# AN ANALYSIS OF

CLAY MINERALS IN RECENT SEDIMENTS FROM THE EAST BAY, GALVESTON

An Abstract of a Thesis Presented to the Faculty of the Graduate School The University of Houston

In Partial Fulfillment of the Requirements for the Degree Master of Science in Geology

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Donald J. Weintritt

August, 1957

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Twenty-one samples of recent sediments taken from a traverse made around East Bay, near Galveston, Texas were subjected to mechanical, differential thermal and base exchange analysis. Special X-ray techniques were employed on the clay fraction by treatment with ethylene glycol and by subjecting them to elevated temperatures up to 550°C.

X-ray analysis proved the most useful technique for discerning minor sample differences, especially as regarding mixed layer sequences.

The distribution of clay minerals by weight was most heavily concentrated in the natural sedimentary trap north of Hanna Reef and along the northern side of East Bay. This suggests that geologically, the bay is retaining, hence gaining sediments, more from the north side than on the side facing Bolivar Peninsula. (Southern shore of East Bay).

As regards diagenesis, Illite, a typical marine-formed clay, was found in all 21 samples. Though quite variable in quantity, the relative proportion of clay minerals in each sample was quite uniform. This is as may be expected in a relatively small lagoonal area not directly fed by an important continental river or stream. Whether the illite is being formed in situ or being transported in from the local quaternary sediments or from older sediments, via the Trinity River for example, can not be established without additional sampling of the provenance areas.

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### I. INTRODUCTION

The purpose of this study is to present the results of clay mineral analyses of recent sediments collected in the East Bay region near Galveston Texas. (Fig. 1). The significance of the data in relation to diagenetic changes and the environment of accumulation are also considered.

The mechanics of the breakdown, transportation and deposition of nonclay minerals has in the past been well covered by geologists and is now a principle well understood. These principles of sedimentation are adequately covered by Krumbein and Pettijohn (1), Twenhofel (2), Krumbein and Sloss (3), and others.

A new sedimentary frontier following on the wake of the development of special equipment allowing classification of the clay minerals is the analysis of the type and amount of clay mineral found in continental and marine areas. These studies are now under vigorous pursuit for various economic and academic reasons.

The clay minerals are small, have high absorptive capacity and hence, are sensitive to environmental changes. This fact, in lieu of the realization that some 50 percent of the sedimentary column consists of shales, is sufficient



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reason to devote considerable study in behalf of the clay minerals.

#### II. THE NATURE OF THE CLAY MINERALS

While most data on the structure, composition, properties, occurrence and mode of origin of the clay minerals has been primarily for the benefit of either Soils or Ceramic interests, geologic correlations are now being extended. The bulk of such correlations are as yet so recent that they are found in detail only in monographic form in the literature.

Two useful texts for clay mineral research studies are by Brindley (4), and Grim (5). They adequately review the current status of the science. A brief description of the clay minerals pertinent to this investigation follows:

## (A) Montmorillonite

Montmorillonite is a sheet type silicate whose structure is usually considered to resemble that of pyrophyllite. The pyrophyllite structure is composed of two outer layers of silica tetrahedra tied together at three corners to form a hexagonal exygen set. The inward-pointing apical exygen of the tetrahedra, bogether with coplanar hydroxyl ions, surround an inner layer of estahedrally coordinated aluminum ion. Montmorillonite differs from pyrophyllite in having isomorphous substitutions, usually of magnesium or iron for aluminum in the octahedral layer, or of aluminum for silicon in the tetrahedral layer, thereby creating a net negative charge, which is satisfied by exchangeable cations largely on the basal oxygen net of the outer tetrahedral layers. The commonest cations held by montmorillonite are calcium, sodium and magnesium. One or more molecular layers of water may enter between each of the unit sheets of montmorillonite as above described. The amount of water that can be adsorbed is dependent upon the amount of water available, the nature of the exchangeable cation held by the montmorillonite, and possibly upon other unknown factors.

Although most earlier workers have tacitly assumed montmorillonite to be a homogeneous mineral, detailed studies and especially studies of marine sediments show that mixtures are far more common than previously realized.

Another important factor of geologic significance as a determinant in the properties of montmorillonite is the nature of the exchangeable cation or cations held between unit sheets. On drying at normal atmospheric humidities, sodium montmorillonite will hold one molecular layer of water between unit sheets, whereas calcium or magnesium montmorillonites will hold two molecular layers. Zero, one and two molecular layers of adsorbed water give rise respectively to basal (001) spacings of 9.6, 12.4, and 15.4 A. Almost all air dried montmorillonites are an

interlayed mixture of two or three of these components and therefore show intermediate basal spacings. The characteristics of mixed layer sequences of this nature have been discussed by Hendricks and Teller (6). Their presence and means of identification in marine sediments were covered by Johns, Grim and Bradley (7), Hathaway and Carroll (8), and Weaver (9). Simple mixed layer sequences give sharp (001) reflections, but may be detected by the absence of integral orders originating from (001) planes. (002) reflections are usually present, but they do not occur at "expected" positions. Asymmetrical (001) reflections are given only by mixed layer sequences with three or more components.

## (B) Kaolinite

In general, the determination of kaolinite by X-ray diffraction is simple. The prominent (001) (002) basal reflections at 7.14 A and 3.57 A are usually adequate for identification.

The structure is composed of a single silica tetrahedral sheet and a single alumina octahedral sheet combined in a unit so that the tips of the silica tetrahedron and one of the layers of the octahedral sheet form a common dayer. All of the tips of the silica tetrahedron point in the same direction and toward the center of the unit made of the silica and octahedral sheets. The chloritic clay minerals may be confused with the kaolinite minerals.

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However, usually the third order at 4.7 A is seen to indicate the presence of chlorite. Chlorites rich in iron frequently give weak first and third-order reflections, and differentiation from kaolinite is particularly difficult.

Kaolinite on heating to 550°C tends to lose its crystalline character, whereas chlorite at this temperature is only partially dehydrated, causing increased intensity of the 14 A reflection.

# (C) Illite

Illites can usually be identified on the basis of X-ray diffraction by their (000) spacings with the (001) at about 10 A. The 10-A line in Illite usually has a band trailing off in intensity toward the low angle region. This unlike in true micas is due to substitutional variations. Most of the illite clay minerals are dioctahedral though trioctahedral forms are locally common.

The structure of illite differs from that of montmorillonite by having a greater charge deficiency and by having most of this greater charge located in the surface layer of tetrahedrally cooridinated ions. Also in the case of illite the balancing cation between the layers is largely potassium. The effect of having the charge deficiency close to the basal surface combined with the small ionic size of the potassium ion renders the layers nonexpandable and the ions nonexchangeable.

## (D) Chlorite

like layers. The layers are continuous in the <u>a</u> and <u>b</u> dimensions and are stacked in the <u>c</u> direction with basal cleavage between the layers. The mica layer is unbalanced by substitution of aluminum for silicon, and this deficiency of charge is balanced by an excess charge in the brucite sheet of aluminum for magnesium.

The chlorite structure consists of alternate mica-like and brucite-

The characteristic 14 A spacing is not changed with moderate heating, nor is it changed by treatment with ethylene glycol as it is in the montmorillonites.

# (E) Mixed-layer minerals

Mixed layer structures are of two different types: regular and random. The random mixed layer mineral is very common in sedimentary rocks. Weaver (9) suggests that if minor environmental differences can be determined by clay mineral. studies the mixed layer clays hold the answer.

The trioctahedral clay minerals including montmorillonite, illite, chlorite and vermiculite form similar flake shaped layers. The type of interlayer cation determines the clay type. A non-mixed layer clay will contain the same type of cation between each layer. However, if 5 or 10 layers contain one type of cation and another 10 or 15 layers contain a different cation, a mixed layer clay is the result.

The cause of mixed layer minerals preferentially adsorbing certain cations in preference to other cations is probably due to minor difference in substitution within the octahedral and tetrahedral layers.

### III. SAMPLES USED IN THIS STUDY

A brief description is given below of the samples used in this study. All samples were collected by Dr. Fan in the summer of 1951. Samples collected were of very recent deposition; i.e., the aqueous mud of slurry-like consistency. Figure 2 shows the traverse followed around East Bay. Geographic data were taken from USGS Chart 1282, "Galveston Bay and Approaches."

<u>Samples 1 through 6.</u> The first leg of the traverse lies in a line extending from Port Bolivar north to Smith Point. These samples represent a dividing line between Galveston Bay to the West and East Bay to the East. Hanna Reef Bisects the traverse along this line.

Samples 6 through 10. These samples were collected along the northern side of East Bay approximately one mile from shore, east, to a point near Robinson Bayou.

Samples 10 through 12. This leg of the traverse extends from Robinson Bayou, south, to Marsh Point. East Bay at this point is about two miles wide.



Samples 12 through 21. Samples collected along this traverse extend

from Marsh Point, west to Baffle Point along the northern side of Bolivar Peninsula.

On the basis of area, the samples collected encompass an area of approximately 60 square nautical miles.

## IV. EXPERIMENTAL RESULTS

A. Particle Size Analysis and Methods of Sample Preparation

1. Procedure: Samples were dispersed on a Hamilton Beach #30 mixer for 15 minutes at high speed. The amount taken on each sample varied in a rough proportion to the amount of high viscosity clay. That is, samples obviously high in clay were lower in weight dispersed than samples high in sand.

Dispersion of the clay was facilitated by the addition of a solution of sodium hexa-meta phosphate to maximum fluidity.

After stirring at high speed, samples were aged overnight or longer to allow the samples to come to equilibrium. Samples were restirred five minutes at low speed, then poured over standard 8 inch sieves.

screen sizes used were 20, 100, 200, and 325 mesh. In each case, a gentle stream of water was used to assure that all sizes

finer than that indicated were washed through to the collection vessel.

The plus 20 mesh fraction in all cases consisted of shell fragments, weeds, etc., and consequently was neither measured nor retained for study. The 100, 200, and 325 mesh fractions were dried to a constant weight at  $110^{\circ}$ C and the amount retained in each case was recorded.

The minus 325 mesh fraction was filtered from 3 to 7 times, to zero or trace  $Cl^-$  and  $SO_{l_2}$  = content. Filtration was carried out on a Baroid laboratory filter press, Model 302 using distilled water. Details of the equipment may be found in an article by Weintritt and Perricone (10). The solids free filtrate after each filtration was tested for cloride ion with AgNO<sub>3</sub> and sulfate ion with an acidified solution of BaCl<sub>2</sub>.

After each sample was adjudged free from soluble salt and therefore no danger of flocculation due to residual salts was feared, the samples were redispersed and separated into two fractions (-44,  $\neq$ 2 micron) and (-2 micron) by Stokes law sedimentation measurements. The separation was continued until all particles greater than 2

micron settled out in the required period of time for the given height of the sedimentation tube used. This usually required from two to four settling periods.

The purpose of the separation was to concentrate all of the elay mineral into the -2 micron fraction. The absence of the 26.4 degree 2 0 quartz peak in all of the samples X-rayed indicates the separation was quite good.

2. Results: The objective of this investigation was characterization of the clay fraction. Hence, the weight dilution of the clay sample by non-clay minerals is of greater importance than their routine identification and classification by the usual size sorting charts.

Figures 3 through 6 show the percentage of each particle size fraction in relation to its weight dilution of the clay mineral.

From Figure 3 it is shown that the plus 100 mesh fraction is present in the largest amount on the south side of East Bay (#13 to 21) and also in the area from Hanna Reef south to Port Bolivar (#1 to 3).

In Figure 4 an analysis of the -100,  $\neq$  200 mesh non-clay fraction indicates a minimum weight dilution of the sediment in an



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area influenced by the physical barrier of Hanna Reef and the northwest side of East Bay (#4 through 7).

The weight distribution histogram depicted in Figure 5 shows that in the -200,  $\neq$  325 mesh range, the East Bay area has the greatest homogeneity. That is, there is less spread between samples than on any other size fraction.

In the -44,  $\neq 2$  micron size fraction as shown in Figure 6 it is observed that the fine non-clay materials are deposited in roughly the same ratios as those of the clay minerals plotted on Figure 7. From this it may be concluded that the same forces responsible for deposition and retention of the clay minerals in any area of East Bay have had the same effect on the very fine non-clay fraction.

By "a priori" reasoning it appears feasible that as non-marine kaolin and montmorillonite were introduced into the bay, presumably from the Trinity River and nearby bayous, tidal currents and other lagoonal forces that transported the clay minerals to their present deposition site were of sufficient magnitude to carry along the -44,  $\neq 2$  micron fraction in approximately the same ratio. On a weight basis, it will be noted from these two figures that there is about



twice as much clay as non-clay.

It may be concluded from curves 3 through 7 that there are two important factors at the present time - physical and chemical. Figure 8 has been prepared to show this fact in another manner. The first three curves at the upper part of the graph are in an area between Hanna Reef and Port Bolivar. This is also the deepest part of East Bay and therefore indicates stronger tidal currents. In this region, the non-clay minerals predominate while the clay minerals are of lesser weight distribution.

The last three curves are in a shallow area surrounded by Hanna Reef and the land on the north-west side of East Bay. In this area the clay minerals make up the prominent constituent on a weight basis. Further evidence of sedimentary accumulation by particle size lies in the ratio of montmorillonite to kaolinite and illite. Kaolinite and illite will always have a larger particle size than montmorillonite because of the difference in ionic coordination and substitution. Hence, the particle size distribution by weight as shown in Figure 6 on the  $-\frac{1}{4}$ ,  $\frac{4}{2}$  micron size, should be carried over as a proportion in the coarse clay minerals (Kaolinite and illite). Inspection of

# TABLE 1

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# Weight-Size Distribution

		Weight Di	stribution,	Percent	
Particle Size, micron	-840 <u>4149</u>	-149 + 74	- 74 - 44	- 44 - 4 2	- 2
Sample #1	20.92	62.84	7.45	3.86	LL OLL
Sample #2	9.77	46.64	14.99	11.84	16.76
Sample #3	2.53	29.33	11.12	22.40	34.61
Sample #4	0.31	5.14	8.71	30.80	55.03
Sample #5	0.14	2.10	5.11	31.91	60.73
Sample #6	0.15	1.13	4.30	31.95	62.47
Sample #7	1.59	5.59	4.19	26.03	62.60
Sample #8	0.55	12.19	17.80	25.09	LL : 36
Sample #9	0.34	8.04	12.32	30.66	48.63
Sample #10	0.42	20.50	15.24	19.28	44.56
Sample #11	1.15	23.88	14.62	20.46	39,88
Sample #12	1.36	35.86	12.37	14.12	36:29
Sample #13	3.63	60.96	8.72	8.13	18.57
Sample #14	5.34	58.37	15.66	8.84	11.79
Sample #15	24.35	59.19	6.71	3, 95	5.80
Sample #16	26.92	46.74	8.15	6.04	11.26
Sample #17	17.37	61.09	8.11	4.38	9.06
Sample #18	3.14	23.13	9.56	25.85	38.32
Sample #19	4.80	8.97	4.77	26.03	55.44
Sample #20	1.64	19.41	24.03	22.20	39.71
Sample #21	16.78	35.22	21.26	11.97	14.78
Average	6.83	29.82	11.20	18.41	33.74

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b



Figure 7 shows this to be true. A detailed analysis is given in Table 1.

As regards the other factor, that of chemical forces, the identification and quantitative measurement of illite shows that this mineral is present in varying amounts in all 21 samples. The weight size distribution histogram of Figure 7 shows that the amount of illite present in any one sample is equal to about 1/3 of the total amount of clay in all samples. X-ray data suggest that the illite present was formed from degraded or alkali poor montmorillonite. This phase of the investigation will be discussed more thoroughly in the section on X-ray analysis.

### B. X-Ray Analysis

 Procedure: Preparation and selection of the -2 micron fraction was described in the previous section. The fraction reserved for X-ray analysis was left hydrated and stored in sealed jars until needed.

Slides for X-ray analysis were prepared as follows: The dilute elurries were poured onto standard 27X46 mm. microscope slides and allowed to air dry. The dried slides were stored in an atmosphere of 50% relative humidity. The humectant used was sodium nitrate. Slides were aged 24 hours or longer in order to allow adsorbed interplatlet water to reach equilibrium for the type of exchange ions adsorbed on the clay. This technique of orienting the clay micelles was similar to that used by Williams, Neznayko and Weintritt (11) and others.

To insure uniform results, all slides were X-rayed starting at the low angle. This technique allows time to run through the humidity sensitive montmorillonitic basal spacings before the water content is altered by a different water vapor pressure in the room.

On completion of the 50% relative humidity X-ray curves, the oriented slides were placed in a dessicator type vessel containing ethylene glycol and heated to  $60^{\circ}$ C for one hour. This technique was described by Brunton (12). It serves to increase the (00%) spacings of expandable clay minerals and facilitates their identification. The use of ethylene glycol has been used frequently in clay mineralogy since its introduction by Bradley in 1945 (13). MacEwan (14) and others have adequately described the theory of the adsorption of organic liquids by elay minerals. After glycolation, samples were allowed to cool to room temperature and were then X-rayed. The glycolated sample curve served as the basis for the quantitative estimation of the clay minerals using the technique of Hathaway and Carroll (8).

Third step in the treatment of the oriented slides was their heating in a muffle furnace to 400°C at the rate of about 10°C per minute. Samples were cooled rapidly and allowed to come to equilibrium with room conditions; after which, each slide was X-rayed. The fourth step was to again heat the samples to 500 + 550°C and repeat the X-ray analysis.

The above described series of steps were necessary in order to ascertain the presence of mixed layer elay minerals including vermiculites and chlorites. The technique while seemingly complicated is necessary because of the identical spacings of kaolinite, chlorite and some mixed layer clay minerals of questionable crystallographic constitution and chemical composition. The theory of this technique is best described by Johns, Grim and Bradley (7), Weaver (9) and Hathaway and Carroll (8).

All analyses were on a North American Phillips Recording High-

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Samples 6 through 10. These samples were collected along the northern side of East Bay approximately one mile from shore, east, to a point near Robinson Bayou.

Samples 10 through 12. This leg of the traverse extends from Robinson Bayou, south, to Marsh Point. East Bay at this point is about two miles wide. Angle Spectrometer, using  $CuK_{\chi}$  radiation at 35 KV, 18 MA. Normal scanning speed was 1 degree/min. Amplifier settings were: Scale Factor 16, Multiplier 1.0, and a Time Constant of 8. The receiving slit was 0.003 inches. The goniometer was run from 2 to  $40^{\circ}$ , 29.

In order to more clearly define the asymmetry of the montmorillonite (OOl) spacing, about half of the samples were run at 1/2 degree per minute and a higher amplification (Scale Factor of 8). In these cases it was only necessary to scan the geiger muller tube to 14 degrees 20 since the <u>Sin 0</u> values drop off rapidly at higher angles and can therefore be accurately resolved at high chart speeds.

2. Results: Figures 9 and 10 are photographs of several typical spectrometer curves taken after each type of treatment. Because many of the samples were taken close together it was not surprising to note that from the viewpoint of diagenetic differences there was little variation in the results obtained. However, to the extent that each sample was studied the results are sited below: Sample 1.

(a) 50% RH. Mont. (001) - 14.9 Å. No higher orders. Only broad bands.
 Illite (001) - 10°A. Distinct. Other (001) series well defined.
 Kaolinite (001) - 7°A. Distinct.







1.



- (b) <u>60°C. Ethylene Glycol.</u> Mont. (001) shifted to 16°A. No higher orders. Only broad bands. Indicates random interstratification.
  Sample 2.
- (a) <u>50% RH.</u> Mont. (001) peak is broad. Extends from 12.6 to 14 Å.
  No higher orders.
- (b) 60°C. Ethylene Glycol. Mont. (001) peak shifted to 16.7 Å.
- (c)  $400^{\circ}$ C. Mont. peak collapsed to 10 Å. Some rehydration apparent at higher angles probably due to not being cooled and kept in a desiccator. Intensity of 7 Å Kaolin peak not decreased. Indicating that if chlorite had been present dehydroxilization of the 7 Å chlorite peak at  $400^{\circ}$ C would result in a decrease of the relative intensity at 7 Å and an increase of the 14 Å peak.
- (d) <u>550°C.</u> Sample run on a broken fragment at high amplification.
  Kaolin 7 Å peak gone. No residual chlorite at 14 Å, 7 Å or 3.5 Å.
  Sample 3.
- (a) <u>50% RH.</u> Mont. (001) peak at 14.2 Å.
- (b) <u>60°C. Ethylene Glycol.</u> Mont. (001) peak shifted to 16.7 Å.
- (c) <u>550°C.</u> Same as 2 c.
- (d) <u>550°C.</u> Kaolin 7 Å peak gone. All Mont. Collapsed to 10°A.

No chlorite peaks apparent.

Sample 4.

- (a) 50% RH. Mont. (001) peak at 14.2 to 14.9 Å.
- (b) 60°C. Ethylene Glycol. Mont. (001) peak shifted to 16 Å.

Indicates some random interstratification.

- (c) 400°C. Same as 2c.
  - (d) 550°C. Same as 3d.

Sample 5.

- (a) 50% RH. Mont. (001) at 14.4 Å. Peak asymmetric to right. Indicates random interstratification toward the sodium side.
- (b) 60°C. Ethylene Glycol. Mont. (001) shifted to 16.9 Å.
- (c) 400°C. Same as 2c.
- (d) 550°C. Same as 3d.

Sample 6.

- (a) 50% RH. Mont. (001) at 14.2 Å. Assymmetric to right. Same as 5a.
- (b) 60°C. Ethylene Glycol. Mant. (001) shifted to 17.0 Å.
- (c)  $400^{\circ}C$ . Same as 2c.
- (d) 550°C. Same as 3d.

## Sample 7.

- (a) 50% RH. Mont. (001) at 12.9 to 14.2 Å.
- (b) 60°C. Ethylene Glycol. Mont. (001) shifted to double peak at

16.6 and 17.3 Å.

- (c)  $400^{\circ}$ C. Double peak of 7 b apparently unimportant. Intensity of 7 Å peak unchanged.
- (d) <u>550°C.</u> Same as 3d.

Sample 8.

- (a) <u>50% RH.</u> Mont. (001) has shoulder at 12.2°A, 13.6 and a broad peak at 14.0 to 14.7 Å.
- (b) 60°C. Ethylene Glycol. Mont. (001) shifted to 16.7 Å.
- (c) 400°C. Same as 2c.
- (d) <u>550°C</u>. Though 7A Kaolin peak not completely destroyed residual peak could not be chlorite since no significant 14 Å chlorite peak is apparent.

Sample 9.

- (a) 50% RH. Mont. (001) peak at 11.8 and 12.4 Å.
- (b) <u>60°C. Ethylene Glycol.</u> Mont. (001) peak shifted to 16.4 Å. No higher (00\$) series indicates random interstratification.
- (c) 400°C. Same as 2c.
- (d) <u>550°C.</u> Same as 3d.

Sample 10.

(a) 50% RH. Mont. (001) peak at 14.0 Å. Broad peak indicates

random interstratification.

- (b) 60°C. Ethylene Glycol. Mont. (001) peak shifted to 16.7 Å.
- (c) 400°C. Same as 2c.
- (d) 550°C. Same as 3d.

Sample 11.

- (a) <u>50% RH.</u> Mont. (001) at 13.6 to 14.0 Å. Peak asymmetric to right.
  No higher (002) series hence has random mixture of mono and divalent cations.
- (b) 60°C. Ethylene Glycol. Mont. (001) shifted to 16.9 Å.
- (c) 400<sup>9</sup>C. Same as 2c.

Sample 12.

- (a) 50% RH. Mont. (001) peak is broad and asymmetric to the right (14.7Å).
- (b) 60°C. Ethylene Glycol. Mont. (001) peak shifted to 16.7 Å.
- (c) 400°C. Same as 2c.

Sample 13.

- (a) <u>50% RH.</u> Mont. (001) peak is broad at 13.1 Å.
- (b) 60°C. Ethylene Glycol. Mont. (001) peak shifted to 17.0 Å.
- (c)  $500^{\circ}$ C. Sample was test fired between the usual runs of 400 and  $550^{\circ}$ C. to observe effect on 7 Å peak. Results show partial collapse

of Kaolin. Not due to Chlorite since there was no corresponding increase at 14 Å.

Sample 14.

- (a) 50% RH. Mont. (001) exceptionally broad. Shoulder at 11.6, 12.2 and wide peak at 14.0 Å.
- (b) 60°C. Ethylene Glycol. Mont. (001) shifted to 16.7 Å.
- (c) 400°C. Same as 2c.
- (d) 550°C. Same as 3d.

Sample 15.

- (a) 50% RH. Mont. (001) at 14.5°A. Peak asymmetric to right.
- (b) 60°C. Ethylene Glycol. Mont. (001) shifted to 15.5 Å.
- (c) 400°C. Same as 2c.
- (d) 550°C. Same as 3d.

Sample 16.

- (a) <u>50% RH.</u> Mont. (001) peak at 14.0 Å. Broad band. Asymmetric to right.
- (b) 60°C. Ethylene Glycol. Mont. (001) peak shifted to 16.7 Å.
- (c) 400°C. Same as 2c.
- (d) <u>550°C.</u> Same as 3d.

Sample 17.

- (a) 50% RH. Mont. (001) peak at 14.5 Å.
- (b) 60°C. Ethylene Glycol. Mont. (001) peak shifted to 17 Å.
- (c) 400°C. Same as 2c.
- (d) 550°C. Same as 3d.

Sample 18.

- (a) 50% RH. Mont. (001) peak at 11.3 to 13.1.
- (b) 60°C. Ethylene Glycol. Mont. (001) shifted to 16.4 Å.
- (c) 400°C. Same as 2c.
- (d) 550°C. Same as 3d.

Sample 19.

- (a) 50% RH. Mont. (001) peak at 14.0 Å. Asymmetric to the right.
- (b) 60°C. Ethylene Glycol. Mont. (001) peak shifted to 16.8 Å.
- (c) 400°C. Same as 2c.
- (d) <u>550°C.</u> Same as 3d.

Sample 20.

- (a) 50% RH. Mont. (001) peak at 12.9 Å.
- (b) <u>60°C. Ethylene Glycol.</u> Mont. (001) peak shifted to a double peak at 16.7 and 17 Å.

Sample 21.

- (a) 50% RH. Mont. (001) peak at 12.2 Å. Broad.
- (b) 60°C. Ethylene Glycol. Mont. (001) peak shifted to 16.7 Å.
- (c) 400°C. Same as 2c.
- (d) 550°C. Same as 3d.

Quantitative estimation of the clay mineral distribution is given in Table 2. It was obtained from the area under the basal spacings of each mineral after correction for diffraction effects was made. This was done on a nomograph (Figure 11) adapted from Hathaway and Carroll (8).

Expressing the clay mineral content as percent by weight of total sample and plotting the results as an areal correlation around East Bay the curves of Figure 12 were obtained. As expected, the fine particle size montmorillonite is the dominant mineral in the quiet areas north of Hanna Reef and along the north shore of East Bay. The coarser grain clay minerals predominate in the tidal current areas.

### C. Base Exchange Analysis

1. Procedure: Because X-ray data indicated East Bay clay mineral content as being fairly homogenous from sample to sample, only a few representative samples were analyzed.

# TABLE 2

# Quantitative Estimation of

# Clay Mineral Distribution in the

# -2 Micron Fraction

	Clay Mineral Component (Percent)		
	Montmorillonite	Illite	Kaolinite
Sample #1	50	25	25
Sample #2	65	15	20
Sample #3	45	25	30
Sample #4	45	25	30
Sample #5	50	30	20
Sample #6	35	25	40
Sample #7	45	30	25
Sample #8	45	25	30
Sample #9	30	45	25
Sample #10	45	30	25
Sample #11	45	30	25
Sample #12	35	30	35
Sample #13	45	25	30
Sample #14	45	40	15
Sample #15	40	30	. 30
Sample #16	35	35	30
Sample #17	40	30	30
Sample #18	35	45	20
Sample #19	40	35	25
Sample #20	50	25	25
Sample #21	35	40	25
Average	42.86	30.71	26.43

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FIG. 11. Nomograph for Estimating Sample Composition in Percent by Weight from the Basal Peak Areas





CLOCKWISE TRAVERSE AROUND EAST BAY

Approximately 2.5 grams of sample  $(150^{\circ}F.)$  are weighed in duplicate to .0001 grams and transferred to round bottom 50 ml centrifuge tubes provided with rubber stoppers. Each sample is dispersed in 30 ml of normal acetate solution and shaken on a mechanical shaking device for 1 hour at room temperature. The mixtures are allowed to stand for several minutes to permit settling. The solids adhering to the upper part of the tube are rinsed down with the clear supernatent liquid. The tubes are then centrifuged at 2500 rpm for 20 minutes and the clear liquid decanted from the well packed residue into a 250 ml volumetric flask. The residue is suspended in 30 ml of one normal ammonium acetate solution, and the shaking, centrifuging, and decanting operations repeated twice more.

The treated residue is suspended in 25 ml solox and shaken as described for 1/2 hour. The residue is separated by centrifuging at 2500 rpm for 20 minutes. The clear liquid is decanted and the residue treated three times more with Solox with alternate shaking and centrifuging as described.

The residue is suspended in 30 ml cold distilled water and transferred to a Kjeldahl flask. The amount of nitrogen is then determined by the standard Kjeldahl method and from the percent nitrogen, the B.E.C. in me/100 gram sample was calculated.

2. Results: As expected from X-ray data, the B.E.C. of the minus 2 micron fraction shows closely equivalent values. Table 3 shows that samples taken at diammetrically opposite ends of East Bay have nearly identical capacities.

Table 3. Base Exchange Capacity of Samples from the East and West Ends of East Bay

Sample No.	BEC, me/100g
2	64
10	69
11	68
12	62

From a diagentic viewpoint, this means that the alteration of dioctahedral and trioctahedral minerals is proceeding at the same rate at either extreme of the Bay. Likewise, it may be concluded that the important element in the distribution of the clay minerals is largely physical.

D. Differential Thermal Analysis

1. Procedure: Samples of the minus 2 micron fractions were dehydrated in a convection oven at 150°F. and lightly ground with a mortar and pestle to pass a 100 mesh screen. Ground samples were placed in the sample holder for analysis by techniques similar to those described by Grim. Furnace temperature was linerally increased at 10 degrees centigrade per minute up to 1000°C. Some samples were run in a nitrogen atmosphere for comparative purposes.

2. Results: The differential thermograms are of no apparent value in the study of recent sediments of the type found in East Bay. Peak temperatures and slopes are indicative of the thermograms obtained with random mixtures of dioctahedral and tri-octahedral clay minerals.

The use of a nitrogen atmosphere appeared to increase the intensity of the peaks but this, in the case of montmorillonite, is of less importance than the peak temperatures themselves and of the presence of asymmetry or shoulders. Nitrogen had the greatest effect on decreasing the exothermic reactions around  $350^{\circ}$ C.

Several examples are given in Figure 13.

### V. SUMMARY AND CONCLUSIONS

Variations in the type and distribution of the elay minerals are clearly reflected in the spectrometer patterns. For the most part, the East Bay region is fairly homogeneous with respect to preferential alteration of the clay minerals in any one sector. It is not known in fact whether this diagensis is a result of in







FIG. 13(b).

situ transformation or whether all or part of the Illite was carried in by terrestial streams and deposited by tidal currents.

It is fairly obvious that in the restricted area north of Hanna Reef and along the north shore, montmorillonite is especially abundant. Some evidence is presented that suggests the asymmetry of the montmorillonite (OOl) basal spacings toward the higher angles represents mixed layer minerals of random interstratification. It is probable that some of the montmorillonite is of the illitemontmorillonite type.

Chlorite was not positively identified by the methods used, however, the literature states that this does not rule out the possibility that an expandable type of chlorite may be present. To determine this possibility, complete chemical and exchangeable ion analysis would have to be run.

Base exchange capacity figures on the clay fraction appear in line with the amount of montmorillonite present.

Differential thermograms gave smooth average curves, making the use of this type of data of little value for the detailed study of recent sediments.

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