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Preliminary Study of Lake Agassiz Sediments

James D. Ross

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PRELIMINARY STUDY

of

LAKE AGASSIZ SEDIMENTS

by

James D. Ross

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ABSTRACT

The Agassiz sediments have been difficult to study because of their complex stratigraphy and homogeneous lithology. The mineral particle size distribution and the petrography of these sediments were the basis of a preliminary study which allowed a columnar section at Grand Forks to be divided into nine stratigraphic units. These are, proceeding from the bottom to the top: Unit 1, gravelly clay loam; Unit 2, gray clay with gravel; Unit 3, dark gray silty clay loam; Unit 4, grayish brown sand; Unit 5, dark gray clay; Unit 6, gray clay with gravel; Unit 7, dark gray clay; Unit 8, gray clay with silt; Unit 9, brown silty loam. The merits of X-ray mineralogical analysis, particle size distribution and also engineering techniques are evaluated concerning their usefulness in geologic work in glacial Lake Agassiz sediment. The X-ray analysis and particle size distribution were particularly good in determining minor lithologic variations in the sediment. The engineering and paleontological techniques were not as good because of the lack of application and development in studying glacial Lake Agassiz sediments. Future work in these areas may remedy this problem and give a clearer picture of the history of glacial Lake Agassiz.

INTRODUCTION

Purpose of Study

The purpose of this report is to present the results of a preliminary study of glacial Lake Agassiz sediments. A discussion of the methods used, and their value as future tools of study is evaluated.

Many surface studies have been made of the lake sediment (Laird, 1964), but extensive petrographic examinations of the lake sediments have not been made. The lack of such detailed work probably results from the many problems that exist in the lake basin. Some of these are: recognition of the subtle and at the same time complex lithologic changes occurring in both horizontal and vertical sections throughout the lake; association of the sediments with many possible environments; and the lack of good outcrops. All of these compound the difficulty of relating the sediments to the present ideas of Lake Agassiz history. To overcome these problems and at the same time test the feasibility of various methods two areas were studied.

Samples Studied

The most intensive study was carried out on samples from a drill hole 100 feet from the north end of Leonard Hall on the University of North Dakota campus in Grand Forks (for location see page 21 and figure 1). Other samples from the Grand Forks area were tested to determine if the Leonard Hall samples were representative of the sediments of this area. There were two locations both on the west bank of the Red River, one is located when the English Coulee merges with the Red, the other where the Red Lake River merges with the Red (see figure 1).

To determine if the methods used would effectively show differences in the sediment, some comparisons were made with drill hole samples taken from a site two miles north of the town of Pembina (see page 21 and Index

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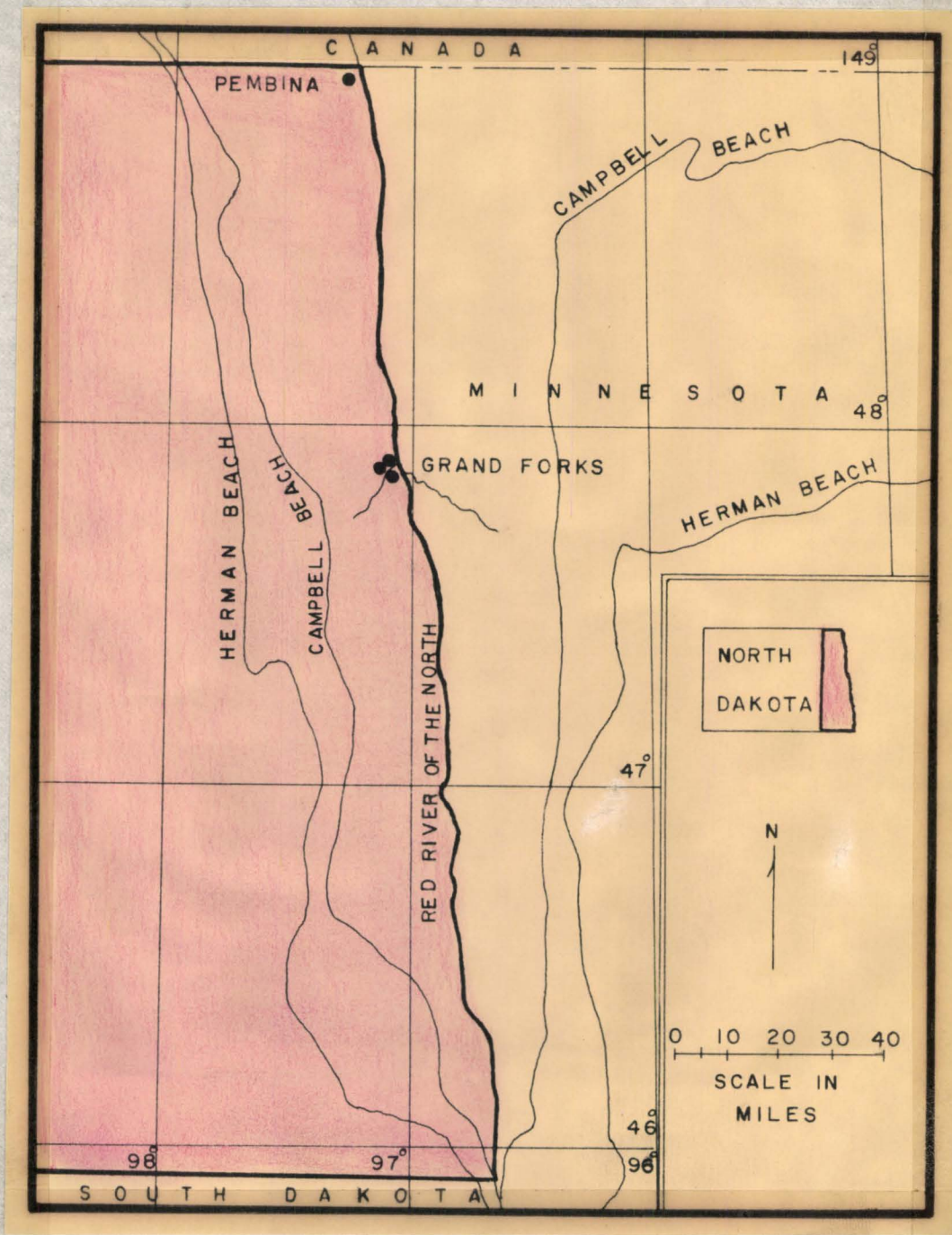
Figure 1. Index Map - Dots mark sample sites.

map figure 1).

Previous Work

Rominger and Rutledge (1952) divided the lake sediment into strati-
graphic units by the use of engineering techniques. The most useful pro-
perties were liquid limit, natural water content and preconsolidation
stress. With these methods a drying surface was determined, and the upper
clays were divided into lithologic units. An unconformity was established
between the upper laminated silt and the lower clays using these properties
and lithologic differences. The methods used in their work rely heavily
upon engineering properties of the sediment, while in this report other
methods of investigation are used to corroborate many of their findings
and has resulted in new information.

ACKNOWLEDGMENTS

samples from the Grand Forks area.

FIELD METHODS

All the core and thin-wall samples were taken by the Soil Exploration Company of Minneapolis, Minnesota. Only core samples from the top 150 feet of sediment in the Grand Forks area were studied. In coring, Soil Exploration Company uses a motorized drilling rig, which easily converts to a small pile driver to facilitate the driving of the core barrel. The auger is drilled the length of auger section (5 feet), it is then pulled out of the hole, a core barrel is connected to the auger shaft and it is driven downward until a one-foot core sample is obtained. After triming these samples are approximately 5 inches long and 1.5 inches in diameter. They are stored in wax-sealed jars to retain their natural moisture.

At Grand Forks in the upper 10 feet, the normal sample interval was 2.5 feet, below this, 5 feet. At 20-foot intervals, thin wall samples were taken. These are large diameter cores (2.5 to 3 inches) which are taken by slowly pressing a thin-edged tube into the sediment. This almost completely eliminates the shearing distortion present in the regular core samples.

The samples from 150 feet to 270 feet were grab samples which represent about five feet of section each. They were taken from a water well drilled by U. S. G. S. on the University Campus at Grand Forks (see table 1 and figure 1). In contrast, all samples at the Pembina site were grab samples. These samples were taken from the circulation mud pits of a water well drilling rig. The time that it takes for the mud to circulate from the pit to the bottom of the hole and back to the pit determines the location of the sample. The sample is caught in a screen at the surface before

it enters the mud pit. After completion of the hole, electric and resistivity logs are sometimes used to check the accuracy of the assigned sample depths. This technique was used at Grand Forks and Pembina sites. Logs were not run. These samples are dried and put in paper envelopes so that they will be available for further study.

LABORATORY METHODS

Petrography

The U. S. Bureau of Soils standard method of soil classification was used in this study. This classification is based on the texture of the soil. The texture of a soil is influenced by the percentages of the various sizes of particles in the soil. The soil particles are grouped into three size classifications as follows:

Particle-Size Classification	Diameter of Particles (Millimeters)	U. S. Standard Sieve
Sand	2.0 to 0.05	#10 to #270
Silt	0.05 to 0.005	(Cannot be separated
Clay	Smaller than 0.005	by sieving)

A soil can be grouped into twelve basic classifications according to the percentages of sand, silt and clay present in the soil. These classifications are shown by a triaxial graph; see Appendix, page 29.

X-ray Analysis

At Grand Forks and Pembina the mineralogy was determined by X-ray analysis. The samples used to determine bulk mineralogy were prepared in the following manner:

1. The cores were split lengthwise and a 40-gram sample, the shape of a triangular prism, was cut from the inside of the split core. The sample was thoroughly mixed to assure that the material would be represent-

ative, then carefully split in half by the cone and quarter method. One

half was wrapped in aluminum foil and returned to the sample bottle along

with the remaining core. From the remaining twenty grams, a five gram

split was heated for 24 hours at 60-80°C. The remaining 15 grams were

used for a size analysis so it was advantageous to calculate the water

content of the material at this time by weighing the five gram portion

before and after heating.

The procedure varies slightly when grab samples are used. Due to the

small amount of material, only one twenty-gram portion for X-ray and size

analysis was used. This assures that a representative sample will remain

in the North Dakota Geological Survey drill-hole files. The preparation

of the bulk sample for X-ray analysis is described in the Appendix, page 29.

The percentage of a mineral is determined from the X-ray analysis by

the height or counts per second at peaks characteristic of the mineral.

The peak heights are proportional to the amounts of the mineral present.

Therefore, the peak heights of a sample of unknown composition can be com-

pared to peak heights of a mineral with a known composition in order to

determine mineral abundances. From this the peak heights or counts per

second necessary for 100% composition of a mineral can be calculated by

a simple proportion.

The 100% composition values for calcite and dolomite were determined

by averaging the measurement of peak widths at one-half intensities of

selected varieties of these minerals. This was done because the crystal-

linity and grain size varies considerably in carbonate rocks, presumably

affecting the quantitative results of an analysis.

The X-ray values for calcite and dolomite were checked against a

chemical analysis of total carbonate (Herrin, Hicks and Robertson, 1958).

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The difference in values was acceptable; this averaged plus or minus 3 percent (Table 1).

The total clay values for 100% composition were determined by averaging the results of an analysis of the clay fraction from a silty clay at depth of 8 feet and a purer clay at 55 feet in the section at Grand Forks.

Crystalline quartz was chosen as a standard for quartz determination. Fairly consistent bulk mineralogy analysis under 100% was noted in the upper samples (to 35 feet). Samples from 35 to 150 feet were consistently over 100%. This could be accounted for by the fact that the values for 100% composition are based on an average of upper and lower clays. The upper clay values may be correct for the upper zone and the lower clay correct for the lower zone, thus the averaging of the sets of figures makes them approximately correct for both zones. The bulk mineralogy of the Pembina samples were consistently over 100%. Possible factors that could account for this are particle size variations, the types of clay minerals present and exposure to drilling mud. (See table 2 for values used in quantitative interpretation of minerals). The clay mineralogy of the Grand Forks site was studied by X-ray analysis of oriented slides. The slides were prepared in the following manner.

1. A suspension of distilled water and 1 gram of sediment from the 15 grams remaining from the size analysis portion of the sample is added to a small plastic vial. This fills it about half full. The mixture is agitated in the Spex Mixer/Mill to ensure that all of the clay is in suspension. There is a problem of the sediment flocculating in distilled water, but shaking in the hand is sufficient to reestablish the suspension long enough to make the analysis take a sample.

2. Allow about 30 seconds to pass, this will let the coarser fractions

TABLE 1

Comparison of the results of a X-ray and chemical analysis of calcite and
dolomite from five selected well sites and depths in Grand Forks area.
Samples 4201 and 4206 are from the Leonard Hall site. Sample 4213 and
4217 are from the English Coulee-Red River site and 4224 is from the
Red Lake River-Red River site See figure 1.

Sample	Mineral in %	X-ray	Chemical
4217-110'	calcite	13	10
	dolomite	9	6
	Total	22	16
4201-55'	calcite	5	6
	dolomite	5	5
	Total	10	11
4206-14'	calcite	4	3
	dolomite	23	26
	Total	27	29
4206-99'	calcite	13	9
	dolomite	23	20
	Total	36	29
4201-100'	calcite	7	8
	dolomite	18	20
	Total	25	28
4201-135'	calcite	7	8
	dolomite	11	11
	Total	18	19
4224-75'	calcite	2	2
	dolomite	13	12
	Total	15	14
4213-45'	calcite	3	4
	dolomite	6	7
	Total	9	11

TABLE 2

Intensity factors necessary for 100% composition used in X-ray quantitative interpretation of minerals in Lake Agassiz sediment at peak position in degrees 2 θ.

Minerals	Peak position in degrees 2 θ (Cu radiation)	Intensity factor (in counts per second per 100%)
Quartz	26.6	11500
Plagioclase	28	5000
K-feldspar	27.5	2200
Calcite	29.4	3200
	Av. 47.5 + 48.5	540
Dolomite	31	5000
Total clay	19.9	284
	34.6	180
	61.9	116

settle to the bottom, then withdraw approximately two milliliters by pipette.
Place this suspension on a one-inch diameter porous glass disk in order to
make the oriented slide. For best results rapid drying is essential, this
inhibits stratification in the sample, especially the clay and fine silt.
The disk is placed on a vacuum filter cup which is mounted on a vacuum
flask. The source of vacuum is an aspirator. A small heated blower and a
heat lamp will assist quick drying. There is also a problem of drying too
quickly, which causes the sediment on the disk to crack and curl. This
problem persists in silty sediments. Careful regulation of vacuum, fan and
heat lamp will help lessen this problem.

3. After drying, the disks are put in a humidifier at $72^{\circ}C$ for one
hour or may be stored there until they are X-rayed.

This technique will permit identification of kaolinite or chlorite, illite,
montmorillonite and mixed layered clays.

4. Glycolation of the samples enables positive identification of
montmorillonite by displacing water layers with ethylene glycol and ex-
panding the lattice to 17 $\overset{\circ}{A}$ for sodium montmorillonite or 15 $\overset{\circ}{A}$ for calcium
montmorillonite. The samples are glycolated by heating in a desiccator
with glycol for at least 1 hour at $60-80^{\circ}C$.

5. To distinguish between kaolinite and chlorite which have peaks
that coincide at 12.5°, the sample must be heated to $550^{\circ}C$ for half an
hour. This will destroy the kaolinite peak leaving the chlorite peak
(Schultz, 1964, p. 6).

Any quantitative assessment of clay mineralogy must be based on various
interpretations and assumptions regarding crystallinity, particle size and
mineral variations, thus greatly reducing the value of this type of inves-
tigation. In this writing only the general variations in abundance of the

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clay minerals will be discussed.

Size Analysis

A rapid method of determining sand, silt and clay, percentages developed by Dr. Frank Karner, (unpublished paper) consists of measuring the clay and the sand fractions, subtracting this from the entire weight of the sample to yield the silt fraction. A comparison of accuracy between this method and conventional methods was carried out by Harlan Friestad (unpublished paper). Differences were within plus or minus 10 percent. In conjunction with the size analysis, a test of disaggregation time was carried out. A 24-hour disaggregation time was found to be sufficient for complete disaggregation (see table 3).

The size analysis method is described in the appendix, page 36.

Engineering Methods

The only engineering method used was the "blows per foot" as determined from Soils Exploration Company's logs. A "blow" consists of dropping a 140-pound weight from a height of 30 inches. An increase in blows per foot indicates an increased strength of the soil.

Paleontology

The use of Ostracods as a geologic tool is somewhat hampered by the wide range of conditions under which Ostracods can exist. In general, the fresh-water Ostracods' food consists of diatoms, bacteria, and organic detritus. They can live in the following environments: (1) temporary ponds and ditches (2) permanent lakes and swamps (3) temporary streams and pools left in stream beds after flow has ceased, (4) permanent streams of all sizes and (5) underground water.

TABLE 3

Variations in particle size distribution with increased disaggregation time and various stirring intervals.

Sample B-1

Time of dis-aggregation:	24 hrs.	24 hrs.	76 hrs.	192 hrs.
Procedure:	stirred 5 min. every 8 hrs.	stirred 5 min. every 24 hrs.	stirred 5 min. every 24 hrs.	stirred 5 min. every 24 hrs.
Sand %	1	1	1	1
B-1 (a) Silt %	70	67	69	64
Clay %	29	32	30	35
Sand %	1	1	1	Not evaluated
B-1 (b) Silt %	69	68	70	
Clay %	30	31	29	

The temperature of the water has little effect on distribution. Relatively few species are affected by the type of bottom.

Candona were found in the glacial Lake Agassiz sediments; the specimens were identified by L. D. Delorme (written communication), Department of Energy, Mines and Resources, Alberta. The genus Candona prefers a mud bottom, probably because of its crawling locomotion. These Ostracods are confined to still waters occurring primarily in plant zones, where wave action is not pronounced. Candona is not seasonal as are many fresh water species. The evidence of shell assemblages could indicate a lake bottom. (Benson and others, p. Q 210-211).

SEDIMENT CHARACTERISTICS

Major Sedimentary Units

The sediments studied from the area have been divided into nine units, the determination of which is based on visual methods. Table 4 compares the general lithology of a Grand Forks section determined by Rominger and Rutledge (1952) with the one taken at Leonard Hall by Soil Exploration Company. Plate I shows the comparative thickness of the units diagrammatically. It is not possible to determine if the sediment is stratified below Unit 5 because only grab samples are available for study. Figures 2, 3, 4, 5, 6, 7 typify color and structure in the upper units.

A stratigraphic section for the Pembina well was not drafted because the sample interval was too great for accurate unit determinations. This site is compared with the Grand Forks site on a particle size and mineral-ogical basis.

Mineralogical Variation

The mineralogical variation in the Grand Forks section is shown on

TABLE 4

Lithologic chart comparing the Leonard Hall section with Rominger and Rutledge, (1952), section at Grand Forks.

Unit	Leonard Hall Grand Forks, N.D.	Grand Forks, N. D. (after Rominger and Rutledge, 1952)
9	Brown and gray brown silty loam to clay with laminations. 20 feet	Brown clay and silt, stratified 20 feet
	unconformity	*unconformity*
8	Gray clay with some lenses of silty loam & Ostrocod shells in lower section 17 feet	Dark blue clay mostly non-stratified. 1ift
	old drying surface	*old drying surface*
7	Dark gray clay, weathered carbonate pebbles, nonstratified. 48 feet	Dark-blue clay, mostly non-stratified, but with some inter-bedded silt at top. 9 feet
6	Gray clay with a little gravel, nonstratified. 39 feet	Dark-blue or black clay, non-stratified, many slicken-side surfaces, high liquid limit and high natural water content. 25 feet
5	Dark gray clay, weathered carbonate pebbles. 18 feet	Dark-blue clay with calcareous concretions, nonstratified. 8 feet
		unconformity
4	Grayish brown sand with limonite concretions. 17 feet	
3	Dark gray silty clay loam undetermined if stratified. 50 feet	Drift 73 + feet
2	Gray clay with a little gravel 34 feet	
1	Gravelly clay, loam. 32 feet	

Figure 2. Unit 6 from Leonard
Hall, Grand Forks.
Sandy gray clay with
a little gravel.
Does not appear strati-
fied. Sample depth
84 feet.

Figure 3. Unit 7 from Leonard
Hall, Grand Forks.
Gray clay with weathered
carbonate pebbles.
Does not appear strati-
fied. Horizontal lines
knife scrapings.
Sample depth 63.5 feet.
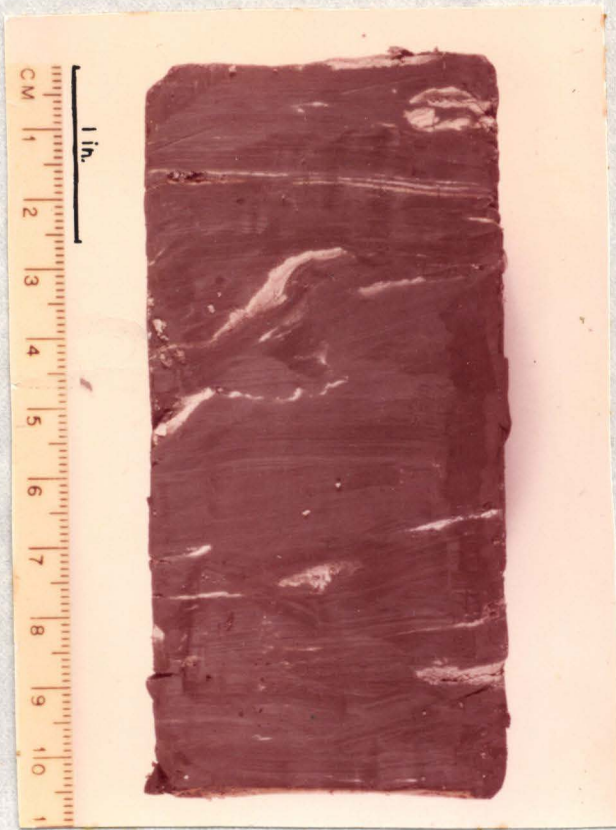
Figure 4. Unit 8 from Leonard Hall, Grand Forks. Gray clay with some lenses and laminations of silty loam and a few lenses of dark gray silty clay. Sample depth 24.5 feet.
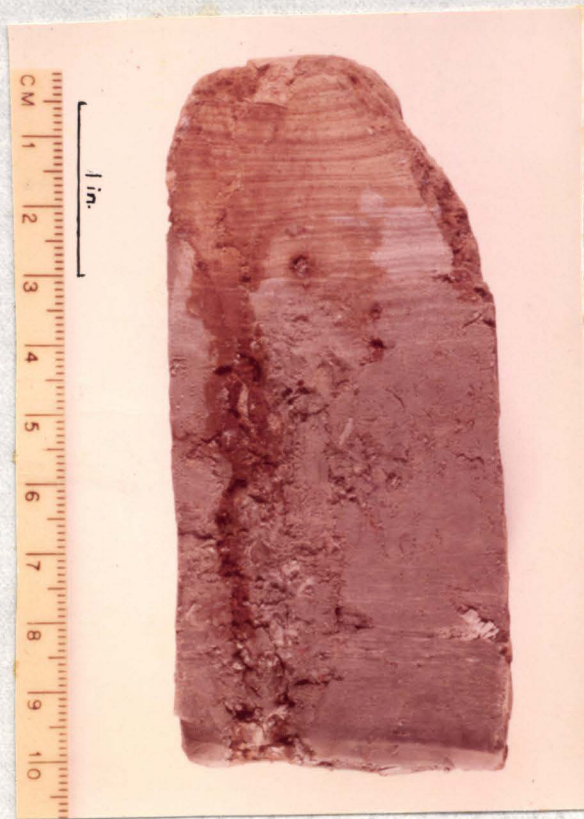
Figure 5. Unit 9 from Leonard Hall, Grand Forks. Gray and brown mottled clay with lenses and laminations of silt. Shows limonite staining and an elongate limonite concretion on the left side. Sample depth 14.5 feet.

Figure 6. Unit 9 (upper section) from Leonard Hall, Grand Forks. A brown and light grayish brown mottled silty clay with lenses of silt and silty clay loam. End view of a limonite concretions can be seen to the right of the center of the core. Sample depth 8 feet.

Figure 7. Unit 9 (upper section) from Leonard Hall, Grand Forks. Randomly oriented, hollow, elongate limonite concretions are common in the silty clay. Sample depth 8 feet.

Plate I. Important aspects of the variation can be summarized as follows:

1. The total carbonate and quartz minerals show sharp increases in the coarser units. Good examples of this are Units 1, 2, 3, 5, and 9.

2. Total clay, the largest constituent, varies inversely with the quartz and total carbonate curves. In Unit 9 Plate I clay values are low, 35 percent, reaching a maximum of 90 percent in Unit 7, then they gradually decrease with minor fluctuations to 35 percent in Unit 1.

3. The mineralogy as a whole is quite sensitive to lithologic variation reflecting the different units by large composition changes. A good example of this is in Unit 6 at the 95-foot level where there are sharp mineral changes that were not detected optically.

4. The core at Pembina has nearly straight-line values for all minerals except total clay, following the same trends noted in the upper 70 feet of the Grand Forks well.

The particle size analysis in many ways reflects the bulk mineralogy this enables the association of a certain particle size distribution with a certain mineral distribution. The quartz and carbonate curves exemplify this by shadowing the sand curve. Plate 1 The composition of the sand is accounted for by quartz and carbonates with small contribution from the feldspar.

The clay size percentage is of the same magnitude as the total clay minerals which indicates that the clay-size sediment is predominately clay mineral. The clay-size curve is smoother and probably more accurate than the total clay curve, due to the problem in determining 100 percent values for clays.

Clay minerals were determined for Unit 7 of the Grand Forks well. The relative values are illustrated by a tracing of the peak of montmorill-

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onite, illite and kaolinite. They generally vary as follows:

1. Relative peak heights of calcium montmorillonite increases towards the top of the section.

2. Although illite has a low peak, it represents a significant portion of the clay minerals and remains fairly constant throughout the section.

3. Kaolinite peaks fluctuate somewhat. They generally increase with depth. Heat treatment of the sample revealed the presence of chlorite. It accounted for less than 20 percent of the kaolinite peak.

This initial examination of the clay minerals indicates that they may be a useful tool in further studies of glacial Lake Agassiz sediments. These general trends may be indicative of environmental or depositional changes in the lake. Much more work must be done to explore these relationships to see if they exist. Data of the mineralogy is in Table 5.

Particle Size Variation

The following general observations were noted in the particle size distribution analysis:

1. The sand fraction is a good indication of stratigraphic units; any minor increase or decrease stands out in the clay units.

2. The silt portion usually follows the sand with coinciding increases and decreases.

3. The clay fraction, the largest size constituent, reflects the sand-free lake clays by consistent values over 60 percent.

The silt fraction increases from 30 percent, the lower part of Unit 9 to 70 percent, the upper sections. This marked increase in silt may be indicative of a drying zone. This is further substantiated by a silt increase

TABLE 5

Particle size distribution and Bulk Mineralogy Data of three Grand Forks sites and one Pembina site.

Location: 100 feet from north end of Leonard Hall on the University of North Dakota campus, in Grand Forks, North Dakota; SE ½, Sec. 5, T. 151 N., R. 50 W. Elevation 830 feet.

Sample Depth (Feet)	Particle Size			Percentage of Mineral Content							
	Sand	Silt	Clay	Quartz	K-feldspar	Plagioclase	Calcite	Dolomite	Total Clay	Total	Absorption Coef.
7.5	3	72	25	27	6	6	1	18	37	95	45
13.5	3	29	68	14	4	6	3	11	63	101	44
25	1	25	74	14	3	4	3	7	75	106	44
35	1	20	79	18	5	5	1	7	71	108	49
45	1	9	90	11	2	4	1	4	84	106	48
50	1	31	68	8	3	5	2	3	84	105	47*
55	1	26	73	8	1	3	3	3	89	107	49
60	1	25	74	8	3	4	4	3	90	111	47*
65	3	21	76	9	3	3	8	8	72	103	47*
75	12	28	60	18	5	5	8	15	48	99	48
85	25	28	47	26	4	9	8	14	47	104	48
95	26	29	45	22	4	5	10	27	31	99	47
100	23	48	29	27	5	7	7	17	43	106	49
105	28	29	43	29	5	8	10	20	41	113	48
115	27	28	45	26	5	6	7	14	35	93	43
125	28	29	43	26	4	8	9	15	39	101	47
130	4	38	58	—	—	—	—	—	—	—	—
135	3	44	53	18	4	6	7	10	61	106	48
158-163	7	55	38	8	4	8	6	5	58	89	45
178-183	1	77	22	11	5	8	7	6	50	87	47
188-193	8	62	30	13	5	7	6	7	63	101	46
208-213	23	36	41	18	4	6	7	4	48	87	47*
243-248	48	26	26	30	7	9	8	11	33	98	48
268-273	49	26	25	28	5	10	9	8	44	104	48

Location: 1200 feet east of bridge that crosses the English Coulee on State Mill Road near the west bank of the Red River in the center of S ½, Sec. 28, T. 152 N., R. 50 W. Elevation 828 feet.

Sample Depth (Feet)	Sand	Silt	Clay	Quartz	K-feldspar	Plagioclase	Calcite	Dolomite	Total Clay	Total	Absorption Coef.
14	1	68	31	21	4	6	2	22	42	97	44
50	1	5	94	9	2	3	2	4	85	105	49
100	27	38	35	29	5	8	9	21	36	108	46

Location: 200 feet south of the Junction of the Red Lake River with the Red River and 150 feet up the west bank of the Red River in SE ¼, Sec. 2, T. 151 N., R. 50 W. Elevation 879.3 feet.

Sample Depth (Feet)	Sand	Silt	Clay	Quartz	K-feldspar	Plagioclase	Calcite	Dolomite	Total Clay	Total	Absorption Coef.
15	4	51	44	24	5	6	4	9	45	93	45
45	1	11	88	9	2	4	3	5	85	108	50
110	64	18	18	48	11	15	7	6	15	102	43

Location: 2 miles north of town of Pembina in a drainage ditch between the Great Northern Railway and the former Highway 81 in the center of the S ½, Sec. 28, T. 151 W., R. 164 N. Elevation 720 feet.

Sample Depth (Feet)	Sand	Silt	Clay	Quartz	K-feldspar	Plagioclase	Calcite	Dolomite	Total Clay	Total	Absorption Coef.
10-15	1	62	37	23	4	6	2	15	60	110	46
31-37	1	9	90	12	3	6	2	7	71	102	45
58-63	1	1	98	10	3	5	2	4	89	113	49
79-84	1	4	96	11	4	4	3	5	86	113	50
100-105	1	4	95	15	4	6	4	7	74	110	49
128-131	1	16	83	17	5	7	4	7	67	107	48
137-142	49	22	29	23	8	11	6	30	22	100	47
184-189	51	27	22	20	5	11	6	31	16	89	47

* Average of Absorption factor of samples above and below.

at a depth of 50 feet which Rominger and Rutledge (1952) recognize as such a zone.

Three more silt zones are recognized at depths of 100, 135 and 100 feet. Care must be taken not to extend the interpretation too far; there are many other factors that could explain the presence of silt zones; see Plate I and Table 4 for graphs and values of particle sizes.

Engineering Interpretations

The data for "blows per foot" were taken from a Soil Exploration Company log at Grand Forks (Plate I). Several other logs were examined to ascertain that the data were not anomalous with other wells in the area. The increase of "blows per foot" to a maximum of 13 at a depth of 45 feet indicates compaction of the sediment. Below this there is a sudden decrease to an average of 5 blows per foot for the rest of the Unit 7. This compacted zone has been recognized by Rominger and Rutledge (1952) as a drying zone and was recorded as far as Crookston, Minnesota and Fargo, North Dakota.

Paleontological Determinations

The use of Ostracods as indicators of environment is somewhat restricted by the varied habitats in which even a single species may exist. Shell fragments were found at the drying zone by Rominger and Rutledge (1952). They state that sedimentation must have stopped long enough to allow this assemblage of life to thrive and then it commenced with the deposition of the same type of material because there are no lithologic changes.

Ostracods are present in the upper part of Unit 9 and at only one point below this, suggesting that they may be used as stratigraphic indicators.

EVALUATION OF ANALYSIS

Validity of Techniques

The U.S. Bureau of Soils Classification was used to describe lithologies. This was convenient since the Soil Exploration Company samples were classified under this system and comparisons could be made. Color determinations were made according to the Munsell Soil Color Chart.

The particle size analysis, although not a standard procedure, has been checked by Harlan Friestad and compared with the North Dakota Geological Survey Standard Procedure A-65 (Lee Clayton, unpublished paper). Friestad found that the size division into sand, silt and clay was sufficient to show significant differences in the sediment and served as a partial basis for the assignment of units.

Quantitative X-ray analysis was based on heights of peaks on diffractometer traces of unoriented powder disks using an empirical absorption-correction method. This is a standard X-ray technique for quantitative data. A chemical determination of calcite and dolomite was made and compared to the X-ray analysis with average deviation of 3 percent.

The engineering data used are taken from Soil Exploration Company's logs and are considered a property of soil strength.

The use of Ostracods to determine drying surfaces is based on the fact that the genus Candona is benthonic and that assemblages of shell fragments might represent changes in the lake environment. Shell fragments are found at only one point below this level which strongly associates them with a nondepositional environment, indicating that the lake may have stabilized temporarily at this time.

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Interpretation

Many possible interpretations of the glacial Lake Agassiz sediments are open to consideration at this point. The study of only one section permits only tentative evaluation. Regardless, an attempt has been made to relate each unit to a depositional environment.

Unit 1 is a typical hard till with angular to well-rounded gravel and sand, having approximately equal proportions of sand, silt, and clay particles.

Unit 2 is a sandy gray clay with less gravel than Unit 1. It is possibly a water-laid till.

Unit 3 appears to be a continuation of the previous decrease in coarse particle sizes from Unit 2. A lake environment may have existed at this time. The amount of sand in this unit corresponds to the amount of sand in the upper sediments (60 ft.), but silt is more abundant and clay less abundant. The upper sediment is generally considered to be a lake deposit. If this is lake sediment, the type of deposition or the origin of the sediment was quite different from the upper lake sediment because of the different silt-clay ratios.

Unit 4 is a sand deposit of fluvial or glacio-fluvial origin. This sand Unit is less than 20 feet thick and appears to be restricted in lateral extent because it is present in only one other well in this area two miles from the Leonard Hall well. These bodies do not appear to be connected.

Unit 5, a gray clay, is only about 20 feet thick. This and unit 7 have similar physical and mineralogical properties suggesting that they are related. These units may represent lake sediment, and the intervening unit 6, which has some gravel in gray clay, may be a lake-deposited till (Edinburg Moraine).

Near the top of Unit 7 is a probable drying surface. This may be the

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first of many fluctuations of the lake level that caused the laminations

of silt between thicker beds of clay in Unit 8.

Unit 9, a complex mixture of sand, silt and clay laminations with

limonite concretions and staining, strongly suggests a fluvial deposition

with probably most of the source material coming from rivers draining into

the lake after it had become swampy and shallow.

Three major primary source areas for the sediments are evident: granitic

shield rocks, Paleozoic carbonates, and Cretaceous shales. The source of

the sediments may be revealed by their mineralogical composition, and by

working from the part to the whole we may be able to apportion a certain

amount of sediment to each possible source.

The Pierre shale, the underlying bedrock in this area, has a typical

composition of 3 percent feldspar, 20 percent quartz, and 78 percent total

clay (Schultz, 1964). If the assumption is made that the total clay in the

lake sediment is contributed by the Pierre shale, a semi-quantitative inter-

pretation of the source of the sediment can be made.

Unit 1 will be used as an example because it is possible for all the

other sediments to be derived from it or related to it in a systematic way.

Unit 1 has a composition of 39 percent total clay, 29 percent quartz, 15 per-

cent feldspar and 18 percent total carbonate. If all the clay in Unit 1 is

attributed to Pierre shale, this would proportionally eliminate all of the

clay, 35 percent of the quartz and 2 percent of the feldspar. The 18 percent

total carbonate can be attributed to the Paleozoic carbonate source. This

leaves 19 percent quartz and 13 percent feldspar unaccounted for. The

remaining minerals do not approximate a granitic composition which should

be roughly 30 percent quartz and 60 percent feldspar. The granitic rocks

in the source areas may have been weathered, changing the feldspar to

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kaolinite, and illite. Thus, granitic rocks may also be contributors to the clay minerals along with the shales.

Further details of the origins would entail a thorough study of clay mineralogy and a comparison of Pierre shale and glacial Lake Agassiz sediments to determine the amounts of kaolinite and illite present in each.

CONCLUSION

In this preliminary study of glacial Lake Agassiz nine sedimentary units were established on the basis of mineralogy, particle size distribution, and, to a lesser degree, by paleontological and engineering techniques.

The methods used were quite sensitive to the lithologic changes in the sediments. The particle size distribution was particularly useful for the establishment of units, although, the classification was only the basic sand, silt and clay distribution.

Quantitative mineralogy of the sediment is useful in establishing petrographic distinctions and in a consideration of source materials.

Engineering methods are considerably useful in working with lithologically homogeneous sediments and are often overlooked as geologic tools.

The study of drill hole samples is one of the best ways to determine the general stratigraphy and hence the history of the lake. At the present, lack of drill hole samples and a lack of means to interpret those that are available, has curtailed such investigations.

Future

Further detailed studies of clay mineralogy will probably yield much information about glacial Lake Agassiz sediments, and may establish source areas of the sediments.

A complete particle size distribution analysis may ascertain the environ-

ment of deposition. This avenue of study has many possibilities; a close sample interval may reveal lithologic changes in the presumably homogeneous clays in the upper units that are so difficult to detect.

Another method that might be useful in working with these clays is an actual hospital type X-ray photo of a thin slab which would show structure not visible to the naked eye.

The problems posed by each unit in this report are numerous. Perhaps, the best way to approach the problems of glacial Lake Agassiz, because of the vast expanse it once covered, is to establish stratigraphy of the lake in a general way and then go back and select specific details necessary for a complete interpretation.

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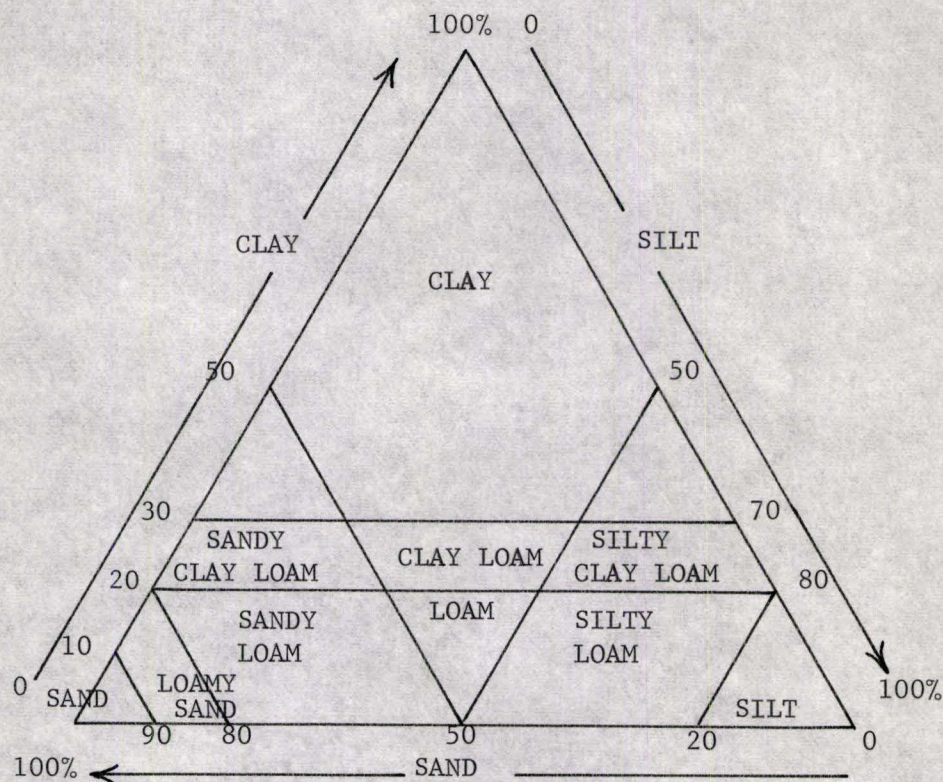   Geological Survey Professional Paper 391-C, pp. 7

APPENDIX

U. S. BUREAU OF SOILS CLASSIFICATION SYSTEM

In tabular form this classification is as follows:

BASIC SOIL CLASSIFICATION	SAND	SILT	CLAY
Sand	90-100%	0-10%	0-10%
Loamy sand	80-90	0-20	0-20
Sandy loam	50-80	0-50	0-20
Loam	30-50	30-50	0-20
Silty loam	0-50	50-80	0-20
Silt	0-20	80-100	0-20
Sandy clay loam	50-80	0-30	20-30
Clay loam	20-50-	20-50	20-30
Silty clay loam	0-30	50-80	20-30
Clay	0-40	0-50	30-100
Sandy clay	50-70	0-20	30-50
Silty clay	0-20	50-70	30-50

The sand present in a soil is classified as coarse (#10 - #20 sieves), medium (#20 - #40 sieves), fine (#40 - #100 sieves), very fine (#100 - #270 sieves), or as well graded. Soils with an appreciable amount of gravel present are classified "with a little gravel" (less than 15%), "with some gravel" (15 to 30%), "with gravel" (30 to 50%), "and gravel" (over 50%). Particles over 3 inches are classified as boulders. Organic soil is classified as "peat" (over 2/3 organic material) or "muck" (1/3 to 2/3 organic material).

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GUIDE FOR X-RAY DIFFRACTION ANALYSIS
by
Dr. Frank Karner (unpublished paper)

X-ray Analysis

A. Splitting and Grinding (for loose sediment modify according to
    particle size; for small samples modify according to sample size):

    1. Retain a large peice for a hand specimen and a chip for a thin
        section. Take several small representative fragments, about
        200 grams, from different parts of the specimen(s). It is
        important to take representative material, so select at random
        using as large a number of fragments as possible.

    2. Crush all material in a large porcelain mortar to fragments less
        than 1/2-inch in diameter and split sample in half by the cone-
        and-quarter method. Wrap half of the sample in aluminum foil
        and label and save (in plastic bag, if material is naturally
        moist). This material may be used for particle size analysis
        and an appropriate amount may be separated through splitting
        at this time.

    3. Crush all material in a 100 gram sample to less than about
        one-eighth inch in diameter. Dry first, if necessary. It will
        should easily pass through the Jones-type splitter. Split sample
        and save one-half as in preceding step.

    4. Crush all material in sample to less than 20 mesh. Using Jones
        splitter, obtain carefully about 4 cc for X-ray determination
        of bulk mineral composition. Save remainder as in preceding
        steps.

    5. Grind the 4 cc sample in the Spex Mixer/Mill (vial number 5004)
        two minutes. Carefully clean all material from vial and carefully

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split 0.8 gram from the total ground sample using the Jones-type splitter. Be certain the splitter is clean and that all material passes through it. Save and label remaining ground material as in preceding steps.

6. Brush as much of the 0.8 grams as possible through a 325 mesh screen with a moderately stiff brush (typewriter eraser brush). In 3-4 minutes half of the material should pass the screen. Grind the coarse remainder for about 5 minutes in a mullite mortar. About half of this should now pass the screen after brushing. Repeat the process of grinding and brushing being certain to regrind all of the coarse material even that trapped between the nylon mesh and the frame. This can be removed by tapping the side of the sieve on the table. Finally, grind any remaining coarse material for 5 minutes in the mortar and add it to that which has been brushed through the screen. Place the 0.8 grams in a plastic vial number (6133) with four small plastic mixing balls and mix in the Spex Mixer/Mill for 2 minutes. The representative, homogeneous sample will be comprised of particles approximately 44 microns in size (usually 30 microns mean particle size for coarse grained materials and finer for fine-grained materials).

B. Packing of Samples in Rotating Holder

After mixing, the vial is overturned on a piece of creased clean paper and tapped so all the material falls out. After the balls have been carefully picked out of the pile with a tweezer, disrupting the pile as little as possible, the material is dumped into the fiber holder (for the standard Philips rotating specimen holder) which has been

placed on a clean glass slide, slotted end up. A spatula is then used
to spread the material evenly in the holder. A properly fitting, smooth-
bottomed vial is then inserted into the back holder and pressed down
very firmly for approximately seven seconds. The pressure is released
on the vial but the holder still pressed on the glass slide for several
seconds. The vial is slowly removed and the holder with its sample is
taken off the glass slide.

C. X-raying of Sample

Do not use the X-ray equipment without explicit permission for each
session. Fill in record book properly.

1. Insert sample holder in rotating specimen holder <u>with beam
   stop in</u>. Tighten set screw with upper surface of sample
   flush with top of rotating specimen holder and replace
   shield.

2. Run sample (usually from 2° to 63° 2θ) under the following
   machine conditions for:

   X-ray Generator (Philips constant potential)

   X-ray Tube (Machlett Cu tube-short anode) 45Kv, 17ma

   Diffractometer (Philips high angle)

   1° /minute scan speed

   1° divergence and anti-scatter slits

   0.006" receiving slit

   Ni filter

   Detector (Philips scintillations, transistorized)

   1 KV

Circuit Panel (Philips)

PHA, width 9V, level 7V

linear scale

1 sec. time constant

1×10^3 counts/sec full scale (rerun off-chart peaks

at appropriate scale, 2×10^3 , 5×10^3, etc.)

Recorder (Bristol, for Philips type circuit panel)

30"/hr. chart speed

MEASUREMENT OF ABSORPTION
by
Dr. Frank Karner (Unpublished paper)

I. Introduction

Correction for absorption is a practical necessity for quantitative analysis by X-ray diffraction techniques. Alexander and Klug (1948) described the basic relationship between diffraction and absorption (Leroux, et al., 1953). An equation expressing the relation as modified by Leroux, et al., (1953) was developed:

$$X_1 = \left(\frac{I_1}{{I_1}_o}\right)\frac{\mu s*}{\mu 1*}$$

where

X_1 = weight fraction of component 1

μ 1* = mass absorption coefficient of component 1

μ s* = mass absorption coefficient of powder sample

I_1 = intensity diffracted at a definite Bragg angle 2 by a crystalline component 1

$(I_1)_o$ = intensity diffracted at a definite Bragg angle 2 by pure crystalline component 1

This formula shows that the weight percentage of component 1 is equal to the ratio of the intensities of the diffraction peaks for component 1 in the sample and pure component 1 after correction for absorption by multiplying by a factor, I.e. the ratio of absorption coefficients for the sample and pure component 1. Leroux, et al., (1953) further established the following relationship in order to experimentally determine the absorption correction:

$$\frac{\mu s*}{\mu 1*} = \frac{P_1 \log (T_s/T_o)}{P_s \log (T_1/T_o)}$$

where

ρ_1 = apparent density of a pure sample of component 1

ρ_s = apparent density of a sample containing weight fraction X_1 of component 1

T_o = intensity of incident X-ray beam

T_1 = intensity transmitted by pure sample of component 1

T_s = intensity transmitted by sample containing weight fraction

The various transmitted intensities are measured and used along with densities of the powders to determine the above absorption ratio which in turn is used as a correction factor for the preceding equation.

II. Measurement

Transmitted intensities are measured using standard Philips holders partly filled to known relative density. These are placed in front of the Ni-filter on the Philips high angle diffractometer and held by a specially designed bracket probably similar to that mentioned by Niskanen (1964). Following Lennox (1957) and Niskanen (1964) the X-ray beam is monochromatized with a sample of ground quartz in the normal sample position and using the 26.7^o 2θ peak with Cu radiation.*

III. Application

X-ray transmission is measured for standard pure mineral samples and used with transmission values obtained for unknown samples to obtain the ratio which is used as the correction for absorption. This is applied by multiplying the ratio $I_1/(I_1)_o$ or the weight percent (X_1) by $\frac{\mu s*}{\mu 1*}$ to give the absorption-corrected weight percentage.

* 4° divergence and anti-scatter slits are used.

Partical Size Analysis Procedure

The semi-quantitative evaluation of clay mineral composition of clay fraction and separation of sand and silt fractions is determined by a procedure designed to obtain representative sand, silt and clay fractions of gravel-poor sediments or rocks which can be easily disaggregated. It will give sand, silt, clay and gravel and pore water percentages.

Pore Water (and Gravel) Content

Weigh the sample to 0.1 gm and dry for about 24 hours at $60\text{-}80^{\circ}$ C. Allow to cool and weigh to nearest 0.1 gm. Weight loss represents pore water content of sample. To obtain gravel content wet sieve the sample through a sieve with 2.00 mm openings. Weigh sieve fraction to nearest 0.1 grams and compare with weight of dried sample to get percentage of gravel.

Disaggregation

1. Weigh sample, estimating to 0.01 gm.

2. Using standard solution of calgon (North Dakota Geological Survey) Standard Procedure A-65) and distilled water prepare 500 ml. of suspension of water, sand, silt, clay and 2 grams of Calgon in 600-1000 ml. beaker. If gravel is present in the sample it may be removed by first dry sieving and then wet sieving with the water to be used for disaggregation. Dry and weigh gravel. Stir suspension for about 5 minutes with magnetic stirrer using as rapid stirring as possible. Let stand for about 24 hours. Stir again for 5 minutes. Repeat stirring and soaking process if disaggregation is not complete.

Clay Determination

Let the suspension stand 75 minutes after above stirring. If sediment

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flocculates (North Dakota Geological Survey, Standard Procedure A-65) follow
recommendation in that procedure. If sediment does not flocculate withdraw
25 ml with pipette at depth of 4 cm. Dry this portion at 60-80° C. and
weigh to nearest .01 gm. Subtract weight of Calgon in 25 ml of suspension.
Multiply by 20 to get total weight of clay in sample. Save out 25 ml of
2 clay fraction by removing a two ml portion with a pipette from a depth
of 2.5 cm after 50 minutes additional settling time. Place suspensions onto
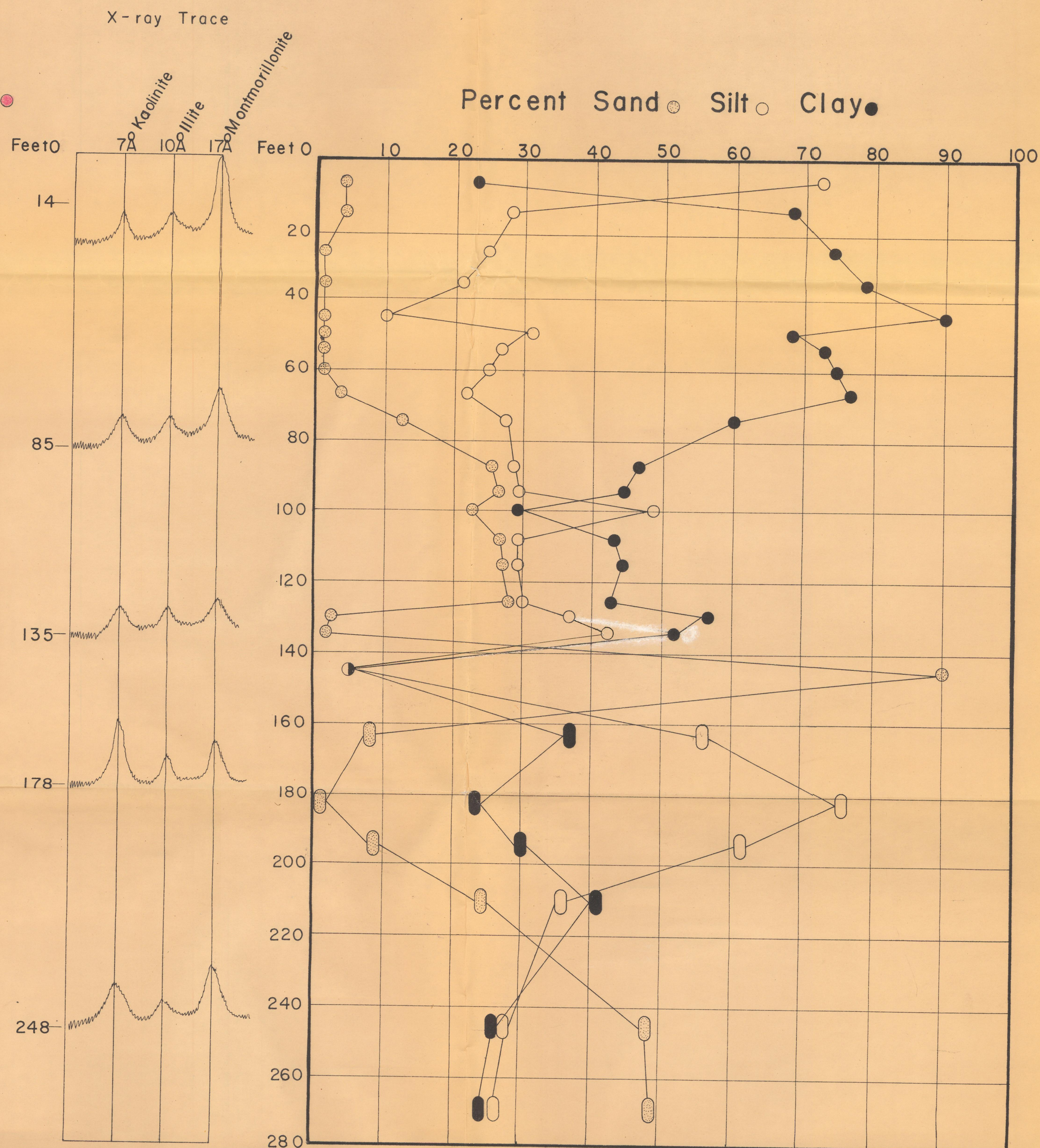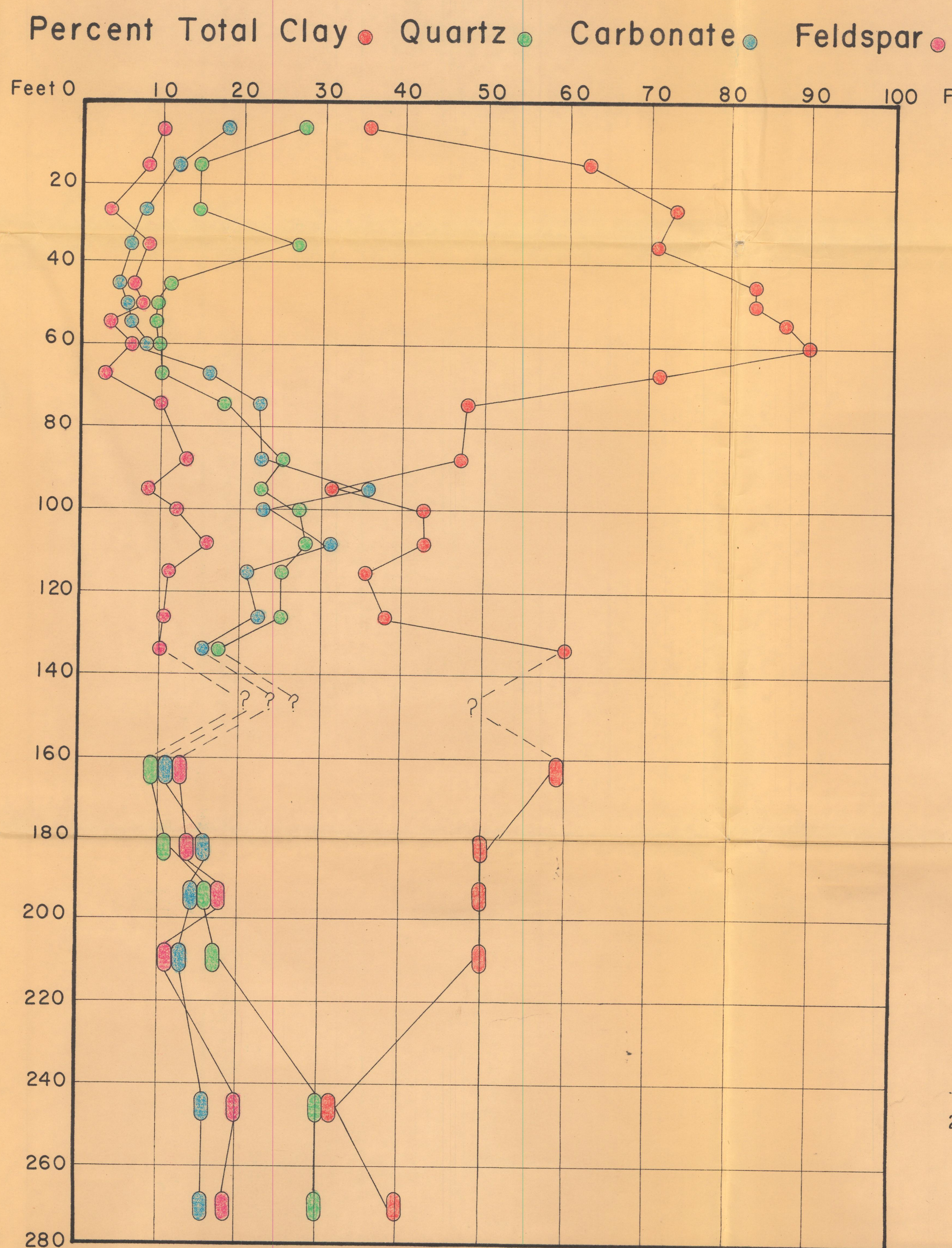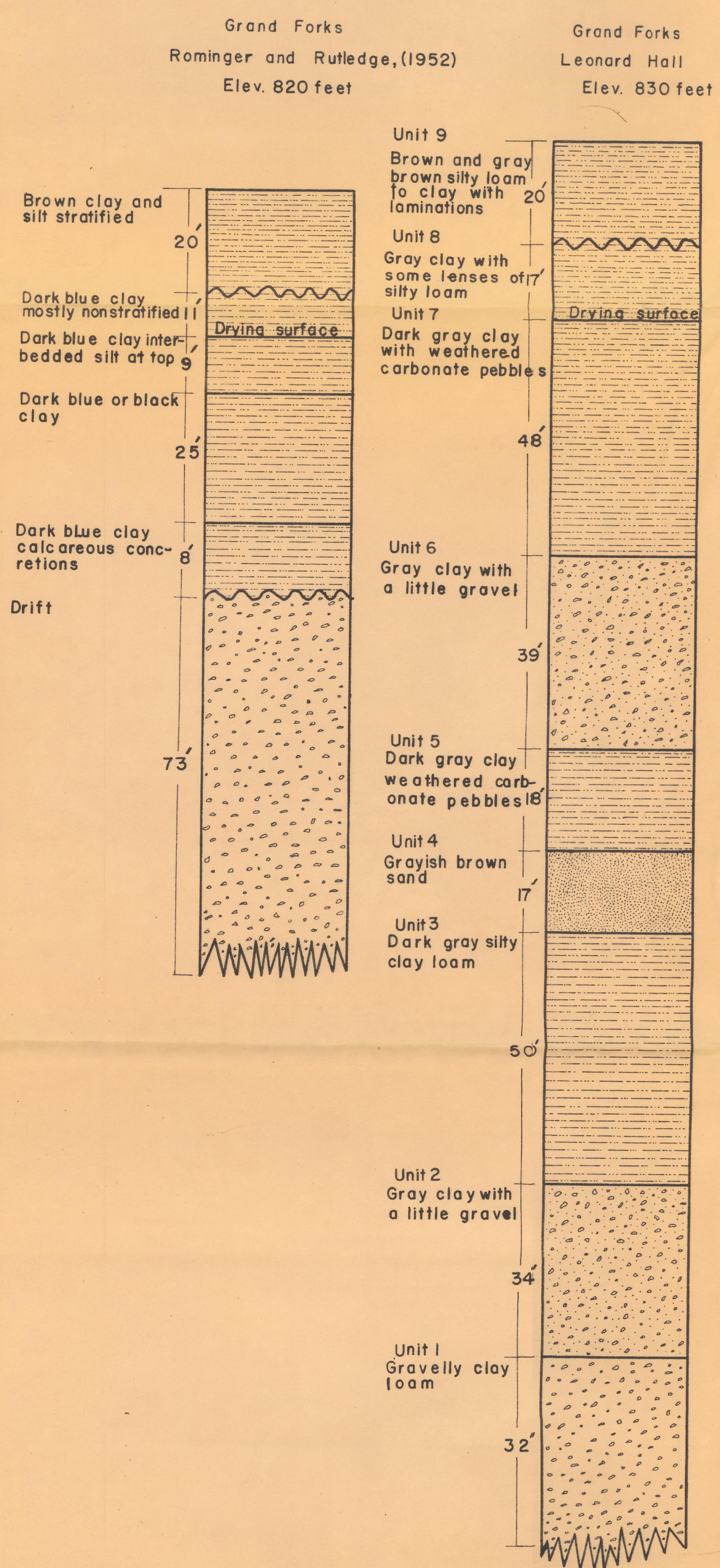two prelabeled glass slides and allow to dry overnight.

Sand-Silt Determination

After 50 additional minutes all silt will have settled to the bottom
of the beaker. Pour off most of the suspension removing most of the clay.
Add about 300 ml of distilled H_2O + calgon solution and mix. Allow to stand
until coarse silt has settled to bottom and pour off and save suspension.
Repeat. Wash sand and coarse silt several times to remove calgon. Mix the
600 ml of silt (plus some clay) suspension. Allow to stand until all silt
has settled to the bottom and pour off the suspended clay. This leaves
sand-free silt with some clay. Most of the clay and some of the silt have
been poured away. Wash and then dry the sand fraction, dry sieve and add the
sediment that passes through the sieve to the silt fraction and weigh the
material on the sieve to get the amount of sand in the sample. Calculate
the amount of silt by difference. Wash and dry the silt-rich fraction and
weigh; it should be fairly close to the calculated value.

Amounts of pore water, sand, silt, and clay can now be calculated and
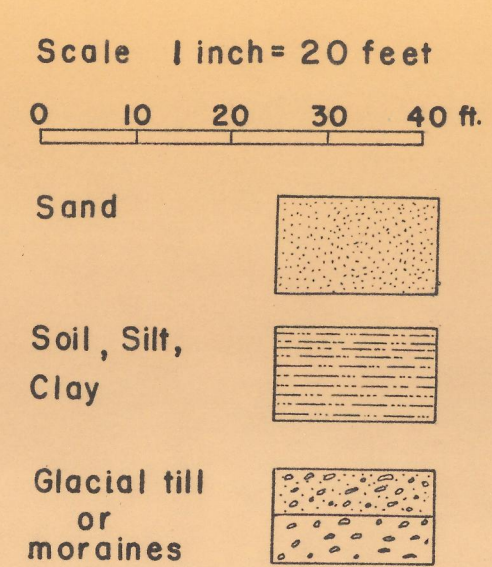representative portions of sand, silt and clay fractions can be X-rayed.

LEONARD HALL SECTION

Grand Forks
Rominger and Rutledge, (1952)
Elev. 820 feet

Grand Forks
Leonard Hall
Elev. 830 feet

Percent Total Clay ● Quartz ● Carbonate ● Feldspar ●

X-ray Trace

Percent Sand ○ Silt ○ Clay ●

PEMBINA SECTION

Legend

Scale 1 inch = 20 feet

0 10 20 30 40 ft.

Sand

Soil, Silt, Clay

Glacial till or moraines

Percent Total Clay ● Quartz ● Carbonate ● Feldspar ●

Percent Sand ○ Silt ○ Clay ●