May 23, 1972

To the Graduate Council:

I am submitting herewith a thesis written by John Walter Odom entitled "A Study of Methods of Measuring Exchangeable Aluminum and Their Correlation with Root Damage from Aluminum Toxicity." I recommend that it be accepted for nine quarter hours of credit in partial fulfillment of the requirements for the degree of Master of Science, with a major in Agronomy.

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A STUDY OF METHODS OF MEASURING EXCHANGEABLE ALUMINUM AND THEIR CORRELATION WITH ROOT DAMAGE FROM ALUMINUM TOXICITY

A Thesis

Presented to

the Graduate Council of

The University of Tennessee

In Partial Fulfillment

of the Requirements for the Degree

Master of Science

---

by

John Walter Odom

June 1972

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ABSTRACT

Although it is known that aluminum ions are toxic to plants, very little is known about the quantity of exchangeable aluminum needed to produce damage. The purpose of this study was to evaluate three common measures of exchangeable aluminum and to correlate the amounts measured with plant root damage. Exchangeable aluminum was measured by titration of the whole soil, fluoride titration and colorimetric determination. All three determinations were found adequate for prediction of aluminum damage. There is a correlation between pH and exchangeable aluminum, but it is not adequate for prediction purposes. It was found that root damage was likely if more than two meq/100g of exchangeable aluminum were present and the pH was less than five. The existance of organic matter-aluminum complexes in the soils studied was not shown.

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CHAPTER I

INTRODUCTION

It is helpful in evaluating soils for growing crops to have some practical measurement which will predict aluminum toxicity. The pH has been used as a criterion for liming for many years, but a low pH does not necessarily lead to aluminum toxicity. It was the objective of this study to evaluate some common measures of exchangeable aluminum as predictors of toxic conditions and to determine criteria for predicting toxic conditions. An attempt was also made to determine whether organic-aluminum complexes existed in the soils studied. It was thought that such complexes might be of importance in interpreting titration curves used for predicting aluminum toxicity.

CHAPTER II

LITERATURE REVIEW

The acidity found in soils generally falls into three categories: strong inorganic acids; aluminum species; and organic matter (2,12,24). Clay minerals are apparently unstable in the presence of hydrogen ions. Any addition of excess free acid causes a breakdown of the clay with release of aluminum ions and neutralization of the hydrogen ions by hydroxide ions from the clay (17,22). High concentrations of hydrogen ions are found only in special situations such as where sulfur is being oxidized. Aluminum ions adsorbed on the clay exchange sites are apparently the main acid species in most acid subsoils. The hydrolysis of aluminum ions in the presence of free salt controls the hydrogen ion concentration in the absence of free acid or organic matter (22). Polymerized aluminum hydroxide interlayers may be important in some subsoils (2,4,9,11,13,14,19,25). These interlayers carry a positive charge and exist as islands between the layers of three-layer clay minerals (2,8). They reduce the Cation Exchange Capacity (CEC) of the soil but prevent collapse of the clays and trapping of potassium (6). Various acid sites on organic matter appear to be the dominant acid factor in surface mineral horizons. The types of organic acid sites found in soils are highly variable and poorly understood (12). It has been suggested that organic matter may complex aluminum ions or aluminum hydroxide polymers (19). Organic matter-aluminum complexes may be important in surface soils which contain smaller amounts of exchangeable

aluminum and more organic matter than subsoils. Free acids, exchangeable aluminum, hydroxy aluminum interlayers and organic acids account for most of the acidity found in soils.

Aluminum is apparently the most common toxic ion in most acid mineral soils. Hydrogen ions are not directly harmful to plants unless the pH is less than four, which may occur during the oxidation of sulfides and in other limited situations. The organic acids found in mineral soils as the result of organic matter decomposition are also normally found as plant metabolites and are usually found in soils in very low concentrations (2). Aluminum is known to suppress plant growth (1,2,10,18). Under toxic aluminum conditions plant main roots are thick, swollen and distorted. Lateral roots have a stubby appearance and never grow normally. The restriction of root systems by aluminum is apparently an inhibition of cell division rather than cell elongation. Aluminum toxicity decreases shoot/root ratios in nutrient solutions over long growth periods. Shoot growth is not affected as early in the growing period as root growth. Shoots are affected by a lack of phosphorus due to reduced translocation (15). Aluminum is the primary toxic component of mineral acid soils and it affects plants by inhibiting cell division and mineral translocation.

Acidity in soils is usually measured in one of the following ways: pH; titration of soil; titration of extract; colorimetry; buffers; or fluorescence. The pH measures the acid concentration but does not give a direct indication of the amount of acidity or a breakdown into types of acidity (2). Titrations of whole soils give an indication of the relative amounts of the different types of acidity

(Figures 1 and 2) but the actual amount measured is a function of the experimental conditions (3,12,16,17,20,21,24). The standard fluoride titration of soil extracts gives values for both total acidity and aluminum. This titration requires the soil to be extracted where the whole soil titration does not (23). Colorimetric methods for determining aluminum in soil extracts are very sensitive but are difficult and time consuming (26). A newer and less well tried fluorimetric method of determining aluminum is said to be even more sensitive than the colorimeter methods (5). Equilibration of buffers with soils is sometimes used as a method of estimating lime requirements. A particular buffer method usually works only for the soils in a limited area and this method, like all laboratory methods for measuring acidity, must be correlated with field experiments in order to predict lime needs (22). All of these methods have been used to measure soil acidity or the exchangeable aluminum component of soil acidity with some success.

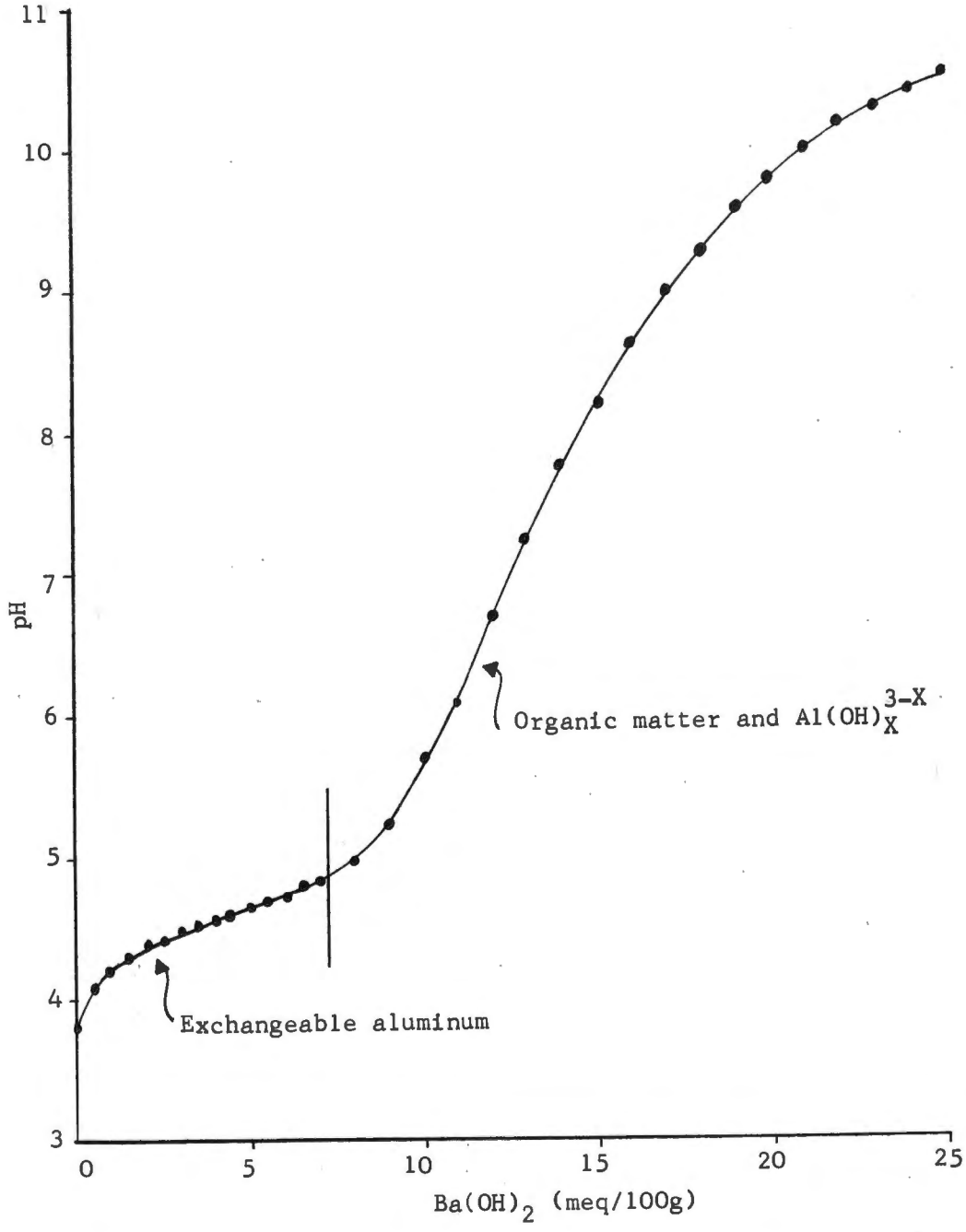


Figure 1. Potentiometric titration of Routon B2g.

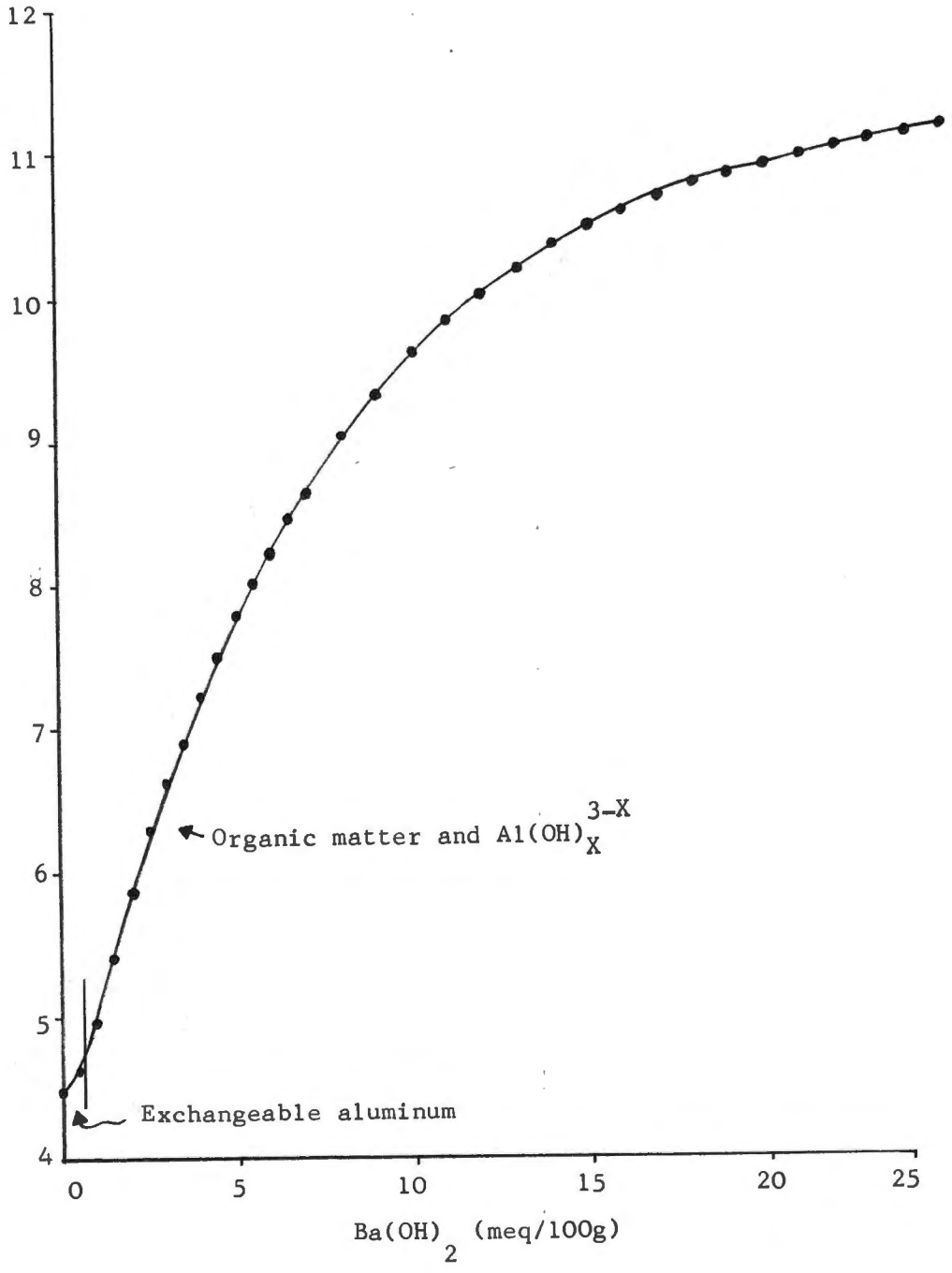


Figure 2. Potentiometric titration of Etowah Ap.

CHAPTER III

METHODS AND MATERIALS

The soils used for this study are listed in Table I. The soils were sampled for this study and are not necessarily representative of the soil types.

Exchangeable aluminum in the soils used was determined by potentiometric titrations of the whole soils, by fluoride titrations of the soil extracts and by colorimetric determinations on the soil extracts. The potentiometric titrations were run on ten grams of oven-dry soil in 200 milliliters of normal potassium chloride under an inert gas atmosphere, using 0.1 N barium hydroxide as the titrant. Potassium chloride was used to speed equilibrium by forcing the aluminum on the exchanger into solution. The inert gas atmosphere was used to avoid carbon dioxide reaction with the titrant during the titration. Barium hydroxide was used in preference to sodium hydroxide because barium exchanges with aluminum better than sodium exchanges with aluminum. Exchangeable aluminum was taken to be the amount of base consumed to the first break (Figures 1 and 2). In preparation for the fluoride titration and the colorimetric method ten grams of each soil were extracted with 200 milliliters of normal potassium chloride over a period of not less than two hours. The standard soil survey fluoride titration was used on each soil. This method consists of titrating the extract to a phenolphthalein endpoint with 0.1 N sodium hydroxide to determine a value for total acidity. Sodium fluoride is then added to complex

TABLE I

SOILS USED IN THE STUDY

Sample Soil	Horizon	Location (county)
1. Muskingum fine sandy loam	A	Knox
2. Muskingum fine sandy loam	B	Knox
3. Routon silt loam	Ap	Henry
4. Routon silt loam	A3g	Henry
5. Routon silt loam	B2g	Henry
6. Sequoia silty clay loam	B	Knox
7. Tellico loam	B	Knox
8. Falaya silt loam	Ap	Fayette
9. Falaya silt loam	IIA2b	Fayette
10. Center silt loam	Ap	Henry
11. Center silt loam	B1g	Henry
12. Center silt loam	B2g	Henry
13. Fullerton cherty silt loam	Ap	Knox
14. Etowah silt loam	Ap	(Saturated with Al^{+3})
15. Etowah silt loam	Ap	Knox
16. Clarksville cherty silt loam	C1	Roane
17. Clarksville cherty silt loam	C1	Meigs
18. Hartsells loam	C1	Fentress

the aluminum and the excess base is back titrated with 0.1 N hydrochloric acid to give a value for exchangeable aluminum (23). The colorimetric method of Yuan and Fiskell was done in triplicate and the average values and standard deviations were calculated (26).

The plants for correlation with exchangeable aluminum and pH were grown in a slurry culture apparatus (Figures 3 and 4). The slurry consisted of five grams of soil, 175 ml of 0.005 N potassium nitrate and 25 ml of distilled water. The indicator plants, Harrison barley and Tennessee 5009 corn, were germinated in an incubator and transferred to the cheesecloth which was wicked into the slurry. After growing for approximately five days the plants were placed visually into damage categories at the end of each run (Figures 5 and 6). Slurry cultures with potassium nitrate were used in order to reduce nutrient effects and allow easy observation of root damage.

The pH values used for root damage correlations were the pH values of the slurries at the end of each run. The salt pH values used for correlation with exchangeable aluminum were taken from the first point of each titration curve. The water pH values used for correlation with exchangeable aluminum were taken in a slurry with a 1:1 soil-water ratio which had been equilibrated for one hour with regular stirring. The salt and water pH values of the samples used in this study are given in Table II.

Two different methods were tried for the detection of organic matter-aluminum complexes in the soils studied. In the first procedure the soils were treated with hydrogen peroxide and heated until the reaction had subsided to destroy organic matter and then titrated as

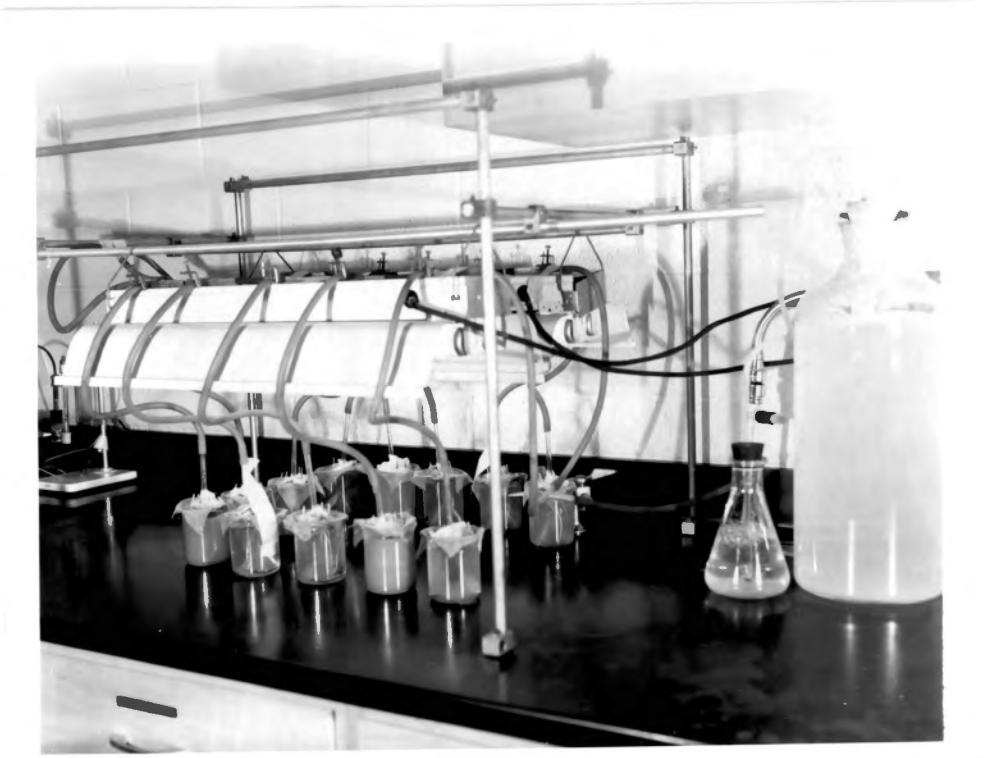


Figure 3. Slurry culture apparatus.



Figure 4. Slurry culture apparatus (close-up).

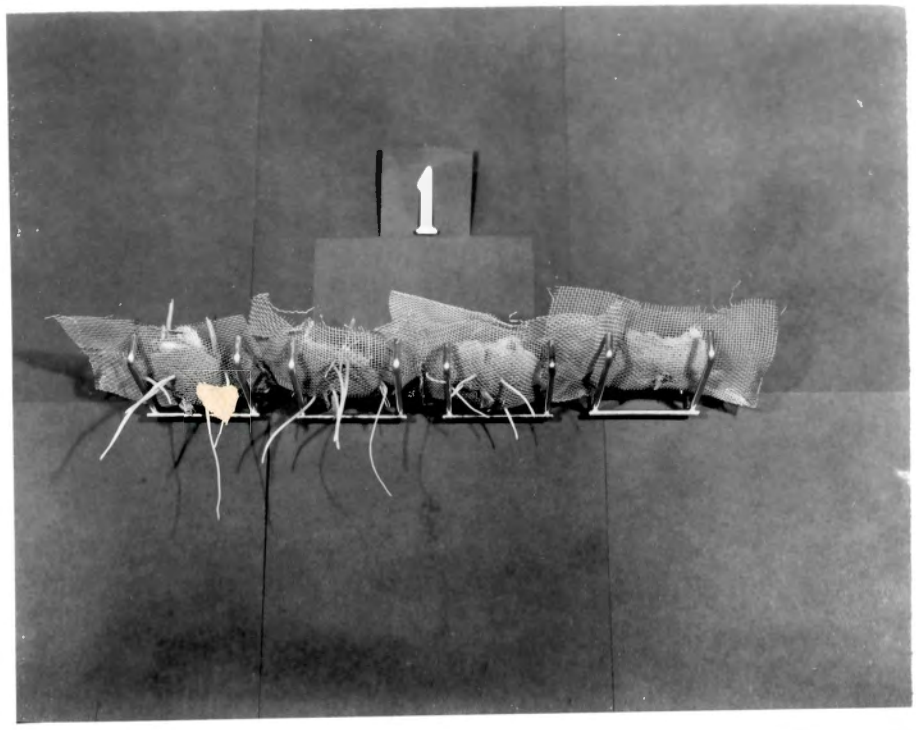


Figure 5. Aluminum damage to corn roots: none, none, intermediate, and severe.

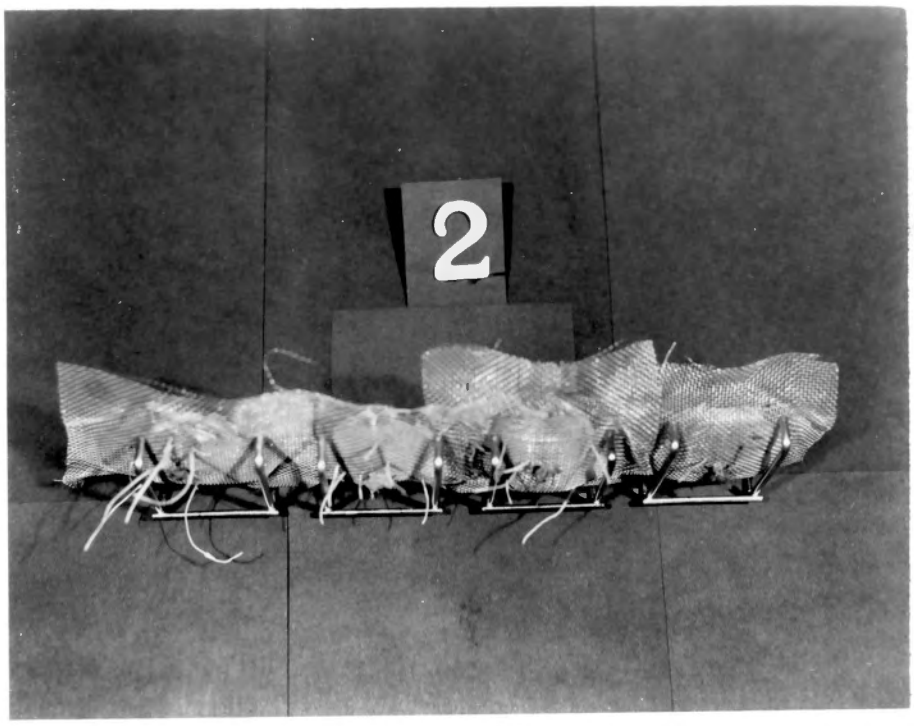


Figure 6. Aluminum damage to corn roots: none, intermediate, intermediate, and severe.

TABLE II

SALT AND WATER pH VALUES OF THE SOILS USED IN THE STUDY

Sample	1:1 Water pH	20:1 Salt pH
1	4.9	4.2
2	4.5	4.0
3	6.2	6.6
4	5.6	5.1
5	4.3	4.1
6	4.2	3.8
7	4.5	4.2
8	4.9	4.4
9	4.5	3.5
10	6.1	6.3
11	4.3	4.3
12	4.3	4.2
13	4.4	4.3
14	4.4	4.3
15	4.6	4.2
16	5.1	4.6
17	4.6	4.7
18	4.4	4.4

described earlier, to measure the amount of aluminum released.

In the other procedure samples of the soils were washed in two 50 ml portions of ion free water with ultrasonic dispersion to remove soluble salts and then washed four times in 50 ml portions of benzene with ultrasonic dispersion to remove organic matter and complexed aluminum. Samples of soils that had been washed with water only and samples that had been washed with both water and benzene were titrated as described on page 7 to measure by difference the soluble aluminum salts and organic matter removed. The water washings and the benzene washings were evaporated to dryness and analyzed for aluminum by the colorimetric method described on page 9.

CHAPTER IV

RESULTS AND DISCUSSION

In Table II, page 14, the results of three different methods for determining exchangeable aluminum are shown. In interpreting the titration curves for the whole soils, the amount of exchangeable aluminum recorded depends upon the point chosen as the break. It is not possible to determine very small amounts of aluminum using this procedure. Colorimetry is then a more appropriate method for the surface soils which were studied because it is more sensitive. Fluoride titration of the extracts is a more widely used method and data for a large number of soils are available. This method is relatively difficult to perform both because of a slow equilibration at the endpoint and because of a very gradual color change of the indicator at the endpoint. This method does not give a measure of exchangeable aluminum if the pH of the soil is higher than the endpoint of the indicator. The colorimetric method for exchangeable aluminum is very difficult and laborious. It also has a rather high standard deviation which necessitates the use of more than one sample. All three methods are adequate for the practical purpose of predicting aluminum damage to crops.

Exchangeable aluminum plotted versus hydrogen ion activity in salt yields a straight line with a correlation coefficient of 0.35 (Figure 7). The one highly deviant point on this plot is the Falaya II A2b which is the only buried surface horizon in the study. The

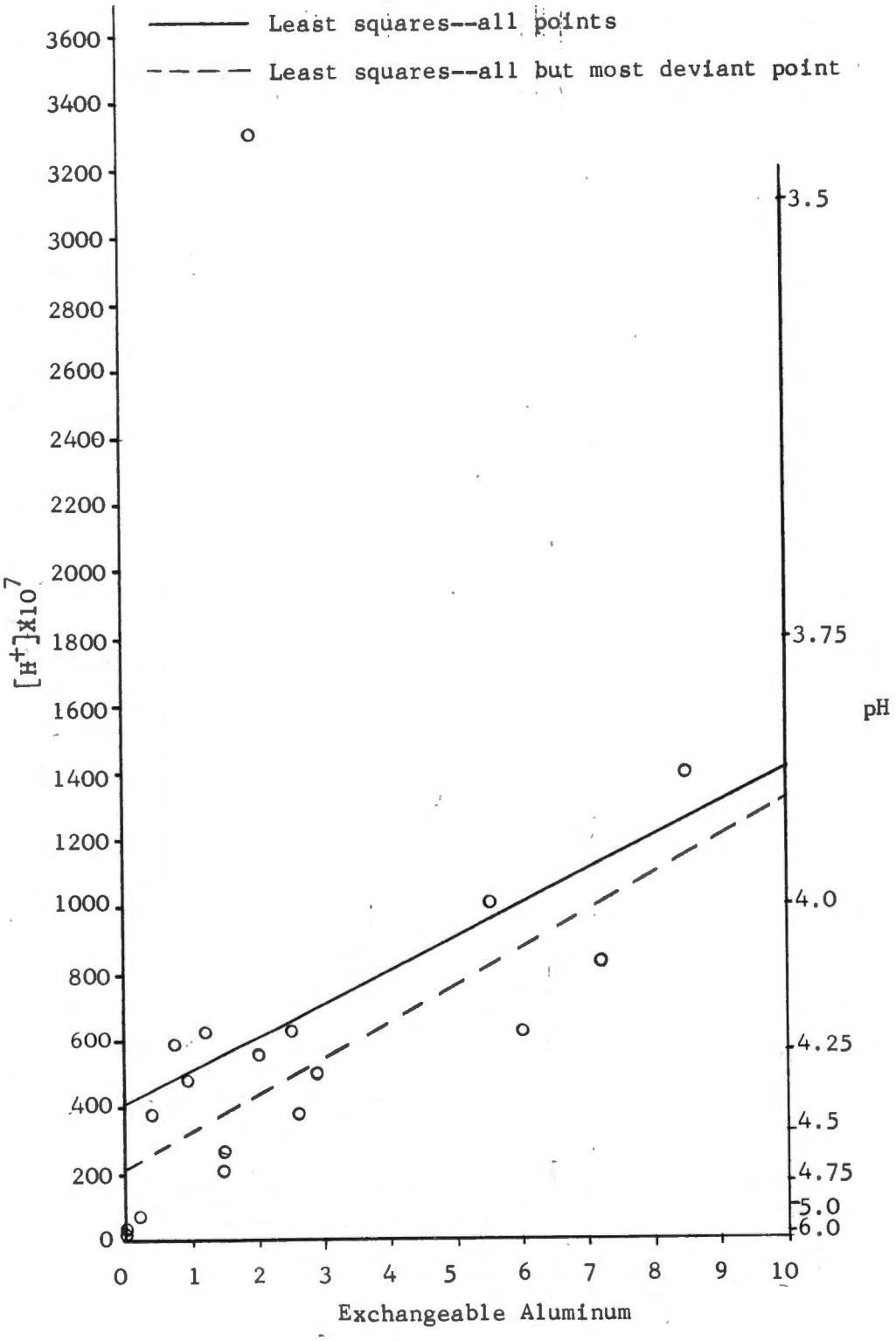


Figure 7. Exchangeable aluminum versus salt pH.

correlation coefficient excluding this point is 0.84. Exchangeable aluminum plotted versus hydrogen ion activity in water yields a straight line with a correlation coefficient of 0.63 (Figure 8). These correlations indicate that the soils used in the study were somewhat similar in cation exchange capacity and organic matter content. The pH is not a good measure of the amount of exchangeable aluminum in the soil because very large changes in exchangeable aluminum are reflected by only small changes in pH. Also, in surface horizons, the pH may be dominated by organic matter which is a nontoxic component of acidity. (Table III).

Plots of root damage as a function of exchangeable aluminum and pH are given in Figures 9 and 10. These plots indicate that for soil horizons with a pH of less than five and more than two meq/100g of exchangeable aluminum root damage from aluminum toxicity is a possibility. Tennessee 5009 corn is apparently more resistant to aluminum damage than Harrison barley as is to be expected since Harrison barley was chosen for its acid sensitivity. It is to be expected that other crops will be more or less sensitive to aluminum damage than these two. There is an indication of increasing damage with decreasing pH at the same amount of exchangeable aluminum. This would suggest an increase in aluminum activity with decreasing pH. The scatter in these graphs may be due to lack of precision in estimating root damage and to a lack of control of the activities of possibly mediating ions such as calcium. The plant roots probably respond to aluminum activity and this plot does not give a direct measure of aluminum activity. This may also add to the scatter of points. Because of the small number of soils

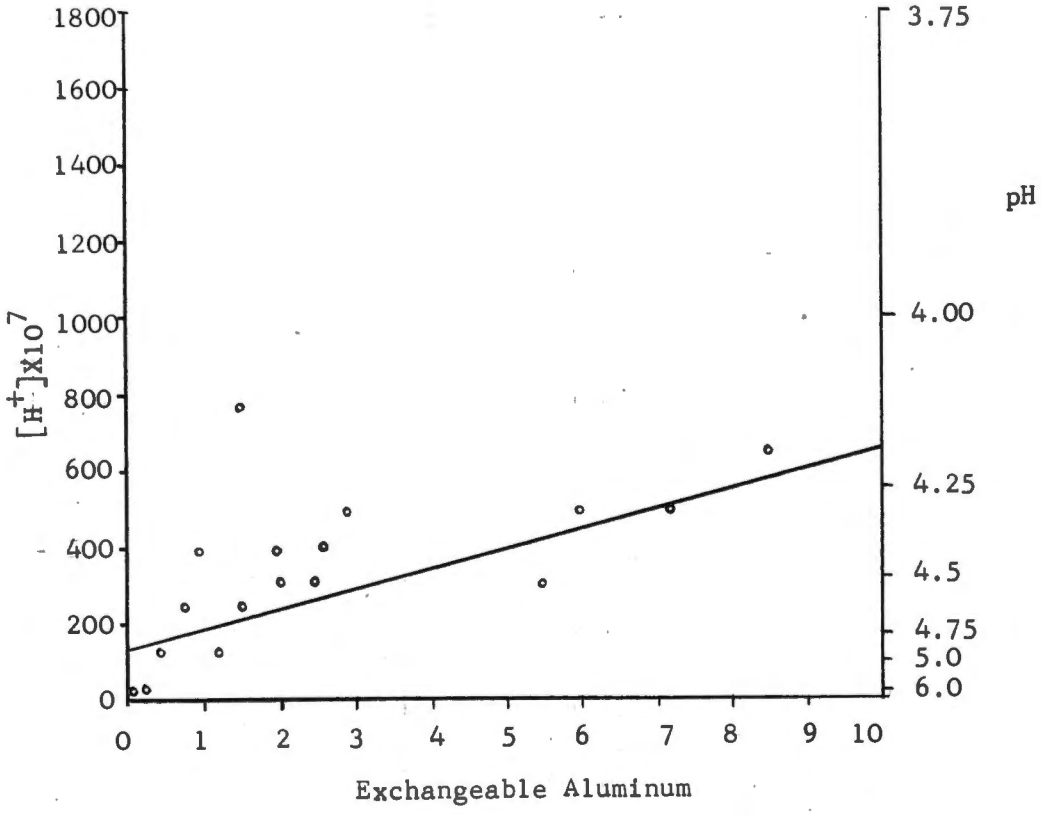


Figure 8. Exchangeable aluminum versus water pH.

TABLE III

EXCHANGEABLE ALUMINUM (meq/100g) BY METHOD

Sample	Titration of Soil	Fluoride Titration	Colorimetry ± S.D.
1	1.2	1.9	0.87 ± 0.21
2	5.5	7.0	5.6 ± 0.4
3	< 1	0	0.031 ± 0.010
4	< 1	0.51	0.19 ± 0.01
5	7.2	6.2	8.3 ± 0.4
6	8.5	9.5	8.2 ± 2.4
7	2.5	2.7	2.1 ± 0.1
8	< 1	0.40	0.24 ± 0.01
9	2.0	2.2	1.7 ± 0.2
10	< 1	0	0.032 ± 0.001
11	2.9	2.9	3.1 ± 0.1
12	6.0	3.9	5.5 ± 0.1
13	< 1	0.60	0.70 ± 0.08
14	2.0	4.2	2.3 ± 0.2
15	< 1	0.23	0.74 ± 0.02
16	1.5	2.1	1.7 ± 0.2
17	1.2	1.7	1.6 ± 0.2
18	2.0	2.6	2.6 ± 0.2

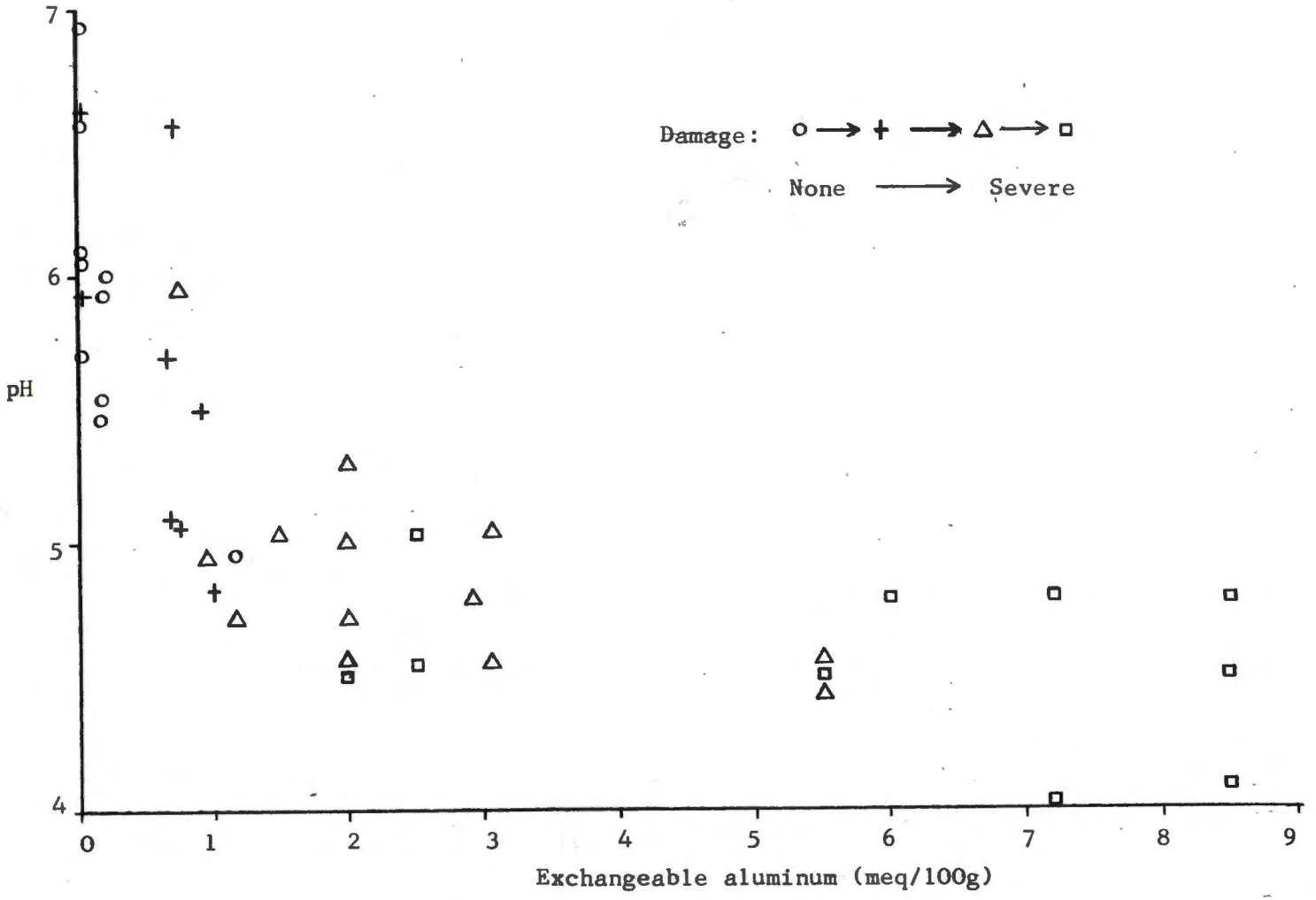


Figure 9. Root damage to Harrison Barley as a function of exchangeable aluminum and pH.

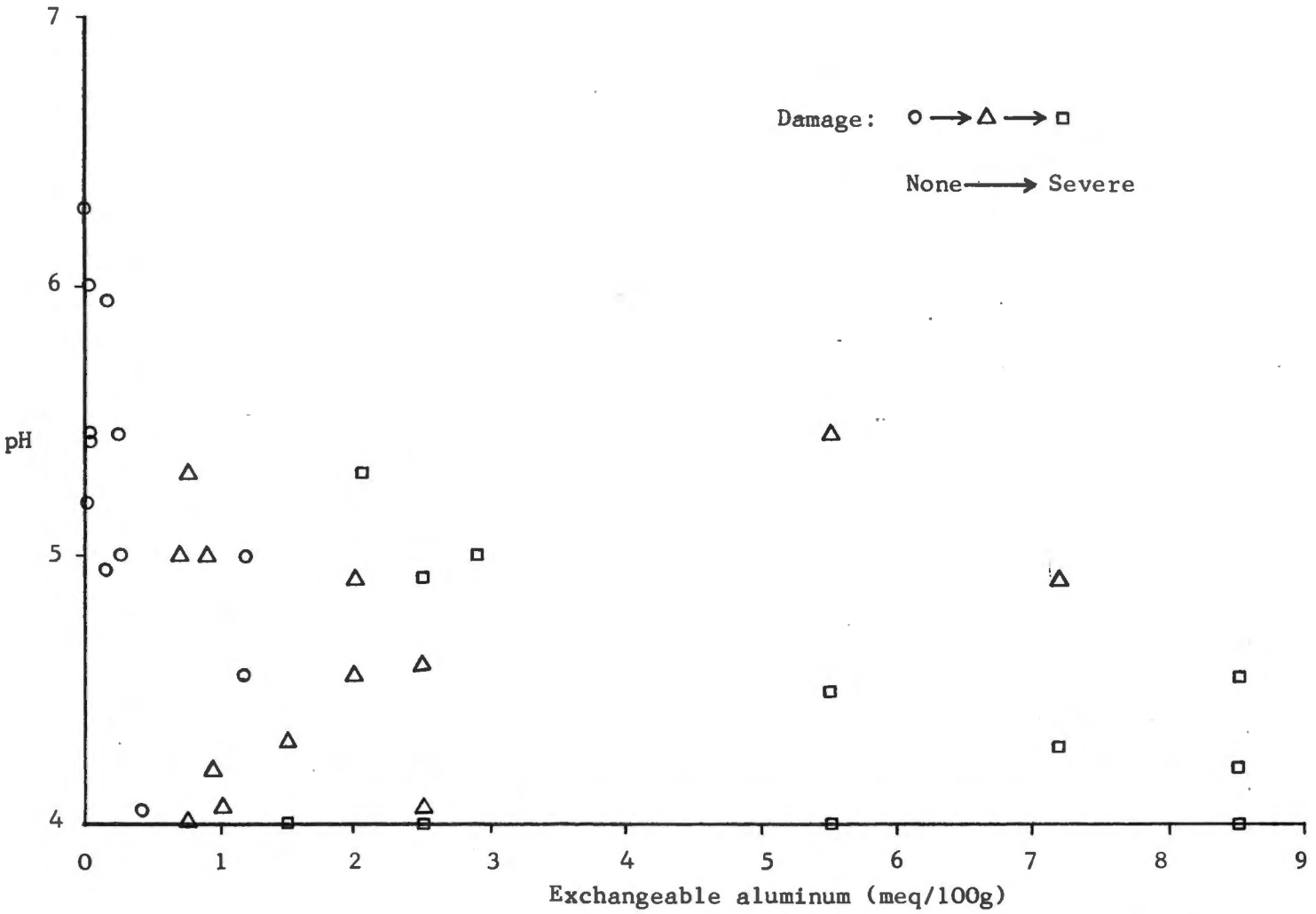


Figure 10. Root damage to Tennessee 5009 Corn as a function of exchangeable aluminum and pH.

used in this study no attempt was made to divide the graphs into surface soils and subsoils. Because of the difficulty in going from the laboratory to the field conservative figures were chosen for the pH and exchangeable aluminum values needed to produce damage and no distinction was attempted for the type of crop.

The attempts to show the existence of organic matter-aluminum complexes gave negative or perhaps conflicting results. It was expected that the titration curves of the soils which had been oxidized with hydrogen peroxide would show less organic and more exchangeable aluminum, since the oxidation of organic-aluminum complexes should release aluminum ions. The curves actually showed a decrease in both organic and exchangeable aluminum. The decrease in exchangeable aluminum may have been brought about by increased complexing with the partially oxidized organic matter or by a change in the clay minerals. The organic matter extractions were successful in extracting organic matter but were not successful in extracting any complexed aluminum. The benzene washings extracted the most organic matter but there was no measurable aluminum in the benzene after the extraction. The water washings removed up to 0.5 meq/100g of aluminum from some soils but this cannot be attributed entirely to organic-aluminum complexes as it may well reflect the presence of soluble salts or hydrolysis of aluminum from the exchanger.

CHAPTER V

CONCLUSIONS

In conclusion, it was found that all three methods for determining exchangeable aluminum were satisfactory for predicting root damage to crops. The relationship between pH and exchangeable aluminum was not judged good enough for prediction purposes. Aluminum damage appears likely at pH values less than five and with more than two meq/100g exchangeable aluminum. The existance of organic matter-aluminum complexes in the soils studied was not shown.

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APPENDIX

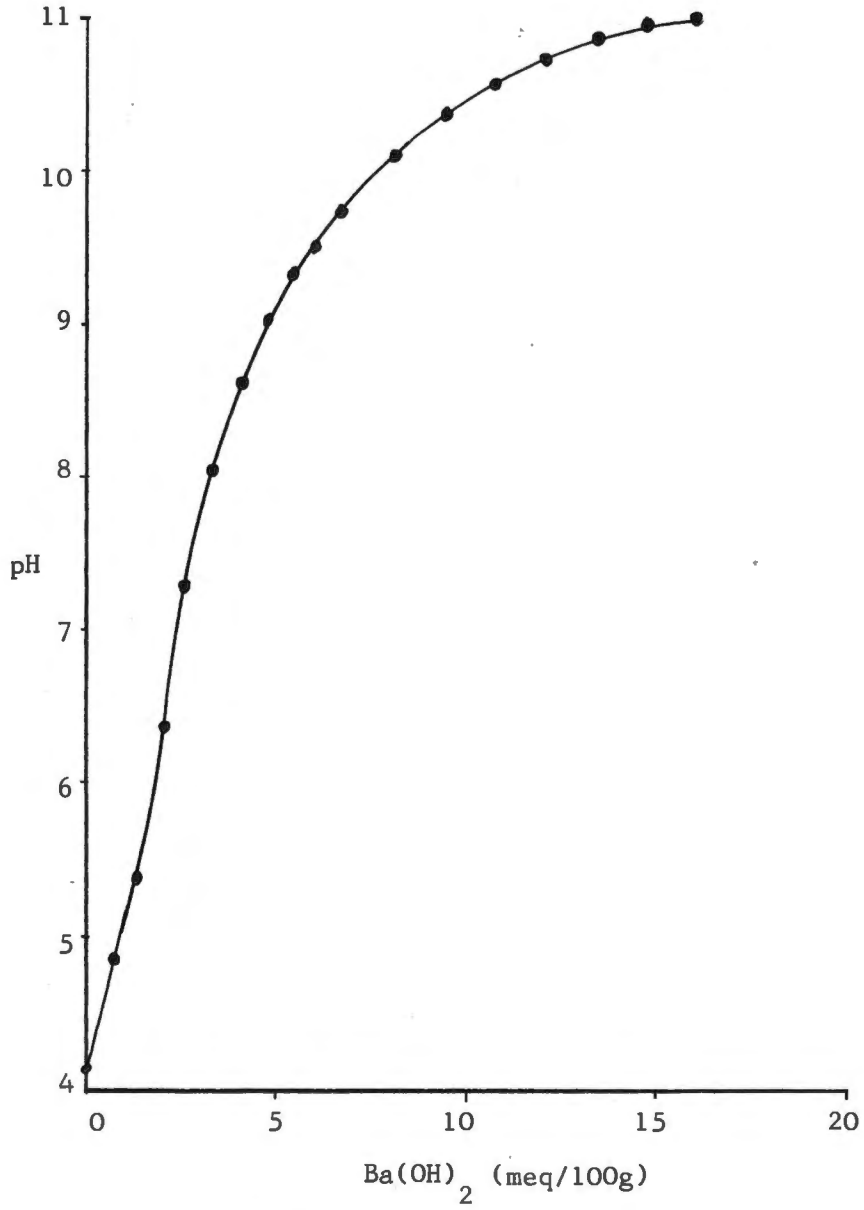


Figure 11. Potentiometric titration of Muskingum A.

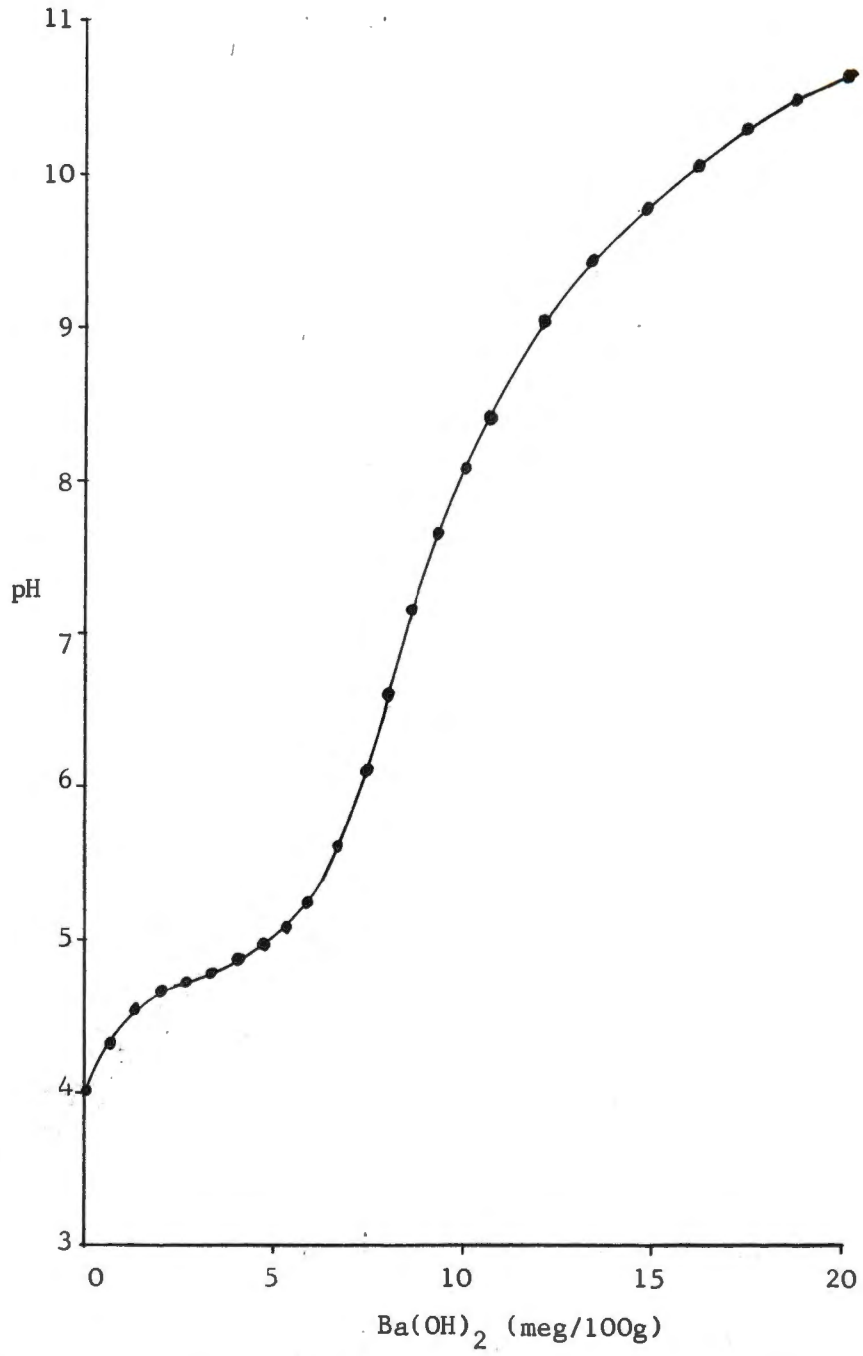


Figure 12. Potentiometric titration of Muskingum B.

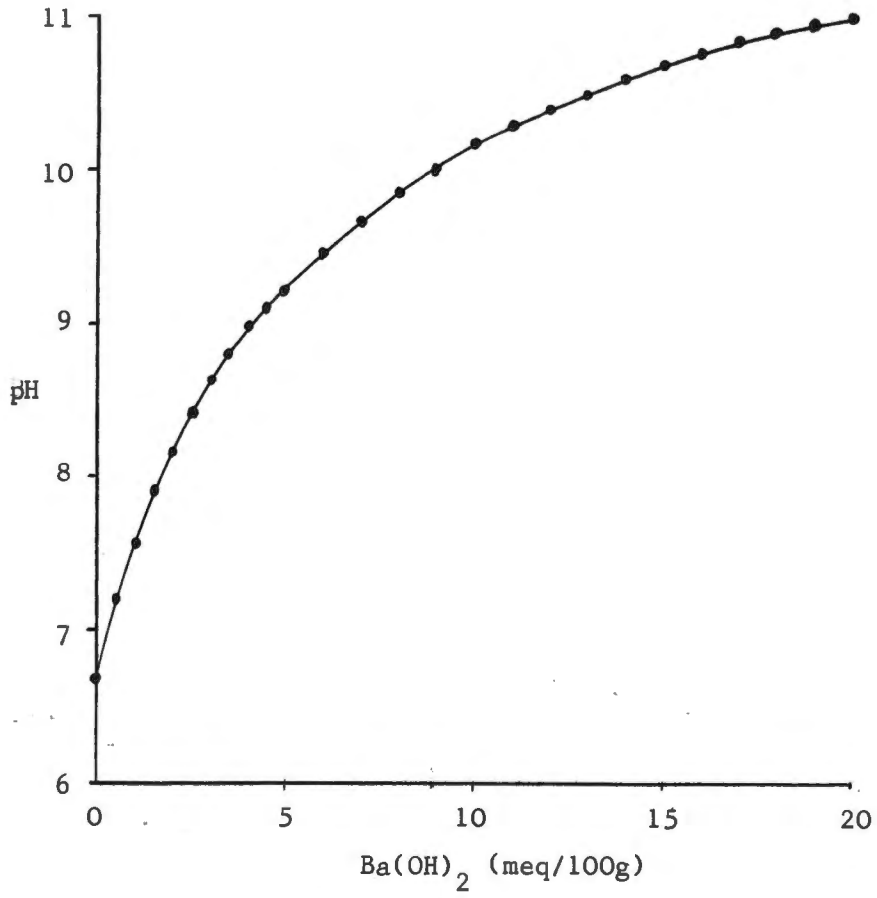


Figure 13. Potentiometric titration of Routon Ap.

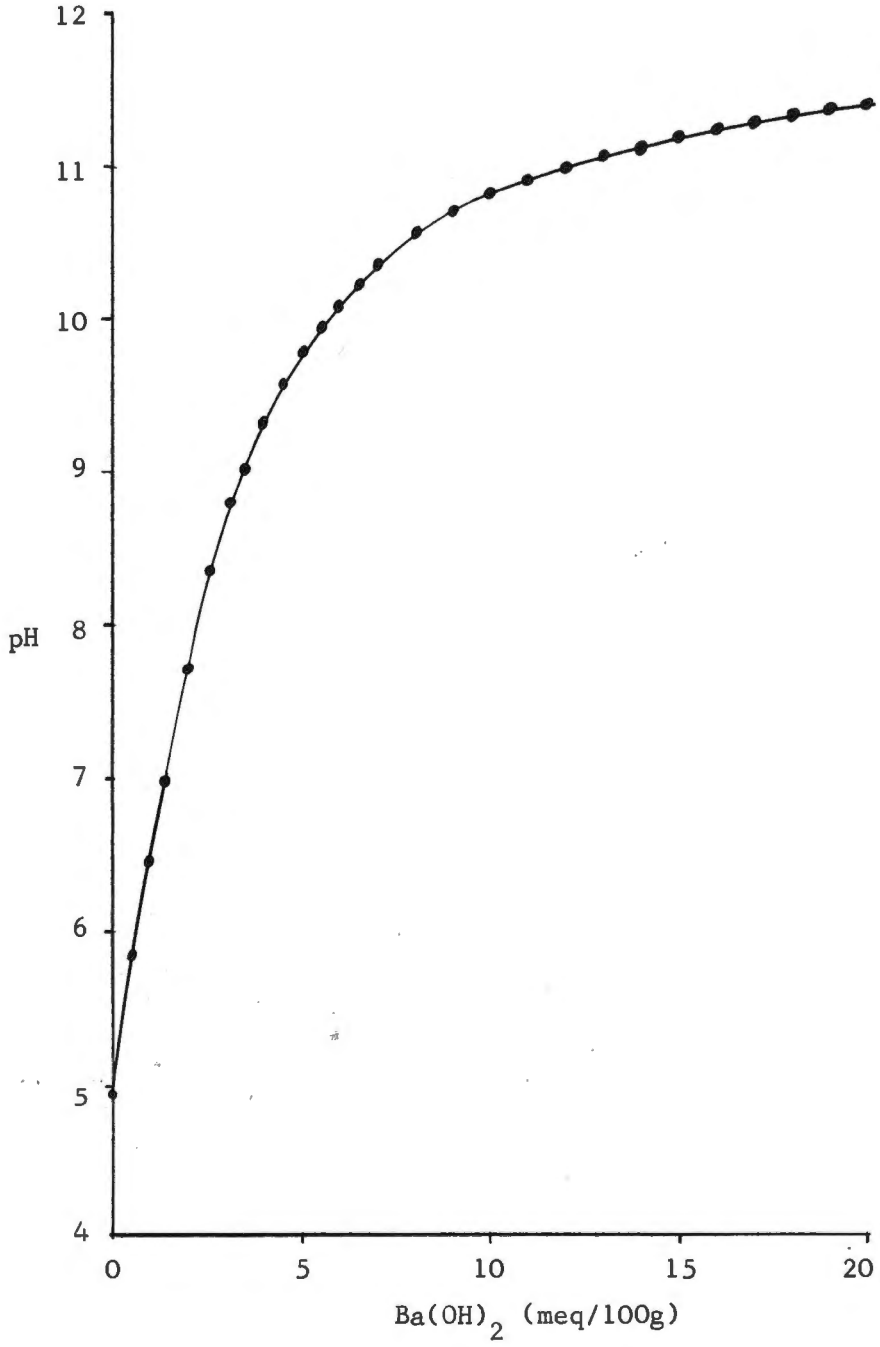


Figure 14. Potentiometric titration of Routon A3g.

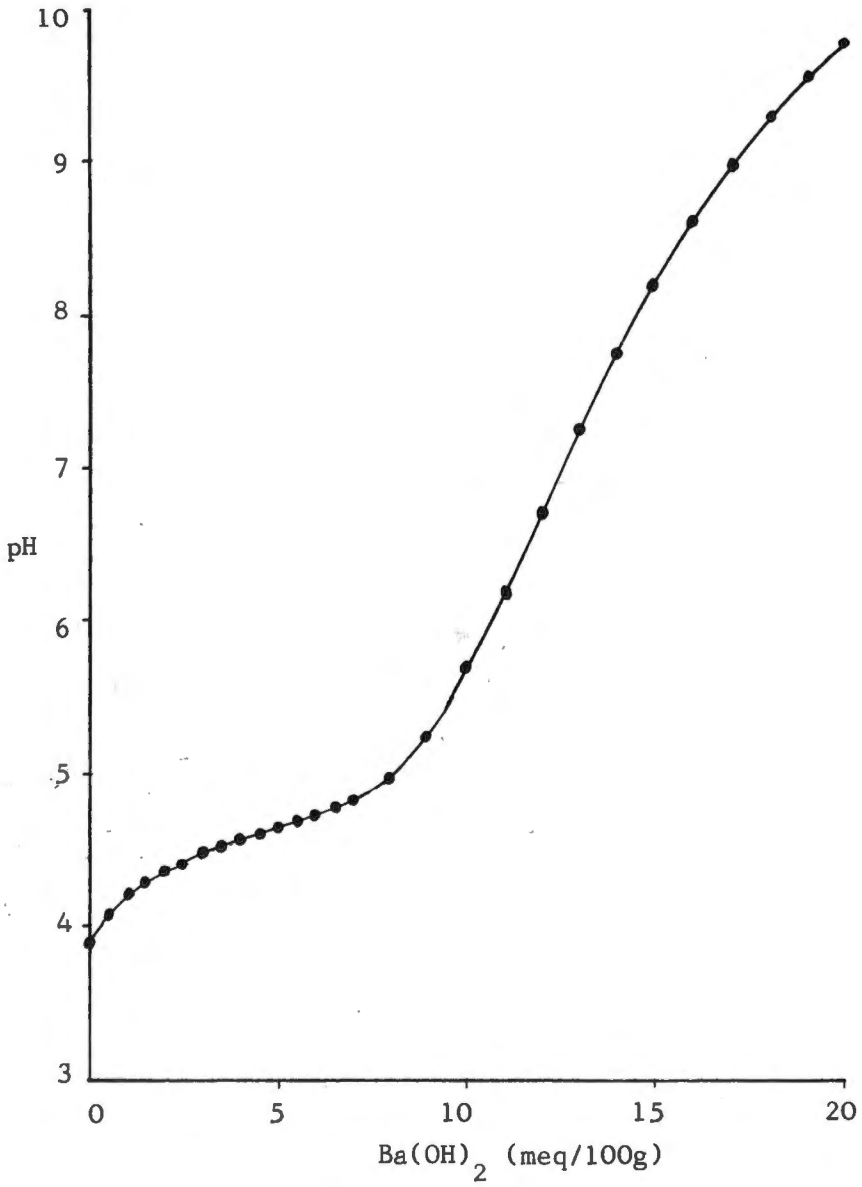


Figure 15. Potentiometric titration of Routon B2g.

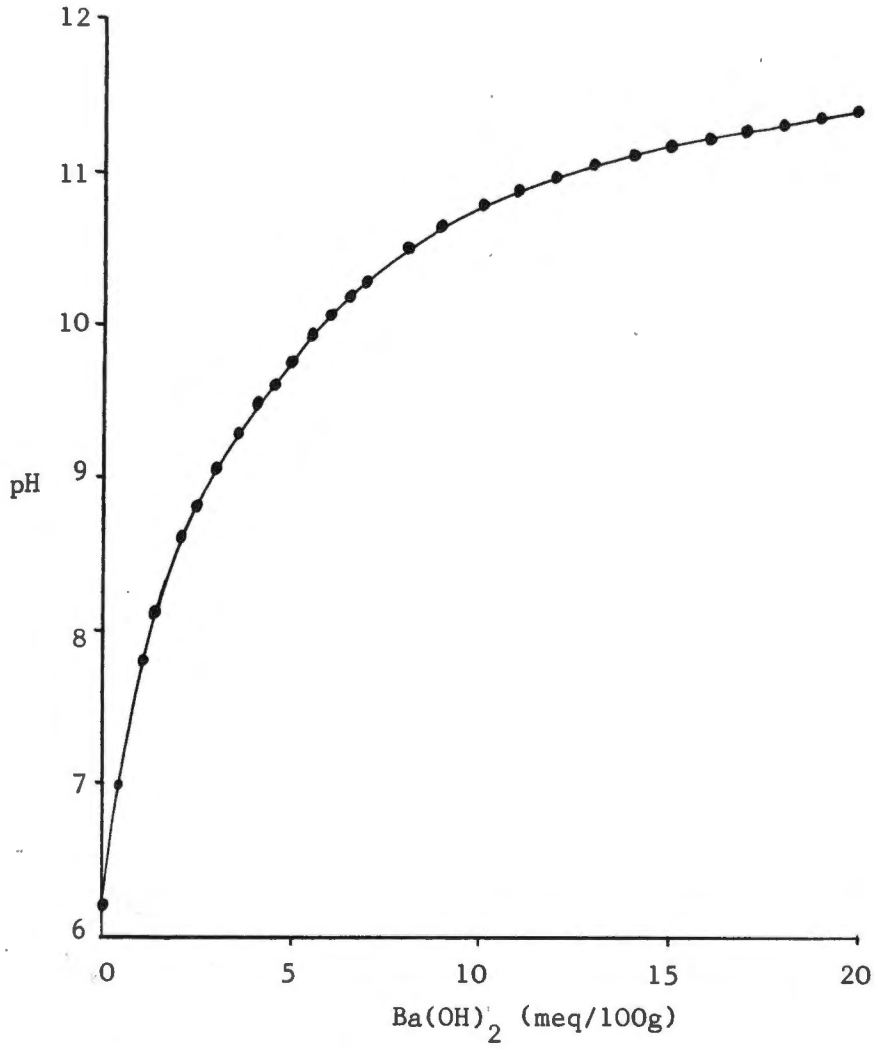


Figure 16. Potentiometric titration of Center Ap.

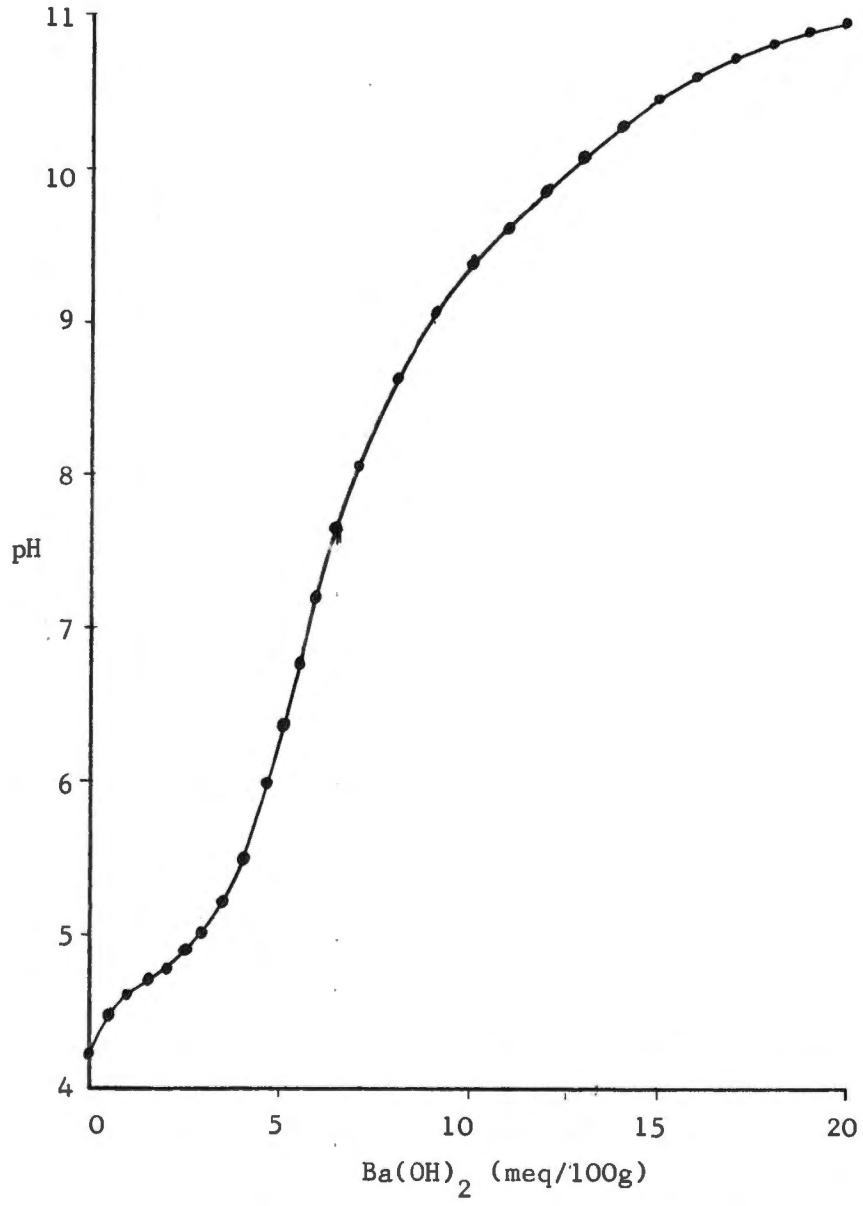


Figure 17. Potentiometric titration of Center Blg.

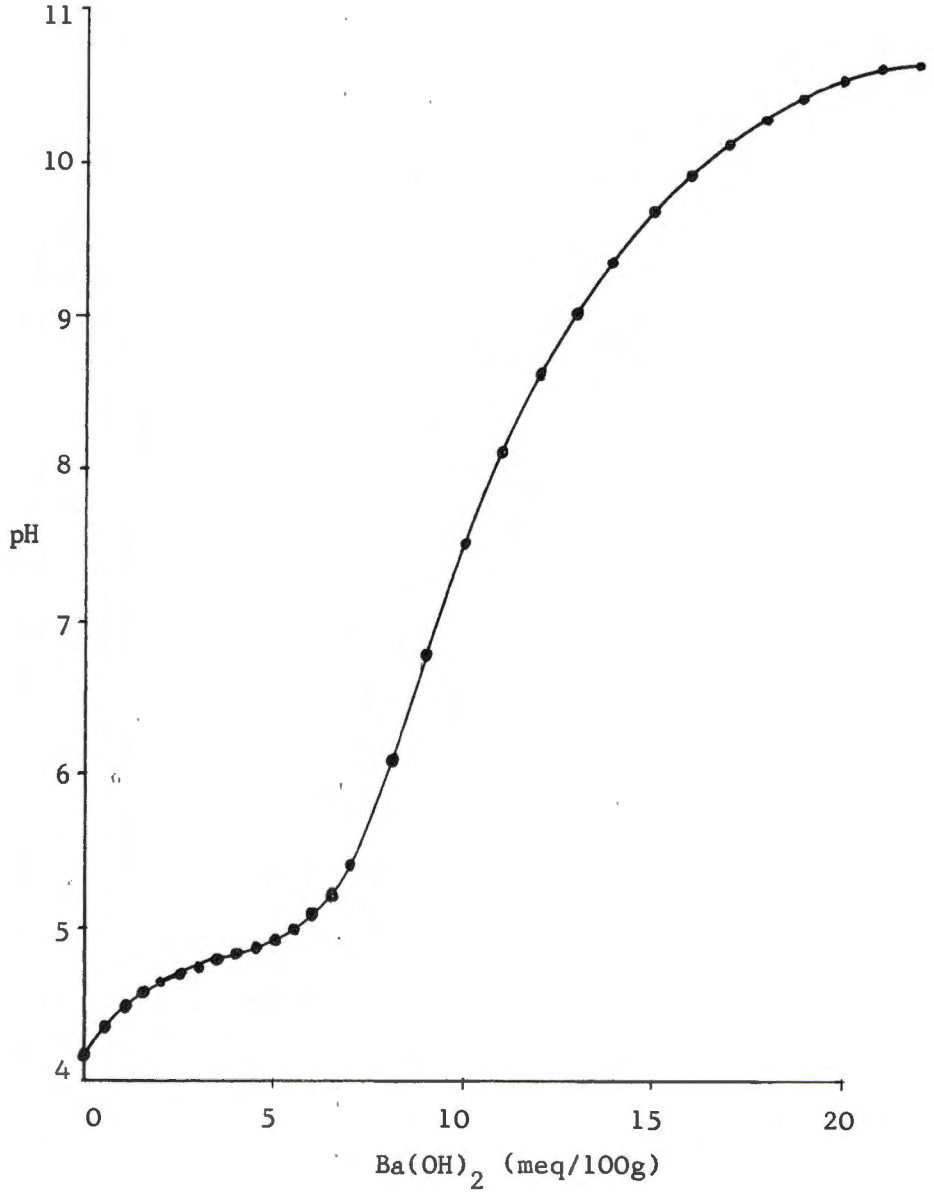


Figure 18. Potentiometric titration of Center B2g.

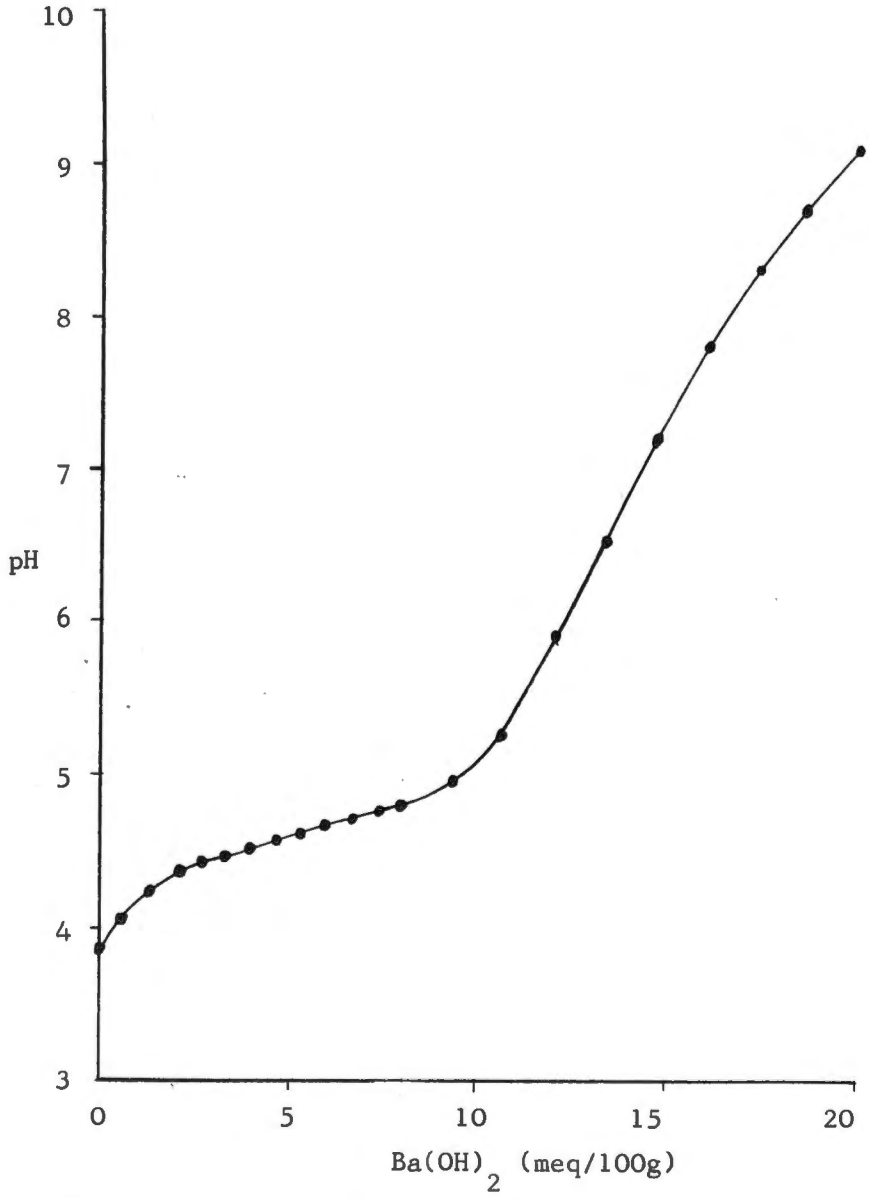


Figure 19. Potentiometric titration of Sequoia B.

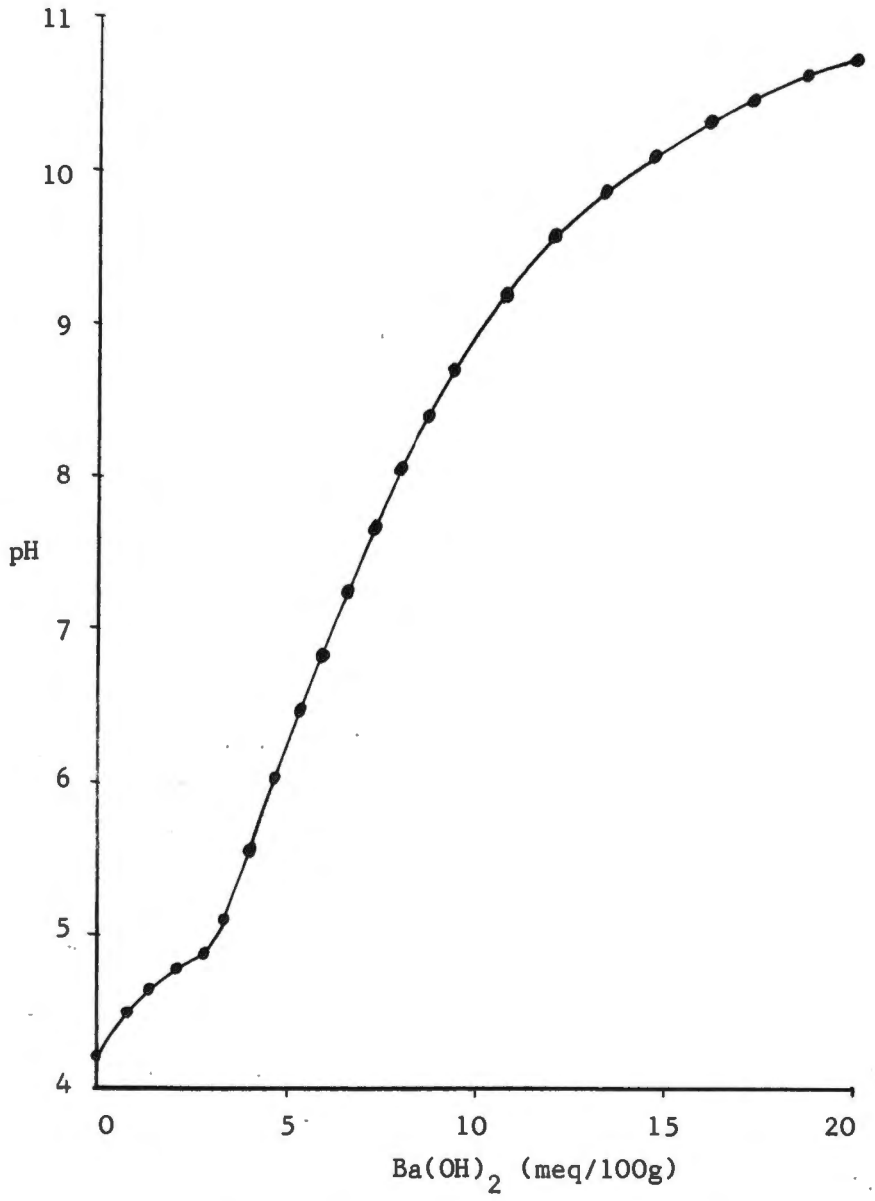


Figure 20. Potentiometric titration of Tellico B.

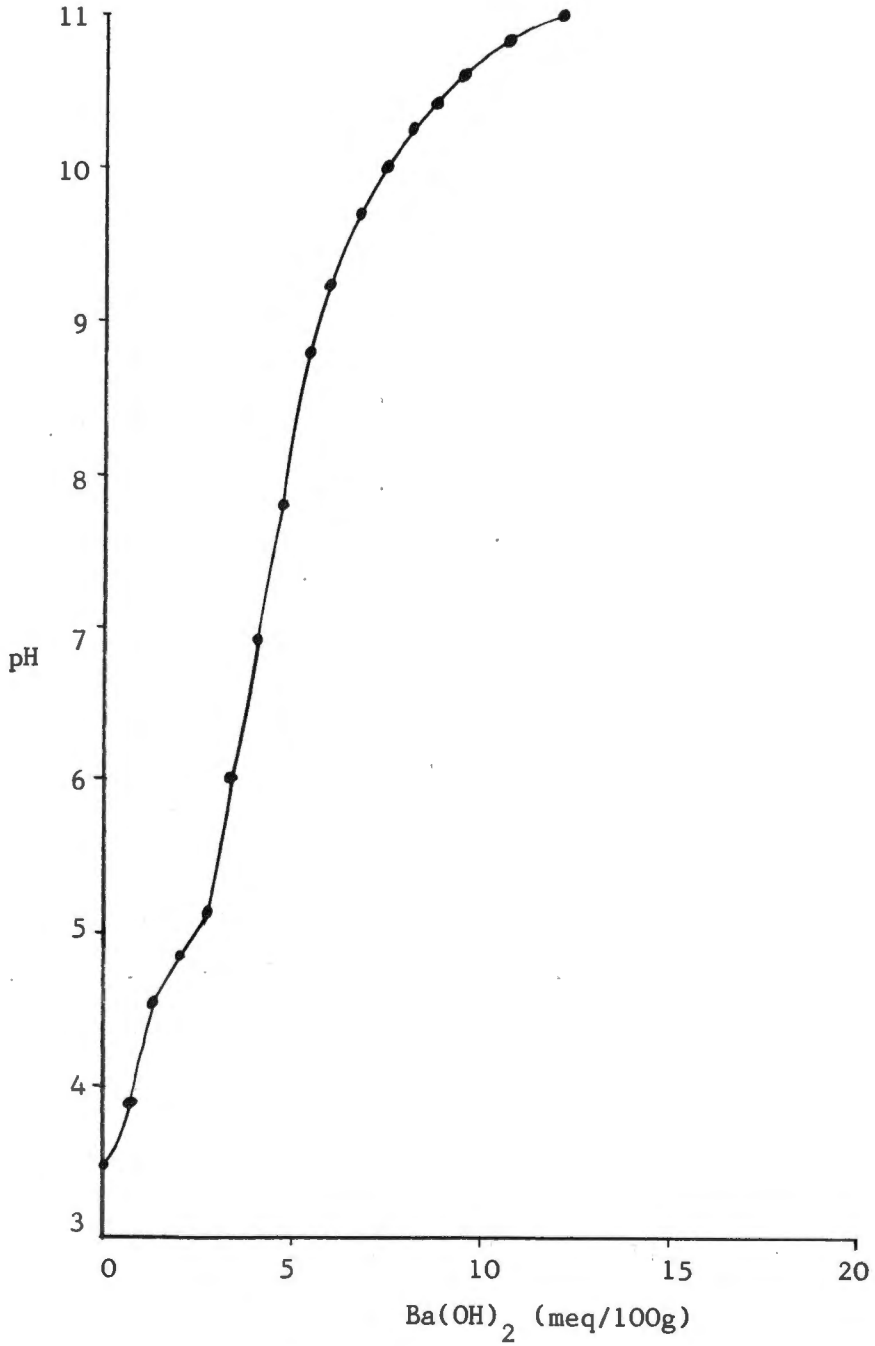


Figure 21. Potentiometric titration of Falaya IIA2b.

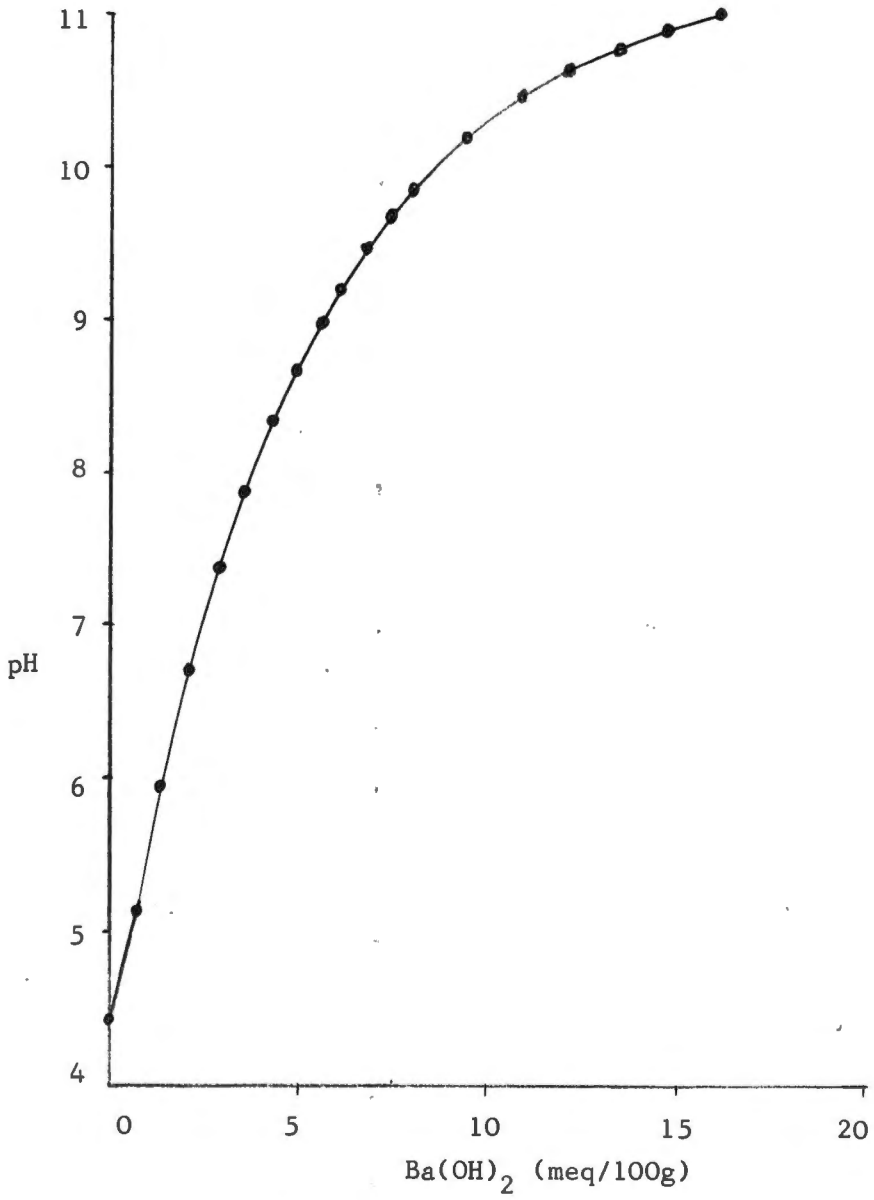


Figure 22. Potentiometric titration of Falaya Ap.

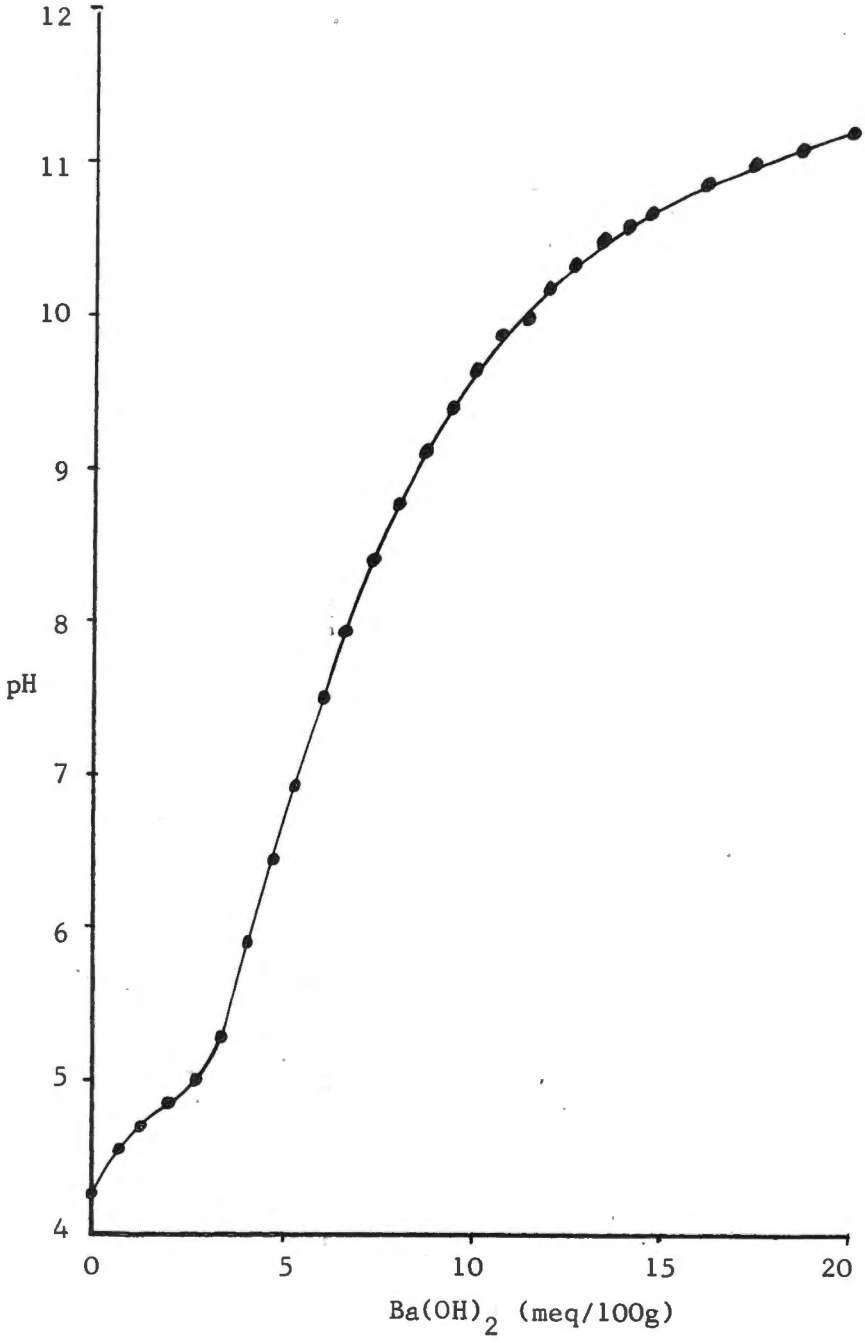


Figure 23. Potentiometric titration of Etowah AP (aluminum saturated).

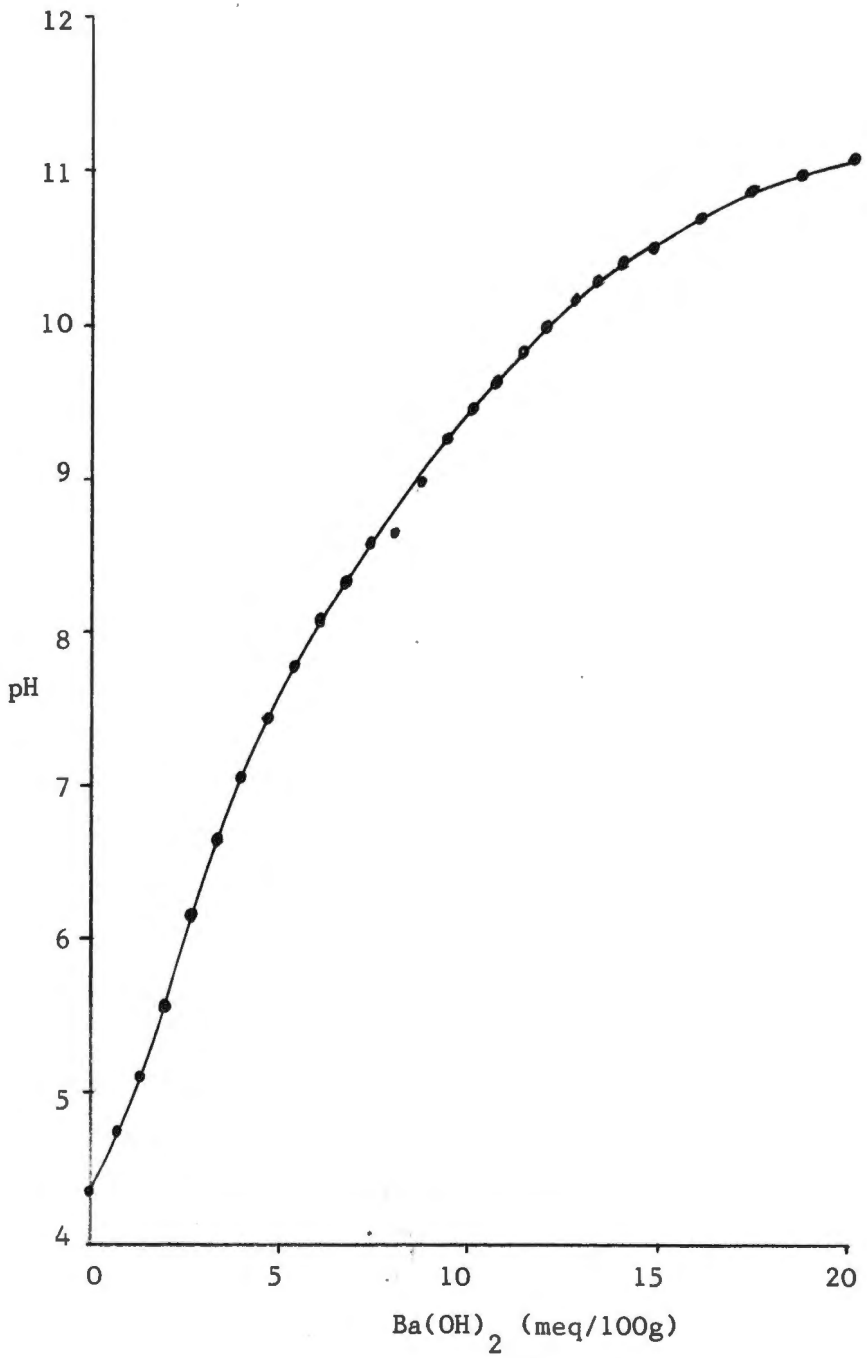


Figure 24. Potentiometric titration of Fullerton Ap.

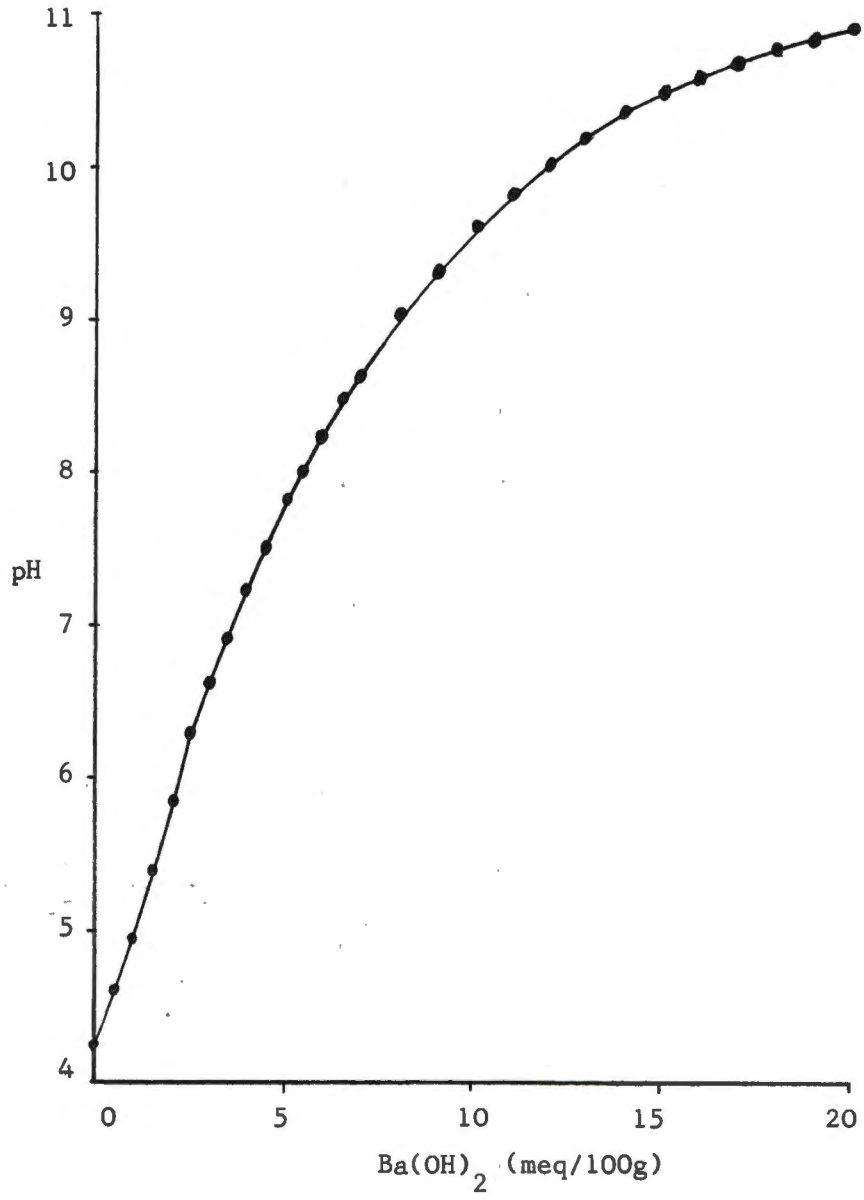


Figure 25. Potentiometric titration of Etowah Ap.

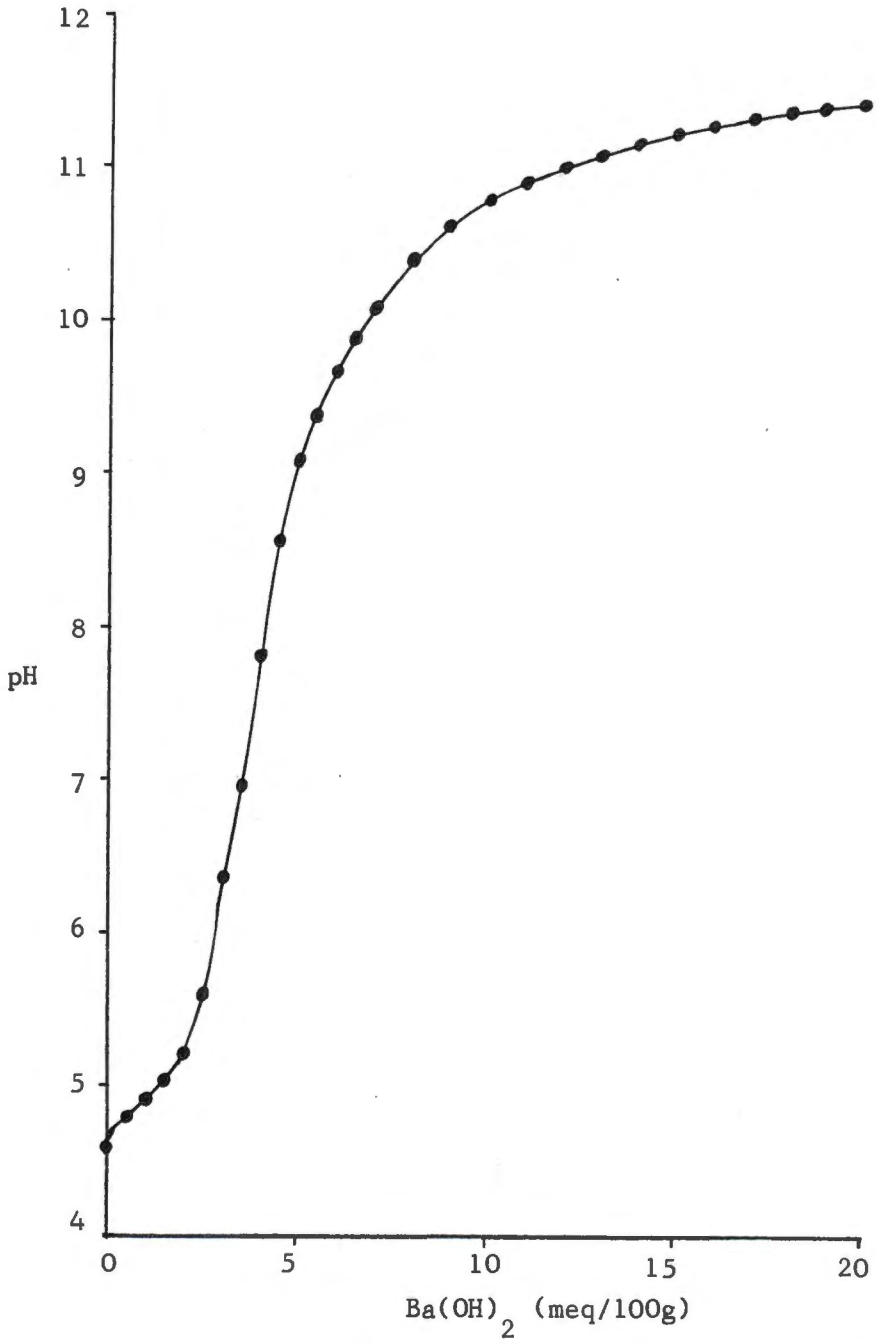


Figure 26. Potentiometric titration of Clarksville Cl (Roane County).

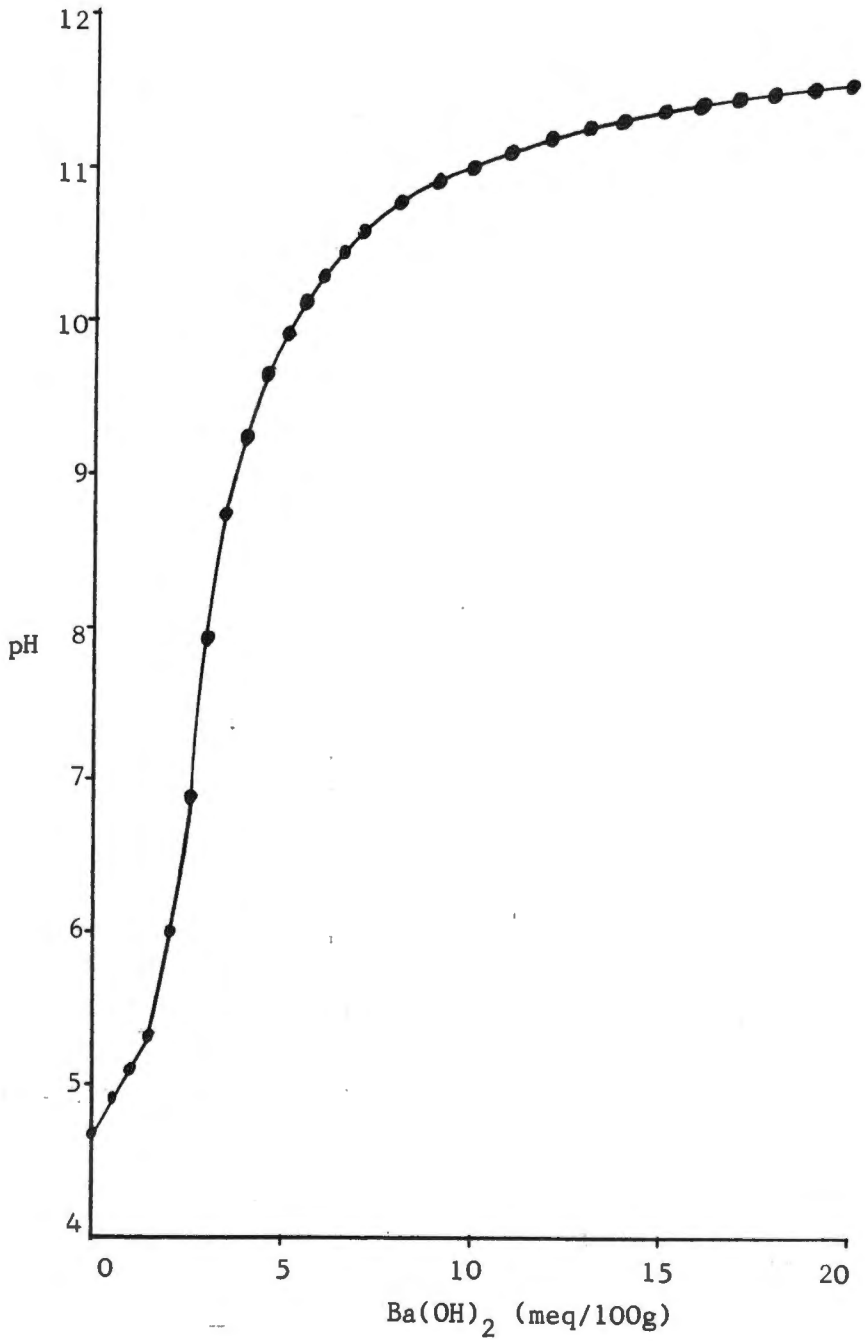


Figure 27. Potentiometric titration of Clarksville Cl (Meigs County).

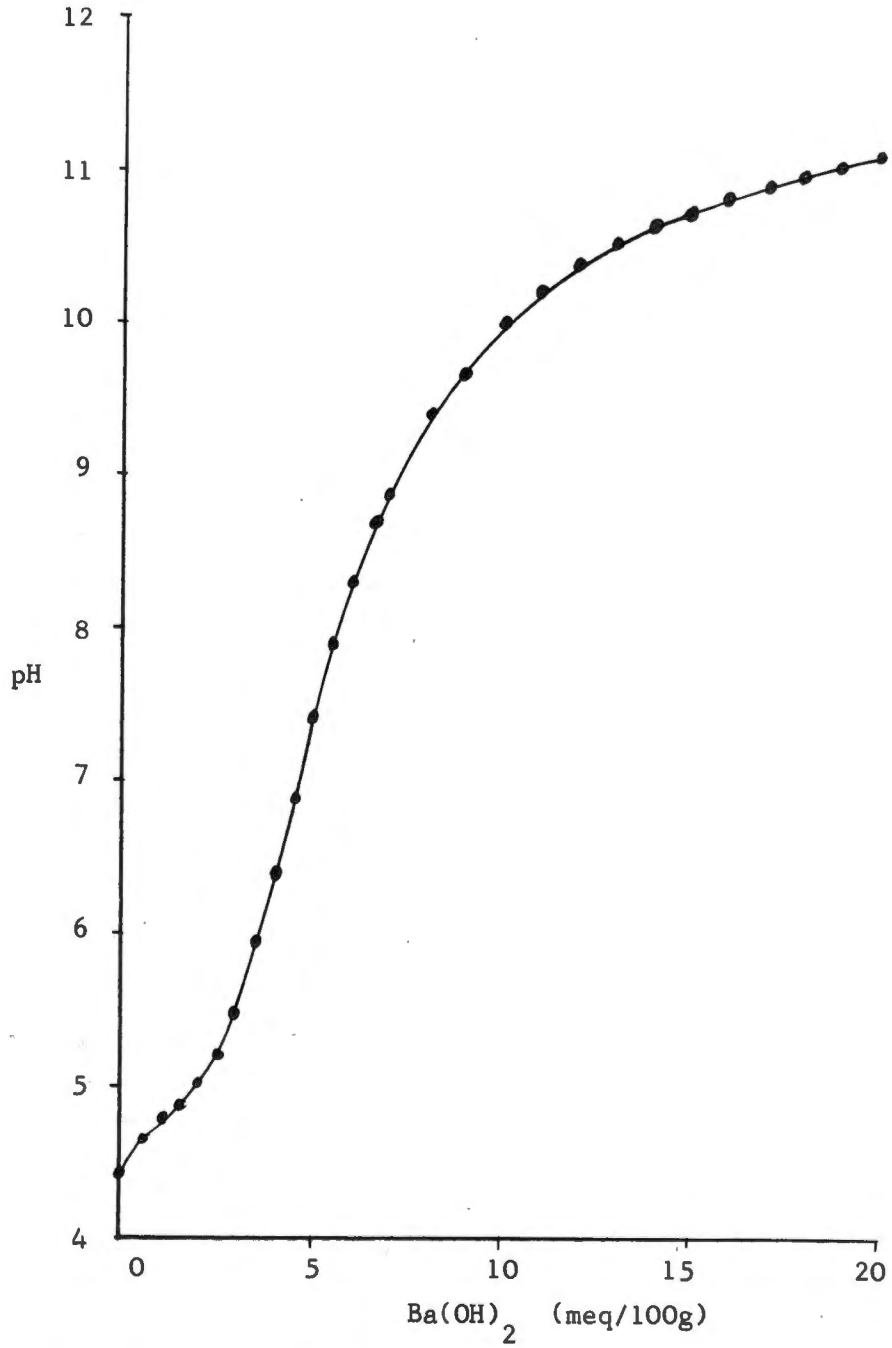


Figure 28. Potentiometric titration of Hartsells C1.

VITA

John Walter Odom was born in Knoxville, Tennessee, on February 24, 1948. He attended elementary school in Henry County and was graduated from E. W. Grove High School of that county in June of 1966. He entered The University of Tennessee at Knoxville in the fall of that year and received a Bachelor of Science degree in Agriculture with a major in Agronomy in August of 1970.

He accepted a National Science Foundation traineeship in the fall of 1970 and began work towards a Master's degree in Agronomy. He received this degree in June of 1972.

He is married to the former Sharon Ann Merica of Alexandria, Virginia.