

HUMIDIFICATION FOR ICAP-AES ANALYSIS OF
SALT EXTRACTS OF SOILS AND COMPARISON
OF SOIL EXTRACTION PARAMETERS
FOR AL, MN, CA, AND MG
DETERMINATIONS

By

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INTRODUCTION

There are two parts to this thesis which involve two separate studies. The first study is laboratory research in which strong salt soil extracts were analyzed using inductively coupled argon plasma atomic emission spectroscopy both with and without argon humidification compared with conventional laboratory test methods. Twenty-five soils were extracted with three strong salt soil extracting solutions and analyzed by three methods.

The second part is laboratory research comparing soil extraction parameters for aluminum, manganese, calcium, and magnesium determinations. Soil extracts were analyzed using inductively coupled argon plasma atomic emission spectroscopy. Two soils were studied using four potassium chloride concentrations, four extraction times, and three soil:solution ratios.

Both parts are presented in a format suitable for publication in *Communications in Soil Science and Plant Analysis*. Laboratory data for both studies are in the appendixes.

PART I
HUMIDIFICATION FOR ICAP-AES ANALYSIS
OF SALT EXTRACTS OF SOILS

ABSTRACT: Use of inductively coupled argon plasma atomic emission spectroscopy (ICAP-AES) has become common in many laboratories for analysis of soils, plants, and water. Major advantages offered by ICAP-AES include rapid simultaneous multi-element analysis. Preliminary investigation showed that results differ between analysis by conventional and ICAP-AES methods. Nebulization problems with strong salt solutions, torch design, and the extreme dryness of the argon gas were all considered to be contributing factors. A Jarrell-Ash¹ Model 9000 ICAP-AES instrument was retrofitted with an argon humidification system and studies were conducted using Mehlich III, 1 M potassium chloride, or 1 M ammonium acetate soil extracts. The results showed that estimates of K, Ca, and Mg concentrations were similar among extracts, with or without humidification. Results for P were lower with humidification than without, but consistently higher compared with conventional colorimetric P analysis. ICAP-AES with or without humidification, provided similar results for Al and Mn; however, results for both elements were lower than those obtained by atomic absorption.

Statistical analysis showed significant differences among methods of

¹Thermo Jarrell Ash Corporation, P.O. Box 9101, Franklin, MA 02038-9101.

analysis regardless of extract. Differences occurred in K and Mg but not Ca when comparing humidification with conventional ICAP analysis.

INTRODUCTION

Inductively coupled argon plasma emission spectroscopy provides several advantages over atomic absorption spectrophotometry (AAS), such as simultaneous multiple element analysis, shorter sampling time, and ability to determine elements which must be otherwise determined by cumbersome wet chemistry methods. However, in order to prove useful, results obtained with ICAP-AES must be comparable in reliability to those obtained by more traditional methodology.

Although ICAP-AES has become the preferred method of analysis for a wide variety of sample types during the past several years, little information is available on analytical problems associated with analyses of soils using concentrated salt extraction solutions. There appears to be agreement among laboratories that the main problem of these solutions is associated with salt accumulations or deposits in or on the sample introduction apparatus of the ICAP-AES. (1,2,3)

While different models of ICAP-AES are based on common principles, variations in features such as torch design, mixing chamber design, and type of nebulizer exist from instrument to instrument. Some

instruments also have a built-in humidification system for the aerosol carrier argon gas while others do not.

This study was conducted to compare analyses obtained by ICAP-AES with or without humidification, with conventional analytical procedures for three strong salt extracting solutions obtained from soils.

MATERIALS AND METHODS

Twenty-five soils were selected from the Oklahoma State University Agronomic Services Laboratory reserve sample bank. Soil samples with pH (1:1 soil:water) values less than 5.0 were chosen to insure that measurable amounts of Al and Mn would be present in the 1 M KCl extract. Initial soil preparation included drying at 75°C for 12 hours and grinding in a Dynacrush soil crusher so that samples would pass a 2 mm screen. All samples were extracted with Mehlich III (M III) (4), 1 M KCl, or 1 M ammonium acetate (NH₄OAC) (5). Potassium, Ca, and Mg were determined in both M III and NH₄OAC extracts using AAS and ICAP-AES. Phosphorus (P) was determined in M III by ICAP-AES and colorimetrically using the modified procedure of Murphy and Riley (6,7). Aluminum and Mn in KCl extracts were determined by ICAP-AES and AAS.

A 1.8 cm³ scoop was used to place soil samples into 50-ml Erlenmeyer flasks. The soil:solution ratios were 1:10, 1:5, and 1:5 for M

III, NH_4OAC , and KCl , respectively. Soils extracted with M III and NH_4OAC samples were shaken on an Eberbach 6150 rotating shaker (240 rpm) for 5 minutes before filtering through Whatman No. 2 filter paper. Soils extracted with KCl were shaken for 10 minutes on the same rotary shaker and filtered through Whatman No. 42 filter paper. All filtrates were collected in 100-ml plastic cups (Solo P35A) and transferred to glass 50-ml test tubes for storage until analyzed.

Conventional Test Methods--AAS and Colorimetric: Potassium, Ca, and Mg were determined in soil extracts using a Perkin-Elmer 2380 atomic absorption spectrophotometer and hollow cathode lamps specific for each element. A single-slot burner head was placed at a 30° angle. The air:acetylene ratio was adjusted to produce a lean, blue flame for all determinations. Aliquots of the extracts to analyzed for Ca and Mg were diluted with lanthanum chloride (LaCl_3) solution to give a final concentration of 1% LaCl_3 . All spectrophotometer settings are listed in Table 1. Aluminum and Mn were determined using a Perkin-Elmer 3030B. Manganese was analyzed using a lean, blue flame; the single-slot burner head was installed so that the light emitted from the hollow cathode tube traveled the entire length of the burner head (0° angle). Aluminum was analyzed using nitrous oxide:acetylene rich, red (reducing) flame with the burner head at a 0° angle.

TABLE 1. Spectrophotometer Settings for AAS.

Element	Wavelength nm	Slit Width mm
K	766.5	2.0
Ca	422.7	0.7
Mg	285.2	0.7
Al	309.3	0.7
Mn	279.5	0.2

A flow spoiler was used in the mixing chamber for all AAS analyses. The flame type, horizontal and vertical burner head positions, wavelength, and position of the hollow cathode lamp were optimized. After optimization, the instrument was set to give a 3-second average reading. After standardization of the blank extracting solution, two known standards were used to set the instrument and a third mid-range standard was used for verification of linearity. The standards were then analyzed again in sequence prior to analyzing the unknown samples. All 25 soil extracts were analyzed before standards were rechecked. The same procedure was followed for each of 3 replications.

ICAP-AES Methods: The ICAP-AES analysis procedure was the same with humidification (ICAP-h) and without humidification (ICAP). The analytical zone was set by aspirating a yttrium nitrate solution and adjusting the sample flow so that the characteristic red zone was set to ± 1 mm from the top of the torch. The horizontal torch position was set by

profiling the instrument with a cadmium solution for maximum gain reading. The vertical torch position was set by determining intensity count ratios of a cadmium solution/deionized water at various positions and setting the torch at the position with the highest ratio. A proportioning pump was used to introduce the solutions into the instrument. Instruments settings for forward R.F. power, torch argon flow, and sample flow were 1.3 kW, 17 liters/min, and 1.6 ml/min, respectively. Auxiliary argon was not used in the non-humidified method but was used for humidified samples at the rate of 1.0 liter/min since the analytical zone set with the yttrium nitrate solution could not be obtained without the additional argon flow. Sample exposure time was 0.7 minutes. The baffle was used in the spray chamber and was positioned 38 mm from the drain hole. The cross-flow nebulizer was used for all extracts. A standard size Jarrell Ash type torch, modified to help prevent salts from depositing at the tip, was used. Modifications included shortening the sample introduction tube by approximately 2 mm, and slightly widening the tip of the sample introduction tube. A N₂ gas purge was used during all analyses. Wavelengths used were 308.21, 766.49, 317.93, 279.08, 257.61, and 178.29 nm for Al, K, Ca, Mg, Mn, and P respectively.

An ARL² humidifier was fitted on the sample argon line for humidified analysis. The set-up consisted of a check valve, the humidifier,

²Applied Research Laboratories, 15300 Rotunda, Suite 301, Dearborn, MI 48120.

another check valve, an excess water trap, and a 2 μ in-line filter. The excess water trap consisted of a 125-ml. plastic bottle heavily wrapped with strapping tape.

The ICAP-AES was standardized with blank extracting solutions and the same high analytical standard used for AAS analysis. The low-and mid-range standards were used for verification after the standardization procedure was completed. All 25 samples were analyzed by averaging four 10-second exposures on each sample. Standards were analyzed again after the last sample to check for drift and corrections were made if necessary.

All analytical results were entered into a personal computer and statistical evaluation was performed using the TurboStat software package (8).

RESULTS AND DISCUSSION

K, Ca, Mg, and P: Significant differences ($\alpha = 0.01$) existed for K and Mg determinations in M III and NH_4OAC extracts by ICAP-AES due to method which included with or without humidification but no differences for Ca were detected. A method x soil interaction was not observed for K or Ca. Interactions were significant for extract x soil, method x extract, and method x soil x extract. F-values are presented in Table 2.

Humidification caused additional problems in operating the instrument, including longer system equilibration time, extreme difficulty in

TABLE 2. F-tests of Effects of Methods of ICAP-AES Analysis, Extracts, and Soils on K, Ca, and Mg Concentrations.

Source	df	F Values		
		K	Ca	Mg
Reps	2			
Methods (M)	1	226.**	0.1	369.**
Extract (E)	1	1031.**	762.**	1351.**
Soils (S)	24	6143.**	3458.**	>10000.**
M x E	1	143.**	99.2**	249.**
M x S	24	0.9	0.3	6.0**
E x S	24	15.1**	18.6**	32.1**
M x E x S	24	3.9**	1.5*	8.4**
EMS***	198			

*, ** indicates significance at $\alpha = 0.005, 0.05$, respectively.
***Error mean square values for K, Ca, and Mg were 10.62, 3173.39, and 67.83, respectively.

igniting the plasma, and an audible noise that occurred at random. Even though this noise does not seem to affect instrument results, occasionally it was associated with loss of the plasma which in turn caused down-time for system re-equilibration and re-standardization.

Significant differences ($\alpha = 0.01$) occurred between humidified and non-humidified ICAP-AES analysis for K and Mg but there were no differences in Ca analyses due to method. Concentrations of K, Ca, and Mg differed significantly ($\alpha = 0.01$) with extractants and soils. F-Values for the analysis of variance evaluating the effect of methods of analysis within extracts are presented in Table 3. While analysis of variance indicates significant differences among methods of analysis, comparison of

TABLE 3. Significant F-Values for Methods within Extracts.

Source	df	F-Values			
		K	Ca	Mg	P
<u>M III</u>					
Reps	2				
Methods (M)	2	22.4**	14.8**	51.3**	3309.**
Soils (S)	24	2661.**	1740.**	>10000.**	2670.**
M x S	48	1.4*	2.7**	4.5**	258.**
EMS	148				
<u>NH₄OAC</u>					
Reps	2				
Methods (M)	2	1850.**	19.6**	>10000.**	--
Soils (S)	24	>10000.**	1338.**	>10000.**	--
M x S	48	20.8**	4.3**	1394.**	--
EMS	148				

*, ** indicate significance at $\alpha = 0.05, 0.01$, respectively.
 ***Error mean square values for M III K, Ca, Mg, and P were 20.66, 5573.39, 61.48, and 102.45, respectively. For NH₄OAc EMS values for K, Ca, and Mg, were 1.45, 5414.71, and 5272, respectively.

means in Table 4 provide additional insight regarding the practical importance of these differences. With the exception of Mg in NH₄OAc extract determined by AAS and P extracted in M III extract and analyzed colorimetrically, very small differences were found between method of determination for K, Ca, Mg, and P for a given extract. These small differences in concentrations would not affect fertilizer recommendations significantly.

Mehlich III P was significantly different ($\alpha = 0.01$) among analytical methods and soils. A significant interaction between methods x soils was

TABLE 4. Mean K, Ca, Mg, and P* ($\mu\text{g/g}$) in Mehlich III and NH_4OAc Soil Extracts Determined by Conventional, ICAP-h, and ICAP Methods of Analysis.

	K		Ca		Mg		P
	M III	NH_4OAc	M III	NH_4OAc	M III	NH_4OAc	M III
Conventional**	85.3	79.9	882.8	811.5	172.4	230.4	53.7
ICAP-h	83.5	79.7	867.2	809.8	167.8	157.9	110.1
ICAP	83.0	74.6	900.3	778.1	166.2	141.2	113.6
LSD (0.05)	0.7	0.2	12.1	11.9	1.3	1.2	1.6

*Only M III data is available for P.

**Conventional is AAS for all elements except P which is colorimetric.

also present. Phosphorus results were higher using ICAP and ICAP-h than for colorimetric analysis. This difference was attributed to complexed P included by ICAP-AES which was not detected with the colorimetric method. Examination of mean values indicate that more variation occurred above $50 \mu\text{g P/g}$ of soil, which is well above the point at which fertilizer recommendations are made.

Regression analyses were performed and correlation coefficients were determined between extracts and among methods. (Table 5 and Table 6)

The 25 soils were ranked in ascending element concentration so that visual comparisons among methods could be made for each element. Results are presented in Figures 1 and 2.

Aluminum and Mn: Use of 1 M KCl extracts for Al and Mn determination presented additional difficulties with and without humidification using ICAP-AES. The KCl soil extracts often extinguished the plasma as soon as the solution was introduced to the plasma. Salts from the extracts deposited at the sample tube tip causing variations in concentrations detected in the extract. Concentrations were generally lower but sometimes increased possibly due to "flaking" of the salts which would introduce more sample into the plasma. With humidification the problems were only lessened in severity. A comparison was made using a 0.5 M KCl soil extract since it was felt that ICAP results could be obtained

TABLE 5. Correlation Coefficients (r) Among Methods of Analysis for K, Ca, Mg, and P Within Extracts.

	M III											
	AAS				ICAP-h				ICAP			
	K	Ca	Mg	P	K	Ca	Mg	P	K	Ca	Mg	P
AAS/P*	0.99	0.99	1.00	0.89	1.00	0.99	1.00	1.00
ICAP-h	0.98	1.00	1.00	0.89
ICAP
	NH ₄ OAC											
	AAS				ICAP-h				ICAP			
	K	Ca	Mg	P	K	Ca	Mg	P	K	Ca	Mg	P
AAS/P*	1.00	0.98	0.96		1.00	0.98	0.97	
		1.00	1.00	1.00	
	

*Colorimetric analysis of P.

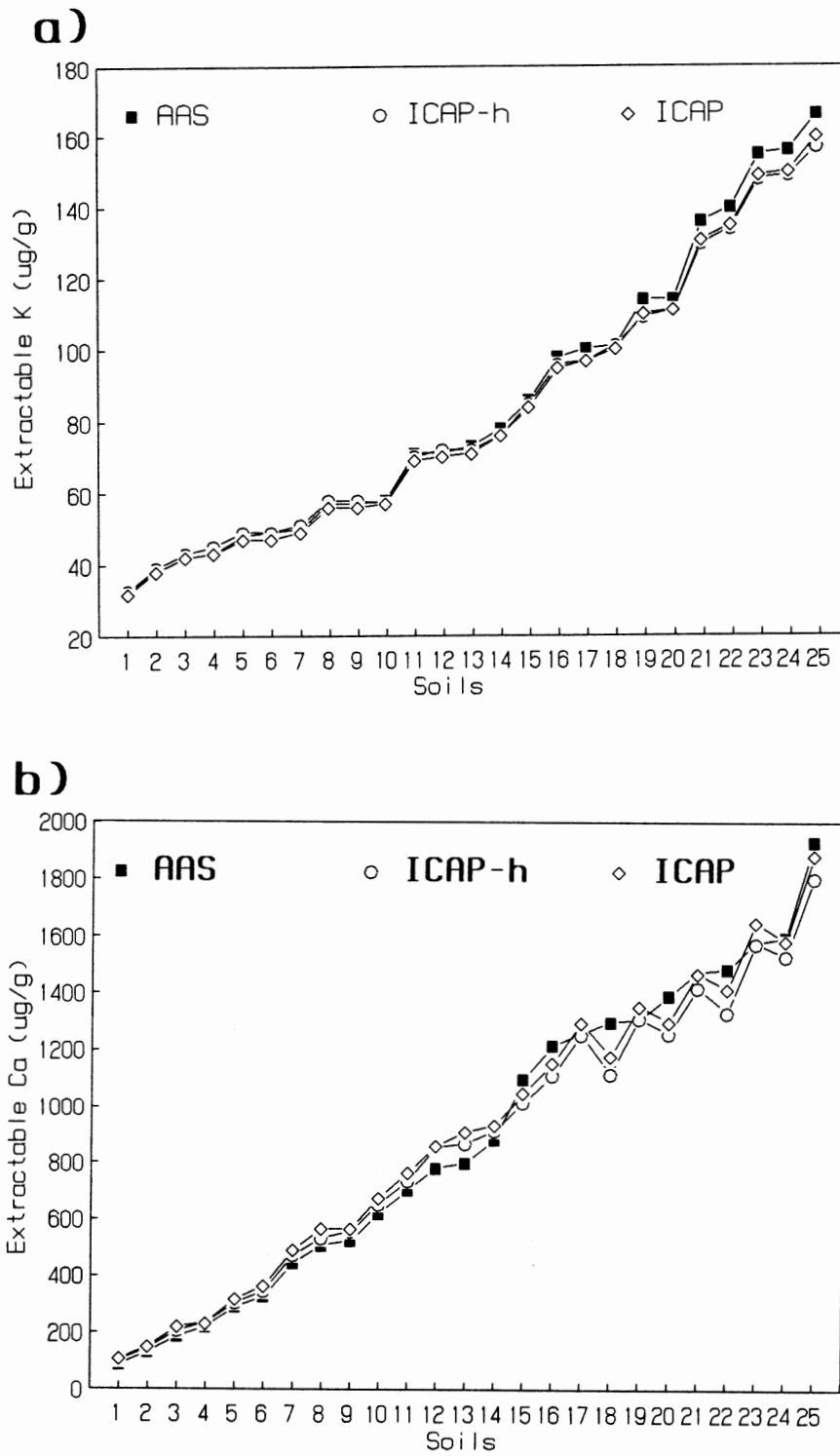
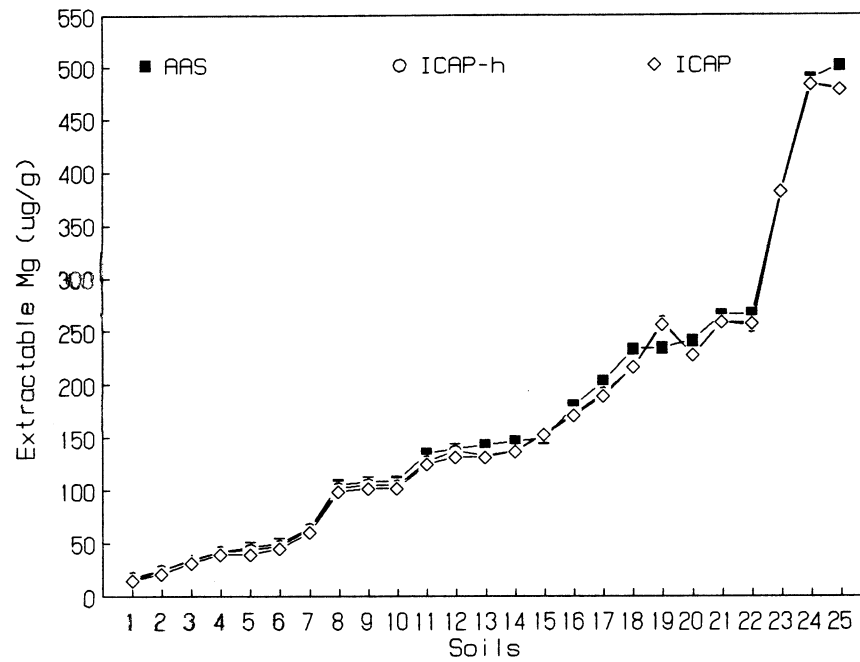


FIGURE 1. Mean Concentrations of Mehlich III Extractable K (a), Ca(b), Mg (c), and P (d) for AAS-Conventional, ICAP-h, and ICAP.

c)



d)

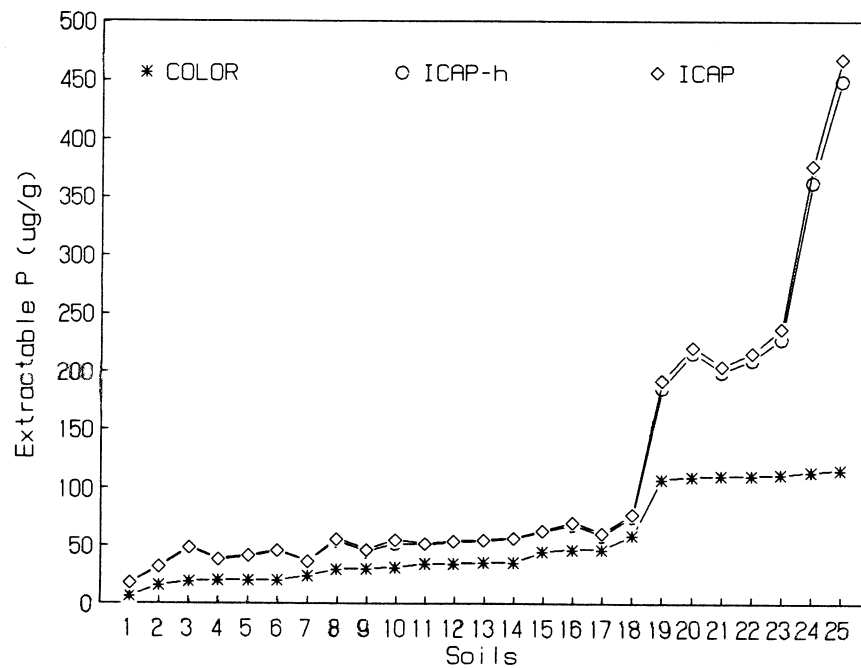


FIGURE 1. (continued)

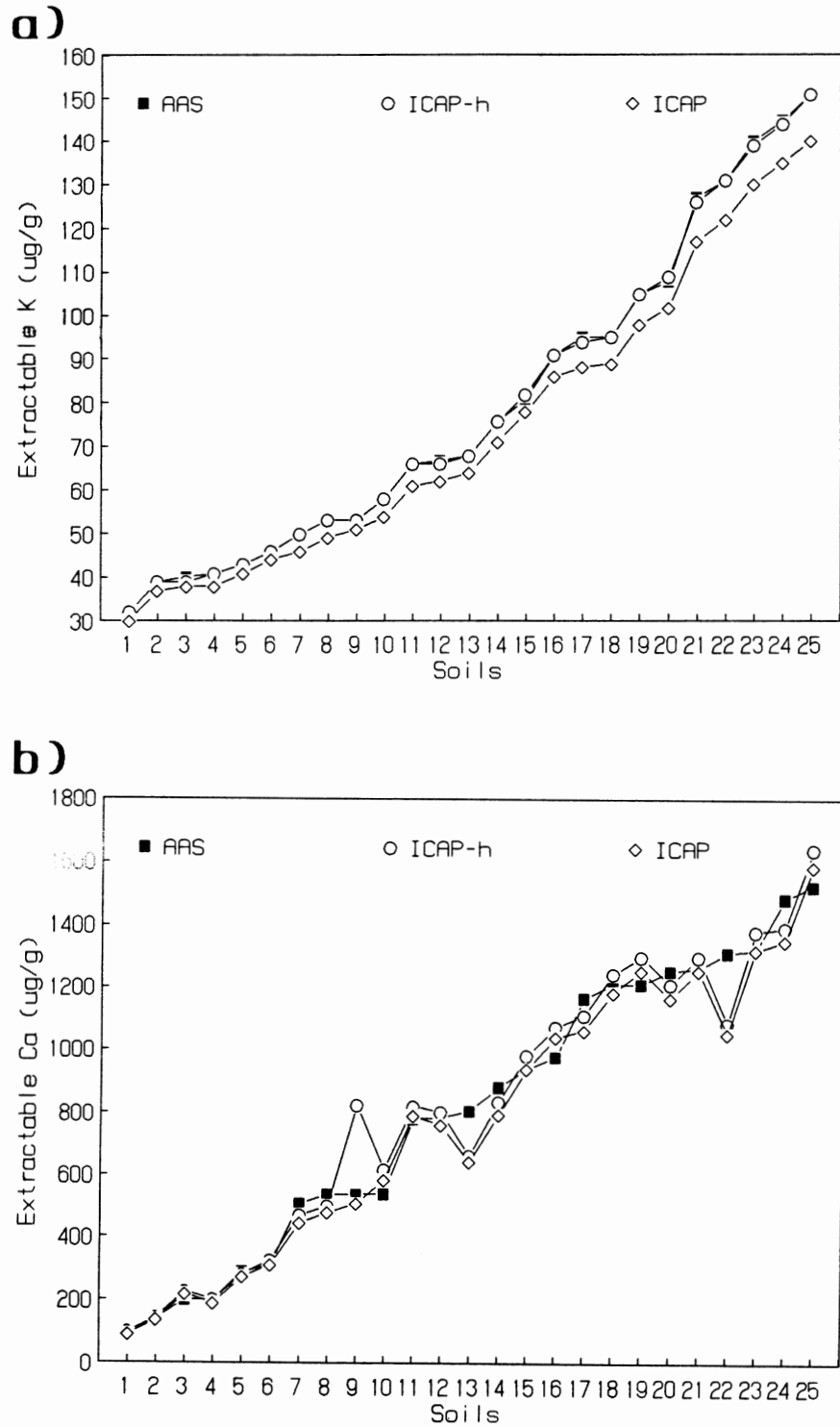


FIGURE 2. Mean Concentrations of Ammonium Acetate Extractable K (a), Ca (b), and Mg (c) for AAS, ICAP-h, and ICAP.

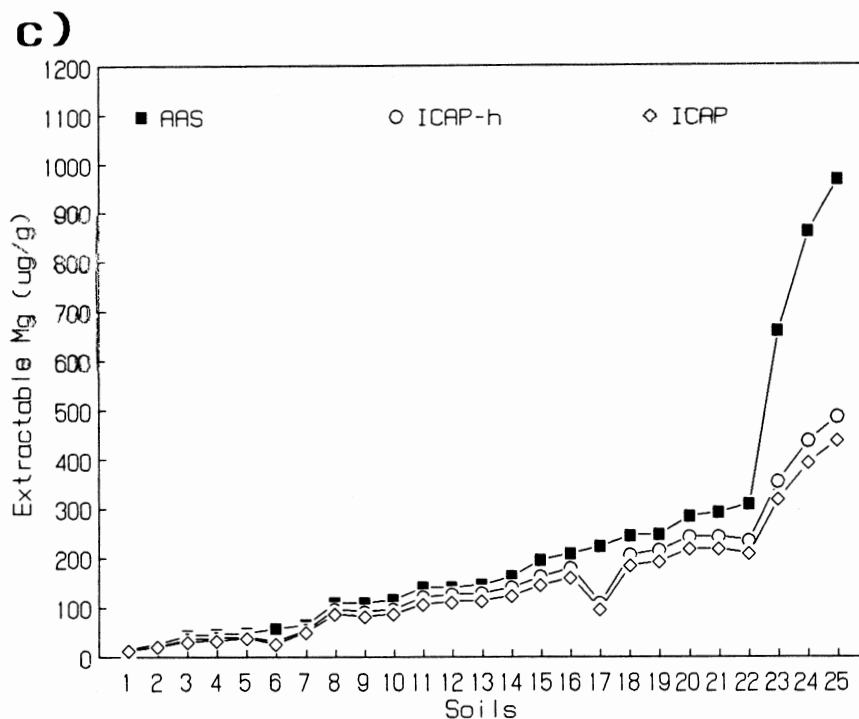


FIGURE 2. (continued)

TABLE 6. Correlations Coefficients for Extracts within Methods (Mehlich III compared to Ammonium Acetate).

Element	AAS	ICAP-h	ICAP
K	1.00	1.00	1.00
Ca	0.98	1.00	1.00
Mg	0.96	1.00	1.00

more quickly using the more dilute solution. Table 7 contains the simple linear regression equations and correlation coefficients for comparison of 0.5 M and 1.0 M extracts for all methods. Of the two solutions, 0.5 M KCl

TABLE 7. Regression Equations and Correlation Coefficient (r) Values Between 0.5 M KCl and 1.0 M KCl Soil Extracts for Al and Mn Analysis Using AAS, ICAP-h, and ICAP.

	AAS		ICAP-h		ICAP	
	Al	Mn	Al	Mn	Al	Mn
Intercept	-6.01	0.04	-2.70	0.92	-0.72	-0.65
Slope	0.96	0.97	0.96	1.04	0.93	1.08
r	0.99	0.99	0.99	0.98	0.98	0.99

with humidification caused the fewest problems with ICAP-AES. The analysis of variance of Al and Mn concentrations in 0.5 M KCl using all analytical methods is shown in Table 8.

TABLE 8. Analysis of Variance for Analysis by AAS, ICAP-h, and ICAP.

Source	df	F Values	
		Al	Mn
Reps	2		
Methods (M)	2	13.6**	201.**
Soils (S)	24	916.**	770.**
M x S	48	2.0**	4.5**
EMS	148		

** indicates significance at $\alpha = 0.01$.

***Error mean square values for Al and Mn were 5573 and 61.5, respectively.

Methods of analyses for the determination of both Al and Mn were significantly different ($\alpha = 0.005$). A significant interaction between

methods and soils was present. Means for Al analyzed by AAS, ICAP-h, and ICAP were 64.0, 59.5, and 60.9 $\mu\text{g/g}$ of soil, respectively with a LSD (0.05) of 2.3. Means for Mn using AAS, ICAP-h, and ICAP were 38.3, 32.2, and 32.5 $\mu\text{g/g}$, respectively with a LSD (0.05) of 0.7. Correlation coefficients (r) for Al\Mn were 0.99\0.99, 0.98\0.99, and 0.99\0.98 for AAS vs ICAP-h, AAS vs ICAP, and ICAP vs ICAP-h. Figure 3 contrasts methods for determination of Al and Mn in 1 M KCl extracts.

CONCLUSIONS

Due to differences encountered when strong salt soil extracting solutions were analyzed using ICAP-AES a study was made of 3 methods, AAS and colorimetric P, ICAP-h, and ICAP. Three extractants, M III, KCl, and NH_4OAC , were used to extract 25 Oklahoma soils. Differences existed between ICAP-h and ICAP for K and Mg analysis regardless of extract. There were no differences in Ca determinations due to method of analysis. Within each extract, significant differences were detected for all elements tested. Examination of means shows that for the soils tested, differences would not change fertilizer recommendations.

Based on this study, ICAP without humidification would be recommended for all elements except M III P tested in each of the three extracts. For P, conventional colorimetric methods would be preferred due to the higher results obtained by ICAP-AES.

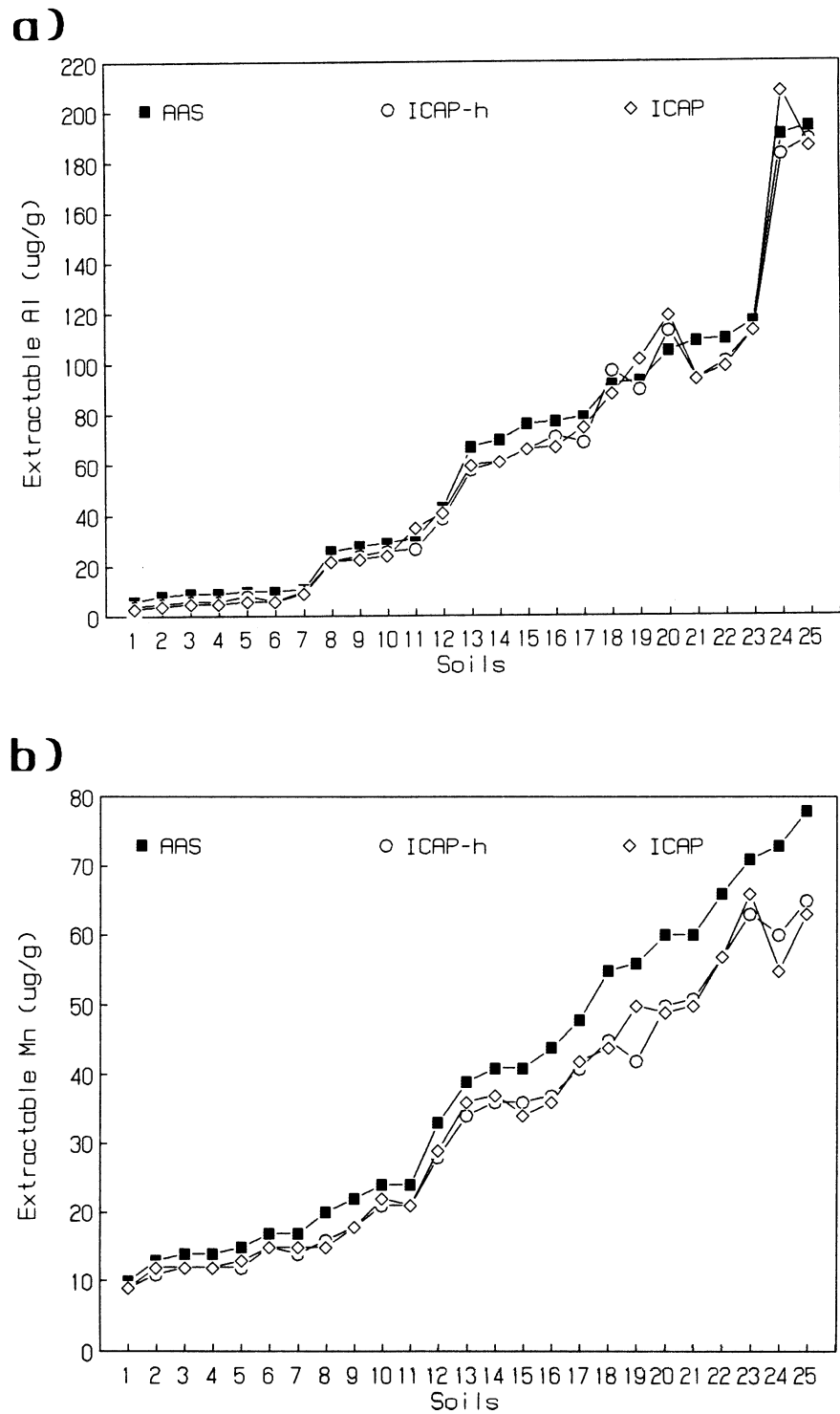


FIGURE 3. Mean Concentrations of 0.5 M Potassium Chloride Extractable Al (a) and Mn (b) for AAS, ICAP-h, and ICAP.

Additional study is needed before determining whether 0.5 M KCl can be used to extract Al and Mn from soils with low organic matter contents (≤ 10 mg/g). Additional study is also needed to determine whether torch design modification and spray chamber modification could reduce the magnitude of differences observed in analyses with strong salt extracting solutions by ICAP-AES.

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PART II
COMPARISON OF SOIL EXTRACTION PARAMETERS
FOR AL, MN, CA, AND MG DETERMINATIONS

ABSTRACT: Inductively coupled argon plasma atomic emission spectroscopy (ICAP-AES) is frequently used for analysis of soil extracts. Advantages include increased detection limits, simultaneous multi-element capabilities, speed, and operator safety due to inert, non-combustible source gas. Previous study showed that instrumentation problems increased with increasing salt content in soil extracting solutions. A Jarrell Ash³ Model 9000 ICAP-AES was used to analyze Al, Mn, Ca, and Mg in KCl soil extracts in a study conducted to examine KCl concentration, soil:solution ratio, and extraction time for two acidic soils in Oklahoma. KCl concentration and soil:solution ratio resulted in differences of extractable Al, Mn, Ca, and Mg in both soils. Extraction time did not effect Al concentration in either soil, but did effect concentration of Mn extracted. Concentration of extractable Ca and Mg varied in one of the soils as a result of extraction time. The most effective extraction parameters selected based on these soils were 0.5M KCl concentration, 10 minute extraction time, and 1:15 soil:solution ratio.

INTRODUCTION

Acid soils are one of the major limiting factors in crop production in the world today (1,2). A number of studies have shown that large amounts

³Thermo Jarrell Ash Corporation, P.O. Box 9101, Franklin, MA 02038-9101.

of plant available Al, and to a lesser extent Mn, are released as soil pH is acidified below 4.5 (3,4,5,6). Liming soils to correct extreme acidity has been a common practice since ancient times.

The SMP Buffer Index (7) is frequently used in routine laboratory analysis to indicate the amount of lime needed to raise the soil pH to 6.8; however, no indication is given for which pH lime application is essential to avoid significant yield reduction or even crop loss. Some studies point out that the SMP buffer lime recommendations are too high when soils require low levels of lime (8). Other work shows that the SMP-estimated amount of lime can even exceed the total acidity in soils (9). Therefore, some attention needs to be given to other analytical testing procedures to determine if perhaps a better indicator for lime application can be developed.

Work by Haynes and Swift (10) showed that as soil pH levels decreased, 1M KCl extractable Al increased. However, additional study is needed to determine whether KCl extractable Al can be useful as an indicator of when a "critical" pH level is being approached. In order to provide a feasible test, such an indicator must not only be accurate and reproducible, but also be rapidly carried out in the laboratory. Previous study has indicated some of the complexities associated with selecting extraction parameters (11, 12). Previous work showed that ICAP-AES could be useful for quick analysis of KCl extracts, but that increasing salt

content of the extracting solution tended to increase instrumentation problems (13,14,15,16).

The objective of this study was to determine soil:solution ratios, extraction times, and KCl concentrations that effectively extract Al, Mn, Ca, and Mg for ICAP-AES analysis.

MATERIALS AND METHODS

Soils from two locations were selected for use in this investigation. Soil I, a Teller fine sandy loam soil (fine-loamy, mixed, thermic Udic Arguistoll) was obtained from a site on the Oklahoma State Agricultural Experiment Station located at Perkins, Oklahoma. Soil II, a Pond Creek silt loam soil (fine-silty, mixed, thermic Udic Arguistoll) was obtained from a long-term cooperated field located near Carrier, Oklahoma. Characteristics of each soil are presented in Table 1. Each soil was air-dried, ground in a Dynacrush⁴ soil crusher, and sieved to pass a 2 mm screen. Experimental design was a split-split plot of four KCl concentrations, four extraction times, and three soil:solution ratios replicated three times.

A 1.8 cm³ scoop was used to place soil into 50-ml Erlenmeyer flasks. Soil:solution ratios (v:v) used were 1:5, 1:10, and 1:15. Concentrations of KCl were 0.125, 0.250, 0.500, and 1.000 M. Samples were placed on an

⁴Custom Laboratory Equipment, Inc. P.O. Box 757, Orange City, FL 32763

TABLE 1. Soil Characteristics* of the Teller and Pond Creek Soils.

	1:1	1:1	SMP	O.M.	C.E.C.	NO ₃ -N	Index	
	H ₂ O pH	KCl pH					B.I.	%
Soil I	4.5	4.0	6.6	1.03	7.2	14	130	363
Soil II	4.8	4.1	6.9	0.75	5.0	60	51	708

*Procedure used for cation exchange capacity was modified from Bower, et al. (17). NO₃-N was analyzed in 0.01 N CaSO₄ soil extract. P and K were analyzed in Mehlich III extract. Actual procedures are discussed in the Oklahoma State University Agronomic Services Laboratory Procedures Manual (18).

Eberbach⁵ 6150 rotating shaker (240 rpm) for 5, 10, 20, and 40 minutes.

After shaking, samples were filtered through Whatman⁶ number 2 filter paper into 100-ml plastic Solo⁷ P35A cups. Filtrates were then transferred to glass 50-ml test tubes prior to ICAP-AES analysis. One replication was completed on each of 3 consecutive days.

The ICAP-AES instrument settings for forward R.F. power, torch argon flow, and sample flow were 1.2 kW, 16 liters min⁻¹, and 1.6 ml min⁻¹, respectively. A N₂ gas purge was used during all analyses. Wavelengths used were 308.21, 317.93, 279.08, and 257.61 nm for Al, Ca, Mg, and Mn, respectively. A standard-size Jarrell Ash type torch was used. A yttrium

⁵Eberbach Corporation, P.O. Box 1024, Ann Arbor, MI 48106.

⁶Whatman International Ltd., Maidstone, England.

⁷Solo Cup Company, Urbana, IL 61801.

nitrate solution was introduced into the plasma and the sample flow adjusted so that the characteristic red zone was set to ± 1 mm from the top of the torch. A cadmium solution was used to optimize horizontal and vertical torch positions. The horizontal position was set by profiling to maximum gain. Vertical position was set by determining cadmium solution/deionized water ratios at various positions with the highest ratio indicating the optimum vertical torch setting.

Calibration and standardization of the ICAP-AES were done by using standards prepared in each concentration of KCl. Samples were analyzed using the average of four 10-second exposures. Standards were analyzed periodically to check for drift and restandardization was performed when drift was significant ($\pm 5\%$).

All ICAP-AES analytical results were analyzed statistically as a split-split plot with the whole plot as KCl concentration, the split plot as extraction time, and the split-split plot as soil:solution ratio.

RESULTS AND DISCUSSION

The concentration of KCl had a significant effect on the concentration of all elements tested in both soils. Time was not a significant factor for extraction of Al for either soil, however, amounts of Mn extracted from both soils and amounts of Ca and Mg from Soil I were affected by extraction time. In Soil II Ca and Mg concentrations extracted

were not significantly affected by extraction time. The soil:solution ratio had a significant effect on Ca in both soils but only on Mg concentration in Soil I. No interaction was observed for extraction time vs KCl concentration on Al or Mn extracted from either soil or for Ca and Mg from Soil I. The interaction of KCl concentration vs soil:solution ratio was significant for Al extracted from both soils and Mn, Ca, and Mg from soil I. F values are reported in Table 2.

TABLE 2. Significant F-values for Effects of KCl Concentrations, Extraction Times, and Soil:solution Ratios on Al, Mn, Ca, and Mg Concentrations.

Source	df	F-values			
		Al	Mn	Ca	Mg
<u>Soil I</u>					
Reps	2				
KCl (C)	3	110.21**	326.04**	195.40**	1454.28**
Time (T)	3	0.27	49.31**	16.92**	14.19**
C x T	9	1.89	1.87	2.31*	2.69*
Ratio (R)	2	14.52**	93.21**	126.46**	9.49**
C x R	6	5.35**	6.70**	4.77**	3.22**
T x R	6	0.88	0.46	0.37	0.68
C x T x R	18	0.82	1.16	0.69	1.26
<u>Soil II</u>					
Reps	2				
KCl (C)	3	1131.83**	130.55**	89.50**	24.30**
Time (T)	3	0.20	3.05*	2.83	1.38
C x T	9	1.14	0.90	0.53	0.67
Ratio (R)	2	26.55**	12.57**	38.31**	2.52
C x R	6	8.13**	1.26	1.59	1.71
T x R	6	0.49	1.57	1.35	1.28
C x T x R	18	0.52	1.09	1.13	1.08

* ** indicates significance at $\alpha = 0.05, 0.01$, respectively.

Mean values for all treatment levels of each element are presented in Table 3. The highest amounts of Mn and Ca were extracted with 0.5M KCl from both soils. However, Al was highest in 1M KCl, and Mg was the same in both 0.5M and 1M KCl. Aluminum levels remained approximately the same regardless of the amount of extraction time. More Ca was extracted from both soils with the 40 minute extraction time. Magnesium levels increased gradually with increased extraction time, but the change was observed only in Soil I and was not statistically significant. As the amount of solution in the soil:solution ratio increased, Al, Mn and Ca also increased. Smaller increases were observed between 1:10 and 1:15 than between 1:5 and 1:10. Magnesium levels remained the same regardless of soil:solution ratio.

Further study of the treatment means shows that the highest amounts of all elements in both soils were obtained with the soil:solution ratio of 1:15. The 40-minute extraction time produced equal or higher means for all elements except Al. Aluminum was the same in Soil II regardless of extraction time, and lowest in Soil I was lowest at 40 minutes probably as a result of secondary reaction. A KCl concentration of 0.5 M was effective for extracting Mn, Ca, and Mg in both soils; however, considerably higher amounts of Al were obtained with 1.0 M KCl. Figures 1 to 3 illustrate the two-way interaction of KCl concentration x extraction time for Al concentration of all soil:solution ratios in both soils. Figure 4 illustrates

TABLE 3. Mean Al, Mn, Ca, and Mg Extracted with Different KCl Concentrations, Extraction Times, and Soil:solution Ratios.

	<u>KCl Concentration (M)</u>					<u>Extraction Time (min.)</u>					<u>Soil:solution Ratio (v/v)</u>			
	0.125	0.250	0.500	1.000	FLSD*	5	10	20	40	FLSD*	1:5	1:10	1:15	FLSD*
Soil I (mg kg ⁻¹)														
Al	3.0	4.6	8.6	13.2	1.5	7.4	7.4	7.5	7.1	1.1	6.1	7.4	8.6	0.9
Mn	83	99	112	106	2	97	99	100	108	1	96	101	103	1
Ca	1049	1275	1480	1344	45	1264	1283	1289	1313	14	1222	1301	1339	15
Mg	281	298	328	328	2	305	307	309	313	3	305	310	310	3
Soil II (mg kg ⁻¹)														
Al	0.8	0.9	3.2	6.3	0.3	2.8	2.9	2.8	2.8	0.3	2.4	2.8	3.3	0.3
Mn	23	27	32	30	1	28	27	29	29	1	27	29	29	1
Ca	640	758	889	796	38	766	764	764	790	22	727	784	803	18
Mg	214	218	244	242	11	228	228	228	234	8	227	230	233	6

*FLSD at $\alpha = 0.05$.

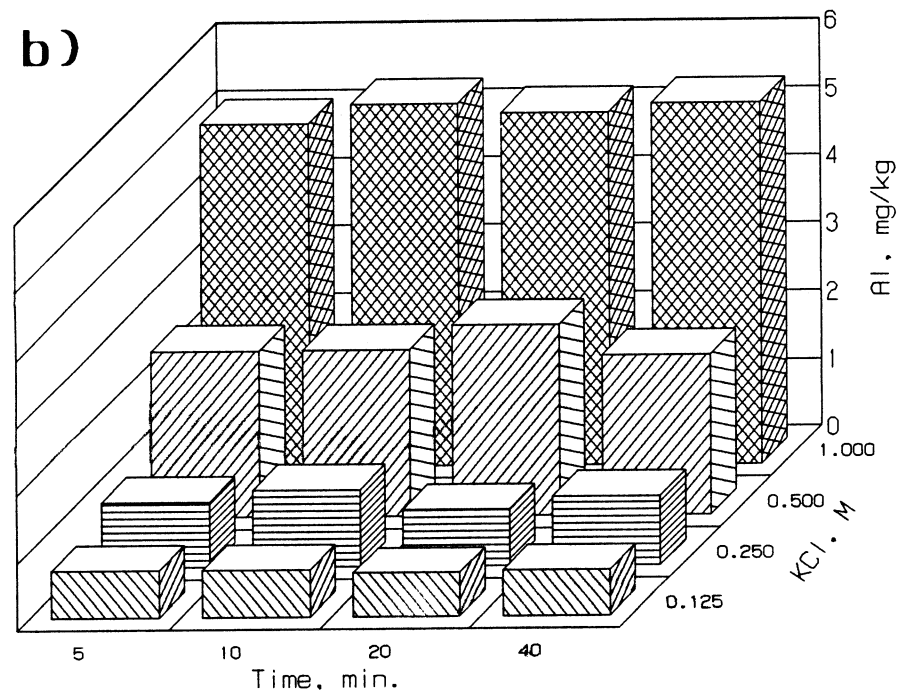
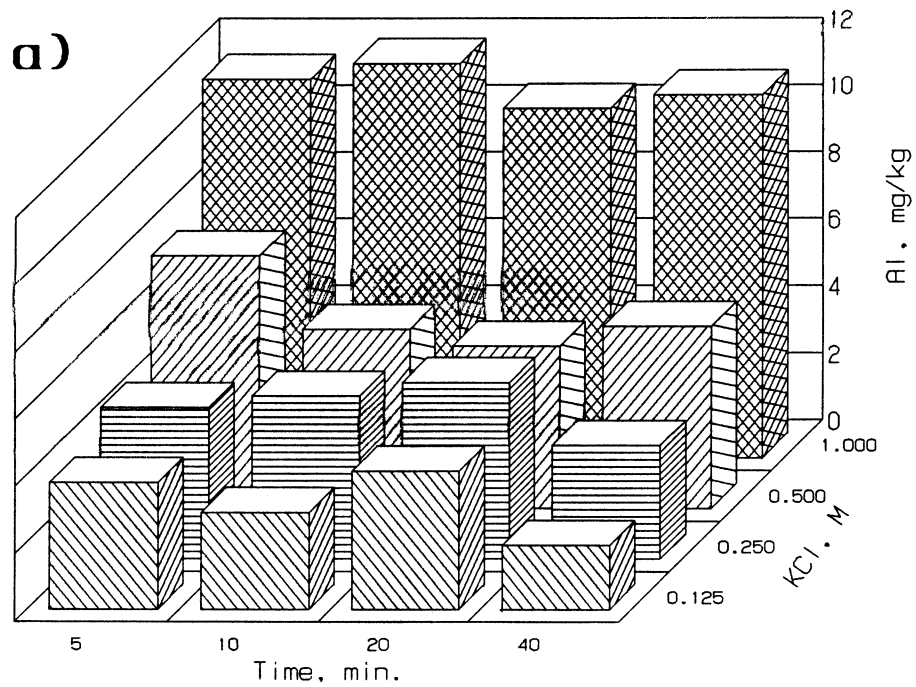


FIGURE 1. Aluminum Concentration x KCl Concentration x Extraction Time for 1:5 Soil:Solution Ratio for Soil I (a) and Soil II (b).

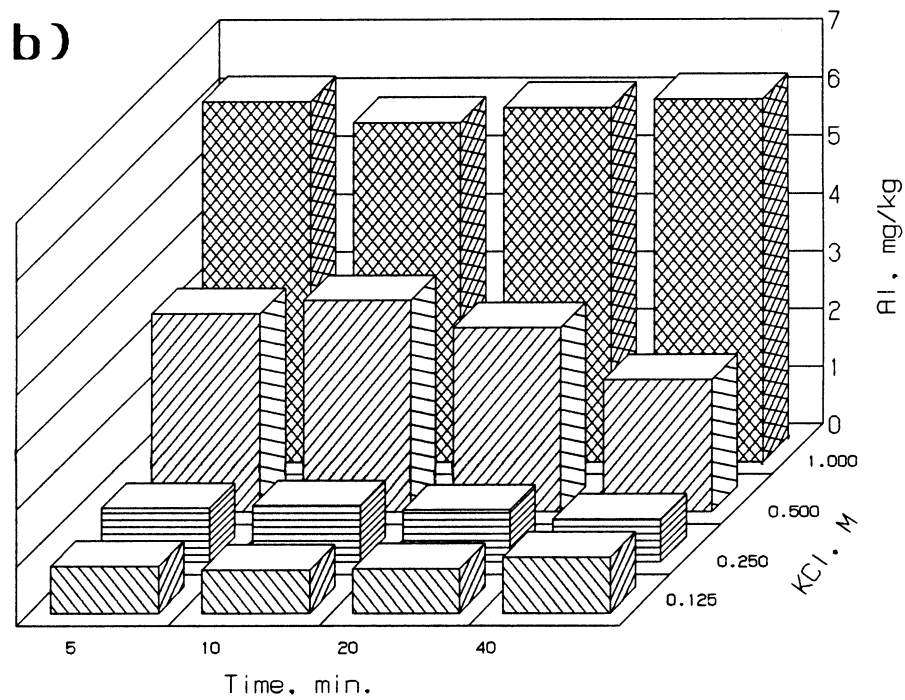
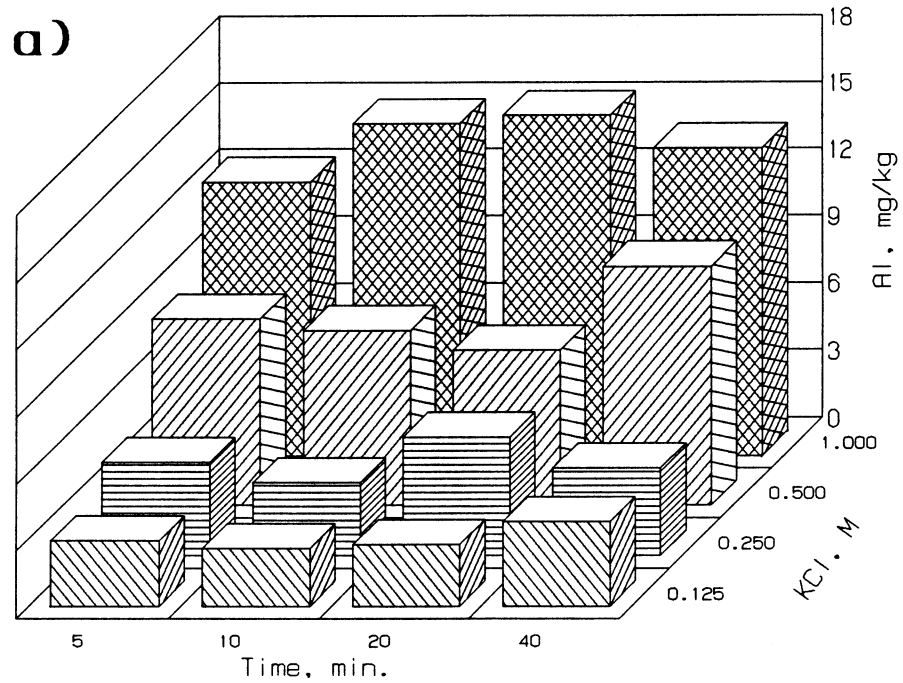


FIGURE 2. Aluminum Concentration x KCl Concentration x Extraction Time for 1:10 Soil:Solution Ratio for Soil I (a) and Soil II (b).

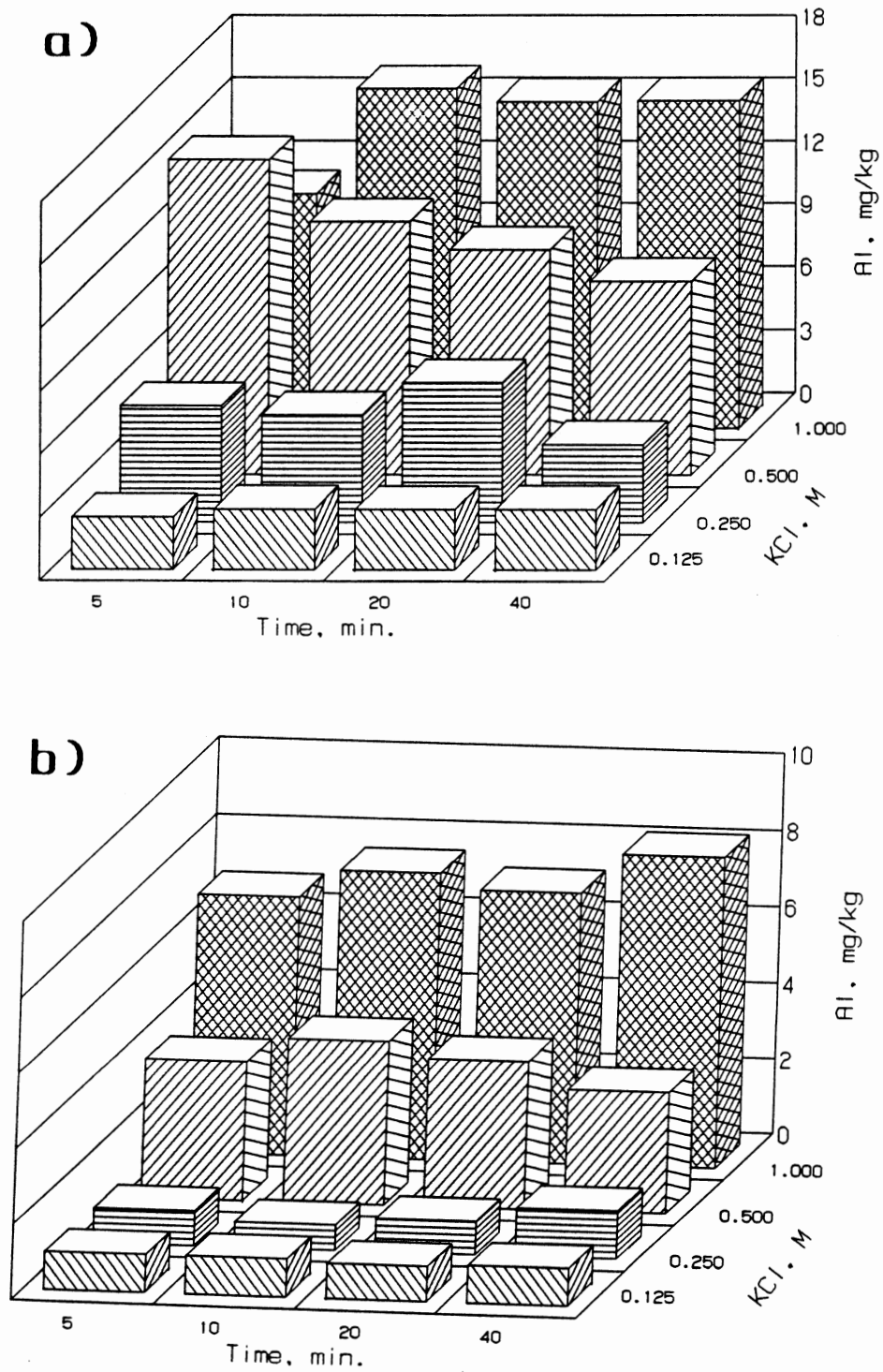


FIGURE 3. Aluminum Concentration x KCl Concentration x Extraction Time for 1:15 Soil:Solution Ratio for Soil I (a) and Soil II (b).

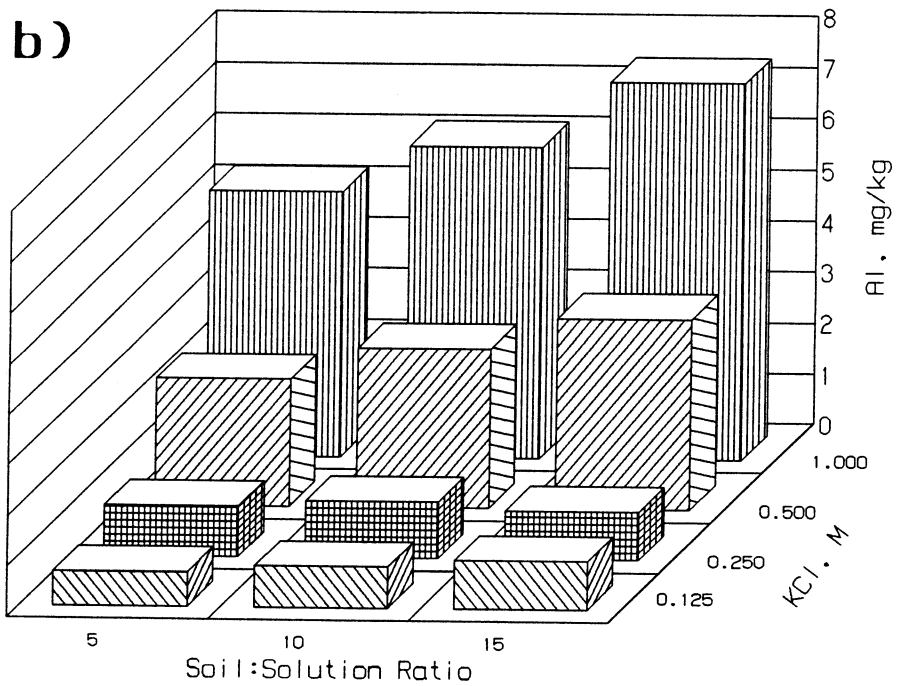
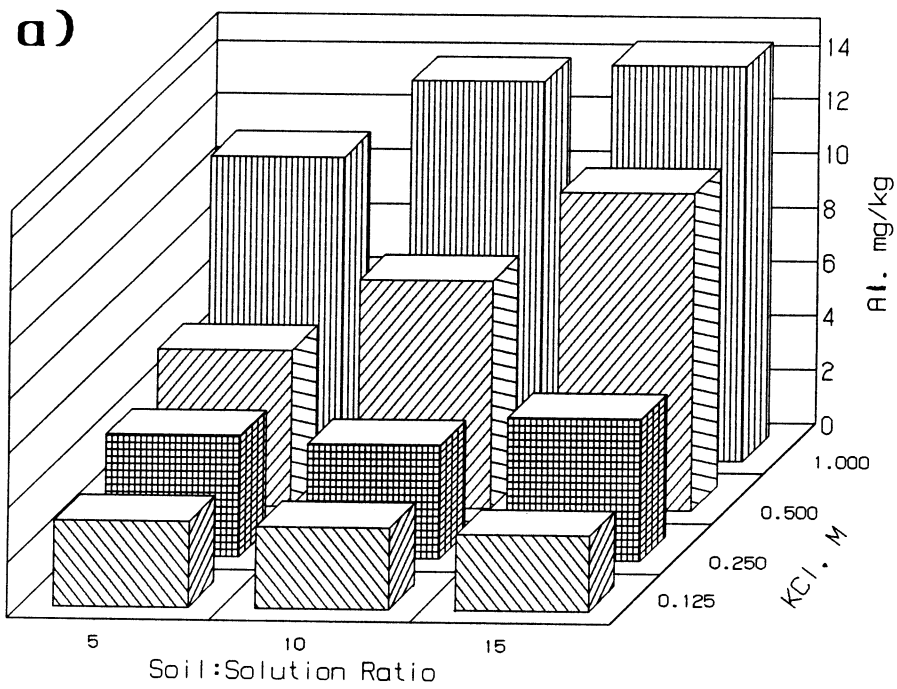


Figure 4. Aluminum Concentration x KCl Concentration x Soil:Solution Ratio Averaged Across All Extraction Times for Soil I (a) and Soil II (b).

the interaction between KCl concentration and soil:solution ratio for KCl extractable Al.

Simple linear regression analysis was performed using all KCl concentrations to see if any relationship existed. There were no correlations found that were useful in predicting 1M KCl concentration from lower KCl concentrations. The ICAP-AES readings using 0.125 M KCl were extremely low and approached the detection limitations of the instrument. Therefore, these values were more erratic than the higher concentrations of KCl used and cannot be considered reliable. Aluminum levels with 0.5 M KCl were far enough above the detection limit of the ICAP-AES that erratic readings did not occur. While all elements determined are certainly important, more emphasis was placed on determination of Al and Mn because of their toxic effect on plants grown in low pH soils. Therefore, the parameters selected for the effective extraction of Al, Mn, Ca, and Mg in these soils was a KCl concentration of 0.5 M, an extraction time of 10 minutes, and a soil:solution ration of 1:15.

CONCLUSION

Soil extraction parameters were compared using two Oklahoma acid soils for determination of Al, Mn, Ca, and Mg by ICAP-AES. This study was carried out to determine which KCl concentration, extraction time, and soil:solution ratio were most effective when using ICAP-AES for analysis of

extracts. Different KCl concentrations and extraction times significantly affected the amount of all elements tested except for extraction time with regard to Al concentration in both soils and Ca and Mg concentration in one soil. Soil:solution ratio also significantly affected extraction of all elements except Mg from one soil. An interaction was observed for concentration vs soil:solution ratio for Al extracted from both soils and for Mn, Ca, and Mg extracted from one of the soils.

Based on the analytical data and instrumentation capability of the ICAP-AES a soil:solution ratio of 1:15 and extraction time of 10 minutes were selected. Use of 1M KCl gave higher results for concentrations of Al, however due to the high salt content in 1 M KCl, use of 0.5 M KCl would be more effective concentration since instrumentation problems do not occur with this concentration.

Additional study is needed on a wider variety of soil types to determine whether or not the trends observed in this study remain the same and whether or not the selected parameters are the most effective for extracting Al, Mn, Ca, and Mg from a wide range of acid soils.

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APPENDIXES

APPENDIX A

MEHLICH III SOIL EXTRACT DATA

Rep	Soil #	AAS				ICAP-h				ICAP			
		K	P mg/kg	Ca	Mg	K	P mg/kg	Ca	Mg	K	P mg/kg	Ca	Mg
1	1	99	21	1245	237	91	42	1310	257	95	46	1370	259
2	1	96	21	1350	236	100	50	1320	260	96	46	1313	253
3	1	99	22	1328	234	95	44	1310	257	95	46	1381	258
1	2	79	110	461	109	71	181	473	104	75	193	497	103
2	2	76	97	435	110	79	191	473	104	77	189	478	101
3	2	79	116	449	109	76	190	483	105	76	197	506	102
1	3	58	47	1360	144	53	57	1259	134	57	62	1314	132
2	3	57	47	1440	141	61	62	1248	132	57	61	1250	128
3	3	58	48	1380	145	57	62	1264	134	57	64	1338	133
1	4	33	21	639	49	29	45	656	48	33	50	683	46
2	4	32	21	606	49	38	51	642	47	33	48	646	44
3	4	33	19	621	52	33	50	663	48	32	51	690	46
1	5	115	32	1185	240	106	51	1113	228	110	56	1171	230
2	5	112	32	1240	244	112	55	1104	225	111	54	1118	224
3	5	115	31	1230	240	110	54	1130	229	109	57	1183	229
1	6	168	31	1095	231	157	52	1023	219	159	56	1060	219

Rep	Soil #	AAS				ICAP-h				ICAP			
		K	P	Ca	Mg	K	P	Ca	Mg	K	P	Ca	Mg
		mg/kg				mg/kg				mg/kg			
2	6	163	30	1140	238	158	58	1006	215	161	56	1014	214
3	6	166	31	1070	234	157	54	1022	218	159	59	1077	218
1	7	142	47	921	202	132	66	915	192	135	69	937	189
2	7	137	46	858	204	136	70	900	189	136	69	902	186
3	7	142	47	870	203	136	72	933	196	135	75	963	192
1	8	72	61	530	105	66	70	554	102	69	78	571	100
2	8	69	60	515	106	74	79	556	102	70	76	548	97
3	8	71	59	531	108	71	77	567	103	69	79	582	99
1	9	58	26	807	139	53	34	846	136	56	35	858	132
2	9	56	24	764	139	62	40	857	138	57	37	831	130
3	9	58	25	772	138	58	39	864	138	56	39	892	134
1	10	43	36	229	46	38	50	230	43	42	54	233	40
2	10	41	35	211	47	48	58	230	44	42	54	225	40
3	10	43	35	215	46	44	56	233	44	42	58	241	41
1	11	49	35	137	24	44	49	143	25	47	52	147	22
2	11	48	35	128	25	53	54	144	25	48	52	141	22
3	11	49	35	128	25	51	55	148	25	47	56	156	23
1	12	43	36	93	17	40	50	99	17	43	55	105	16
2	12	43	36	84	16	48	56	102	18	43	55	101	15
3	12	44	36	78	16	47	60	106	19	43	60	112	16
1	13	57	37	334	66	53	53	340	62	56	57	360	61
2	13	56	36	324	64	61	59	341	62	56	55	343	59
3	13	58	37	320	65	60	58	358	64	56	63	388	62
1	14	50	45	188	44	46	59	207	42	49	64	218	40

Rep	Soil #	AAS				ICAP-h				ICAP			
		K	P	Ca	Mg	K	P	Ca	Mg	K	P	Ca	Mg
		mg/kg				mg/kg				mg/kg			
2	14	49	44	185	40	55	64	205	42	49	62	205	38
3	14	51	45	182	42	53	66	213	43	50	68	235	41
1	15	101	113	512	147	94	222	532	137	96	237	567	138
2	15	98	107	499	145	99	227	525	135	97	227	532	134
3	15	102	116	519	147	98	239	550	140	96	250	604	141
1	16	87	22	1450	266	80	37	1318	254	83	41	1413	258
2	16	83	21	1470	265	87	41	1301	250	84	40	1316	248
3	16	86	20	1545	268	86	45	1383	263	84	45	1520	267
1	17	71	16	1200	264	67	27	1101	256	70	34	1182	260
2	17	69	16	1215	264	74	34	1094	253	70	32	1100	250
3	17	72	16	1180	267	73	35	1153	265	70	35	1259	267
1	18	102	21	820	107	98	34	862	106	99	39	910	103
2	18	99	21	794	108	103	41	854	104	100	37	845	98
3	18	103	21	787	110	103	40	889	107	99	42	973	106
1	19	137	116	1615	149	128	435	1565	154	130	467	1653	154
2	19	133	111	1550	149	133	455	1564	154	132	446	1555	149
3	19	138	121	1585	152	130	460	1598	155	130	491	1754	158
1	20	115	114	1810	180	108	352	1802	172	111	376	1870	171
2	20	113	109	1945	180	114	364	1786	171	111	358	1769	165
3	20	115	120	2050	181	111	373	1827	174	111	397	2015	178
1	21	73	32	722	134	67	40	727	125	71	45	760	125
2	21	72	31	704	135	75	48	728	126	71	46	725	121
3	21	74	31	687	135	74	47	760	130	71	50	821	129
1	22	49	109	289	36	43	207	302	33	48	220	318	32

Rep	Soil #	AAS				ICAP-h				ICAP			
		K	P	Ca	Mg	K	P	Ca	Mg	K	P	Ca	Mg
		mg/kg				mg/kg				mg/kg			
2	22	48	106	298	35	53	217	302	34	47	214	301	31
3	22	49	115	279	35	50	226	314	35	48	233	338	33
1	23	39	7	1215	487	34	15	1237	478	37	19	1275	478
2	23	38	7	1270	489	44	22	1237	476	38	18	1222	470
3	23	39	7	1295	491	41	20	1291	495	38	21	1393	500
1	24	155	111	1440	384	144	192	1393	378	147	200	1431	377
2	24	153	106	1450	386	150	201	1405	379	151	199	1388	372
3	24	157	115	1545	386	150	208	1464	393	149	217	1585	397
1	25	157	113	1510	505	145	200	1503	470	148	212	1555	474
2	25	153	109	1645	499	151	211	1509	470	153	207	1492	465
3	25	158	111	1635	498	152	219	1581	491	150	229	1708	496

APPENDIX B

NH₄OAC SOIL EXTRACT DATA

Rep	Soil #	K	AAS		K	ICAP-h		K	ICAP	
			Ca mg/kg	Mg		Ca mg/kg	Mg		Ca mg/kg	Mg
1	1	91	1130	313	90	1236	235	84	1176	209
2	1	90	1240	310	91	1251	239	87	1196	214
3	1	91	1265	312	92	1245	237	85	1184	211
1	2	76	530	117	75	468	98	70	441	87
2	2	76	505	119	76	474	101	72	447	89
3	2	76	490	115	77	473	100	71	445	88
1	3	58	1240	141	58	1211	126	54	1161	112
2	3	58	1280	143	57	1204	126	55	1168	114
3	3	58	1245	141	59	1227	129	54	1173	114
1	4	32	520	51	32	615	42	30	574	37
2	4	32	555	48	32	613	42	31	585	38
3	4	32	545	49	33	623	43	30	591	39
1	5	105	1100	253	105	1105	217	97	1050	191
2	5	106	1230	251	105	1106	218	99	1065	196
3	5	104	1175	250	106	1121	220	98	1077	197
1	6	151	920	248	149	977	206	139	928	183
2	6	151	935	250	151	983	209	141	938	185
3	6	151	965	246	151	989	208	140	946	186
1	7	131	850	210	130	829	181	121	782	158
2	7	131	895	210	132	832	183	123	795	163
3	7	131	900	209	132	840	183	122	811	165
1	8	66	520	109	65	524	93	61	496	82
2	8	66	540	113	65	524	94	62	503	84
3	8	66	550	112	66	530	95	61	514	86
1	9	53	790	142	54	795	121	50	754	106
2	9	54	795	142	54	802	123	52	761	108
3	9	54	765	141	55	807	124	51	775	110
1	10	39	200	48	39	225	38	36	215	33
2	10	39	205	47	39	227	39	37	217	34
3	10	39	190	47	39	228	38	37	223	35
1	11	50	120	29	49	143	23	46	136	21
2	11	50	155	27	49	145	24	47	137	21

Rep	Soil #	K	AAS		ICAP-h			K	ICAP	
			Ca	Mg	K	Ca	Mg		Ca	Mg
			mg/kg		mg/kg			mg/kg		
3	11	50	150	27	49	145	23	46	141	22
1	12	41	90	18	40	97	14	37	93	14
2	12	41	110	18	41	98	15	38	93	14
3	12	41	95	17	40	97	15	38	95	14
1	13	53	330	67	52	322	57	48	310	50
2	13	53	305	68	53	329	58	49	307	50
3	13	53	295	68	52	325	57	49	319	52
1	14	43	190	43	43	195	36	40	187	32
2	14	44	210	43	44	200	38	41	187	33
3	14	44	200	43	43	198	37	41	195	35
1	15	94	510	149	93	494	128	87	468	113
2	15	95	555	147	95	501	131	88	473	115
3	15	95	540	147	94	500	130	88	486	117
1	16	80	1240	289	82	1297	243	78	1239	216
2	16	81	1260	287	83	1314	248	77	1232	217
3	16	81	1290	287	82	1298	245	77	1301	229
1	17	67	980	295	67	1067	244	63	1025	218
2	17	69	980	296	68	1084	249	64	1020	218
3	17	69	975	294	68	1073	246	65	1079	229
1	18	95	750	111	94	814	95	88	786	85
2	18	95	790	110	95	831	98	88	776	85
3	18	94	800	111	95	820	97	89	811	89
1	19	127	1260	164	125	1366	141	116	1314	124
2	19	127	1375	164	127	1402	145	118	1307	125
3	19	127	1340	163	126	1385	142	118	1355	129
1	20	108	1540	199	108	1623	162	101	1573	145
2	20	109	1495	198	110	1659	167	102	1561	146
3	20	108	1540	198	109	1644	165	102	1628	152
1	21	67	800	228	65	655	108	62	636	98
2	21	67	795	226	67	667	112	62	636	98
3	21	67	820	226	66	665	111	63	658	102
1	22	46	260	58	46	279	31	43	270	27
2	22	47	310	59	47	285	32	44	270	28
3	22	47	295	59	46	282	31	44	281	30
1	23	40	990	983	39	1074	481	37	1028	428
2	23	40	1490	959	40	1103	496	38	1045	437
3	23	41	1470	962	40	1087	485	38	1083	449
1	24	146	1210	667	143	1282	351	133	1229	314
2	24	145	1205	657	146	1322	364	135	1235	317
3	24	146	1220	659	145	1303	357	137	1309	333
1	25	140	1490	870	138	1374	430	128	1327	386
2	25	141	1505	860	140	1415	445	131	1324	388

Rep	Soil #	AAS			ICAP-h			ICAP		
		K	Ca mg/kg	Mg	K	Ca mg/kg	Mg	K	Ca mg/kg	Mg
3	25	140	1460	856	139	1397	438	132	1404	408

APPENDIX C

KCL SOIL EXTRACT DATA

Rep	Soil #	AAS		ICAP-h		ICAP	
		Al mg/kg	Mn mg/kg	Al mg/kg	Mn mg/kg	Al mg/kg	Mn mg/kg
1	1	107.5	75.3	87.2	55.5	87.0	53.8
1	2	208.5	13.4	185.3	12.2	184.0	11.8
1	3	28.5	44.3	22.8	35.5	23.1	35.4
1	4	116.0	55.3	95.7	42.5	99.0	43.1
1	5	10.0	60.9	7.6	50.9	6.5	48.9
1	6	5.5	60.1	4.0	51.8	3.3	48.5
1	7	30.5	78.8	26.3	65.6	24.1	62.1
1	8	10.5	19.1	10.5	16.1	9.1	15.1
1	9	8.5	21.1	6.2	19.0	4.8	18.1
1	10	80.5	40.0	70.1	35.5	67.5	33.5
1	11	79.5	32.3	66.5	28.6	65.5	27.4
1	12	113.0	16.6	97.7	15.2	91.2	14.7
1	13	69.5	56.3	59.5	49.0	58.6	47.4
1	14	72.0	23.1	59.5	21.1	58.4	20.3
1	15	120.5	12.8	108.8	11.7	108.7	11.3
1	16	43.0	66.8	38.6	56.9	37.6	53.5
1	17	8.5	47.0	6.3	41.9	5.5	39.4
1	18	7.5	70.8	5.8	63.0	4.9	62.3
1	19	6.5	9.1	4.9	8.6	4.3	8.0
1	20	110.0	13.7	108.8	12.1	109.9	11.8
1	21	26.5	40.1	22.2	36.9	20.4	34.4
1	22	80.0	23.1	69.9	21.9	69.7	20.4
1	23	193.5	39.0	185.3	35.6	197.1	34.5
1	24	95.0	16.6	89.8	15.3	96.3	14.5
1	25	31.5	12.4	26.7	11.8	33.1	11.7
2	1	92.0	72.7	101.0	61.9	88.7	55.9
2	2	200.0	14.9	192.4	12.3	187.7	12.4
2	3	28.5	44.8	25.8	38.7	22.6	36.4
2	4	111.0	55.0	108.5	47.9	100.3	44.5
2	5	11.0	60.8	7.9	52.0	6.3	49.6

Rep	Soil #	AAS		ICAP-h		ICAP	
		Al mg/kg	Mn mg/kg	Al mg/kg	Mn mg/kg	Al mg/kg	Mn mg/kg
2	6	7.0	61.0	4.1	52.7	3.2	50.3
2	7	31.0	77.7	26.7	67.3	23.4	63.5
2	8	11.5	20.8	10.6	16.3	8.8	15.4
2	9	9.5	22.9	5.8	18.5	4.7	18.1
2	10	79.5	42.5	70.0	35.5	67.6	34.9
2	11	77.0	34.5	66.1	28.3	64.6	28.6
2	12	111.0	18.1	93.7	14.6	91.5	15.3
2	13	70.5	57.0	58.6	47.2	57.5	49.0
2	14	71.0	25.1	57.8	19.9	58.5	21.1
2	15	121.5	14.4	106.2	10.9	107.2	11.8
2	16	44.5	66.7	36.6	52.8	38.0	55.5
2	17	11.5	48.6	5.8	38.2	5.3	40.9
2	18	11.0	71.7	5.4	57.9	4.5	64.3
2	19	9.0	10.3	4.5	7.8	3.9	8.2
2	20	110.0	15.3	105.3	11.3	112.5	12.1
2	21	27.5	41.4	20.0	32.3	20.7	36.0
2	22	80.0	24.9	64.1	19.3	72.7	21.7
2	23	189.0	39.8	170.5	31.6	202.0	36.4
2	24	94.5	18.1	84.7	13.8	98.7	15.1
2	25	32.5	13.8	25.0	10.6	33.5	12.1
3	1	77.5	71.7	101.5	62.7	88.0	56.0
3	2	173.5	14.3	188.4	12.1	187.7	12.6
3	3	28.0	44.2	24.4	36.4	22.8	36.6
3	4	102.0	53.9	101.3	44.5	97.0	44.0
3	5	7.5	58.8	7.4	47.5	5.8	49.9
3	6	5.5	59.4	3.8	48.5	3.0	50.9
3	7	26.5	76.8	24.9	61.1	23.0	64.1
3	8	11.0	20.3	10.1	15.1	8.9	15.8
3	9	8.0	22.1	5.7	16.9	4.5	18.7
3	10	71.0	41.0	73.1	36.6	66.3	34.8
3	11	71.5	33.3	65.5	26.2	68.0	30.1
3	12	102.0	17.6	89.8	11.2	98.5	16.3
3	13	62.0	56.1	54.4	29.9	64.3	52.9
3	14	64.5	24.6	67.0	22.9	64.9	22.8
3	15	108.0	13.9	122.7	12.8	122.9	12.7
3	16	42.5	64.9	42.9	60.8	46.5	60.9
3	17	9.5	46.9	7.0	43.7	6.8	45.0
3	18	8.5	71.4	6.2	67.2	5.8	71.4
3	19	7.5	9.9	5.5	9.2	5.2	9.7

Rep	Soil #	AAS		ICAP-h		ICAP	
		Al	Mn	Al	Mn	Al	Mn
		mg/kg		mg/kg		mg/kg	
3	20	96.0	14.8	124.7	13.4	135.4	13.8
3	21	24.5	40.9	23.3	38.1	25.3	39.6
3	22	76.5	24.1	73.0	22.4	84.0	23.1
3	23	191.0	39.1	192.8	36.2	225.9	36.9
3	24	90.5	17.5	95.1	15.8	111.7	15.6
3	25	29.0	13.2	27.8	12.0	37.9	12.3

APPENDIX D

SOIL I DATA--TELLER SOIL

KCl (M)	Time (min.)	Rep	Soil:Solution Ratio	Al	Mn	Ca	Mg
				mg kg ⁻¹			
0.125	5	1	5	3.8225	74.400	938.00	260.35
0.125	5	2	5	3.6005	76.300	952.50	265.50
0.125	5	3	5	3.7900	76.100	944.00	271.50
0.125	5	1	10	2.3110	80.890	1035.00	277.80
0.125	5	2	10	3.2180	83.020	1054.00	281.50
0.125	5	3	10	3.1440	84.740	1067.00	282.60
0.125	5	1	15	1.8600	81.990	1063.35	280.80
0.125	5	2	15	2.1135	87.525	1132.05	293.85
0.125	5	3	15	3.4410	88.845	1115.55	290.25
0.125	10	1	5	2.2845	80.100	1014.00	277.65
0.125	10	2	5	4.2785	80.650	997.50	278.25
0.125	10	3	5	2.0270	78.650	977.00	277.15
0.125	10	1	10	2.3110	80.040	1021.00	271.10
0.125	10	2	10	2.2290	86.220	1076.00	285.70
0.125	10	3	10	3.1440	83.540	1065.00	280.80
0.125	10	1	15	1.8600	85.545	1108.35	288.60
0.125	10	2	15	3.4695	88.095	1121.55	289.65
0.125	10	3	15	3.2295	86.490	1113.45	287.10
0.125	20	1	5	3.9495	79.200	1036.00	281.50
0.125	20	2	5	4.5115	81.000	983.50	274.75
0.125	20	3	5	3.7755	79.850	969.00	274.20
0.125	20	1	10	1.8880	79.450	1009.00	268.60
0.125	20	2	10	3.2180	84.620	1062.00	281.70
0.125	20	3	10	3.0590	87.760	1122.00	294.80
0.125	20	1	15	3.0465	84.915	1069.95	277.95
0.125	20	2	15	2.1135	89.805	1142.40	295.95
0.125	20	3	15	3.3570	90.060	1144.05	297.45
0.125	40	1	5	1.4235	77.600	955.00	263.00
0.125	40	2	5	2.5410	79.400	965.00	269.45
0.125	40	3	5	1.7365	81.600	979.00	276.05
0.125	40	1	10	2.5080	85.170	1059.00	278.00
0.125	40	2	10	4.6300	88.040	1078.00	288.40

KCl (M)	Time (min.)	Rep	Soil:Solution Ratio	Al	Mn	Ca	Mg
				mg kg ⁻¹			
0.125	40	3	10	4.1350	88.340	1082.00	287.40
0.125	40	1	15	2.4105	84.735	1063.35	276.15
0.125	40	2	15	2.1135	88.980	1114.80	288.15
0.125	40	3	15	4.0365	91.500	1145.55	295.65
0.250	5	1	5	5.6850	88.850	1156.00	285.05
0.250	5	2	5	5.9850	89.300	1168.00	291.65
0.250	5	3	5	1.7255	86.850	1134.50	281.35
0.250	5	1	10	6.2970	93.860	1247.00	287.60
0.250	5	2	10	1.7980	101.500	1309.00	304.00
0.250	5	3	10	4.4510	97.910	1270.00	304.70
0.250	5	1	15	3.0885	99.210	1311.75	289.80
0.250	5	2	15	6.9675	105.030	1369.95	304.80
0.250	5	3	15	6.4380	101.220	1344.90	310.05
0.250	10	1	5	3.4490	89.850	1213.50	286.80
0.250	10	2	5	5.7100	91.800	1153.50	290.35
0.250	10	3	5	5.2650	92.850	1191.00	297.15
0.250	10	1	10	2.6030	93.550	1226.00	282.90
0.250	10	2	10	2.1880	101.300	1293.00	298.40
0.250	10	3	10	5.0460	95.330	1251.00	300.50
0.250	10	1	15	8.1135	100.440	1320.30	288.60
0.250	10	2	15	2.5170	106.950	1359.45	300.75
0.250	10	3	15	4.6110	100.335	1317.00	301.80
0.250	20	1	5	5.4550	94.800	1204.00	294.75
0.250	20	2	5	6.2900	92.200	1167.50	291.95
0.250	20	3	5	3.9000	92.350	1181.50	298.00
0.250	20	1	10	4.9510	95.910	1251.00	283.80
0.250	20	2	10	6.8630	103.300	1321.00	303.00
0.250	20	3	10	4.1180	98.190	1280.00	307.60
0.250	20	1	15	4.4625	100.905	1310.25	284.85
0.250	20	2	15	8.2710	109.080	1419.45	309.45
0.250	20	3	15	7.0905	105.015	1331.85	305.85
0.250	40	1	5	4.2365	93.100	1202.50	294.35
0.250	40	2	5	2.5625	98.500	1238.00	299.40
0.250	40	3	5	3.2330	98.600	1244.50	308.80
0.250	40	1	10	1.6290	102.900	1298.00	298.30
0.250	40	2	10	4.3750	107.100	1355.00	309.80
0.250	40	3	10	5.7700	103.200	1310.00	311.90
0.250	40	1	15	3.0450	106.830	1359.30	296.70
0.250	40	2	15	1.8435	108.165	1465.05	309.15

KCl (M)	Time (min.)	Rep	Soil:Solution Ratio	Al	Mn	Ca	Mg
				mg kg ⁻¹			
0.250	40	3	15	6.1770	101.985	1328.10	303.60
0.500	5	1	5	8.9400	100.400	1311.50	311.05
0.500	5	2	5	9.9500	105.300	1389.00	321.45
0.500	5	3	5	3.6070	106.350	1414.50	322.50
0.500	5	1	10	5.9060	109.300	1453.00	325.50
0.500	5	2	10	11.8400	111.200	1484.00	331.40
0.500	5	3	10	7.2480	112.400	1519.00	330.30
0.500	5	1	15	12.1800	106.965	1417.50	316.50
0.500	5	2	15	13.8915	109.740	1512.00	318.45
0.500	5	3	15	18.9300	105.120	1436.10	293.85
0.500	10	1	5	3.5065	98.800	1343.00	313.20
0.500	10	2	5	10.2850	109.400	1419.00	330.60
0.500	10	3	5	2.1225	106.300	1411.50	320.85
0.500	10	1	10	12.4200	108.900	1394.00	315.80
0.500	10	2	10	3.7920	115.600	1524.00	335.10
0.500	10	3	10	7.2140	117.100	1574.00	336.60
0.500	10	1	15	12.5835	109.020	1473.45	323.55
0.500	10	2	15	13.5900	114.150	1524.00	331.65
0.500	10	3	15	9.8655	119.475	1614.00	339.45
0.500	20	1	5	4.2445	112.900	1436.50	332.35
0.500	20	2	5	5.6550	110.100	1411.50	322.15
0.500	20	3	5	4.5300	110.700	1459.00	332.30
0.500	20	1	10	5.2680	107.000	1387.00	316.40
0.500	20	2	10	7.6840	114.500	1537.00	334.20
0.500	20	3	10	7.7850	119.600	1588.00	344.30
0.500	20	1	15	12.9360	105.195	1425.60	312.45
0.500	20	2	15	10.3185	116.685	1551.00	335.40
0.500	20	3	15	8.7585	117.600	1581.00	333.75
0.500	40	1	5	8.8600	111.650	1435.00	329.10
0.500	40	2	5	2.5670	114.550	1468.50	336.80
0.500	40	3	5	4.7650	113.100	1486.50	337.75
0.500	40	1	10	10.7700	112.800	1427.00	322.30
0.500	40	2	10	11.8100	118.100	1553.00	337.80
0.500	40	3	10	9.3950	119.900	1576.00	340.90
0.500	40	1	15	12.2310	115.965	1527.00	331.20
0.500	40	2	15	8.4555	120.705	1590.00	340.50
0.500	40	3	15	6.8700	122.790	1626.00	341.70
1.000	5	1	5	13.2000	96.750	1231.00	316.50
1.000	5	2	5	13.1150	106.800	1373.50	334.50

KCl (M)	Time (min.)	Rep	Soil:Solution Ratio	Al	Mn	Ca	Mg
				mg kg ⁻¹			
1.000	5	3	5	7.5700	96.800	1250.00	310.20
1.000	5	1	10	12.3200	99.560	1269.00	317.70
1.000	5	2	10	13.0500	106.200	1374.00	330.20
1.000	5	3	10	11.1600	103.800	1338.00	324.90
1.000	5	1	15	10.6590	98.640	1298.10	314.85
1.000	5	2	15	16.8300	107.775	1401.45	330.60
1.000	5	3	15	5.8770	106.560	1421.55	350.55
1.000	10	1	5	13.2650	102.000	1307.50	321.70
1.000	10	2	5	14.1950	104.300	1312.00	328.00
1.000	10	3	5	7.8550	103.800	1330.00	330.05
1.000	10	1	10	14.8400	104.400	1346.00	329.70
1.000	10	2	10	18.1600	110.300	1417.00	340.10
1.000	10	3	10	11.3600	104.000	1320.00	321.60
1.000	10	1	15	15.4950	102.630	1330.50	322.20
1.000	10	2	15	16.3350	100.275	1312.20	303.60
1.000	10	3	15	16.5300	109.500	1412.70	332.25
1.000	20	1	5	11.3400	104.050	1290.50	324.45
1.000	20	2	5	11.1400	104.450	1321.00	329.85
1.000	20	3	5	8.7650	101.700	1272.00	318.85
1.000	20	1	10	12.7500	100.100	1267.00	309.70
1.000	20	2	10	18.5100	111.700	1390.00	337.50
1.000	20	3	10	14.2100	113.500	1431.00	343.60
1.000	20	1	15	13.7220	101.775	1301.40	313.95
1.000	20	2	15	16.0950	107.595	1339.20	324.00
1.000	20	3	15	16.7400	107.625	1421.25	331.65
1.000	40	1	5	13.7300	106.050	1314.50	333.80
1.000	40	2	5	8.6350	106.800	1340.50	337.15
1.000	40	3	5	10.0800	110.400	1356.00	339.50
1.000	40	1	10	9.7960	109.000	1336.00	327.00
1.000	40	2	10	13.5100	111.700	1413.00	336.30
1.000	40	3	10	17.7300	112.200	1399.00	339.90
1.000	40	1	15	14.6445	107.625	1358.25	326.25
1.000	40	2	15	15.1350	108.840	1350.45	328.65
1.000	40	3	15	16.8900	115.965	1452.90	339.30

APPENDIX E

SOIL II DATA--POND CREEK SOIL

KCl (M)	Time (min.)	Rep	Soil:Solution Ratio	Al	Mn	Ca	Mg
				mg kg ⁻¹			
0.125	5	1	5	0.7180	17.055	584.50	200.15
0.125	5	2	5	0.7045	20.085	586.00	199.25
0.125	5	3	5	0.6745	22.550	562.00	200.75
0.125	5	1	10	0.5610	21.010	625.10	210.40
0.125	5	2	10	0.5900	23.090	641.50	214.90
0.125	5	3	10	1.2460	24.340	641.00	212.60
0.125	5	1	15	0.7575	21.675	664.20	213.45
0.125	5	2	15	0.7575	23.835	668.85	218.70
0.125	5	3	15	1.4025	22.440	666.30	215.40
0.125	10	1	5	0.7040	20.260	580.00	200.55
0.125	10	2	5	0.7045	21.675	581.50	202.10
0.125	10	3	5	0.6885	22.480	574.50	203.20
0.125	10	1	10	0.5610	21.240	619.20	206.20
0.125	10	2	10	0.5050	23.580	635.00	211.30
0.125	10	3	10	1.1330	22.950	641.40	211.80
0.125	10	1	15	0.8430	21.495	645.00	208.65
0.125	10	2	15	0.7575	24.195	672.90	217.65
0.125	10	3	15	1.3170	23.910	682.50	220.50
0.125	20	1	5	0.5770	18.525	586.00	198.50
0.125	20	2	5	0.7045	22.700	595.50	205.05
0.125	20	3	5	0.6675	22.270	586.50	206.80
0.125	20	1	10	0.5610	21.460	601.20	199.90
0.125	20	2	10	0.7170	23.920	639.80	210.90
0.125	20	3	10	0.9910	23.750	643.00	212.80
0.125	20	1	15	0.6315	22.890	653.70	209.55
0.125	20	2	15	0.7575	25.410	676.50	217.95
0.125	20	3	15	1.3170	26.295	658.95	215.25
0.125	40	1	5	0.7040	19.860	589.00	199.25
0.125	40	2	5	0.7045	20.540	579.50	197.50
0.125	40	3	5	0.6390	23.030	582.00	205.20
0.125	40	1	10	1.2100	39.050	1131.00	386.50
0.125	40	2	10	0.7170	24.140	635.30	210.20

KCl (M)	Time (min.)	Rep	Soil:Solution Ratio	Al	Mn	Ca	Mg
				mg kg ⁻¹			
0.125	40	3	10	0.9350	24.100	644.70	214.50
0.125	40	1	15	0.7575	23.250	643.05	205.65
0.125	40	2	15	0.8010	24.390	657.75	210.75
0.125	40	3	15	1.3170	25.185	680.10	218.25
0.250	5	1	5	1.3015	23.885	709.00	214.35
0.250	5	2	5	0.8485	24.540	699.00	216.10
0.250	5	3	5	0.7540	24.575	702.50	217.30
0.250	5	1	10	1.2570	25.850	738.60	210.80
0.250	5	2	10	0.7190	27.000	771.20	218.80
0.250	5	3	10	0.8420	27.180	760.00	223.70
0.250	5	1	15	1.0260	25.380	781.05	206.70
0.250	5	2	15	0.5400	28.980	797.70	218.10
0.250	5	3	15	1.2630	28.305	785.25	223.35
0.250	10	1	5	1.7310	23.450	701.50	210.45
0.250	10	2	5	1.0005	25.645	708.00	218.20
0.250	10	3	5	0.6815	25.520	714.50	222.95
0.250	10	1	10	1.1420	24.380	754.90	211.30
0.250	10	2	10	0.7190	28.790	782.30	222.90
0.250	10	3	10	1.1030	26.410	761.80	225.00
0.250	10	1	15	0.9825	25.935	768.00	203.70
0.250	10	2	15	0.4050	28.005	813.60	220.05
0.250	10	3	15	0.6540	27.750	803.10	224.85
0.250	20	1	5	0.5855	23.930	690.50	206.60
0.250	20	2	5	0.8990	26.840	720.50	221.35
0.250	20	3	5	1.0295	27.755	714.50	223.45
0.250	20	1	10	0.6980	27.030	736.10	206.00
0.250	20	2	10	0.7190	29.550	793.80	226.10
0.250	20	3	10	1.3060	30.250	754.00	223.70
0.250	20	1	15	0.9825	26.835	794.55	211.35
0.250	20	2	15	0.5400	29.640	811.50	219.15
0.250	20	3	15	1.1325	29.565	797.40	225.45
0.250	40	1	5	0.8000	23.975	719.50	213.45
0.250	40	2	5	0.8390	26.750	727.00	218.55
0.250	40	3	5	1.4355	27.640	733.50	230.40
0.250	40	1	10	0.6840	26.620	757.00	211.80
0.250	40	2	10	0.7190	29.860	815.90	229.20
0.250	40	3	10	0.8420	28.510	766.80	226.60
0.250	40	1	15	1.0695	24.435	804.30	210.45
0.250	40	2	15	0.7635	27.945	791.40	210.30
0.250	40	3	15	2.0010	29.055	815.55	229.65

KCl (M)	Time (min.)	Rep	Soil:Solution Ratio	Al	Mn	Ca	Mg
				mg kg ⁻¹			
0.500	5	1	5	1.7115	28.435	804.00	237.80
0.500	5	2	5	2.9530	28.980	836.00	238.65
0.500	5	3	5	2.7265	29.510	859.00	243.15
0.500	5	1	10	3.7920	29.590	862.00	240.50
0.500	5	2	10	2.6840	30.790	880.70	245.50
0.500	5	3	10	3.8920	33.300	905.70	243.70
0.500	5	1	15	3.0960	50.085	950.85	251.70
0.500	5	2	15	3.2220	31.935	961.20	250.65
0.500	5	3	15	4.7310	32.580	959.40	249.75
0.500	10	1	5	3.4895	27.965	796.50	235.40
0.500	10	2	5	1.6945	30.640	833.00	240.90
0.500	10	3	5	2.2230	30.525	844.00	241.40
0.500	10	1	10	4.4290	30.100	872.90	241.60
0.500	10	2	10	3.4560	32.440	894.40	247.10
0.500	10	3	10	3.1710	31.940	914.20	245.40
0.500	10	1	15	3.4725	30.510	890.10	242.40
0.500	10	2	15	4.6305	32.685	934.05	243.60
0.500	10	3	15	4.8825	32.565	954.90	251.25
0.500	20	1	5	3.8085	38.945	813.50	233.20
0.500	20	2	5	2.1645	31.980	860.50	245.45
0.500	20	3	5	2.4830	33.575	874.00	246.95
0.500	20	1	10	3.6240	31.550	819.60	232.10
0.500	20	2	10	2.5170	33.510	907.90	247.00
0.500	20	3	10	3.4900	34.940	901.50	242.70
0.500	20	1	15	4.0260	31.125	896.25	244.95
0.500	20	2	15	3.3225	33.690	939.75	247.80
0.500	20	3	15	4.3785	34.845	950.55	251.25
0.500	40	1	5	1.8790	26.130	848.00	240.00
0.500	40	2	5	2.4830	29.665	884.50	243.90
0.500	40	3	5	2.7600	32.965	891.00	248.00
0.500	40	1	10	1.3090	28.530	873.50	243.70
0.500	40	2	10	1.6110	33.500	914.30	248.30
0.500	40	3	10	3.9930	33.830	915.20	246.10
0.500	40	1	15	1.7115	31.380	891.60	237.90
0.500	40	2	15	3.4725	32.745	958.35	250.50
0.500	40	3	15	4.3785	35.340	917.10	239.10
1.000	5	1	5	5.6800	26.850	739.50	235.85
1.000	5	2	5	5.4150	27.730	774.50	240.35
1.000	5	3	5	4.0845	30.785	749.00	238.60
1.000	5	1	10	6.2430	28.860	801.10	243.90

KCl (M)	Time (min.)	Rep	Soil:Solution Ratio	Al	Mn	Ca	Mg
				mg kg ⁻¹			
1.000	5	2	10	5.5120	29.130	810.60	242.10
1.000	5	3	10	6.9730	30.110	806.60	244.70
1.000	5	1	15	7.0230	32.355	787.65	234.45
1.000	5	2	15	7.4715	30.180	849.90	245.55
1.000	5	3	15	5.9265	30.015	838.35	243.30
1.000	10	1	5	6.2250	26.910	724.50	231.80
1.000	10	2	5	4.9145	29.445	773.50	241.85
1.000	10	3	5	4.8815	28.675	759.50	238.95
1.000	10	1	10	4.4500	29.000	792.90	243.40
1.000	10	2	10	6.6080	27.950	804.10	241.10
1.000	10	3	10	6.5750	30.700	816.10	243.80
1.000	10	1	15	8.9160	28.695	795.60	238.95
1.000	10	2	15	6.7245	31.155	854.25	246.75
1.000	10	3	15	6.9735	29.985	822.90	237.15
1.000	20	1	5	4.6155	28.195	752.00	240.35
1.000	20	2	5	5.5850	30.130	779.50	245.40
1.000	20	3	5	5.4150	30.400	776.50	244.90
1.000	20	1	10	5.6780	29.130	780.80	237.50
1.000	20	2	10	6.2430	31.850	827.70	247.10
1.000	20	3	10	6.4750	32.480	796.80	237.60
1.000	20	1	15	7.9695	30.120	800.40	241.95
1.000	20	2	15	5.8770	29.685	776.40	237.00
1.000	20	3	15	7.5210	32.775	812.85	238.20
1.000	40	1	5	4.7815	29.280	764.50	243.55
1.000	40	2	5	5.2650	32.460	832.50	257.10
1.000	40	3	5	5.9450	32.445	789.50	245.70
1.000	40	1	10	5.3130	29.210	774.40	234.10
1.000	40	2	10	5.5790	31.900	832.90	245.70
1.000	40	3	10	7.9360	31.580	817.60	245.40
1.000	40	1	15	8.1195	30.135	797.85	237.30
1.000	40	2	15	8.1195	27.630	797.85	237.75
1.000	40	3	15	8.2680	33.255	860.55	245.85

VITA

Debra L. Minter

Candidate for the Degree of

Master of Science

Thesis: HUMIDIFICATION FOR ICAP-AES ANALYSIS OF SALT
EXTRACTS OF SOILS AND COMPARISON OF SOIL EXTRACTION
PARAMETERS FOR AL, MN, CA, AND MG DETERMINATIONS

Major Field: Agronomy

Biographical:

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