

MINERALOGICAL ANALYSIS OF SOIL CLAYS INVOLVING VERMICULITE-CHLORITE-KAOLINITE DIFFERENTIATION

by

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ABSTRACT

The content of kaolinite plus halloysite is differentiated quantitatively from chlorite by differential dissolution, even in those samples wherein each mineral has 500°C heat instability of the 7 Å peak. This differentiation is possible because the structures of kaolinite and halloysite are destroyed on loss of the 7 Å peak, producing amorphous material which is rapidly soluble in 0.5 N NaOH, whereas the chlorite structure remains largely intact with only partial dehydration of the octahedral layer. The amorphous residue of kaolinite-halloysite is dissolved from the other mineral components of the heated sample with sodium hydroxide and the dissolved silica and alumina are allocated to kaolinite. The percentages of kaolinite thus obtained for Elliott, B₃-C horizon, 2-0.2 μ fraction, are 3.8 and 3.7 based on silica and alumina, respectively. Similarly, the percentages of kaolinite obtained for Buchanan, B₁ horizon, 2-0.2 μ fraction, are 14.9 and 13.8 based on silica and alumina, respectively. Collapse of the vermiculite basal spacing to 10 Å on K saturation and heating to 300°C, together with the clear differentiation of kaolinite by differential dissolution analysis (D.D.A.) above, removed the possibility of misinterpretation of the chlorite-like diffractometer tracing by kaolinite and vermiculite occurring together. Other clays, from Davidson and Susquehanna soils, had 44 and 42 to 43 percent kaolinite, based on silica and alumina, respectively. The vermiculite and montmorillonite contents of soil clay samples are quantitatively determined by specific surface. The mica percentages are based on the K₂O after exclusion of potassium in feldspars. Water loss in the 400°C-to-ignition range gives an important quantitative basis for chlorite.

INTRODUCTION

Differentiation of kaolinite from chlorite, and the latter from vermiculite, is a problem because the 7 Å x-ray diffraction peak common to the three minerals is not fully differentiable by x-ray diffraction combined with heat treatments. The heat instability of the 7 Å peak of some chlorites in sediments (Bradley, 1954) at temperatures below 500°C has prevented differentiation of such chlorite from kaolinite and halloysite by criteria which are applicable for the typical mineral species. The resistance to collapse on heating of certain intergradient layer silicates of soils having 7 Å and 14 Å spacings (Brown, 1953; Rich and Obenshain, 1955; Tamura, 1957; Dixon and Jackson, 1959, 1960) confounds their differentiation from typical chlorite, especially in samples in which the latter is interstratified with vermiculite (Jackson and others, 1954). An intergradient chlorite-vermiculite-montmorillonite is defined (Dixon and Jackson, 1959) as one of the series of layer silicates having

heat stability of the 14Å spacing, cation exchange capacity, and specific surface intermediate between those of the nonexpansible mineral chlorite and the two expansible and collapsible minerals, vermiculite and montmorillonite.

Kaolinite and halloysite were successfully differentiated from an aluminous chlorite having typical heat stability in a synthetic mixture (Hashimoto and Jackson, 1960), the sample being heated at 500°C to destroy the kaolinite or halloysite structure and subsequently the resulting residue being dissolved with NaOH. Although heating at 500°C may weaken or destroy the 7Å chlorite peak, the chlorite structure is left largely intact with only partial dehydration of the octahedral layer (Brindley and Robinson, 1951, p. 193), thereby accounting for this mineral not dissolving in the NaOH.

The objective of the present study was to apply the differential dissolution analysis (D.D.A.) method of Hashimoto and Jackson (1960) to soil clays having strong 7 and 14Å diffraction peaks and to check the results against the other available criteria for vermiculite, kaolinite, and chlorite in the presence of illite and small amounts of other minerals.

SOILS EMPLOYED

Elliott soils are moderately well drained Brunizem soils, formed under prairie vegetation in southeastern Wisconsin and northeastern Illinois, on dolomitic silty clay loam till. The B₃-C horizon sample analyzed was taken by the second author, in Vermillion Co., Illinois (section 35, T 21 N, R 14 W), on the Clay Conference field trip, October 8, 1956.

Morley soils are moderately well drained, weak Gray-Brown Podzolic soils formed on dolomitic clayey till with or without a loess covering. Morley soils are associated with Elliott soils. The B-horizon sample analyzed was taken by the second author in May, 1956, from Racine Co., Wisconsin.

Maquoketa shale, an upper Ordovician formation containing dolomite and pyrite, is considered to be a possible component of the parent material of the Morley soils. The sample was taken by the second author in May, 1956, from Dodge Co., Wisconsin.

Buchanan stony loam is an imperfectly drained soil formed from extremely acid colluvial parent material occurring along 3–15 percent slopes of the mountains, associated with Lycoming soil, according to the Soil Survey of Union Co., Pennsylvania, 1946 (Jeffries and Yearick, 1949). The B₁-horizon sample analyzed came from the 15 to 25 in. depth (Union Co., no. 152), and was kindly furnished by Dr. C. D. Jeffries especially for tests of the D.D.A. and specific surface methods. The pH value was listed as 4.5.

Susquehanna soils are Red Podzolic soils which occur in the Atlantic and Gulf Coastal Plains and are derived chiefly from thick beds of acid heavy clays. The sample analyzed is of Susquehanna fine sandy loam, clayey B horizon, 6–22 in., from 2 miles west of Montgomery, Alabama, supplied by Dr. J. R. Taylor, Jr., for the collection of the second author.

Davidson soils include reddish lateritic soils derived from dark basic rocks. The Davidson clay sample of Alabama analyzed was kindly furnished, at the suggestion of Dr. G. D. Smith, by Dr. R. W. Pearson, as one similar to the one reported by Pearson and Ensminger (1949), to be employed as a reference sample for testing new methods. It was used in analyses previously reported (Jackson and others, 1954).

PROCEDURES

Clay fractions of the six soil samples were dispersed, fractionated and prepared for x-ray diffraction analysis with a General Electric diffractometer, according to published procedures (Jackson, 1956, pp. 31-188). Free oxides (Mehra and Jackson, 1960) were removed before fractionation from all samples except the Elliott and Maquoketa shale, and were removed from the latter separated clay fractions prior to elemental analysis. Mono-interlayer specific surface measurements were made by the method of Kinter and Diamond (1958) as modified by Mehra and Jackson (1959). Elemental analyses were made by the semimicrochemical method of Corey, Jackson, Whittig, and Swindale (Jackson, 1958, pp. 278-300).

The kaolinite content is based on the difference between elements dissolved by 0.5 N NaOH (2.5-min boiling) from samples heated at 500°C (4 hr) and 110°C (Hashimoto and Jackson, 1960). Elements dissolved from samples dried at 110°C (110°/NaOH) are considered to arise from amorphous material. Sample sizes ranged from 40 to 100 mg and the ratio of sample to NaOH solution was kept at 1 mg per ml. The clays were dried from benzene to facilitate trituration of aggregates into complete dispersion prior to the brief boiling in NaOH. Mica composition is based on 10 percent K₂O equals 100 percent (Jackson, 1956, pp. 543-544; Mehra and Jackson, 1959) after allowance for the small amounts of feldspar present based on x-ray diffraction data. Expansible mineral percent (vermiculite plus montmorillonite) is based on 775 m² per g equals 100 percent (Mehra and Jackson, 1959). Individual percentages for vermiculite and montmorillonite are based on specific surface and relative x-ray intensities of the two minerals. Allocation of elements corresponding to the percentages of amorphous material, feldspar by x-ray intensities, mica, vermiculite, montmorillonite, kaolinite, gibbsite (from Al₂O₃ dissolved by 110°/NaOH), and anatase from percent TiO₂ were made using chemical formulas (Jackson, 1956, p. 539) with changes of octahedral cations on a chemical equivalent basis to adjust for isomorphous substitution as indicated by the elemental analyses.

Differences between water loss in the 400°C-to-ignition range and those calculated for the several minerals determined independently were used for chlorite content. Silica remaining after subtraction of elements attributed to chlorite was allocated to quartz. Final adjustments in percent composition of each mineral were made to obtain the optimum agreement with all data employed.

ILLITE-RICH SOIL CLAYS

The coarse clay fractions of the Elliott and Morley soils contain illite (mica), vermiculite, chlorite, feldspar and quartz as indicated by x-ray diffractometer tracings (Fig. 1). The presence of high-charge vermiculite is shown by decrease in intensity of 14Å peak and increase in intensity of 10Å peak as a result of K saturation at room temperature in the presence of glycerol. The effect of Sr saturation and glycerol solvation in expanding vermiculite of Morley soil to greater than 14Å (comparable to Walker, 1957) differentiates it from chlorite and emphasizes the importance of saturation with the appropriate cation in diffraction analysis. Identical x-ray results were obtained from Sr saturation by washing as by boiling, indicating an ordinary exchange reaction. The expansion to 18Å of some vermiculite as a result of the Sr saturation and glycerol solvation indicates ease of 14-to-18Å conversion and hence transitional properties of clay vermiculite toward montmorillonite. The Sr ion is larger and presumably less hydrated than Mg or Ca and thus may seat in the silica hexagonal network on one side of the interspace, thus decreasing the effective layer charge (producing montmorillonite-like properties). An increase in expansion of the vermiculite of this clay was also produced by the 500°C preheating and subsequent NaOH treatment followed by Mg saturation and solvation (Fig. 1). This heating-extraction treatment thus appears also to exert a charge-lowering effect on vermiculite. A similar increase in 18Å material at the expense of 14Å material has been reported for Crosby soil clay (Dixon and Jackson, 1959).

The gain in intensity of the 14Å peak on heating at 500°C or more confirms the presence of chlorite (solid curves, Fig. 1). Loss of the 7Å peak with apparently simultaneous gain of the 14Å peak on heating at 500°C may indicate a high-iron chlorite, but partial hydration of the octahedral layer due to weathering (Droste, 1956) could also account for the 500°C instability of the chlorite. The total iron oxide content and the high 7Å versus 14Å peak intensities for the two soil clays indicate the presence of iron chlorite (Brindley and Robinson, 1951, p. 187).

The shoulder produced by Elliott and Morley clays between 10 and 14Å indicates reinforcement of interstratified 14Å diffraction peaks and collapse of some 14Å spaces to 10Å, resulting from heating at 500°C and above (Fig. 1, solid curves). This shoulder at about 12.6Å was produced by interstratified material, in which some layers collapsed at the temperatures at which chlorite layers reinforce the 14Å peak (500° and 550° or 600°C). A chlorite-vermiculite (or montmorillonite) interstratified system would have collapsed on heating below 500°C. This material, which produces the shoulder, resisted collapse on heating to higher temperatures than intergrades in Davidson (Fig. 2) and other soils (Dixon and Jackson, 1960) where appreciable collapse is evident on heating below 500°C. Heat stability of the precursor of the 12.6Å material at 300° and 400°C suggests a different origin from the intergrades studied in more southern soils which presumably formed

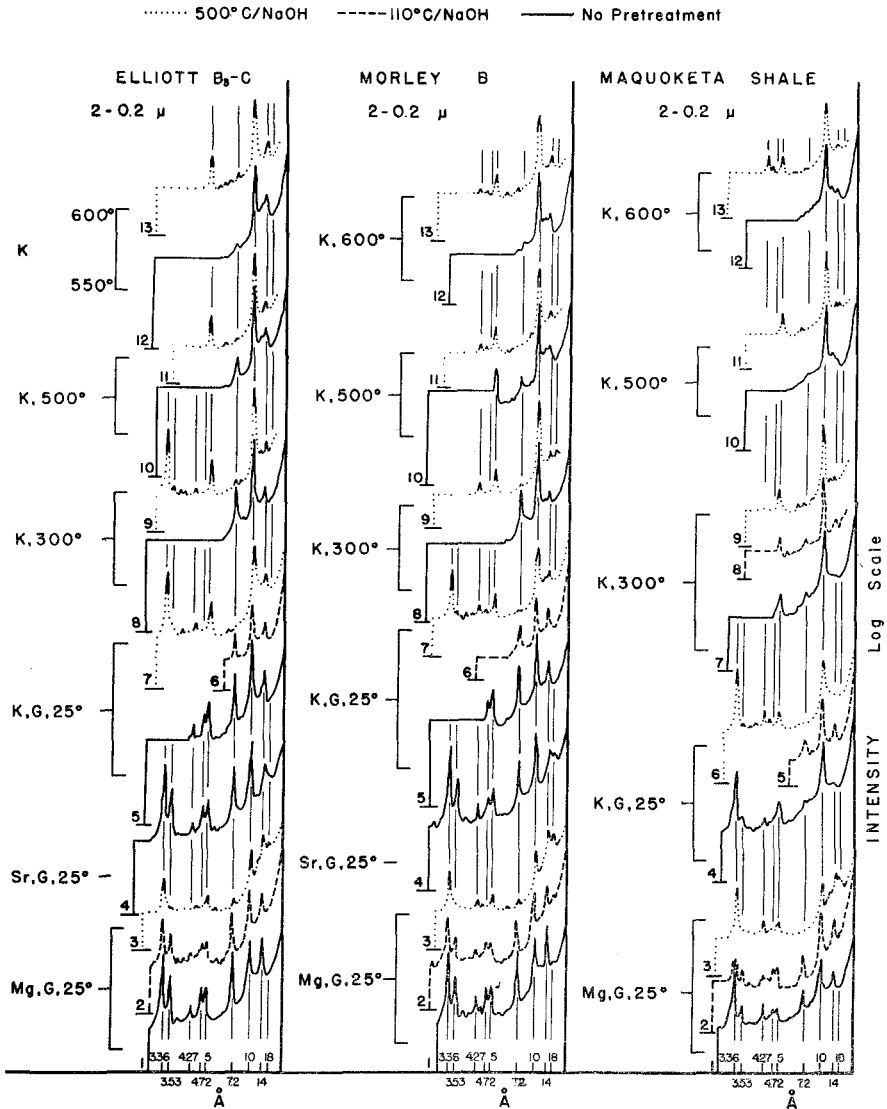


FIGURE 1.—X-Ray diffraction patterns of illite-rich coarse clays from soils, showing the effects of heating and subsequent 0.5 N NaOH boiling treatments to remove amorphous material, kaolinitic residue and interlayer aluminum.

by the precipitation of gibbsitic interlayers in expansible minerals. Also, intergrades formed in soils which have been reported (Dixon and Jackson, 1960) have not shown reinforcement in the 10–14 Å region analogous to that produced by chlorites reported here (Fig. 1). The weathering of chlorite to collapsible material (Whittig and Jackson, 1955; Droste, 1956) is a possible explanation for the 500°C unstable material interstratified in the chloritic components of these samples. The formation of collapsible and expansible material from chlorite would seem especially likely on weathering of hydrated chlorite suggested by Bradley (1955, p. 100).

Some collapse of typical chlorite on heating has been reported (Martin, 1955; Brindley and Robinson, 1951, p. 195); collapse of sedimentary chlorite also has been reported (Bradley, 1954, p. 328). Martin (1955, pp. 135, 138) reports collapse on heating (550°C for 0.5 hr) from 15.2 to 14.0 Å for a sample of thuringite (44–2 μ) and from 14.5 to 14.0 Å for a 2 μ sample of clinochlore. Martin (1955, p. 138) reports a cation exchange capacity of 32 meq per 100 g and 25 mg of glycol per g sorbed for the 44 to 2 μ sample of thuringite. Martin (1955, p. 138) reported glycol sorption values ranging from 6 to 106 mg per g and cation exchange values from 3.8 to 47 meq per 100 g for various size fractions of chlorites. The instances reported of some collapse of chlorites on heating together with the rather high cation exchange and glycerol sorption values reported by Martin (1955) indicate the presence of collapsible layers interstratified with chlorite layers even in samples selected for properties typical of chlorite. Bradley (1954, p. 328) reports collapse of some layers of a chlorite from a sediment owing to incomplete filling of the brucite layer.

The evidence for collapsible layers in chlorite samples indicates that collapse of chloritic material such as reported here may be inherent in the chlorite prior to weathering. This interpretation is supported by the presence of similar material in silt fractions of Maquoketa shale which reinforced in the 10–14 Å region on heating at 500° and 600°C (Dixon, 1958, p. 36). This sample of Maquoketa shale contained 38 percent CaCO₃ equivalent, suggesting that weathering of silicates was not extensive. Interstratification of chlorite with more collapsible silicates is consistent with their genesis in hydrothermal deposits in which chlorites and montmorillonite often form in adjacent zones that grade into one another (Grim, 1953, p. 325).

The decrease in intensity of the 7 Å peak on heating at 500°C might suggest the presence of considerable kaolinite in Elliott and Morley samples. The dissolution data (Table 1) indicate the presence of about 5 percent kaolinite. Such data tend to be somewhat high owing to the solubility of other minerals in NaOH. Allocation of water loss above 400°C (Table 2) to the quantity of hydroxyl in vermiculite indicated by specific surface data and in mica on the basis of K₂O leaves only about enough water to account for the chlorite indicated by the 14 Å and 4.72 Å peaks which persist on heating at 400°C. Thus, if kaolinite is present it is only about 2 percent (Table 3)—within the error of the water analysis. This D.D.A. result is in great contrast

TABLE 1.—KAOLINITE AND EXPANSIBLE LAYER SILICATE DETERMINATIONS BASED ON DIFFERENTIAL DISSOLUTION IN NaOH AND SPECIFIC SURFACE BY A MONO-INTERLAYER GLYCEROL METHOD

Soil Clay, 2-0.2 micron Fraction	0.5 N NaOH Boiling Applied after Heating						Kaolinite		Planar Specific Surface	
	110°C [400°C]			500°C			on	on	as	as
	SiO ₂ %	Al ₂ O ₃ %	Fe ₂ O ₃ %	SiO ₂ %	Al ₂ O ₃ %	Fe ₂ O ₃ %	SiO ₂ %	Al ₂ O ₃ %	m ² /g	% Vr + % Mt
Elliott	2.38	0.27	n.d.	4.13 (1.75)	1.72 (1.45)		3.8	3.7	130	17
Morley	1.98	0.33	n.d.	4.13 (2.15)	2.00 (1.67)	0.93	4.6	4.2	118	15
Maquoketa	1.51	0.13	n.d.	4.65 (3.14)	1.57 (1.44)		6.8	3.6	58	8
Susquehanna	3.14	1.94	0.20	23.5 (20.4)	18.8 (16.9)	0.73	43.9	42.8	105	14
Davidson	3.73 [4.01]	5.35 [5.53]	0.33 [0.47]	24.0 (20.3)	22.2 (16.8)	0.54	43.6	42.4	132	17
Buchanan	2.87	1.95	0.30	9.81 (6.94)	7.42 (5.47)	0.54	14.9	13.8	135	17

* Vr—vermiculite; Mt—montmorillonite. () = Net for kaolinite.

to that for truly kaolinitic clays (next section). Allocation of the remainder of the elements on the basis of mineral formulas (Jackson, 1956, p. 539) is in good agreement with the allocation based on water loss above 400°C for these two clays. Cation exchange values for the coarse clay fractions of Elliott, 25.2 meq per 100 g, and Morley, 25.0 meq per 100 g, measured as Ca replaced by NH₄, also support the mineralogical interpretations. The Maquoketa coarse clay contains the same minerals, in different proportions, as the Elliott and Morley coarse clays (Fig. 1, Table 3).

TABLE 2.—ELEMENTAL ANALYSES OF COARSE CLAY FRACTIONS OF SOME SOILS SELECTED FOR VARIABLE CONTENTS OF ILLITE, VERMICULITE, CHLORITE AND KAOLINITE

Sample	Heating Weight Loss, Percent			Elemental Analysis Data, Percent							
	110-400°C	400-540°C	540°C-ign.	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	K ₂ O	Na ₂ O	TiO ₂
	Illite-rich clays										
Elliott	2.01	2.95	2.02	49.6	22.1	6.28	3.19	0.18	6.17	0.40	1.24
Morley	2.33	2.95	1.32	52.6	20.6	5.71	2.98	0.18	5.98	0.36	1.13
Maquoketa	1.92	2.54	2.02	56.9	18.2	4.83	2.22	0.10	5.58	0.36	0.97
	Kaolinite-rich clays										
Susquehanna	3.43	7.06	1.62	44.0	31.2	3.05	0.49	0.04	1.83	0.24	2.24
Davidson	4.33	7.49	2.21	40.4	34.4	2.06	0.45	0.02	1.91	0.28	2.13
Buchanan	3.75	3.94	1.69	51.6	26.1	3.55	1.15	0.07	4.10	0.35	1.18

TABLE 3.—MINERAL AND AMORPHOUS MATERIAL[†] CONTENT OF SOIL COARSE CLAY FRACTIONS, 2-0.2 μ, BASED ON ALLOCATION OF COMBINED DATA

Sample	Composition Expressed in Percent*									
	Fl	Qr	Mi	Chl	Vm	Mt	Kl	Gb	Am	An
Elliott	6	10	50	8	17	0	2	0	3	1
Morley	6	15	50	8	10	5	2	0	2	1
Maquoketa	10	20	45	6	8	0	2	0	2	1
Buchanan	5	12	41	7	17	0	14	0	5	1
Susquehanna	5	5	18	(5)	12	8	40	0	5	2
Davidson	5	4	19	(5)	15	2	40	8	4**	2

* Fl—feldspars; Qr—quartz; Mi—micas; Chl—chlorites; Vm—vermiculites; Mt—montmorillonite series; Kl—kaolinite plus halloysite; Gb—gibbsite; Am—amorphous SiO₂ and Al₂O₃; An—anatase; reported percentages are on samples from which free Fe₂O₃ was extracted.

** Amorphous SiO₂ only. () Largely intergradient chlorite-vermiculite.

KAOLINITE-RICH CLAYS

The Buchanan coarse clay (Fig. 2) is an example of a mixture including 17 percent vermiculite and 14 percent kaolinite which would give excellent criteria for the presence of much chlorite on the basis of x-ray spacings of 14Å and 7Å without heat treatments. Jeffries and Yearick (1949) reported chlorite, "hydrous mica" and quartz as the constituents of the total clay fraction. Dr. C. D. Jeffries kindly furnished this important reference soil sample for further study and standardization of methods. The data presented here for the coarse clay show 7 percent chlorite, 40 percent mica, and 12 percent quartz (Table 3), together with the 31 percent of vermiculite and kaolinite (mentioned above).

Heating the K-saturated Buchanan sample at 300°C produced collapse of the 14Å spacing to 10Å (Fig. 2), thus showing the presence of typical vermiculite. Loss of the 7Å peak on heating at 500°C without reinforcement of the 14Å peak suggests that kaolinite rather than chlorite produced most of the 7Å peak. The dissolution data (Table 1) show that indeed 14 percent kaolinite-halloysite is present. Sharpening of the 10Å peak produced by samples heated after 110°C/NaOH relative to the sample which was not NaOH-treated indicates the presence of a small amount of intergradient chlorite-vermiculite (Dixon and Jackson, 1959).

The Susquehanna and Davidson soil clay samples are each shown to be high in kaolinite by the presence of large 7.2 and 3.57Å peaks and the absence of 4.7 and 14Å chlorite peaks on heating at 300°C (Fig. 2). Confirmation was obtained in that heating at 500°C destroyed the kaolinite peaks without the chlorite reinforcement at 14Å. Kaolinite and halloysite are included together in the present discussion; the presence of both has been confirmed for the Davidson samples (Jackson and others, 1954, p. 237). The differences in silica and alumina dissolved between 110°/NaOH and 500°/NaOH allocates to about 43 percent kaolinite (Table 1) in each of the two soil clays. Mica is indicated by the 10 and 5Å peaks produced by each sample while Mg saturated and glycerated, and on the basis of K₂O content (Table 2) they each contain about 18 percent mica.

The x-ray diffractometer tracings (Fig. 2) show that Susquehanna coarse clay contains much kaolinite and intergradient chlorite-expandable layer silicate, together with mica, quartz and montmorillonite (Table 3). The D.D.A. value of 40 percent kaolinite in the coarse clay can be compared to 50 percent estimate of kaolinite in the fine clay by Coleman and Jackson (1946) for this same soil sample. Their 20 percent montmorillonite compares to our 8 and 10 percent montmorillonite and vermiculite, respectively, by specific surface. Pearson and Ensminger (1949) reported hydrated alumina, kaolinite, montmorillonite and quartz for the total clay fraction of a different Susquehanna soil sample.

The present data (Table 3) for Davidson coarse clay are consistent with the reports of hydrated alumina, kaolinite and quartz by Pearson and Ensminger

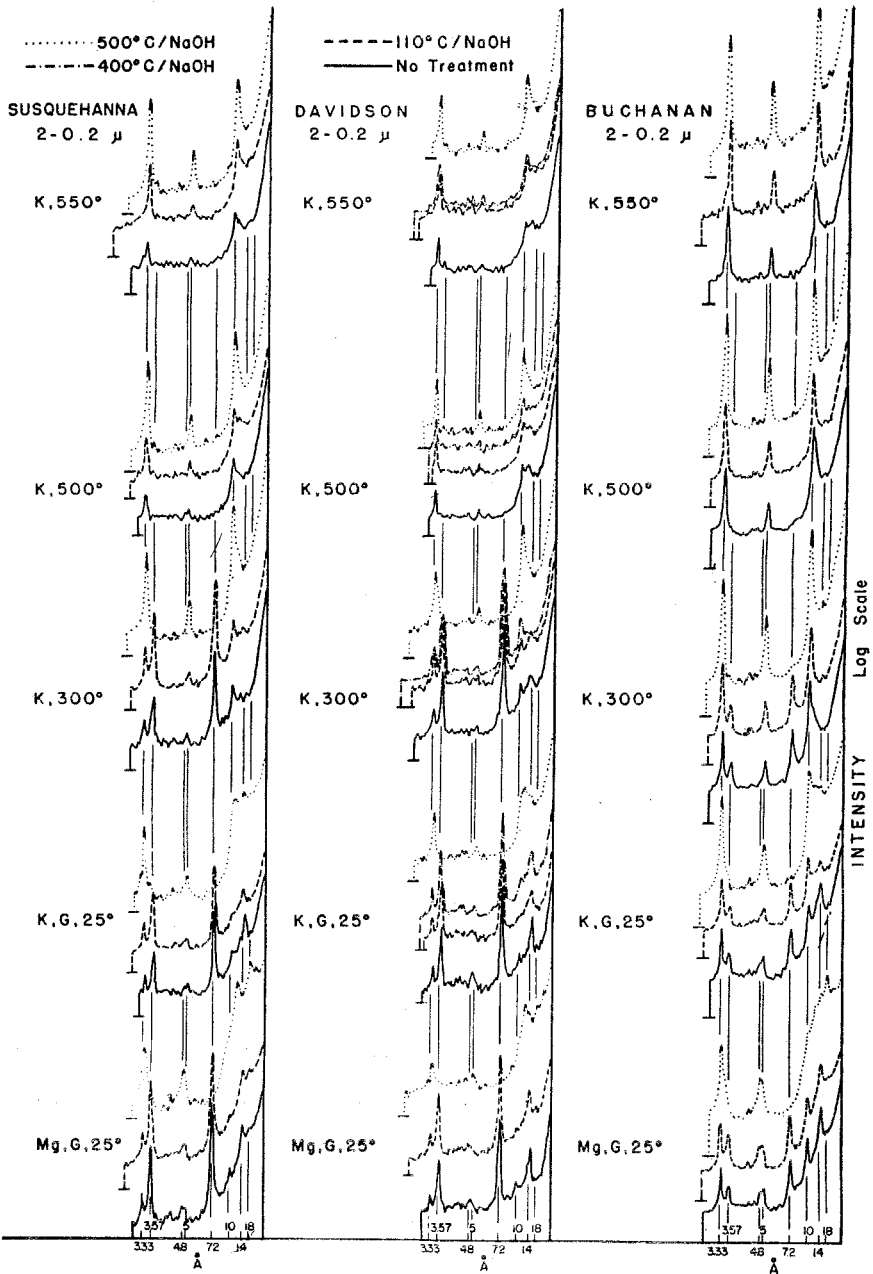


FIGURE 2.—X-Ray diffraction patterns of kaolinite-rich coarse clays from soils, showing the effects of heating and subsequent 0.5 N NaOH boiling treatments to remove amorphous material, kaolinitic residue and interlayer aluminum.

(1949) for a similar sample. The unidentified 14Å component noted by Pearson and Ensminger (1949) and identified as interstratified chlorite with vermiculite or montmorillonite or both (Jackson and others, 1954) is identified here as intergradient chlorite–expansible layer silicate (as defined in the introduction). The D.D.A. results herein, with the 500°/NaOH and to a lesser extent 400°/NaOH treatments removing interlayer Al and permitting collapse to 10Å on heating, marks it as an intergradient chlorite–vermiculite instead of interstratified chlorite–vermiculite. Gibbsite was reported in fractions of the same soil sample of Davidson (Jackson and others, 1954, p. 237) and is indicated here (Table 3) by the larger water loss in the 110° to 400°C range and high percent Al₂O₃ (Table 2).

CONCLUSIONS

Kaolinite is shown to be differentiated quantitatively from chlorite in soil clays by NaOH dissolution of the residue of kaolinite made soluble by heating at 500°C and analysis of the silica and alumina dissolved. Prior dissolution of amorphous material in sodium hydroxide and analysis for silica and alumina are necessary for the allocation to kaolinite. This method of differential dissolution analysis (D.D.A.) applied to the clays from tills derived from the Lake Michigan basin herein greatly increases the chlorite percentage reported, at the expense of kaolinite. A recent paper by the present authors showed that the D.D.A. method gave an increased kaolinite percentage, at the expense of chlorite, in one clay of this basin. At the same time, previously estimated kaolinite contents in soils of the Piedmont and Coastal Plain of southeastern United States are decreased little in the present report, while chlorite, vermiculite and intergradient clays are more quantitatively determined.

Vermiculite is differentiated qualitatively from chlorite and kaolinite by thermo-x-ray diffraction analysis, including the effects of potassium saturation and heating to 300°C. Quantitative analysis for vermiculite is effected by specific surface determinations.

Intergradient chlorite–expansible layer silicates have been differentiated from chlorite through disruption of the interlayer material by heating at 400° or 500°C and dissolving with sodium hydroxide, thus permitting collapse to 10Å on heating. Elemental analysis, including H₂O–OH thermogravimetric analysis, is especially important for accurate quantitative mineralogical analysis of polycomponent clay samples such as soil clays.

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286 EIGHTH NATIONAL CONFERENCE ON CLAYS AND CLAY MINERALS

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