

A CHEMICAL AND MINERALOGICAL CHARACTERIZATION AND
GENESIS STUDY OF THE ZANEIS AND KINGFISHER SOIL
SERIES OF THE REDDISH PRAIRIE GREAT SOIL GROUP

By

Harrell D. Molthan

Bachelor of Science

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Submitted to the faculty of the Graduate School
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Thesis Approved:

Fenton Gray

Thesis Adviser

John V. Reed

Robert Warden

Dean of the Graduate School

452807

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INTRODUCTION

Very little research has been completed in regard to the characterization and genesis of the soils in the United States and practically none on the Reddish Prairie Soils.

A movement is now underway to collect the basic chemical and mineralogical data of the soils of the United States in hopes that a complete characterization study, for at least the more important soils, will be available in the near future. The objective of this report is to furnish this data for two soils of the Reddish Prairie Great Soil Group.

The Reddish Prairie soils were chosen because of the lack of information about these soils and because of their great extent in Oklahoma. Two representative soils of this group, the Zaneis and Kingfisher, were selected in hopes that the information obtained from them could be applied, in a broad sense, to the other closely related soils. They were also selected in order to establish how closely they are related, and if they are different enough to justify separation into two separate series.

The ultimate objective of chemical and mineralogical studies is their correlation with plant growth factors. Therefore, data of this type will be a valuable aid, not only in soil classification, but to all the fields of study involving the soil.

REVIEW OF LITERATURE

The Significance of Clay Mineralogy

It is a well established fact that clay minerals are of fundamental importance in the soil. Since their recognition as crystalline materials (14, 22)¹, many detailed investigations have been conducted to determine the specific types and amounts of clay minerals present in soils. Data of this type have been used in many ways for a better understanding of the inherent properties and characteristics of the soil.

Mineralogical data, such as the type and amount of clay mineral present and its location in the soil profile, have been used as an index for the chemical and physical weathering and the soil formation processes that have taken place in the formation of the soil studied (8, 17, 19, 20, 25, 26). Several investigators (8, 20, 25, 26, 40) used this information, along with a basic chemical and physical characterization, to establish the genesis of grassland soils in studies very similar to the one presented in this report.

Judging from these investigations, there seems to be no set procedure for using this information to the best advantage. Due to the many and varied factors of soil formation, one has to collect as much

¹Figures in paranthesis refer to literature cited.

data as is deemed necessary and from this data, attempt to reconstruct the series of events that led to the formation of the soil in question.

Methods of Analysis

There are several methods currently employed for the identification of clay minerals as to type and amount. Usually, two or more of these methods are employed since no one method seems adequate for soil clays.

A brief review of each of the methods used in this investigation, its scope and limitations, are as follows:

X-ray Analysis

X-ray diffraction serves as a major tool in the characterization of clay minerals. Since its introduction by Hendricks and Fry (14) and Kelley, Dore, and Brown (22), a great deal of time and effort has been spent in developing methods and techniques which would permit more positive identification and characterization of clay minerals with X-ray diffraction.

Early investigators (14, 28, 34) established that each group of minerals have X-ray diffraction lines that are common to that group, although several lines are common to all of the clay minerals.

Whiteside and Marshall (39) used the X-ray diffraction lines shown in TABLE I for positive identification of specific clay minerals. The lines shown for vermiculite were suggested by Gruner (12).

From the values in TABLE I, it can readily be seen where some confusion might result. Further confusion was added when several later investigators found some montmorillonites could give lines as low as 14 or 15 A. This means that montmorillonite, vermiculite, and chlorite

can all give X-ray diffraction maxima at about 14 Å. Chlorite also gives a basal spacing of 7 Å which corresponds to kaolinite.

TABLE I
X-RAY DIFFRACTION LINES OF MINERALS

Common to all minerals	Quartz	Kaolins	Chlorite	Illite	Verm- iculite	Montmor- illonite
<u>angstrom units</u>						
4.4	4.2	7.2	14.0	10.0	14.0	17.4
2.5	3.3	3.6	7.0	5.0	9.2	3.1
				3.3	5.3	

Bradley (1) and MacEwan (28) found that montmorillonite can be separated from vermiculite and chlorite by complete glycerol or glycol solvation of the sample, after first saturating with a divalent cation, prior to X-ray analysis. This treatment results in the glycol, or glycerol, entering the interlayer space and expanding the clay to give an (001) basal spacing of 17 to 17.7 Å.

Kunze (24) found that some montmorillonites will not retain a double layer of glycol in the interlayer space when dried under atmospheric conditions, and that data obtained from such samples can lead to the misinterpretation of mixed layer minerals unless certain precautions are taken in the preparation of samples. He found by allowing the samples to reach equilibrium with an atmosphere of ethylene glycol, after careful glycol solvation, this difficulty was overcome.

Walker (38) found that vermiculite can be readily identified because its X-ray maxima shifts to 10 Å after heating to 300 to 500°C if sufficient precautions are taken against rehydration. This, and

the fact that glycol or glycerol solvation has no effect upon its basal spacing, may be used as an identification procedure (38).

Brindley and Robinson (2) report that heating at 550 to 600°C for one-half hour is sufficient to decompose minerals such as kaolinite, halloysite, and chamosite, but that most true chlorites will not decompose at this temperature. This gives a means for distinguishing chlorites from kaolinite.

Grim, Bradley, and Brown (11) found illite to be essentially unchanged by any of the treatments mentioned here and it still gave strong diffraction patterns at 10, 5, and 3.3 $\overset{\circ}{\text{A}}$.

By use of these procedures, montmorillonite, vermiculite, illite, kaolinite, chlorite and quartz can usually be identified by X-ray diffraction analysis.

Ethylene Glycol Retention

Dyal and Hendricks (6) introduced ethylene glycol retention as a measure of the surface area of clay minerals. They attempted to obtain a mono-molecular layer of glycol over the entire surface, both internal and external, of a hydrogen saturated clay sample. The weight of glycol retained by the total surface area of the clay was calculated and another sample of the H-clay was heated at 600 $\overset{\circ}{\text{C}}$ to collapse all the expanding lattice minerals prior to glycol solvation. From these samples, the external surface area was calculated. The internal surface due to expansion of lattices could then be calculated by difference. This procedure does not give the exact equilibrium point for a mono-molecular layer because the samples continuously lose glycol.

Martin (29) revised this procedure to include a free glycol surface and saturation of the sample with a divalent cation. He found by using the free glycol surface that the surface of the clay sample will reach an equilibrium with the atmosphere of glycol and the weight of the sample will remain almost constant (± 0.3 mgm). Using this method, the equilibrium point is readily obtained with good accuracy.

The results of the two methods can best be shown by inspection of TABLE II.

TABLE II
GLYCOL SORPTION OF REFERENCE CLAY MINERALS

	Kaolinite	Halloysite	Illite	Montmorillonite
	<u>mgm/gm sample.</u>			
Dyal and Hendricks	6.7	20	73	255
Martin	17.0	56	80	283

Martin (29) made no attempt to correlate glycol retention with surface area. He believed the retention value was unique and reproducible for any given clay and saw no need to introduce another possible source of error by attempting to calculate surface area.

In a later paper, Dyal and Hendricks (7) reported glycol retention for a series of montmorillonite and kaolinite mixtures. They found a linear relationship between glycol retention and per cent montmorillonite. They believed that surface area determinations were very indicative of the type and amount of clay present.

Total Non-exchangeable Potassium

Total non-exchangeable potassium has long been used as an index

for the amount of illite, or hydrous mica, present in clays despite a great deal of disagreement as to the percentage K_2O of illite. A great deal of this disagreement is probably due to previous reports where hydrous micas were used for potassium determinations and a considerable amount of variation was found as to total potassium content. Using the present system of dividing hydrous micas into their component parts (17, 18, 29, 32), a good reason for this variation has been established.

It has recently been proposed that the illite percentage of a sample, below which the illite content is unlikely to fall, be calculated on the basis of a 10 per cent K_2O content in illite, the K_2O attributed to feldspar, if present, first being excluded (18). Schmehl and Jackson (35) found the illite content calculated from this value gave very good agreement with other data.

Differential Thermal Analysis

The volatilization of water, whether adsorbed or derived from lattice OH ions, is accompanied by an endothermic reaction. This is reflected by a lag in the temperature of the sample relative to that of an anhydrous reference mineral that is heated at the same rate. On the otherhand, energy is set free as a result of molecular rearrangements. The differential thermal analysis shows the temperatures at which these effects take place. An endothermic dip in the differential thermal curve indicates the loss of water, and exothermic reactions are reflected by rises in the temperature above that of the reference material (21).

Orcel and Gallere (31) first showed the endothermic dips and exothermic peaks of D.T.A. graphs to have value in the investigation of

clay minerals. Since that time a great deal of work has been devoted to the study of D.T.A. as a qualitative and quantitative tool for estimating presence and quantity of clay minerals in mineralogical and soil samples. It has been shown that the temperatures at which these dips and peaks take place are highly characteristic for kaolinite and fairly so for montmorillonite (21). Unfortunately, the thermal curve for hydrous mica is not always clearly distinguishable from that of montmorillonite in soil clays. (21).

At the present time, the D.T.A. of clay minerals has many limitations for both quantitative and qualitative analysis, but it can be a valuable tool when used with other methods. It will detect some minerals present in small amounts that X-ray would miss. It can also give a good quantitative estimation of the kaolinite minerals (27).

Cation Exchange Capacity

Grim (10) states that the cation exchange capacity of clays stems from two sources. One source is the broken bonds created by mechanical loss of molecules around the edges of silica-alumina units. The other source results from unsatisfied charges within the lattice structures due to atomic substitution.

Owing to different particle sizes, amounts of substitution, and interlayer expansion, the clay minerals have been found to have widely varying cation exchange capacities. Some have a wide enough difference to be of aid in a quantitative estimation of the amount of clay minerals in a sample.

Ormsby and Sand (32) found base-exchange determinations made on montmorillonites, illites, and mixed-layer aggregates to be a good method for the analysis of mixed-layer aggregates. They found a

linear relationship between the base-exchange capacity and per cent of montmorillonite in montmorillonite-illite mixtures. The results of this work indicate that the cation exchange capacity of pure illite is 15 m.e./100gm, which is considerably lower than the value found in older literature, and montmorillonite was reported to have a exchange capacity of 95 m.e./100gm. These values were substantiated by Fry singer and Thomas (9) who reported the cation exchange capacity of montmorillonite and illite to be 98 to 109 m.e./100gm, depending on the source, and 16.8 m.e./100gm respectively.

Judging from the results of several investigators (10, 15, 21, 23), the cation exchange capacity of kaolinite will vary from 3 to 15 m.e./100 gm, depending on the size of the particles present.

The exchange capacities of chlorite and vermiculite also exhibit a good deal of variation. Grim (10) gives the base-exchange of chlorite and vermiculite to be 10 to 40 m.e./100gm and 100 to 150 m.e./100gm respectively.

Martin (29) and Ormsby and Sand (32) found the hydrous micas to have varying exchange capacities. They attributed this to the fact that the hydrous micas seem to be intermediate, or interstratified, montmorillonite-illite complexes, and the exchange capacity will depend upon the predominating mineral.

From the data listed here, it is easy to see why cation exchange capacity is not very well adapted to the qualitative determination of clay minerals. It does, however, lend itself very well to a quantitative estimation once the types of clay minerals present are established.

MATERIALS AND METHODS

Soils

The soils used in this investigation are of the Zaneis and Kingfisher soil series. The two series occur extensively on the erosional upland of central and north central Oklahoma. The Kingfisher series occurs a little to the west of the area occupied by the Zaneis series.

Morphologically, the two series appear to be very closely related. Both soils were developed from similar parent materials and under similar environments. In fact, some question has arisen as to whether they should be classified as different soils.

For this study, four Zaneis and two Kingfisher soils were chosen. Two sites, considered to be representative of the series, were selected in each of three counties; Payne, Oklahoma, and Kingfisher. The soil at each site was sampled in detail. The samples were brought to the laboratory, allowed to air dry, and crushed to pass a 60 mesh sieve. The samples were then stored in cardboard ice cream cartons until used.

The profile description and site locations of each soil are as follows:

Zaneis Loam No. 1

Collected by: Fred Dries, Fenton Gray, W.R. Bain, and H.D. Molthan.

Location: 2 mi E and 2 mi N of Stillwater, 950 ft W of N quarter corner or 1782 ft E of NW corner of sec 7, T 19 N, R 3 E.

Site: Normal erosional upland with convex surface and gradient of about 2 per cent. The area is in a native meadow mostly of tall grasses adjoining a cultivated field to the east.

Profile:

A₁ 0-9" Dark brown (7.5YR 4/2, 3/2, m) loam or silt loam; moderate medium granular; friable; porous; roots abundant; pH 5.9; grades to horizon below.

B₁ 9-15" Dark reddish-brown (5YR 3/3) clay loam; weak subangular blocky; friable; permeable; pH 5.6; roots abundant; grades to horizon below.

B₂ 15-24" Dark reddish-brown (5YR 3/3, 2/3, m) heavy clay loam; weak subangular blocky; more clayey than horizon above; moderately friable; permeable; pH 5.7; porous; roots abundant; some fine ferruginous concretions; grades to horizon below.

B₃ 24-36" Reddish-brown (5YR 4/3, 3/3, m) sandy clay loam; mottled with yellowish-red (5YR 4/6); weak subangular blocky; moderately permeable; few ferruginous concretions; roots abundant; pH 6.0.

C 36-46" Reddish-brown sandy clay loam with seams of weathered sandstone; weakly stratified with heavier clayey material; pH 6.2.

D_{3c} 46" + Dark reddish-brown (2.5YR 3/4) well indurated sandstone; very slightly friable; pH 6.4.

The parent material develops in thinly bedded sandy rocks of the Chase group of the Wellington formation which is here composed of alternating bands of sandstone and clays.

Zaneis Loam No. 2

Collected by: Fred Dries, Fenton Gray, Ruel Bain, and H.D. Molthan.

Location: $1\frac{1}{2}$ miles W of Oklahoma State University Dairy Barn, 1520 ft E of SW corner sec 8, T 19 N, R 2 E.

Site: Normal erosional upland with weak convex surface and gradient of 2 percent. Area is a native tall grass prairie.

Profile:

A₁ 0-11" Very dark reddish-gray (5YR 4/2, 3/2, m) loam or silt loam; moderate medium granular; porous; friable; permeable; roots abundant; pH 5.9; grades to

B₁ 11-23" Yellowish-red (5YR 4/8, 3/4, m) clay loam; prismatic to weak subangular blocky; friable; permeable; porous; roots abundant; pH 5.6.

B₂₋₁ 23-32" Yellowish-red (5YR 5/6, 3/4, m) silty clay loam; structure same as above; very hard dry; pH 5.7.

B₂₋₂ 32-42" Dark reddish-brown (5YR 3/4, m, 4/6, crushed dry) silty clay loam; structure same as above; few ferruginous concretions; pH 5.8.

B₃₋₁ 42-48" Yellowish-red (5YR 5/6, 4/6, m) sandy clay; stratas of soft weathered sandstone; more iron concretion than above; weak subangular blocky; pH 5.9.

B₃₋₂ 48-58" Dark red (2.5YR 3/6) weak subangular blocky; iron concretions increase with depth; some stratified layers of rather raw shale and weathered sandstone; pH 6.2.

C 58-78" Dark reddish-brown (2.5YR 4/4, 3/4, m) definitely weathered sandstone and shale; pH 6.5.

The parent material develops from alternating bands of sandstones and shales of the Chase group of the Wellington formation.

Zaneis Loam No. 3

Collected by: H.M. Galloway, Ruel Bain, H.D. Molthan, and Andy Levin.

Location: Due N of Edmond between Sante Fe railroad and section line road in NW SW sec 13, T 14 N, R 3 W and .62 mi S of NW corner sec 13.

Site: Gently sloping erosional upland; surface convex; gradient 2 per cent.

Profile:

- A₁ 0-11" Brown (7.5YR 4/2, 3/2, m) loam or silt loam; moderate medium granular; friable; porous and permeable; pH 6.4; grades to the layer below.
- B₁ 11-16" Brown (7.5YR 4/3, 3/2, m) clay loam; moderate medium granular; friable to firm; crumbly moist; many pores and worm holes; pH 6.1; grades slowly to the layer below.
- B₂₋₁ 16-27" Reddish-brown (6YR 4/3) heavy silty clay loam ; moderate medium subangular blocky; firm; many fine black, soft concretions; porous; clay films on peds; pH 6.2.
- B₂₋₂ 27-39" Reddish-brown (5YR 4/4) light clay; compound moderate coarse prismatic and moderate coarse subangular blocky; very firm; very hard dry; crumbly moist; sides of peds have distinct clay films; crushed, red (3.5YR 4/6); many incipient black concretions; pH 6.4; grades to the layer below.
- B₃ 39-52" Red (2.5YR 4/6) light clay with a few yellowish red mot-tles and rounded black spots which are ferruginous films; moderate medium prismatic and moderate coarse subangular blocky; firm; very hard dry; sides of peds darker than interiors; clay films indistinct; pH 6.5; grades to layer below.

C₁ 52-64" Red (2.5YR 4/8) sandy clay streaked and mottled with light brown (7.5YR 6/4). This is partially weathered intercalated soft thin bedded sandstone and red silty clay beds; pH 6.9; grades to layer below.

C₂ 64-81" Stratified dark reddish-brown coarse sandy loam and slightly hardened red sandstone with occasional lenses of red clay, which has cuboidal breakage; pH 7.0.

Roots are well distributed in A₁, B₁, and B₂₋₁ and are largely between peds in the B₃ and C. This develops in the upper part of the Garber formation which is very close to the contact with the Hennessey formation.

Zaneis Loam No. 4

Collected by: Fenton Gray, Ruel Bain, and Harrell Molthan.

Location: 0.6 mi west of Waterloo store on Oklahoma-Logan county line, 380 feet west of the north quarter corner sec 2, T 14 N, R 3 W.

Site: Gently sloping erosional upland with weak convex surface and gradient of about 2 per cent. Site is in a fence row with a vigorous growth of tall prairie grasses including big and little bluestems, Indian, and switchgrass and with some side oats grama.

Profile:

A₁ 0-8" Dark brown (8.5YR 4/2, 2/2, m) loam; weak fine to medium granular; friable; porous; numerous roots; pH 6.3; grades to the horizon below.

A₃ 8-14" Dark brown (7.5YR 4/3, 3/3, m) light clay loam; moderate medium granular; friable moist; roots slightly less numerous than in A₁; many fine pores; pH 6.2; grades to

B₁ 14-22" Dark reddish-brown (5YR 4/3, 3/3, m) clay loam; weak coarse

granular to weak subangular blocky; faces of peds have colloidal coatings; some roots are present; pH 6.3; abrupt boundary with next layer.

B₂₋₁ 22-33" Dark reddish-brown (5YR 4/4, 3/4, m) heavy clay loam; strong medium subangular blocky and weak coarse prismatic; pronounced colloidal coating on peds; hard when dry; pH 6.3; grades to the layer below.

B₂₋₂ 33-42" Reddish-brown (5YR 5/5, 4/4, m) light sandy clay; moderate medium prismatic and moderate medium subangular blocky; very firm; very hard when dry; slowly permeable; discontinuous dark coatings of peds; a few fine pores; pH 6.5; boundary with next horizon rather abrupt.

C₁ 42-54" Red (2.5YR 5/6, 4/6, m) fine sandy loam with seams of partly weathered sandstone; contains a few small clay balls; friable when moist; pH 6.6; rock color and hardness change slightly in lower part where it grades to

C₂ 54-64" Red (2.5YR 5/8, 4/8, m) weakly consolidated sandstone which is friable when moist and hard when dry; pH 6.7.

Roots are well distributed down to and including the B₂₋₂. In the C₁ and C₂ they are very small in size and few in number and are mostly in seams and cracks.

The parent material forms in weakly consolidated sandstones which are part of the upper Garber formation near the contact with the Hennessey formation.

Kingfisher Silt Loam No. 1

Collected by: Carl Fisher, H.M. Galloway, Ruel Bain, Jim Culver, and Andy Levin.

Location: 2.0 miles E of main corners in city of Kingfisher on highway 33, 1500 ft E of NW corner sec 24, T 16 N, R 7 W.

Site: A native pasture which is dominately of short and mid grasses including buffalo, blue grama, side oats grama, and scattered clumps of little bluestem. Gradient $1\frac{1}{2}$ to 2 per cent, weak convex.

Profile:

A₁ 0-10" Brown (7.5YR 4/3, 3/3, m) silt loam; moderate medium granular; friable; a few fine pores; abundant fine roots; pH 6.5; grades to the layer below.

B₁ 10-14" Reddish-brown (5YR 4/3, 3/3, m) silty clay loam; strong medium subangular blocky; firm; a few holes, worm casts and pores; pH 6.4; grades to the horizon below.

B₂₋₁ 14-21" Reddish-brown (2.5YR 4/4, 3/3, m) silty clay loam; compound moderate medium prismatic and strong coarse subangular blocky; firm; very hard dry; continuous clay films on peds which are about one value darker than interiors; roots are well distributed along the prisms and through the peds; pH 6.4; grades through a 3-inch transition to layer below.

B₂₋₂ 21-32" Reddish-brown (5YR 4/4) silty clay loam with a few small red spots; compound moderate medium prismatic and moderate coarse subangular blocky; very firm; very hard when dry; pH 6.7; sides of ped have dark reddish brown clay film coatings which are one value darker than the interiors; grades to the layer below.

B₃ 32-42" Yellowish-red (5YR 4/6, crushed 5/6) silty clay loam; with structure like horizon above; sides of peds are slightly coated with dark reddish-brown films; a few fine soft black films; very firm;

very hard dry; pH 7.1; grades indistinctly to the layer below.

C₁ 42-52" Red (3.5YR 5/6, 4/6, m) heavy silty clay loam; compound weak medium prismatic and weak blocky; very firm; very hard dry; a few very fine pores; pH 7.7; grades shortly to the layer below.

C₂ 52-65" Red (3.5YR 5/6, 4/6, m) heavy silty clay loam; weak medium blocky; about like the C₁ horizon but has a few white sandy grains in the mass; noncalcareous; grades to layer below; pH 7.8.

C₃ 65-74" Red (3.5YR 5/6) silty clay loam with seams of fine grained noncalcareous sandstone about 1/8 inch thick; a thicker band of this flat-bedded sandstone limits further digging at 74 inches; pH 8.3.

This parent material is formed from the Cedar Hills sandstone member of the Hennessey formation. Root are well distributed in all layers down to and including the C₁. Below this they are sparse and occur only in natural cracks.

Kingfisher Silt Loam No. 2

Collected by: Carl Fisher, H.M. Galloway, Glenn Williams, and Harrell Molthan.

Location: $\frac{1}{2}$ mi W of Okarche in SESENE sec 36, T 13 N, R 8 W.

Site: Is a native pasture mostly of short and mid grasses. Gradient $1\frac{1}{2}$ per cent, weak convex.

Profile:

A₁₋₁ 0-7" Reddish-brown (5YR 4/4, 3/4, m) silt loam; moderate medium granular; friable; permeable; pH 6.4; grades to

A₁₋₂ 7-14" Same as above except that structure is slightly stronger; pH 7.0; this grades through a 2-inch transition to the layer below.

A₃ 14-19" Reddish-brown (5YR 4/4, 3/4, m) heavy silt loam; moderate medium granular; friable; numerous pores and fine root holes; pH 7.2;

grades to the horizon below.

B₂₋₁ 19-28⁰⁰ Reddish-brown (2.5YR 4/5, 3/5, m) silty clay loam; moderate medium subangular blocky; firm; weak clay films on peds which are darker than interiors; pH 7.3; grades to

B₂₋₂ 28-36⁰⁰ Reddish-brown (2.5YR 4/4, 3/4, m) heavy silty clay loam; compound coarse prismatic and weak medium blocky; firm; slowly permeable; pH 7.3; clay films apparent; surfaces darker than interiors; grades to the layer below.

B₃ 36-44⁰⁰ Red (2.5YR 4/6, 3/6, m) silty clay loam much like layer above but without the clay films; a few black spots and partly weathered sandstone fragments; pH 7.3; grades to the layer below.

C₁ 44-56⁰⁰ Red (2.5YR 4/8, 3/8, m) heavy loam between seams of slightly hardened fine grained sandstone; slightly or indistinctly laminated with silty strata; pH 7.8; changes slightly to an easier digging, less hard layer below.

C₂ 56-68⁰⁰ Heavy silty clay loam which is of slightly hardened silty rocks; this rests on a thicker, hardened layer of sandstone at 68 inches which prohibits deeper digging; pH 8.2.

There is good root distribution through the B₂₋₂. Below this the roots are smaller, less abundant, and occupy only the natural cracks between peds.

The parent material is weathered from the Cedar Hills sandstone member of the Hennessey formation.

Methods of Analysis

Chemical Analysis

The chemical analysis of each sample was made as follows: The pH was determined with a Beckman glass electrode pH meter on a 1:1 soil-water paste. The organic matter was determined by the potassium dichromate wet oxidation method of Schollenberger (36). Total nitrogen content was determined by the Kjeldahl method according to Harper (13). Total phosphorus determinations were made by perchloric acid digestion and development of the molybdate color complex according to a procedure by Shelton and Harper (37). The exchangeable cations were determined by a modification of the method of Peach et al. (33). The exchangeable cations were displaced with ammonium acetate and extracted by leaching. The leachate was digested with perchloric acid, brought to volume with distilled water, filtered through a chloride saturated anion exchange column, and the exchangeable cations were determined on a Beckman DU Flame Spectrophotometer with Photomultiplier. The cation exchange capacity was determined by the ammonium saturation and distillation method of Peach et al. (33).

Clay Separation and Fractionation

A 500 gram sample of each major horizon was selected for clay separation and placed in a 1000 ml beaker for removal of organic matter. The organic matter was removed with H_2O_2 as outlined by Jackson, Whittig, and Pennington (19). The sample was washed to remove the end products of oxidation by centrifuging, decanting, and redispersing with distilled water. This operation was repeated until the soil tended to remain in suspension after centrifuging.

The sample was then washed from the centrifuge tubes with a 1 N Na_2CO_3 solution and saturated with sodium for dispersion.

With this method of dispersion, no further treatment was needed throughout the cycle of clay separation. It has also been shown that this method removes small quantities of organic matter not removed by H_2O_2 treatment (19).

The dispersed sample was transferred to an 18 liter carboy and diluted to the 18 liter mark with distilled water. The sample was thoroughly shaken and allowed to stand undisturbed for sedimentation.

At a predetermined time, as calculated by Stokes Law, a siphon was lowered into the suspension to the calculated settling depth of the less than 2 micron size particles, and the suspension containing these particles was siphoned off (21). The carboy was refilled with distilled water, and the separation was repeated until a sufficiently large sample was obtained (about 40 liters).

The suspension containing the less than 2 micron size fraction was collected in a 50 liter carboy, thoroughly mixed, and centrifuged with a Sharples super centrifuge to fractionate the sample into the 2 to 0.2 micron and less than 0.2 micron size fractions.

For this separation, the suspension was conducted directly from the 50 liter carboy into the centrifuge bowl through a siphon tube fitted with a screw clamp to regulate flow. A constant head was maintained by lowering a glass tube, through the stopper containing the siphon tube, into the carboy until it rested about one inch from the bottom of the carboy, and firmly securing the stopper. This procedure proved very satisfactory, as once the desired rate of flow was established it

remained constant throughout the operation.

To effect the separation, an arbitrary centrifuge speed was chosen, and the rate of flow necessary for the desired fractionation calculated from a variation of Stokes Law (39). The density of the less than 0.2 micron particles was assumed to be 2.20.

The material collected on the plastic sleeve of the centrifuge bowl was redispersed and cycled through the centrifuge for more complete separation. This operation was repeated until the liquid passing the centrifuge was clear. With a low concentration of suspensoid, the separation was usually complete after four or five runs through the centrifuge.

The suspension containing the less than 0.2 micron size fraction was transferred from the collecting carboys to five gallon crocks and flocculated with 1 N magnesium acetate. The supernatant liquid was decanted and the flocculated material transferred to a 2000 ml Erlenmyer flask for storage.

The material collected on the centrifuge bowl sleeve after the last centrifugation, the 2 to 0.2 micron fraction, was washed from the sleeve into a 2000 ml Erlenmyer flask with distilled water, flocculated with a 1 N magnesium acetate solution, the supernatant liquid decanted, and stored.

Preparation of Samples for Analysis

A sufficient quantity of each sample for analysis was selected and saturated with magnesium ions as outlined by Kunze and Rich (3). The procedure was modified to omit the alcohol washings and continue washing with distilled water until the sample remained in suspension

after centrifugation¹

Slurry samples for X-ray analysis were drawn off at the prescribed time and stored in sealed polyethylene bottles. The remainder of the sample, after the washing was completed, was dried at 47°C, crushed to pass a 60 mesh sieve, and stored in sealed plastic vials.

Methods of Analysis

The slurry samples for X-ray analysis, along with samples of the dry Mg-clay for D.T.A., were sent to Dr. G. W. Kunze, Agronomy Department, Texas A&M College, College Station, Texas, for analysis.

An approximately 0.2 gram sample of the Mg-clay was weighed on an analytical balance and the ethylene glycol retention value determined by a variation of Martin's procedure (3). The results were reported as mgm ethylene glycol/100gm sample.

Total non-exchangeable potassium was determined by the HF acid dissolution method of Jackson (16), and results are reported as per cent K₂O. Final determinations were made on a Beckman DU Flame Spectrophotometer with Photomultiplier.

For cation exchange capacity determinations, an adaptation of the Gedreiz NH₄Cl method outlined by Kelley (21) was used. A 500 mgm sample of the Mg-clay was placed in a 12 x 100 mm Allihn filter tube with fritted disc, which had been placed on a suction flask, and leached with a 1 N NH₄Cl solution to displace magnesium.

¹Modification suggested by Dr. G.W. Kunze, Agronomy Department, Texas A&M College, College Station, Texas, in a personal communication.

Ten milliliters of the leaching solution were added to the sample in the filter tube, the sample was swirled periodically for 30 to 45 minutes, and then allowed to stand for two hours without suction. At the end of the two hour period gentle suction was applied, and the leachate was collected in a test tube which had been placed inside the suction flask. The sample was further leached with 10 milliliter portions of the leaching solution until a total volume of approximately 50 milliliters of leachate was obtained.

The leachate was transferred to a 50 ml volumetric flask, the volume adjusted to 50 milliliters, and the magnesium content was determined with a Beckman DU Flame Spectrophotometer with Photomultiplier. The results are reported as m.e. Mg/100gm sample.

RESULTS AND DISCUSSION

Particle Size Analysis

The particle size distribution of the Zaneis No. 1 profile (TABLE III) reveals a pronounced clay bulge in the B₂ horizon with a corresponding accumulation of the fine silt fraction, but to a lesser degree. An interesting feature of this profile is the marked increase of coarse silt from the D to the A horizon. This is accompanied by a decrease in the fine sand fraction, while the very fine sand remains fairly constant. This, and the increase of fine silt in the upper half of the profile, suggests either stratification of parent material or the coarse particles present in the parent material are actually aggregates composed of smaller particles and have weathered into their component parts and undergone eluviation and illuviation as the solum has developed.

The particle size analysis of the Zaneis No. 2 profile (TABLE III) indicates a normal soil that has developed in place from uniform parent material. There has apparently been some movement of fine silt along with the eluviation and illuviation of the clay fraction. The coarse fractions decrease in the B horizon, as would be expected, due to the accumulation of clay and show a slight increase in the A horizon due to the loss of clay sized particles.

TABLE III
 PARTICLE SIZE DISTRIBUTION OF THE ZANEIS NO. 1
 AND ZANEIS NO. 2 PROFILES*

		<u>Size Class and Diameter of Particles (in mm.)</u>					
Depth inches	Horizon	Coarse and Med. Sand 2-.25 %	Fine Sand .25-.1 %	Very Fine Sand .1-.05 %	Coarse Silt .05-.02 %	Fine Silt .02-.002 %	Clay <.002 %
<u>Zaneis No. 1</u>							
0-9	A ₁	0.6	12.1	26.0	31.8	11.9	17.6
9-15	B ₁	0.6	11.5	22.5	26.5	13.2	25.7
15-24	B ₂	0.7	9.2	16.5	21.5	16.9	35.0
24-36	B ₃	0.3	15.6	23.5	19.7	10.6	30.3
36-46	C	0.3	21.0	27.3	17.8	9.7	23.9
46 +	D	0.5	61.3	23.9	2.6	3.1	8.6
<u>Zaneis No. 2</u>							
0-11	A ₁	0.3	19.9	28.4	25.5	8.4	17.5
11-23	B ₁	0.2	16.1	24.1	20.7	8.4	30.5
23-32	B ₂₋₁	0.3	14.2	23.3	20.3	9.2	32.7
32-42	B ₂₋₂	0.5	14.9	24.3	22.6	9.9	27.8
42-48	B ₃₋₁	0.8	15.6	23.4	24.7	8.8	26.7
48-58	B ₃₋₂	0.7	17.6	23.3	24.1	9.2	25.1
58-78	C	0.4	18.3	26.6	23.1	10.0	21.6

*Mechanical analysis by S. C. S. laboratory, Lincoln, Nebraska.
 Samples S570kla60-3-(1-6) and S570kla60-4(1-7).

The mechanical analysis of Zaneis No. 3 (TABLE IV) reveals the same type of distribution as was observed in Zaneis No. 1, but is not as pronounced as in Zaneis No. 1. It appears that these two soils have been affected by the same, or quite similar processes.

The particle size distribution of the Zaneis No. 4 profile (TABLE IV) does not conform to a standard pattern. The particles larger than 0.1 mm increase from the C₂ horizon up through the B₃ horizon. The clay content of the C₁ horizon is less than that of the C₂. Both the fine and coarse silt fractions exhibit a continuous decrease from the A₁ horizon to the C₂ horizon, with a large drop occurring between the B₁ and B₂ horizons. This drop in silt is accompanied by a large increase in sand as well as clay. It is difficult, if not impossible, to visualize any soil forming process that could account for this type of distribution. Apparently, there has been a definite stratification of parent material in this profile. The disconformity among horizons expressed here is also evident in the clay mineral composition to be discussed later.

The mechanical analysis of the Kingfisher No. 1 profile (TABLE V) reveals an interesting situation. There is a noticeable disconformity between the upper and lower halves of the profile, with the break occurring between the B₂₋₂ and B₃ horizons. There are more coarse sized particles (>0.02 mm) and more clay in the upper half of the profile, and more fine silt in the lower half. It would appear from this data that there has been stratification of parent material, and the upper portion of this profile did not necessarily weather from the same material that the lower half did.

TABLE IV
 PARTICLE SIZE DISTRIBUTION OF THE ZANEIS NO. 3
 AND ZANEIS NO. 4 PROFILES*

		<u>Size Class and Diameter of Particles (in mm.)</u>					
Depth inches	Horizon	Coarse and Med. Sand	Fine Sand	Very Fine Sand	Coarse Silt	Fine Silt	Clay
		2-.25 %	.25-.1 %	.1-.05 %	.05-.02 %	.02-.002 %	<.002 %
<u>Zaneis No. 3</u>							
0-11	A ₁	2.4	20.6	20.3	29.4	11.7	15.6
11-16	B ₁	2.9	20.0	17.8	21.9	12.7	24.7
16-27	B ₂₋₁	3.1	24.4	19.7	15.9	10.8	26.1
27-39	B ₂₋₂	3.2	26.8	20.0	13.3	8.5	28.2
39-52	B ₃	4.4	29.1	19.5	12.1	6.4	28.5
52-64	C ₁	7.8	34.1	17.0	10.9	4.9	25.3
64-81	C ₂	25.4	31.2	9.0	10.5	7.2	16.7
<u>Zaneis No. 4</u>							
0-8	A ₁	0.8	12.7	21.1	36.0	13.5	15.9
8-14	A ₃	0.7	12.9	20.1	32.9	13.0	20.4
14-22	B ₁	1.0	10.6	18.2	29.1	14.6	26.5
22-33	B ₂₋₁	0.7	13.7	24.4	17.7	10.7	32.8
33-42	B ₂₋₂	0.5	16.5	30.9	17.4	7.6	27.1
42-54	C ₁	0.5	14.7	42.3	15.6	5.6	21.3
54-64	C ₂	0	6.5	54.5	11.4	3.0	24.6

*Mechanical analysis by S.C.S. laboratory, Lincoln, Nebraska.
 Samples S570kla55-2-(1-7) and S570kla55-3-(1-7).

TABLE V
 PARTICLE SIZE DISTRIBUTION OF THE KINGFISHER NO. 1
 AND KINGFISHER NO. 2 PROFILES*

		<u>Size Class and Diameter of Particles (in mm.)</u>					
Depth		Coarse	Fine	Very	Coarse	Fine	Clay
inches	Horizon	Med. Sand	Sand	Fine Sand	Silt	Silt	
		2-.25	.25-.1	.1-.05	.05-.02	.02-.002	<.002
		%	%	%	%	%	%
<u>Kingfisher No. 1</u>							
0-10	A ₁	1.1	1.4	11.3	50.9	15.6	19.7
10-14	B ₁	1.2	1.0	8.3	40.1	14.3	35.1
14-21	B ₂₋₁	1.2	1.1	8.4	35.3	13.7	40.3
21-32	B ₂₋₂	0.8	0.7	5.6	39.7	20.8	32.4
32-42	B ₃	0.2	0.2	1.8	39.0	30.6	28.2
42-52	C ₁	0	0.1	1.9	41.5	31.5	25.0
52-65	C ₂	0	0.1	1.8	42.5	32.8	22.8
65-74	C ₃	0.7	0.5	2.4	44.3	32.9	19.3
<u>Kingfisher No. 2</u>							
0-7	A ₁₋₁	0.3	0.6	22.4	52.7	11.1	12.9
7-14	A ₁₋₂	0.2	0.5	21.1	48.2	12.6	17.4
14-19	A ₃	0.3	0.8	19.6	48.7	11.2	19.4
19-28	B ₂₋₁	0.3	0.5	18.0	42.6	10.8	27.8
28-36	B ₂₋₂	0.2	0.5	17.7	43.2	10.5	27.9
36-44	B ₃	0.2	0.6	18.4	47.6	10.8	23.4
44-56	C ₁	0.1	0.7	19.2	52.0	11.3	16.7
56-68	C ₂	0.9	0.7	19.9	45.5	13.3	19.7

*Mechanical analysis by S. C. S. laboratory, Lincoln, Nebraska.
 Samples S570kla37-10-(1-8) and S570kla37-11-(1-8).

The particle size distribution of the Kingfisher No. 2 profile (TABLE V) is very similar to that of Zaneis No. 2, in that the same overall pattern is exhibited by both soils. From this data it appears that this profile also developed from uniform parent material.

The only noticeable difference between the Kingfisher and Zaneis profiles, from inspection of TABLES III, IV, and V, is that the Kingfisher profiles are higher in silt while the Zaneis profiles are higher in sand. The clay contents of the two series are quite comparable. This would indicate that the Kingfisher profiles developed from a silty parent material, whereas the Zaneis profiles were derived from more sandy parent material.

Chemical Analysis

The chemical analyses are presented in TABLES VI, VII, and VIII. The exchange complex of all six profiles is predominately saturated with Ca and Mg. The K and Na saturation is considerably lower. The two Kingfisher profiles are higher in exchangeable bases, especially Na and Ca, and the exchangeable bases exceed the exchange capacity of most horizons. This is especially true in the parent material of both profiles. The C₃ horizon of Kingfisher No. 1 and the C₂ horizon of Kingfisher No. 2 were treated with dilute HCl and washed with distilled water to remove soluble carbonates prior to analysis for exchangeable cations. This same phenomena has been observed by other investigators in soils from similar environments and was attributed to sparingly soluble carbonates (3).

The pH of the solum appears to be inherited directly from the

TABLE VI

CHEMICAL MEASUREMENTS OF THE ZANEIS NO. 1 AND NO. 2 PROFILES

Horizon	Depth	pH with 1:1 soil-water ratio	% Organic Matter	% Nitrogen	Total phos. p.p.m.	CEC m. e. /100gms.	Exchangeable Cations m. e. /100 gms.			
							Ca	Mg	K	Na
<u>Zaneis No. 1</u>										
A ₁	0-9"	5.90	2.75	0.195	210.4	13.50	7.000	3.618	0.396	0.423
B ₁	9-15"	5.61	2.12	0.153	164.0	18.76	9.150	5.222	0.358	0.423
B ₂	15-24"	5.65	1.57	0.125	122.4	21.52	12.300	6.990	0.460	0.462
B ₃	24-36"	6.00	0.45	0.076	73.6	16.30	8.650	5.962	0.396	0.577
C	36-46"	6.20	0.30	0.075	57.6	12.07	6.525	4.565	0.294	0.596
D	46 + "	6.40	0.00	0.059	25.6	6.93	4.850	3.372	0.205	0.596
<u>Zaneis No. 2</u>										
A ₁	0-11"	5.91	2.50	0.165	168.0	11.49	4.85	3.207	0.460	0.308
B ₁	11-23"	5.60	1.50	0.130	156.0	16.08	6.65	4.359	0.320	0.385
B ₂₋₁	23-32"	5.70	1.22	0.113	137.6	15.91	6.65	5.058	0.358	0.577
B ₂₋₂	32-42"	5.79	0.71	0.086	88.0	14.02	6.05	5.674	0.320	0.577
B ₃₋₁	42-48"	5.90	0.42	0.079	61.6	14.30	6.10	5.674	0.320	0.577
B ₃₋₂	48-58"	6.20	0.17	0.067	45.6	13.51	6.42	6.743	0.320	0.577
C	58-78"	6.45	0.07	0.065	41.6	11.40	5.35	5.140	0.256	0.654

TABLE VII

CHEMICAL MEASUREMENTS OF THE ZANEIS NO. 3 AND ZANEIS NO. 4 PROFILES

Horizon	Depth	pH with 1:1 soil-water ratio	%	%	Total phos. p. p. m.	CEC m. e. /100 gms.	Exchangeable Cations m. e. /100 gms.			
							Organic Matter	Nitrogen	Ca	Mg
<u>Zaneis No. 3</u>										
A ₁	0-11"	6.41	2.72	0.136	137.6	10.67	6.875	3.742	0.499	0.385
B ₁	11-16"	6.10	1.67	0.102	128.0	15.05	8.550	5.222	0.358	0.423
B ₂₋₁	16-27"	6.21	0.97	0.077	131.2	16.97	9.175	6.250	0.396	0.577
B ₂₋₂	27-39"	6.40	0.55	0.057	70.4	17.80	9.175	6.990	0.422	0.462
B ₃	39-52"	6.53	0.37	0.048	70.4	16.83	9.300	6.620	0.460	0.654
C ₁	52-64"	6.88	0.22	0.040	76.8	15.57	7.275	5.592	0.396	0.654
C ₂	64-81 "	7.00	0.07	0.039	83.2	10.50	4.025	3.372	0.473	0.423
<u>Zaneis No. 4</u>										
A ₁	0-8"	6.31	2.85	0.146	163.2	10.77	7.325	3.454	0.665	0.423
A ₃	8-14"	6.22	1.77	0.105	137.6	13.37	6.750	4.354	0.396	0.769
B ₁	14-22"	6.28	1.52	0.099	131.2	16.97	7.450	5.058	0.396	0.923
B ₂₋₁	22-33"	6.28	0.97	0.077	80.0	21.48	10.500	6.456	0.524	0.654
B ₂₋₂	33-42"	6.49	0.72	0.065	70.4	17.24	8.650	5.428	0.243	0.846
C ₁	42-54"	6.55	0.32	0.048	54.4	11.52	5.350	4.359	0.256	0.423
C ₂	54-64"	6.72	0.22	0.042	44.8	12.65	7.200	5.058	0.320	0.846

TABLE VIII

CHEMICAL MEASUREMENTS OF THE KINGFISHER NO. 1 AND KINGFISHER NO. 2 PROFILES

Horizon	Depth	pH with 1:1 soil-water ratio	% Organic Matter	% Nitrogen	Total phos. p.p.m.	CEC m. e./100 gms.	Exchangeable Cations m. e./100 gms.			
							Ca	Mg	K	Na
<u>Kingfisher No. 1</u>										
A ₁	0-10"	6.5	3.50	0.161	340.5	16.08	10.000	5.058	0.921	1.000
B ₁	10-14"	6.4	1.85	0.102	240.0	20.50	9.900	7.812	0.665	0.423
B ₂₋₁	14-21"	6.4	1.60	0.086	176.0	25.86	11.550	9.910	0.742	0.462
B ₂₋₂	21-32"	6.7	0.87	0.057	200.0	20.74	9.700	9.416	0.524	0.615
B ₃	32-42"	7.1	0.52	0.046	270.0	17.62	7.800	7.812	0.410	0.659
C ₁	42-52"	7.7	0.32	0.036	306.0	15.50	10.000	7.812	0.410	1.385
C ₂	52-65"	7.8	0.25	0.034	348.0	15.29	8.050	7.648	0.396	0.659
C ₃	65-74"	8.3	0.12	0.027	336.0	13.03	13.000	5.140	0.396	0.615
<u>Kingfisher No. 2</u>										
A ₁₋₁	0-7"	6.4	2.05	0.102	216.0	9.59	6.030	3.043	0.806	0.423
A ₁₋₂	7-14"	7.0	1.50	0.077	176.0	11.18	7.700	4.030	0.742	0.462
A ₃	14-19"	7.2	1.27	0.067	162.4	11.83	8.300	5.350	0.499	0.320
B ₂₋₁	19-28"	7.3	0.97	0.063	156.0	16.63	10.900	6.250	0.460	0.731
B ₂₋₂	28-36"	7.3	0.85	0.048	156.0	17.27	10.900	6.990	0.524	0.615
B ₃	36-44"	7.3	0.45	0.038	152.0	13.24	8.300	7.155	0.422	1.154
C ₁	44-56"	7.8	0.37	0.029	136.0	8.44	4.350	6.538	0.307	2.308
C ₂	56-68"	8.2	0.15	0.027	196.0	9.09	12.200	9.211	0.294	3.462

parent material. The higher the pH of the parent material the higher the pH observed in the solum. The Zaneis No. 1 and No. 2 are the most acid and the two Kingfisher profiles the most basic, with Zaneis No. 3 and No. 4 intermediate.

All six profiles exhibit an accumulation of organic matter, nitrogen, phosphorus, and to a lesser degree Ca, in the A_1 horizon. Judging from the exchangeable bases and changes in pH from horizon to horizon, the leaching process does not seem to have greatly affected these soils. This would indicate that calcification was the predominate process of soil formation for all six profiles.

In comparing the two series it is observed that the two Kingfisher profiles are higher in phosphorus, especially in the parent material, and are more basic. Organic matter and nitrogen content of the two series are comparable. The overall analysis reveals the two Kingfisher profiles to be of higher inherent fertility than the 4 Zaneis profiles.

Clay Mineralogy

The results of the X-ray analysis and D.T.A. are presented in TABLES IX and X. The interstratified (Is) interpretation of the X-ray analysis is apparently hydrated micas that are poorly ordered and not uniformly hydrated. This is reflected by the low angle scatter ($<8^\circ 2\theta$) of the X-ray spectrographs for the Mg saturated, glycol solvated samples (Figures 1 to 12) and the fact that when the sample was K saturated and heated there was a reduction of scatter and a significant increase in intensity of the $10 \overset{\circ}{\text{A}}$ peak.

The montmorillonite-chlorite (M/C) interstratified interpretation

TABLE IX

IDENTIFYING CRITERIA FOR CLAY MINERALS OF THE ZANEIS NO. 1, NO. 2, AND NO. 3 PROFILES***

Horizon	Coarse clay 2.0-0.2 microns					Fine clay <0.2 microns				
	CEC me./100g.	% K ₂ O	mg/g* Glycol Retent.	X-ray Analysis	D. T. A.	CEC me./100g.	% K ₂ O	mg/g* Glycol Retent.	X-ray Analysis	D. T. A.
					<u>Zaneis No. 1</u>					
A ₁	25	2.5	80	I ₂ , K ₂ , Q ₂ , I _s ?	K, V	65	1.7	192	I ₂ , K ₂ , I _s ? **	K, Hm
B ₂	33	2.8	102	M ₂ , I ₂ , K ₂ , Q ₃	K, M	78	1.3	235	K ₂ , I _s ?	K, Hm
C	24	2.3	75	K ₂ , I ₂ , V/C ₂ , Q ₃	K, V	63	1.6	210	I ₂ , K ₂ , I _s ?	K, Hm
D	29	2.3	88	K ₂ , I ₂ , V/C ₂ , Q ₃	K, V	59	1.9	177	I ₂ , K ₂ , I _s ?	K, Hm
					<u>Zaneis No. 2</u>					
A ₁	22	3.0	71	I ₂ , K ₂ , Q ₃	K, V	56	1.9	188	I ₂ , K ₂ , I _s ?	K, Hm
B ₂	29	2.5	86	I ₂ , K ₂ , Q ₃	K, V	56	1.8	163	I ₂ , K ₂ , I _s ?	K, Hm
C	24	2.7	73	I ₂ , K ₂ , V/C ₂ , Q ₃	K, V	58	2.0	182	I ₂ , K ₂ , I _s ?	K, V
					<u>Zaneis No. 3</u>					
A ₁	30	2.7	82	I ₂ , K ₂ , Q ₃ , V/C?	K, V	61	1.9	187	I ₂ , K ₂ , I _s ?	K, Hm
B ₂	39	2.7	108	I ₂ , K ₂ , Q ₃ , V/C?	K, V	82	1.6	214	I ₂ , K ₂ , I _s ?	K, Hm
C	37	3.1	88	I ₂ , K ₂ , Q ₃ , V/C?	K, V	71	2.0	206	I ₂ , K ₂ , I _s ?	K, Hm

* Pure montmorillonite retains approximately 280 mg/g glycol

** I - Illite, M - Montmorillonite, K - Kaolinite, V - Vermiculite, C - Chlorite, Q - Quartz,
I_s - Interstratified. Subscript refers to relative amount: 1 - greater than 40%, 2 - 10 to
40%, 3 - less than 10%

***X-ray analysis and D.T.A. by Dr. G.W. Kunze, Agronomy Department, Texas A&M College.

TABLE X

IDENTIFYING CRITERIA FOR CLAY MINERALS OF THE ZANEIS NO. 4 AND THE KINGFISHER NO. 1 AND NO.2 PROFILES***

Horizon	Coarse clay 2.0-0.2 microns					Fine clay <0.2 microns				
	CEC me./100g.	% K ₂ O	mg/g* Glycol Retent.	X-ray Analysis	D.T.A.	CEC me./100g.	% K ₂ O	mg/g* Glycol Retent.	X-ray Analysis	D.T.A.
<u>Zaneis No. 4</u>										
A ₁	21	2.7	58	I ₂ ,K ₂ ,Q ₂ ,I _s ?	K, V	70	2.2	209	I ₂ ,K ₂ ,I _s ? **	K, Hm
B ₂	48	3.0	111	M/C ₂ ,I ₂ ,K ₃ ,Q ₃	M, K	106	1.3	137	M/C ₂ ,I ₂ ,K ₃	M, K
C	32	3.1	78	I ₂ ,K ₂ ,V/C ₂ ,Q ₃	K, V	72	1.8	192	I ₂ ,K ₂ ,I _s ?	K, Hm
<u>Kingfisher No. 1</u>										
A ₁	21	4.5	63	I ₂ ,K ₂ ,Q ₂ ,I _s ?	V, K	72	2.7	210	I ₂ ,K ₃ ,I _s ?	V, K
B ₂	29	4.1	77	I ₂ ,K ₂ ,Q ₃ ,V/C _?	V, K	81	2.2	214	I ₂ ,K ₃ ,I _s ?	V, K
C	37	3.7	100	M/C ₂ ,I ₂ ,K ₂ ,Q ₃	M, C, K	68	2.0	166	M/C ₂ ,I ₂ ,K ₂	M, C, K
<u>Kingfisher No. 2</u>										
A ₁	21	4.5	64	I ₁ ,K ₂ ,Q ₂ ,I _s ?	V, K	66	3.5	186	I ₁ ,I _s ?	I, Hm
B ₂	30	4.1	80	I ₁ ,K ₂ ,Q ₃ ,V/C _?	K, V	77	3.1	199	I ₁ ,K ₃ ,I _s ?	I, K
C	31	4.2	92	I ₁ ,K ₂ ,Q ₃ ,V/C _?	V, K, C	65	3.9	198	I ₁ ,K ₃ ,I _s ?	Hm, K

* Pure montmorillonite retains approximately 280 mg/g glycol.

** I - Illite, M - Montmorillonite, K - Kaolinite, V - Vermiculite, C - Chlorite, Q - Quartz, I_s - Interstratified. Subscript refers to relative amount: 1 - greater than 40%, 2 - 10 to 40%, 3 - less than 10%.

*** X-ray analysis and D.T.A. by Dr. G.W. Kunze, Agronomy Department, Texas A&M College.

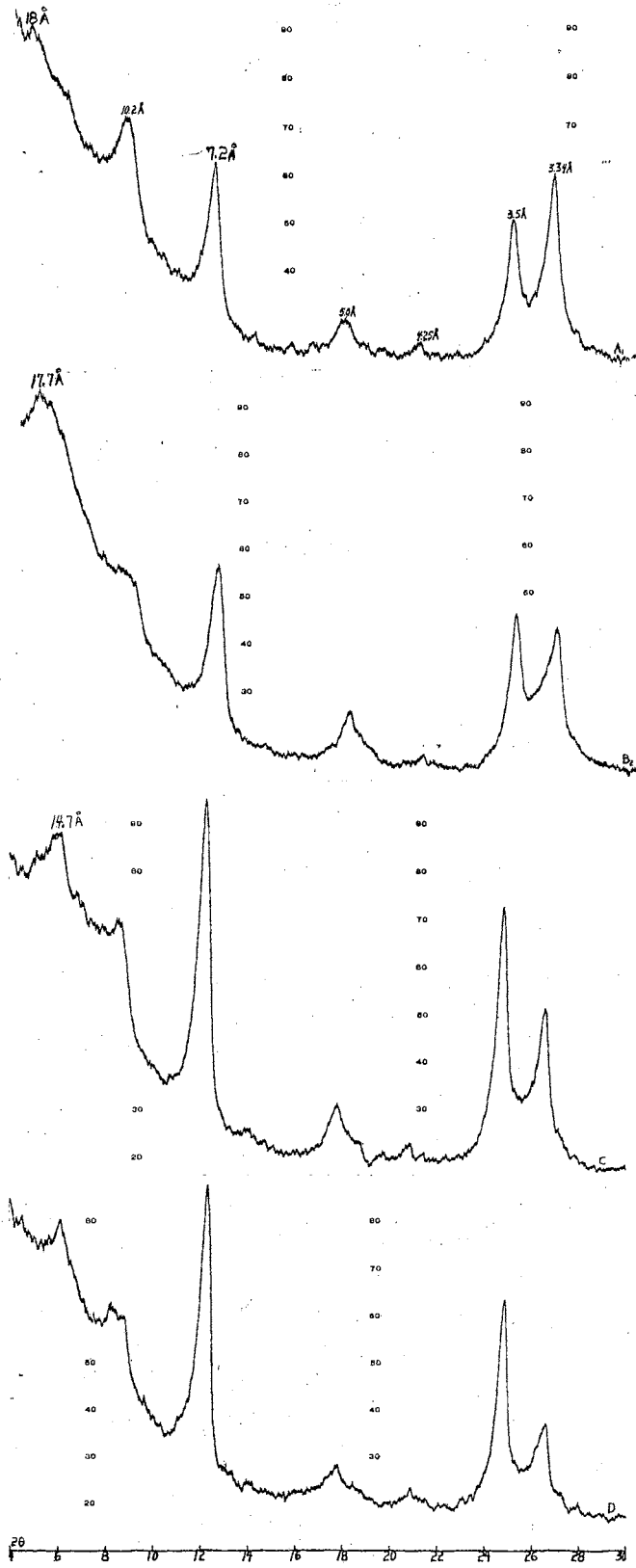


Fig. 1. X-ray spectrographs of Zaneis No. 1 coarse clay fractions

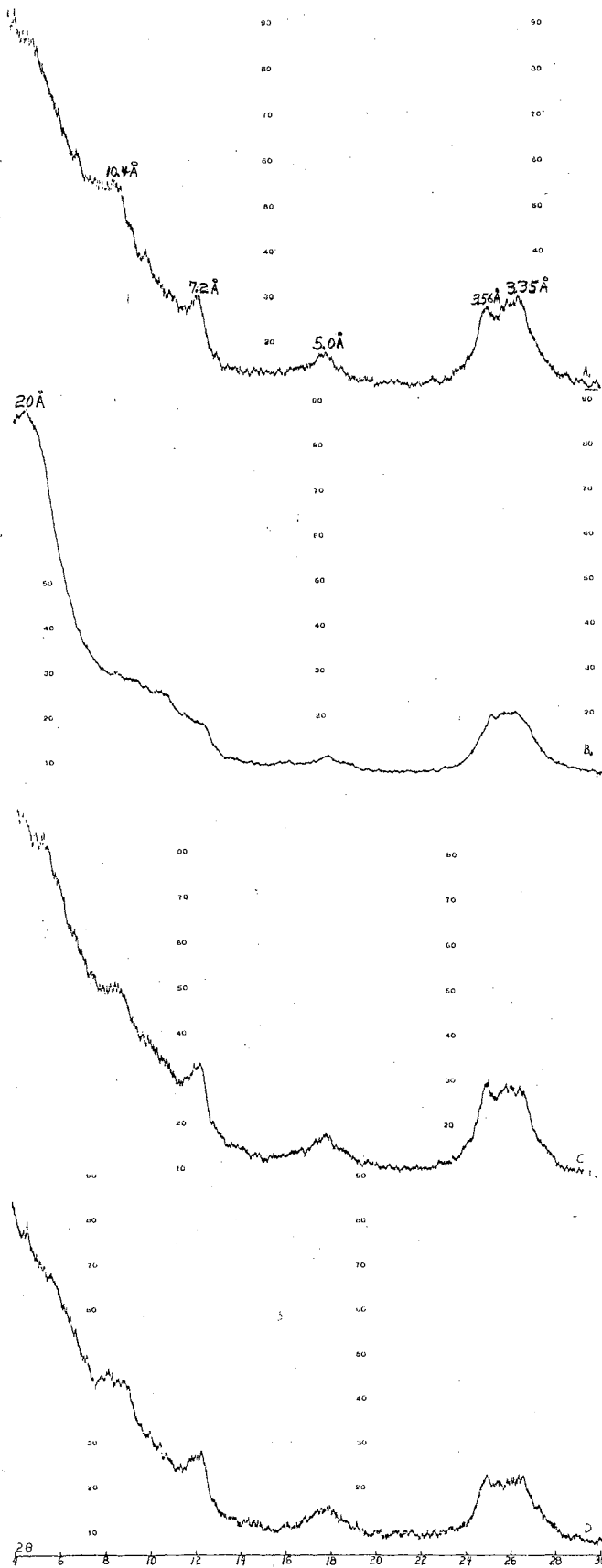


Fig. 2. X-ray spectrographs of Zaneis No. 1 fine clay fractions.

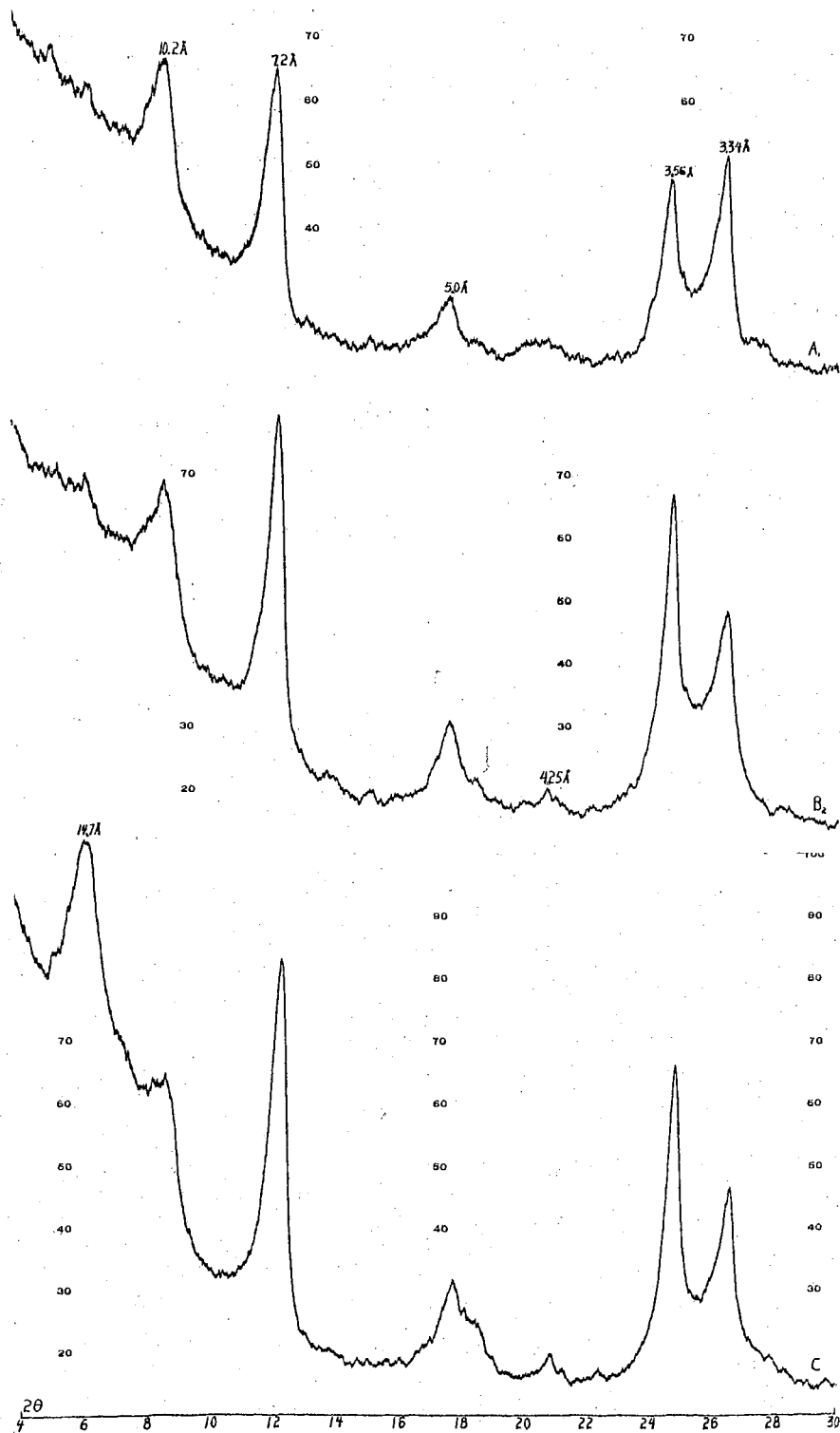


Fig. 3. X-ray spectrographs of Zaneis No. 2 coarse clay fractions.

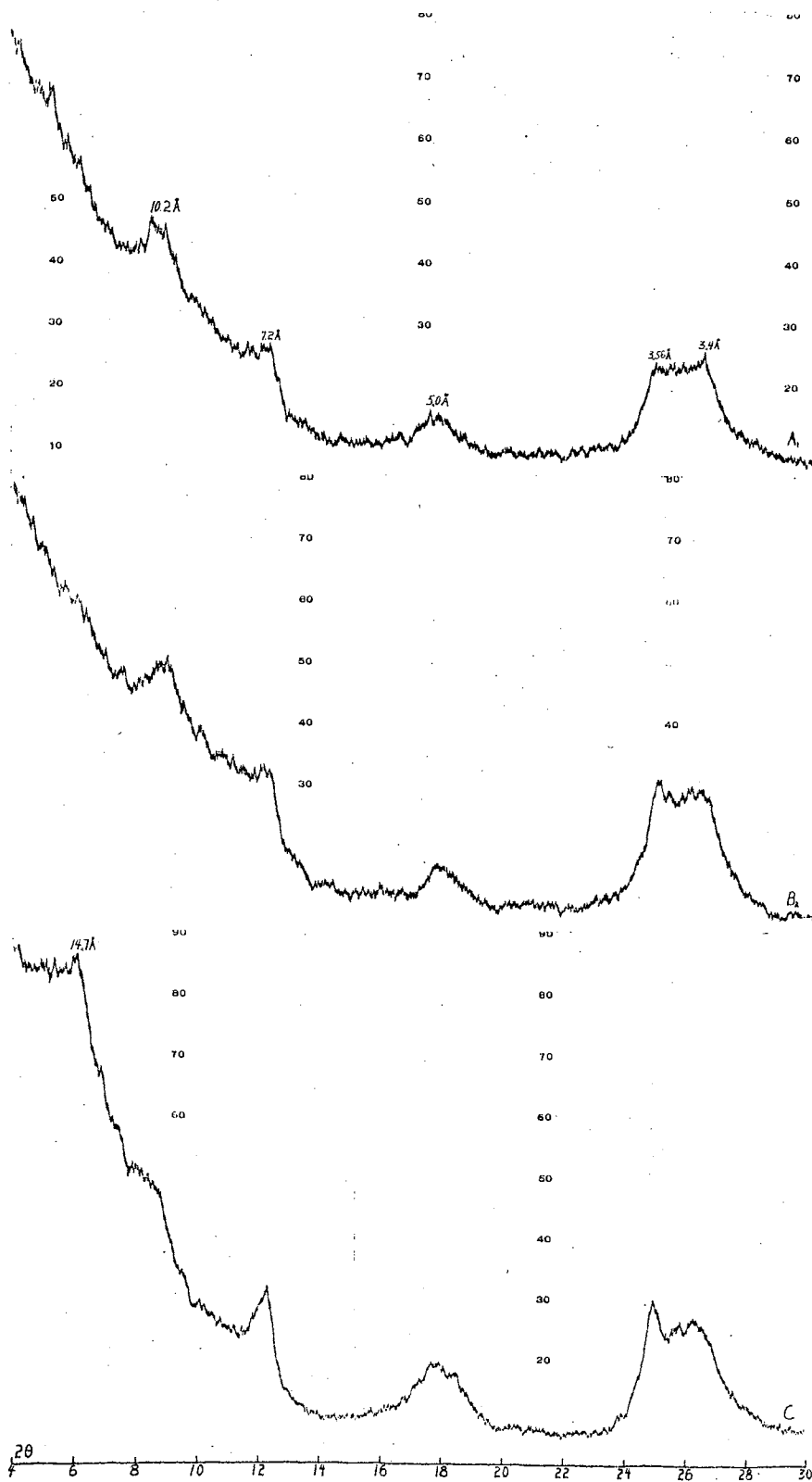


Fig. 4. X-ray spectrographs of Zaneis No. 2 fine clay fractions.

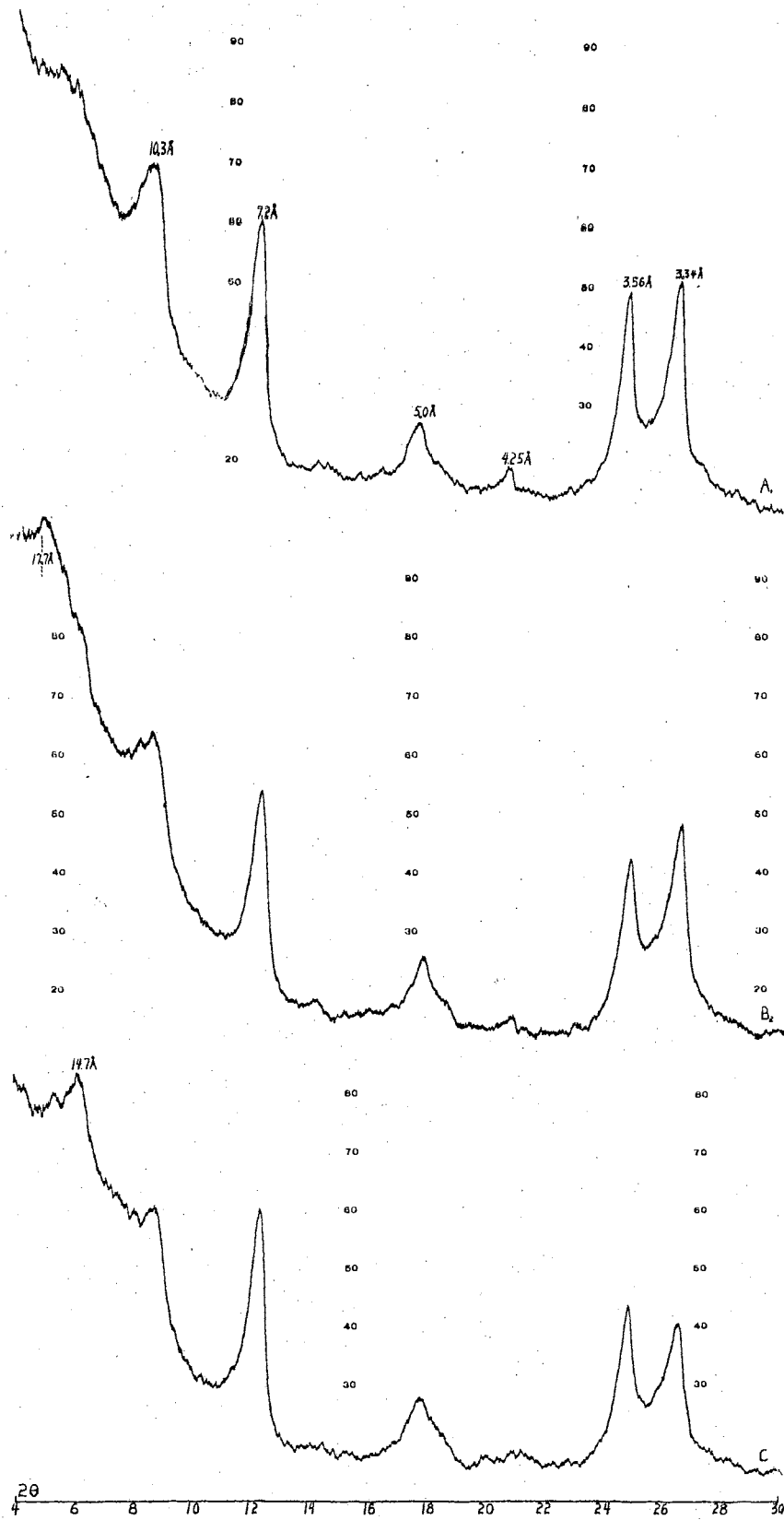


Fig. 5. X-ray spectrographs of Zaneis No. 3 coarse clay fractions.

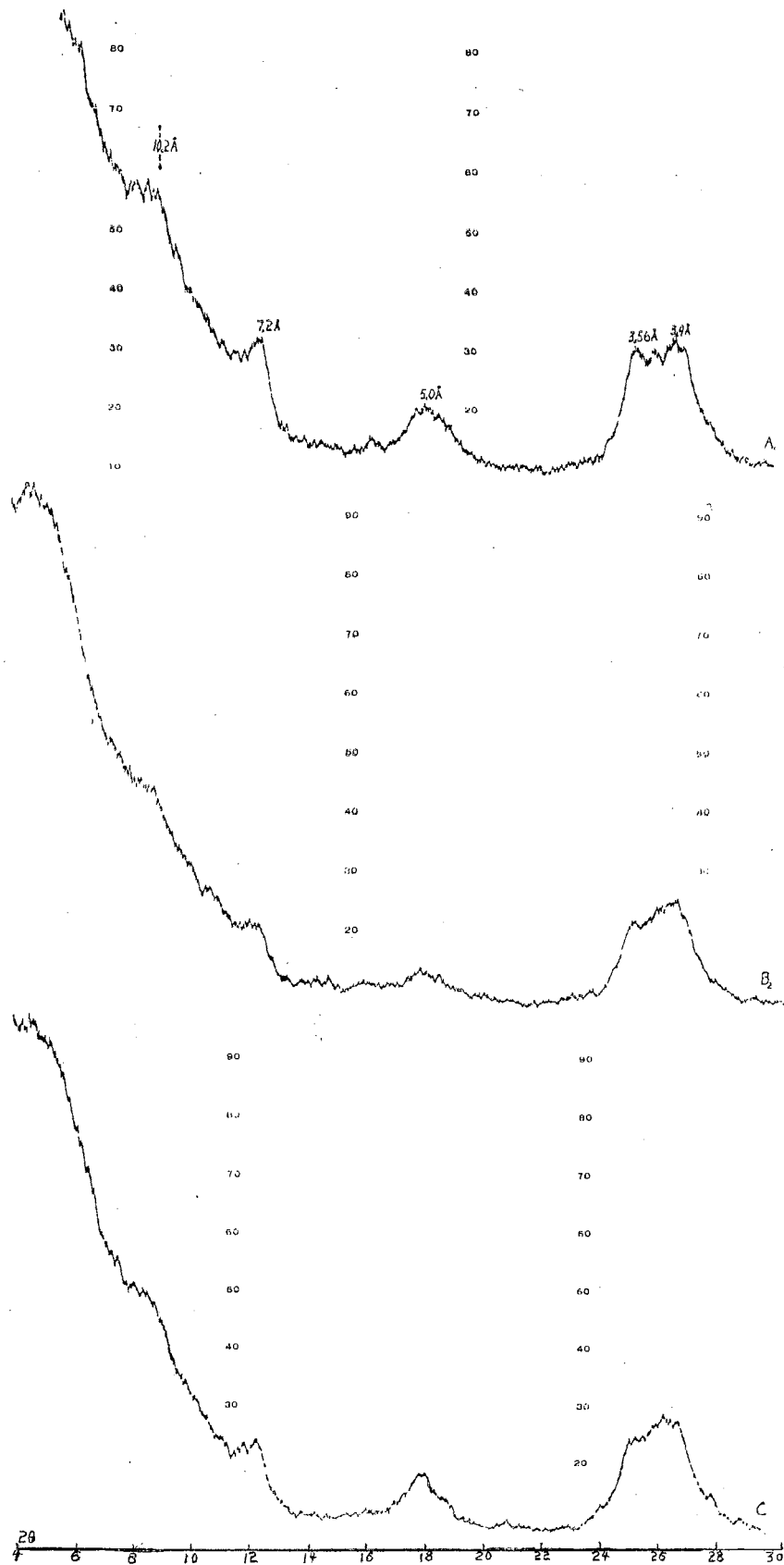


Fig. 6. X-ray spectrographs of Zaneis No. 3 fine clay fractions.

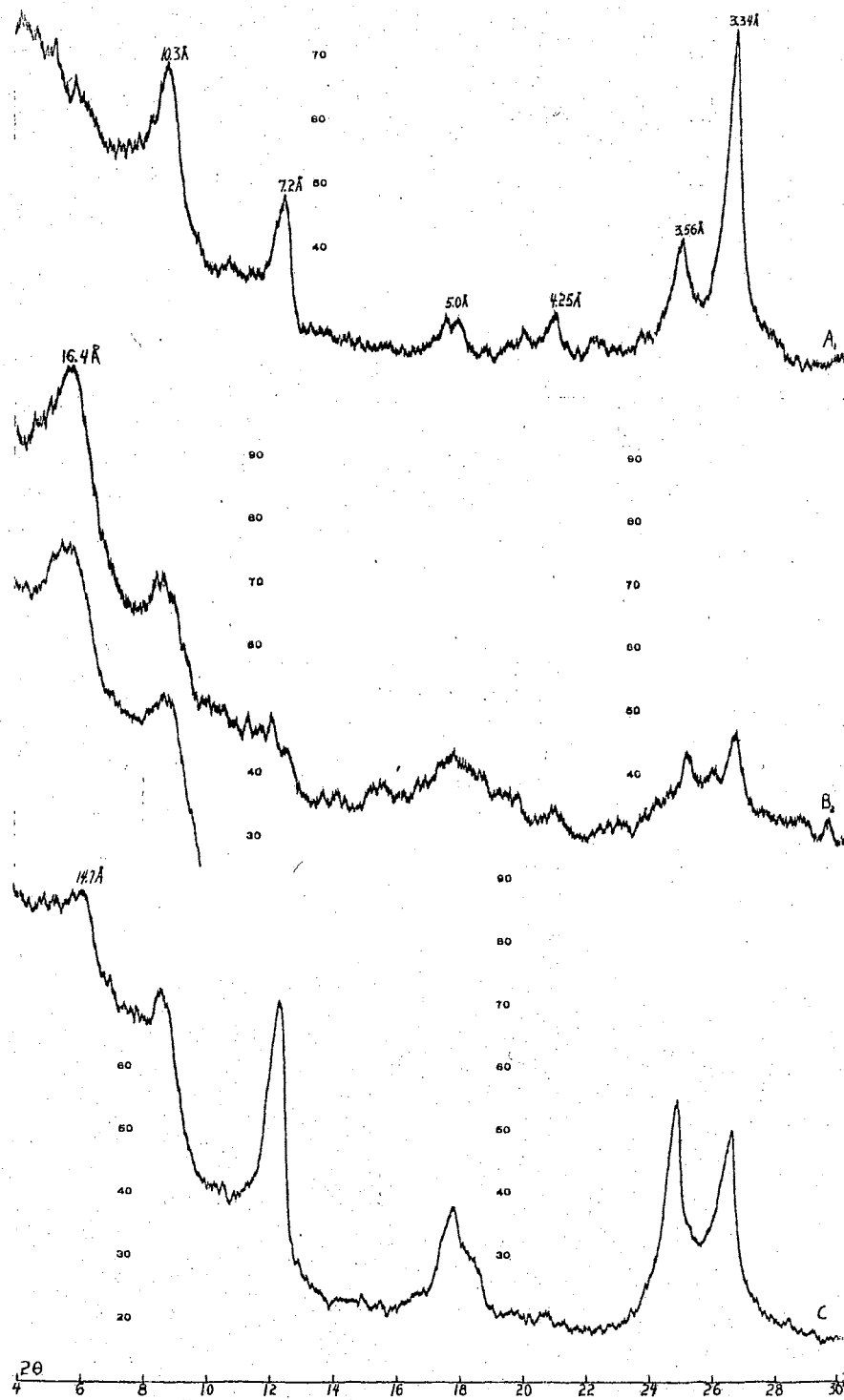


Fig. 7. X-ray spectrographs of Zaneis No. 4 coarse clay fractions.

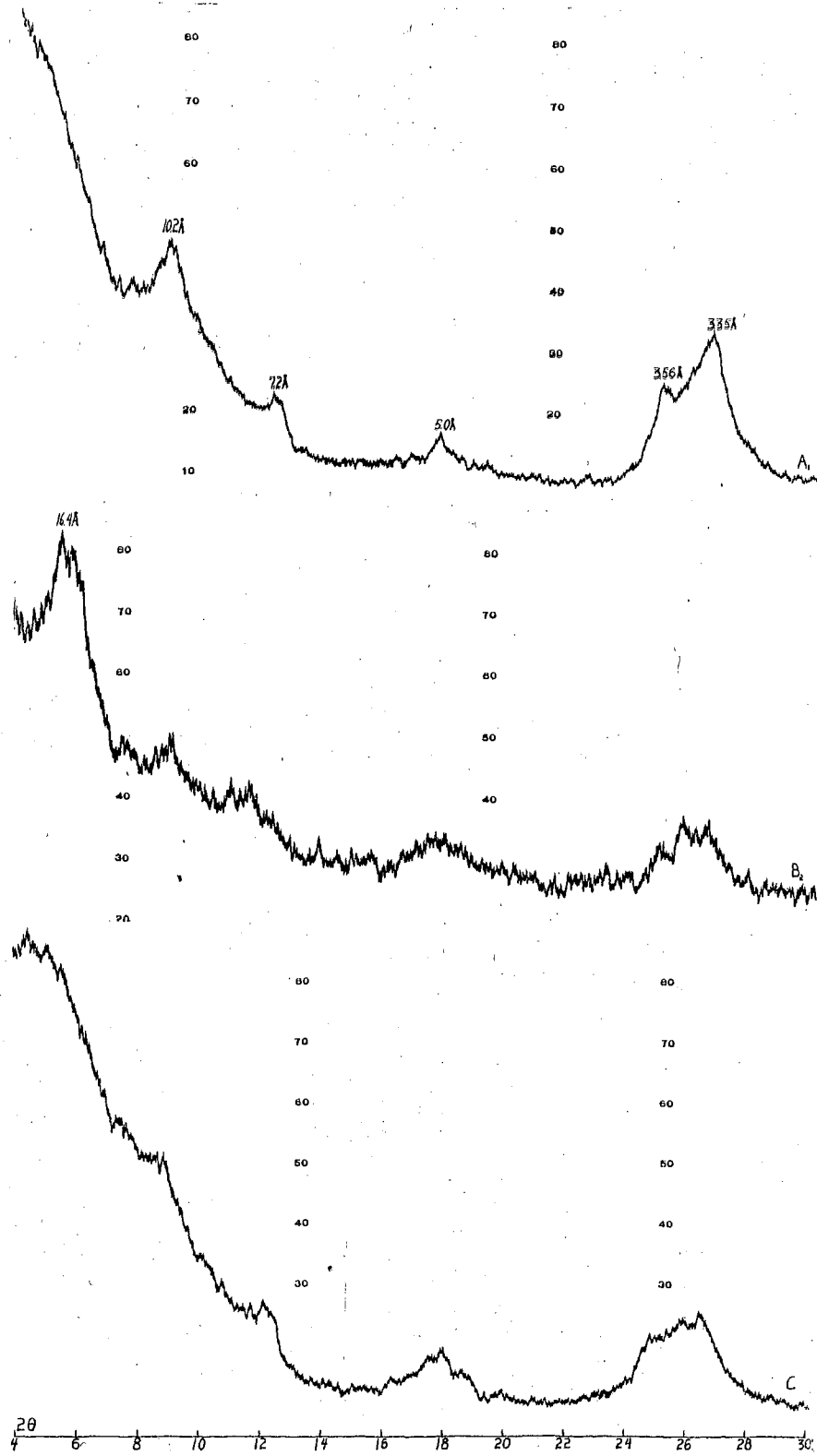


Fig. 8. X-ray spectrographs of Zaneis No. 4 fine clay fractions.

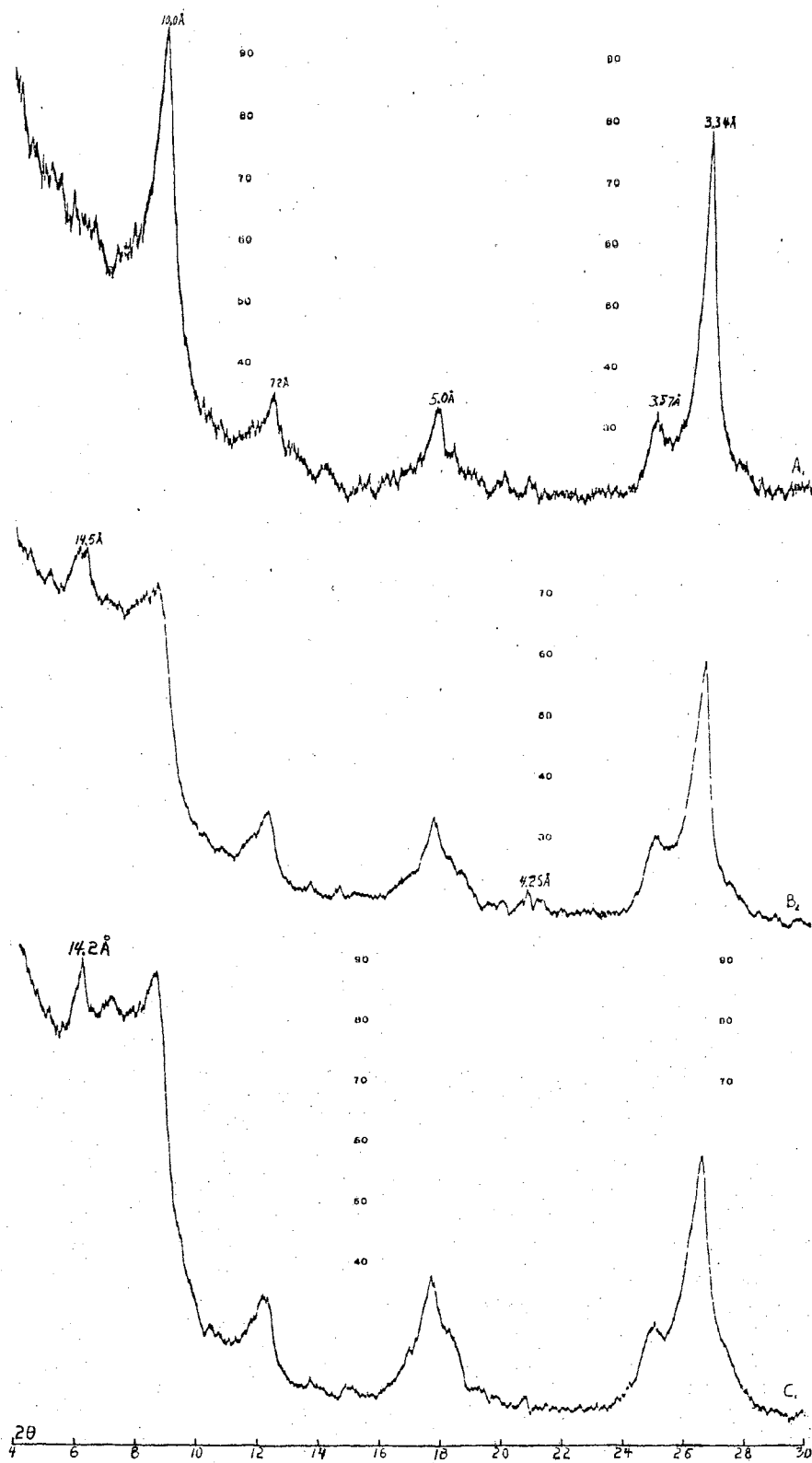


Fig. 9. X-ray spectrographs of Kingfisher No. 2 coarse clay fractions.

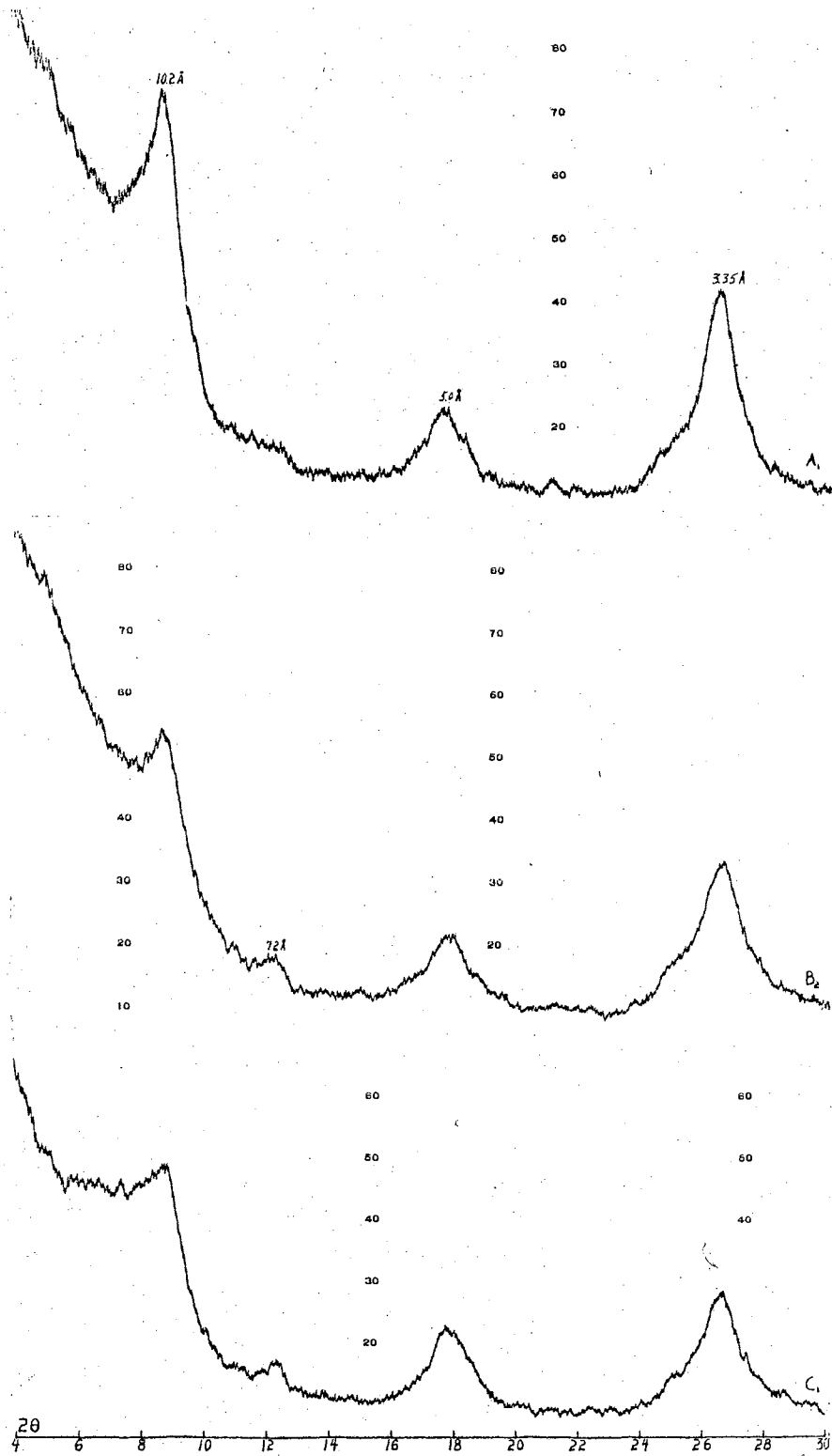


Fig. 10. X-ray spectrographs of Kingfisher No. 2 fine clay fractions.

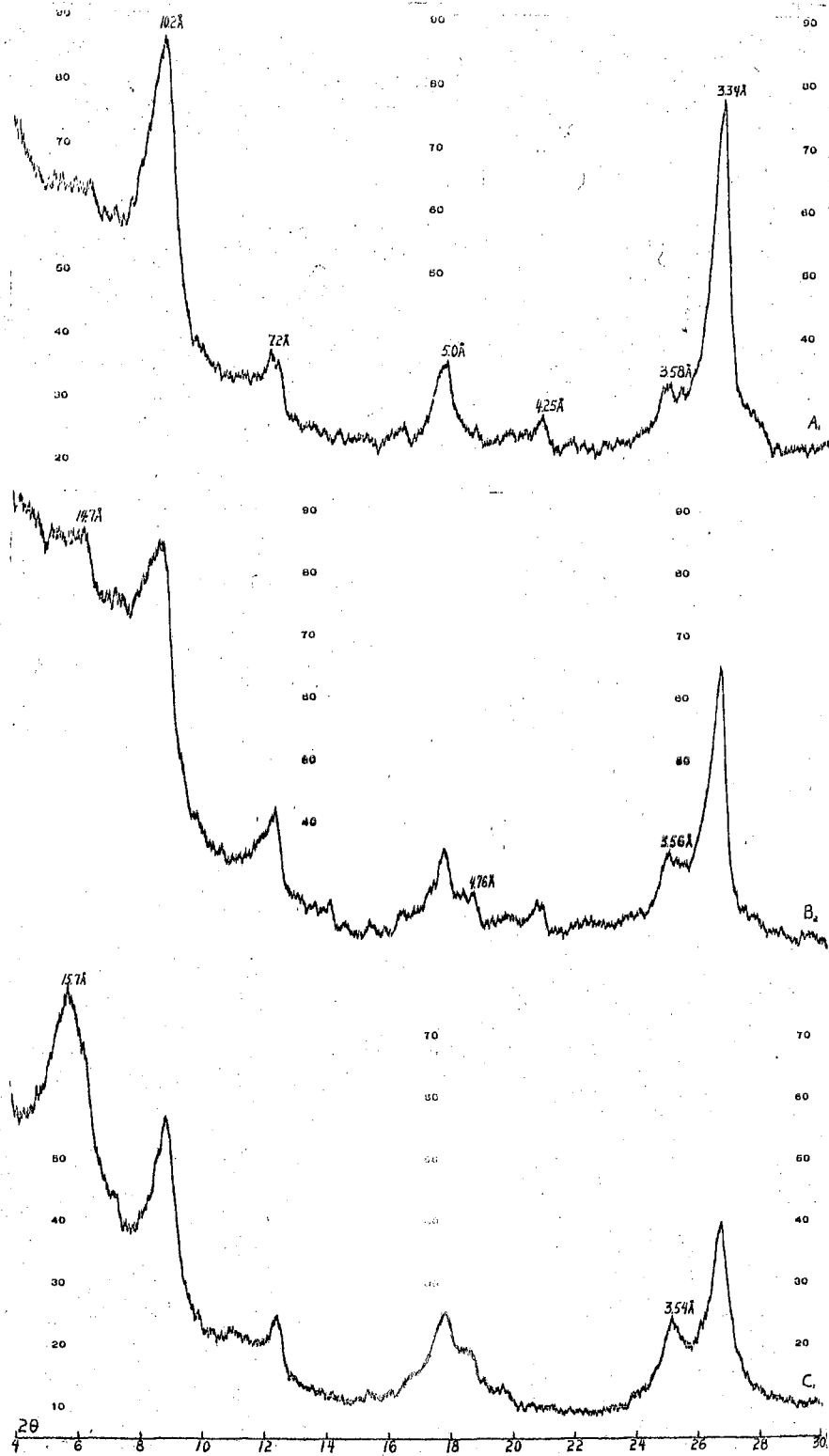


Fig. 11. X-ray spectrographs of Kingfisher No. 1 coarse clay fractions.

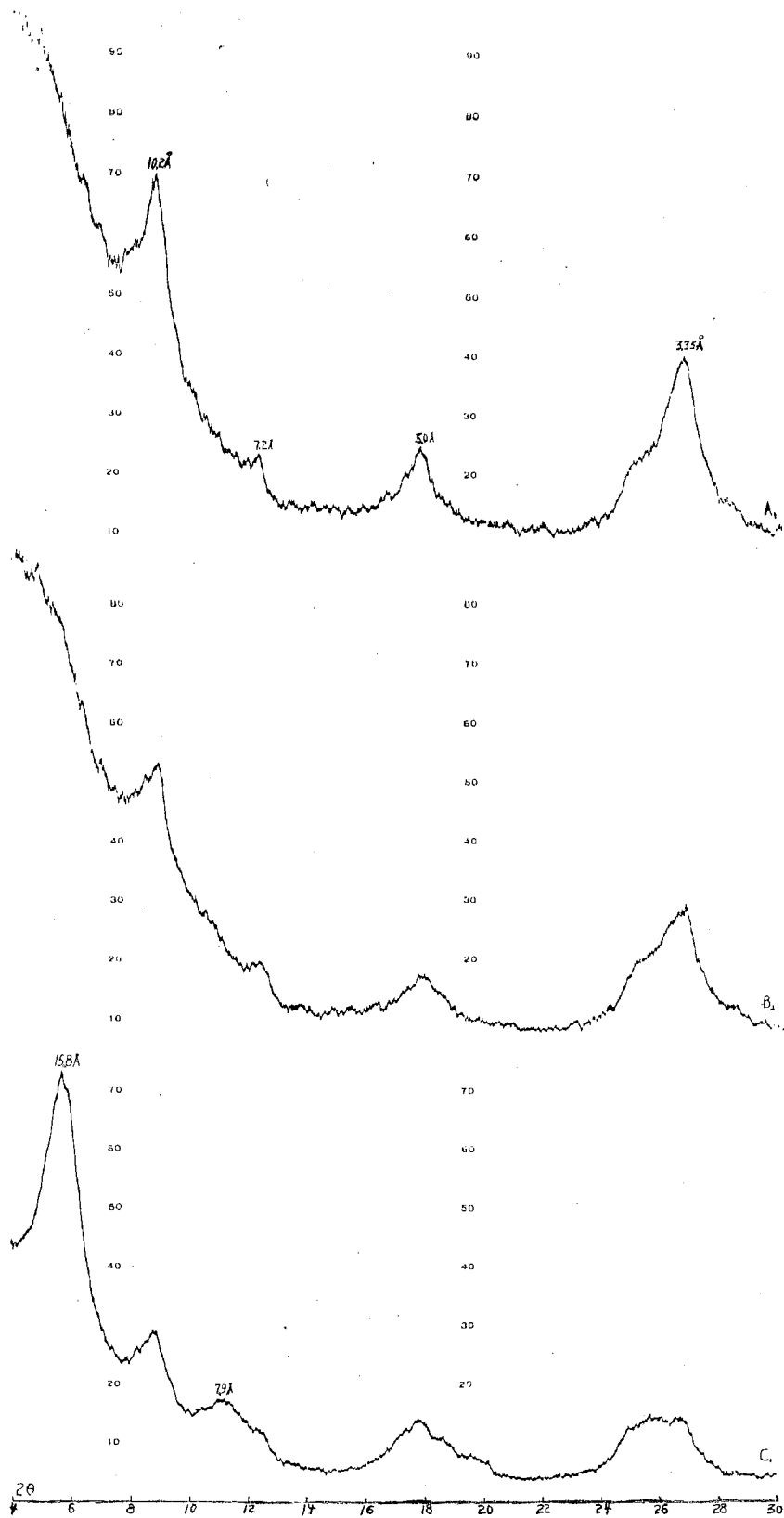


Fig. 12. X-ray spectrographs of Kingfisher No. 1 fine clay fractions.

for the clay fractions of the B horizon of Zaneis No. 4 and C horizon Kingfisher No. 1 was based on a strong peak at approximately 16 \AA for the Mg saturated, glycol solvated samples (Figures 7, 8, 9, and 10) and the fact that this spacing of the K saturated samples, heated to 600°C , did not approach 10 \AA as would be expected if the interstratification had been either montmorillonite-vermiculite or montmorillonite-illite. The D.T.A. of the C horizon of Kingfisher No. 1 was also indicative of chlorite in that it gave a strong high temperature endotherm at 840°C for both size fractions (Figure 13).

The vermiculite-chlorite (V/C) interstratification, common in the coarse clay fractions, was based on a 14.7 \AA peak for the Mg saturated, glycol solvated samples and a reduction in intensity of this peak when the sample was K saturated and heated, along with a 14 \AA peak remaining in the unheated K-sample. Much of this material appears to be vermiculite or vermiculite-like hydrated micas, with lesser amounts of chlorite.

The vermiculite interpretation of the D.T.A., common in the coarse clays, can be montmorillonite as well, since both minerals give the same double low temperature endotherm (Figure 14). The vermiculite interpretation was made in conjunction with the X-ray results.

The hydrated mica interpretation of the D.T.A., common in the fine clays, was based on the low temperature endotherm which though not a double endotherm, was much more intense than commonly associated with illite (Figure 14).

From the cation exchange capacities and ethylene glycol retention

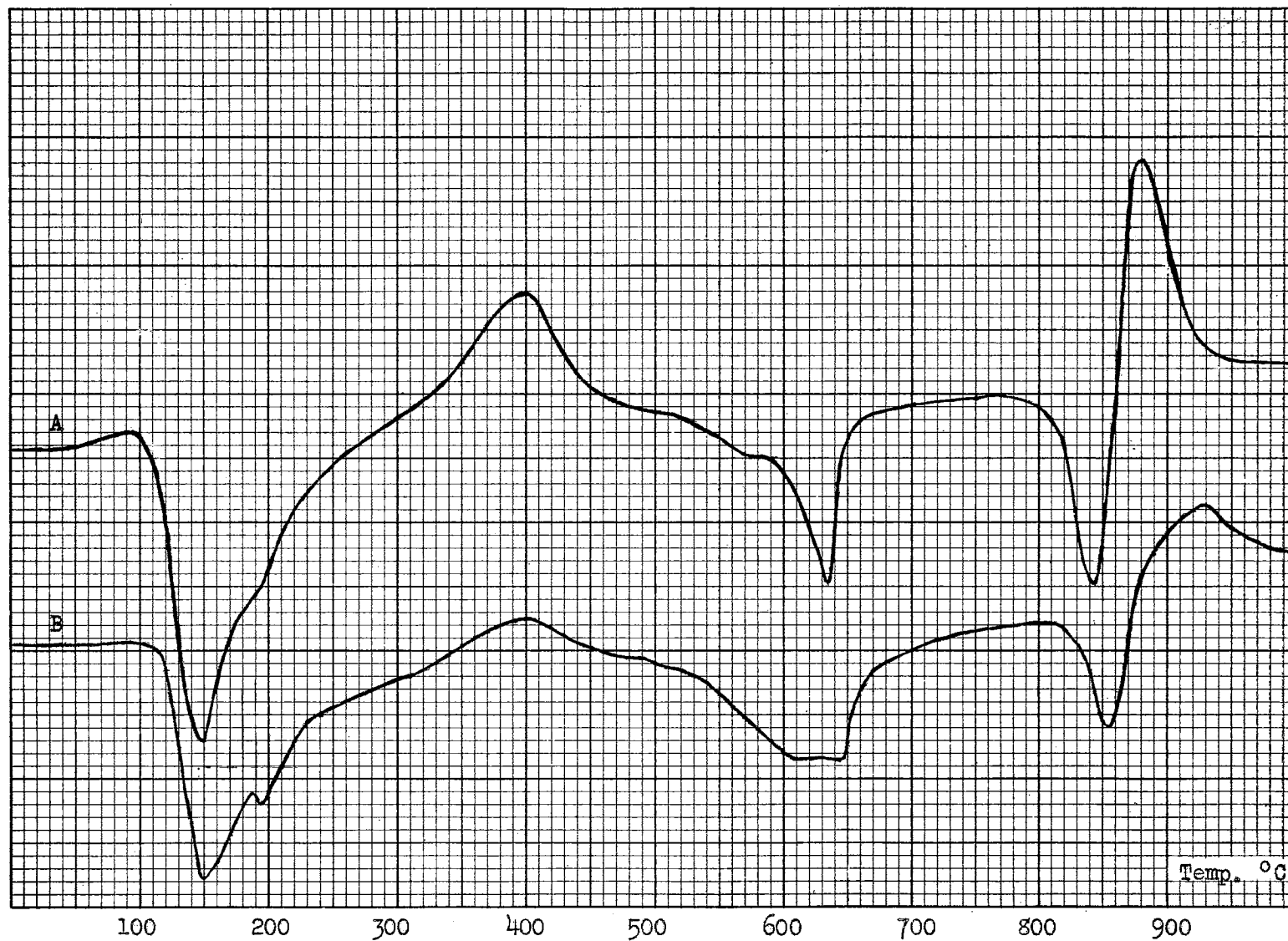


Fig. 13. Differential thermal curves of the fine (curve A) and coarse (curve B) clay fractions of the Kingfisher No. 1 C horizon.

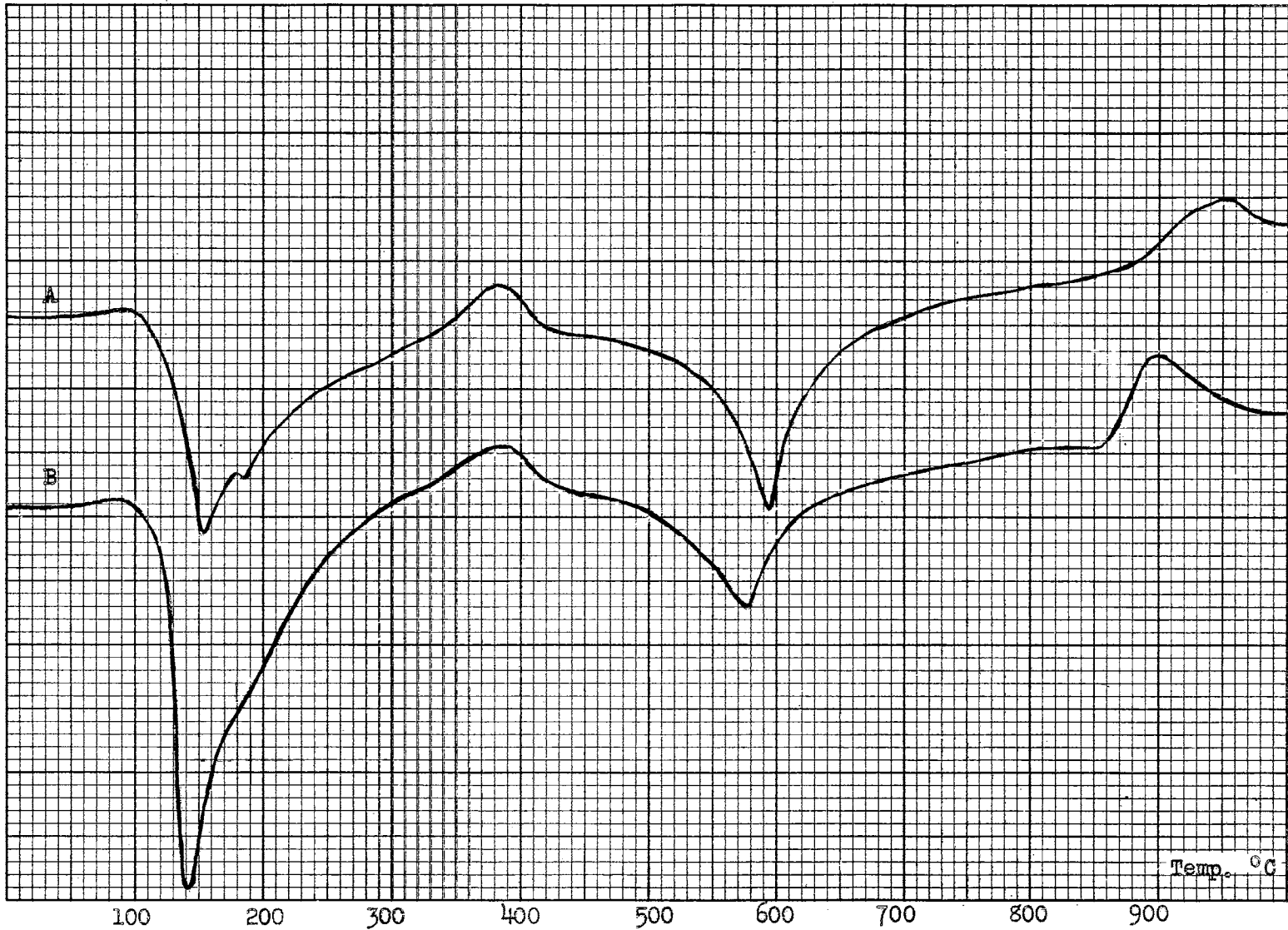


Fig. 14. Differential thermal curves for typical fine (curve B) clay fraction and coarse (curve A) clay fraction.

values, TABLES IX and X, it is apparent that a high exchange, expanding lattice mineral must comprise a large percentage of the fine clay fractions of all six profiles. If one assumes the 10% K_2O value for illite (18), uses the approximate percent of kaolinite from X-ray and D.T.A., and lets a montmorillonitic type mineral constitute the remainder of the sample, then calculates the theoretical cation exchange capacity and ethylene glycol retention value from this data, it compares quite favorably with the values obtained experimentally. From these calculations it appears that the interstratified, or hydrated mica, component of the fine clay fractions of these soils is primarily composed of a montmorillonitic type clay mineral, and that this mineral is the major constituent of the fine clay fractions where it is observed.

The types and relative amounts of clay minerals present in Zaneis No. 1 are fairly uniform for the A, C, and D horizons as reflected by the cation exchange capacity, percent K_2O , X-ray analysis, and D.T.A. (TABLE IX, Figures 1 and 2). This is especially true for the fine clays. The only noticeable difference is the appearance of the interstratified complex (1s) in the coarse clay of the A horizon. The B horizon is somewhat different. The characteristic 17.7 \AA peak of montmorillonite appears in the X-ray spectrograms of the coarse clay fraction (Figure 1) and a 20 \AA peak, possibly due to poorly ordered montmorillonite, appears in the X-ray of the fine clay fraction along with the absence of the 10 \AA illite peak (Figure 2). There is also an increase in the cation exchange capacity and ethylene glycol retention of both fractions and a decrease in K_2O content of the fine clay fraction (TABLE IX), indicating an increase in the montmorillonite content. Seemingly, the vermiculite-

chlorite (V/C) component of the coarse clay fraction has been converted into an interstratified complex and finally to a montmorillonitic type of mineral in the A and B horizons, and the montmorillonite has been eluviated and accumulated in the B horizon. The question remains as to just how much actual difference there is between these minerals. Has there actually been a conversion of the 2:1 layer silicates or merely a lessening of the bonding strength between lattices allowing wider expansion, and consequently a mineral more like montmorillonite? Unfortunately, this question cannot be answered without a more complete analysis.

From the data presented in TABLE IX the clay minerals present in the Zaneis No. 2 profile appear to be uniform throughout the profile as to type and relative amounts. This uniformity is also expressed in the X-ray spectrographs (Figures 3 and 4). Although the vermiculite-chlorite present in the coarse clay fraction of the C horizon does not show up in the X-ray analysis of the A and B horizons, the D.T.A. reveals it is present. The cation exchange capacities, percent K_2O , and glycol retention values are unusually uniform for the fine clays. If there has been any weathering of clay minerals in this profile it is not evident from this data.

The Zaneis No. 3 profile also exhibits a very uniform composition of clay minerals (TABLE IX). Other than a selective type of eluviation and illuviation favoring an increase of high-exchange, expanding lattice mineral in the B horizon, as reflected by an increase in the cation exchange capacity and glycol retention along with a decrease in percent K_2O , there is no apparent difference in the composition of the clay

fractions of the A, B, and C horizons of this profile. This trend is also reflected in the X-ray spectrographs (Figures 5 and 6). The diffraction patterns are very similar except for an increase in intensity of the low angle scatter ($<8^{\circ} 2\theta$) in the B horizon. Again, any weathering of clay minerals is not evident from this data.

The Zaneis No. 4 profile exhibits some unusual clay properties. From inspection of the X-ray spectrographs (Figures 7 and 8) and the data presented in TABLE X it is evident that the B horizon is quite different, and appears to be unrelated to the A and C horizons which are similar to each other. An abrupt change was also noted in the field between these horizons. It hardly seems probable that weathering is the answer. If it were, at least a detectable amount of the montmorillonite-chlorite interstratified mineral (M/C) should be present in the A horizon also. It does not seem likely that all of this material was transported into the B horizon by the processes of eluviation and illuviation. A better answer seems to be stratification of parent material as reflected by the mechanical analysis discussed previously. More will be said about this in the discussion of Kingfisher No. 1.

Another interesting feature of the B horizon is the marked similarity of the X-ray spectrographs (Figures 7 and 8) and ethylene glycol retention values of the fine and coarse clay fraction, but quite a drastic change in cation exchange capacity (TABLE X). The cation exchange capacity of the fine clay fraction more than doubles with only a small increase in ethylene glycol retention. This would tend to indicate the presence of amorphous or poorly crystallized constituents as a major component of the fine clays. Also a large, unexplained exothermic peak appears

in the differential thermal graphs of the fine clay that is not present in the coarse clay (Figure 15). Several peculiarities were also noted while preparing this sample. All of this points to the need for further investigation before any definite conclusions can be reached.

The clay mineral properties of the Kingfisher No. 2 are presented in TABLE X. The clay minerals present in the B and C horizons appear to be very uniform as to both type and relative amount. The cation exchange capacities, per cent K_2O , and ethylene glycol retention values exhibit little variation. The X-ray spectrographs of these two horizons (Figures 9 and 10) are quite similar, also indicating uniformity of minerals. The only noticeable difference is a slight increase in intensity of the low angle scatter ($<8^\circ$ 2θ) in the fine clay fraction of the B horizon, indicating more of the hydrated mica component. This is also reflected by an increase in the cation exchange capacity and a decrease in per cent K_2O , but it is not accompanied by an increase in glycol retention as would be expected.

The A horizon is slightly different in that it appears to have more illite than the other two horizons. The X-ray spectrographs (Figures 9 and 10) give exceptionally good illite peaks for all three orders of reflection (10, 5, and 3.34° \AA). This apparent increase of illite is not reflected by the K_2O content of the fine clay fraction, and there is only a slight increase in the K_2O content of the coarse clay fraction, but there is a noticeable drop in the cation exchange capacity and glycol retention value of the coarse clays.

There does appear to have been some weathering of clay minerals in the A horizon. Apparently, the vermiculite-chlorite (V/C) present in the coarse clays of the B and C horizon has been altered to a

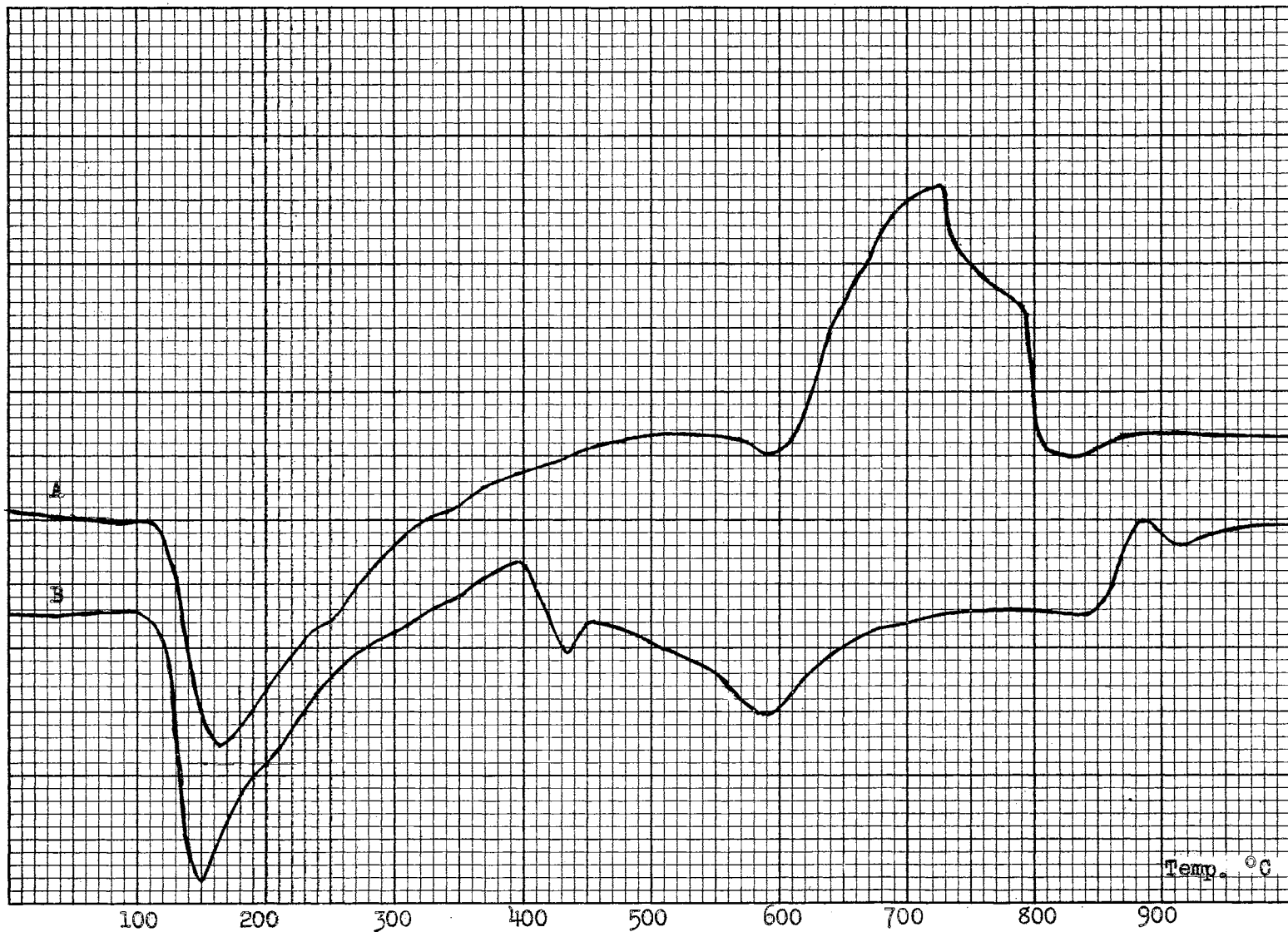


Fig. 15. Differential thermal curves for the fine (curve A) and Coarse (curve B) clay fractions of the Zaneis No. 4 B horizon.

hydrated mica type mineral of smaller size in the A horizon, then part of this mineral incorporated into the fine clay fraction has undergone eluviation and accumulated in the B horizon. This would account for the slight increase of the other minerals in the A horizon coarse clays and apparent increase of the hydrated mica in the B horizon fine clays. Weathering is also reflected in the loss of kaolinite from the A horizon fine clay fraction. However, all of these changes are slight, indicating that weathering has not proceeded to any great extent.

The clay mineralogical properties of Kingfisher No. 1 (TABLE X) appear to give evidence of intensive weathering. The data for the coarse clay fraction suggests the conversion of the montmorillonite-chlorite (M/C) found in the C horizon to a vermiculite-chlorite (V/C) component in the slightly weathered B horizon and to the interstratified (Is) or hydrated mica component present in the more highly weathered A horizon. These changes are accompanied by a reduction of cation exchange capacity and ethylene glycol retention and an increase in per cent K_2O . All of this suggests weathering or conversion of the 2:1 layer silicates in favor of the illitic component. It is also interesting to study the X-ray diffraction patterns of the coarse clay fractions (Figure 11). The large M/C peak (at 15.7 Å) of the C horizon appears to be cut off, leaving only the base, in the B horizon and is then completely cut off, leaving only low angle scatter, in the A horizon. However, when these samples were K saturated and heated, the 15.7 Å peak of the C horizon shifted only slightly to 14.2 Å, but there was a reduction of scatter and significant shift to 10 Å in the A and B horizons. This tends to rule out the possibility of a masking effect by some other component in the A and B horizons,

and establish that the minerals are actually different.

The trend present in the fine clay fraction is somewhat reversed from the coarse clay fraction. The M/C present in the C horizon is not detectable in the A and B horizons (Figure 12), but the cation exchange capacities and ethylene glycol retention values as well as the K_2O content increase in the fine clays of the A and B horizons (TABLE X). This would indicate weathering in favor of a clay mineral with greater exchange properties and higher glycol retention values as well as an illitic type mineral, with the former component being of smaller particle size and the bulk of it falling into the fine clay fraction. However, when all of this is viewed in the light of other data, another possibility is revealed. An inspection of the data for Kingfisher No. 2 (TABLE X) reveals the clay minerals present in the coarse clay fractions of the A and B horizons of the two profiles to be the same, and only slightly different, in that there is more illite present in the No. 2 profile, for the fine clay fraction; however, the C horizons are quite different. By comparing the clay properties of the C horizon of Kingfisher No. 1 with the B horizon of Zaneis No. 4 (TABLE X) a noticeable similarity is found. Apparently, the same mineral, the M/C, is present in these two horizons, but is not present in any other horizon of any profile studied. These facts very strongly suggest the possibility of stratification of parent material.

It will be remembered that the mechanical analysis of Zaneis No. 4 and Kingfisher No. 1, discussed previously, also suggested stratification. All of this leads to the question that perhaps there is some relation between the clay minerals of the B horizon of the Zaneis No. 4 and the C horizon of the Kingfisher No. 1, and also a relation

between the clay minerals of the A and B horizons of Kingfisher No. 1 and Kingfisher No. 2.

More interesting facts are brought to light by observing the exact location of these profiles in relation to their respective geological formations. It will be remembered that the Zaneis No. 4 is located on the Garber formation and the Kingfisher No. 1 and No. 2 on the Cedar Hills sandstone member of the Hennessey formation. The site location of Zaneis No. 4 is very close to the contact of the Garber and the Hennessey shales, the Kingfisher No. 1 is located very close to the contact of the Cedar Hills sandstone and the Hennessey shales, and the Kingfisher No. 2 well over into the Cedar Hills sandstone. It would seem possible that the C horizon of the Kingfisher No. 1 is actually material from the Hennessey shales and that only the A and B horizons are derived from Cedar Hills sandstone materials. The mechanical analysis also points to this possibility. Inspection of TABLE V shows that the lower half of the profile has a marked increase in the fine silt fraction and very little sand, indicating a finer textured parent material. The upper half of the profile has only a moderate amount of fine silt with more sand, as does the Kingfisher No. 2, indicating a coarser textured parent material. It would also seem plausible that there could be an interstratification of materials from both formations at the site of Zaneis No. 4, and that the B horizon of Zaneis No. 4 is derived principally from Hennessey shale material, not from the Garber sandstones as the remainder of the profile apparently is since it strongly resembles the other Zaneis profiles as to clay mineralogy. This

would explain why the C horizon of Kingfisher No. 1 appears to be related to the B horizon of Zaneis No. 4, while the A and B horizons appear to be related to Kingfisher No. 2.

In light of the data presented here, stratification of parent material seems to be a better answer for the differences observed in the clay mineralogy of the Kingfisher No. 1 and Zaneis No. 4 profiles than does weathering of clay minerals.

Stratification as postulated here cannot be proven until data is available for the clay mineralogy of the Hennessey shales in the vicinity of the Zaneis No. 4 and Kingfisher No. 1. It does, however, seem feasible. The Oklahoma geology map¹ shows the Zaneis No. 4 to be located on a narrow finger of the Garber sandstones, bounded on both sides by the Hennessey shales. This location should increase the possibility of random stratification. The map also reveals the Kingfisher No. 1 to be just over into the Cedar Hills sandstones, which overlay the Hennessey shales. This location should give a good chance for overlapping of the two formations as suggested by the data presented in this report.

The geology of this whole area is rather uncertain, as a great deal of disconformity is observed in the Permian deposits of Oklahoma. This is probably due to the fact that these deposits were derived from streams rising to the north, east, and south, depositing their loads of sediments into a southwestward retreating sea (5). Oaks (30)

¹Geologic Map of Oklahoma by H. D. Miser, U. S. Geological Survey. Prepared cooperatively by the U. S. Geological Survey and the Oklahoma Geological Survey (1954).

has postulated the early existence of a large river, flowing from the southeast across southern Oklahoma, depositing a large delta represented by the upper portion of the Garber-Wellington formation of central Oklahoma. Westward tilt of strata and deep post Permian erosion apparently destroyed the rest of the delta (30). Two other deltas, one comparable to the modern Nile delta, have been proposed by Chenoweth (4) in this area. Under these conditions, it would seem that stratification is not only possible, but probable. This leaves the question as to whether any changes in clay minerals noted in these soils are actually due to weathering or stratification of parent material. The uniformity of minerals present in the fine clay fraction of some of the profiles investigated in this report leads to the conclusion that there has been some weathering of the coarse clay fractions, but it is difficult to ascertain to what degree.

It appears that the present regime of soil forming factors has had little effect in altering the clay minerals present in these soils. Seemingly, the clay minerals present in the solum are, for the most part, inherited directly from the clay minerals present in the parent material. Apparently, these minerals were laid down in a sedimentary parent material in a weathered state and are therefore resistant to further weathering.

The exact percentage of each clay fraction was not determined. However, from the separation of the fractions their relative amounts were estimated. The fine clay fraction composes about 70 to 80 per cent of most horizons and exhibits a tendency toward accumulation in the B horizon. The D horizon of Zaneis No. 1 showed the highest per cent of coarse particles. The two fractions were about 50-50

in this horizon.

From the data reported in TABLES IX and X the relative amounts of clay present in the fine clays were estimated. The coarse clays were too varied to give any better estimate than that of the X-ray analysis and D.T.A., except for illite. The 10 per cent K_2O value for pure illite (18) was used as an index for illite content. Multiplying the per cent K_2O (TABLES IX and X) by 10 gives a good estimation of illite. The relative amount of kaolinite indicated by the X-ray results and D.T.A. was used for kaolinite content. The remainder of the sample was assumed to be an expanding lattice, high exchange montmorillonitic mineral. The theoretical values calculated from this data were then compared with the experimentally measured cation exchange capacity and ethylene glycol retention value and found to give good agreement.

The results of these calculations indicate that the B horizon fine clays of Zaneis No. 1 and No. 3 are about 70-75 per cent a montmorillonitic mineral, while the remainder of both profiles are about 60-65 per cent this mineral. The fine clays of Zaneis No. 2 are more uniform and appear to be about 55-60 per cent montmorillonitic throughout the profile. The Zaneis No. 4 A and C horizon fine clays are about 60-65 per cent montmorillonitic. From the available data the clay content of the B horizon cannot be calculated. The fine clays of Kingfisher No. 2 are about 60-65 per cent montmorillonitic throughout the profile. The Kingfisher No. 1 A and B horizon fine clay fractions appear to be about 70 per cent montmorillonitic mineral, and the montmorillonite-chlorite (M/C) interstratified mineral appears to compose about the same percentage of the C horizon.

In comparing the Zaneis and Kingfisher profiles it will be noted that the clay minerals present are quite similar. The only difference found in this investigation is that the Kingfisher profiles contain more illite and less kaolinite. The hydrated mica content, which appears to be primarily a montmorillonitic mineral, is about the same and dominates the clay fraction of both series.

SUMMARY AND CONCLUSIONS

Two soil series of extensive area in the Reddish Prairie of central Oklahoma were examined to determine the type and relative amounts of clay minerals present in the major horizons.

Four profiles of the Zaneis series and two profiles of the Kingfisher series were sampled by horizon and sub-horizon to the depth of the slightly weathered parent material. A chemical and mechanical analysis was then made for each horizon. Only the major horizons were selected for clay analysis. The clay fraction of each of the selected horizons was separated by sedimentation. This fraction was further fractionated into the 2- to 0.2 micron size fractions.

The material found in these two fractions was examined by chemical and physical analysis, X-ray, and D.T.A. From the results of these examinations, the clay minerals present in each of the two size separates of the clay fraction were determined as to type and relative amount.

The results of the chemical analysis indicated calcification to be the predominate soil forming process of both series, and revealed the Kingfisher profiles to be higher in phosphorus and exchangeable bases.

The mechanical analysis revealed the Kingfisher profiles to be considerably higher in silt content; whereas, the Zaneis profiles

are higher in sand. It also revealed a strong possibility of stratified parent materials, especially in the Zaneis No. 4 and Kingfisher No. 1.

The results of the clay mineral study indicate the fine clays dominate the clay fractions of all six profiles, and a montmorillonitic mineral dominates the fine clays, with lesser amounts of illite and kaolinite. The coarse clay fractions exhibit more diversity with montmorillonite, illite, kaolinite, and vermiculite all being present as major components, and quartz and chlorite present in lesser amounts. Illite dominates the coarse clay fractions of both Kingfisher profiles and is a major constituent of the fine clays. The only notable difference between the two series, as to the clay mineralogy, is the higher concentration of illite in the Kingfisher series at the expense of the kaolinite content. Content of the other minerals is comparable. The clay mineral content throughout the profile is fairly constant for most profiles. This is not true for the Zaneis No. 4 and Kingfisher No. 1, but stratification appears to play the dominant role in these two profiles.

A montmorillonite-chlorite interstratified mineral appeared in both clay fractions of the B horizon of Zaneis No. 4 and C horizon of Kingfisher No. 1, but was not found in any other horizon or any other profile. The occurrence of this mineral, geological location, and mechanical analysis were used to postulate the stratification of parent material from two different geological formations at the site locations of these two profiles.

Very little evidence of clay mineral weathering was found in these soils. It appears that the clay minerals present in these profiles

are for the most part inherited directly from the clay minerals present in the parent material, and deposited as a sedimentary parent material in a weathered state and are resistant to further weathering. Consequently, the present regime of soil forming factors has had little effect in altering these minerals.

The major morphological differences between the two soil series studied exist in the nature of the parent material. It appears that the soil materials of the two Kingfisher profiles were deposited under more of a marine environment, whereas the Zaneis soil materials were more of a fresh water deposit. This is reflected in the more silty texture, higher base concentration, and higher illite and phosphorus content of the Kingfisher soils.

More basic morphological data is needed to better classify the Reddish Prairie soils.

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VITA

Harrell Dean Molthan

Candidate for the Degree

of

Master of Science

Thesis: A CHEMICAL AND MINERALOGICAL CHARACTERIZATION AND GENESIS
STUDY OF THE ZANEIS AND KINGFISHER SOIL SERIES OF THE
REDDISH PRAIRIE GREAT SOIL GROUP

Major: Agronomy

Biographical:

Personal data: Born February 5, 1933, at Ft. Smith,
Arkansas, son of Harrell C. and Lucille Molthan.

Education: Graduated from Bokoshe High School, Bokoshe,
Oklahoma, 1950. Undergraduate work at Oklahoma Agri-
cultural and Mechanical College, Stillwater, Oklahoma,
1950 to 1953, 1955 to 1957. Graduate study at Oklahoma
State University, 1959 to 1960.

Experience: Farm reared. Farm labor during summers from
from 1951 to 1953. U. S. Army 1953 to 1955. Phillips
Petroleum Company summer 1956. Graduate assistant,
1957 to 1960, Oklahoma State University.

Member: Agronomy Club and Phi Sigma

Date of Final Examination: May, 1960