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Seasonal Variability of pH and Lime Requirements in Several Southern Michigan Soils When Measured in Different Ways[1]

J. B. COLLINS, E. P. WHITESIDE AND C. E. CRESS[2]

Prairie View

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Seasonal Variability of pH and Lime Requirements in Several Southern Michigan Soils When Measured in Different Ways[1]

J. B. COLLINS, E. P. WHITESIDE AND C. E. CRESS[2]

ABSTRACT

The seasonal variability of pH and lime requirement of the plow layer of 13 soil series, at 19 sites, by various methods on field-moist, air-dry and oven-dry samples were observed from May through September 1966 near East Lansing, Mich. Several factors associated with variation in pH were also observed.

At the beginning of the season, the mean glass electrode-pH of air-dry and oven-dry samples measured in each of three suspending media—water, 1.0*N* KCl, and 0.01*M* $CaCl_2$—were each significantly lower than the corresponding field-moist pH. On air-dry samples, pH measurements with the glass electrode in water are significantly correlated with those in 0.01*M* $CaCl_2$ ($r = 0.96$) and in 1.0*N* KCl ($r = 0.92$) or those measured with the Truog kit ($r = 0.90$).

The pH of field-moist samples in water showed a maximum seasonal variability of 1.6 pH units and an average variation of 0.8 pH unit. The pH of samples in 1.0*N* KCl showed the least average seasonal variability of the methods used, 0.2 pH units. Electrical conductivity gave the highest correlation ($r = -.82$) with seasonal variability of pH on air-dry samples in water. The glass electrode pH on air-dry samples in 1.0*N* KCl were better correlated ($r = .63$) with percentage base saturation than those in water ($r = .53$) and slightly better than those in 0.01*M* $CaCl_2$ ($r = .61$).

Four of 11 sites studied had a lime requirement early in the season but each of the 11 sites had a lime requirement in mid-summer.

Additional Key Words for Indexing: pH and moisture conditions; pH and suspending media; errors in pH measurements.

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PERHAPS the most important chemical test of a soil material as a medium for plant growth is its pH value. The solubility and availability of many nutrients are closely related to soil pH.

This study was conducted from May through September 1966, to investigate the seasonal variability of the pH of the plow layer of 13 soil series at 19 sites in southern Michigan by various procedures; to observe several of the possible factors influencing the seasonal variations; and to evaluate the errors in the determinations of soil pH. In addition, the lime requirement was determined by several methods and the results compared.

LITERATURE REVIEW

It has frequently been observed (2, 8, 17, 21) that pH values of soil samples taken from the same site at different times during the year show considerable variation. Seasonal variations in soil reaction are of much practical importance where this chemical test is used to determine fertilizer (particularly micronutrient) and lime requirements of soils. Soil reaction is also one of the criteria used in the new soil classification system (7th Approximation) to classify certain soils at the family level, and it has commonly been used as a criterion for differentiation among soil series.

Several investigators (2, 21) have reported a decrease in soil pH during the summer months. In some instances, a variation during the year of as much as 1.0 pH unit has been reported. Baver (2) attributed the decrease in soil pH to the accumulation of soluble salts. Others (17, 19) have reported findings similar to those of Baver. Schofield et al. (19) indicated that soil pH measured in 0.01 *M* $CaCl_2$ should be independent of the initial amount of salts present in nonsaline soils. Many European workers have also attempted to minimize seasonal variations in soil pH by measuring pH in 1*N* KCl (17).

Huberty (8) obtained lower pH with oven-dry samples than with air-dry samples and observed that the oven-dry pH was no more variable than the air-dry pH. Rost et al. (18)

found that all but one of 144 soils became more acid upon air drying, and concluded that the only reliable indication of conditions existing in the field is obtained when pH is determined on field-moist samples. Van Der Paauw (21) observed that soil pH gradually increases in periods of high rainfall and decreases in periods of low rainfall.

Seasonal variability of soil pH has been observed to be associated with soil organic matter. Arrhenius (1) found little change with a humus rich soil but a change of 0.9 pH unit with a soil low in organic matter.

The relationship between differences in soil pH and base saturation has been the subject of many investigations (10, 12). Morgan (12) pointed out that the relationship between pH and base saturation may be fairly constant within a soil type, but that it may vary widely between soil types. Mehlich (10) reported that the pH and base saturation relationship is almost solely influenced by the nature of the exchange complex.

The reproducibility of soil pH values with the glass electrode has been the subject of several investigations (3, 4, 15, 17, 19). Coleman et al. (4) concluded from their study that the measured emf, which is interpreted in terms of pH, includes the activity of hydrogen ions and a junction potential. Peech et al. (15) pointed out that the error due to the junction potential should not exceed 0.25 pH unit. They and Schofield et al. (19) indicated that the error due to the junction potential could be minimized by measuring pH in a flocculated soil suspension. Clark (3) pointed out that the error due to the junction potential is essentially eliminated by insuring that the ionic strength of the salts in solution is greater than 0.005 *M*.

There has been little agreement among investigators (1, 5, 8, 11, 16) as to the proper ratio of soil to water that should be used for pH measurements. McGeorge (11) found that the increase in pH is most rapid for soil-water ratios below 1:8. Pierre (16) observed that the hydrogen ion activities of some soils were not affected by changing the soil-water ratio from 1:2 to 1:50. Gillespie and Hurst (5) found no consistent differences in the pH of soils using soil-water ratios of 1:1 and 1:2. Their results varied from a -0.15 to a $+0.25$ of a pH unit, based on 1:1 as a standard.

In 1930 the soil reaction committee of the International Society of Soil Science (7) adopted a 1:2.5 soil-water ratio as the standard. The procedures used in this study for pH in water (1:1) and in 0.01*M* $CaCl_2$ (1:2) are those recommended jointly by the American Society of Agronomy and the American Society of Testing and Materials in Agronomy no. 9, *Methods of Soil Analysis, Part 2*, 1965.

EXPERIMENTAL PROCEDURE AND SOIL SITES

Nineteen sites representing 13 soil series were used in this study. All were mineral soils except Houghton which was organic. The surface texture and natural drainage of the mineral soils varied from clay loam to sand and from well to poorly drained, as shown in Table 1. The sites selected were not close to gravel roads, dead furrows, lime or manure piles, or burned muck areas. Also, the sites were uniform with respect to topography, texture, natural drainage, vegetation or crop, color of surface, and pH. The initial pH of air-dry samples measured in water varied from 5.2 to 8.0.

Uniformity of the pH at each site was ascertained by the variability of pH among six individual cores and a composite sample which consisted of 20 cores. Each of the six individual cores, representing .16 of the area of each plot, was 7.62 cm in diameter and extended to a depth of from 15.24 cm–20.32 cm (the plow depth). The 20 cores of the composite sample were 2.54 cm in diameter and extended to a depth of from 15.24 cm–20.32 cm. These 20 cores were collected as per instructions in Extension Bulletin E-498 of Michigan State University.

After the sites were located and established, one composite sample was collected from each once a montn. Ten of the sites were sampled from May through September; the other sites were sampled from June through September. A subsample from the composite sample of each site was placed in a plastic bag and brought into the laboratory. Part of each subsample was refrigerated at 5C, until the field-moist pH could be determined. The remainder of the subsample was placed on a laboratory bench and covered with wrapping paper while air drying. Determinations of pH on the air-dry samples were made after 3 to 4 weeks of drying. The pH of the field-moist samples was determined from 1–3 days after collection of the samples. A portion of the field-moist sample from each site was dried in an oven for 24 hr at 38–43C, and the pH of these oven-dried samples was also determined.

Table 1—Texture, slope and erosion class, natural drainage, and variations in the pH of field moist core samples collected in the initial sampling of each site

Soil site	Site no.	Texture (surface)	Slope and erosion	Natural drainage	Means and determination standard deviations (S_D)	
Blount	1	Clay loam	A1	Somewhat poorly	7.09	0.134
Ceresco no. 1	2	Sandy loam	A1	Somewhat poorly	7.00	0.122
Ceresco no. 2	3	Sandy loam	A1	Somewhat poorly	7.84	0.155
Colwood no. 1	4	Loam	A1	Poorly	8.11	0.155
Colwood no. 2	5	Loam	A1	Poorly	7.76	0.144
Chelsea	6	Sand	B1	Well	5.68	0.125
Hillsdale no. 1	7	Sandy loam	B1	Well	6.65	0.122
Hillsdale no. 2	8	Sandy loam	B1	Well	6.22	0.122
Houghton	9	Muck	A1	Poorly	7.01	0.099
Lapeer	10	Sandy loam	A1	Well	7.35	0.096
Nekoosa no. 1	11	Sand	A1	Somewhat poorly	6.91	0.144
Nekoosa no. 2	12	Sand	B1	Somewhat poorly	7.72	0.101
Oakville	13	Sand	B1	Well	6.31	0.122
Pewamo no. 1	14	Clay loam	A1	Poorly	7.50	0.119
Pewamo no. 2	15	Clay loam	A1	Poorly	7.50	0.119
Plainfield	16	Sand	B2	Well	6.14	0.169
Spinks no. 1	17	Loamy sand	B1	Well	6.60	0.127
Spinks no. 2	18	Loamy sand	B1	Well	6.70	0.131
St. Clair	19	Clay loam	B1	Well	7.54	0.110
					Combined S_D	0.139

METHODS

All samples were crushed and sieved through a 2-mm sieve prior to analysis, and all determinations were run in duplicate, except where otherwise indicated.

The pH was measured on the field-moist, air-dry, and oven-dry samples with the glass electrode. Using 0.01*M* $CaCl_2$ and 1*N* KCl as the suspending media, a 1:2 soil-liquid ratio was used. The field-moist and air-dry pH was also determined with the Hellige-Truog colorimetric kit.

The cation exchange capacities were determined by the sodium saturation method described by Jackson (9). The total exchangeable bases were determined by the method described by Bray and Willhite, as reported by Jackson (9). Electrical conductivity measurements were made using the procedure of Greweling and Peech (6). Total organic matter was determined by the method of Mitchell as described by Jackson (9).

Lime requirement was evaluated by the following three methods (i) Shoemaker et al., (20) buffer method; (ii) pH and texture; (iii) exchange acidity—difference between CEC and total exchangeable bases.

RESULTS AND DISCUSSION

Evaluation of the Errors Encountered in the Measurement of Soil pH with the Glass Electrode

To evaluate the errors in soil pH measurements with the glass electrode, consideration was given to: (i) the variations in duplicate pH measurements made on composite field-moist samples from each of the 19 sites, compared to the variability of duplicate measurements made on six core samples from each of these sites; (ii) the variability of the pH of several standard air-dry laboratory check samples measured repeatedly during this study; (iii) the effects of

different operators, different pH meters, and different times on the pH of five air-dry soil samples; and (iv) the reproducibility of soil pH measurements made on these air-dry samples—in water, in 0.01*M* $CaCl_2$, and in 1.0*N* KCl.

Variability of Field Moist pH Values Within Each of the 19 Soil Sites

The variability of field-moist pH values of the first composite samples collected from each plot and each of the six single-core samples collected from .16 of each plot were employed to determine the variability of the pH within each plot. These pH values were measured in water with one pH meter, by one operator on the same day. The determination standard deviation for the individual sites ranged from a low of 0.096 to a high of 0.185 pH unit, and for the combined analyses it was 0.138 pH unit (Table 1). The individual F statistics for testing significance among cores and the composite sample within each plot were all nonsignificant. In fact, the F tests deviated only slightly from 1.0. Thus, it can be concluded that the determination variability is consistent from site to site and that the variability in pH measurements of a particular soil site at a given time is due mainly to determination errors rather than to sampling or site variability.

Variability of pH Measurements Made on Standard Laboratory Check Samples

The pH values of six air-dry check samples used in this investigation were each measured several times during this study, on approximately the same date that the pH of 19 sites used in this study was measured.

The variability of the pH of each of the check samples was 0.3 pH unit or less during the season. The analysis of variance for these data was performed and the standard error of the mean was calculated to be 0.062 pH unit. With this statistic, t tests were performed and none of the measured mean pH values of the six check samples differed significantly from their "known value," using the .01 probability level t test criterion. Hence, it can be concluded that the standard deviation, which equals 0.152 pH unit, is mainly a determination standard deviation.

The Effects of Different Operators and Different pH Meters on the Variability of Soil-pH Values

Each of three operators measured the pH of five air-dry soil samples with one pH meter on the same day in the following suspending media: H_2O, 0.01 *M* $CaCl_2$, and 1.0 *N* KCl. A three-way analysis of variance (five soils, three operators, and three suspending media) was performed on the data; soils and media were considered fixed and operator was random in that analysis. Significant differences were found for all main effects and a soil by media interaction (Table 2). Therefore, the variability of soil-pH measurements made by different operators must be considered in studies of this type.

In addition, the pH of the above five soil samples was measured in the three suspending media by one operator using two pH meters. The LSD .05 statistic, calculated using these data, was found to be 0.09 pH unit for the operators and 0.06 pH unit for the pH meters. By these criteria differences between the operators and pH meters were significant and nonsignificant at the 5% level, respectively. Hence, variation in pH measurements made under these conditions with the different pH meters are not important.

Table 2—Three way analysis of variance (5 soils, 3 operators, 3 suspending media) of pH measurements on air-dry samples in water, 0.01*M* $CaCl_2$ and 1.0*N* KCl

Source	df	MS
Soil	4	1.96**
Operator	2	0.13**
Soil × operator	8	0.01
Suspending media	2	3.62**
Soil × suspending media	8	0.04**
Operator × suspending media	4	0.01
Error	16	0.01

* Significant at 5% level. ** Significant at 1% level.

Reproducibility of pH as Measured in Three Suspending Media—One operator employed a single pH meter and made 10 pH measurements on the above five soil samples at hourly intervals on one day. The standard deviations of pH measured in water, in 0.01 *M* $CaCl_2$ and in 1.0 *N* KCl were ±0.08, ±0.07, and ±0.08, respectively. The hypothesis of homogeneous variances could not be rejected at the 5% level. Hence, it can be concluded that there are no significant differences in the reproducibility of soil pH values as measured in each of the three suspending media on a single day on air-dry samples.

Variability of Soil pH Values Measured in 0.01 *M* $CaCl_2$ on Two Different Dates

The pH of all the air-dry samples from each of the 19 sites used in this study was measured in 0.01 *M* $CaCl_2$ on two different dates by one operator using a single pH meter. The regression of March 21 values on April 13 values was calculated (Fig. 1): $Y = 0.04 + 0.99X$. The standard error of the estimate equals 0.15 pH unit. As an approximate measure of significance at the 5% level on an individual basis, the LSD was calculated using the standard error of the estimate. Hence, in individual soil-pH

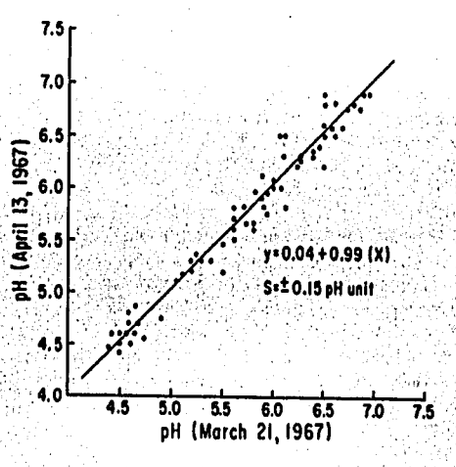

Fig. 1—Comparison of pH values on air-dry samples from 19 sites measured in 0.01*M* $CaCl_2$ on two dates.

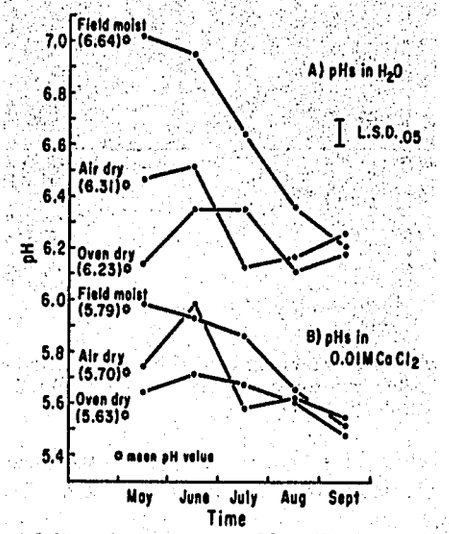

Fig. 2—Variability of average monthly pH of ten sites measured on field-moist, air-dry or oven-dry samples in two suspending media.

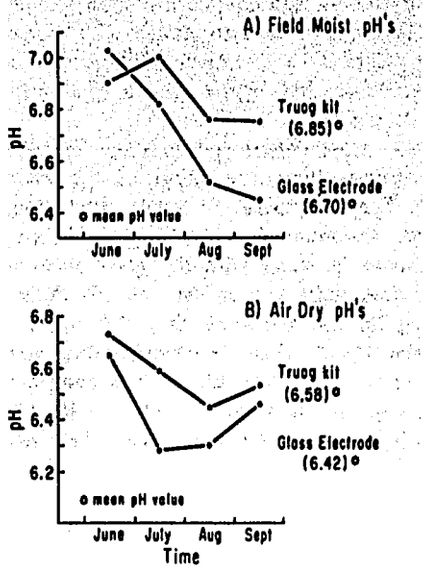

Fig. 3—Comparison of average monthly pH values on field-moist and air-dried samples of the 19 sites measured with the Truog kit and in H_2O with a glass electrode.

measurements where difference exceeds 0.42 pH units the difference will be beyond reasonable chance fluctuation. When more than one determination, say n, is made per sample, the error is reduced by $1/\sqrt{n}$.

Seasonal Variability of Average Monthly Soil-pH Values as Measured on Samples at Three Moisture Conditions and in Three Suspending Media

Regardless of suspending media, the average monthly soil pH of field-moist samples showed a steady decrease in values from May through September, and the pH on oven-dried and air-dried samples showed a cyclic seasonal trend (Fig. 2) (Averaging of H^+ concentrations instead of pH gives lower average values and slightly greater variations in pH during the season.).

Monthly differences between soil-pH values measured on samples at the three moisture conditions were significant at certain times during the early part of this study. In fact, the calculated LSD at the .05 probability level equals 0.1 pH unit (see Fig. 2). This calculation was based on a site by time by moisture condition interaction. In May, June, or July, the average monthly pH values of air-dried and oven-dried samples measured in water were as much as 0.55 and 0.88 pH units lower, respectively, than the corresponding pH of field-moist samples (Fig. 2A). Similarly, the average monthly pH of air-dried and oven-dried samples measured in 0.01 M $CaCl_2$ were as much as 0.28 and 0.34 pH units lower, respectively, than the corresponding pH of field-moist samples (Fig. 2B). However, differences between the means of the pH of air-dried and oven-dried samples measured in either water or 0.01 M $CaCl_2$ were less than 0.1 pH unit and nonsignificant. At the conclusion of this study in September, there were no significant differences between pH values measured on field-moist, air-dry or oven-dry samples in water or in 0.01 M $CaCl_2$.

Average monthly pH values of field-moist samples measured in water and in 0.01M $CaCl_2$ showed seasonal variations of 0.80 and 0.47 pH units, respectively (Fig. 2). However, the pH of oven-dry and air-dry samples measured in 0.01M $CaCl_2$ were just as variable as the corresponding pH measured in water. The seasonal mean pH (in parenthesis Fig. 2) in 0.01M $CaCl_2$ were lower than the mean pH values in water when measured on samples at the following moisture conditions by the following amounts: field-moist, 0.85 pH unit; oven-dry, 0.60 pH unit; and air-dry, 0.61 pH unit.

The maximum variation of average monthly pH of air-dry samples measured in 1.0N KCl was 0.2 pH unit. This variation is about one-half of that observed for the corresponding pH measured in water and in 0.01M $CaCl_2$. The average monthly pH measured in water was the highest at all times during this study and those in 1.0N KCl were the lowest. The seasonal mean pH of air-dry samples measured in 0.01M $CaCl_2$ and in 1.0N KCl was 0.6 and 1.0 pH unit lower, respectively, than the corresponding pH measured in water.

The simple correlations between the pH of air-dry samples measured in water compared to corresponding measurements in 0.01M $CaCl_2$ or in 1.0N KCl (all with glass electrode) and with the Truog kit in water were 0.96, 0.92, and 0.90, respectively. Each of these was significant at the less than 0.01 probability level. The linear regression equations were calculated to be:

$\hat{y} = -1.09 + 1.09\ (X)$, where $\hat{y}$ = predicted 0.01M $CaCl_2$ pH and X = H_2O pH

$\hat{y} = -1.31 + 1.05\ (X)$, where $\hat{y}$ = predicted 1.0N KCl pH and X = H_2O pH

$\hat{y} = 0.01 + 1.02\ (X)$, where $\hat{y}$ = predicted Truog kit pH and X = H_2O pH

These correlations and equations are sufficiently strong to indicate that it is possible to predict with reasonable accuracy the pH of air-dry samples of these soils in either of the above suspending media once a standard suspending medium is chosen.

The pH values determined with the Truog kit showed seasonal trends similar to those of the glass electrode (Fig. 3). The field-moist pH values determined with this kit showed less seasonal variability than the corresponding pH measured in water with the glass electrode. On the average, field-moist and air-dry soil pH determined with the Truog kit was not more than 0.3 and 0.2 pH units higher, respectively, than the corresponding glass electrode pH. Therefore, the Truog kit, as it is commonly used by soil surveyors in Michigan, appears to be a satisfactory field kit for pH determinations when the above are considered.

Seasonal Variations in pH of Individual Sites

The pH values of field-moist samples measured in water of each of the 19 sites studied showed the above-mentioned seasonal variability. The maximum variability of pH values for the individual sites varied from 0.5 pH unit for site no. 6 to 1.6 pH unit for site no. 14. Measurements in 0.01*M* $CaCl_2$ showed significant seasonal variations on only 14 of the 19 sites.

Only 11 of the 19 sites showed seasonal variability when the pH of air-dry samples was measured in water. The variability of the pH of the other eight sites fell within the error deduced in this study for pH measurements.

The pH of air-dry samples showed significant seasonal variability on only seven of the sites when measured in 1.0*N* KCl and 10 showed significant seasonal variability when measured in 0.01*M* $CaCl_2$. Figure 4 shows the seasonal variability of the pH of Hillsdale sandy loam, site no. 7, measured on field-moist or air-dried samples in three suspending media. The seasonal variations of the pH of this site are similar to the average trends in Fig. 2, but the salt solutions failed to decrease seasonal fluctuations in pH at this site. These situations warrant additional study.

Several Factors Influencing the Seasonal Variability of Soil pH Values

To help explain the seasonal variations in soil pH-measurements, the field moisture content, total exchangeable metallic cations, electrical conductivity, and organic matter content were determined on each sample collected from each site during the study. In addition, the cation exchange capacity was determined on one sample from each site. Note that it was assumed that there were no significant changes in the cation exchange capacity of any site during the study. Statistical analyses of these data were made and the resulting significant correlations between pH and these soil properties are shown in Table 3.

The relationship between pH and base-saturation percentage was quite variable and the correlations were not very high, but were significant at the 1% level (Table 3). Note that pH of air-dried samples in 0.01*M* $CaCl_2$ or in

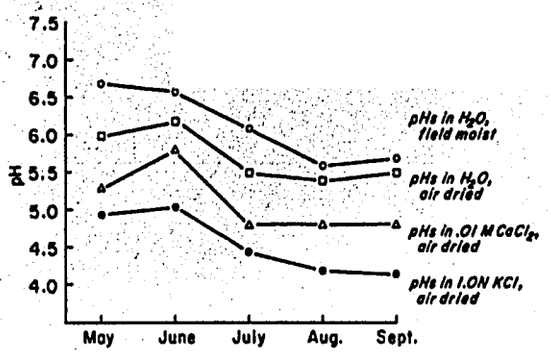

Fig. 4—Seasonal variability of the pH of Hillsdale sandy loam, site 7, when measured on field-moist or air-dried samples in three suspending media.

1.0*N* KCl gave a higher correlation with base-saturation percentage than the pH measured in water. Since the pH vs. base-saturation percentage relationship was just as variable in those samples for which the cation exchange capacity was actually measured as it was in the other samples collected from each site during the season, the above-mentioned assumption of no significant seasonal changes in cation exchange capacity seems reasonable.

Electrical conductivity was negatively correlated with soil pH of the field-moist or air-dry samples (Table 3). The negative correlation shows that an increase in electrical conductivity, due probably to the presence of soluble salts, is associated with a decrease in soil pH values. The field-moisture percentage was also negatively correlated with electrical conductivity as would be expected if this water were effectively diluting any salts present. The higher correlation of electrical conductivity with the pH of air-dry samples also supports these relationships.

Organic-matter percentages were positively and significantly correlated with the pH of field-moist samples, but they were only slightly and nonsignificantly correlated with the pH of air-dry samples. This difference is evidently associated with the positive and significant correlation of organic-matter percentage with field-moisture percentage and of field-moisture percentage with the pH of field-moist samples.

Hence, this evidence indicates that soluble salts are probably responsible for most of the seasonal variability of the pH of most of the soil sites studied. However, the pH of air-dry samples of some sites showed seasonal variability when measured in 0.01*M* $CaCl_2$ and even in 1.0*N* KCl. This indicates that other factors besides soluble salts

Table 3—Simple correlations between other soil properties and field moisture percentages or soil pH as measured with a glass electrode

Other soil properties	Field moisture percentage	pH's measured on field moist or air dried samples, in three suspending media: Field moist H_2O	Air dried H_2O	Air dried 0.01 M $CaCl_2$	Air dried 1.0 N KCl
Base saturation %		0.49**	0.53**	0.61**	0.63**
Electrical conductivity	-0.73**	-0.34**	-0.82**		
Organic matter %	0.76**	0.59**	0.09		
Field moisture		0.29**			

** Significant at 0.01 level.

are responsible for the observed variation in the measured pH of some soils, e.g. Fig. 4. Recent studies of Mortland (13) and Mortland and Raman (14) show that clay films in equilibrium with atmospheres of different relative humidities vary greatly in their surface acidity with the structure of the clay and the exchangeable cation on the clay. These situations deserve further study.

Seasonal Variability of Lime Requirement

The variation of lime requirement was evaluated on air-dry samples collected from 11 sites. On the average, the pH plus texture method showed the maximum-average-seasonal variability of lime requirement, which was 2.24 ton/ha (1.0 ton/acre) (Fig. 5). The maximum-average variations in lime requirements by the buffer method were comparable to, but greater than, those of the exchange-acidity method. The pH plus texture and S.M.P. buffer methods showed a cyclic seasonal trend, with the maximum lime requirement in midsummer (July). Contrary to these, lime requirement evaluated by the exchange-acidity method tended to fluctuate during the season and was low in August as well as in June. If site no. 14 is excluded, the variation in the average lime requirement by the exchangeable-hydrogen method during the season was only 0.67 ton/ha (0.3 ton/acre).

Four of the 11 sites had a lime requirement at the beginning of the season, but each of the 11 sites had a lime requirement in July or in August. The lime requirement of about one-half of the sites varied between 3.36 and 2.5 ton/ha (1.5 and 2.5 tons/acre) during the season by each of the three methods. The maximum seasonal difference in lime requirement for any of the sites studied was 8.96 ton/ha (4 tons/acre) with use of the exchange acidity method on samples from site no. 14. Hence, seasonal variability should be considered when lime recommendations are made on some sites.

Standardization of pH for Soil Classification Purposes

In the characterization of soils similar to these for classification purposes the data indicate that air-dry or oven-dry samples in a salt solution should be used to reduce seasonal variability in glass electrode pH measurements (Fig. 2). Where seasonal variations in pH occur those also need to be characterized.

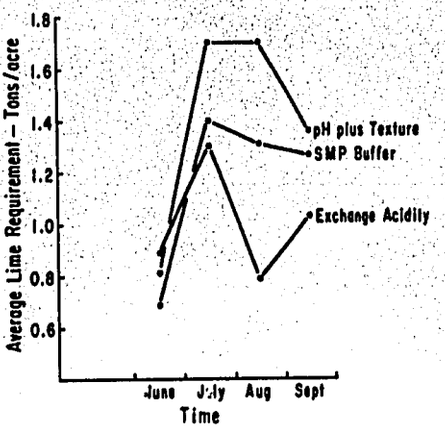

Fig. 5—Seasonal variability of average monthly lime requirements determined by three methods.

LITERATURE CITED

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