium held on exchange sites of humic acid is not physically fixed and remains highly available for plant uptake, as demonstrated in corn (Jones, 1948). Zhang et al. (2013) found that humic acid coating of clay minerals increased K adsorption by kaolinite, illite, and montmorillonite; however, no significant effects of humic acid were observed in fixed K amount. Olk and Cassman (1995) discovered that the mobile humic acid fraction was able to reduce K fixation in vermiculite and increase its exchangeability and plant availability, whereas Ca-humate had no apparent effect on K availability. Cation exchange sites on soil organic matter are more accessible than those on 2:1 phyllosilicate planar and wedge sites (Jardine and Sparks, 1984b); therefore, the adsorption kinetics on organic matter exchange sites are faster than clay exchange sites (Wang and Huang, 2001).

Role of Redox Potential

Potassium, having only one oxidation state in aqueous or soil environments, is not directly affected by reduction–oxidation (redox) potential; however, redox potential may affect soil constituents with which K reacts (Lindsay, 1979). The effect of redox potential is more important for biotite and its derivative clay minerals having structural Fe than for muscovite (Sparks and Huang, 1985). The reduction of structural Fe in smectite results in interlayer collapse (Lear and Stucki, 1989; Stucki et al., 1984, 1996, 2002; Stucki and Tessier, 1991) and increased layer charge and K fixation (Chen et al., 1987). Reduction of structural Fe does not affect exchangeable K, but fixed interlayer K is positively correlated with smectite Fe(II) content (Khaled and Stucki, 1991). In contrast, illite releases K upon Fe reduction, possibly through change in electrostatic attraction between K⁺ and hydroxyl groups (Shen and Stucki, 1994).

In four montmorillonitic soils from South Dakota, a single redox event did not change the soil K fractionation or affect plant K uptake (Schindler et al., 2003). Schindler et al. (2003) presumed these soils, containing only 2 to 5% total Fe, were predominated by Mg-montmorillonite, which is less affected by redox changes. This demonstrates that not only the location of the isomorphic substitution (i.e., tetrahedral or octahedral layer) but also the substituting cation and its redox properties are important in predicting soil K dynamics.

Plant Availability of Nonexchangeable Potassium

In addition to exchangeable K, nonexchangeable K may significantly contribute to plant K uptake. Early assessments of plant available K frequently involved greenhouse experiments to classify the K supplying power of major soils through repeated cropping sequences with alfalfa (*Medicago sativa* L.), clover (*Trifolium repens* L.), millet (*Setaria italica* (L.) P. Beauv.), ryegrass (*Lolium perenne* L.), or other plant species tolerant of multiple harvests; these experiments were conducted on soils from Alabama (Gholston and Hoover, 1949), Arkansas (Legg and Beacher, 1952), Indiana (Rouse and Bertramson, 1950), Iowa (Tabatabai and Hanway, 1969), Kentucky (Sutton and Seay, 1958), Minnesota (Weber and Caldwell, 1965), Mississippi (Gholston and Hoover, 1949), New York (Chandler et al., 1945), North Dakota (Sudjadi, 1969), Ohio (Evans and Simon, 1950), Pennsylvania (Grissinger and Jeffries, 1957), Wisconsin (Attoe, 1949), and the Northern Plains (Reitemeier et al., 1950).

These successive cropping experiments across diverse soils generally indicated that plant K uptake was greater than change in exchangeable K from cropping, indicating that nonexchangeable K is releasable upon plant uptake of solution K. These results have been confirmed more recently in Australia (Moody and Bell, 2006). As soil K reserves are depleted with successive harvests, tissue K concentration and plant K uptake also decrease, indicating that nonexchangeable K release is often not sufficient for sustained plant growth upon intensive cropping.

Bray and DeTurk (1939) suggested that K fixation and release serve to protect fertilizer K from leaching and luxury consumption yet providing K release later for the growing crop. While working with two Wisconsin silt loams, Attoe (1949) found that one soil fixed 31 to 56% of applied K and the other soil fixed little K, yet fertilizer K recovery was high at 80 to 98% and 92 to 98%

for seven oat (*Avena sativa* L.) crops, respectively. Mortland et al. (1957) demonstrated that fixed K in montmorillonite and vermiculite could be easily released through cropping. The plant availability of fixed K from illite or muscovite is much lower, and their release kinetics are slower (Mortland et al., 1957). Although fixed K in vermiculite is obtainable by plants, the kinetics of adsorption and desorption on vermiculite are much slower than those for montmorillonite (Jardine and Sparks, 1984b; Mortland et al., 1957). This challenges the ability of rapid routine soil testing methods to desorb plant available K in vermiculitic soils.

Upon intensive crop K removal, soils will approach a minimum exchangeable K level (Attoe, 1949; Gholston and Hoover, 1949; Tabatabai and Hanway, 1969) that is supported by nonexchangeable K and structural K release. The minimal exchangeable K concentration has been correlated with clay content (Tabatabai and Hanway, 1969) and nonexchangeable K release rate to Ca-resin (Havlin and Westfall, 1985). Havlin and Westfall (1985) believed that the K supplying power of soils could be reasonably determined from initial exchangeable K levels.

During intensive cropping with alfalfa, NH4OAc or HNO₃ extractants (exchangeable K and nonexchangeable K methods, respectively) were not consistent predictors of residual K for subsequent crop uptake in Ohio soils (Richards and McLean, 1961). In Minnesota, Weber and Caldwell (1965) noted that correlation of NH4OAc and HNO₃ extractable K with bromegrass (*Bromus inermis* Leyss.) K uptake decreased with each successive harvest. When exchangeable K is low and plant K uptake is dependent on nonexchangeable K release, such static soil testing methods fail to account for K release kinetics that affect crop response to K. Havlin and Westfall (1985) measured the nonexchangeable K release rate of 12 calcareous Great Plains soils and found the release rate constant to Ca-resin to be linearly correlated with relative K uptake and relative yield of alfalfa.

As interlayer K is removed through cropping, net K removal increases K fixation capacity (Liu et al., 1997b; Simonsson et al., 2007, 2009; Tributh et al., 1987) and even NH₄⁺ fixation capacity (Liu et al., 1997b). Long-term addition of fertilizer K and manure K has been shown to decrease K selectivity (Goulding and Talibudeen, 1984a) and increase K release rate (Simonsson et al., 2007).

Rhizosphere Effects

Plant root absorption of K⁺ establishes a low concentration gradient near the root surface that induces diffusion to the root and release of interlayer K (Mengel and Kirkby, 2001). The release of nonexchangeable K by plant roots is directly related to the ability of plant roots to reduce rhizosphere solution K concentration (Fergus and Martin, 1974). Interlayer K release to Italian ryegrass (*Lolium multiflorum* Lam.) was observed when solution K concentration reached 80 μ mol dm⁻³ (Hinsinger and Jaillard, 1993). Root uptake can induce interlayer K release at higher rhizosphere solution K concentrations if low NH₄⁺ and high Ca²⁺ or Mg²⁺ are present to allow 2:1 interlayer expansion (Moritsuka et al., 2004; Vetterlein et al., 2013).

In corn, solution K and exchangeable K content of the rhizosphere is lower than the bulk soil (Zeng and Brown, 2000), which provides a concentration gradient for nonexchangeable K release. Rhizosphere K concentration may increase at later growth stages if mass flow to the root exceeds root K influx rate (Vetterlein and Jahn, 2004). Low soil water content will increase solution K in the bulk soil through a solution concentrating effect (Zeng and Brown, 2000); however, rhizosphere solution K and root K influx will be lower due to reduced diffusion to the root (Mackay and Barber, 1985; Seiffert et al., 1995; Zeng and Brown, 2000).

Plant roots exude organic anions and acids into the rhizosphere which serve to modify soil pH or complex ions for uptake (Mengel and Kirkby, 2001). Organic acids, such as oxalic and citric

acid, facilitate K release from K-bearing minerals (Song and Huang, 1988). Interlayer K release of micas was increased by sodium tetraphenylboron (NaBPh₄) extraction, which provides low solution K concentration for interlayer K release; however, structural K release from K-feldspars was greater with oxalic acid, which provides H⁺ for K-bearing mineral dissolution (Song and Huang, 1988). Scott and Welch (1961) demonstrated short-term corn K uptake and successive extractions using a NaCl-HCl solution continued to release nonexchangeable K whereas NaBPh₄ did not after one extraction. This suggests that H⁺ attack, in addition to low solution K, are needed to release structural K from K-feldspars. The microcline content (9 to 16%) of the silt fraction in Indiana soils was correlated (r 0.73) to HNO₃ extractable K (Phillipe and White, 1952). Using organic acids, mineral K release was: biotite > microcline = orthoclase > muscovite (Song and Huang, 1988). However, Sadusky et al. (1987) noted that H-resin released far more K from high K-feldspar sand fractions than oxalic acid did.

Role of Particle Size

The clay-size fraction is generally considered the seat of K-supplying power in soils. For eight Iowa soils, the clay fraction provided 60% of HNO₃ extractable K, even though total K content was greater in coarser particle-size fractions (Pratt, 1952). In calcareous Great Plains soils, Havlin et al. (1985) showed that the clay fraction contributed 65 to 85% of nonexchangeable K released to Ca-resin in 7000 h. The relative proportion of HNO₃ extractable K of total K increases as particle size decreases (Pratt, 1952), which is also supported by release kinetics of illite to NaBPh₄ (Cox and Joern, 1997). Soil aggregate size has a minimal effect on increased early ryegrass K uptake and dry matter yield with smaller aggregates, but there is no effect on final tissue K concentration or total K uptake (Tabatabai and Hanway, 1968).

In low exchangeable K soils, nonexchangeable K and structural K release to plants may be greater from the silt (Olsen and Shaw, 1943) or sand fractions (Parker et al., 1989a). In loessderived soils, the silt and sand fractions have been shown to release amounts of nonexchangeable K available for plant uptake that is comparable to the whole soil (Mengel et al., 1998). In Delaware Coastal Plain soils that were low in exchangeable K yet non-responsive to K fertilizer application, the availability of subsoil K and the release of nonexchangeable K, largely existing as K-feldspar in the sand fraction (Parker et al., 1989b), were attributed as significant sources of plant available K (Parker et al., 1989a). Sadusky et al. (1987) measured considerable amounts of nonexchangeable K released to H-resin from these sandy soils after 30 d. The high K supplying potential of Atlantic Coastal Plain sandy soils with low exchangeable K had been previously observed in the 1930s in Florida (Bryan, 1937). MacLean and Brydon (1963) commented that mineralogy or particle size did not address the great variability in K release to H-resin or HNO₃ for 11 diverse Canadian soils, admitting that the degree of mineral weathering and soil management may influence fixation and release characteristics of soils.

Long-term Effects of Cropping on Potassium Mineralogy

Illite and mixed-layer smectite–illite serve as the source of plant available nonexchangeable K (Barré et al., 2008b). Plant K uptake has been shown to expand clay interlayers (Doll et al., 1965) and deplete illitic material from clays (Conyers and McLean, 1968), thus modifying illite to mixed-layer smectite–illite (Tributh et al., 1987). The association between defined clay mineral classes and nonexchangeable K may be difficult to interpret in mixed mineralogical settings. Rouse and Bertramson (1950) found that HNO₃ extractable K was related to the relative intensity of the 0.99 nm X-ray diffraction (XRD) peak of fine and coarse clay fractions in Indiana soils. Mortland (1958) demonstrated that the K content of weathered biotite

could be correlated with the 1.4 nm/1.0 nm peak ratio of XRD patterns. The 1.4 nm/1.0 nm peak ratio of clay-sized phlogopite correlated well ($r^2 = 0.84$ to 0.86) with alfalfa K uptake (Naderizadeh et al., 2010). Barré et al. (2007b) applied a center of gravity concept to XRD patterns of 2:1 clay minerals showing that the weighted interlayer spacing could be related to clay K content and soil K mass balance of greenhouse pots. This technique was successfully applied to long-term field plots in France (Barré et al., 2008a).

Long-term agricultural experiments in Illinois (Velde and Peck, 2002) and Sweden (Simonsson et al., 2009) have shown that net crop K removal depletes illitic nonexchangeable K and leads to formation of K-depleted mixed-layer smectite–illite. At the University of Illinois Morrow Plots, continuous corn production decreased the K content of the clay fraction from 13.9 to 12.4 g K kg⁻¹ between 1913 and 1996 (Velde and Meunier, 2008). The addition of K fertilizer starting in 1955 restored K-depleted smectite–illite to an illitic form (Velde and Peck, 2002). In Denmark, long-term K depletion transformed vermiculite–illite (XRD peak at 1.34 nm) to vermiculite (XRD peak at 1.43 nm) (Liu et al., 1997a).

Mixed-layer smectite–illite and vermiculite–illite minerals exhibit some degree of flexibility and reversibility that allows them to serve as important buffers that provide K to plants or store fertilizer K (Barré et al., 2008b; Velde and Peck, 2002). DeMumbrum (1959) demonstrated that laboratory K removal from micas or K addition to vermiculite produced interstratified 2:1 phyllosilicates. In greenhouse experiments, Barré et al. (2007b) showed that nonexchangeable K reserves in 2:1 interlayers could be rapidly modified by net soil K addition or removal by plants. Corn roots inoculated with mycorrhizae decreased illitic material in the rhizosphere after 40 d (Paola et al., 2016). Interestingly, the rhizosphere was enriched with illitic material relative to the bulk soil after 130 d (Paola et al., 2016). Paola et al. (2016) attributed this to internal plant K

cycling that allowed K recapture from the root in the small volume of K-depleted 2:1 phyllosilicates in the rhizosphere. Increased rhizosphere solution K when mass flow exceeds plant K uptake (Vetterlein and Jahn, 2004) could also provide a mechanism for illite enrichment in the rhizosphere.

For surface soil horizons, nonexchangeable K reserves in 2:1 interlayers may be replenished through nutrient uplift by plants, which deposits K at the soil surface for its subsequent fixation (Barré et al., 2007a). Under long-term alfalfa production in China without K fertilization, exchangeable K declined over time, but the total K and illite content of surface horizons increased for 15 years via nutrient uplift before being depleted to mixed-layer smectite–illite over the following six years (Li et al., 2011). A 15 year cereal-sugar beet (*Beta vulgaris* L. spp. *vulgaris*) rotation in Germany also showed illite enrichment at the soil surface relative to the subsoil (Vetterlein et al., 2013). Long-term soil K balance difference at Rothamsted, U.K. did not produce noticeable changes in 2:1 phyllosilicate content nor agree with changes in total K content (Singh and Goulding, 1997); the lack of agreement was attributed to nutrient uplift and leaching.

For North American grassland soils, enrichment of illite in A horizons relative to C horizons has been observed in North Dakota (Redmond and Whiteside, 1967) and Saskatchewan (St. Arnaud and Mortland, 1963). The clay-size mica K content of Saskatchewan soils was also higher in A horizons (Somasiri et al., 1971). Velde (2001) found that mixed-layer smectite–illite was the most common clay mineral assemblage over a range of climates and parent materials of the central United States. Barré et al. (2009) hypothesized that nutrient uplift was responsible for illite enrichment in the surface soils of temperate grasslands soils. In forest systems, nutrient uplift has been also been identified to increase exchangeable K (Johnson et al., 2008), nonexchangeable K, and 1.0/1.4 nm peak ratio of A horizons (Tice et al., 1996).

Effect of Soil Water Content and Sample Drying on Exchangeable Potassium

Early research into K fixation showed that soils near their minimum exchangeable K level released K upon drying to air-dry water content (Attoe, 1947; Reitemeier et al., 1948). Soils with low exchangeable K have been frequently reported to have higher NH4OAc extractable K after sample drying (Grava et al., 1961; Luebs et al., 1956; Scott et al., 1957; Scott and Smith, 1957; Sutton and Seay, 1958). When Scott et al. (1957) added K to soils with low exchangeable K prior to drying, the soil would fix K. Soils with high native exchangeable K also fixed K upon drying (Cook and Hutcheson, 1960). Scott et al. (1957) proposed that K would be fixed upon drying if initial exchangeable K levels were raised above the fixation-release equilibrium between exchangeable K and nonexchangeable K forms; the fixation-release equilibrium depends on initial exchangeable K level and clay mineralogy (Dowdy and Hutcheson, 1963a). Recent investigations support that soil K dynamics depend on initial exchangeable K concentration and its fixation-release equilibrium, which differ based on soil parent material and mineralogy (Haby et al., 1988; Martins et al., 2015; Schneider, 1997).

Scott et al. (1957) observed that some soils reached a point of maximum K fixation between 2 to 6% soil water and began to release K as the sample was dried beyond that soil water content, yet other soils would continue to fix K as they dried. Dowdy and Hutcheson (1963a) revealed that vermiculite would fix K above 4% soil water and montmorillonite would fix K below 4% soil water. Illite does not fix added K upon drying but releases interlayer K (DeMumbrum and Hoover, 1958), and it serves as the source of exchangeable K increase upon drying (Dowdy and Hutcheson, 1963a). In a vermiculite-illite mixture, drying initially released K but fixed K upon remoistening; net fixation occurred with as little as 10% vermiculite (DeMumbrum and Hoover, 1958). Dowdy and Hutcheson (1963a) proposed that these release and fixation reactions could occur

simultaneously within the same mixed-layer clay mineral. Freezing and thawing may release K from montmorillonite and fix K in illite (Fine et al., 1941).

Higher drying temperatures increase K release upon drying (Burns and Barber, 1961; Haby et al., 1988) because higher drying temperatures remove more interlayer water and structural water producing clay configuration changes (Sparks and Huang, 1985). Drying temperatures above 60 °C are also likely to release additional K from residues and microbial biomass (Roberts, 1968).

The fixation and release dynamics of air-drying soil prior to cropping can increase plant K uptake in low exchangeable K soils (Attoe, 1947; Barber et al., 1961; Dowdy and Hutcheson, 1963b; Scott and Smith, 1957) or decrease plant K uptake in high exchangeable K soils (Dowdy and Hutcheson, 1963b); however, these phenomena would not affect plants under field conditions because air-dry soil water contents are far below the wilting point for plants.

Temporal Variation in Exchangeable Potassium

Spatial variation in exchangeable K across landscapes and fields has been well documented and incorporated into precision nutrient management (Franzen and Peck, 1997; Mallarino, 1996; Sawchik and Mallarino, 2007). Temporal variation in exchangeable K has also been frequently observed; however, its integration into fertilizer recommendations has been neglected. Roberts (1968) declared that fertilizer recommendations based on a single sampling time would have inherent problems due to temporal K variation at sampling. Furthermore, yield response to K fertilization may vary from year to year at the same location (Randall et al., 1997a). Exchangeable K levels can vary between years without a clear relationship to soil K mass balance (Mallarino et al., 1991a, 2011; Peck and Sullivan, 1995; Randall et al., 1997b; Vitko et al., 2009). At two longterm locations in south central Minnesota, annual variation in exchangeable K prevented calculation of exchangeable K drawdown or buildup rates (Randall et al., 1997b). Within the year, exchangeable K is generally highest in winter or spring and lowest in late summer or fall; these trends have been documented in Arkansas (Keogh and Maples, 1972), Delaware (Liebhardt and Teel, 1977), Indiana (Bell and Thornton, 1938), Illinois (Peck and Sullivan, 1995), Minnesota (Grava et al., 1961), North Dakota (Rakkar et al., 2015), Ohio (Lockman and Molloy, 1984), and Wisconsin (Vitko et al., 2009). The seasonal range in exchangeable K is greater for high K soils than low K soils; however, the coefficient of variation is greater for low K soils (Lockman and Molloy, 1984).

Rouse and Bertramson (1950) observed in Indiana that exchangeable K frequently, and occasionally HNO₃ extractable K, would increase from fall to spring. In West Virginia, exchangeable K was highest during winter months, which was attributed to freezing and thawing, and lowest in early summer, which was attributed to plant uptake, microbial immobilization, leaching, and fixation without freeze–thaw events (Childs and Jencks, 1967). In sandy Australia soils, seasonal variation in exchangeable K, being highest in autumn, was inversely related to microbial biomass K and organic matter K (Roberts, 1968); in highly leachable sands, microbial K immobilization and organic matter K serve as important K reserves.

Over a nine year soil sampling period from 1986 to 1994 in Illinois, Peck and Sullivan (1995) observed a cyclical variation in exchangeable K with low levels in late summer and high levels in midwinter, albeit great variability was observed in exchangeable K levels throughout that time. They attributed the exchangeable K increase over fall and winter to soil water content and release of interlayer K through drying and freezing. Franzen (2011), using data from Peck and Sullivan (1995), constructed a time-series unobserved components model that identified a seasonal variation component in exchangeable K whose periodicity paired with the seasonal variation component in soil water content. In Mississippi, the temporal variation in exchangeable K followed

a similar pattern between years that the authors described as a sinusoidal pattern (Oldham et al., 2013, 2015). Luebs et al. (1956) even noted a seasonal relationship between exchangeable K and the seasonal change in vapor pressure in the laboratory where their soil samples were stored.

The exchangeable K increase in October observed by Grava (1961) coincides with corn senescence, which would allow leaching of K from corn tissues into the soil. In corn and soybean, plant tissue K content decreases following physiologic maturity and initiation of plant senescence as K is leached from tissues (Oltmans and Mallarino, 2015). Potassium leaching from plant residues increases with rainfall (Oltmans and Mallarino, 2015; Rosolem et al., 2005). In Iowa, Oltmans and Mallarino (2015) found that most residue K had leached from soybean residues and about half of residue K had leached from corn residues by January. Residue K lost from corn and soybean accounted for 16 and 54% of variation between fall and spring soil test K levels in Iowa, respectively (Oltmans and Mallarino, 2015).

Mallarino et al. (2011) noted that classification of responsive, low exchangeable K soils was best in June, which would correspond with the highest exchangeable K level of the year. Mallarino et al. (2011) also observed that exchangeable K variability was lowest in June when soil samples would generally be consistently moist. Soils with high soil water content exhibit less variability upon sample drying than soils with low soil water content (Burns and Barber, 1961).

Potassium Fertilizer Application

Potassium Fertilizer Sources

Plant roots absorb K^+ from the soil solution (Mengel and Kirkby, 2001). Commercial K fertilizers are highly soluble salts that readily bear K^+ upon dissolving in soil water. Potassium fertilizer products are primarily used as dry granular fertilizers but may also be mixed into solution and suspension fertilizers (Stewart, 1985). The most common K fertilizer is muriate of potash

(KCl, 500-510 g K kg⁻¹), which comprises 95% of fertilizer K used in the United States (Stewart, 1985). Potassium chloride is the highest analysis K fertilizer and generally the cheapest K source to U.S. farmers. Sulfate of potash (K₂SO₄, 420 g K kg⁻¹) is another common K source that provides S, which may be important in areas with S deficiencies or to potato production where high residual soil Cl may be an issue. Potassium sulfate is produced by reacting KCl with H₂SO₄ or by further refining potassium magnesium sulfate salts. Both processes add additional cost per unit K for K₂SO₄ over KCl (Barber et al., 1985; Stewart, 1985). Potassium magnesium sulfate salts such as kainite (KCl•MgSO₄•3H₂O, 160 g K kg⁻¹) or langbeinite (K₂SO₄•2MgSO₄, 170 g K kg⁻¹) may be used in areas requiring Mg and S (Stewart, 1985). Potassium nitrate (KNO₃, 370 g K kg⁻¹) application is largely limited to high-value specialty crops (Stewart, 1985).

The Cl component of KCl has been recognized to alleviate Cl deficiencies in small grains (Engel et al., 1994; Fixen et al., 1986; Timm et al., 1986); however, Cl has received minimal attention in corn yield responses to KCl. Liebhardt and Munson (1976) identified that K was responsible for reducing stalking lodging in corn. Corn is classified among the least sensitive crops to Cl deficiency with a deficiency threshold of 0.05 to 0.11 mg Cl g⁻¹ in shoots (Xu et al., 2000). Nevertheless, some Cl deficiencies have been recognized in irrigated corn on sandy soils with low organic matter. Heckman (1995, 1998), working in New Jersey, reduced stalk rot incidence with KCl over K_2SO_4 ; however, grain yield increases from Cl were infrequent and usually small. Small corn grain yield increases have been occasionally observed in Kansas soils with low Cl (Lamond and Leikam, 2002). Chloride toxicity is an issue for some vegetable and fruit crops (Xu et al., 2000); however, corn is tolerant to high soil Cl levels. Parker et al. (1985) were unable to induce phytotoxicity in corn shoots containing 32.7 mg Cl g⁻¹ at the highest Cl treatment of 728 μ g g⁻¹.

Potassium Fertilizer Placement

Potassium fertilizer is often broadcast applied as dry granular fertilizers to the soil surface and incorporated with tillage (Havlin et al., 2005). Fertilizer efficiency can be improved by placing K in concentrated bands near the root zone, requiring less K to achieve the same yield as broadcast application (Welch et al., 1966). Banded fertilizer application reduces the soil volume that can fix K, thus providing higher solution K concentration for plant uptake (Mengel and Kirkby, 2001). In reduced tillage systems with limited or no soil mixing, soil nutrients may become vertically stratified and concentrated near the soil surface and unavailable to plant roots (Randall et al., 1985). To counter nutrient stratification, fertilizer may be deep-banded near a 15 cm depth.

Increased early K uptake with deep-banded K placement in no-till occasionally translated into small grain yield increases in Iowa (Mallarino et al., 1999). Deep-banded K placement increased corn grain yield about 0.3 Mg ha⁻¹ over broadcast or shallow-banded placement in no-till (Bordoli and Mallarino, 1998) and ridge till (Borges and Mallarino, 2001) systems. In Ontario, shallow-banded K rates of 42 to 50 kg K ha⁻¹ in spring increased corn grain yield in four of five sites years regardless of fall K application in no-till and strip-till (Vyn and Janovicek, 2001). If soil K levels are sufficient, banded K placement techniques rarely improve corn grain yield response over broadcast in no-till systems (Ebelhar and Varsa, 2000; Fernández and White, 2012). High CEC soils may benefit from banded K placement to provide increased solution K concentration for plant uptake (Bell et al., 2009).

The co-application of multiple fertilizers in the same fertilizer band is often logistically favorable for farmers wanting to make one fertilizer application. Co-application of anhydrous ammonia and KCl in fertilizer bands reduces K fixation and increases exchangeable K within the fertilizer band (Stehouwer and Johnson, 1991); the mechanism was previously discussed.

Concentrated monoammonium phosphate, a common P fertilizer, has been shown to enhance K release from microcline and micas, likely through mineral dissolution fostered by phosphate complexation and H⁺ attack (Zhou and Huang, 2006, 2007). Some fertilizer combinations may be antagonistic, however. In calcareous soils, the co-application of banded P and K fertilizers may decrease the diffusion and solubility of P by increasing formation of low solubility Ca-P complexes through Ca^{2+} displacement from cation exchange sites by added K⁺ (Akinremi and Cho, 1993).

Potassium Fertility Recommendations

Soil Testing: Calibration and Interpretation

Soil fertility concerns itself with soluble and easily accessible nutrients to plants (Mengel and Kirkby, 2001). Soil testing is commonly employed to assess the plant available nutrient supply of soils and the fertilizer requirements of field crops (Peck and Soltanpour, 1990). Plant available nutrients are those that will be taken up by a crop in a growing season (Cope and Evans, 1985). Soil testing methods must be correlated with nutrient uptake and calibrated with some desired crop effect, which is usually the marketable yield (Corey, 1987). A well calibrated soil test should indicate: (i) the adequacy of the soil nutrient supply for optimum yield and (ii) the quantity of fertilizer required for optimum yield if the soil nutrient supply is deficient (Cope and Evans, 1985; Eckert, 1994; Evans, 1987). The soil test level where optimum yield is achieved has been called the adequacy level, critical level, or sufficiency level. This may also be considered as the soil test level below which crop response to fertilization should be expected or above which crop response should not be expected.

The calibration process requires numerous field experiments, across a range of soils from deficient to sufficient nutrient supply, to identify the soil test level adequate for maximum economic production without fertilization (Cope and Evans, 1985; Evans, 1987). Greenhouse

experiments may be useful for comparing extractants, soils, and crops (Dahnke and Olson, 1990), but they have limited value for field application because of the differences between root exploration in pots and field conditions (Cope and Evans, 1985). As crop yield potential, production practices, and soil test levels change, soil test calibrations should be verified and updated (Evans, 1987).

Bray (1944) found that corn yield response could be related to NH₄OAc extractable K; he noted that the relationship with relative corn grain yield of unfertilized field plots was a clearer relationship than using absolute yield increase. Expressing crop response as relative yield is generally more useful than absolute yield because soil and climatic conditions, which vary by site and year, also affect yield (Evans, 1987). Bray (1944) recognized that corn yield response to K could vary by soil type, thus he calculated separate response curves for different soil types in Illinois.

Sufficiency Level versus Basic Cation Saturation Ratios

Soil testing concerns itself not only with methodology and calibration but also the interpretation of soil test results (Peck and Soltanpour, 1990). The interpretation of exchangeable K for fertilizer recommendations generally follows the sufficiency level of available nutrient (SLAN) or the basic cation saturation ratio (BCSR) concept (Eckert, 1987; Haby et al., 1990; McLean, 1977). The SLAN concept is based on a definable quantify of nutrient that can be used to predict yield response; this is the sufficiency level discussed above. The SLAN concept also provides soil test indices to indicate likelihood of response (Eckert, 1987). Since soil CEC affects the solution K concentration in relation to exchangeable K (Bell et al., 2009), some researchers have modified soil test K sufficiency levels for soil CEC (Cope and Evans, 1985; McLean, 1976).

The BCSR concept is based on an "ideal soil" with specific ratios of basic cations on cation exchange sites. This interpretation, suggesting 2 to 5% K saturation of cation exchange sites, was based on alfalfa research in New Jersey and Missouri (Kopittke and Menzies, 2007). The ideal ratios have been universally extrapolated to other crops and regions. The validity of the BCSR concept has been challenged for agronomic and economic reasons (Liebhardt, 1981; Olson et al., 1982). Soil test calibration research from the 1930s in Illinois showed that calibrations for exchangeable K should not be modified because of consideration for other exchangeable cations (e.g., Ca, Mg) or CEC (Bray, 1937). In Ohio, McLean et al. (1983) observed that corn grain yield decreased when K saturation was increased to 4.3%. In general, the BSCR concept tends to increase K fertilizer rate recommendations and cost compared to the sufficiency approach (Haby et al., 1990). Kopittke and Menzies (2007), in their review of the BCSR concept, demonstrated that soil fertility and crop productivity can be maintained over a range of cation ratios as long as the absolute amount of individual nutrients is sufficient. When relative cation percentages are multiplied by soil CEC, the BCSR concept often results in nutrient levels that are sufficient when recognized as a quantity (Eckert, 1987).

Profitability of Yield Response

As yield response approaches its maximum and yield increase per additional nutrient input decreases, economics of yield response should be considered (Evans, 1987). A profitability component has been built into current N recommendations for corn in multiple states of the North Central region, including those of the central Corn Belt (Sawyer and Nafziger, 2005) and North Dakota (Franzen, 2014). However, the profitability component has often been neglected in K recommendations. The net economic return of fertilizer K application decreases or may be negative on soils with high exchangeable K (Mallarino et al., 1991a, 1991b; Randall et al., 1997a).

Economic critical levels for soil K testing methods were calculated for corn in Iowa by Mallarino and Blackmer (1994).

Fertilizer recommendations may include build-up, maintenance, or drawdown rates to adjust soil test levels near the sufficiency level (Eckert, 1994; McLean, 1977; Olson et al., 1987). Soil testing methods provide results that are dependent on soil quantity-intensity relationships (Mengel and Kirkby, 2001); therefore, build-up recommendations should include some allowance for soil buffering capacity (Eckert, 1987). The sufficiency level itself, however, may not always be the goal of fertility management because economic return may be greatest by fertilizing for optimum yield at some soil test level below the sufficiency level (Eckert, 1987). Fertilizing for crop requirements below the sufficiency level also prevents nutrient build-up to excessive levels where leaching and erosion result in economic loss of fertilizer inputs and subsequent environmental issues (Olson et al., 1987). Potassium, unlike N and P (Gilliam et al., 1985), does not have specific environmental issues relating to water quality, but its loss from soils maintained at high soil K levels increases economic losses and accelerates depletion of geologic mineral K reserves.

Soil Testing: Soil Potassium Testing Methods

Soil K tests are designed to easily and rapidly assess plant available K, which has usually involved the estimation of solution and exchangeable K (Helmke and Sparks, 1996; Warnacke and Brown, 2012). Soil testing for exchangeable K is affected by extraction method, soil water content, sample drying, date of soil sampling, and antecedent K additions (Haby et al., 1990). Some of these factors have been previously discussed in other sections; yet, some aspects pertinent to fertilizer recommendations require further discussion.

Ammonium Acetate

The recommended method for assessing plant available K in the North Central region of the United States employs 1 M NH₄OAc (pH 7.0) extraction on air-dry or oven-dry soil with drying temperatures less than 40 °C (Gelderman and Mallarino, 2012; Warnacke and Brown, 2012). Volk and Truog (1934) found that soil extracted with 1 M NH₄OAc (pH 6.8) for 5 min was able to extract 96% of total exchangeable K extracted through leaching with NH₄OAc; the 5-min extraction with 1 M NH₄OAc was a superior extractor of exchangeable K than other salt solutions or dilute acids. Hanway and Scott (1959) confirmed that NH₄OAc extracted more K from moist or dried samples than other common extraction solutions of that time (i.e., dilute HCl and Morgan's solution). For corn, NH₄OAc extractable K and tissue K concentration of young plants and ear leaves at R1 growth stage often correlate well and provide exchangeable K critical levels for sufficient tissue K concentration (Mallarino et al., 1991b; Mallarino and Higashi, 2009; Schindler et al., 2002; Woodruff and Parks, 1980).

Early soil test calibrations for corn established the exchangeable K critical level for 90% relative corn grain yield at 90 mg K kg⁻¹ in Illinois (Bray, 1944) and 72.5 mg K kg⁻¹ in Tennessee (Winters, 1946). More recent soil test calibrations for NH₄OAc extractable K on air-dry soil have frequently experienced weak correlations with corn yield response; exchangeable K critical levels were calculated at 95 mg K kg⁻¹ in Pennsylvania (Beegle and Oravec, 1990) and 82 to 123 mg K kg⁻¹ (varied by regression model) in Iowa (Mallarino and Blackmer, 1994). No relationship between exchangeable K and corn yield response could be established in Minnesota (Randall et al., 1997b). In irrigated high-yield corn production in Nebraska, exchangeable K only accounted for 11% of variation in corn K uptake (Wortmann et al., 2009), although Nebraska soils have been

noted to contain substantial amounts of K-feldspar (Sautter, 1964) and illite (Abed and Drew, 1966) that may release K upon cropping.

Boiling Nitric Acid

Boiling HNO₃ has been a standard method to assess nonexchangeable K (Helmke and Sparks, 1996; Martin and Sparks, 1985). Strong acids such as HNO₃ can release more K than H-resin, suggesting that some structural K is released by the method as well (Martin and Sparks, 1983). Boiling HNO₃ has been used to estimate plant K uptake (Reitemeier et al., 1948) and often correlates with plant K uptake better than exchangeable K (Garman, 1957; Rouse and Bertramson, 1950). Mallarino and Blackmer (1994) found that HNO₃ predicted corn grain yield response better than NH₄OAc. Nevertheless, others have found that plant K uptake is better correlated with exchangeable K than HNO₃ extractable K (Weber and Caldwell, 1965).

Sodium Tetraphenylboron

In soils where nonexchangeable K is a significant source of plant available K (e.g., illitic or vermiculitic soils), the standard NH₄OAc extraction method has often been an inadequate means to assess plant available K (Cox et al., 1999; Rouse and Bertramson, 1950). Sodium tetraphenylboron extraction can remove interlayer K from 2:1 phyllosilicates (Cox et al., 1996). Sodium tetraphenylboron solution precipitates K⁺ from the soil solution, thus allowing interlayer expansion and K release from micas and illite (Cox and Joern, 1997). A 5-min extraction by NaBPh₄ was identified as a superior predictor of wheat K uptake compared to NH₄OAc in illitic soils from Indiana (Cox et al., 1999).

Extraction times for the NaBPh₄ method of Cox et al. (1996) have been adjusted in attempts to better assess plant available nonexchangeable K. Extraction times have ranged from 1 to 15 min for readily plant available K (Bilias and Barbayiannis, 2016; Cox et al., 1999; Wang et al., 2010),

30 to 60 min for slowly plant available nonexchangeable K (Carey et al., 2011; Murashkina et al., 2007b; Wang et al., 2010), and 4 to 16 h for reserve nonexchangeable K (Carey et al., 2011). Wang et al. (2010), who varied the NaCl concentration of the NaBPh₄ extraction solution, showed that NaCl as an additional ion-exchanger greatly increased extractable K; however, NaBPh₄ extraction without NaCl, where K release would be controlled mainly by solution K concentration, produced better correlations with ryegrass leaf K concentration. Sodium tetraphenylboron had no advantage over NH₄OAc in predicting corn leaf K concentration of montmorillonitic soils from South Dakota because these montmorillonitic soils with average 17% tetrahedral layer charge had low K fixation potential (Schindler et al., 2002).

In greenhouse experiments, the relationship between soil K mass balance and NH₄OAc extractable K varied greatly across nine Indiana soils, but the slopes of the relationship between soil K mass balance and NaBPh₄ extractable K were consistent across soils (Cox et al., 1999). Wang et al. (2010) found that the 60-min NaBPh₄ extraction without NaCl was an effective extractant across four Chinese soil types of different K fixation and nonexchangeable K release characteristics, whereas NH₄OAc was only effective for relating K availability within soil types. In California soils, the relationship between nonexchangeable K extracted using NaBPh₄ was correlated with K fixation potential for individual soils, but the relationship was not consistent across soils (Murashkina et al., 2007b).

Ion-exchange Resins

Skogley (1994) criticized chemical extractions methods for not being mechanistically related to plant uptake of nutrients and offered ion-exchange resins as an alternative. Ion-exchange resins mimic plant uptake in their being dependent on solution K activity and rate of diffusion (Schaff and Skogley, 1982; Skogley and Schaff, 1985). Resins have high CEC that provides a sink

for solution K and facilitate nonexchangeable K release (Helmke and Sparks, 1996). Ion-exchange resins have been calibrated for crops in Brazil where exchangeable K is low and 1:1 clays predominate (Cantarella et al., 1998). Potassium exchange reactions on 1:1 clays are much faster than those on 2:1 clays, especially vermiculite (Jardine and Sparks, 1984b). Potassium release to Ca-resin is rapid in kaolinitic soils, but it proceeds slowly in smectitic soils, which may not reach equilibrium even after 25 d (Nilawonk et al., 2008).

A mixed-bed, ion-exchange resin capsule was developed by researchers in Montana to extract cationic and anionic nutrients simultaneously from soil (Skogley, 1992; Yang et al., 1991). Skogley (1994) showed that corn K uptake correlation with mixed-bed resin K was slightly better $(r^2 = 0.28)$ than NH₄OAc $(r^2 = 0.24)$; however, neither method was a strong predictor of corn K uptake. Sherrod et al. (2003) remarked that soil chemical properties (e.g., pH, carbonates) and resin counter-ion (e.g., H⁺, HCO₃⁻) may affect the soil-resin environment and nutrient extraction. Hydrogen-resin extractable K exhibited a relationship with cumulative K uptake (r = 0.79) in Pakistan soils where cumulative plant K uptake had been correlated with soil mica content (r = 0.71) (Rahmatullah and Mengel, 2000). Overall, soil test calibration research for resin K methods is lacking for soils with 2:1 clays.

Soil Testing: Sample Handling

Prior to laboratory analysis, soil samples are usually dried and ground for convenience of handling and to facilitate better soil mixing to produce a homogenous mixture for subsampling (Gelderman and Mallarino, 2012). However, it has been known for decades that soil sample drying changes the quantity of extractable K from the field condition (previously discussed). Recent studies continue to demonstrate that soil sample drying increases NH₄OAc extractable K (Barbagelata and Mallarino, 2013; Martins et al., 2015; Vitko et al., 2009). Exchangeable K is

even sensitive to changes to relative humidity in the air (Attoe, 1947), which may change during the year in the area where samples are stored (Luebs et al., 1956). The relationship between NH₄OAc extractable K on air-dry and field-moist soil samples is often not predictable because it varies with clay content, clay mineralogy, soil water content, and exchangeable K level (Barbagelata and Mallarino, 2013; Rakkar et al., 2015; Vitko et al., 2009). Early research showed that exchangeable K from field-moist soil correlated better than air-dry exchangeable K with corn K uptake (Luebs et al., 1956) and millet K uptake (Sutton and Seay, 1958).

Multistate North Central regional greenhouse studies with millet showed that field-moist exchangeable K correlated better than air-dry exchangeable K with K uptake, tissue K concentration, and relative grain yield (Barber et al., 1961). Barber et al. (1961) commented that relative grain yield was related to soil K supply on soils below 100 mg kg⁻¹ field-moist exchangeable K, which was the exchangeable K critical level for millet in his experiments. These results were corroborated in North Central regional field studies for alfalfa (Hanway et al., 1961) and corn (Hanway et al., 1962); these studies also found that subsoil K improved regression for alfalfa and corn. A critical reading of the corn study by Hanway et al. (1962) reveals that they used multiple regression involving subsoil K, fertilizer K, soil texture, and plant population to predict corn yield and yield increase; they did not examine relative grain yield response as Bray (1944) suggested. Depending on the inclusion of subsoil K, the advantage of field-moist soil over air-dry soil was apparent or nearly disappeared (Table 3). The relationship was not consistent for predicting both grain yield and yield increase, however. Without inclusion of subsoil K, exchangeable K from field-moist soil analysis was moderately superior in predicting grain yield increase.

Table 3. Effect of soil sample drying on exchangeable K and inclusion of subsoil K on degree of correlation for multiple regression models predicting corn grain yield and yield increase to K in the North Central regional K studies on corn.[†]

		\mathbb{R}^2		
Dependent variable	Inclusion of subsoil K	Field moist	Air dry	
Grain yield	Yes	0.62	0.53	
	No	0.35	0.34	
Yield increase	Yes	0.42	0.39	
	No	0.32	0.24	

[†] Adapted from Hanway et al. (1962)

Although correlations between NH₄OAc extractable K and yield were often stronger using field-moist soil, the procedure was not widely adopted because of the difficulties of handling moist soil samples and limited calibration data on extraction of K from moist soil samples (Gelderman and Mallarino, 2012); the Iowa State University Soil Testing Laboratory abandoned the method in 1989. Barbagelata and Mallarino (2013) recalibrated the 1 M NH₄OAc method for corn and soybean in Iowa and found that K extracted from field-moist soil ($r^2 = 0.39$ to 0.58) correlated better with grain yield response to K fertilization than K extracted from oven-dry soil ($r^2 = 0.24$ to 0.27). The critical levels for corn using the moist K test were lower (51 to 82 mg K kg⁻¹) than the dry K test (144 to 301 mg K kg⁻¹); the range of critical levels encompasses differences between regression models. Hanway et al. (1962) noted that no significant corn grain yield increases occurred above 80 mg K kg⁻¹ using field-moist soil. Recently, Iowa State University has reinstituted K recommendations for corn and soybean using the moist K test (Mallarino et al., 2013). Evaluation of the air-dry and field-moist NH₄OAc K methods in North Dakota found neither method to be strong predictors of corn yield response (Rakkar et al., 2015).

RECALIBRATION OF SOIL POTASSIUM TEST FOR CORN IN NORTH DAKOTA Abstract

Potassium fertilizer recommendations for corn (Zea mays, L.) are commonly guided by yield response calibrations to soil test K. The standard soil test K method for the North Central region employs 1 M NH₄OAc (pH 7) on air-dry or oven-dry soil, but sample drying affects the amount of extractable K. In addition, yield responses to K fertilization may be inconsistent on low and high K soils. Potassium rate trials were established in southeastern North Dakota to evaluate soil K test methods and their relationship with corn yield response to K fertilization. Soil test methods evaluated were NH₄OAc on air-dry and field-moist soil, sodium tetraphenylboron, and a mixed-bed ion-exchange resin. Soil sample drying increased NH₄OAc extractable K by a factor of 1.27 on average (range 0.7 to 2.8), which varied by soil test K level and site. NH₄OAc extractable K from air-dry soil (DK) was the best predictor of relative grain yield response ($r^2 = 0.45$ to 0.53) with soil test K critical levels ranging from 107 to 149 mg K kg⁻¹, depending on regression model. Nevertheless, DK only predicted significant grain yield responses at 10 of 19 sites. Corn tissue K concentration was related to soil test K ($r^2 = 0.56$ to 0.84), but tissue K concentration exhibited no relationship with grain yield response. Potassium-feldspar, muscovite, and illite content had mixed relationships with relative yield response and did not improve weak relationships between soil test K and yield response.

Introduction

Fertilizer recommendations are commonly guided by yield response calibrations to soil test levels of a particular nutrient. The present K fertilizer recommendations for corn (*Zea mays* L.) in North Dakota were developed in the late 1970s and early 1980s (Dahnke and Vasey, 1981; Wagner et al., 1977) when soil test K levels were very high across much of the state (Dahnke et al., 1982, 1992b) and native soil K fertility was sufficient for most crop K requirements with the exception of particularly sandy soils (Zubriski and Moraghan, 1983). In 1980, 3% of soil tests in North Dakota indicated medium or lower soil test K levels (<130 mg kg⁻¹) (Nelson, 1979). Decades of net soil K removal through grain export without maintenance K fertilizer applications (Cassel et al., 1973; Dahnke et al., 1986; IPNI, 2012) has increased the number of low testing K soils in North Dakota. In 2010 and 2015, 17 and 16% of soil tests in North Dakota, respectively, had exchangeable K levels below the current 160 mg K kg⁻¹ critical level (Fixen et al., 2010; IPNI, 2016).

The standard soil K test method for soils in the North Central region of the United States employs a neutral ammonium acetate extraction solution (1.0 M NH₄OAc at pH 7.0) on soil that has been air-dried or oven-dried at temperatures less than 40 °C (Warnacke and Brown, 2012). This method, however, has come under scrutiny because soil sample drying prior to laboratory analysis (Barbagelata and Mallarino, 2013) and date of soil sampling (Franzen, 2011; Vitko et al., 2009) have been shown to affect soil test K results. Moreover, yield responses to K fertilizer application may be inconsistent on both low and high soil test K soils (Rakkar et al., 2015).

For decades, soil sample drying has been shown to increase NH₄OAC extractable K in low K soils (Attoe, 1947; Grava et al., 1961; Luebs et al., 1956; Scott and Smith, 1957). Soil sample drying may also fix K and decrease NH₄OAC extractable K in high K soils (Cook and Hutcheson, 1960; Scott et al., 1957). The relationship between exchangeable K from air-dry and field-moist soil samples is often not predictable and varies with clay content, clay mineralogy, soil water content, and soil test K level (Barbagelata and Mallarino, 2013; Martins et al., 2015; Vitko et al., 2009). In Iowa, NH₄OAC extractable K from field-moist soil exhibited a superior predictive relationship to corn yield response to K fertilization than using air-dry soil (Barbagelata and

Mallarino, 2013). However, neither NH₄OAC extractable K from air-dry soil nor field-moist soil was shown to be an adequate predictor of corn yield response to K fertilization in North Dakota (Rakkar et al., 2015).

In soils where nonexchangeable K is a significant source of plant available K (e.g., illitic or vermiculitic soils), NH₄OAc may provide an inadequate assessment of plant available K (Cox et al., 1999; Rouse and Bertramson, 1950). Sodium tetraphenylboron (NaBPh₄) extractant, which has the ability to remove nonexchangeable K from 2:1 phyllosilicates (Cox et al., 1996), was identified as a superior predictor of wheat K uptake over NH₄OAc in illitic soils from Indiana (Cox et al., 1999). Soils of eastern North Dakota are generally dominated by smectitic or mixed mineralogy (USDA-NRCS, 2006); however, some soils with mixed mineralogy contain appreciable illite (Franzen, 2015) where NaBPh₄ extraction may be justified. Ion-exchange resins, which have high CEC to serve as a sink for solution K to facilitate nonexchangeable K release (Helmke and Sparks, 1996), have been explored for their ability to mimic plant K uptake in being dependent on solution K activity and rate of diffusion (Schaff and Skogley, 1982; Skogley and Schaff, 1985). Ion-exchange resins have been calibrated for crops in low K, 1:1 clay soils (Cantarella et al., 1998); however, calibration data is lacking for resins on soils containing 2:1 clays, whose K exchange kinetics are much slower than 1:1 clays (Jardine and Sparks, 1984b; Nilawonk et al., 2008).

The objective of this study was to revise the K fertilizer recommendation for modern corn production in North Dakota through: (i) evaluation of the NH₄OAc extraction method on air-dry and field-moist soil as well as novel soil tests including NaBPh₄ and an ion-exchange resin and (ii) assessment of soil test K relationship with yield response to establish soil test critical levels.

Methods and Materials

Field Study

On-farm fertilizer K rate trials were established in southeastern North Dakota at thirteen locations in 2015 and six locations in 2016 (Table 4); this region of North Dakota has the highest concentration of corn production and the greatest range of soil test K levels for evaluating crop response to K fertilization in the state. The experiments were organized as randomized complete block design with four replications and six K rate treatments on corn. The experiment unit size was 3.0 m by 9.1 m. The K treatments were 0, 28, 56, 84, 112, and 140 kg K ha⁻¹ applied as fertilizer-grade KCl (500 g K kg⁻¹) granules hand-broadcasted prior to planting. All sites received uniform broadcast applications of N as Limus-treated urea (460 g N kg⁻¹) (BASF Corporation, Research Triangle Park, North Carolina, USA) and P as fertilizer-grade monoammonium phosphate (110 g N kg⁻¹, 230 g P kg⁻¹) following the North Dakota corn fertility recommendations (Franzen, 2014) as well as 112 kg ha⁻¹ pelletized gypsum (180 g S kg⁻¹) to reduce non-K nutrient deficiencies in the trials. Fertilizer treatments were shallowly incorporated (5-8 cm) by farmercooperators, except on no-till sites. Farmer-cooperators chose corn hybrid, planted corn, and applied herbicides and other inputs on experiment areas when they conducted those activities on the rest of the field (Table 5). Initial soil samples were collected from non-fertilized check and fallow plots from 0- to 15-cm and 15- to 30-cm depths and thereafter collected biweekly until harvest from the 0- to 15-cm depth only. Six soil cores were taken from each plot at each sampling date and composited for analysis. Soil samples (0-60 cm) were collected from check plots in late June to assess background soil Cl level; the soil Cl test has not been calibrated for corn in North Dakota (Franzen, 2016). All sites had at least 28 kg Cl ha⁻¹ (equal to 28 kg K ha⁻¹ rate of KCl),

except site LS15 with 1.0 kg Cl ha⁻¹. Check plots at site LS15 received 28 kg Cl ha⁻¹ as CaCl₂ equal to the lowest KCl rate.

After physiologic maturity, corn grain was hand-harvested as whole corn ears from one interior 9.1-m row, shelled using an Almaco corn sheller (Almaco, Nevada, Iowa), weighed for grain yield, and measured for grain moisture and test weight using a GAC 500 XY moisture tester (Dickey-John Corp., Auburn, Illinois). Grain yield was corrected to 145 g kg⁻¹ grain moisture.

In 2016 only, plant samples were collected for tissue K analysis at growth stages V5-V6 and VT (Ritchie et al., 1996). Whole plants were collected at growth stage V5-V6, and ear leaves were collected at growth stage VT. Six plants were sampled each time from an interior row not intended for grain harvest.

			Soil classification†		
Site	Latitude (N)	Longitude (W)	Series	Particle-size	Great Group
AB15	46°59'31.00"	97°22'51.85"	Gilby	Fine-loamy	Aeric Calciaquoll
AR15	47°06'40.61"	97°16'50.50"	Glyndon	Coarse-silty	Aeric Calciaquoll
B15	46°14'27.40"	96°59'28.95"	Glyndon	Coarse-silty	Aeric Calciaquoll
C15	45°57'03.79"	96°46'34.73"	Mantador	Coarse-loamy	Aquic Pachic Hapludoll
D15	46°18'27.26"	96°49'09.81"	Gardena	Coarse-silty	Pachic Hapludoll
F115	46°00'30.67"	96°37'49.39"	Doran	Fine	Aquertic Argiudoll
F215	45°58'43.94"	96°41'49.91"	Gilby	Fine-loamy	Aeric Calciaquoll
LN15	46°42'05.14"	97°15'58.11"	Kindred	Fine-silty	Typic Endoaquoll
LS15	46°40'41.01"	97°17'05.70"	Hecla	Sandy	Oxyaquic Hapludoll
M15	46°16'18.93"	97°28'41.11"	Arveson	Coarse-loamy	Typic Calciaquoll
P15	47°01'39.36"	97°06'49.55"	Kindred	Fine-silty	Typic Endoaquoll
V15	46°50'03.61"	97°55'04.04"	Embden	Coarse-loamy	Pachic Hapludoll
W15	46°30'32.43"	97°00'42.32"	Garborg	Sandy	Typic Endoaquoll
AK16	46°58'42.62"	97°25'18.65"	Glyndon	Coarse-silty	Aeric Calciaquoll
CF16	46°23'55.57"	96°55'28.32"	Mantador	Coarse-loamy	Aquic Pachic Hapludoll
GD16	47°09'57.62"	97°03'04.40"	Galchutt	Fine	Vertic Argialboll
LB16	46°27'51.33"	97°10'37.17"	Garborg	Sandy	Typic Endoaquoll
MO16	46°18'13.28"	96°51'34.23"	Wyndmere	Coarse-loamy	Aeric Calciaquoll
VC16	46°53'26.12"	97°55'00.18"	Swenoda	Coarse-loamy	Pachic Hapludoll

 Table 4.
 Locations, soil types, and soil characterization for nineteen field trials.

† U.S. Soil Taxonomy (Soil Survey Staff, 1999, 2014).

Site	Year	Prev. crop	Tillage†	Planting date	Hybrid‡	Population §
						plants ha ⁻¹
AB15	2015	Wheat	CT	24 Apr.	PF 92-G84	71566
AR15	2015	Soybean	CT	25 Apr.	D 36-30	68142
B15	2015	Dry bean	ST	18 Apr.	C 3899	73164
C15	2015	Soybean	CT	1 May.	R 4B953	74120
D15	2015	Soybean	CT	18 Apr.	C 3899	70175
F115	2015	Corn	CT	26 Apr.	GC 95/33	67963
F215	2015	Soybean	CT	2 May	P 9917RR	77109
LN15	2015	Soybean	CT	1 June	P 9366	78413
LS15	2015	Soybean	CTnw	5 May	P 9366	64800
M15	2015	Soybean	CT	26 Apr.	NS 9505	83564
P15	2015	Soybean	CT	28 Apr.	P 9284R	73642
V15	2015	Wheat	NT	29 Apr.	NK N17P-3110	41662
W15	2015	Soybean	CT	29 Apr.	PF 76S92	79380
AK16	2016	Wheat	CT	3 May	PF 92G84	76945
CF16	2016	Corn	CT	22 Apr.	D 48-12	82368
GD16	2016	Soybean	CT	30 Apr.	NT 5B782	55530
LB16	2016	Soybean	CT	23 Apr.	P 9284R-NM61	74778
MO16	2016	Soybean	CT	22 Apr.	P 9703AM	84700
VC16	2016	Wheat	NT	5 May	NK N17P-3000	50210

Table 5. Agronomic information for nineteen field trials.

[†] CT, conventional till; CTnw, conventional till but not worked; NT, no-till; ST, strip-till.

‡ C, Cropland; D, Dekalb; GC, Gold Country; NK, NK; NS, Nuseed; NT, NuTech; P,

Pioneer; PF, Peterson Farms; R, REA.

§ Average population at harvest.

Soil and Tissue Analysis

Soil samples from non-fertilized check and fallow plots were hand-homogenized and split into two subsamples. One subsample was air-dried following the procedure suggested for the North Central Region of the U.S. (Gelderman and Mallarino, 2012), ground to pass through a 2mm sieve, and analyzed for NH₄OAc extractable K (Warnacke and Brown, 2012). A 2 g air-dry soil sample was weighed and extracted with 20 mL 1 M NH₄OAc at pH 7.0 and shaken for 5 min in 50-mL Erlenmeyer flasks. The extract was filtered through Whatman No. 2 filter paper (GE Healthcare Bio-Sciences, Pittsburgh, Pennsylvania) and analyzed for extractable K by atomic absorption spectroscopy (AAS). The other subsample was kept at field-moisture, stored in a plastic re-closeable bag, and refrigerated at 4 °C until analysis. The field-moist sample was prepared following the direct sieving procedure described by Gelderman and Mallarino (2012). Field-moist soil was passed through a 2-mm sieve, and soil water content was immediately determined by drying a 6 g moist subsample to air-dryness and constant weight. A 2 g air-dry equivalent mass of field-moist soil was weighed and extracted with 20 mL 1 M NH₄OAc at pH 7.0, thereafter, following the same analysis procedure described for the air-dry method.

Sodium tetraphenylboron extraction, following the procedure of Cox et al. (1999), was used to determine the most reactive and total nonexchangeable K fractions using 5-min and 168-h extraction times, respectively. A 0.5 g air-dry soil sample was weighed into a 50-mL Folin-Wu digestion tube with 3 mL extraction solution (0.2 M NaBPh₄ + 1.7 M NaCl + 0.01 M EDTA). After the extraction period (5-min or 168-h), 25 mL of quenching solution (0.5 M NH₄Cl + 0.11 M CuCl₂) was added to arrest K extraction. Digestion tubes were placed in digestion block at 150 °C until precipitate dissolved completely (30 to 60 min). The suspension was diluted to 50 mL with deionized water, mixed, and allowed to settle. A 20-mL aliquot of supernatant was transferred to a 50-mL centrifuge tube and acidified with three drops of 6 M HCl to prevent Cu²⁺ precipitation. The extract was diluted with deionized water (1:10) and analyzed for NaBPh₄ extractable K by AAS.

Resin extractable K was determined using a mixed-bed, cation- and anion-exchange resin capsule (UNIBEST Inc., Walla Walla, WA) based on the design of Skogley (1992). A 30 g air-dry equivalent mass of 2-mm sieved, field-moist soil was weighed and incubated with a resin capsule and 30 mL deionized water for 168 h at constant 20 °C. After the incubation period, the resin

capsule was washed with deionized water to remove attached soil and leached with 50 mL 2 M HCl using a manual slow-drip leaching apparatus (UNIBEST Inc., Walla Walla, WA). The resin capsule leachate was analyzed for resin extractable K using AAS.

The estimated cation exchange capacity (ECEC) was determined by summation of extractable cations (Ca, Mg, K, Na, and neutralizable soil acidity) using 1 M NH₄OAc at pH 7.0 on air-dry soil samples (Warnacke and Brown, 2012). Semi-quantitative mineral identification and clay speciation was conducted by Activation Laboratories Ltd. (Ancaster, Ontario, Canada) using X-ray diffraction and quantified using the Rietveld method on a composite field-moist soil sample from each site; their diagnostic procedure did not distinguish between smectite species (Borchardt, 1989). Soil texture was determined by the hydrometer method (Gee and Or, 2002).

Plant samples were dried at 65°C to constant weight, ground, and analyzed for tissue K using acetic acid extraction (Raun et al., 1987; Franzen and Peck, 1997). A 0.20 g tissue sample was extracted with 2% v/v acetic acid and shaken for 5 min in 100-mL centrifuge tubes. The extract was filtered through Whatman No. 2 filter paper and analyzed for tissue K by AAS.

Data Analysis

Data were analyzed for individual site years using SAS version 9.4 (SAS Institute, 2013) to determine if yield responses to K fertilizer were significant using PROC GLM. Relative grain yield was calculated by dividing the average yield of the 0 kg K ha⁻¹ treatment by the maximum average treatment yield at each experiment site. Relative grain yield of the unfertilized treatment and extractable K by various soil test K methods were regressed to fit linear-plateau, quadratic-plateau, quadratic, and exponential models using PROC NLIN. If the model fit was significant (p < 0.10), soil test K critical levels were calculated at 90, 95, 99, and 100% of the maximum relative yield predicted by each model. Mineralogical analyses were related to soil test K and yield
response using PROC PRINCOMP to identify principal components between yield response and mineralogical variables. Response sites were also partitioned by smectite/illite ratio of their claysized fraction using PROC CLUSTER with Ward's minimum variance method, and significance of clusters were verified with PROC GLM. Since the variance of variables may be unequal, variables were standardized to a Z-score using PROC STANDARD prior to cluster analysis. Relative grain yield and NH₄OAc extractable K from air-dry soil were regressed using PROC NLIN for significantly clustered sites.

Results and Discussion

Comparison of Soil Potassium Testing Methods

Soil K levels of 19 K trials in southeastern North Dakota exhibited a wide range from 69 to 444 mg kg⁻¹ NH₄OAC extractable K from air-dry soil (DK) in late May (Table 6). Sodium tetraphenylboron extractable K (TBK) for the 5-min and 168-h extraction periods was considerably greater than either DK or NH₄OAC extractable K from field-moist soil (MK), indicating considerable reserves of nonexchangeable K in most soils (Table 6). Resin extractable K was lower than other methods at all sites. The percent K saturation across sites ranged from 0.7 to 4.6%, thus including the 2 to 5% K saturation range suggested by promoters of the basic cation saturation concept for adequate K nutrition (Kopittke and Menzies, 2007).

The correlation between DK and MK was strong across sites and sampling dates ($r^2 = 0.90$, Table 7). The 5-min TBK extraction was strongly correlated with DK ($r^2 = 0.88$) and MK ($r^2 = 0.80$). The strong correlations of 5-min TBK with DK and MK suggest that the 5-min TBK extraction removed mostly exchangeable K and nonexchangeable K in close proximity to the exchangeable phase. The 168-h TBK extraction was moderately correlated with exchangeable K methods (Table 7). Percent K saturation was moderately correlated with DK and MK, poorly

correlated with 168-h TBK, and negatively correlated with ECEC ($r^2 = 0.17$, p = 0.01, n = 37). Resin extractable K exhibited a weak relationships with 5-min and 168-h TBK (Table 7). Although the resin and TBK extraction methods are employed for nonexchangeable K assessment (Cox et al., 1996; Helmke and Sparks, 1996), the methods clearly operate by different mechanisms and extract non-proportional amounts exchangeable and nonexchangeable K. Resin extractable K was positively correlated with K saturation; no significant negative correlation was recognized with ECEC ($r^2 = 0.11$, p = 0.16, n = 19).

Site	DK†	MK	TBK	TBK	RK	K sat.	ECEC
			5 min	168 h			
			- mg kg ⁻¹			%	cmol _c kg ⁻¹
AB15	108	102	235	713	17.2	2.2	15.5
AR15	90	84	282	1255	8.3	1.0	33.8
B15	160	199	384	2456	22.1	1.6	31.5
C15	87	103	284	1206	13.0	2.1	20.1
D15	109	101	249	1293	12.3	1.4	28.4
F115	169	143	497	2607	7.8	1.8	31.7
F215	100	82	250	1157	7.2	0.9	36.3
LN15‡	444	428	1146	3475	79.5	4.1	29.1
LS15	198	201	257	709	87.1	4.6	9.5
M15	134	129	284	1160	13.8	1.4	38.2
P15	163	143	548	2637	13.7	2.1	28.8
V15	203	235	551	2195	25.3	2.8	17.1
W15	111	135	223	707	21.7	2.4	11.5
AK16	163	115	366	1898	14.0	1.4	30.6
CF16	84	56	166	797	12.0	0.7	30.5
GD16	84	55	225	1140	5.8	1.3	16.8
LB16	69	45	160	689	12.0	0.8	23.0
MO16	114	69	265	1221	12.7	1.6	18.9
VC16	201	171	504	2515	13.7	2.2	24.1

Table 6.Soil test K values at nineteen trials sampled in late May by various soil testmethods.

[†] DK and MK, NH₄OAc extractable K on air-dry and field-moist soil, respectively; TBK, tetraphenylboron extractable K; RK, resin extractable K; K sat., K saturation of ECEC; ECEC, estimated cation exchange capacity.

‡ Sampled in early June.

		Coefficient of correlation (r ²)					
		DK	MK	TBK	TBK	K sat.	RK
Method [†]	Range‡			5 min	168 h		
n§		1304	1304	168	168	37	19
DK	39 - 612	1.00	0.90***	0.88***	0.56***	0.55***	0.46**
МК	22 - 607		1.00	0.80***	0.49***	0.61***	0.50***
TBK 5 min	127 - 1203			1.00	0.77***	0.42***	0.24*
TBK 168 h	663 - 3578				1.00	0.20**	0.03
K sat.	0.55 - 4.58					1.00	0.80***
RK	5.84 - 87.1						1.00

Table 7. Correlation of various soil test K methods.

*, **, *** Significant at the 0.05, 0.01, and 0.001 probability level, respectively.

[†] DK and MK, NH₄OAc extractable K on air-dry and field-moist soil, respectively; TBK, tetraphenylboron extractable K; RK, resin extractable K; K sat., K saturation.

[‡] Units: mg kg⁻¹ for DK, MK, TBK 5 min, TBK 168 h, RK; % for K saturation.

§ Number of samples per comparison.

Effect of Soil Sample Drying on Exchangeable Potassium

Sample drying increased NH₄OAc extractable K by 1.27 times on average (p < 0.01, n = 1365, range 0.69 to 2.75), showing that sample drying causes an overestimation of exchangeable K from field-moist soil conditions. The correlation between DK and MK was strong ($r^2 = 0.90$, Table 7, Figure 4); however, MK was generally lower than DK, particularly for high K soils (Figure 4). The absolute difference between DK and MK expressed no clear relationship with soil test K level across sites (Figure 5A), but the relative difference (DK/MK ratio) across sites followed an exponential decay relationship ($r^2 = 0.36$) showing more K being released by drying for low K soils (Figure 5B). The obverse scenario of regressing the MK/DK ratio against DK did not exhibit any significant relationship, confirming that MK is the independent factor in the sample drying effect.

Although the correlation between DK and MK was strong across sites ($r^2 = 0.90$, Table 7) because of the wide range of soil test K values, the correlation between DK and MK for individual

sites ranged from weak to strong ($r^2 = 0.31$ to 0.84, Table 8). The absolute and relative differences between DK and MK were more strongly related to soil test K level for individual sites than across all sites (Table 8), which suggests that the soil-specific properties that control K release and fixation dynamics upon drying (e.g., soil water content, texture, mineralogy, soil test K) are not consistent across sites. For individual sites, the DK/MK ratio exhibited a stronger relationship than the absolute K difference with MK (Table 8), which indicates that the magnitude of K release upon drying appears to be scaled to soil test K level. The DK/MK ratios for individual sites followed exponential decay relationships, except for sites LS15 and VC16 that exhibited weak liner relationships between the DK/MK ratio and MK level (Table 8).



Figure 4. Relationship between NH4OAc extractable K from air-dry (DK) and field-moist (MK) soil across all trials.

The absolute difference between DK and MK reached zero as soil test K increased for most soils (Table 8) indicating the equilibrium exchangeable K level between release and fixation as proposed by Scott et al. (1957). Some soils always released K upon drying so that no equilibrium point could be calculated. Unlike other sites, site LS15 fixed K upon drying, although its relationship between absolute or relative K difference and soil test K level were very weak ($r^2 = 0.05$, Table 8).



Figure 5. Relationship between the difference (A) and ratio (B) of NH4OAc extractable K from air-dry (DK) and field-moist (MK) soil and NH4OAc extractable K from field-moist (MK) soil across all trials.

	DK† (mg kg	⁻¹)	DK-M	IK (mg k	g ⁻¹)		DK/MK		
Site	Equation [‡]	r^2 §	Equation [‡]	X-int.¶	r ²	p>F	Equation [‡]	r^2	p>F
AB15	41.7 + 0.652X	0.51	41.7 - 0.348 <i>X</i>	120	0.23	< 0.001	$0.943 + 2.88e^{-0.0324X}$	0.41	< 0.001
AR15	66.2 + 0.461X	0.31	66.2 - 0.539X		0.38	< 0.001	$1.01 + 5.35e^{-0.0379X}$	0.74	< 0.001
B15	71.3 + 0.605X	0.63	71.3 - 0.395X	180	0.42	< 0.001	$0.796 + 1.73e^{-0.0120X}$	0.53	< 0.001
C15	48.9 + 0.664X	0.66	48.9 - 0.336X	145	0.34	< 0.001	$1.01 + 2.92e^{-0.0306X}$	0.66	< 0.001
D15	41.1 + 0.697X	0.83	41.1 - 0.303 <i>X</i>	136	0.47	< 0.001	$0.973 + 3.11e^{-0.0326X}$	0.76	< 0.001
F115	70.8 + 0.822X	0.63	70.8 - 0.178X		0.07	0.029	$1.29 + 37.7e^{-0.0528X}$	0.74	< 0.001
F215	52.5 + 0.726X	0.79	52.5 - 0.274X		0.35	< 0.001	$1.16 + 5.07e^{-0.0411X}$	0.76	< 0.001
LN15	179 + 0.651X	0.65	291 - 0.636X	457	0.52	< 0.001	$0.942 + 3.21e^{-0.00774X}$	0.53	< 0.001
LS15	-23.9 + 1.13X	0.81	-23.9 + 0.133X	180	0.05	0.063	0.869 + 0.000710X	0.05	0.072
M15	48.2 + 0.778X	0.84	48.2 - 0.222 <i>X</i>		0.30	< 0.001	$1.08 + 3.53e^{-0.0311X}$	0.72	< 0.001
P15	90.4 + 0.688X	0.53	90.4 - 0.312 <i>X</i>		0.19	< 0.001	$1.13 + 3.82e^{-0.0212X}$	0.64	< 0.001
V15	79.4 + 0.639X	0.61	79.4 - 0.361 <i>X</i>	220	0.34	< 0.001	$0.886 + 2.70e^{-0.0148X}$	0.38	< 0.001
W15	27.5 + 0.736X	0.78	27.5 - 0.264 <i>X</i>	104	0.31	< 0.001	$0.869 + 1.16e^{-0.0210X}$	0.40	< 0.001
AK16	69.4 + 0.709X	0.68	69.4 - 0.291 <i>X</i>		0.27	< 0.001	$0.631 + 1.59e^{-0.00722X}$	0.60	< 0.001
CF16	32.0 + 0.677X	0.70	32.0 - 0.323X	99	0.34	< 0.001	$-0.785 + 2.74e^{-0.00493X}$	0.60	< 0.001
GD16	31.8 + 0.740X	0.71	31.8 - 0.260X		0.24	< 0.001	$0.886 + 1.48e^{-0.0221X}$	0.49	< 0.001
LB16	35.3 + 0.559X	0.52	35.3 - 0.441 <i>X</i>	80	0.40	< 0.001	$0.997 + 3.56e^{-0.0527X}$	0.67	< 0.001
MO16	49.8 + 0.563X	0.41	48.2 - 0.412 <i>X</i>		0.30	< 0.001	$1.13 + 6.42e^{-0.0550X}$	0.72	< 0.001
VC16	79.3 + 0.635X	0.56	79.3 - 0.366X	217	0.30	< 0.001	1.49 - 0.00226X	0.37	< 0.001

 Table 8.
 Regression equations for NH4OAc extractable K from air-dry soil and its absolute difference and ratio with NH4OAc extractable K from field-moist soil.

[†] DK and MK, NH₄OAc extractable K on air-dry and field-moist soil, respectively.

 $\ddagger X$, NH₄OAc extractable K from field-moist soil (MK), mg kg⁻¹.

§ All models significant at 0.001 probability level.

¶ X-int., X-intercept where (DK-MK) = 0; not shown where predicted outside of soil test K range.

Corn Grain Yield Response to Fertilization

The wide range of soil test K levels in the trials encompassed the 160 mg kg⁻¹ DK critical level employed in the present K fertilizer recommendations for corn in North Dakota (Franzen, 2014). The present DK critical level predicted grain yield response at 10 of 19 sites. Corn yield increased in response to K fertilization at only five of 12 sites with DK below the 160 mg kg⁻¹ critical level (Table 9). Moreover, two sites with DK above the 160 mg kg⁻¹ critical level had yield increases. Two sites (F115 and GD16) had significant yield decreases at the high 140 kg K ha⁻¹ rate compared with lower K rates and exhibited quadratic relationships between yield and K rate (Table 9). Yield decreases at comparable high K rates have been occasionally witnessed by Clover and Mallarino (2013) and Laboski and Andraski (2016).

Site LS15 had a confounded yield response because of the in-season application of CaCl₂ on the 0 kg K ha⁻¹ treatment to raise soil Cl levels to match the lowest KCl rate. Although no statistical difference was identified between treatments, the yield response to CaCl₂ on the 0 kg K ha⁻¹ treatment compared to the KCl treatments alludes to some yield affect by the Cl treatment (Table 9). The KCl treatments were applied in April prior to planting and high rainfall in May 2015, which likely leached Cl from the KCl treatments on the sandy-textured soil at site LS15 (Table 4); as a result, the CaCl₂ treatment provided Cl in excess of the KCl rates. The potential Cl deficiency, which was not controlled in the experiment design, confounded treatment responses at site LS15. There was no plant tissue analysis conducted in 2015 to confirm Cl deficiency. Corn yield increases from Cl have been documented on irrigated sands in New Jersey by Heckman (1995, 1998).

					Grain	yield					p>F	
				Fertilize	r K ra	te (kg K ha ⁻¹))				Tre	nd‡
Site	DK†	0	28	56		84	112	140			L	Q
	mg kg ⁻¹				— kg l	na ⁻¹						
AB15	108	9755	9742	9706		9627	10409	10438		0.90	0.36	0.58
AR15	90	9639	10437	10383		10772	10506	10556		0.60	0.19	0.28
B15	159	13882	13511	13786		13201	13480	13635		0.91	0.62	0.53
C15	87	12938	12151	13363		14276	13743	13015		0.28	0.26	0.26
D15	109	13126	13413	12807		12860	12601	12555		0.82	0.23	0.95
F115	169	12131 b§	12675 ab	12472	ab	13301 a	12663 ab	10993	c	0.03	0.19	< 0.01
F215	100	11472 c	12007 bc	12415	abc	11665 bc	13251 a	12640	ab	0.06	0.02	0.79
LN15	444	10352	10123	9931		10133	11025	10557		0.64	0.34	0.48
LS15	198	10715	9714	9667		10449	10177	10656		0.35	0.60	0.11
M15	134	10297 c	10614 c	10841	bc	10797 bc	11672 ab	12289	a	0.04	< 0.01	0.30
P15	163	11897	11756	11636		12216	12153	11661		0.86	0.87	0.69
V15	203	6732	6636	6655		6465	6680	7574		0.60	0.29	0.19
W15	111	11545 bc	11121 c	11426	c	12066 ab	12462 a	12182	a	0.01	< 0.01	0.63
AK16	163	12143	11641	12602		11776	13059	11187		0.12	0.74	0.22
CF16	84	13596	14254	13687		13841	15476	15137		0.15	0.03	0.48
GD16	84	10419 b	10934 b	10953	b	11685 a	12006 a	10587	b	< 0.01	0.02	< 0.01
LB16	69	10077 c	11951 b	12027	b	13093 a	11909 b	12456	ab	< 0.01	< 0.01	0.01
MO16	114	15148	14579	15159		14644	14505	14339		0.80	0.28	0.86
VC16	201	8985 c	10862 a	10299	ab	9351 bc	9376 bc	10800	a	0.01	0.29	0.98

 Table 9.
 Corn grain yield response to K fertilization and initial soil test K.

† DK, NH4OAc extractable K from air-dry soil.

‡ L, linear; Q, quadratic.

§ Within rows, treatment means followed by the same letter are not significantly different, LSD(0.10).

66

The relationship between the relative yield of unfertilized treatments and soil test K by various soil test methods is shown in Figure 6. Linear-plateau, quadratic-plateau, quadratic, and exponential regression models between relative yield response and soil test K using various methods are reported in Table 10. Model differences between coefficients of determination for each soil test K method demonstrate implicit model bias as recognized by Mallarino and Blackmer (1992, 1994). The critical levels for each soil test K method were generally similar across models in spite of model bias (Table 11). The sites LN15, LS15, V15, and VC16 were omitted from regression because of very high soil test K level, non-K nutrient deficiency, poor plant population and no-till, and no-till, respectively.

The DK method was the strongest predictor of yield response ($r^2 = 0.45$ to 0.52) with 95% yield response critical levels ranging from 86 to 103 mg K kg⁻¹ depending on model (Table 11). The DK method was a better predictor of yield response to K fertilization than MK, which contrasts the results of Barbagelata and Mallarino (2013) in Iowa. The MK method had a significant relationship with relative yield only when fitted to quadratic-plateau and exponential models. The 95% critical levels for MK ranged from 56 to 62 mg K kg⁻¹ (Table 11), which were lower than respective DK critical levels. Model fit for MK was inferior to comparable to the model fit for DK, showing no advantage of NH₄OAc extraction on field-moist soil over the standard method of using air-dried soil. Even though the 160 mg kg⁻¹ DK critical level predicted only 10 of 19 yield responses, the DK method remains the best method for predicting yield response in North Dakota. When significant yield responses (Table 9) were reclassified to the 95% yield response critical of 100 mg kg⁻¹ DK (Table 11), the DK method predicted only three yield responses for the six sites below 100 mg kg⁻¹ DK and failed to predict four yield responses above the critical level. Overall,

the weak to moderate relationships between NH₄OAc extraction methods (i.e., exchangeable K) and relative yield response suggest other factors influence plant available K.

Sodium tetraphenylboron extractable K (TBK) had a significant relationship with relative yield response for 5-min TBK using linear-plateau, quadratic-plateau, and quadratic models (Table 10); no significant relationship between 168-h TBK and relative yield response was identified. The 5-min TBK extraction was an inferior predictor of yield response compared to either NH4OAc method. The NaBPh4 extraction with its ability to extract a portion of nonexchangeable K did not provide a better estimate of plant available K than NH4OAc extractable K. Schindler et al. (2002) observed that NaBPh4 had no advantage over NH4OAc to estimate corn K uptake in montmorillonitic soils due to their diminished capacity to fix K. This would also apply to the smectite-dominated mineralogy of most soils in this study (Table 15).

Resin extractable K had no significant relationship with relative yield response and could only be fitted to a linear model with no plateau (Table 10). Skogley (1994) showed only a moderately weak relationship ($r^2 = 0.28$) between mixed-bed resin K and corn K uptake. Moreover, Sherrod et al. (2003) remarked that soil chemical properties (e.g., pH, carbonates) and resin counter-ion (e.g., H⁺, HCO₃⁻) may affect the soil-resin environment and nutrient extraction.

Percent K saturation of ECEC, calculated as the NH₄OAc extractable K percentage of total NH₄OAc extractable cations, had a significant relationship with relative yield response for all models (Table 10); however, its relationship with relative yield response was always weaker than the DK sufficiency approach. Moreover, the K saturation critical levels were all less than 1.8% (Table 11), which is lower than the 2 to 5% K saturation range promoted under the basic cation saturation ratio concept (Kopittke and Menzies, 2007). In agreement with Liebhardt (1981) and

McLean et al. (1983), these findings further demonstrate that the basic cation saturation ratio concept is agronomically inferior to the sufficiency concept.



Figure 6. Relationship between relative yield of unfertilized corn and various soil test K methods: (A) DK and (B) MK, NH₄OAc extractable K on air-dry and field-moist soil, respectively; TBK, tetraphenylboron extractable K for (C) 5 min and (D) 168 h; (E) RK, resin extractable K; and (F) K sat., K saturation. Single dots indicate sites excluded from regression.

Model	Equation [†]	$X < \ddagger$	Maximum§	r ²	p>F
Linear-plateau†					
DK¶	31.5 + 0.664 <i>X</i> ‡	93	93	0.49	0.017
МК	32.6 + 0.998X	61	93	0.47	0.022
TBK 5 min	74.2 + 0.0636X	333	95	0.33	0.090
TBK 168 h	81.9 + 0.00704X	2028	96	0.30	0.117
RK	84.2 + 0.528X		n.p.#	0.16	0.140
K sat.	74.1 + 12.7X	1.56	94	0.42	0.037
Quadratic-plateau*					
DK	$-5.2 + 1.72X - 0.00750X^2$	115	94	0.52	0.012
МК	$-34.8 + 3.67X - 0.0264X^2$	69	93	0.47	0.022
TBK 5 min	$60.5 + 0.178X - 0.000230X^2$	389	95	0.36	0.068
TBK 168 h	$77.3 + 0.0148X - 0.00000293X^2$	2522	96	0.31	0.109
RK	n.f.††				
K sat.	$62.1 + 35.9X - 10.2X^2$	1.77	94	0.39	0.052
Quadratic					
DK	$38.0 + 0.821X - 0.00292X^2$		95	0.45	0.026
MK	$80.2 + 0.128X - 0.000200X^2$		95	0.30	0.118
TBK 5 min	$76.9 + 0.0155X - 0.000160X^2$		96	0.35	0.078
TBK 168 h	$76.9 + 0.0155X - 0.00000315X^2$		96	0.31	0.109
RK	n.f.				
K sat.	$61.5 + 36.5X - 10.0X^2$		95	0.41	0.044
Exponential					
DK	94.2 - 1535e ^{-0.0650X}		94	0.52	0.012
MK	92.7 - 4790e ^{-0.127X}		93	0.45	0.028
TBK 5 min	n.f.				
TBK 168 h	97.6 - 23.9e ^{-0.00106X}		95	0.31	0.112
RK	n.f.				
K sat.	94.4 - 65.1e ^{-2.38X}		94	0.36	0.069

Table 10. Models fitted to relationships between relative yields of unfertilized corn and plant available K evaluated by various soil test K methods.

[†] Equation applies only for *X* values less than values at which the segmented model joins (i.e., critical values corresponding to 100% yield level shown).

 $\ddagger X =$ soil test K value (mg kg⁻¹ for DK, MK, TBK 5 min, TBK 168 h, RK; % for K sat.).

§ Maximum relative yield predicted by model in percent (e.g., plateau, maximum, asymptote).

¶ DK and MK, NH₄OAc extractable K on air-dry and field-moist soil, respectively; TBK, tetraphenylboron extractable K; RK, resin extractable K; K sat., K saturation.

n.p., no model plateau.

†† n.f., no model fit.

		Critical values					
		DK‡	MK	TBK	TBK	RK	K sat.
Model	Yield level [†]			5 min	168 h		
	%			- mg kg ⁻¹ -			%
Linear-plateau	100	93	61	333	§		1.56
	99	92	60	318			1.48
	95	86	56	258			1.19
	90	79	52	183			0.82
Quadratic-plateau	100	115	69	389			1.77
	99	104	64	323			1.47
	95	90	56	243			1.09
	90	80	51	183			0.80
Quadratic	100	149		460			1.81
	99	120		355			1.51
	95	99		262			1.13
	90	83		190			0.84
Exponential	99	113	68				1.79
	95	103	62				1.50
	90	97	59				1.32

Table 11. Critical values of soil test K for various soil test methods as determined by various models.

[†] Critical values calculated to attain 90, 95, 99, or 100% of the maximum relative yield predicted by a model (Table 10).

‡ DK and MK, NH4OAc extractable K on air-dry and field-moist soil, respectively;

TBK, tetraphenylboron extractable K; RK, resin extractable K; K sat., K saturation. Critical values are not shown when the fit of the model was not significant (p <

0.10).

Corn Plant Tissue Potassium Response to Fertilization

Plant tissue K analysis from 2016 trials revealed that K fertilization increased tissue K concentration at growth stage V5 at four sites and at growth stage VT at three sites (Table 12). For site LB16, the application of KCl at any rate increased tissue K to similar levels; therefore, an orthogonal contrast was required to identify fertilizer response over the check. Only sites GD16 and LB16, which had significant tissue K increases, also had significant grain yield response (Table 9). Sites AK16 and CF16 with increased tissue K from fertilization had no corresponding

grain yield increase, indicating luxury consumption. Tissue K responses to fertilization without concomitant grain yield increases have been frequently reported (Mallarino and Higashi, 2009; Randall et al., 1997a). Site VC16 did not have increased tissue K concentration at growth stage V5 or VT; however, VC16 still had a 1.9 Mg ha⁻¹ grain yield increase over the check treatment with 28 kg K ha⁻¹ (Table 9). Although most plant K is accumulated by growth stage R1 (Bender et al., 2013; Ciampitti et al., 2013), insufficient K nutrition at site VC16 sometime after growth stage VT manifested itself as a grain yield response to fertilization.

Soil test K using either DK or MK expressed clear relationships with tissue K concentration at both growth stages V5 and VT (Figure 7). Soil test K often expresses good relationships with tissue K (Clover and Mallarino, 2013; Mallarino and Higashi, 2009; Tyner, 1947; Walker and Peck, 1975). The DK and MK methods were comparable in their model fit with tissue K concentration (Table 13), and stronger than soil test K relationships with relative grain yield response. The growth stage V5 tissue K concentration of unfertilized corn plants had a plateaulike relationship with soil test K with maximum tissue K predicted at 34 to 35 g K kg⁻¹ on a dry matter basis (Table 13). For growth stage V5, the soil test K critical levels for 95% tissue K concentration varied from 102 to 134 mg kg⁻¹ DK and 97 to 143 mg kg⁻¹ MK, depending on regression model (Table 14). The soil test K critical levels for growth stage V5 tissue K concentration are generally higher than those for relative grain yield response (Table 11). The growth stage VT ear leaf tissue K concentration was linearly related with soil test K (Figure 7), but it had no maximum tissue K concentration to indicate K sufficiency in plant tissue at growth stage VT. A linear relationship between soil test K and growth stage VT ear leaf tissue K concentration was also reported by Mallarino and Higashi (2009). The quadratic model had a significant relationship between soil test K and growth stage VT ear leaf tissue K concentration (Table 13); however, inclusion of the quadratic component had no significant improvement over the linear model because the quadratic component was not significant for either DK (p = 0.40) or MK (p = 0.21) regression models.

Although the regression relationships between soil test K and tissue K concentration were strong (Table 13), the relationship between tissue K concentration and absolute grain yield (Figure 8A) or relative grain yield response (Figure 8B) at either growth stage V5 or VT was weak. Tissue K concentration did not address inconsistences between soil test K and grain yield response or provide a consistent indicator of sufficient K nutritional status across sites. Tissue K concentration and grain yield may have strong relationships within fields (Tyner, 1947; Walker and Peck, 1975); however, relating tissue K and grain yield across multiple sites is more tenuous (Mallarino and Higashi, 2009).

Clover and Mallarino (2013) determined that the optimum tissue K concentration to maximize corn grain yield in Iowa was 20.2 to 25.1 g K kg⁻¹ for whole plants at V5-V6 growth stage and 10.2 to 11.0 g K kg⁻¹ for ear leaves at growth stage R1. Sites GD16 and LB16 had tissue K concentrations within or below those critical ranges for their respective growth stage and had concomitant grain yield responses. Site CF16 had tissue K concentration within the critical range at growth stage VT, but it did not have a grain yield response. Site VC16 had tissue K concentrations greater than the critical ranges of Clover and Mallarino (2013) at both growth stages, yet it had a grain yield response. In North Dakota, tissue K concentration did not consistently predict grain yield response.

			Tissue K	concentratio	n			1	p>F	
		Fe	ertilizer K ra	ate (kg K ₂ O	ha ⁻¹)			Tre	nd‡	no K vs.
Site	0	28	56	84	112	140		L	Q	with K
Whole plant (growth stage V5)										
AK16	33.8 b†	33.9 b	39.5 ab	40.2 ab	43.7 a	42.7 a	0.05	< 0.01	0.56	0.04
CF16	28.8 c	33.9 b	36.0 ab	38.4 a	38.3 a	36.8 ab	< 0.01	< 0.01	< 0.01	< 0.01
GD16	24.0 d	32.9 c	34.9 bc	38.4 abc	42.4 a	42.2 ab	< 0.01	< 0.01	0.11	< 0.01
LB16	21.2	30.5	30.2	30.7	33.1	37.1	0.16	0.02	0.67	0.02
MO16	21.9	27.5	27.2	30.5	33.2	29.0	0.24	0.05	0.21	0.04
VC16	33.7	36.7	37.5	38.3	36.8	39.9	0.58	0.12	0.69	0.13
]	Ear leaf (gro	owth stage V	/T)					
AK16	13.8	13.6	14.0	14.3	14.7	14.5	0.83	0.22	0.98	0.57
CF16	10.0 c	11.0 bc	11.7 b	12.2 ab	13.0 a	13.3 a	< 0.01	< 0.01	0.40	< 0.01
GD16	10.5 c	13.4 bc	15.6 ab	16.2 ab	16.9 ab	17.8 a	0.01	< 0.01	0.19	< 0.01
LB16	9.3	12.1	12.5	12.8	12.1	13.3	0.42	0.10	0.34	0.05
MO16	11.2	11.6	11.7	11.7	12.6	11.5	0.80	0.48	0.49	0.42
VC16	18.3	18.7	18.6	19.4	19.1	19.4	0.09	0.14	0.86	0.28

 Table 12. Plant tissue K analysis for 2016 fertilizer K trials.

[†] Within rows, treatment means followed by the same litter are not significantly different, LSD(0.05).

‡ L, linear; Q, quadratic.

75



Figure 7. Relationship between tissue K concentration at growth stage V5 (A, B) or VT (C, D) and NH4OAc extractable K on air-dry (A, C) and field-moist (B, D) soil.

Model	Equation [†]	$X < \ddagger$	Maximum§	r ² ¶
		mg kg ⁻¹	g kg ⁻¹	
	Growth stage V5			
Linear-plateau†				
DK#	-1.73 + 0.331 <i>X</i> ‡	107	34	0.57
MK	3.15 + 0.298X	103	34	0.61
Quadratic-plateau [†]				
DK	$-22.6 + 0.865X - 0.00332X^2$	130	34	0.58
MK	$-6.25 + 0.592X - 0.00217X^2$	136	34	0.60
Quadratic				
DK	$-2.85 + 0.446X - 0.00131X^2$		35	0.56
MK	$1.91 + 0.401X - 0.00120X^2$		35	0.57
Exponential				
DK	$34.4 - 123e^{-0.0327X}$		34	0.59
MK	35.3 - 57.5e ^{-0.0243X}		35	0.59
	Growth stage VT			
Linear				
DK	5.67 + 0.0648X			0.83
MK	6.55 + 0.0737X			0.84
Quadratic				
DK	$4.15 + 0.0959X - 0.000130X^2$			0.83
МК	$4.84 + 0.120X - 0.000240X^2$			0.85

Table 13. Regression models fitted to relationships between tissue K concentration and soil test K.

[†] Equation applies only for *X* values less than values at which the segmented model joins (i.e., critical values corresponding to 100% yield level shown).

 $\ddagger X$, soil test K value.

§ Maximum tissue K concentration predicted by model (e.g., plateau, maximum, asymptote).

¶ All models are significant (p < 0.001).

DK and MK, NH4OAc extractable K on air-dry and field-moist soil, respectively.

	Tissue K	Critical val	ues
Model	level*	DK‡	MK
	%	mg kg ⁻¹	
Linear-plateau	100	107	103
	99	106	102
	95	102	97
	90	97	92
Quadratic-plateau	100	130	136
	99	120	123
	95	108	109
	90	99	97
Quadratic	100	170	167
	99	154	149
	95	134	128
	90	118	112
Exponential	99	180	207
	95	130	143
	90	109	115

Table 14. Critical values of soil test K for whole plant tissue K at growth stage V5 as determined by various models.

[†]Critical values calculated to attain 90, 95, 99, or 100% of maximum tissue K predicted by a model (Table 13).

Constructed by Construction and MK, NH4OAc extractable K on air-dry and field-moist soil, respectively.



Figure 8. Relationship between tissue K concentration at growth stage V5 or VT and grain yield (A) and relative grain yield (B).

Role of Soil Mineralogy in Grain Yield Response

Inconsistent yield responses to K fertilization were observed on low and high soil test K soils in North Dakota by Rakkar et al. (2015). A post-hoc mineralogical analysis of these sites indicated that a number of non-responsive, low soil test K sites had substantial amounts of K-feldspar and/or illite (Franzen, 2015). Mineralogical analysis for this study showed that some sites contained appreciable amounts of K-feldspar, muscovite, and/or illite (Table 15). All soils contained 50 to 100 g kg⁻¹ K-feldspar, and responsive and non-responsive sites were distributed throughout that K-feldspar range. The illite content of the clay fraction was more variable between responsive and non-responsive sites.

Potassium-feldspars may serve as a large source of plant available K if plant demand is high (Martin and Sparks, 1983; Parker et al., 1989b; Sadusky et al., 1987), which may explain nonresponsive, low soil test K soils with high K-feldspar content. Potassium-bearing 2:1 phyllosilicates, such as mica and illite, may also provide K available for plant uptake (Barré et al., 2008b; Conyers and McLean, 1968; Hinsinger and Jaillard, 1993; Velde and Peck, 2002).

A principal component analysis of relative yield response, DK, and selected K-bearing minerals did not indicate any strong relationships between yield response and any other variable (Table 16). The first principal component (0.502 proportion of variance) showed that DK, illite, smectite, and muscovite content were weakly to moderately related. The second principal component (0.203 proportion of variance) indicated that relative yield and K-feldspar were inversely related, whereas the third principal component (0.146 proportion of variance) showed relative yield and K-feldspar to be positively correlated yet negatively related to illite content. Potassium release from mineral K or nonexchangeable K forms may provide enough K to satisfy plant K requirements in some low soil test K, non-responsive soils; however, the relative grain

yield response to fertilization does not exhibit a clear relationship with soil test K or the quantity of K-bearing minerals in the whole soil or clay fraction. The kinetics that govern K release from these minerals under plant demand remains to be elucidated for soils in North Dakota.

		V	Whole soil		< 2µm-fraction			
Site	Quartz	Plagioclase	K-feldspar	Muscoviteillite	Smectite	Illite	Kaolinite	
				- g kg ⁻¹				
AB15	420	284	99	18	840	140	20	
AR15	379	275	95	30	850	120	30	
B15	365	183	63	38	790	160	50	
C15	416	227	64	26	850	120	30	
D15	458	219	60	23	820	150	30	
F115	385	154	56	30	870	100	30	
F215	382	183	74	19	790	140	70	
LN15	334	216	69	66	700	250	50	
LS15	524	178	55	<idl‡< td=""><td>520</td><td>410</td><td>70</td></idl‡<>	520	410	70	
M15	397	176	86	34	740	200	60	
P15	340	174	92	36	830	140	30	
V15	365	182	56	17	650	300	50	
W15	394	207	62	18	470	480	50	
AK16	450	225	56	38	700	250	50	
CF16	434	204	53	<idl< td=""><td>770</td><td>160</td><td>70</td></idl<>	770	160	70	
GD16	581	193	61	28	770	190	40	
LB16	564	178	50	19	720	220	60	
MO16	439	188	48	<idl< td=""><td>780</td><td>180</td><td>40</td></idl<>	780	180	40	
VC16	481	217	55	<idl< td=""><td>810</td><td>160</td><td>30</td></idl<>	810	160	30	

Table 15. Soil mineral composition for K-bearing and select minerals[†].

[†] Analysis conducted by Activation Laboratories Ltd. (Ancaster, Ontario, Canada) using X-ray diffraction and quantified using the Rietveld method.

‡ <IDL, below instrument detection limit.

	Princi	Principal component			
	1	2	3		
Eigenvalue	3.012	1.218	0.878		
Proportion of variance	0.502	0.203	0.146		
	Correlatio	n with eig	envector		
Variable	1	2	3		
Relative yield	0.287	-0.488	0.658		
DK†	0.534	-0.181	0.034		
Illite‡	0.479	-0.151	-0.454		
Smectite:	0.429	0.094	0.197		
Muscovite	0.449	0.379	-0.305		
K-feldspar	0.132	0.744	0.478		

Table 16. Principal component analysis between relative yield, NH₄OAC extractable K, and content of selected minerals.

[†] DK, NH₄OAc extractable K on air-dry soil.

‡ Illite and smectite of clay fraction expressed on whole soil basis.

Bray (1944) recognized that corn yield response to K could vary by soil type and calculated separate response curves for different soil types in Illinois. Cluster analysis partitioned sites into two groups by the smectite/illite ratio of their clay-sized fraction (Figure 9). The average smectite/illite ratio of higher smectite soils (group 1) and lower smectite soils (group 2) were 6.6 and 3.1, respectively, and significantly different between groups (p < 0.001). With the exception of site W15, all sites still contained more smectite than illite (Table 17), which agrees with the smectitic and mixed mineralogical classification of soils in eastern North Dakota (USDA-NRCS, 2006). The average DK of group 1 and 2 soils were 118 and 115 mg kg⁻¹, respectively, and not significantly different (p = 0.87). The relationship between DK and relative yield response showed visual differences between group 1 and group 2 soils (Figure 10); however, regression relationships between DK and relative yield were not significant (p < 0.10) for group 1 or group 2 soils using various regression models (Table 18). As with regression across sites earlier (Figure 6;

Table 10), sites LN15, LS15, V15, and VC16 were omitted from cluster analysis and subsequent regression because of very high soil test K level, non-K nutrient deficiency, poor plant population and no-till, and no-till, respectively; inclusion of these sites in cluster analysis did not improve regression.

Although model fit for group 1 or group 2 soils was not significant (p < 0.10), group 2 soils with lower smectite and higher illite expressed stronger regression relationships with relative yield response than group 1 soils with higher smectite (Table 18). The DK critical levels predicted by linear-plateau and quadratic-plateau models (i.e., the segmented model joint) are lower for group 2 soils, suggesting that soils with more illite may have a lower critical level. The DK critical levels for group 2 soils are similar to those predicted across all soils (Table 11) for the respective model; the higher smectite group 1 soils appear to require higher soil test K. While these regression relationships are not significant, these differences between group 1 and group 2 soils suggests that soils with different clay mineralogy should be treated differently in soil test K calibration, which warrants further investigation as Bray (1944) acknowledged.



Figure 9. Cluster tree of K trials by smectite/illite ratio using Ward's minimum variance cluster analysis.

Group	Site	Smectite/illite ratio
1	AB15	6.00
1	AR15	7.08
1	C15	7.08
1	D15	5.47
1	F115	8.70
1	F215	5.64
1	P15	5.93
2	AK16	2.80
2	B15	4.94
2	CF16	4.81
2	GD16	4.05
2	LB16	3.27
2	M15	3.70
2	MO16	4.33
2	W15	0.98

Table 17. Classification of K trials by smectite/illite ratio.

† Classification by Ward's minimum variance cluster analysis (Figure 9).

‡ Ratio of smectite/illite in clay-sized fraction.



Figure 10. Relationship between relative yield of unfertilized corn and NH4OAc extractable K on air-dry soil (DK) for high smectite/illite group 1 soils (A) and low smectite/illite group 2 soils (B).

Model	Equation [†]	$X < \ddagger$	Maximum§	r^2	p>F
		mg kg ⁻¹	%		
Linear-plateau†					
Group 1¶	71.8 + 0.195X;	115	94	0.30	0.487
Group 2	28.5 + 0.701X	93	94	0.59	0.106
Quadratic-plateau [†]					
Group 1	$57.8 + 0.519X - 0.00184X^2$	141	94	0.27	0.527
Group 2	$-35.9 + 2.40X - 0.0111X^2$	108	94	0.59	0.107
Quadratic					
Group 1	$46.8 + 0.695X - 0.00247X^2$		96	0.31	0.475
Group 2	$39.3 + 0.780X - 0.00271X^2$		95	0.49	0.188
Exponential					
Group 1	94.8 - 139e ^{-0.0369X}		95	0.25	0.556
Group 2	94.1 - 2119e ^{-0.0695X}		94	0.58	0.115

Table 18. Regression models fitted to relationships between relative yield of unfertilized corn and NH4OAC extractable K from air-dry soil for high and low smectite/illite soils.

[†] Equation applies only for *X* values less than values at which the segmented model joins (i.e., critical values corresponding to 100% yield level shown).

‡ X, NH₄OAC extractable K from air-dry soil.

§ Maximum relative yield predicted by model (e.g., plateau, maximum, asymptote).

¶ Group 1 and 2, high and low smectite/illite ratio soils, respectively, classified in Table 17.

Summary and Conclusions

The standard soil K test in the North Central region employs a neutral 1.0 M ammonium acetate extraction on air-dry or oven-dry soil; however, the method has come under scrutiny because sample drying has been shown to affect the amount of extractable K and its relationship with yield response to K fertilization is often weak. Soil sample drying was shown to significantly increase NH4OAc extractable K from its field-moist condition by a factor of 1.27 on average and the amount of NH4OAc extractable K released upon drying was greater for low K soils. The absolute and relative amount of K released upon drying varied among the soils, and no clear relationship existed across soils.

The current soil test critical level of 160 mg kg⁻¹ predicted corn grain yield response to K fertilization at only 10 of 19 sites; nevertheless, relative grain yield response was best related to NH₄OAc extractable K from air-dry soil. The NH₄OAc K method on field-moist soil had no advantage over the standard method. Sodium tetraphenylboron and resin extraction methods, which have the ability to assess a portion of nonexchangeable K, were inferior methods as well. Tissue K concentration was increased in response to K fertilization at four of six sites. Tissue K was well related to soil test K; however, there was no relationship between tissue K concentration and grain yield response to K fertilization. Potassium-feldspar, muscovite, smectite, and illite content had mixed relationships with relative yield response. Partitioning sites by smectite/illite ratio prior to regression did not improve relationships between soil test K and yield response, but it provided some indication that soils with smectitic or illitic mineralogy may have different soil test K critical levels.

SEASONAL VARIATION IN SOIL TEST POTASSIUM LEVELS

Abstract

Potassium fertilizer recommendations are commonly guided by yield response calibrations to soil test K; however, the temporal variation in soil test K between years and within a year may confound soil test calibration and interpretation. At 19 K fertilizer trials on corn in North Dakota, NH4OAc extractable K from air-dry (DK) and field-moist (MK) soil was recorded biweekly during the growing season from non-fertilized cropped and fallow plots. Soil test K was affected by sampling date for 18 of 19 sites. Soil test K was generally highest in spring and lowest in late summer before increasing slightly in fall after crop senescence. Cubic regression modeled soil test K change using DK at 12 sites and MK at 17 sites. Maximum and minimum soil test K was predicted to occur between day of year 132 and 164 and day of year 222 and 242, respectively. Seasonal difference between spring and fall soil test K ranged from 16 to 83 mg kg⁻¹ for DK and 19 to 88 mg kg⁻¹ across sites, which would be large enough to change soil test K interpretation classes for fertilizer K recommendations. Interpretation of soil test K results for directing fertilizer K recommendations or recording long-term soil test K trends requires caution and recognition of the seasonal variation and potential change in soil test K associated with the date of soil sampling.

Introduction

Fertilizer recommendations are commonly guided by yield response calibrations to soil test levels of a particular nutrient (Peck and Soltanpour, 1990); however, seasonal variability in soil test levels may confound soil test calibration and interpretation (Kowalenko, 1991; Sabbe and Marx, 1987). Soil test K levels are known to vary between years and within a year. Soil test K levels vary between years without a clear relationship to soil K mass balance (Mallarino et al., 1991a, 2011; Peck and Sullivan, 1995; Randall et al., 1997b; Vitko et al., 2009). At two long-term locations in south central Minnesota, annual variation in soil test K prevented calculation of soil test K drawdown or buildup rates (Randall et al., 1997b). Moreover, yield response to K fertilization may vary from year to year at the same location (Randall et al., 1997a).

Within a year, soil test K is generally highest in spring and lowest in late summer or fall (Bell and Thornton, 1938; Grava et al., 1961; Liebhardt and Teel, 1977; Lockman and Molloy, 1984; Peck and Sullivan, 1995; Rakkar et al., 2015; Vitko et al., 2009), following seasonal patterns in plant K uptake and soil water dynamics. Over winter, soil test K increases in response to freeze–thaw events (Childs and Jencks, 1967; Peck and Sullivan, 1995; Rouse and Bertramson, 1950) and leaching of K from plant residues (Oltmans and Mallarino, 2015; Rosolem et al., 2005).

Over a nine year soil sampling period from 1986 to 1994 in Illinois, Peck and Sullivan (1995) observed a cyclical variation in soil test K with low levels in late summer and high levels in midwinter, albeit great variability was observed in soil test K levels throughout that time. Franzen (2011), using data from Peck and Sullivan (1995), constructed a time-series unobserved components model that identified a seasonal variation component in soil test K whose periodicity paired with the seasonal variation component in soil water content. In Mississippi, temporal variation in soil test K followed a similar pattern between years that the authors described as a sinusoidal pattern (Oldham et al., 2013, 2015). Luebs et al. (1956) even noted a seasonal relationship between exchangeable K and the seasonal change in vapor pressure in the laboratory where their soil samples were stored. Roberts (1968) remarked that fertilizer recommendations based on a single sampling time would have inherent problems from temporal K variation at sampling.

The objective of this study was to assess the seasonal variation in soil test K during the growing season, examining NH₄OAc extractable K from air-dry and field-moist soil, to improve soil test K interpretation for fertilizer recommendations.

Methods and Materials

Field Study

On-farm fertilizer K rate trials were established in southeastern North Dakota at thirteen locations in 2015 and six locations in 2016 (Table 4). The experiments were organized as randomized complete block design with four replications and six K rate treatments on corn and one non-fertilized fallow treatment to assess soil K changes without plant uptake. The experiment unit size was 3.0 m by 9.1 m. The K treatments were 0, 28, 56, 84, 112, and 140 kg K ha⁻¹ applied as fertilizer-grade KCl (500 g K kg⁻¹) granules hand-broadcasted prior to planting. The experiment unit size was 3.0 m by 9.1 m. Farmer-cooperators planted corn and applied herbicides and other inputs on experiment areas when they conducted those activities on the rest of the field. Initial soil samples were collected from non-fertilized check and fallow plots from 0- to 15-cm and 15- to 30- cm depths and thereafter collected biweekly until harvest from the 0- to 15-cm depth only. Six soil cores were taken from each plot at each sampling date and composited for analysis.

Soil Analysis

Soil samples from non-fertilized check and fallow plots were hand-homogenized and split into two subsamples. One subsample was air-dried following the procedure suggested for the North Central Region of the U.S. (Gelderman and Mallarino, 2012), ground to pass through a 2mm sieve, and analyzed for NH₄OAc extractable K (Warnacke and Brown, 2012). A 2 g air-dry soil sample was weighed and extracted with 20 mL 1 M NH₄OAc at pH 7.0 and shaken for 5 min in 50-mL Erlenmeyer flasks. The extract was filtered through Whatman No. 2 filter paper (GE Healthcare Bio-Sciences, Pittsburgh, Pennsylvania) and analyzed for extractable K by atomic absorption spectroscopy (AAS). The other subsample was kept at field-moisture, stored in a plastic re-closeable bag, and refrigerated at 4 °C until analysis. The field-moist sample was prepared following the direct sieving procedure described by Gelderman and Mallarino (2012). Field-moist soil was passed through a 2-mm sieve, and soil water content was immediately determined by drying a 6 g moist subsample to air-dryness and constant weight. A 2 g air-dry equivalent mass of field-moist soil was weighed and extracted with 20 mL 1 M NH₄OAc at pH 7.0, thereafter, following the same analysis procedure described ^{for} the air-dry method.

Data Analysis

Soil test K data was analyzed as a randomized complete block design with split-plot in time arrangement with SAS version 9.4 (SAS Institute, 2013) using PROC GLM to determine if changes in soil test K were significant over time or between cropped or fallow surfaces. The SAS macro PDGLM800 (Saxton, 1998) was used to simplify PROC GLM output for interpretation. Soil test K data were regressed to fit cubic functions over time using PROC NLIN. The first and second derivatives of the cubic functions were calculated to identify maximum and minimum soil test K during the growing season. Soil water content was standardized to a Z-score within each site using PROC STANDARD prior to regression analysis over time using PROC NLIN. The time modeling did not use PROC UCM as employed by Franzen (2011) because soil samples in this study were collected during only one sampling season cycle.

Results and Discussion

Effect of Sampling Date on Soil Test Potassium

Soil test K measured throughout the growing season from late May to early September (2015) or late September (2016) revealed that soil test K did change from spring to fall. The effect

of soil sampling time on NH₄OAc extractable K from air-dry (DK) and field-moist (MK) soil was significant (p < 0.05) at 18 of 19 sites (Table 19). The main effect of a cropped or fallow soil surface was only significant for DK and MK at three sites. The interaction between surface and soil sampling time was significant at 15 of 19 sites. The DK method was higher than the MK method at most soil sampling dates (Figures 10, 11, and 12), in agreement with Grava et al. (1961) and Peck and Sullivan (1995).

For cropped plots, soil test K was generally highest in late May or June and reached its lowest level in late summer before slightly increasing in fall (Figures 10, 11, and 12), agreeing with the cyclical soil test K observations of Peck and Sullivan (1995) in Illinois. These trends were consistent with seasonal trends in plant K uptake and soil water use. In fall, soil test K increased as K was leached from senesced corn tissue after physiologic maturity (Oltmans and Mallarino, 2015). For fallow plots, soil test K trends were of lesser magnitude and remained largely constant throughout the growing season, although variation occurred between sampling dates (Figures 10, 11, and 12). Soil test K from fallow plots consistently decreased at site LS15 and consistently increased at sites P15 and AK16. Soil test K values for individual sites are reported in the Appendix. Soil water content was higher on fallow plots because of reduced evapotranspiration in the absence of plant water uptake. The average soil water content to air-dryness of cropped and fallow treatments across sites and sampling dates was 0.158 and 0.172 g g⁻¹, respectively, which were significantly different (p < 0.001), with ranges of 0.021 to 0.263 g g⁻¹ and 0.039 to 0.261 g g⁻¹, respectively.

	p>F							
		DK†			MK			
Site	S‡	Т	$\mathbf{S} \times \mathbf{T}$	S	Т	$\mathbf{S} \times \mathbf{T}$		
AB15	0.64	< 0.01	< 0.01	0.69	< 0.01	< 0.01		
AR15	0.68	< 0.01	< 0.01	0.51	< 0.01	< 0.01		
B15	0.25	< 0.01	0.01	0.14	< 0.01	< 0.01		
C15	0.94	0.04	0.02	0.72	0.02	0.01		
D15	< 0.01	0.32	0.01	0.01	0.08	0.01		
F115	0.47	< 0.01	0.10	0.47	< 0.01	0.02		
F215	0.07	< 0.01	< 0.01	0.08	< 0.01	< 0.01		
LN15	0.26	< 0.01	0.05	0.28	< 0.01	0.07		
LS15	0.04	< 0.01	0.02	0.04	< 0.01	< 0.01		
M15	0.37	< 0.01	< 0.01	0.30	< 0.01	< 0.01		
P15	0.33	< 0.01	0.02	0.65	< 0.01	< 0.01		
V15	0.33	< 0.01	< 0.01	0.23	< 0.01	< 0.01		
W15	0.02	< 0.01	< 0.01	0.03	< 0.01	< 0.01		
AK16	0.18	< 0.01	0.09	0.18	0.08	< 0.01		
CF16	0.02	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01		
GD16	0.05	< 0.01	< 0.01	0.05	0.01	< 0.01		
LB16	0.08	< 0.01	< 0.01	0.07	< 0.01	< 0.01		
MO16	0.25	< 0.01	< 0.01	0.11	< 0.01	< 0.01		
VC16	0.22	< 0.01	0.10	0.24	< 0.01	0.12		

 Table 19. Analysis of variance (ANOVA) for sampling time on cropped and fallow treatments for nineteen sites.

[†] DK and MK, NH₄OAc extractable K on air-dry and fieldmoist soil, respectively.

‡ S, surface (cropped vs. fallow); T, time (day of year).





Figure 11. Soil test K from air-dry (DK) and field-moist (MK) soil from cropped and fallow plots over time for seven of thirteen 2015 sites. Bars are standard error.


Figure 12. Soil test K from air-dry (DK) and field-moist (MK) soil from cropped and fallow plots over time for six of thirteen 2015 sites. Bars are standard error.



Figure 13. Soil test K from air-dry (DK) and field-moist (MK) soil from cropped and fallow plots over time for 2016 sites. Bars are standard error.

Modeling of Soil Test Potassium over Time

Regression analysis of soil test K relationships in Figures 10, 11, and 12 showed that the effect of sampling date could be modeled by a cubic function. For cropped plots, cubic relationships with sampling date were significant (p < 0.10) at 12 of 19 sites for DK and 17 of 19 sites for MK (Tables 20 and 21). Using the first and second derivatives of cubic function inflection points, the regression equations predicted maximum soil test K to occur in spring between day of year (DOY) 132 and 164 (12 May to 13 June) and minimum soil test K to occur in fall between DOY 222 and 242 (10 Aug. to 30 Aug.). Sampling date bias due to the biweekly sampling interval is acknowledged for the range of dates predicted for maximum and minimum soil test K. Site MO16 had no maximum or minimum as its soil test K levels decreased continuously throughout the growing season.

The seasonal difference between spring and fall soil test K ranged from 16 to 83 mg kg⁻¹ for DK and 19 to 88 mg kg⁻¹ across sites with significant cubic relationships (Tables 20 and 21), which would be great enough to change soil test K interpretation classes for fertilizer recommendations (Franzen, 2014) at most sites simply depending on a spring versus fall soil sampling time. The soil test K range between spring and fall sampling dates varied considerably across sites, which prevents simple adjustment of spring or fall soil test K results to a different sampling date for interpretation. However, the difference in soil test K between spring and fall sampling dates and the average soil test K were positively correlated for DK ($r^2 = 0.37$, p = 0.04, n = 12) and MK ($r^2 = 0.59$, p < 0.01, n = 13), indicating that the change in soil test K from spring to fall appears to be scaled by soil test K level for those sites with significant regressions (Tables 20 and 21).

	M	odel par	ameters†				Maxi	mum K	Mini	Minimum K	
Site	a	b	с	d	r^2	p>F	Time	Value	Time	Value	
				10-6			DOY	mg kg ⁻¹	DOY	mg kg ⁻¹	
AB15	-1039	19.8	-0.110	196	0.88	0.03	149	113	226	68	
AR15	-1585	26.8	-0.139	234	0.67	0.18	168	114	228	89	
B15	-2659	45.7	-0.239	405	0.79	0.08	163	194	230	134	
C15	-1415	24.2	-0.126	215	0.45	0.45	169	111	222	95	
D15	-613	12.3	-0.068	119	0.88	0.03	150	113	230	82	
F115	-370	9.71	-0.056	100	0.50	0.39	141	174	230	140	
F215	-1052	19.2	-0.103	178	0.39	0.53	158	113	227	84	
LN15	595	3.50	-0.047	121	0.60	0.38	‡		215	368	
LS15	-1003	21.7	-0.125	226	0.92	0.01	140	207	230	123	
M15	-1890	32.5	-0.168	282	0.97	< 0.01	164	155	234	107	
P15	-538	11.4	-0.060	102	0.20	0.81	166	177	225	167	
V15	-2499	44.0	-0.231	393	0.77	0.09	163	236	230	176	
W15	-1669	29.7	-0.161	279	0.98	< 0.01	156	120	228	68	
AK16	-146	5.61	-0.033	59	0.88	< 0.01	137	162	232	136	
CF16	-232	5.74	-0.033	59	0.96	< 0.01	134	81	242	43	
GD16	-167	4.52	-0.026	47	0.92	< 0.01	140	85	228	69	
LB16	-128	3.67	-0.022	40	0.80	0.02	132	68	231	49	
MO16	399	-3.54	0.012	-10	0.90	< 0.01	n.c.§	n.c.	n.c.	n.c.	
VC16	-254	7.57	-0.041	69	0.54	0.17	152	201	240	178	

Table 20. Cubic regression models of NH₄OAc extractable K from air-dry soil over time on cropped plots.

‡ Maximum or minimum not reported where predicted outside sampling range.

§ n.c., not calculable using first and second derivatives.

	М	odel par	ameters†				Maxi	mum K	Mini	Minimum K	
Site	a	b	с	d	r^2	p>F	Time	Value	Time	Value	
				10-6			DOY	mg kg ⁻¹	DOY	mg kg ⁻¹	
AB15	-1329	24.6	-0.136	241	0.99	< 0.01	149	107	228	49	
AR15	-1326	23.4	-0.126	216	0.94	0.01	156	88	231	42	
B15	-1207	25.2	-0.145	262	0.96	< 0.01	141	202	227	121	
C15	-83	4.54	-0.032	66	0.86	0.04	‡		222	61	
D15	-1246	23.1	-0.128	227	0.79	0.08	150	106	227	55	
F115	-623	14.0	-0.081	147	0.86	0.04	137	144	231	84	
F215	-1270	22.9	-0.125	219	0.86	0.04	153	86	228	40	
LN15	2600	-25.1	0.081	-60	0.61	0.37			200	332	
LS15	-509	13.1	-0.076	136	0.95	< 0.01	135	206	238	131	
M15	-1326	23.9	-0.127	214	0.84	0.05	156	129	238	71	
P15	-242	7.63	-0.047	90	0.86	0.04			226	100	
V15	-1232	26.9	-0.158	291	0.88	0.03	138	245	223	157	
W15	-1109	21.9	-0.124	221	0.94	0.01	144	136	230	65	
AK16	-413	8.48	-0.043	71	0.69	0.06	162	123	245	103	
CF16	-478	8.62	-0.045	73	0.74	0.03	159	62	247	37	
GD16	-388	7.08	-0.036	58	0.69	0.06	163	63	250	44	
LB16	-106	2.34	-0.011	16	0.78	0.02	163	50			
MO16	180	-1.64	0.008	-20	0.79	0.02	n.c.§	n.c.	n.c.	n.c.	
VC16	-479	10.2	-0.050	78	0.51	0.21	165	184	263	148	

Table 21. Cubic regression models of NH₄OAc extractable K from field-moist soil over time on cropped plots.

‡ Maximum or minimum not reported where predicted outside sampling range.

§ n.c., not calculable using first and second derivatives.

Cubic regression for combined sites where individual cubic relationships were significant revealed that 2015 sites could be successfully summarized into one cubic function, indicating that the periodicity of soil test K change was consistent across low and high soil test K sites in 2015 (Figure 14A). The cubic regression equations for DK (Eq. 2) and MK (Eq. 3) using 2015 sites were:

$$DK = -1655 + 30.0 DOY + -0.161 DOY^2 + 0.000279 DOY^3$$
(Eq. 2)

$$MK = -888 + 18.2 DOY + -0.104 DOY^2 + 0.000187 DOY^3$$
(Eq. 3)

where DK and MK, NH₄OAc extractable K from air-dry and field-moist soil, respectively; DOY, day of year. The 2015 combined regression for MK was stronger ($r^2 = 0.22$, p < 0.01, Eq. 3) than the combined regression for DK ($r^2 = 0.16$, p = 0.02, Eq. 2). The maximum and minimum soil test K values occurred at DOY 157 and 228, respectively, for DK and DOY 142 and 229, respectively, for MK. The combined maximum and minimum soil test K dates lie within the range of dates for individual sites (Tables 20 and 21). Although 2016 sites exhibited strong sinusoidal relationships for five of six individual sites, the sites were unable to be summarized into one function (Figure 14B) because of late season rainfall variability across the study region (Figure 15B). The 2016 combined cubic regressions for DK and MK methods had p-values of 0.33 and 0.20, respectively.



Figure 14. Soil test K from air-dry (DK, closed circles) and field-moist (MK, open circle) soil over time for cropped plots of combined sites from 2015 (A) and 2016 (B).

The range of dates predicted by regression equations for cropped plots (Tables 20 and 21) provides soil sampling professionals with target dates for soil sampling activities depending on their interpretation of soil test K values. If one wished to know the potential maximum soil test K for the growing season, a spring sampling date would be preferable. Conversely, a fall sampling date would be preferred if one wanted to know the potential minimum soil test K during the

growing season. Kowalenco (1991) conservatively preferred a fall sampling date identifying lower soil test K for directing fertilizer recommendations.

Franzen (2011), using the nine-year soil sampling data of Peck and Sullivan (1995), identified that the seasonal variation in soil test K and soil water content were related. Our study region received limited rainfall in late summer and early fall of 2015, which resulted in decreased soil water content (Figure 15A) causing increased K fixation and decreased K diffusion (Olk et al., 1995; Zeng and Brown, 2000) to reduce soil test K in late summer. Since soil water content varied greatly between individual sites, the standardization of soil water content within each site clarified the decreasing trend in soil water content for cropped plots across sites (Figure 15C). Fallow plots, having no plant water uptake, did not exhibit an apparent decrease in soil water content having a stronger relationship with sampling date across sites than the non-standardized soil water content (Table 22). Soil water content maximum and minimum were predicted at DOY 151 (31 May) and DOY 256 (13 Sept.), respectively.

In 2016, the study region was dry during spring and early summer, followed by scattered rains in late summer that differentially affected soil water content across sites (Figure 15B). No trend in soil water content or standardized soil water content (Figure 15D) was apparent across 2016 sites. Cubic regression between soil water content and sampling date was not significant (p < 0.10) for 2016 cropped or fallow plots (Table 22). The lack of trend between soil water content and sampling date in 2016 was reflected in the poor combined regression for soil test K in 2016 (Figure 14B). Variation in seasonal soil water content challenges the robustness of combined site modeling of soil test K over time.



Figure 15. Soil water content to air-dryness (A, B) and its standardized Z-score within a site (C, D) over time for 2015 (A, C) and 2016 (B, D) sites.

		Model parameters ⁺							
Year	Surface	a	b	c	d	r^2	p>F		
				10-6	10-9				
		Y =	$Y = Soil H_2O$ content to air-dryne						
2015	Cropped	-0.732	0.0145	-80.0	125	0.12	0.007		
2015	Fallow	-0.0566	0.00321	-10.0	19.6	0.01	0.791		
2016	Cropped	0.0917	0.00117	-4.87	5.82	0.01	0.927		
2016	Fallow	0.286	-0.00254	17.0	-35.8	0.03	0.571		
		$\mathbf{Y} = \mathbf{Z}$	Z-score of so	oil H ₂ O co	ontent to	air-dryn	ess		
2015	Cropped	-20.8	0.348	-1830	2995	0.39	< 0.001		
2015	Fallow	11.5	-0.188	1070	-2000	0.09	0.026		
2016	Cropped	-1.66	0.0197	-60.0	23.8	0.02	0.769		
2016	Fallow	6.09	-0.131	852	-1690	0.10	0.126		
			2	2					

 Table 22. Cubic regression equations for soil water content over time for cropped and fallow plots.

[†] Cubic model: $Y = a + bDOY + cDOY^2 + dDOY^3$; where DOY, day of year.

For fallow plots, modeling soil test K trends was less successful with significant regression relationships (p < 0.10) identified at 3 of 19 sites for DK and 5 of 19 sites for MK (Tables 23 and 24). Since soil test K on fallow plots expressed less overall change than on the cropped plots (Figures 10, 11, 12), the low number of sites with significant cubic relationships was expected. Without plant K uptake or water use on fallow plots, soil test K trends with seasonal maximums and minimums were less apparent. Regression equation for sites GD16 and LB16 did predict MK maximums in fall (Table 24), showing that soils with lower soil test K (<90 mg kg⁻¹ MK) may increase over time in the absence of plant K uptake. No combined site regression for soil test K was attempted for fallow plots because of the low number of individual sites with significant regression relationships.

	М	lodel par	ameters†				Maxi	mum K	Mini	Minimum K	
Site	a	b	с	d	r^2	p>F	Time	Value	Time	Value	
				10-6			DOY	mg kg ⁻¹	DOY	mg kg ⁻¹	
AB15	-1410	23.4	-0.119	196	0.66	0.65	172	99	231	79	
AR15	-944	16.0	-0.082	137	0.56	0.31	181	103	215	100	
B15	-3975	63.2	-0.313	506	0.95	< 0.01	177	216	235	168	
C15	92	0.594	-0.006	15	0.32	0.64	‡		190	97	
D15	-524	9.57	-0.048	78	0.73	0.12	186	114	220	113	
F115	480	-4.63	0.022	-30	0.17	0.84			161	170	
F215	-169	5.04	-0.029	53	0.26	0.72	154	122	206	118	
LN15	-5151	85.4	-0.426	698	0.38	0.66	178	479	229	433	
LS15	-1981	36.1	-0.193	332	0.85	0.04	158	216	229	156	
M15	-1229	22.1	-0.118	206	0.65	0.21	162	126	221	105	
P15	-379	7.76	-0.038	62	0.71	0.14	n.c.§	n.c.	n.c.	n.c.	
V15	117	1.14	-0.006	10	0.02	0.99	163	188	233	187	
W15	-1680	28.2	-0.144	238	0.48	0.41	170	133	232	105	
AK16	-19	3.34	-0.019	36	0.47	0.25	147	169	211	164	
CF16	-166	3.84	-0.019	31	0.22	0.67	165	81	250	71	
GD16	-6	1.57	-0.009	17	0.18	0.74	163	85	189	85	
LB16	271	-2.94	0.014	-20	0.27	0.56			172	69	
MO16	62	0.616	-0.004	8.5	0.12	0.12			221	84	
VC16	208	0.614	-0.007	16	0.67	0.07			217	193	

Table 23. Cubic regression models of NH₄OAc extractable K from air-dry soil over time on fallow plots.

‡ Maximum or minimum not reported where predicted outside sampling range.

§ n.c., not calculable using first and second derivatives.

	Model parameters†						Maxi	mum K	Mini	Minimum K	
Site	a	b	с	d	r^2	p>F	Time	Value	Time	Value	
				10-6			DOY	mg kg ⁻¹	DOY	mg kg ⁻¹	
AB15	-796	14.3	-0.076	131	0.59	0.27	160	78	228	57	
AR15	-619	11.7	-0.064	114	0.36	0.57	157	79	218	66	
B15	-3142	51.6	-0.260	427	0.43	0.48	173	214	233	166	
C15	36	1.27	-0.010	23	0.28	0.69	‡		202	70	
D15	-584	10.8	-0.056	96	0.65	0.20	178	107	211	105	
F115	-844	16.7	-0.094	171	0.57	0.30	152	129	215	107	
F215	-58	2.97	-0.018	35	0.18	0.83	138	100	205	95	
LN15	-5729	99.9	-0.530	916	0.64	0.33	164	444	221	361	
LS15	120	2.36	-0.018	36	0.79	0.08			238	160	
M15	-746	14.3	-0.079	142	0.44	0.46	156	99	216	83	
P15	-268	6.63	-0.038	72	0.61	0.25	153	110	201	106	
V15	183	0.466	-0.006	17	0.69	0.16			185	173	
W15	-394	9.23	-0.052	93	0.46	0.44	147	137	225	115	
AK16	-193	4.60	-0.022	36	0.78	0.02	n.c.§	n.c.	n.c.	n.c.	
CF16	-227	3.75	-0.015	19	0.50	0.22	205	75			
GD16	126	-1.63	0.011	-20	0.81	0.01	269	101			
LB16	283	-4.34	0.026	-50	0.66	0.07	214	64	135	52	
MO16	291	-4.38	0.027	-50	0.74	0.03	228	87			
VC16	325	-2.66	0.016	-30	0.11	0.87	229	201			

Table 24. Cubic regression models of NH₄OAc extractable K from field-moist soil over time on fallow plots.

‡ Maximum or minimum not reported where predicted outside sampling range.

§ n.c., not calculable using first and second derivatives.

Summary and Conclusions

The date of soil sampling affected soil test K levels at 18 of 19 sites using NH₄OAc extraction on air-dry and field-moist soil alike. Soil test K was highest in spring and reached its lowest value in late summer before slightly increasing in fall in response to leaching of corn tissue K after senescence. Trends in soil test K were of lesser magnitude and more variable for fallow plots, which lacked crop K uptake and water use. For most sites, the magnitude of soil test K change was great enough to change soil test K interpretation classes, which would lead to different fertilizer K recommendations simply based on a spring versus fall soil sampling time. The change in soil test K could be modeled using cubic functions, whose maximum and minimum soil test K were predicted between day of year 132 and 164 and day of year 222 to 242, respectively. Seasonal trend in soil test K was influenced by soil water content during the growing season. Depending on the interpretation of soil test K results for directing fertilizer K recommendations, a soil fertility program may use spring or fall soil sampling dates to ascertain the potential maximum or minimum soil test K. Long-term recording of soil test K should sample fields at the same time each year to reduce the intra-seasonal temporal variation in soil test K.

GENERAL CONCLUSIONS

Soil sample drying was shown to significantly increase NH₄OAc extractable K from its field-moist condition by a factor of 1.27 on average and the amount of NH₄OAc extractable K released upon drying was greater for low K soils. The absolute and relative amount of K released upon drying varied among the soils, and no clear relationship existed across soils. Consequently, there is no simple conversion factor or equation between the amounts of NH₄OAc extractable K from air-dry or field-moist soil.

In 2016, tissue K concentration was increased in response to K fertilization at four of six sites. Tissue K concentration was well related to soil test K, predicting the soil test K critical level for 95% maximum tissue K concentration for growth stage V5-V6 at 102 to 134 mg kg⁻¹ NH₄OAc extractable K from air-dry soil. There was no soil test K critical level predicted for tissue K concentration at growth stage VT as the relationship with soil test K was linear. There was no relationship between tissue K concentration and grain yield response to K fertilization.

The current soil test K critical level of 160 mg kg⁻¹ NH₄OAc extractable K from air-dry soil predicted corn grain yield response to K fertilization at only 10 of 19 sites; nevertheless, relative grain yield response was best related to NH₄OAc extractable K from air-dry soil, predicting the soil test K critical level for 95% yield response at 86 to 103 mg kg⁻¹ NH₄OAc extractable K from air-dry soil. The NH₄OAc extraction method on field-moist soil had no advantage over the standard soil test K method using air-dry soil. Sodium tetraphenylboron and resin extraction methods, which have the ability to assess a portion of nonexchangeable K, were inferior methods in predicting yield response. Principal component analysis showed that there were good relationships among NH₄OAc extractable K from air-dry soil and muscovite, smectite, and illite content; however, K-feldspar, muscovite, smectite, and illite content had mixed relationships

with relative yield response. Partitioning sites by smectite/illite ratio prior to regression did not improve relationships between soil test K and yield response, but it provided some indication that soils with smectitic or illitic mineralogy may have different soil test K critical levels.

The date of soil sampling affected soil test K levels at 18 of 19 sites using NH₄OAc extraction on air-dry and field-moist soil alike. Soil test K was highest in spring and reached its lowest value in late summer before slightly increasing in fall in response to leaching of corn tissue K after senescence. Trends in soil test K were of lesser magnitude and more variable for fallow plots, which lacked crop K uptake and water use. The change in soil test K could be modeled using cubic functions, whose maximum and minimum soil test K were predicted between day of year 132 and 164 and day of year 222 to 242, respectively. Seasonal trend in soil test K was influenced by soil water content during the growing season.

Overall, the results of this study indicate that the present fertilizer K recommendations for corn in North Dakota (Franzen, 2014) do not adequately predict corn grain yield response to K fertilization, only being correct about half of the time. Nevertheless, the NH₄OAc extraction method using air-dry soil, employed in the present recommendations, remains the best predictor of yield response over other extraction methods investigated. Seasonal variation in soil test K from spring to fall was often great enough to change soil test K interpretation classes, which may affect fertilizer K recommendations. Soil sampling in fall when soil test K is lower likely leads to more frequent fertilizer K applications than soil sampling in spring when soil test K is higher. For long-term recording of soil test K, individuals should sample fields at the same time each year to reduce intra-seasonal temporal variation in soil test K while tracking annual variation in soil test K.

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DOY†		DK		МК					
_	C‡	F	Mean	С	F	Mean			
			r	ng kg ⁻¹					
140	108 ab§	72 ef	90 abc	102 ab	74 cdefg	88 a			
154	116 a	100 abc	108 a	109 a	81 bcde	95 a			
170	109 ab	99 abc	104 ab	95 abc	71 defgh	83 ab			
189	75 ef	88 cde	81 c	76 cdef	83 bcd	80 ab			
205	85 cdef	95 bcd	90 abc	54 ghi	67 defghi	60 c			
216	70 ef	76 def	73 c	50 hi	59 fghi	55 c			
229	67 f	83 cdef	75 c	49 i	65 defghi	57 c			
251	84 cdef	86 cdef	85 bc	61 efghi	75 cdefg	68 bc			
Mean	89	87		74	72				

Table A1. Soil test K over time of cropped and fallow surfaces at site AB15.

‡ C, cropped; F, fallow.

§ Means followed by same letter are not statistically different for given comparison (surface, time, or surface × time), Tukey-Kramer HSD(0.05).

DOY†			Ι	OK			МК						
	C‡		F	F Me		C			F		Mean		
						— mg k							
141	90	cdef§	77	f	84	с	84	ab	81	ab	83	a	
155	116	ab	100	bcde	108	ab	83	ab	65	abcde	74	ab	
170	119	a	104	abcde	112	a	86	a	75	abcd	81	а	
188	94	cdef	88	def	91	abc	77	abc	82	ab	80	a	
203	99	bcde	103	abcde	101	abc	56	cdef	69	abcde	63	abc	
216	86	ef	90	cdef	88	bc	43	f	54	def	49	с	
229	94	cdef	105	abcd	100	abc	49	ef	65	bcdef	57	bc	
251	97	cde	107	abc	102	abc	56	cdef	80	ab	68	abc	
Mean	99		97				67		72				

Table A2. Soil test K over time of cropped and fallow surfaces at site AR15.

[†] DOY, day of year; DK and MK, NH₄OAc extractable K on air-dry and field-moist soil, respectively.

‡ C, cropped; F, fallow.

DOY†			Ι	ЭK				МК					
	C‡		F		Mean			С		F		Mean	
							mg kg	g ⁻¹					
141	159	bcd§	134	d	147	c		198	abc	177	abcde	188	ab
154	213	a	188	abc	200	a		199	ab	172	bcde	186	abcd
170	187	abc	210	ab	198	a		176	abcde	199	abc	187	abc
191	168	abcd	215	a	192	ab		155	bcde	246	a	200	a
209	143	cd	178	abcd	161	abc		112	e	168	bcde	140	d
222	145	cd	180	abcd	162	abc		121	de	162	bcde	141	bcd
236	148	cd	166	abcd	157	bc		127	cde	153	bcde	140	cd
251	152	cd	180	abcd	166	abc		135	bcde	186	abcd	160	abcd
Mean	164		181					153		183			

Table A3. Soil test K over time of cropped and fallow surfaces at site B15.

‡ C, cropped; F, fallow.

§ Means followed by same letter are not statistically different for given comparison (surface, time, or surface × time), Tukey-Kramer HSD(0.05).

DOY†			D	K			MK						
	С‡		F		Mean		С		F		Mean		
						— mg	g kg ⁻¹						
142	87	bc§	98	abc	92	ab	103	a	80	abc	91	a	
155	119	a	101	abc	110	a	96	ab	69	bc	82	ab	
170	106	ab	102	abc	104	ab	77	abc	81	abc	79	ab	
191	89	bc	84	c	86	b	71	abc	64	bc	67	ab	
209	103	abc	97	bc	100	ab	75	abc	65	bc	70	ab	
222	91	bc	100	abc	96	ab	56	c	80	abc	68	ab	
236	91	bc	100	abc	95	ab	57	c	68	bc	63	b	
251	107	ab	103	abc	105	ab	77	abc	81	abc	79	ab	
Mean	99		98				76		73				

Table A4. Soil test K over time of cropped and fallow surfaces at site C15.

[†] DOY, day of year; DK and MK, NH₄OAc extractable K on air-dry and field-moist soil, respectively.

‡ C, cropped; F, fallow.

DOY†			DK	_		MK					
	C‡		F		Mean	С		F		Mean	
						- mg kg ⁻¹					
141	109	ab§	102	ab	106	101	abc	97	abc	99	
155	116	ab	106	ab	111	112	a	96	abc	104	
170	112	ab	117	ab	115	110	ab	107	ab	108	
191	94	ab	120	ab	107	72	abc	108	ab	90	
209	87	b	114	ab	101	58	c	102	abc	80	
222	86	b	111	ab	98	63	bc	96	abc	80	
236	94	ab	125	а	109	82	abc	109	ab	95	
251	90	ab	122	ab	106	75	abc	113	a	94	
Mean	98	b	115	a		84	b	104	a		

Table A5. Soil test K over time of cropped and fallow surfaces at site D15.

‡ C, cropped; F, fallow.

§ Means followed by same letter are not statistically different for given comparison (surface, time, or surface × time), Tukey-Kramer HSD(0.05).

DOY†		DK			MK						
	C‡	F	Mean		С		F		Mean		
					— mg kg ⁻¹ -						
142	169	155	162	b§	143	а	123	abc	133	a	
155	185	188	186	a	136	ab	135	ab	136	a	
170	181	174	177	ab	138	ab	129	abc	134	a	
191	134	138	136	c	104	abcd	112	abcd	108	bc	
209	152	169	160	b	78	d	95	bcd	87	c	
222	157	189	173	ab	103	abcd	127	abc	115	ab	
236	152	192	172	ab	88	cd	119	abcd	103	bc	
251	152	177	165	ab	94	bcd	137	ab	116	ab	
Mean	160	173			111		122				

Table A6. Soil test K over time of cropped and fallow surfaces at site F115.

[†] DOY, day of year; DK and MK, NH₄OAc extractable K on air-dry and field-moist soil, respectively.

‡ C, cropped; F, fallow.

DOY†			D	Ж]	MK		
	C‡		F		Mean		С		F		Mean	
						- mg kg ⁻¹						
142	100	cdef§	118	abcde	109	b	82	c	101	abc	92	ab
155	124	abc	131	ab	128	а	89	bc	98	abc	94	ab
170	126	ab	127	ab	126	а	92	abc	101	abc	96	a
191	75	f	110	bcde	93	с	57	d	100	abc	78	abc
209	92	ef	117	abcde	104	bc	44	d	83	c	64	c
222	95	ef	142	а	118	ab	53	d	113	а	83	abc
236	97	def	121	abcd	109	b	54	d	95	abc	75	bc
251	94	ef	138	а	116	ab	59	d	109	ab	84	ab
Mean	100		125				66		100			

Table A7. Soil test K over time of cropped and fallow surfaces at site F215.

‡ C, cropped; F, fallow.

§ Means followed by same letter are not statistically different for given comparison (surface, time, or surface × time), Tukey-Kramer HSD(0.05).

DOY†		DK			MK					
	C‡	F	Mean		С	F	Mean			
				mg l	xg ⁻¹					
152	444	416	430	ab§	428	432	430	а		
170	462	507	484	а	332	425	378	ab		
187	363	463	413	b	370	455	413	a		
205	345	411	378	b	244	333	289	c		
225	370	458	414	b	322	383	352	b		
238	410	436	423	b	369	385	377	ab		
251	388	448	418	b	369	451	410	ab		
Mean	397	448			348	409				

Table A8. Soil test K over time of cropped and fallow surfaces at site LN15.

[†] DOY, day of year; DK and MK, NH₄OAc extractable K on air-dry and field-moist soil, respectively.

‡ C, cropped; F, fallow.

DOY†			D	K			МК						
	C‡		F		Mean		С		F		Mean		
						— mg k	g ⁻¹						
140	198	abc§	194	bcd	196	b	201	ab	198	ab	199	ab	
154	219	ab	225	а	222	a	210	а	203	ab	207	a	
175	176	cdef	209	ab	193	b	175	bcd	189	abc	182	bc	
191	148	fgh	178	cde	163	c	165	cde	168	cde	167	cd	
205	134	gh	155	efgh	144	cd	149	def	166	cde	157	de	
225	136	gh	167	def	151	cd	132	f	169	cde	151	de	
238	129	h	155	efgh	142	d	142	ef	176	bcd	159	de	
251	135	gh	163	efg	149	cd	132	f	162	cde	147	e	
Mean	159	b	181	а			163	b	179	a			

Table A9. Soil test K over time of cropped and fallow surfaces at site LS15.

‡ C, cropped; F, fallow.

§ Means followed by same letter are not statistically different for given comparison (surface, time, or surface × time), Tukey-Kramer HSD(0.05).

DOY†	DK								\mathbf{N}	IK		
	C‡		F		Mean		С		F		Mean	
						mg kg ⁻¹ –						
141	134	abcd§	110	cde	122	bcd	129	a	92	cd	111	a
154	151	а	136	abc	143	а	120	abc	99	bcd	109	а
175	150	а	121	bcde	136	ab	128	а	92	cd	110	а
191	140	ab	112	cde	126	abc	123	ab	92	d	107	а
209	113	bcde	104	e	109	d	76	de	61	e	68	b
222	113	bcde	113	bcde	113	cd	81	de	87	de	84	b
236	107	de	115	bcde	111	cd	85	de	90	d	87	b
251	114	bcde	127	abcde	121	bcd	81	de	94	cd	87	b
Mean	128		117				103		88			

 Table A10.
 Soil test K over time of cropped and fallow surfaces at site M15.

[†] DOY, day of year; DK and MK, NH₄OAc extractable K on air-dry and field-moist soil, respectively.

‡ C, cropped; F, fallow.

DOY†			Ι	ЭK			МК					
	C‡		F		Mean		С		F		Mean	
							– mg kg ⁻¹ –					
141	163	b§	136	c	149	c	143	a	106	bcdef	125	а
155	191	a	160	bc	175	a	133	ab	111	bcdef	122	a
170	173	ab	157	bc	165	ab	122	abcd	106	bcdef	114	ab
190	166	ab	153	bc	159	bc	123	abc	107	bcdef	115	ab
205	171	ab	169	ab	170	ab	90	f	95	def	93	c
216	166	ab	159	bc	163	abc	92	ef	100	cdef	96	bc
229	175	ab	176	ab	176	a	103	cdef	115	bcdef	109	abc
251	173	ab	174	ab	173	a	102	cdef	119	abcde	111	abc
Mean	172		160				114		108			

 Table A11.
 Soil test K over time of cropped and fallow surfaces at site P15.

‡ C, cropped; F, fallow.

§ Means followed by same letter are not statistically different for given comparison (surface, time, or surface × time), Tukey-Kramer HSD(0.05).

DOY†			D	K				MK						
	C‡		F	F			C		F		Mean			
						m	g kg ⁻¹							
140	203	bc§	191	bc	197	abc	234	ab	178	bc	206	ab		
154	255	a	186	bc	220	a	259	a	173	c	216	a		
170	224	ab	187	bc	205	ab	213	abc	170	c	191	abc		
189	209	bc	193	bc	201	abc	181	bc	177	bc	179	cd		
205	204	bc	200	bc	202	abc	163	с	170	c	166	cd		
216	177	c	179	c	178	с	156	с	168	c	162	d		
237	189	bc	189	bc	189	bc	183	bc	185	bc	184	bcd		
251	194	bc	193	bc	193	bc	183	bc	186	bc	184	bcd		
Mean	207		190				197		176					

 Table A12.
 Soil test K over time of cropped and fallow surfaces at site V15.

[†] DOY, day of year; DK and MK, NH₄OAc extractable K on air-dry and field-moist soil, respectively.

‡ C, cropped; F, fallow.

DOY†			D	Ж				МК						
	C‡		F		Mean		_	С		F		Mean		
						— mg	kg ⁻¹	l						
141	111	bcd§	110	bcd	111	abc		134	ab	134	ab	134	а	
154	120	abc	131	ab	125	a		130	ab	140	a	135	a	
175	112	bcd	121	abc	117	ab		123	abc	116	abcd	119	b	
191	95	cde	148	а	122	ab		89	def	138	a	113	b	
209	71	e	107	bcd	89	d		59	g	98	cde	79	d	
222	72	e	114	bcd	93	cd		69	fg	122	abc	95	c	
237	74	e	108	bcd	91	d		71	efg	107	bcd	89	cd	
251	86	de	122	abc	104	bcd		69	fg	119	abc	94	с	
Mean	93	b	120	a				93	b	122	a			

 Table A13.
 Soil test K over time of cropped and fallow surfaces at site W15.

‡ C, cropped; F, fallow.

§ Means followed by same letter are not statistically different for given comparison (surface, time, or surface × time), Tukey-Kramer HSD(0.05).

DOY†		DK					M	K	
	C‡	F	Mean		С		F		Mean
					mg kg ⁻¹ ·				
137	163	170	166	a§	115	bcdefg	113	bcdefg	114
153	154	161	157	abc	116	bcdefg	126	abcde	121
168	157	167	162	ab	130	abc	132	ab	131
181	151	163	157	abc	124	abcdef	129	abcd	127
194	146	164	155	abc	105	defg	129	abcd	117
209	139	160	149	abc	105	defg	124	abcdef	115
224	129	156	142	c	100	fg	143	a	122
237	141	168	154	abc	102	efg	137	ab	119
252	138	158	148	bc	107	cdefg	141	а	124
267	147	176	161	ab	97	g	146	a	122
Mean	146	164			110		132		

 Table A14.
 Soil test K over time of cropped and fallow surfaces at site AK16.

† DOY, day of year; DK and MK, NH4OAc extractable K on air-dry and field-moist soil, respectively.

‡ C, cropped; F, fallow.

DOY†			J	DK				МК						
	C‡		F		Mean		С		F		Mean			
	-						- mg kg ⁻¹ —							
137	84	a§	75	abc	80	а	56	bcdefg	49	defg	52	cd		
153	75	abc	87	а	81	a	54	cdefg	69	abcd	62	bc		
168	77	abc	83	ab	80	а	74	abc	79	а	77	а		
181	69	abcde	84	ab	76	ab	54	cdefg	73	abc	64	b		
194	66	bcdef	77	abc	71	abc	45	efg	65	abcde	55	bcd		
209	56	def	77	abc	67	bc	43	efg	71	abcd	57	bcd		
224	48	f	74	abcd	61	c	40	fg	73	abc	56	bcd		
237	52	ef	84	а	68	bc	37	g	80	а	58	bcd		
252	53	ef	79	ab	66	bc	39	fg	78	ab	59	bc		
267	59	cdef	77	abc	68	bc	35	g	62	abcdef	48	d		
Mean	64	b	80	а			48	b	70	a				

 Table A15.
 Soil test K over time of cropped and fallow surfaces at site CF16.

† DOY, day of year; DK and MK, NH4OAc extractable K on air-dry and field-moist soil, respectively.

‡ C, cropped; F, fallow.

§ Means followed by same letter are not statistically different for given comparison (surface, time, or surface × time), Tukey-Kramer HSD(0.05).

DOY†			Ι	OK						MK		
	С‡		F		Mean		С		F		Mean	
							mg kg ⁻¹					
138	84	abcde§	86	abcd	85	a	55	cdefgh	56	bcdefgh	56	c
153	83	abcdef	79	bcdefg	81	ab	63	bcdefgh	60	bcdefgh	62	abc
168	78	cdefgh	83	abcdef	81	ab	70	abcde	64	bcdefgh	67	ab
181	76	cdefgh	82	bcdef	79	ab	62	bcdefgh	68	abcdefg	65	abc
194	74	defghi	90	ab	82	ab	54	defgh	73	abcd	63	abc
209	64	ij	83	abcdef	74	bc	50	gh	74	abc	62	abc
224	61	j	72	efghij	67	c	52	efgh	75	ab	64	abc
237	66	hij	83	abcdef	75	bc	51	fgh	73	abcd	62	abc
252	68	ghij	94	a	81	ab	53	efgh	86	а	69	a
267	72	fghij	87	abc	80	ab	49	h	70	abcdef	59	bc
Mean	73		84				56		70			

 Table A16.
 Soil test K over time of cropped and fallow surfaces at site GD16.

† DOY, day of year; DK and MK, NH4OAc extractable K on air-dry and field-moist soil, respectively.

‡ C, cropped; F, fallow.

DOY†				DK						MK		
	C‡		F		Mean		С		F		Mean	
	-					mg kg ⁻¹						
137	69	abcd§	76	ab	73	а	45	defg	52	cdef	48	bcd
153	61	cdefg	63	abcdef	62	bc	51	cdef	52	cdef	51	abc
168	68	abcd	67	abcd	68	ab	57	bcde	67	ab	62	а
181	62	bcdefg	66	abcde	64	abc	44	efg	59	bcd	51	abc
194	52	efg	78	a	65	abc	42	fgh	62	abc	52	abc
209	50	fg	64	abcdef	57	c	39	fgh	60	bc	50	abcd
224	47	g	70	abcd	59	bc	43	efg	75	а	59	ab
237	51	fg	69	abcd	60	bc	38	fgh	67	ab	52	abc
252	56	defg	76	ab	66	ab	32	gh	60	bc	46	cd
267	57	defg	74	abc	66	abc	27	h	48	cdef	37	d
Mean	57		70				42		60			

 Table A17.
 Soil test K over time of cropped and fallow surfaces at site LB16.

‡ C, cropped; F, fallow.

§ Means followed by same letter are not statistically different for given comparison (surface, time, or surface × time), Tukey-Kramer HSD(0.05).

DOY†			DK				МК						
	C‡		F		Mean		С		F		Mean		
						– mg kg ⁻¹ -							
137	114	a§	93	bc	103	a	69	abc	59	bcdef	64	bc	
153	88	bcd	81	bcdefg	84	bc	66	abcd	58	cdef	62	bc	
168	88	bcd	90	bcd	89	b	78	a	75	ab	76	а	
181	81	bcdefg	84	bcdefg	82	bc	56	cdef	65	abcde	61	bc	
194	84	bcdefg	94	b	89	b	57	cdef	67	abc	62	bc	
209	70	fg	82	bcdefg	76	c	50	ef	67	abc	59	bc	
224	70	g	79	cdefg	74	c	57	cdef	77	а	67	ab	
237	71	fg	88	bcd	79	bc	50	def	69	abc	60	bc	
252	73	efg	85	bcde	79	bc	45	fg	62	abcde	54	c	
268	77	defg	87	bcdef	82	bc	32	g	45	fg	38	d	
Mean	81		86				56		65				

 Table A18.
 Soil test K over time of cropped and fallow surfaces at site MO16.

† DOY, day of year; DK and MK, NH4OAc extractable K on air-dry and field-moist soil, respectively.

‡ C, cropped; F, fallow.

DOY†		DK			МК						
	C‡	C‡ F		Mean		F	Mean				
				m	g kg-1						
137	201	209	205	a§	171	182	176	abc			
153	194	204	199	ab	181	184	183	abc			
168	187	202	195	ab	192	192	192	ab			
181	215	200	208	а	204	204	204	а			
194	184	197	190	ab	158	190	174	abc			
209	175	190	182	b	155	175	165	bc			
224	172	190	181	b	160	181	170	bc			
237	181	205	193	ab	173	214	194	ab			
252	173	200	187	ab	159	199	179	abc			
267	180	207	193	ab	144	174	159	с			
Mean	186	201			170	190					

 Table A19.
 Soil test K over time of cropped and fallow surfaces at site VC16.

 \dagger DOY, day of year; DK and MK, NH4OAc extractable K on air-dry and field-moist soil, respectively.

‡ C, cropped; F, fallow.