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Exchangeable Potassium and Clay Minerals in Selected Iowa Soil Profiles¹

J. J. HANWAY, R. L. HANDY, and A. D. SCOTT

Abstract. Exchangeable K values and X-ray diffraction patterns were obtained for soil samples from several modal profiles of nine different soil series in Iowa. Exchangeable K in "moist" subsoils was consistently low and almost always lower than in the "moist" surface soil. Drying increased exchangeable K in all the soils, but the increase due to drying varied between soil series and with depth in the profile of each series. Reversion of exchangeable K to a non-exchangeable form on rewetting the oven-dry samples generally increased with depth in the profile but the exchangeable K never decreased to the original "moist" level. X-ray diffraction analyses showed the presence of montmorillonite, chlorite, illite and/or mica, and kaolinite in all the profiles, with montmorillonite predominating. Vermiculite was present in some of the soil series. No relationship between the clay mineral content and the amount of exchangeable K could be established.

Potassium (K) is held on the clay minerals in soils in exchangeable and non-exchangeable forms. The amount of exchangeable K in Iowa soils, however, depends on the moisture status of the soil (Luebs, *et al.* 1956; Scott, *et al.*, 1957). Drying generally increases the amount of exchangeable K, especially in subsoil samples. The K released on drying may revert to a non-exchangeable form on rewetting, but the amount of such reversion varies greatly between different soil samples. The amount of exchangeable K also depends on the kind and amount of clay minerals in the soil. Most of the K on montmorillonite and kaolinite is readily exchangeable. Much of the K in illite and vermiculite, on the other hand, is not readily exchangeable because it is located between the layers of a contracted lattice. A large amount of the K in Iowa soils is presumed to be present in this non-exchangeable form.

It is well known that the amount of clay varies markedly between different soils and between different horizons of any one soil type found in Iowa. However, much less is known about the kinds of clay minerals present. On the basis of differential thermal analysis, chemical composition, and base exchange capacity, Russell and Haddock (1940) calculated that in five Iowa soils (Clarion, Grundy, Tama, Webster and Weller) the average percentages of montmoril-

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lonite, illite, and kaolinite were 60, 30, and 10, respectively. Peterson (1946) used X-ray diffraction of the less than one micron clay, differential thermal analysis, and base exchange capacity in studying different Iowa soils and concluded that the predominant mineral in Clinton, Tama, Clarion, Webster, and Marshall soils was montmorillonite with possibly some kaolinite and in the Marshall soil possibly some illite. Only in a Gosport soil was the clay predominantly kaolinite. In an earlier study, Peterson (1944) used differential thermal analysis to study the clay minerals in different soils and concluded: (1) the platy structure in certain horizons of the Clinton, Fayette, Marion, and Edina soils was associated with a higher content of kaolinitic clays in these soils as compared to Clarion, Webster, Tama, and Marshall soils that have no horizons with a platy structure; (2) in general montmorillonite gradually increased with depth in gray brown podsols and planosols but not in prairie soils; and (3) Lindley and Shelby soils developed on Kansas and/or Nebraskan till had a higher proportion of kaolinite than did other Iowa soils. No studies relating the amount of exchangeable K to the kinds and amounts of different clay minerals in Iowa soils have been reported.

The purpose of this study was to obtain more information concerning the kinds of clay minerals in the profiles of different Iowa soils, to determine the amount of exchangeable K in Iowa soils, and to determine if any relationship could be found between the kinds of clay minerals and the amount of the exchangeable K in these soils under different moisture conditions.

METHODS AND MATERIALS

Soil samples were collected from different horizons of modal profiles of nine different soil series. The number of profiles of each series sampled and the counties in which they were located are shown in Table 1. The soil samples were stored in paper cartons in a cool (40°F), constant temperature room for several months prior to analyses. During this storage the moisture content of the soil samples decreased to between 5 and 10 percent. Therefore, the soil samples were not truly field moist when analyzed, but are referred to as "moist" in this paper to distinguish them from oven-dried samples.

Exchangeable K was determined on the "moist" and oven-dry (110°C. for 24 hours) soil samples by extracting 2 gm. soil with 10 ml. neutral, 1 N NH₄OAc and determining K in the extract with a flame photometer. To determine if the K released to an exchangeable form on drying reverted to a fixed form during moist storage, 2 ml. of water was added to 1.5 gm. samples of the "moist" and oven-

Table 1
Numbers and Locations of Modal Profiles of Different Soil Series
Included in This Study

Soil series	Number of profiles sampled	Counties in which profiles were located
Carrington	6	Mitchell, Linn
Tama	5	Marshall, Scott
Clinton	5	Jefferson
Fayette	6	Clayton, Winneshiek
Mahaska	2	Jefferson
Edina	4	Wayne, Davis, Van Buren
Shelby	3	Shelby
Marshall	3	Shelby
Weller	4	Decatur, Jefferson

dry soils and, after 90 days storage, exchangeable K was extracted as described above.

Samples from each horizon of two of the modal profiles of each soil series were selected for X-ray diffraction analysis. Each sample was air-dried and pulverized to pass a 200 mesh sieve. A representative portion of the material passing the sieve was treated with ethylene glycol to expand the montmorillonite, and packed wet into a sample holder. The surface was smoothed with a glass slide to increase preferred orientation and increase intensities of basal reflections. All analyses were made with a Geiger counter diffractometer utilizing a 1° beam slit, 0.2° detector slit, 3 second time constant, 2° per minute scan rate, 1000 cps full scale, and $\text{CuK}\alpha$ radiation. Selected samples were given various other treatments prior to or without glycolation, to confirm the identification of the several clay minerals.

Quantitative analysis by X-ray diffraction without the use of internal standards is at best an estimate, since diffraction intensities depend not only on the amount, composition, and crystallinity of a mineral, but also on sample packing, amount of excess glycol, degree of preferred orientation, and absorption of X-rays by the samples. With $\text{CuK}\alpha$ radiation, absorption is particularly sensitive to the presence of iron; therefore, clay diffraction intensities may be lower in an iron-rich B horizon than in an underlying C, even though the B contains more clay mineral. Nevertheless, to summarize the X-ray diffraction data, quantitative estimates were attempted by a peak area method in which peaks were approximated by triangles. Overlapping peaks were separated and carefully evaluated with regard to symmetry and total area. Peak areas were calculated and referred to a geometric scale to give more emphasis to minor minerals. The scale is as follows:

<i>Area, in. ²</i>	<i>Scale</i>	<i>Rating</i>
0	0	Undetectable
<0.05	t	trace

Area, in. ²	Scale	Rating
0.05—0.1	1	scarce
0.11—0.2	2	minor
0.21—0.4	3	moderate
0.41—0.8	4	abundant
0.81—1.6	5	very abundant
1.61—3.2	6	predominant

RESULTS and DISCUSSION

Profile distribution of exchangeable K. The distribution of exchangeable K in the profiles of the nine different soil series when K was extracted from the "moist" and oven-dry soil samples is shown in Figure 1. The ranges of exchangeable K shown in Figure 1 for the "moist" and oven-dry samples from the various depths include the data from all of the profiles of each soil series.

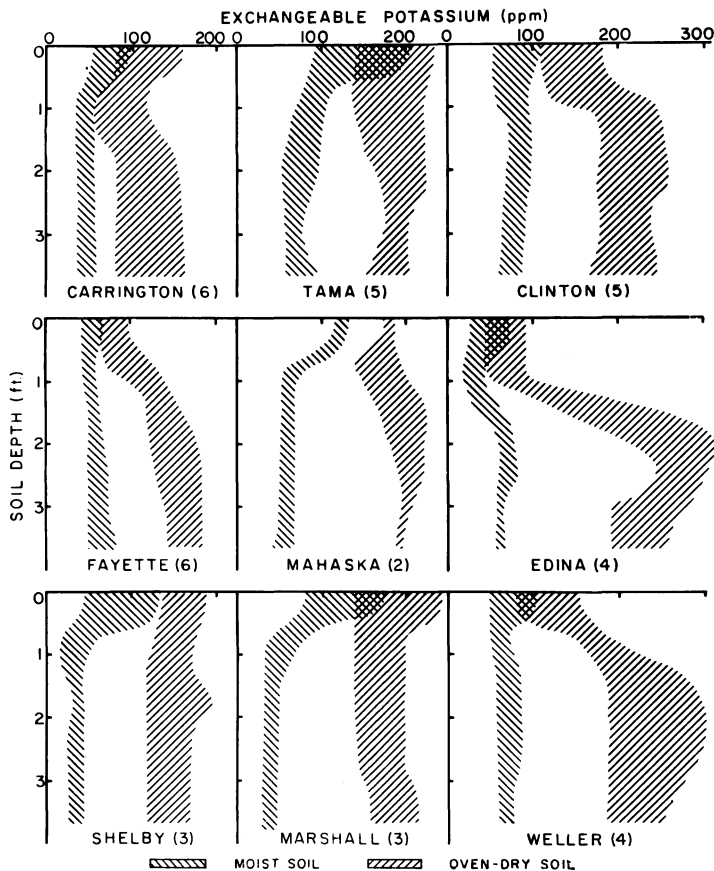


Figure 1. Profile distribution of exchangeable K as determined on "moist" and oven-dry (10° C., 24 hours) samples of nine soil types. (The numbers in parentheses indicate the numbers of profiles of each soil series that were included in establishing the range in exchangeable K shown for the "moist" and oven-dry soils.)

Except for the surface soil samples, the variability in exchangeable K in the "moist" samples within a soil was relatively small. It can also be seen that the profile of different soil series differed more than the profiles within a soil series. The "moist" value for the surface soils did vary widely in some cases, notably in the Tama, Shelby, and Marshall soils.

Exchangeable K in the "moist" samples was relatively constant with depth throughout the profiles of the Clinton, Fayette, and Weller soils but decreased with depth in the Carrington, Tama, Mahaska, Shelby, and Marshall soils and increased in the B horizon of the Edina profile. These data also illustrate the low level of exchangeable K found in subsoils under moist conditions. Other studies have shown that these "moist" values are slightly higher than would have been obtained with truly field-moist samples. The Marshall and Shelby profile samples did not get as dry as the others, and it is believed that they show more correctly that the exchangeable K in subsoils in the field is often very low, and in Iowa is almost lower than in the corresponding surface soils.

Drying increased exchangeable K in all samples, but the amount of K released on drying varied with soil type and with depth in each profile. Thus, the profile distribution of exchangeable K in each soil depended on the moisture content of the soil samples at the time of extraction of exchangeable K. Subsoil samples generally released more K on drying than did the surface soils.

Exchangeable K in the oven-dry samples was relatively constant throughout the profiles of the Carrington, Tama, Mahaska, Shelby, and Marshall soils (although it decreased somewhat in the A_3-B_1 horizon of the Carrington profile), but was higher in the subsoil than in the surface soil in the Clinton, Fayette, Edina, and Weller profiles. All the soils in the first group have developed under grass vegetation. In the latter group, except for the Edina, the soils were developed under forest vegetation. The Edina is a highly weathered planosol developed under grass. Further studies on this relationship of exchangeable K to clay distribution in the profile and to native vegetation would, therefore, appear warranted.

Reversion of K from exchangeable to non-exchangeable forms during moist storage. Luebs, *et al.* (1956) have shown that some of the K that has been released by drying will revert to a non-exchangeable form on rewetting. It was of interest, therefore, to determine if the amount of reversion varied with depth in the profile and from one soil series to another. The amount of reversion that occurred when oven-dry samples from selected profiles of the different soil series were stored in water for 90 days is shown in Figure 2. The exchangeable K values obtained after rewetting the "moist" sample are also shown by dotted lines for comparison.

It can be observed, by comparison of the "moist" values in Figures 1 and 2, that there was little change in exchangeable K in these "moist" samples due to storage in water for 90 days. Although some release of K had occurred in these "moist" soil samples that were partially dried, rewetting appeared to result in very little reversion of this to a non-exchangeable form.

In most of the soil profiles there was only a small amount of reversion in oven-dry samples from the upper horizons. The amount of reversion increased with depth in the profile, but in no case did reversion decrease the exchangeable K level to that found in the "moist" soil. Also, it can be seen that the profile pattern in reversion varied markedly from one soil series to another. These results

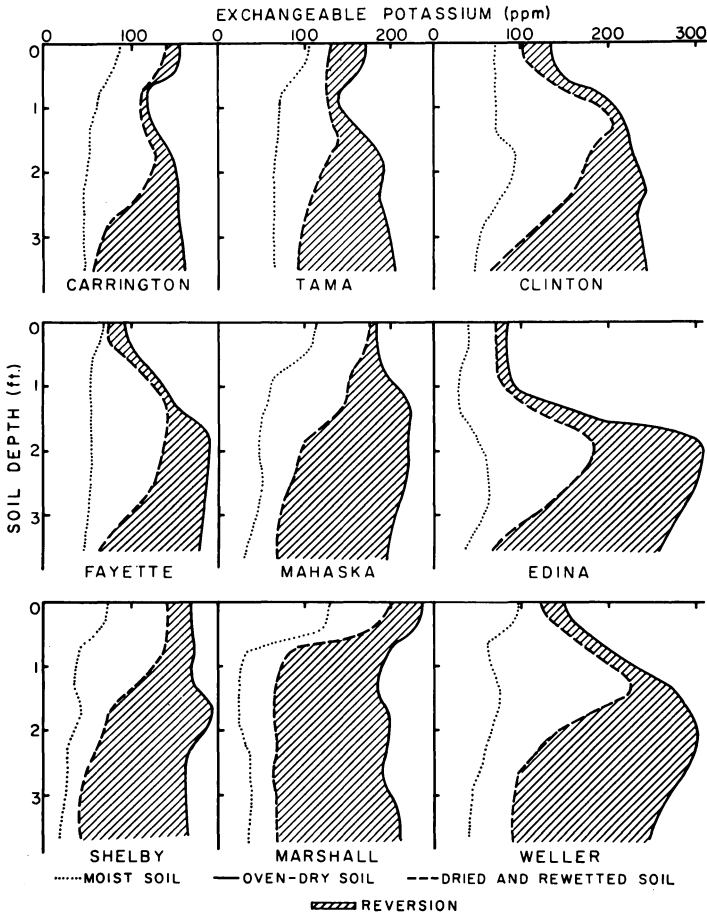


Figure 2. Exchangeable K in oven-dry (110° C., 24 hours) and "moist" Iowa soil profile samples that have been stored in water for 90 days. The exchangeable K values obtained with the oven-dry samples are also shown for comparison.

indicate that the process of K release on drying is only partially reversible and that rewetting of dried soil samples, especially from the surface horizon, will not result in exchangeable K values similar to those obtained for undried soils.

X-ray diffraction patterns for profile samples. X-ray diffraction patterns were obtained for the various profile samples to provide information concerning the kinds and relative amounts of different clay minerals. The relative intensities of the X-ray diffraction peaks indicating 17 Å, 14 Å, 10 Å, and 7 Å spacings in glycolated samples are reported in Table 2. X-ray diffraction patterns for the different horizons of a Marshall silt loam profile (No. 39) are shown in

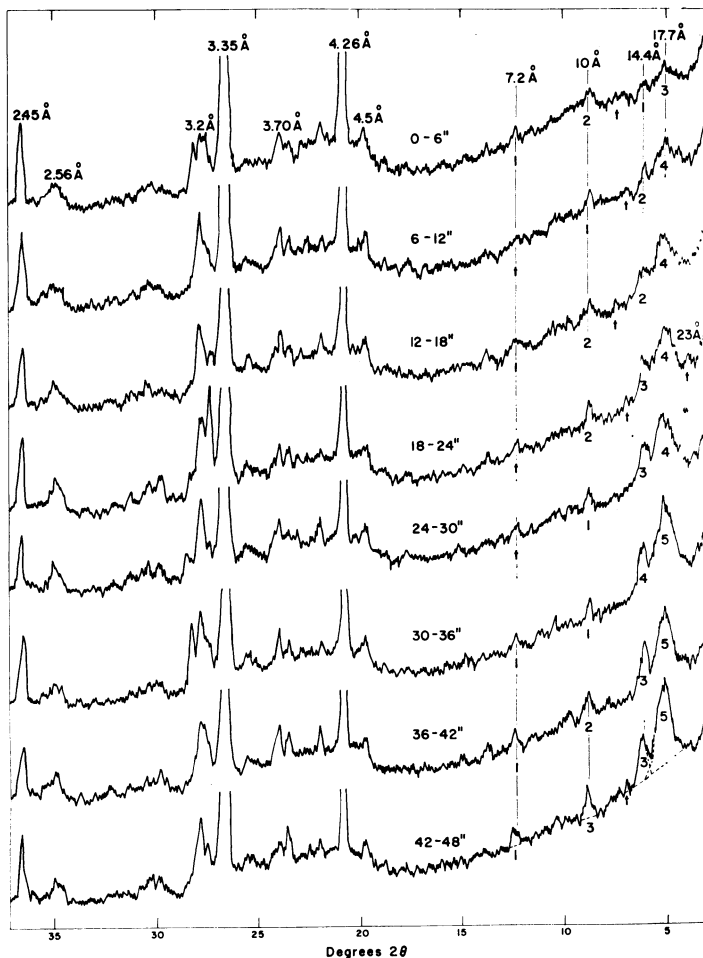


Figure 3. X-ray diffraction patterns for glycolated samples from different horizons of a Marshall silt loam profile (profile No. 39).

Figure 3. The relative intensity ratings of different peaks are shown in this figure to illustrate the size of peaks represented by the data in Table 2.

Relative intensity ratings for samples from similar horizons of different profiles of the same soil series usually agree within one scale unit and never differ by more than two units. The relative intensities of the peaks tend to be lower and more variable for the A horizon samples than for the subsoil samples. Interferences as a result of the higher organic matter contents of the A horizons could be responsible for much of this variability and the lower intensities.

The largest diffraction peak for nearly all glycolated samples was in the neighborhood of a 17.7 Å spacing. In untreated samples heated to 700° C for 0.5 hour this 17 Å peak was replaced by a large 10 Å and a smaller 9.6 Å peak. These results indicate that a montmorillonite-type mineral is present in all these soil profiles. Broadness of this peak suggests a very fine crystallite size in the C-axis direction.

Relative intensity ratings of 2 to 6 were obtained for the 17 Å peaks in the B and C horizons of all profiles, with ratings of 4 and 5 predominating. This indicates an abundant amount of montmorillonite in the parent material and in the zone of clay accumulation. The relative intensities of 17 Å peaks for samples from A horizons were lower and more variable, ranging from a trace to 5.

Most of the profile samples gave a diffraction peak indicating a spacing of 14.4 to 14.0 Å. This peak could be due to chlorite and/or vermiculite. Therefore, fresh material from about one-third of the samples was heat-treated at 700° C for 0.5 hour and re-run. As shown in Figure 4, heat treatment reduced the 14 Å peak, but failed to eradicate it, indicating the presence of chlorite. On the other hand, wetting a fresh sample with water usually removed the 14 Å peak or masked it with a strong, sharp 15 Å peak. According to Barshad (1950), calcium-saturated vermiculite immersed in water has an 001 spacing of 15 Å. Furthermore, this 15 Å peak was not observed in soil samples (such as Edina 32 A) that did not exhibit a 14 Å peak when treated with glycol. Thus, the 14 Å peak probably indicates vermiculite plus trace amounts of chlorite. Intensities of the 15 Å and other clay mineral peaks in the water-wet samples were considerably increased by preferred orientation. Sharpest peaks occurred after setting a few minutes.

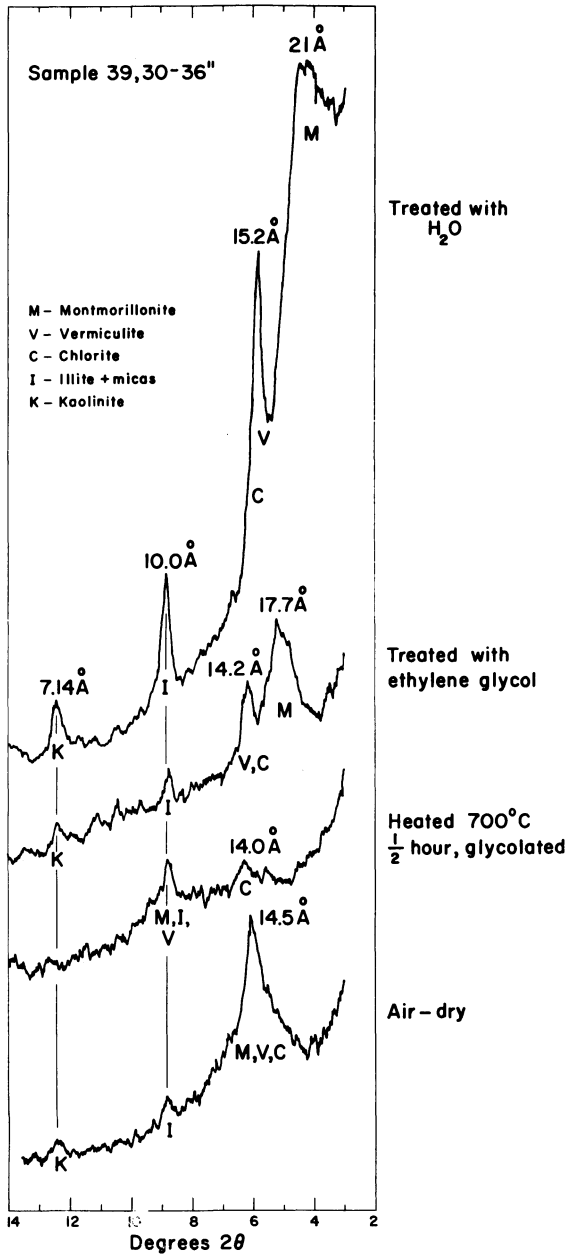


Figure 4. X-ray diffractograms comparing the effects of different treatment on the 30 inch to 36 inch depth sample from Marshall silt loam profile (No. 39).

Table 2
Relative Intensities of X-ray Diffraction Peaks for Samples from Profiles of Different Soil Series in Iowa

Soil Series	Sampling Depth (Inches)		Genetic Horizon		Relative Intensities *								Interlayer Spacing (A)					
	Profile		I	II	I	II	17A		14A		10A		7A		I	II		
	I	II					I	II	I	II	I	II	I	II				
Carrington 13	15	0	0															
		6	6	A ₁	A _p	i	4	2	1	1	2	t	2	16, 13			
		14	12	A ₃ -B ₁	A ₃ -B ₁	1	3	2	1	1	2	1	t		20, 12	12		
		26	18	B ₂	A ₃ -B ₁	4	3	3	2	1	1	2	1			
		27	24	B ₂	B ₂	3	3	3	2	2	t	1	t	12		
			30		B ₃		4		2		t		1			
			36		C	C	3	4	1	1	1	1	1			
			48		C	C	4	5	2	2	1	t	3	2		
			0	0														
			6	6	A _p	A _p	1	1	t	1	1	1	t	t	16, 13, 11	12		
Tama 1	6	6	6	A ₃	A ₃	2	2	1	1	1	1	t	t	20, 16, 12	13			
		13	15	B ₂	B ₂	3	3	3	1	2	1	1	t			
		19	20	B ₂	B ₂	3	3	3	1	2	1	1	t			
		25	26	B ₂	B ₂	4	4	2	3	2	1	1	t	20			
		31	31	B ₃	B ₃	4	4	3	3	1	t	t	1	24			
				C	C	4	4	3	2	1	1	1	1	11	11			

Soil Series		Sampling Depth (Inches)		Genetic Horizon		Relative Intensities *								Interlayer Spacing (A)	
						17A		14A		10A		7A			
I	Profile II	I	II	I	II	I	II	I	II	I	II	I	II		
1	Tama	37	42	C		4	3	2	1			24			
		49	0												
7	Clinton	5	6	A _p	A _p -A ₂	2	3	1	2	0	2	t	t	12	
		12	12	A ₂	B ₁	4	4	3	3	1	1	1	2	24	
		18	18	B ₁	B ₁	4	4	2	3	1	1	0	t		
		26	24	B ₂	B ₂	4	5	2	2	t	1	t	1		
			30	B ₃	B ₂	4	4	2	2		t		1		
			36		B ₃		4		3	1	1	t	t		
			42	B ₃	B ₃ -C	4	5	2	3	1	2		t		
			48	C		4		2		1		t			
		0	0												
20	Fayette	6	6	A _p -A ₂	A _p -A ₂	t	1	1	t	1	1	t	t	12	
		12	12	B ₁	B ₁	2	3	1	2	1	1	t	t	16, 12.5	
		18	18	B ₁	B ₁	3	3	2	2	1	t	t	t	24	

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Soil Series		Sampling Depth (Inches)		Genetic Horizon		Relative Intensities *								Interlayer Spacing (A)	
		I	II			17A		14A		10A		7A		I	II
I	Profile II	I	II	I	II	I	II	I	II	I	II	I	II		
20	Fayette	24	24	B ₂	B ₂	5	4	3	3	1	t	1	t
		36	36	B ₂	B ₂	5	4	4	3	2	1	1	t
		48	48	B ₃	B ₃	4	4	3	3	1	1	t	1	12
		0	0												
28	Mahaska	8	6	A _p	A _p	3	1	t	t	1	t	0	t
		13	13	A ₃ -B ₁	A ₃ -B ₁	4	3	t	1	t	2	t	t	12
		19	19	B ₂	B ₂	4	5	1	t	t	2	0	1	24
		26	26	B ₂	B ₃	4	5	1	1	t	2	t	1	24
		33	36	B ₃	C	4	5	1	2	t	2	t	t
		40		C	C	4		2		t		1		24
		48		C	C	4		t		1		t	
		0	0												
27	Edina	8	8	A _p	A _p	2	2	1	t	1	1	t	t	24, 12
		15	14	A ₂	A ₂	2	2	t	t	1	2	t	t
				B ₂	A ₂	5	3	1	t	t	t	t	0

Soil Series		Sampling Depth (Inches)		Genetic Horizon		Relative Intensities *								Interlayer Spacing (A)		
						17A		14A		10A		7A		I	II	
I	Profile II	I	II	I	II	I	II	I	II	I	II	I	II			
27	Edina	32	22	19	B ₃	B ₂	5	5	2	t	1	1	t	t	12
			29	28	C	B ₃	5	5	1	t	t	t	t	t
			36	37	C	C	5	5	3	t	t	t	t	t
			48	48	C	C	5	5	3	t	t	t	t	t
37	Shelby	38	0	0	A _p	A _p	4	3	1	t	2	1	t	t	12
			6	6	B ₂	B ₁	5	3	t	t	1	1	1	1
			12	12	B ₂	B ₂	5	4	3	1	t	1	1	2	12
			18	18	B ₂	B ₂	4	5	2	1	1	2	t	2	12	24
			24	24	B ₂	B ₂	4	5	2	1	1	2	t	2	12	24
			30	30	B ₂	BC	4	3	3	t	1	1	1	1	12
			36	33	B ₃	C	4	3	2	t	2	2	1	1	12	12
			42	39	C	C	4	2	3	t	2	2	1	1
34	Marshall	39	48	48	C	C	4	4	0	1	1	1	2	1	11
			0	0	A _p	A _p	3	3	1	1	2	2	t	1	12
			6	6	B ₁	A ₃	4	4	3	2	1	1	1	t	13

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Soil Series		Sampling Depth (Inches)		Genetic Horizon		Relative Intensities *								Interlayer Spacing (A)	
		I	II			17A		14A		10A		7A		I	II
I	Profile II	I	II	I	II	I	II	I	II	I	II	I	II	I	II
Marshall 34	39	12	12												
		18	18	B ₂	B ₂	5	4	3	2	2	2	1	1	12
		24	24	B ₂	B ₂	5	4	2	3	2	2	1	t	12
		30	30	B ₂	B ₂	5	4	3	3	1	1	1	t	12
		36	36	B ₃	B ₃	5	5	3	4	2	1	1	1
		42	42	C	C	5	5	3	3	2	2	1	1	11
Weller, Decatur Co. 24	25	48	48												
		0	0	A _p	A _p	2	1	1	1	1	1	t	t	12
		6	5	A ₂ -B ₁	A ₅ -B ₁	4	3	1	2	2	1	t	1
		11	10	B ₂	B ₂	5	5	1	2	2	1	t	t	24, 12
		20	20	B ₃	B ₃	5	5	1	t	1	1	1	0	12
		24	29	B ₃ -C	C	6	4	1	2	1	1	0	t	12
		36	36	C	C	5	5	1	1	1	1	t	1	11	20
		48	48												

Soil Series		Sampling Depth (Inches)		Genetic Horizon		Relative Intensities *								Interlayer Spacing (A)	
						17A		14A		10A		7A		I	II
I	Profile II	I	II	I	II	I	II	I	II	I	II	I	II		
Weller, Jefferson Co. 30 33		0	0	A _p	A _p	1	3	1	1	t	1	t	t	16	12
		7	6	A ₂	A ₂	4	5	2	1	2	1	t	t	20, 11
		15	13	B ₂	B ₂	5	5	2	2	2	1	t	t	11	24, 12
		23	22		B ₃		5		2		2		t	
			32	B ₃	C	5	5	2	2		1	t	t
		36	40	C	C	5	5	2	3	1	t	t	t
		48	48												

* 0-No detectable peak
t-trace
1-scarce
2-minor

3-moderate
4-abundant
5-very abundant
6-predominant

i-all in interlayer

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The 14 Å peaks were generally highest in samples from the B and C horizons. Very low intensity 14 Å peaks were observed for almost all surface soil samples. The 14 Å peaks were especially prominent in B and C horizon samples from the Fayette and Marshall profile with intensity ratings of 3 and 4. The Mahaska, Edina, and Weller profiles had only minor 14 Å peaks (except for a C horizon sample from one Edina and one Weller profile). B and C horizon samples from the other profiles generally had intensity ratings of 2 or 3. One Shelby profile had consistently larger peaks than the other.

Small peaks indicating a 10 Å spacing characteristic of micas and illite were common. No attempt was made to distinguish these minerals. Probably both classes of minerals were present, but apparently in small amounts since low relative intensity ratings of 1 and 2 predominated.

There was a small diffraction peak at a spacing of 7 Å in most of the samples. This spacing may be due to kaolinite on a second order reflection from chlorite. The 7 Å peak disappeared when the samples were heated at 700° C. This means that kaolinite was present, and the chlorite (indicated by 14 Å spacing, above) probably a low iron variety. The relative intensities of the 7 Å peaks for samples from these profiles was consistently low—generally only a trace. Only one C horizon sample from a Carrington profile had a rating as high as 3.

In some samples a small degree of random interlayering was indicated by small, broad X-ray diffraction peaks at several spacings. Most commonly these spacings were in the neighborhood of 11 Å to 12 Å; however, others occurred at 13 Å, 16 Å, 20 Å, and 22-24 Å. Indications of interlayering were present for fewer than half of the samples, and, where present, the relative intensity rating usually showed only a trace. Interlayering, or incomplete expansion of the montmorillonite, was most pronounced in several A horizon samples.

Other minerals indicated by X-ray traces in Figure 3 include quartz, which gave large, off-scale peaks at 4.21 Å and 3.35 Å and a sharp peak at 2.45 Å. A smaller, apparent 3.70 Å peak was the $K\beta$ reflection from the 3.35 Å quartz spacing (the filter thickness selected eliminated 95 percent of the $K\beta$ radiation). Sharp single, double, or triple peaks in the neighborhood of 3.2-3.4 Å are attributed to feldspars. Other broader peaks at about 4.5 Å, 3.5 Å, and 2.5 Å are non-diagnostic diffractions from the clay minerals.

GENERAL DISCUSSION AND CONCLUSIONS

These data from several modal profiles of each soil series indicate that the exchangeable K content and the effect of drying on the ex-

changeable K content are characteristic of each soil series. In undried samples the exchangeable K contents were higher and more variable in the surface soil than in the subsoil, and were consistently very low in the subsoil horizon. Drying increased exchangeable K in all of the soil samples, but the increase was generally much greater in subsoil than in surface soil samples. Reversion of K from exchangeable to non-exchangeable forms on rewetting dried soil samples occurred in all samples, but the amount of reversion generally increased with depth in the profile.

The X-ray diffraction analyses indicated that montmorillonite, chlorite, illite or mica, and kaolinite were in all of the profiles. The predominant micaceous mineral in all samples, however, was montmorillonite. Vermiculite was present in some of the soil series. These results agree with those of earlier investigators; however they did not consider vermiculite in their studies. The X-ray diffraction patterns for different profiles of any one soil series were very similar. The greatest difference between soil series was in the relative intensities of the 14 Å peaks obtained for samples from the B and C horizons. Soils of the Marshall and Fayette series had 14 Å peaks of high intensity, whereas soils of the Mahaska, Edina, and Weller series had 14 Å peaks of low intensity.

The X-ray data indicated differences in the amounts of montmorillonite, vermiculite, and mica in the various profile samples; but it was not possible to develop a relationship between the clay mineral composition and the exchangeable K in the profiles. The differences in relative intensities of the 14 Å (vermiculite) peak between different soil series do not appear to be related to any of the differences in amounts of exchangeable K in these profiles. Differences between surface and subsoil sample were observed in both the X-ray diffraction patterns and exchangeable K, but no consistent relationship was evident.

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