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Amedee, Gaston; Peech, Michael

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9. ABSTRACT

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THE SIGNIFICANCE OF KCl-EXTRACTABLE Al(III) AS AN INDEX TO LIME REQUIREMENT OF SOILS OF THE HUMID TROPICS[1]

G. AMEDEE AND M. PEECH
Cornell University, Ithaca, New York

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ABSTRACT

The significance of KCl-extractable Al(III) in acid soils of the humid tropics for determining lime requirement was evaluated by comparing the amounts of Al(III) extracted with 0.1 *N* KCl and 1 *N* KCl. The cumulative amounts of Al(III) removed upon successive extractions of the soils with 1 *N* KCl were found to be greater than those removed by exhaustive successive extractions with 0.1 *N* KCl. This could, in part, be attributed to the release of H^+, owing to the increase of the surface negative charge in the presence of the higher electrolyte concentration at pH values above the zero point of charge, which in turn would be expected to result in an equivalent amount of KCl-extractable Al(III).

The value of (pH-⅓pAl) for the first and subsequent 0.1 *N* KCl extracts was found to remain remarkably constant and nearly equal to 2.8, corresponding to the solubility of gibbsite. This would indicate that the concentration of Al(III) in 0.1 *N* and 1 *N* KCl extracts was determined by the solubility of precipitated $Al(OH)_3$ and amorphous $Al(OH_3$ originally present in the soil. Gibbsite was found to be virtually insoluble in 1 *N* KCl at pH values above 4. The significant conclusion that may be drawn is that the amount of Al(III) extracted by 1 *N* KCl does not represent the intrinsic property of the acid soils of the humid tropics.

INTRODUCTION

In acid soils exchangeable aluminum is generally recognized as the predominant cation as determined by exchange process with a neutral salt solution, like KCl. This determination is based on the assumption that aluminum ions are counter-ions balancing excess permanent negative charge on the surface of the solid phase. This method for determining exchangeable aluminum by extraction, however, is not satisfactory. It has been shown that the aluminum extracted by a salt solution represents both exchangeable as well as nonexchangeable aluminum (Dewan and Rich 1970; Rich 1970). The method for determining exchangeable aluminum by extraction with 1 *N* KCl, as applied to acid soils of the humid tropics, is quite ambiguous inasmuch as permanent charge of these soils is low. These soils are known to contain appreciable amounts of crystalline and amorphous iron and aluminum oxides and hydroxides, kaolinite and halloysite. Consequently, they have a large hydroxyl surface from exposed octahedral AlOH and FeOH groups. The charge of these materials is known to be largely pH dependent (Deshpande et al. 1964; Sumner and Davidtz 1965; Tweneboah et al. 1967). Van Raij and Peech (1972) have shown that soils that occur extensively in the humid tropics possess different electrochemical properties as compared to most soils of the temperate regions. They are characterized by exhibiting a constant surface potential. The charge of these soils could be increased by increasing the concentration of salt as well as pH. Consequently, the concentration of KCl used in the extraction of aluminum from many acid soils of the humid tropics should have a significant effect on the electric charges of the soil particles. Moreover,

[1] Agronomy Paper No. 1112, Department of Agronomy, Cornell University, Ithaca, N.Y. 14853. Supported by grant csd 2834 to Cornell University by the Agency for International Development under the Institutional Grants Program established by Section 211(d) of the Foreign Assistance Act of 1961 as amended in 1966. Paper based on a thesis by the senior author in partial fulfillment of his Ph.D. requirements.

it is conceivable that the aluminum hydroxide and gibbsite in these soils should be readily soluble at any given pH in KCl solution and the value of ($pH-\frac{1}{3}pAl$) of the soil extracts should give in indication of the degree of saturation of the extract with respect to aluminum hydroxide. The value for ($pH-\frac{1}{3}pAl$) in aqueous solutions in equilibrium with gibbsite was found to be 2.83 (Frink and Peech 1962b). No data, however on the solubility of freshly precipitated or amorphous $Al(OH)_3$ and gibbsite in 1 *N* KCl could be found in the literature; nor could the value of ($pH-\frac{1}{3}pAl$) for 1*N* KCl extracts be calculated owing to the lack of information as to the magnitude of the activity coefficients of Al^{3+} and $AlOH^{2+}$ in 1 *N* KCl. The activity coefficients of Al^{3+} for 0.1 *N* KCl, however, can be calculated accurately using the Debye-Hükel equation. The object of this investigation was to study the significance of KCl-extractable aluminum which is commonly used for estimating the lime requirement of acid soils of the humid tropics.

MATERIALS AND METHODS

Description of Soils

Four highly-weathered acid soils of the humid tropics were used in this investigation. Two were subsoils of an Oxisol and an Ultisol from Puerto Rico. The other two were samples of Ap and B2 horizons of an Oxisol collected in the State of São Paulo, Brazil. One surface sample of a New York soil was also included for comparison. The names used in this study were adapted from the Soil Taxonomy of the United States (Soil Survey Staff 1970). A description and some properties of the soils are given in Table 1. The zero point of charge of soils 1 and 2 was known to be 3.9 and 6.2, respectively (Van Raij and Peech 1972), but the zero point of charge of soils 3 and 4 was not determined.

Analytical methods and procedures

In one experiment, 5-g samples of the air-dried soils 3, 4, and 5 were weighed into 90-ml glass centrifuge tubes, mixed for 5 min with 50 ml of 1.0 *N* KCl using a motor stirrer, and centrifuged for 15 min at 2100 rpm in an International centrifuge, Model K. The clear supernatant solution was decanted and saved for the determination of pH and aluminum by the aluminon method (Frink and Peech 1962a). This extraction procedure was repeated successively 20 times. A correction was applied for the occluded salt solution after each extraction in bringing the total volume of the extracting solution to 50 ml. The same extraction procedure was repeated with the same soils using 0.1 *N* KCl extracting solution.

In another experiment, a 10-g sample of soils 1 and 2 was brought to pH 3.0, 3.5, 4.0, and 4.5 in a 50-ml beaker by adding a known amount of standard HCl to the soil suspension containing 20 ml of 0.1 *N* KCl. Soils 4 and 5 were adjusted similarly to pH 3.0 and 3.5. The suspensions were placed in a humidified chamber for 1 week and the pH values were maintained approximately constant by addition of standard HCl.

TABLE 1

Description and some properties of the soils studied

Soil no.	Soil name	Depth	Organic matter	pH in		Exchangeable	
				H_2O	0.01 *M* $CaCl_2$	Ca^{2+}	Mg^{2+}
		cm	%			me/100 g	me/100 g
1	Acrorthox-Ap (Brazil)	0–20	4.32	5.30	4.80	1.76	0.67
2	Acrorthox-B2 (Brazil)	100–200	1.14	5.90	5.70	0.33	0.12
3	Haplorthox-B1 (Puerto Rico)	61–91	1.09	5.00	4.25	0.50	0.52
4	Tropohumult-B1 (Puerto Rico)	61–91	0.59	4.61	4.00	1.60	0.20
5	Fragiochrept-Ap (New York)	0–15	3.23	4.40	4.00	0.99	0.21

At equilibrium, the soil suspensions were then transferred to 90-ml glass centrifuge tubes using additional 0.1 *N* KCl to effect the transfer. The volume was adjusted to 50 ml, and, after stirring, the suspensions were allowed to stand overnight. The soil suspensions were then centrifuged and the supernatant solution was decanted. The pH values and aluminum concentrations were then determined in the extracts by the glass electrode and the aluminon method (Frink and Peech 1962a). The same determinations were made using 0.01 *M* $CaCl_2$ instead of 0.1 *N* KCl as the extracting solution.

RESULTS AND DISCUSSION

Effect of KCl concentration on the amount of exchangeable aluminum extracted

The cumulative amounts of Al(III) removed upon successive extractions by the two concentrations of KCl have been plotted in Figs. 1, 2, and 3. The pH values, the total amount of Al(III) extracted, and the values of (pH-$\frac{1}{3}$pAl) for the 0.1 *N* KCl soils extracts are given in Table 2. The analytical results of the experiment in which the pH of the soils was adjusted to different values and the calculated values for (pH-$\frac{1}{3}$pAl) are shown in Table 3.

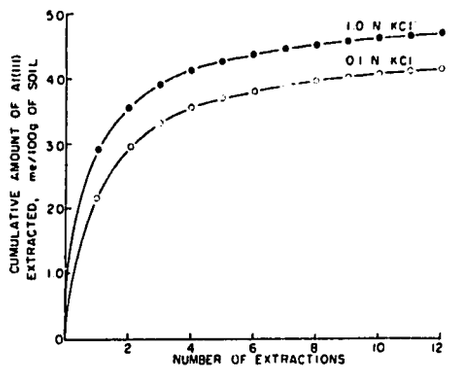


FIG. 1. Cumulative amounts of Al(III) extracted by 0.1 *N* and 1.0 *N* KCl from Haplorthox-B1 (soil 3).

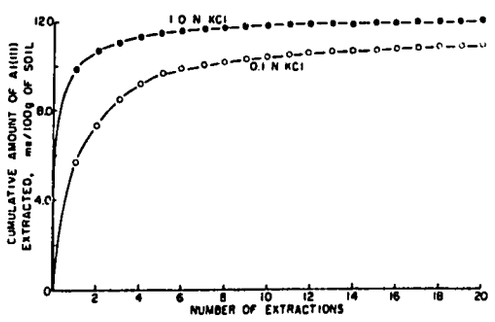


FIG. 2. Cumulative amounts of Al(III) extracted by 0.1 *N* and 1.0 *N* KCl from Tropohumult-B1 (soil 4).

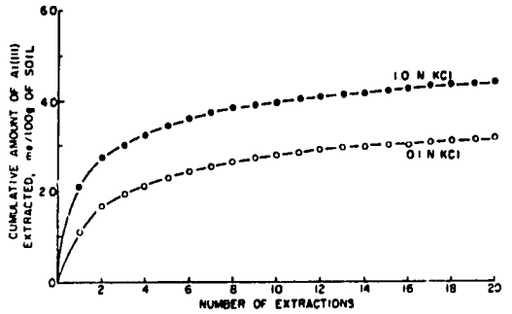


FIG. 3. Cumulative amounts of Al(III) extracted by 0.1 *N* and 1.0 *N* KCl from Fragiochrept-Ap (soil 5).

The amount of aluminum obtained even upon exhaustive, successive extraction by 1 *N* KCl was found to be always greater than that extracted by 0.1 *N* KCl as shown in Figs. 1, 2, and 3. Hence, the inefficiency of the 0.1 *N* KCl solution to exchange Al^{3+} cannot be explained by the mass action effect. It is evident that the cumulative amount of aluminum obtained upon exhaustive extractions is dependent on the concentration of KCl. The soils from Puerto Rico (soils 3 and 4) are dominated by iron and aluminum oxides and are known to exhibit a constant surface potential. The New York soil, 5, is known to contain a large amount of pH-dependent charge[2] even though the clay fraction has been found to consist of illite, and other 2:1 clay minerals having a permanent charge. Presumably, the surfaces of these materials are coated with hydrous iron and aluminum oxides which essentially determine the colloid chemical properties. One possible mechanism of this salt effect on the amount of aluminum extracted can be explained by the following stepwise reactions:

$$ROH + KCl \rightarrow RO^{-}...K^{+} + HCl \quad (1)$$

$$Al(OH)_3 + 3HCl \rightarrow AlCl_3 + 3H_2O \quad (2)$$

where *R* represents an atom of Fe(III) or Al(III) belonging to the solid phase (Van Raij and Peech 1972). On the negative side of the zero

[2]D. O. Ataga. 1968. Cation exchange properties of some acid soils. Ph.D. thesis. Cornell University, Ithaca, N.Y. 14853.

TABLE 2

Values of (pH-⅓pAl) for the soils extracts upon successive extraction of the soils with 0.1 N KCl

Soil no.	Soil name	Extraction number	pH	Total Al	pAl	pH-⅓pAl
				mM		
3	Haplorthox-B1 (Puerto Rico)	1	4.20	0.73	3.90	2.90
		2	4.35	0.26	4.35	2.90
		3	4.45	0.12	4.68	2.89
		4	4.49	0.07	4.92	2.85
4	Tropohumult-B1 (Puerto Rico)	1	4.00	1.89	3.51	2.83
		2	4.11	0.58	4.02	2.77
		3	4.22	0.38	4.17	2.83
		4	4.30	0.23	4.38	2.84
5	Fragiochrept-Ap (New York)	1	3.85	0.38	4.17	2.46
		2	3.98	0.18	4.50	2.48
		3	4.05	0.09	4.80	2.45
		4	4.12	0.06	4.98	2.46

TABLE 3

The value of (pH-⅓pAl) for the soils extracts as found by extracting the soils with 0.1 N KCl and 0.01 M $CaCl_2$ adjusted to different pH values

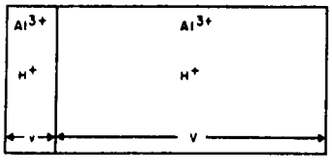
Soil no.	Soil name	0.1 N KCl				0.01 M $CaCl_2$			
		pH	Total Al	pAl	pH-⅓pAl	pH	Total Al	pAl	pH-⅓pAl
			mM				mM		
1	Acrorthox-Ap (Brazil)	3.40	2.82	3.33	2.29	3.55	2.76	3.15	2.50
		3.75	1.15	3.72	2.51	3.76	0.36	3.99	2.43
		4.26	0.19	4.50	2.76	4.19	0.13	4.44	2.71
		4.70	0.02	5.52	2.86	4.61	0.01	5.61	2.74
2	Acrorthox-B2 (Brazil)	3.55	3.61	3.24	2.47	3.55	2.59	3.18	2.49
		3.81	1.91	3.48	2.65	3.85	1.02	3.57	2.66
		4.20	0.33	4.26	2.78	4.15	0.21	4.23	2.74
		4.60	0.03	5.31	2.83	4.55	0.02	5.28	2.79
4	Tropohumult-B1 (Puerto Rico)	3.28	6.87	2.97	2.29	3.31	3.01	3.12	2.27
		3.65	5.33	3.06	2.63	3.68	1.86	3.30	2.58
		3.90*	4.09	3.18	2.84	3.95*	0.93	3.60	2.75
5	Fragiochrept-Ap (New York)	3.35	3.57	3.24	2.27	3.39	2.27	3.24	2.31
		3.72	2.14	3.45	2.57	3.80	0.91	3.60	2.60
		3.95*	1.42	3.63	2.74	4.29*	0.25	4.23	2.88

* Unadjusted soil pH.

point of charge, the effect of the addition of salt should be to increase the surface negative charge of the soil particles by dissociation of the –OH groups. The hydrogen ions released should, in turn, dissolve the aluminum hydroxide present in the soils to produce equivalent amounts of Al^{3+} provided, of course, there is no re-adsorption of the released H^+ by some other soil surfaces. Thus the greater the concentration of salt in such a system, the greater should be the amount of H^+ released by dissociation of the surface hydroxyl groups and the greater should be the amount of "exchangeable" aluminum extracted from the soil.

The activity of Al^{3+} and $AlOH^{2+}$ in the 0.1 *N* KCl extract can be calculated (Lindsay et al. 1959). The pH of the extract was found to increase upon successive extractions but the value of (pH-⅓pAl) remained constant (Table 2). Because the value of (pH-⅓pAl) in the presence of gibbsite in equilibrium with the soil solution was found to be constant and very nearly equal to 2.8 (Lindsay et al. 1959; Frink and Peech 1962b), it is suggested that the soils from Puerto Rico were in equilibrium with gibbsite in the first and subsequent extractions. It is conceivable that Al^{3+} in the double layer solution could be readily precipitated as $Al(OH)_3$ upon dilution with 1 *N* KCl solution. In a previous study (Frink and Peech 1963) of the hydrolysis of Al^{3+} in dilute aqueous solutions, Al^{3+} was found to precipitate as $Al(OH)_3$

upon dilution. As an example of the mechanism of precipitation of $Al(OH)_3$ in KCl, consider the following system:



where v is the volume of the double layer solution containing Al^{3+} and H^+ and V the volume of KCl solution added at the time of the extraction. Upon dilution with KCl, the concentrations of Al^{3+} and H^+ in the double layer solution are exchanged and diluted by a factor of $v/(V + v)$. The ion activity ratio $(Al^{3+})^{1/3}/(H^+)$ must be changed accordingly in the process to maintain equilibrium with gibbsite. If H^+ in the double layer solution were to be diluted tenfold, for example, Al^{3+} would have to be diluted a thousandfold. Thus the ion activity ratio $(Al^{3+})^{1/3}/(H^+)$ must increase or the ion activity product $(Al^{3+})(OH^-)^3$ should increase with dilution and some of the exchangeable Al^{3+} must be precipitated as $Al(OH)_3$. The degree of saturation of the extracts with respect to $Al(OH)_3$ is indicated by the value of (pH-$^1/_3$pAl) listed in Table 2. It should be noted that the values of (pH-$^1/_3$pAl) obtained for the New York soil would suggest undersaturation with respect to amorphous $Al(OH)_3$.

As shown in Table 3, when HCl was added to the soil suspensions, the system remained undersaturated with respect to $Al(OH)_3$. This would suggest that aluminum ions were not generated rapidly enough upon dissolution of $Al(OH)_3$ in the presence of H^+. Contrary to expectation, it was observed that the amounts of aluminum extracted were not related to the zero point of charge of the soils. The lower the pH of the suspensions, the greater was the amount of aluminum extracted from the soils. Apparently the concentration of Al(III) in 0.1 N KCl and 0.01 M $CaCl_2$ was determined by the solubility of some amorphous $Al(OH)_3$.

SOLUBILITY OF FRESHLY PRECIPITATED Al $[OH]_3$ AND GIBBSITE IN 1 N KCl

Procedure

In order to determine the extent of solubility of amorphous aluminum hydroxide in KCl, a study of Al(III) released in KCl upon successive extraction of freshly precipitated $Al(OH)_3$ with 1 N KCl was made following the same procedure used in the determination of Al(III) in soils. Freshly-precipitated $Al(OH)_3$ was prepared by dissolving $AlCl_3$ in an appropriate amount of H_2O and neutralizing the solution by adding slowly a concentrated solution of NaOH from a buret. The mixture was stirred constantly with a magnet bar until $Al(OH)_3$ was precipitated. Then the gel was washed four times with H_2O by centrifugation and dialyzed for 5 days until the conductance of the dialyzate was about that of distilled H_2O. The pure gel was dried on a steam hotplate at 45°C to avoid any crystallization. The x-ray diffraction pattern of the powder did not indicate the existence of any crystalline form of $Al(OH)_3$. The zero point of charge was determined by potentiometric titration and found to be 8.1 which is in good agreement with values reported by previous investigators (Schuylenborgh 1949; Jackson 1963). It should be noted, however, that the zero point of charge of $Al(OH)_3$ is not absolute. It has been shown (Schuylenborgh 1951) that the value obtained depends on the method of preparation.

Gibbsite used in this study was a commercial gibbsite from Aluminum Company of America, Pittsburg, Pennsylvania, labeled as C-33. A description of this material can be found elsewhere (Kittrick 1966). The sample was washed four times with H_2O using a centrifuge. The zero point of charge was found to be 4.8 as determined by the potentiometric method.

A 0.2-g sample of the freshly precipitated $Al(OH)_3$ and gibbsite was extracted with 50 ml of 1 N KCl adjusted to pH 3.0, 4.0, 4.5. An extraction with unadjusted 1 N KCl solution was also carried along for comparison. This extraction procedure was repeated eight times using the same method that has been already described for the determination of aluminum in soils.

In still another experiment, different amounts of standard HCl were added to a mixture of 0.2 g of $Al(OH)_3$ and 20 ml of 1 N KCl. The mixture was allowed to react for 1 week after which time an additional volume of 1 N KCl was added to bring the volume to 50 ml. Then, the suspensions were mixed and allowed to stand overnight. The pH values and the Al(III) concentrations were determined in the extracts after centrifugation.

RESULTS AND DISCUSSION

The cumulative amount of Al(III) extracted by 1 *N* KCl from freshly precipitated $Al(OH)_3$ and gibbsite have been plotted against the number of extractions in Fig. 4. It can be seen in Fig. 4 that when the 1 *N* KCl extracting solution was adjusted to pH 3.0 or 4.0, a constant amount of Al(III) was extracted during each extraction of the freshly precipitated $Al(OH)_3$. When KCl was adjusted at pH 4.5 or unadjusted, the concentration of aluminum decreased slightly upon successive extraction. It is obvious that freshly precipitated $Al(OH)_3$ is readily soluble in 1 *N* KCl and, as expected, the lower the pH of the KCl, the greater was the solubility of $Al(OH)_3$, whereas gibbsite was found to be virtually insoluble in 1 *N* KCl at pH values above 4. The results of the experiment in which different amounts of acid were added to freshly precipitated $Al(OH)_3$ suspended in 1 *N* KCl are shown in Table 4. It can be seen that the solubility of $Al(OH)_3$ became significant only after saturation of the surface with H^+ by adsorption. Consequently, it was observed that, upon successive extraction of the freshly precipitated $Al(OH)_3$ with adjusted 1 *N* KCl extracting solution, a constant amount of H^+ was adsorbed during each extraction. As a result, the pH of the successive extracts remained constant. Upon saturation of the surface, the added H^+ should react with $Al(OH)_3$ to form aluminum ions in the solution. At any given pH

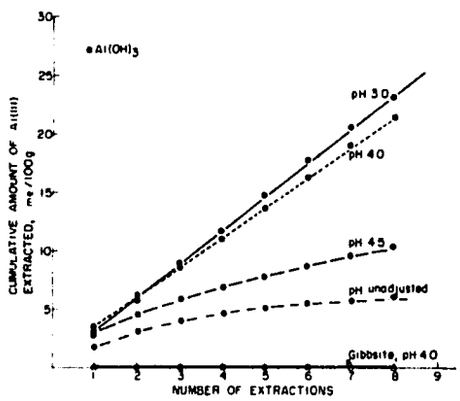


FIG. 4. Cumulative amounts of Al(III) extracted from freshly precipitated $Al(OH)_3$ and gibbsite by 1 *N* KCl adjusted to different pH values. The values given on the ordinate should be multiplied by 10 for the curve showing the amount of aluminum dissolved at pH 3.

TABLE 4

Solubility of freshly precipitated $Al(OH)_3$ in 1 N KCl to which different amounts of HCl had been added

HCl added	pH of extract	Al (III) found in extract	H^+ adsorbed
me/100 g		me/100 g	me/100 g
0	5.30	3.6	—
20	4.90	7.7	15.9
60	4.60	38.1	25.5
123	4.45	102.7	23.9
290	4.22	270.0	23.4

of the extract from the freshly precipitated $Al(OH)_3$, the amount of Al(III) extracted with 1 *N* KCl was found to be much greater than that extracted from the soil. The reason for this discrepancy is not clear. Perhaps the freshly precipitated material has a much greater specific surface than the amorphous $Al(OH)_3$ encountered in soils or the rate of dissolution may differ in the two cases. In any case, it should be obvious that the release of Al(III) from acid tropical soils cannot be attributed to the solubility of gibbsite which is virtually insoluble in 1 *N* KCl. Moreover, the determination of aluminum in highly weathered soils by the extrapolation procedure, as suggested by some investigators (Skeen and Sumner 1967), by plotting the cumulative amounts of Al(III) extracted as a function of the number of extractions with 1 *N* KCl solution adjusted to the pH of the soil is also subject to error, inasmuch as amorphous $Al(OH)_3$ is readily dissolved in acid extracting solution. Also the amount of $Al(OH)_3$ dissolved should be considerably lower during the first three or four extractions because of the common ion effect, resulting from exchange of the adsorbed Al^{3+} by the added salt solution, on the solubility of $Al(OH)_3$. The linear portion of the curve obtained is due to the dissolution of some amorphous $Al(OH)_3$ and the degree of the slope should be determined by the solubility of $Al(OH)_3$. The greater the slope of the linear portion of the curve, the lower will be the apparent value for the amount of "exchangeable" aluminum obtained by extrapolation. Thus the amount of exchangeable aluminum determined by this procedure will vary, depending on the pH of the extracting solution and the amount of amorphous $Al(OH)_3$ existing in the soil.

It should be obvious that the amount of

KCl-extractable Al(III) does not represent an intrinsic property of acid soils of the humid tropics. Thus the extraction of aluminum from soils with extracting solution having low pH values and/or high salt concentration could lead to erroneous evaluation of the lime requirement of soils of the humid tropics. Because of appreciable amounts of pedogenic aluminous chlorite in these soils and its great capacity to react with lime, the estimation of the lime requirement of these soils should be based on some method which would reflect more accurately the neutralization of soil acidity by lime under field conditions. In practice, however, the actual amount of lime that should be applied to bring the soil to some desired pH value will be determined by the kind of crop to be grown (Pearson 1975) and the amount of acid-forming fertilizer that is to be applied.

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