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Ionic Ratios and Crop Performance. I. Vanadate and Phosphate on Soybean*

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With 3 figures and 4 tables

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Abstract

Resin-extraction techniques obtain a suite of extractable elements/ions from a single sample. They are, therefore, capable of characterizing interactions between nutrients and toxic ions on plant growth and of aiding in identification of genetic characteristics of plants required for optimal resource use efficiency. To demonstrate the usefulness of this technique, a field experiment was established on a 3.24-ha site that included five soil mapping units. The site was divided into three equal portions and planted with corn (*Zea mays* L.), soybean (*Glycine max* L. Merr.) and wheat (*Triticum aestivum* L.). Crops were rotated annually and each rotation was divided into 360 plots measuring about 3 m by 30 m. Soybean varieties designated 9091 and 9061 were planted in consecutive strips of 4 rows at 0.75 m row spacing. Soil samples were extracted with anion and cation resin exchangers. Resin exchangers were eluted with acid and the eluates examined by inductively coupled plasma atomic emission spectroscopy. Soil samples were also examined for bicarbonate extractable P, organic C, total N and pH. Seed yields were different between varieties and soils ($P < 0.05$). Seed yields ranged from about 2,390–3470 kg ha⁻¹. Seed yields of variety 9061 declined by as much as 20% (about 540 kg ha⁻¹) as the resin extractable V:(V + P) ratio increased to 0.15 mol mol⁻¹. Resin extractable V differed between soil mapping units. Soil bicarbonate extractable P, organic C, total N and pH had minimal effects on seed yield.

Key words: vanadium — phosphorus — soybean — *Glycine max* — resin-extraction

Introduction

Within a climatic area, much of the variability in crop yield can be attributed to soil water relationships, plant parasites, variation in mineral nutrition, and interplant competition for light, water and nutrients. As the area of interest becomes smaller,

the variability of many factors in crop yield diminishes. Most plant nutritional work has focused on factors present in inadequate amounts to achieve 'maximal' or 'optimal' yield. To this end a number of analytical procedures have been developed to determine adequacy in the production environment. Most of these methods rely on extraction of rather easily exchanged ions by using a salt solution in which the element of interest is absent.

Little attention is given to concentrations of extractable ions in soil which inhibit crop production because the cost of mitigating the offending specie usually seems prohibitive. Passive exchange of extractable ions from soil through use of ion exchange resins provides a suite of elements based on their ease of displacement and relative concentrations (Olness et al. 1990, Qian et al. 1992; Yang et al. 1991). This type of extraction tends to mimic plant extraction of nutrients from soils. In many cases, resin extraction provided correlations of extracted amounts (concentrations) with plant response that were superior to conventional methods (for example, Møller and Morgensen 1953, Amer et al. 1955, Salomon and Smith 1957, Arnold 1958, Moser et al. 1959, Semb et al. 1959, Walmsley and Cornforth 1973, Sibbesen 1977, Van Raij et al. 1986; Skogley et al. 1990).

Use of resins has been inhibited because of technical difficulties in separating the resin from the soil. Advances have been made in separating resins from soil during the extraction procedure by encasing the resins in fine mesh synthetic plastic bags (Sibbesen 1977). More recently we suggested use of dialysis tubing made from re-constituted cellulose; this permits evaluation of the extracted chemistries directly after elution with inductively coupled plasma emission spectroscopy ICP-AES without further separation (Olness et al. 1989). By using this technique,

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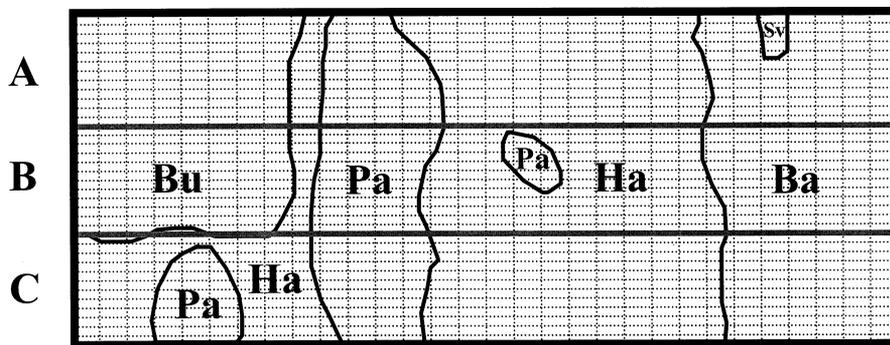


Fig. 1: Soil mapping units and cropping sequence within the experimental site. The soil abbreviations are: Ba, Barnes; Ha, Hamerly; Pa Parnell; Bu, Buse; and Sv, Svea. Areas A, B, and C were planted to maize, wheat, and soybean in annual rotation

we were able to distinguish soils based on the suite of ions extracted with exchange resins (Olness and Rinke 1994). The soils used in our initial studies were collected from widely separated regions. Obtaining a suite of ions permits evaluation of effects of several chemical species with a single extract and it also permits the evaluation of interactions between a broader range of elements. In an application of resin extraction to a 3-ha field, we were able to distinguish differences between adjacent soil mapping units (Rinke and Olness 1996). We chose to examine the analytical data to determine if (1) adjacent soil mapping units differed in extractable ions (2) seed yield differed between soil mapping units (3) seed yields were correlated with extractable ions, and (4) seed yield data contained evidence of interactions between resin extractable ions.

Materials and Methods

The field site consisted of 3.24 ha measuring 108 m by 300 m on the Barnes–Aastad Association’s Swan Lake Research Farm located 24 km NNE of Morris, MN. Soil taxonomic units were delineated by a detailed soil survey conducted by the local NRCS Soil Survey (Fig. 1). The four major soils contained in the site were Barnes loam (fine-loamy, mixed Udic Haploboroll), Buse loam (fine-loamy, mixed, Udorthentic Haploboroll), Hamerly clay loam (fine-loamy, frigid, Aeric Calciaquoll) and Parnell clay loam (fine, montmorillonitic, frigid typic Argiaquoll; Fig. 1). Less than 0.1 % of the area was Svea sandy loam (fine-loamy, mixed, Pachic Udic Haploboroll; USDA 1972) and these plots were omitted from the study. The site was divided into 3 equal portions and planted with soybean, maize, and wheat and crops were rotated annually. Each crop area was further subdivided into 360 plots measuring 3 m by 10 m and planted with soybean or maize in 1995 and soybean, wheat, and maize in 1996. Soybean varieties are designated 9091 and 9061. Each plot consisted of 4 rows of soybean planted at a rate of 65 seeds m^{-2} in 0.75-m row spacing. Each variety was planted continuously (that is, without subplot borders)

over the 300-m length of the field. Soybean seed yields were taken from the central 8 m of the two central rows of each plot with a plot combine and samples were withdrawn for further characterization. Soil samples were taken from alternate plots by compositing 5 core samples (0–60-cm) taken from the centre two rows. The cores were segmented into 15-cm increments. The soils were dried at about 60 C and ground for chemical analyses. Resin extractions were conducted by using the procedure described by Olness and Rinke (1994) with the exception that the extracting solution contained 20 % (v/v) methanol to inhibit microbial activity. In 1995, extractions were carried out over a 5-day period with a brief manual shaking twice daily. The procedure was changed to include slow continuous shaking in 1996 (rapid mechanical shaking is avoided to preclude abrasion of the membrane). After elution, extracts were submitted to the Analytical Research laboratory of the University of Minnesota, St. Paul for ICP-AES analysis. Soil pH was determined on 1:2 soil:water pastes with a radiometer autotitrator. Total N and total C were determined with a LECO Model CN2000 combustion furnace operating at 1350 °C with an IR detector for CO_2 and a thermal conductivity detector for N_2 (LECO 1994a and b). Organic C was determined by the difference between total C and inorganic C. Soil ammonium (NH_4^+ -N) and nitrate-N (NO_3^- -N) concentrations were determined with an AlpKem autoanalyzer model RFA-300 (OI Analytical, College Station, TX) and phenol (Patton and Crouch 1977) and after Cd reduction to nitrite with N-1-naphthylethylenediamine (American Public Health Association 1976), respectively. Inorganic C was determined by using the automated volumetric technique of Wagner et al. (1998). Available P was determined by the $NaHCO_3$ extraction method (Kuo 1996).

Because numbers of plots within a soil mapping unit varied, SAS PROC GLM was used to analyse yield and soil chemical data (Ray 1982). Field plots within which the mapping changed were excluded from statistical analyses; yields could not be reliably assigned to mapping units in these plots. Also, yields from a few plots were noticeably affected by excess water which reduced plant population densities; therefore, seed yields from plots with $<2000 \text{ kg ha}^{-1}$ were also excluded. This decision removed about 5–8 yield values ($<2.5\%$) each year from

Table 1: Selected soil characteristics¹

Name	Depth cm	pH g kg ⁻¹	Carbon ²	
			Organic	Total
Barnes loam	0–15	7.26	22.9	26.6
	15–30	7.24	19.0	23.6
	30–45	7.57	7.3	27.0
Hamerly clay loam	0–15	7.57	29.5	39.6
	15–30	7.58	22.5	35.9
	30–45	7.67	8.2	27.4
Parnell silty clay loam	0–15	7.53	34.9	43.1
	15–30	7.51	25.8	35.4
	30–45	7.59	7.9	16.7
Buse loam	0–15	7.61	31.4	39.7
	15–30	7.66	24.7	36.1
	30–45	7.90	8.7	35.1

¹ Values represent means of $n \geq 24$ for Barnes, Buse, Hamerly, and Parnell soils.

² Total C = Organic C plus carbonate C.

Table 2: ANOVA of soybean seed yield

Source	DF	P > F	Significance ¹
1995			
Variety	1	0.106	ns
Soil	3	0.005	**
1996			
Variety	1	0.020	**
Soil	3	0.072	*

¹ ns, non-significant; * Significant at $P = 0.1$; ** Significant at $P = 0.05$.

the data set; all came from Hamerly clay loam and Parnell clay loam mapping units.

Results and Discussion

Soil mapping units within the field site are rather small and none of the soils could be viewed as a classic representative. However, seed yields differed between soils (Table 2). In both 1995 and 1996, seed yields from the Buse loam (average 2.96 Mg ha⁻¹ and 2.97 Mg ha⁻¹, respectively) were less than those from the Barnes loam (average 3.45 Mg ha⁻¹ and 3.22 Mg ha⁻¹, respectively) despite the fact that these soils generally have similar characteristics. Yields from the Parnell and Hamerly soils were intermediate to those of the Barnes and Buse soils. Variances in seed yields were too large to enable measurement of differences in 1995 but yields of 9091 were greater than 9061 in 1996.

When evaluating seed yields, the ANOVA procedure assumed plots within soils were not measurably different. However, plots were arbitrarily assigned to a mapping unit based on overall characteristics of the profile even though they varied continuously. Arbitrary assignment of plots to a mapping unit enhances variance and obscures some subtle effects. Some plots assigned to a mapping unit were, in fact, only marginally different from plots in adjacent mapping units. In spite of this violation of convention/assumption, differences in yields between soils are clearly evident (Table 2).

Available P is an important determinant in soybean yield and extractable P data were examined for possible explanations of the seed yields. Mean NaHCO₃-extractable-P (B-P) concentrations in the surface 15-cm depth zone ranged from 10.5 µg g⁻¹ for the Buse loam to 16.8 µg g⁻¹ for the Parnell clay loam (Table 3). The mean B-P-values ranked in the 'upper medium' to 'high' range and application of P to such soils is usually considered unlikely to realize economic benefit. Resin-extractable P (R-P) values roughly paralleled those of the B-P. Resin-P is obtained as a fraction of all anions and the reservoir of resin adsorption sites is deliberately set at much less than the total extractable anion complement in a sample while B-P extraction attempts to remove all extractable-P. Thus an exact correlation would indicate that other chemistries were in near constant proportions to extractable-P and this result seems extremely unlikely.

The Parnell clay loam consistently had greater

Table 3: Soil Extractable P

Soil	Method ($\mu\text{g g}^{-1}$)	
	Bicarbonate	Resin ¹
1995		
Barnes	11.9 b ²	0.859 b ³
Hamerly	15.4 a	1.12 ab
Parnell	15.1 a	1.41 a
Buse	11.1 b	1.14 ab
1996		
Barnes	11.4 b	4.50 b
Hamerly	10.5 b	2.46 b
Parnell	16.8 a	13.2 a
Buse	10.9 b	2.17 b

¹The extraction procedure was modified slightly by including continuous rolling of the tubes after 1995 to improve extraction efficiency. The site in 1995 was adjacent to site of 1996.

²Values for NaHCO_3 extractable P followed by the same letter are not different ($P < 0.05$).

³Values for resin extractable P followed by the same letter in a given year within a column are not different ($P < 0.05$).

extractable-P concentrations than the other soils; this might be expected because this soil occupies the depressional areas that, over a period of time, should have received finer particles resulting from natural erosion. Cultivation accelerates erosion but the soil has been cultivated for only about a century and cultivation was initially only moderately intensive and often avoided depressions. The Hamerly clay loam occupies areas intermediate between the Parnell clay loam and the Barnes and Buse soils; extractable-P concentrations in this soil were generally similar to the Barnes and Buse soils with the exception of B-P in 1995. The Barnes and Buse soils occupy similar landscape positions, have similar textures, and the extractable-P from these soils was similar for both B-P and R-P-tests.

Thus, while (1) soybean seed yields increased weakly with extractable P concentrations (data not shown) (2) soils differed with respect to extractable P concentrations, and (3) differences in seed yields between soils were observed, the results seemed inconsistent with both realized climate and soil fertility. The inconsistencies were particularly pronounced for the Barnes and Buse soils.

While other ion extractions also differed between

Table 4: Resin-Extractable vanadium in soil

Soil	Vanadium ¹ (mol g^{-1})
1995	
Barnes	0.165 c
Hamerly	0.611 a
Parnell	0.669 a
Buse	0.403 b
1996	
Barnes	0.358 b
Hamerly	2.831 a
Parnell	2.206 a
Buse	2.935 a

¹The extraction procedure was modified slightly by including continuous rolling of the tubes after 1995 to improve extraction efficiency. Site of 1995 is adjacent to site of 1996.

Values for resin extractable V followed by the same letter in a given year are not different ($P < 0.05$).

soils (Rinke and Olness 1996), most variances in individual forms were inadequate in explaining variances in soybean yield. The relative R-E concentrations of three ions, however, were quite different; these were Li, Co, and V (Table 4). These three elements are seldom surveyed in the agronomic literature. Relative concentrations of R-E Li and Co seemed unrelated to soybean seed yield. However, seed yield tended to decrease as the concentration of R-E V increased. Again, the relationship was rather weak. The Barnes loam has less R-E V than the other soils. In all cases, V was extracted on the anion exchange resin and was probably, in view of local redox conditions, in the vanadate form (VO_3^- ; +5 valence).

Vanadium was a subject of more intense evaluation in the 1970s. At that time it was thought that it might be both an essential element and in deficient supply. However, work by Singh (1971, *Zea mays*), Hara et al. (1976; *Brassica*), Wallace et al. (1977, *Phaseolus*), and Davis et al. (1978, *Hordeum vulgare*) showed loss of yield potential with increases in vanadium content in the nutrient solution. Loss of yield potential in maize occurred when vanadium concentrations exceeded $50 \mu\text{g l}^{-1}$ (Singh 1971). In 1983, Cantley et al. showed that VO_3^- was a potent inhibitor of ATPase activity and interest in vanadium as a plant nutrient subsequently waned. At about the same time, Bowman (1983) showed

that VO_3^- and H_2PO_4^- exhibited mutual competition for accumulation in the common bread mold *Neurospora crassa*. More recently, Sklenar et al. (1995), working with phosphate concentrations similar to those found in soils, showed that increasing ambient VO_3^- concentrations decreased H_2PO_4^- uptake rates by maize roots.

Vanadate has slightly larger dimensions than its H_2PO_4^- analog with the V-O bond being about 0.5–0.6 nm longer than the P-O bond but the atomic weights (99 vs. 97) are very similar (Clark 1975, Toy 1975). The observations of Bowman et al. (1983) and Sklenar et al. (1995) suggest a direct competition between VO_3^- and H_2PO_4^- . If V is involved in an interaction with P, it seems reasonable to consider mole fraction relationships. Using VO_4^- as a mole fraction of only Group 5 elements (As, V, and P) provided a closer explanation of yield variance but the greatest resolution was obtained when only VO_3^- and H_2PO_4^- were used (Fig. 2). The data from 1995 are unconvincing but, with an improvement in the extraction procedure, the results in 1996 are significant ($P < 0.05$). Interestingly, when the data of Singh (1971), Hara et al. (1977), Wallace et al. (1977), and Davis et al. (1978) are plotted on the same mole fraction basis they show very close agreement (Fig. 3).

Differences between varieties are obvious (Fig. 2). The intercepts of the relationships ranged from 3.1 to 3.2 Mg ha^{-1} indicating rather uniform potential seed yields among the cultivars. The results are consistent with a multiple allelic character. The fact that varieties differ in their apparent response to vanadium offers hope for development of improved cultivars. In this regard, Wilkinson and Duncan (1993) also found varietal differences in response to the presence of VO_3^- in sorghum (*Sorghum bicolor* L.) so the prospect seems to hold potential.

Variety Pi 9091 seems relatively unaffected by resin-extractable V. This variety is resistant to development of Fe-chlorosis, a visible necrosis caused by inadequate Fe early in the growth of soybean. Variety Pi 9061 is regarded as moderately sensitive to Fe-chlorosis and it suffers an appreciable yield loss (as much as 20%) as the V:(V + P) ratio approaches 0.15. Because V is a potent inhibitor of plasma lemma ATPases involved in nutrient accumulation by the plant, its presence may accentuate Fe-chlorosis when Fe is marginally available. Soils in the region are rather rich in total Fe but amounts of accessible Fe are often marginally adequate for soybean. The expression of Fe-chlorosis is climatically sensitive and this further suggests

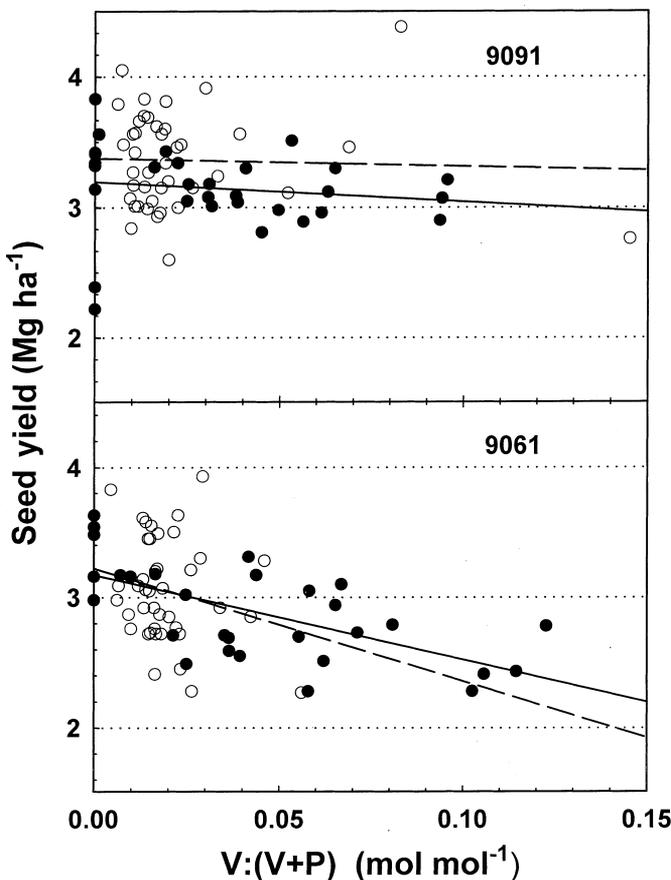


Fig. 2: Soybean seed yield as a function of V:(V + P) molar ratios in 1995 and 1996. Slope of the regression models were nonsignificant in 1995 but significant for 9061 in 1996. Dashed lines represent data collected in 1995 and solid lines represent data collected in 1996

that the rate of root growth, which would be slowed in the presence of V (Mikuš et al. 1995), is a factor in the expression of Fe-chlorosis. The fact that V toxicity can occur without visible symptoms or significant biochemical signal within the plant makes this a very insidious poison.

In summary, circumstantial evidence has been obtained for a subtle V by P interaction with soybean seed yield. The evidence of a V by P interaction was enabled through resin-extraction techniques. Expression of a V by P interaction appears genotypically dependent; this supports a conclusion that V is an inhibitive factor in soybean seed yield. Yield losses of as much as 20% were observed with molar V:(V + P) ratios of ≤ 0.15 . We have insufficient information on the natural occurrence of V and relative resin-extractable V:P ratios to discuss the potential economic implications of this observation at other sites.

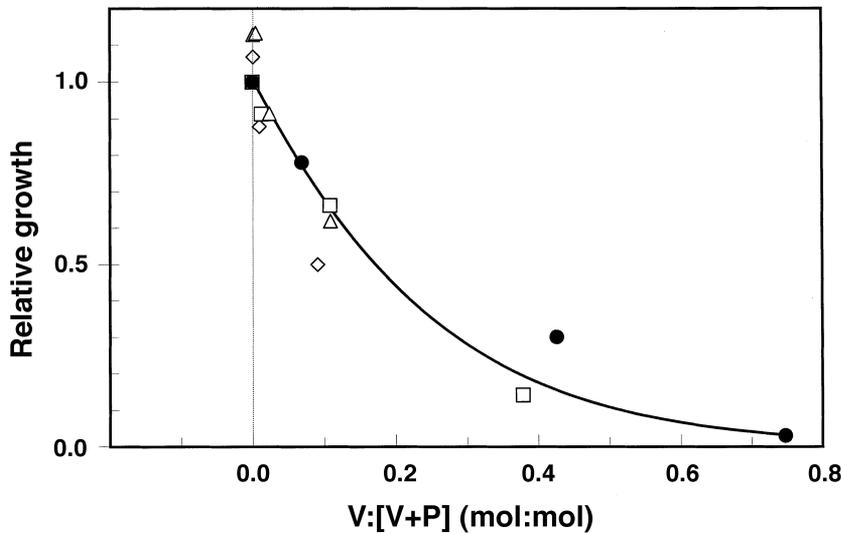


Fig. 3: A construction of the effect of V:(V + P) ratios on plant productivity for data extracted from literature sources. The solid line is a General Energy Model for Limited Systems fit to the data; $Y = a*(b + \{(e^{-k(x-f)} - e^{k(x-f)}) / (e^{-k(x-f)} + e^{k(x-f)})\})$, where $a = 2$, $b = 1$, $f = -0.217$, and $k = 2.5$ with $R^2 > 0.94$. Symbols represent data from: Δ , Singh (1971); \diamond Wallace et al. (1977); \square , Hara et al. (1977); and \bullet , Davis et al. (1978)

Zusammenfassung

Ionische Verhältnisse und Ernteerträge. I. Vanadate and Phosphate auf Sojabohnen

Durch Harzextraktionstechniken erhält man eine Reihe von ausziehbaren Elementen/Ionen aus einer einzigen Probe. Es ist daher möglich, Interaktionen zwischen den Nährstoffen zu beschreiben und die Wirkung toxischer Ionen auf das Pflanzenwachstum zu bestimmen sowie genetische Eigenschaften von Pflanzen zu identifizieren, die für eine optimale Nutzungseffizienz benötigt werden. Um die Eignung dieser Technik zu demonstrieren, wurde ein Feldexperiment auf einer 3,24 ha großen Fläche mit fünf Kartierungseinheiten durchgeführt. Der Standort war in drei gleichgroße Abschnitte aufgeteilt; es wurden Mais (*Zea mays* L.), Sojabohne (*Glycine max* L. Merr.), Weizen (*Triticum aestivum* L.) angebaut. Die Fruchtfolge wurde jährlich geändert und jede Fruchtfolge wurde in 360 Parzellen mit einer Größe von 3 m mal 30 m aufgeteilt. Drei Sojabohnensorten (9091, 9061 und 704), wurden in aufeinanderfolgenden Streifen von 4 Reihen in Abständen von 0,75 m Abständen angesät. Die Bodenproben wurden mit Anionen- und Kationenharzaustauschern behandelt. Die Harzaustauscher wurden mit Säure ausgewaschen und die Eluate induktiv und in Verbindung mit einer plasmaemissionstechnischen Spektrografie untersucht. Die Bodenproben wurden auf Bikarbonat, auswaschbares P, organisches C, gesamt N und pH untersucht. Die Samenerträge waren für Sorten und Böden ($p < 0,05$) unterschiedlich. Die Samenerträge variierten von 2390 kg bis 3470 kg ha⁻¹. Die Samenerträge der Sorten 704 und 9061 verringerten sich um 20 % (etwa 540 kg ha⁻¹), wenn sich das Verhältnis des extrahierbaren V:(V + P) auf 0,15 Mol/Mol erhöhte. Das extrahierbare V war für die verschiedenen Bodeneinheiten unterschiedlich. Bodenbikarbonat, extrahierbares P, organisches C, gesamt N und pH hatten nur geringfügige Auswirkungen auf den Samenertrag.

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