# EFFECT OF CHEMICAL ACTION ON THE VOLUME CHANGE CHARACTERISTICS OF LAKE AGASSIZ CLAY

A THESIS

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## Synopsis

The effects of various inorganic chemicals on the volume change characteristics of the expansive clay soil of the Winnipeg area have been investigated.

Chloride, sulphate and hydroxide salts were used as immersing solutions for standard one-dimensional consolidation tests. These results were compared to those using distilled water as a swelling solution.

Differential Thermal Analyses revealed that, for each salt, exchange took place between the exchangeable cations held in the interlayer water of the clay minerals and the cations in the solution.

It was found that the amount of swelling of the soil was partially dependent on the valence of the exchangeable cation; a monovalent ion like sodium absorbed more water and thus produced greater swelling than the trivalent ion aluminum. The chloride salts tended to reduce the swelling of the soil while the sulphates and hydroxides generally increased the amount of swelling. Calcium hydroxide, which had a considerable stabilizing effect, was an exception to the rule.

The effect of the ions upon the compressibility of the soil was small in comparison to the effect on the swelling characteristics.

## Acknowledgements

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In addition, the help of Professor Michael A. Zwarich with the X-ray analysis and ion exchange determination was greatly appreciated.

# List of Symbols

a	ratio of swelling index to compressive index (Cs/Cc)
$a_{ m V}$	coefficient of compressibility (cm <sup>2</sup> /gm)
Cc	compression index (slope of virgin compression branch of e - log P curve)
Cs	swelling index (slope of rebound branch of e - log P curve)
e	void ratio of soil (volume of voids/volume of soil)
e <sub>0</sub>	initial void ratio (in field)
Δe	change in void ratio
( <u>a</u> e/e <sub>0</sub> )c	relative amount of compression (change in void ratio from initial to that under maximum compressive load/initial void ratio)
( <b>∆</b> e/e <sub>0</sub> )s	relative amount of swelling (change in void ratio from initial to that when maximum swelling occurs/initial void ratio)
Н	original thickness of clay layer
ΔH	compression of clay layer
$\mathrm{m}_{\mathrm{V}}$	coefficient of volume compressibility (cm <sup>2</sup> /gm) (compression of soil per unit of original thickness per unit increase in pressure)
n	porosity of soil
Δn	change in porosity
P	intergranular pressure (Tons/ft <sup>2</sup> )
Pc	pre-consolidation pressure
Po	initial pressure
Ps	swelling pressure
ΔP	change in pressure
μ	1 micron (10 <sup>-6</sup> meter)

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## Chapter I

#### Introduction

The soil in the Winnipeg area of Carada has, for many years, been a constant source of trouble with regard to foundation engineering and construction. The problem is a layer of highly plastic, fine grained lacustrine clay which has a thickness of from twenty to forty feet and in some places is even as deep as eighty-five feet. This was deposited during the recession of the last glacier as the fine grained sediment settled out of the waters of the glacial Lake Agassiz. This clay, which has a fairly low safe bearing capacity, is highly compressible and can exert tremendous swelling pressures.

To overcome this difficulty, most medium and large size structures in Winnipeg are founded on caissons or piles which extend to the glacial till beneath the clay, where good bearing can be obtained. However this method is uneconomical for supporting light buildings, road and airfield pavements. In these latter instances much costly damage occurs due to the vertical movement of the clay layer beneath building floors and foundations, and pavement subgrades.

It is therefore desireable to stabilize this clay so as to reduce its consolidation and swelling characteristics. Much work has been done, and is being done, in stabilizing soils for road and airport subgrades using portland cement, calcium chloride and various types of lime compounds. These agents have been shown to bind the soil particles together reducing the permeability and increasing the shear strength of the soil.

The purpose of this investigation is to determine what effect various common inorganic salts and/or cations have on the volume change characteristics of the Lake Agassiz clay. To accomplish this, a series of one-dimensional consolidation tests were performed using these salts in the immersing solution. The resulting consolidation and swelling characteristics are compared to those when distilled water is used. These results are correlated to the changes in the chemical structure of the soil, especially of the clay minerals, as evidenced by differential thermal analysis and X-ray diffraction analysis.

#### Chapter II

#### Theoretical Review

### (a) Clay Mineralogy

In soil mechanics, a clay is generally considered as a material which develops plasticity when mixed with a limited amount of water. Clay has a large specific area.

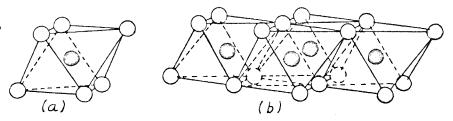
There are many different clay minerals but all have a flat plate-like structure composed of two basic units of atomic lattice arrangements. The one consists of two sheets of closely packed oxygens or hydroxyls in which aluminum, magnessium or iron are embedded in octahedral coordination, so that they are equidistant from six oxygens or hydroxyls (Figure 1). If aluminum is predominant, the structure is called gibbsite; if magnesium, then it is brucite. The structure is electrically balanced by the omission of some aluminum ions depending on whether aluminum or magnesium, etc. is present.

The other unit is composed of silica tetrahedrons in which a silicon atom is equidistant from four oxygens, or hydroxyls if required to balance the structure. The tetrahedrons are arranged to form a hexagonal network, the bases of which are in one plane giving a sheet composition. The oxygen tips all point in the same direction. (Figure 2).

The various clay minerals are made up of combinations and variations of these two structural units.

Montmorillonite clay has a structure formed by two silica tetrahedrons with a central alumina octahedral sheet.

# FIGURE 1

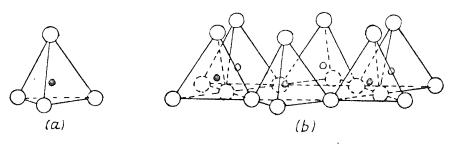


O AND ()= HYDROXYLS O ALUMINUMS, MAGNESIUMS, ETC.

DIAGRAMMATIC SKETCH SHOWING
(a) SINGLE OCTAHEDRAL UNIT
(b) THE SHEET STRUCTURE OF THE OCTAHEDRAL UNITS

( FROM GRIM)

# FIGURE 2



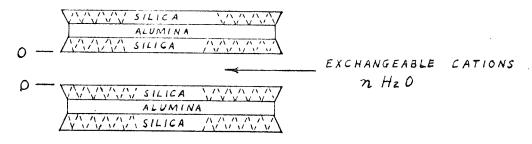
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DIAGRAMMATIC SKETCH SHOWING
. (a) SINGLE SILICA TETRAHEDRON

(b) THE SHEET STRUCTURE OF SILICA TETRAHEDRONS

(FROM GRIM)

# FIGURE 3



MONTMORILLONITE STRUCTURE

The tips of the two tetrahedron sheets face each other and abut on the central layer. With this arrangement the oxygen layer of one unit is adjacent to that of the next (Figure 3).

Exchangeable cations occur between the silica layers, and the spacing between these layers depends to an extent upon the size of the cation. Also the thickness of the water layer between the units depends upon the nature of the exchangeable cation. For example, the presence of Na will give rise to a greater spacing than will Ca and will affect the physical properties of the clay.

The lattice of montmorillonite always has an unbalance of charge caused by the substitution of ions of different valence in the tetrahedral and/or octahedral sheet such as Al<sup>3/2</sup> for Si<sup>4/2</sup> and Mg<sup>f/2</sup> for Al<sup>3/2</sup>. This is partly compensated for by the substitution of OH for O in the octahedral layer, but the net charge deficiency is balanced by exchangeable cations absorbed between the unit layers and around their edges.

The expanding lattice structure of montmorillonite causes it to swell considerably in the presence of water. Three layered expanding clay minerals such as montmorillonite are often called bentonite.

Illite clay is similar to mica, and also has a structure composed of two silica tetrahedral layers with a central alumina octahedral layer. The unit is the same as montmorillonite except that some of the silicon ions are always replaced by aluminums and the resultant charge deficiency is balanced by potassium ions.

The structural unit layers of illite are in a relatively fixed position, so that the polar ions cannot enter between them and cause expansion. Also the interlayer balancing cations are not exchangeable. It is possible that all gradations can exist between illite and montmorillonite making classification difficult. Thus the separation of these two must frequently be purely arbitrary with, in general, definitely expanding material being called montmorillonite and non-expanding being called illite.

The clays of the Winnipeg area are predominantly montmorillonite and illite as will be discussed later in Chapter 4.

However other clay minerals such as Kaolinite, Vermiculite and Chlorite are also present.

Kaolinite has a structure composed of one silica tetrahedral layer and one alumina octahedral layer. The charges within
each structural unit are balanced. The units are closely spaced and
regularly stacked with strong bonding between successive layers (i.e.
no swelling).

Vermiculite consists of an alteration of mica and double water layers. Like montmorillonite, it has a high ion exchange capacity and an expanding lattice structure. However this expansion is limited.

Chlorite is composed of alternate mica-like and brucitelike layers and is a reasonably stable structure.

Clay minerals have the property of sorbing certain anions and cations and retaining these in an exchangeable state. These ions are held around the outside of the silica-alumina clay mineral

structural units. The exchange reaction generally does not affect the structure of the silica-alumina packet, but the nature of the exchangeable ion may influence substantially the physical properties of the material.

Ion exchange capacity is generally measured in terms of milliequivalents per 100 grams of soil at pH 7.

Both cation exchange and anion exchange occur but more information is available about the former than about the latter.

The common exchangeable cations are calcium, magnesium, hydrogen, potassium, ammonium and sodium in order of general relative abundance. Montmorillonite and vermiculite, as previously mentioned have a high cation exchange capacity in the range of 80-150 milliequivalents per 100 gms. Illite and chlorite are lower with a range of 10-40 med per 100 gms and kaolinite is very low with a range of 3-15 med per 100 gms.

The common anions in clay minerals are sulphate, chloride, phosphate and nitrate. Investigation of anion-exchange reactions is very difficult. The general relative abundance of these anions is not known but their exchange capacities are generally much lower than the cation exchange capacities for each clay mineral.

#### (b) Consolidation

Dealing with soils for engineering purposes presents an additional problem not encountered in the use of other materials such as steel and concrete. Where strength is the only consideration in these latter cases, settlement and swelling must also be accounted for in soils as well as the shear strength or bearing capacity.

Terzaghi(2) first introduced the theory of consolidation and developed an expression for the computation of settlement of structures located above a confined stratum of normally loaded clay.

In the soil structure, buth the soil particles and the water in the voids are considered to be incompressible. When a load is applied an excess pressure is set up in the pore water. Since a liquid cannot sustain shear stresses, the water drains away through the soil, dissipating the pore pressure and leaving the soil structure to carry the entire applied load. The load squeezes the soil particles closer together filling the voids vacated by the water and consolidation takes place.

The addition of the weight of a fill or structure increases the pressure on the clay layer from the overburden pressure  $p_0$  to a value  $p_0 \not = \Delta p$ . The corresponding void ratio decreases from  $e_0$  to e.

Thus 
$$e_0 - e = \Delta e = a_V \cdot \Delta p$$
 (1)

or 
$$a_V \left(\frac{cm^2}{gm}\right) = \underline{\Delta} e$$
 (2)

 $\mathbf{a}_{\mathbf{v}}$  is the coefficient of compressibility for the given pressure range.

The decrease in porosity,  $\Delta$ n per unit of the original soil volume, corresponding to the decrease in void ratio  $\Delta$ e is given by the expression

$$\Delta n = \frac{\Delta e}{1 \neq e_0} \tag{3}$$

Therefore 
$$\Delta n = \frac{a_V}{1 \neq e_O}$$
  $\Delta p = m_V \Delta p$  (4)

$$m_{V} \left( \frac{cm^{2}}{gm} \right) = \frac{a_{V}}{1 \neq e_{O}}$$
 (5)

 $\mathbf{m}_{\mathbf{v}}$  is the coefficient of volume compressibility. This gives the compression of the clay layer, per unit of the original thickness, due to a unit increase in pressure.

With a clay thickness of H and a pressure increase of  $\Delta p$ , the thickness of the clay is reduced by

$$\Delta H = H \cdot \Delta p \cdot m_V \tag{6}$$

In a semi-logarithmic plot the field consolidation curve (K) becomes a straight line (Figure 4) which can be expressed by the equation

$$e = e_o - Cc \cdot \log_{10} \left( \frac{p_o \neq \Delta p}{p_o} \right) \tag{7}$$

Cc is called the compression index and is the slope of the straight line part of the consolidation curve.

A decompression curve is also fairly straight over a considerable pressure range and can be given by

$$e = e_1 \neq Cs \cdot \log_{10} \left( \frac{p_0 \neq \Delta p}{p_0} \right)$$
 (8)

Cs is called the swelling index.

Combining equation (7) with (2) and (5) gives

$$a_{V} = \frac{Cc}{\Delta p} \cdot \log_{10} \left( \frac{p_{o} \neq \Delta p}{p_{o}} \right)$$
 (9)

or 
$$m_V = \frac{Cc}{\Delta p (1 \neq e_0)} \cdot \log_{10} \left( \frac{p_0 \neq \Delta p}{p_0} \right)$$
 (10)

Substituting m<sub>v</sub> in (6) results in

$$\Delta H = H \cdot \frac{Cc}{1 \neq e_0} \cdot \log_{10} \left( \frac{p_0 \neq \Delta p}{p_0} \right)$$
 (11)

This represents the compression of a clay layer under an increase in load  $\Delta p$ .

If the clay is remolded, the e - log p curve changes and since the curve is straight over a large pressure range it can be represented by the expression

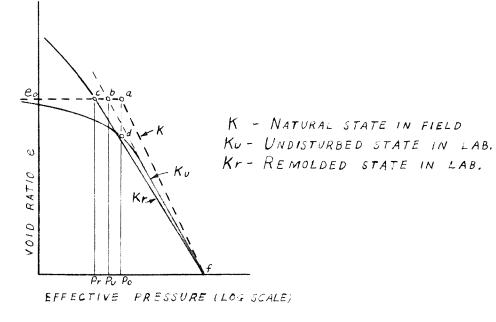
$$e = e_o - Cc' \cdot \log_{10}\left(\frac{p_o \neq \Delta p}{p_o}\right)$$
 (12)

Cc and Cs are determined in the laboratory from the e - log p curve of a one-dimensional consolidation test. The Cc value obtained from a laboratory test closely represents that of the natural state in the field.

When fine grained bentonitic soils are exposed to water at a low confining pressure considerable swelling occurs. The swelling pressure is taken as the pressure on the curve at which the soil is consolidated to its original void ratio before swelling occurred. (Figure 5).

The values obtained from the e - log p curve of one-dimensional consolidation tests are used to estimate the total amount of settlement or heaving which will occur under a fill or structure which is placed on a clay soil.

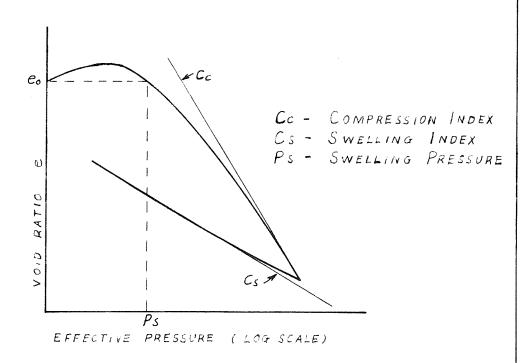
## FIGURE 4



RELATIONSHIP BETWEEN CANDP FOR ORDINARY CLAY

(FROM TERZAGHI AND PECK)

# FIGURE 5



TYPICAL & - LOG P CURVE FOR SWELLING CLAYS

## Chapter III

#### Literature Review

## (a) Clay Structure

In the previous chapter the basic structure of the clay minerals was discussed. Lambe (4) presents a further explanation discussing not only the arrangement of the soil particles but also the electrical forces acting between them. He states that electrical forces act between all soil particles but only become important when they are large in comparison to the forces related to mass. This applies in the case of a colloid which has a large specific surface and the electrical forces held near the surface are dominant. Clay size particles, especially montmorillonite, are usually in the colloidal range, which is less than 1 micron. The net negative charge carried by clay particles are balanced by exchangeable ions. When a clay particle is placed in water, the cations (plus a smaller number of anions) swarm around the colloid as illustrated in Figure 6a. Figure 6b shows the distribution of ions with distance from the clay particle surface and Figure 6c is a curve of the electrical potential with distance. The swarm of counterions is called the double layer. The counterions are also called "exchangeable" since they can be replaced.

In addition, there are two other forces acting on the clay particles. The Helmholtz or zeta potential is a repulsive force existing at the boundary between the inner and outer parts of the diffuse double layer and is much less than the potential

which can be calculated for the surface charge density. Van der Waal's forces are attractive forces which depend on the density and size of the particles and are also negligible.

The Gouy-Chapman theory shows how the potential-distance curve, i.e. double layer, varies with characteristics of the dispersion medium. Equations have been developed which show that if potential energy is reduced when adjacent particles approach each other, they will "flocculate" or form aggregates, but if the energy of the system increases when the particles approach each other, they will disperse. Variables in the soil-water system affect the colloidal stability. Generally a tendency toward flocculation is caused by:

increasing: electrolyte concentration

: ion valence: temperature

decreasing : dielectric constant

: size of hydrated ion

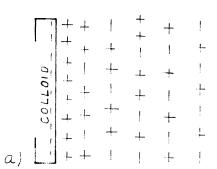
Hq:

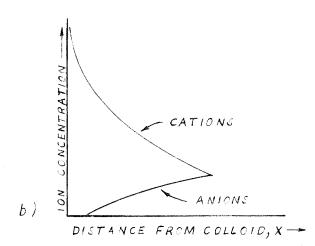
: anion absorption

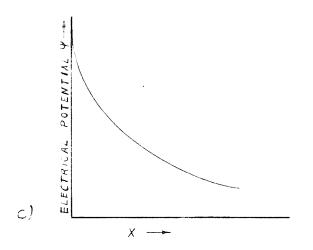
The water in clay can be arbitrarily divided into three types depending on the relative magnitude of force between the clay and water. The absorbed water is held strongly by the soil; the double layer is all the water attracted by the soil; and free water is not attracted to the soil at all. Figure 7 illustrates a montmorillonite particle and its adjacent water layers.

The above mentioned variables in the soil-water system together with the amount of free water affect not only the shear strength characteristics of soil but also the volume change characteristics.

# FIGURE 6

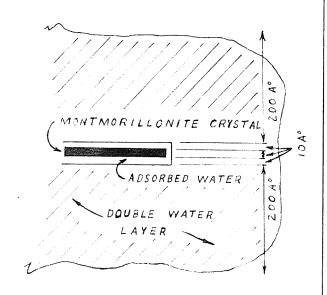






DIFFUSE DOUBLE LAYER

FIGURE 7



TYPICAL MONTMORILLONITE

PARTICL'E

1000 A° BY 10 A°

\* IFROM LAMBEL

(FROM LAMBE)

In the second paper of the series (Ref. 5), Lambe discusses "The Engineering Behaviour of Compacted Clay". He mentions the the effect of chemical reactions in the soil are important in the engineering use of soil.

## (b) Swelling of Soils

Swelling characteristics of clay soils have been the subject of many papers, (Ref. 6-15). Discussions are generally centered on the identification of swelling type soils and how and why swelling takes place.

Several simple tests have been suggested for means of identifying potential swelling soils. The most common proposal is the use of the Atterberg Limits. Kantey and Brink<sup>(10)</sup> have offered the following criteria for determining expansive soils:

- (1) the linear shrinkage should be greater than 8%
- (2) the liquid limit should be greater than 30%
- (3) the plasticity index should be greater than 12%.

Seed et al<sup>(12)</sup> and Holtz<sup>(7)</sup> provide other relationships also using Atterberg Limits.

The use of the activity ratio (Plastic Index/% Clay Size) has also been suggested as a criterion but this is not widely agreed upon.

Probably the best routine test for determining an expansive soil is the free swell test as suggested by Holtz<sup>(7)</sup> or the standard one-dimensional consolidation test described by Hardy<sup>(15)</sup>.

Actually the only positive identification of clays subject to volume change is by microscopic examination, X-ray diffraction and differential thermal analysis. However these tests are

time consuming, require relatively expensive equipment and specially trained personnel.

It is generally agreed that several factors are necessary in order that swelling occurs in soils. An exposure of the soil to water and/or a reduction in the external load in combination with a clay of a high specific surface are the most important points. Montmorillonite and illite clays both fall into this category but montmorillonite is much more expansive due to its surface chemical composition. This surface structure is such that water molecules are strongly attracted to it. Thus the thickness of double layer water between each clay particle is greatly increased (i.e., swelling occurs).

Swelling has also been explained by an osmotic interpretation (Ruiz 6). The cationic concentration is much higher in the vicinity of the clay particles than it is in the free water. The difference in ionic concentration gives rise to an osmotic pressure difference that causes water to flow from the point of low concentration to the point of high concentration (i.e., into the soil). The swelling pressure may be evaluated by the van't Hoff equation:

$$P = RT (C_C - C_O)$$

where P = osmotic or swelling pressure

R = gas constant

T = absolute temperature

Cc = concentration of ions at the point midway between
 the two interacting plates

Co = concentration of ions away from the influence of the clay particles

The amount of swelling in soils depends on several other factors. It has been shown that swell and swell pressure increase with increasing density, decreasing water content, increasing random-

ness of structure (flocculent structure), and decreasing electrolyte concentration in the absorbed water. Swelling pressure decreases as swelling occurs.

The exchangeable cations present also affect the swelling. For example clays having monovalent ions such as sodium will take up more water than those having divalent ions such as calcium.

In any engineering work dealing with expansive soils it is necessary to determine the amount of swell and swelling pressure that will develop under the anticipated conditions. This is quite possible with sufficient testing although Dyregrov and Hardy (14) illustrate the variability in answers which are obtained from different swelling tests.

Holtz and Gibbs (13) give examples where hydraulic structures were constructed on expansive clays. By considering the swelling properties of the soil, these structures were built keeping distress to a minimum.

# (c) Ion Exchange and Stability

Grim (16) presents a discussion on the fundamentals of ion exchange. He points out that an understanding of ion exchange reaction is essential to an understanding of the nature of properties of the soils. This is important because it is the mechanism of ion exchange which provides the most practicable means of modifying the characteristics of soils.

Winterkorn (17) gives an example of stabilizing gravel road surfaces with a clay binder by chemical means. The compounds were applied by spraying the road surface with aqueous solutions.

"The road was greatly improved and necessary maintenance decreased by treatment with sodium and potassium chlorides, silicates, and phosphates, while treatment with calcium chloride made the road worse. Laboratory tests on samples taken from the treated sections showed that the beneficial influence of the sodium and potassium silicates, phosphates and chlorides was due to the same cause, namely their dispersive effect and not to any cementing action on the secondary aggregates. The calcium-salt treatment on the other hand, resulted in further repression of the hydration of the clay aggregates and in a consequent increase in the permeability of the road surface".

An interesting point made by Winterkorn is that it is not necessary to work with homo-ionic soils in the field. He states that a Na - soil characteristic can be achieved with about 15 per cent Na - saturation of the exchange complex and the saturation proportion required for Fe - and Al - soil characteristics is even less.

Winterkorn also mentions successful stabilization tests using Na - soap solutions and organic cations.

Goldberg and Klein (18) performed a series of tests treating two expansive clays with calcium hydroxide. They found that additions of calcium hydroxide in excess of 2 per cent considerably reduced the swelling characteristics of the soil. These benefits increased with increasing amounts of salt up to about 6 per cent. Increased resistance of the soil to deformation under

load was also noted.

"X-ray diffraction and differential thermal studies of the several materials included in this investigation indicate the complete conversion of all calcium hydroxide employed in treatment to other forms".

". . .it is shown that a portion of the calcium hydroxide is converted to calcium carbonate under conditions similar to those which occur in the field".

Bolt (19) did compression tests on calcium and sodium montmorillonite and on sodium illite and compared his results with theoretically calculated values. In general he got a good comparison. He concluded, "...that the compressibility of pure clay suspensions and paste can be accounted for very well by the consideration of the interaction between the electric double layers formed on the clay particles".

At a symposium<sup>(20)</sup> sponsored by the ASCE four papers were presented by Grim, Taylor, Rosenqvist and Lambe on the physico-chemical properties of soils. Grim<sup>(20a)</sup> discusses the structure and composition of clay minerals, their origin and methods of identifying the clay minerals.

Taylor (20b) presents a complete discussion on ion exchange and its applicability to swelling and compressibility of soils. He concludes that, "...it is evident from the results obtained where detailed investigations have been carried out in individual cases that the Gouy-Chapman theory of diffuse double layer provides a satisfactory basis for the study of many clay soils, and

may be used to derive useful information about probable mechanical behaviour from the results of chemical tests".

Rosenqvist's paper (20c) on "Soil-Water Systems" discusses influence of the water phase and the effect of water upon mechanical properties of the soil. He also mentions the influence of surface ions on mechanical properties of the soil.

Lambe (20d) presents an interesting paper on "The Role of Soil-Technology". He explains the various components of soil technology and emphasizes the contributions of soil technology to soil engineering. In particular he shows how technology has made important contributions in the analysis of stability and settlement problems and improved techniques of soil stabilization, especially by chemical treatment.

At the Conference on Soil Stabilization (21) held at the Massachusetts Institute of Technology in June 1952, papers on all types of soil stabilization were presented. These included work done by chemical means, electrical, bitumens and other admixtures as well as mechanical methods. The papers by Woods and Yoder, and Murray discuss the effect of chemicals on the physical properties of the soil. However little mention is made of chemical changes in the soil structure and particular ion exchange phenomena. In the few years since that time, much work has been done in the field of ionic stabilization.

## Chapter IV

#### Laboratory Investigation

## (a) Description of Soil

The soil used in this investigation was a typical clay soil of the Winnipeg area. It was obtained from a site on the campus of the University of Manitoba from the excavation for a new building. Four adjacent block samples were manually dug out from the excavation floor at a depth of nine to ten feet. Keeping the proper orientation, the blocks were trimmed and wrapped in a clear plastic wrap to reduce moisture loss. They were immediately taken to the soils laboratory where samples were taken to determine the moisture content. The block samples were then wrapped in aluminum foil, coated with wax and stored in a humidity chamber.

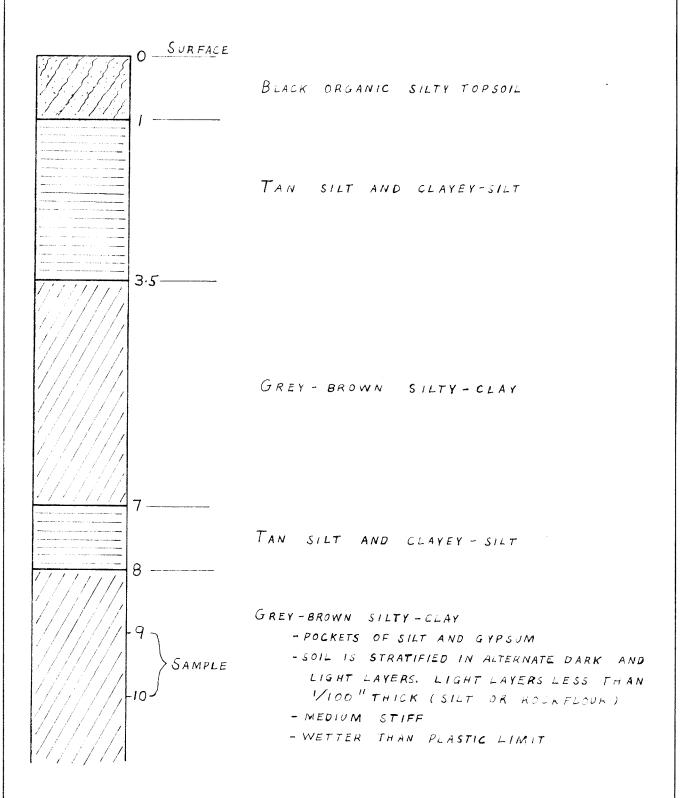
A depth of nine to ten feet was chosen for the test samples as this is the approximate depth where foundations and basement floor slabs are located for many structures. Thus it is at this elevation where the greatest amount of settlement or heaving would occur. In addition, the sample from this depth is above the water table.

One block of soil approximately one foot cube was used for the entire testing. The soil could be described as a grey-brown silty-clay with small pockets of silt and gypsim and very thin horizontal stratifications of silt or rock flour (See soil profile Figure 8). The silt stratifications, which have a thickness of less than 1/100 inch, are similar to varves but are not uniform enough for the soil to be classified as a varved clay.

# FIGURE 8

# SOIL PROFILE

SITE: EXCAVATION FOR ADDITION TO EDUCATION BUILDING, UNIVERSITY OF MANITOBA.



The natural moisture content was found to vary from 46% to 65% with 56% being a representative average. The degree of saturation was approximately 96%.

To identify the soil and to determine the physical characteristics and the physico-chemical properties, three samples were taken from the block. A horizontal slice was taken from the top, one from the bottom and a vertical slice from one side. The three samples, which gave practically identical results, were prepared and tested according to ASTM, Procedures for Testing Soils, 1958 (22).

The average specific gravity was found to be 2.765 and is estimated to be accurate to 0.005. From grain size analysis the soil was found to consist of, on the average, 93% clay size particles, 6% silt and 1% sand. In determining the Atterberg Limits a slight modification to the ASTM Procedure was employed; after air drying and powdering the soil was cured in a moist condition for 24 hours to give more consistent results. The liquid limit was found to be 101 and the plastic limit 35 giving a plasticity index of 66. A shrinkage limit of 17 and a linear shrinkage of approximately 17% were also determined. The activity ratio of the soil, that is the ratio of the plasticity index to the per cent finer than 0.002 mm, is 0.73.

When these results are plotted on Casagronde's plasticity chart, this soil is classified as a CH type, that is an inorganic clay of high plasticity or fat clay. According to Kantey and Brink (10) This would be classed as an expansive soil.

Maray diffractometer analysis were also done to determine the mineralogical composition of the soil and corroborate the results of the differential thermal analysis. The procedure used was that of M. L. Jackson, "Soil Chemical Analysis, Advanced Course". The organic material and the carbonates were first removed. The soil was then fractionated, by centrifuging into three size ranges: fine silt  $5\mu$  to  $2\mu$ , coarse clay  $2\mu$  to  $0.2\mu$  and fine clay, less than  $0.2\mu$ . Two slides were prepared for each fraction, one with the soil saturated with magnesium, Mg<sup>f,f</sup> and the other saturated with potassium, K<sup>f</sup>. In addition to the initial X-ray, each sample saturated with potassium was heated to  $300^{\circ}$ C and X-rayed again, then reheated to  $550^{\circ}$ C and X-rayed a third time. This gave a total of twelve X-rays for each sample.

Instead of photographing the fringes produced by the X-ray, the results were automatically recorded on graphs. Position of the peaks on these graphs corresponded to the different d-spacings for the various minerals and compounds and thus identification was possible.

Montmorillonite was the predominant mineral in the fine clay fraction and totaled approximately 45% of the complete sample. This is in good accord with that found by others, Elson (23). The coarser sizes were made up of illite, vermiculite, kaolinite and chlorite with some quartz and feldspar present.

The carbonate content was slightly less than 5% and organic matter probably about 2%. Differential thermal analyses show that the composition of the soil is predominantly interlayered illite and montmorillonite. Dolomite, organic compounds and a trace of free iron are also present. Calcium is the main exchangeable cation in the interlayer water. (thermogram for sample 1b, Appendix B).

The ion exchange capacity of the soil was also determined. The exchangeable ions in the soil are leached out with a 1:1 mixture of amnonium acetate and ethyl alcohol. The total exchange capacity was found to be 40-50 milliequivalents per 100 grams. This was almost entirely calcium, Caff and magnesium, Mgf, with slightly more calcium. Exchangeable sodium and potassium ions were very low with approximately 1 meg per 100 grams each.

The pH of the soil was 7.5 which also indicates a calcium montmorillonite soil.  $Grim^{(1)}$ 

#### (b) Consolidation Testing

A total of thirty-three one-dimensioned consolidation tests were performed using solutions of thirteen different salts for swelling in addition to distilled water. A consolidometer bank consisting of six loading frames was used to compress the soil samples. Weights were applied to a pan and transferred through a system of pulleys with a 1:10 ratio to a yoke and thence to the sample. Plate I shows six tests in progress.

The consolidometer dish which contained the clay sample and the swelling solution consisted of a lucite bowl with a lucite-covered brass base and a dial gauge support. The lucite was used

as it is chemically inert and not corroded or affected by strong acids or bases.

Floating ring tests were used throughout so as to reduce the side friction effect and produce a more uniform pressure distribution throughout the thickness of the sample. The rings were of brass and had an interior diameter of 2.04 inches with a height of 7/8 inch and thickness of 1/4 inch.

Two sets of rings were used during the testing. The first six were painted with a clear enamel paint to keep the metal from being in contact with the soil and solution and to prevent corrosion and tarnishing of the rings. Before a test was set up, the interior of the ring was coated with a very fine teflon-like spray so as to further reduce side friction. The painted rings proved unsatisfactory especially at the end of the test when the samples were dried in the oven; the paint would run and it would be necessary to refinish them before use again. However the actual test results using these rings were quite satisfactory.

The second set of rings were identical to the first except they were teflon coated instead of painted. These were much easier to use as they prevented corrosion, reduced side friction, needed no refinishing and were easy to clean. The first rings were used for fourteen tests and the teflon-coated ones were employed in the last eighteen tests.

While the height of the rings were 7/2 inch, the average initial sample height was only 0.61 inch. The sample was trimmed about 1/4 inch short so as to allow swelling without the

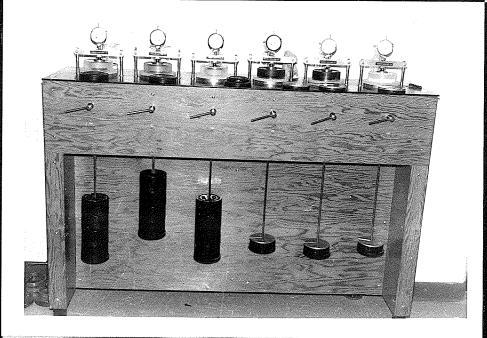


PLATE I
Consolidometer Bank with Six Tests in Progress

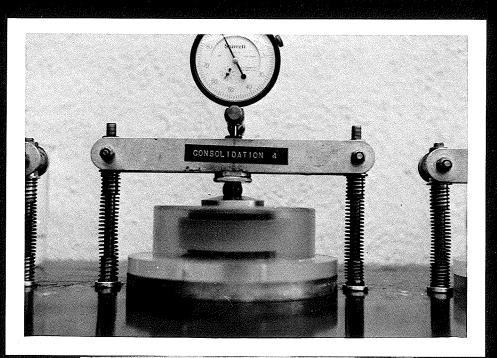


PLATE II
Soil Sample under Consolidation

o.30. A cutter with the exact same interior diameter as the rings was used to cut the samples from small chunks which were in turn cut from the larger block. The soil sample was transferred from the cutter to the ring and the ends were trimmed parallel about 1/4 inch shorter than the ring. The weight of the soil and ring were recorded and a sample height measured using a dial gauge reading to 0.001 inch.

The clay sample was centered in the ring, a piece of filter paper and a porous corundum stone situated on either side and the ensemble placed in the consolidometer and positioned under the load frame. A 1/2" steel ball bearing was used to get a uniform pressure distribution from the yoke to the loading cap. A dial gauge reading to 0.001 inch was set up to measure the deflection of the sample. Plate II illustrates the entire arrangement.

When the set up was complete and the initial reading taken on the dial gauge, the first load was applied to the sample. Deflections were read at the following times: 5, 10, 15 and 30 secs., 1, 2, 4, 8, 15 and 30 mins., and 1, 2, 4 and 24 hours. After the two minute reading for the initial load, the water or salt solution, whichever the case, was added to produce swelling in the soil. The level of the liquid was kept to about mid-height of the sample. After 24 hours more solution was added to completely immerse the sample and swelling allowed to continue until maximum was reached after about 40 to 48 hours. The remaining loads were added, every 24 hours.

A load increment ratio of 1.75 was used, that is P2/P1 = 1.75 or  $\Delta$ P/P1 = 0.75. The following pan loads were applied: 0.3, 0.5, 0.8, 1.4, 2.5, 4.3, 7.5, 13, 23 and 40 pounds. The 40 pound weight, which produces a pressure of 8.82 tons per square foot on the sample, is the maximum capacity of the loading frames. In the unloading cycle every second increment was used, that is from 40 lbs. to 13, to 4.3, to 1.4, to 0.5 lbs. The loads were removed at four hour intervals and unloading completed in one day.

Tests #1 to #4 were slightly different. The load increment ratio was the same but the maximum load was 35 lbs. or 7.72 tons/sq. ft. These were run mainly to investigate the effect of differences in unloading cycle upon the swelling index Cs. No. 1 was unloaded every increment at 24 hour intervals, #2 every second increment at 24 hour intervals, #3 every second increment at 4 hour intervals. On #4 the loading was started at a pressure higher than the overburden pressure in the field and the unloading was every second increment at four hour intervals.

Some variation was noted in the compressive indices but the swelling indices for the four were practically constant. Thus it was assumed that unloading every second increment at four hour intervals would give good results for the swelling index and cut the time of the test almost in half.

When the unloading was completed, the clay sample and ring were dried in an oven at 110°C to obtain the dry weight of the soil and to determine the initial and final moisture contents.

The salt solutions used in this testing program, with the exception of a few cases, had a one molar concentration, that is one formula weight of salt per litre of solution. (As the solution evaporated from the open consolidometers the concentration would increase slightly.) A reagent grade salt was used for each except for sodium chloride where a common table brand was used. The solutions of sodium sulphate and potassium sulphate were super-saturated and some salt precipitated out during the test. The sodium chloride and the ammonium chloride solutions were at the saturation point. The calcium hydroxide was insoluble and thus held in a suspension.

Tests 1 to 4, as mentioned previously, had swelling solutions of distilled water. Tests 5 to 8 had solutions of potassium chloride, calcium chloride, magnesium chloride and aluminum chloride respectively.

Test 9 was an exception to the others. It was done on a Farnell consolidation frame. In this case the sample was loaded to 18 tons/sq. ft., more than twice the capacity of the other frames, and a recycling phase and unloading was done. A fixed ring 2.50 inches in diameter and 0.75 inch high, giving the same L/D ratio as the other tests, was used. The swelling solution was distilled water.

Test 10 is another with a swelling solution of distilled water and unlike #1 to #4 was loaded in the same manner as the other tests with salt solutions. Tests 11 to 14 were with solutions

of sodium sulphate, sodium chloride, potassium sulphate and magnesium sulphate. Test 15 had a solution of aluminum sulphate with a 0.5 molar concentration. In this case it was not possible to get more salt into solution.

Tests 16, 17 and 18 were with swelling solutions of potassium hydroxide, sodium hydroxide and calcium hydroxide respectively. As mentioned previously, the calcium hydroxide would not go into solution and was held in a suspension.

In test 19 distilled water was again used for swelling to compare with #10 as a considerably higher swelling pressure was found in #10 than in nos. 1 to 4.

Test #20 was an extra. The swelling solution was 20% liquid detergent (Joy) and 80% distilled water.

No. 21 again had a solution of potassium chloride only this time with a 2 molar concentration.

All the consolidation tests up to this point were on soil samples that were relatively undisturbed. For the remainder of the tests, except for #28, the soil was remolded by working by hand and most of the above salts were used in the swelling solutions again. Tests nos. 22 to 27 employed solutions of distilled water, sodium chloride, calcium chloride, aluminum chloride, potassium hydroxide and sodium hydroxide respectively.

In test #28 the soil sample again was in a relatively undisturbed state and the swell solution was ammonium chloride with a 1 molar concentration (saturation point).

Nos. 29-31 had solutions of aluminum sulphate (again 0.5 molar), magnesium sulphate and potassium sulphate respectively.

The last two tests were slightly different. The soil samples were remolded with the dry powdered salt (mixed in) and distilled water was used for swelling. No. 32 had approximately 5% calcium chloride per dry weight of soil. No. 33 had about 5% calcium hydroxide and 10% distilled water. This would roughly approximate what is often used in road stabilization.

The results of these thirty-three tests are plotted as e - log P curves in Appendix A and are tabulated in Table 1.

## (c) Differential Thermal Analysis

After each consolidation test was completed and the soil dried in the oven, a differential thermal analysis was run on it to determine the changes in the soil caused by the salts.

The first step was to powder the dry soil disc from the consolidation test and pass it through a #60 sieve. A representative sample, about 3 grams, was taken using a sample splitter, spread on a watch glass and placed in a room with a controlled humidity of about 45% for at least 24 hours.

The differential thermal apparatus used was Model DTA-13M manufactured by Robert L. Stone Co., Austin, Texas. The recorder is by Leeds and Northrup. Plates III and IV show the assembly including the furnace and specimen holder block, the auxiliary furnace on the cooling rack, the gas bottles and the recorder.

The specimen holders have been drilled in a cylindrical rod of chrome nickel steel which is supported by an aluminum tube.

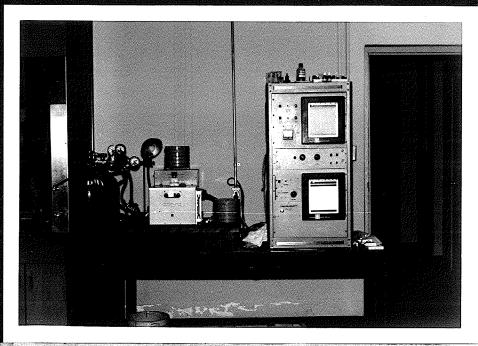


PLATE III

Differential Thermal Analysis Apparatus Heating Furnace Removed Showing Specimen Holder

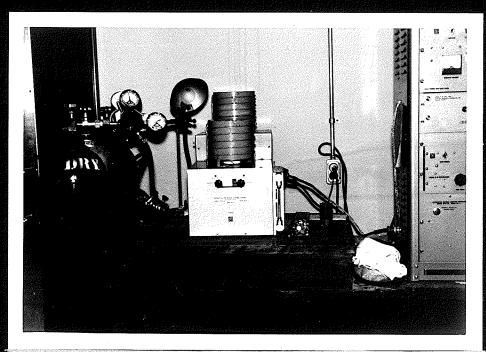


PLATE IV

Heating Furnace in Place and Auxiliary Furnace on Cooling Rack for DTA.

There are two specimen holders, one for the sample to be tested, and one for the inert sintered aluminum oxide compound with which the sample is compared. Thermocouple wires lead to each of the holders and are arranged so that the potentials oppose each other.

With the application of heat substances undergo exothermic and endothermic reactions. These reactions can be picked up by the sensitive recorder and are graphed as exothermic or endothermic peaks. Each peak is characteristic of a particular mineral or compound; as it is unlikely that any two minerals have chemical bonds of identical strength, they will decompose, oxidize, or change phase at different temperatures. Also the amplitude and shape of the peaks are important. When the sample is the same as the standard compound no differential reaction takes place and a straight line is recorded on the graph.

Two runs were made for each sample. The one was heated to 600°C in a nitrogen atmosphere. This suppressed the oxidation of the organic matter and allowed the interlayer water curve of the lower half of the clay curve to be recorded. The second run was heated to 1000°C. An oxygen atmosphere was used up to between 500° and 600°C and then was changed to a carbon dioxide atmosphere which was continued to 1000°C. This allowed the organic matter to be recorded followed by the upper half of the clay curve.

The heating rate in these tests was about 10°C per minute although this varied slightly.

Differential thermal analyses were also done on samples of the original untreated clay (same samples as for X-ray analysis,

as previously mentioned) so that the treated samples could be compared for changes which took place in the soil structure.

The thermograms from the differential thermal analyses are reproduced in Appendix B.

### Chapter V

#### Hesults of Tests

### (a) Consolidation Tests

The results of the consolidation tests which were performed using the various salts in the swelling solutions are plotted
in the form of e - log P curves. These are reproduced in Appendix
A and the values of compressive index, swelling index, swelling
pressure, etc. are summarized in Table 1.

In each of the tests a straight line was not obtained on the virgin compression part of the e - log P curves. The compressive index, Cc, was taken to be the slope of the line between the two points of maximum pressure, i.e. 5.08 and 8.82 tons per sq. ft. This slope would not be the true compressive index as the straight line part of the curve was not reached. However this value is quite adequate for comparing the effects of the different salts as the same points are used in each case. This is illustrated in the tests with the distilled water. Cc values of 0.500 and 0.492 were found in tests nos. 10 and 19. But in the first four tests where the samples were loaded to a lower pressure than in the above cases, the compressive index ranged from 0.408 to 0.482. In test No. 9 a Cc value of 0.572 was found when the sample was loaded to 18 tons per sq. ft.

The swelling index, Cs, was taken to be the slope of the line between the maximum pressure, 8.82 tons per sq. ft. and 0.32 tons per sq. ft. in the unloading cycle. These two points give

TABLE 1
SUMMARY OF CONSOLIDATION TEST RESULTS

TES'	. CONDITION	SWELLING SOLUTION	pH of SOLUTION	Pe T/FT <sup>2</sup>	Cc	Cs	Ps T/FT <sup>2</sup>	a = <u>Cs</u> Cc
1 2	Relatively Undisturbed	11	7.	0.9	0.408	0.218	0.35 .28	0.533 .491
3	11 H	11		1.15	.482	.209	.28	.433
4 5 6	"		,		.475	.228	7.1	.480
<i>5</i>	11	KCl (1 M) CaCl <sub>2</sub> .2H <sub>2</sub> O (1 M)	6. 4.5		.380 .400	.116	.14	.305
7	11	MgCl <sub>2</sub> .6H <sub>2</sub> O (1 M)	4.5		.403	.150	.13 .14	.352 .372
8	11	A1C13.6H2O (1 M)	2.		.400	.110	.06	.278
9	£\$	Distilled H2O	0	2.2	.572	.234	.65	.409
10	17	п	6.5	1.3	.500	.210	.58	.420
11	Ħ	Na <sub>2</sub> SO <sub>4</sub> (1 M)	5.5		.420	.158	.98	.376
12	t 9	NaCl (l M)	4.		.518	.172	.34	.332
13	‡f	$K_2SO_4$ (1 M)	6.		.347	.085	.56	. 245
14	††	MgSO4.7H2O (1 M)	5.		•500	.158		.316
15	. !!	$A12(S0_4)_3.18H_20(0.5M)$			•500	.100	•53	.200
16	78	KOH (1 M)	13.		.458	.082	.44	.179
17	11	NaOH (1 M)	13.		.466	.100	1.3	.215
18	<b>!</b> \$	$Ca(OH)_2$ (1 M)	12.5		.448	.163	.21	.364
19	F	Distilled H <sub>2</sub> O	6.5	1.45	.492	.199	•3 <b>5</b>	.404
20	1 <b>1</b> Ff	20% liquid detergent	8.5		.541	.160	•35	.296
21		KCl (2 M)	4.5		.426	.116	.Ol	.272
22	Remolded	Distilled H <sub>2</sub> O	6.5		.608	.178	.42	.292
23 24	11	NaCl (1 M)	5.		.465	.128	.15	.275
25	11	CaCl <sub>2</sub> .2H <sub>2</sub> O (1 M)	4.5		.480	.125	.23	.260
26	Ħ	A1Cl <sub>3</sub> .6H <sub>2</sub> O (1 M) KOH (1 M)	2.		.445	.076	.20	.171
27	łt .	NaOH (1 M)	13. 13.		.473 .568	.067 .104	.30	.142
28	Rel.Undisturb		5 <b>.</b>		.375	.118	.45 .15	.183 .317
29	Remolded	$Al_2(SO_4)_3.18H_2O(0.5M)$	3.		.408	.103	•15 •35	.252
30	u	MgSO <sub>4</sub> .7H <sub>2</sub> O (1 M)	5°		•530	.133	•43	.251
31	tt .	$K_2SO_4$ (1 M)	6.		.475	.110	.26	.232
-	Remolded with	Distilled H <sub>2</sub> O	5.		.415	.127	.12	.306
	5% CaCl2.2H2O	~			•41)	•1~ /	0 I.C	• )00
33	Remolded with 5% Ca(OH) <sub>2</sub> & 10% H <sub>2</sub> O	Distilled H2O	6.		0.591	.052	0.0	.088

the best average slope of the unloading part of the curve and like the compressive index is satisfactory for comparing the effects of the various salts.

The swelling pressure for these soil samples was determined by the "free swell" test. It is believed by some authorities that this test is invalid and that a "constant volume" test should be used to accurately determine the swelling pressure. However Dyregrov and Hardy (14) feel that the "free swell" test is preferable in some cases. Either way, a good comparison is obtained, as in the above indices, on the effect of the salts on the swelling pressure of the soil.

In the tests with the distilled water, the pre-consolidation pressure (Pc) of the soil was estimated by the Casagrande method. Values ranged from 0.9 to 1.45 tons per sq. ft. with one as high as 2.2 tons per sq. ft. The existing overburden pressure in the field was approximately 0.5 or 0.6 tons per sq. ft. Thus the soil is in a slightly over-consolidated state.

In general, the effect of the salts was to reduce the swelling index and the compressive index (in most cases). The swelling pressure was also reduced except where the sodium salts were used. These effects will be discussed more fully in the next chapter.

In order to compare the results produced by the different salts on the volume change characteristics of the soil, new plots were made which eliminated the effect of the initial void ratio.

Each void ratio, e, at the various loads on the test samples was

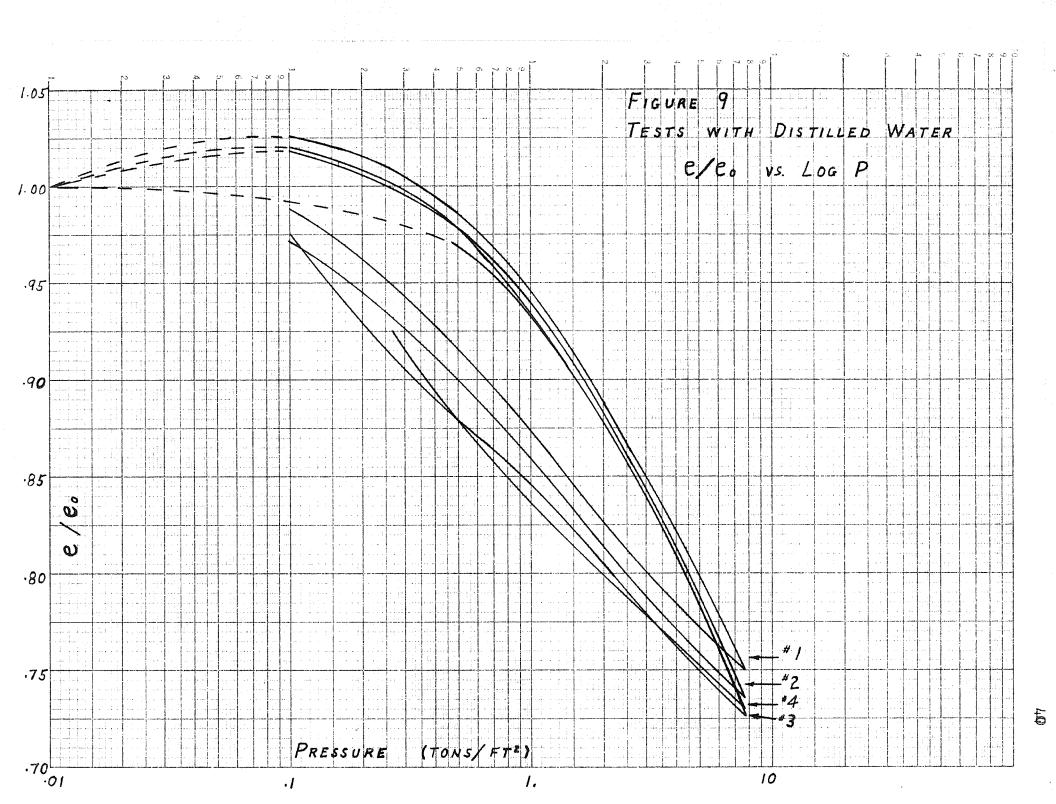
divided by the initial void ratio,  $e_0$ , and new curves were drawn of  $e/e_0$  vs  $\log P$ . Dividing by  $e_0$  eliminates the effect that the initial density and moisture content have on the swelling characteristics of the soil. Thus each curve begins at a common point and the relative amounts of swelling and consolidation can easily be determined. (See Figures 9-18).

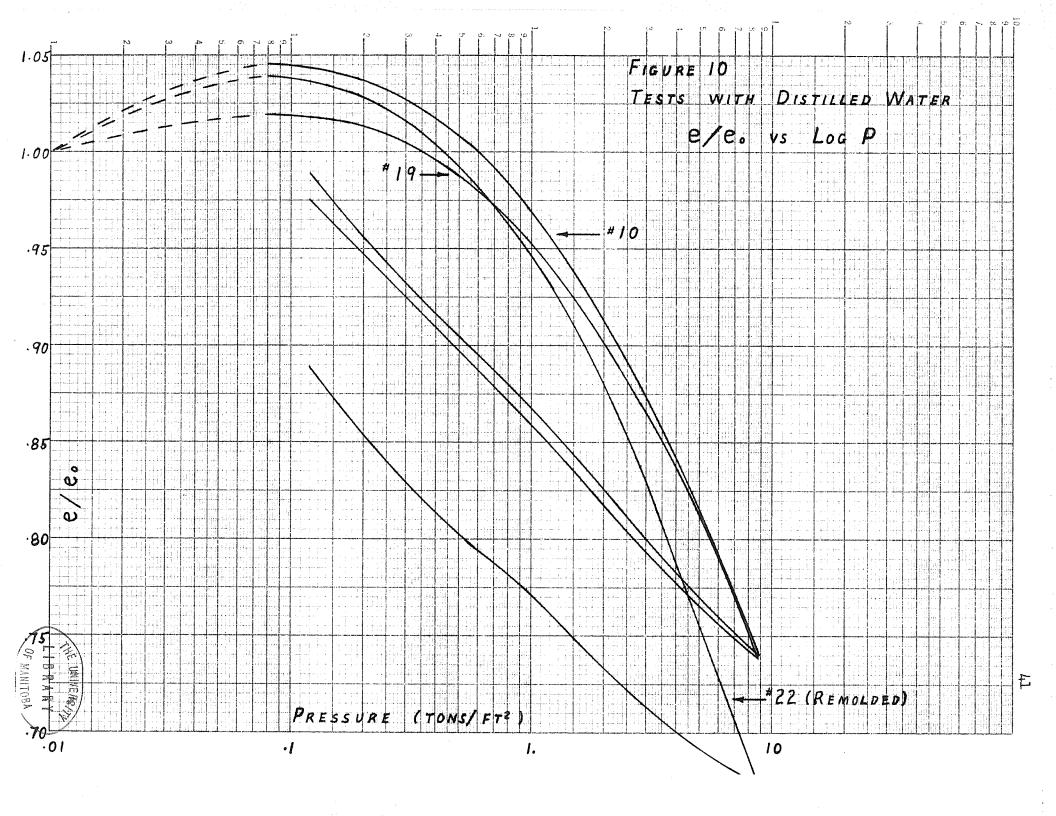
### (b) Differential Thermal Analysis

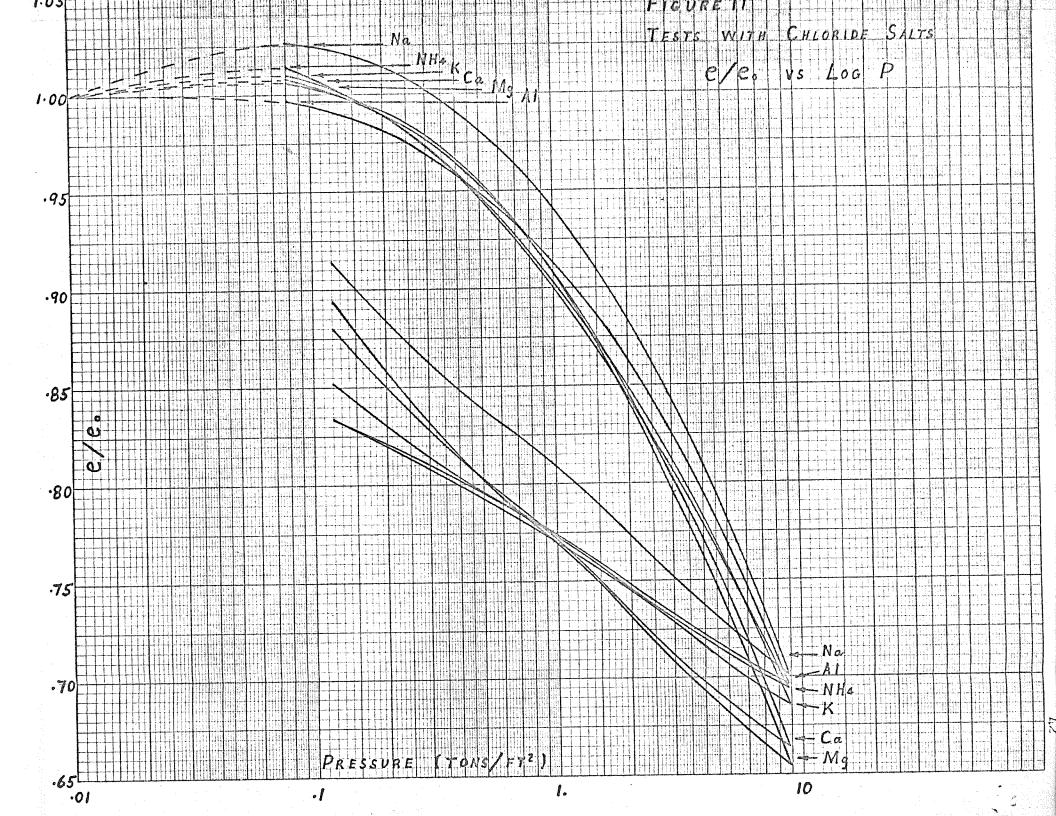
In the previous chapter a description of the soil composition was given. Differential thermal analysis showed that the mineral composition was predominantly interlayered illite and mont-morillonite with small amounts of carbonates, quartz, free iron and organic matter. Calcium was found to be the main exchangeable ion in the interlayer water of the clay.

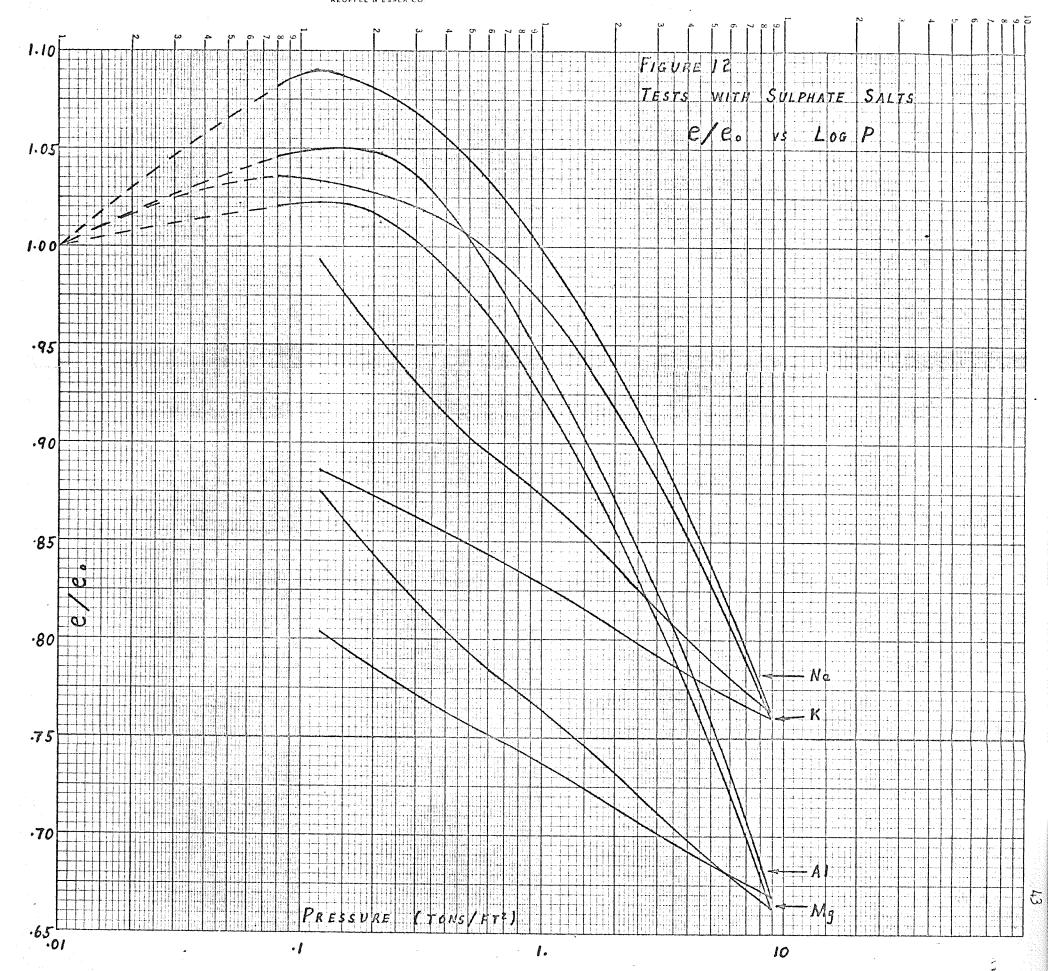
Subsequent differential thermal analyses, which were done on the samples used in the consolidation tests, in which salts were added to the swelling solutions, revealed that exchange took place between the calcium ion in the interlayer position and the cations in the solution. In general no other change in the soil composition was noted. The thermograms from the various tests are reproduced in App@ndix B.

In the tests with distilled water no change from the original soil structure occurred except for a slight intensification of the calcium exchange ion. This was also the case when the sample was remolded before consolidation.





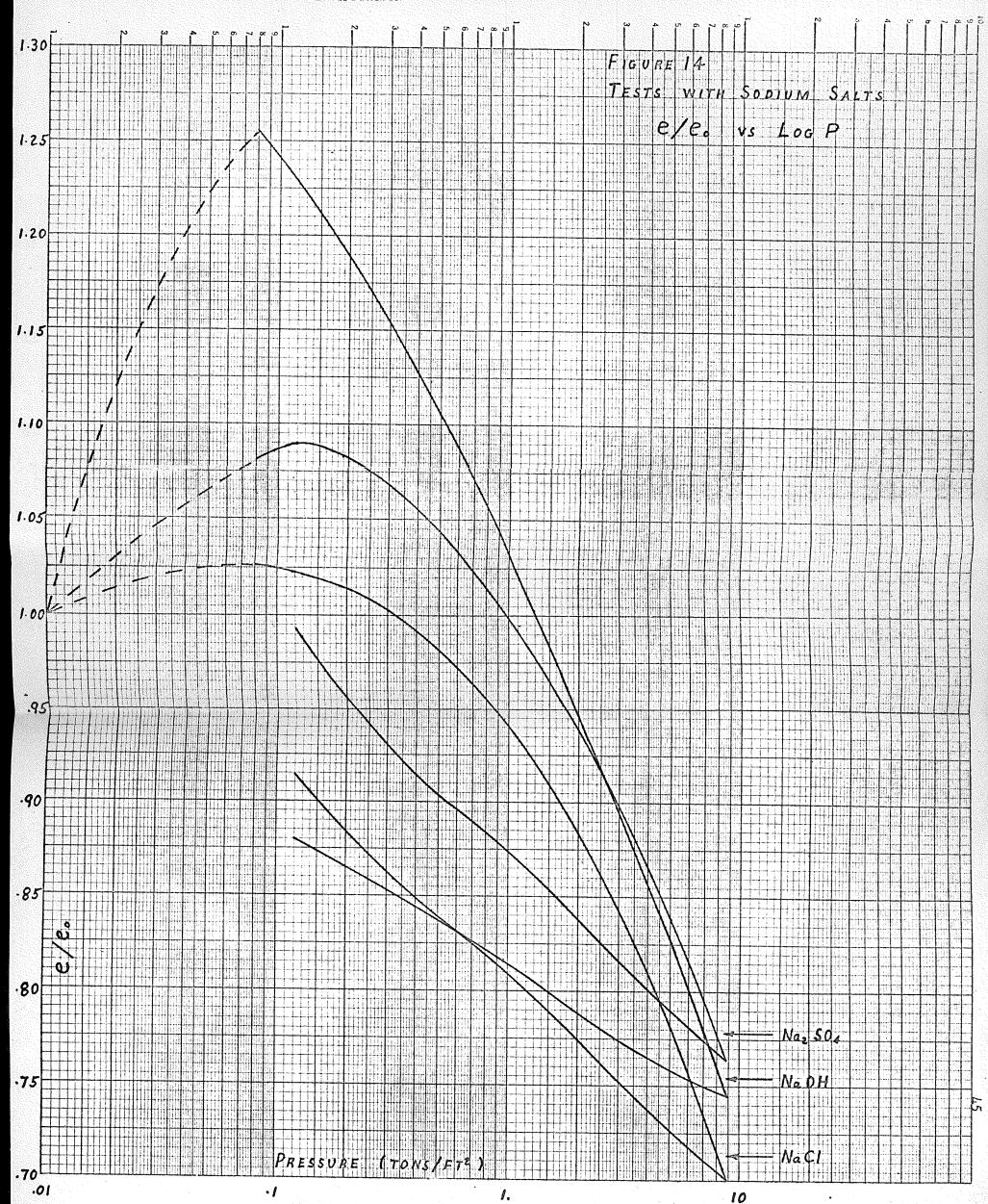


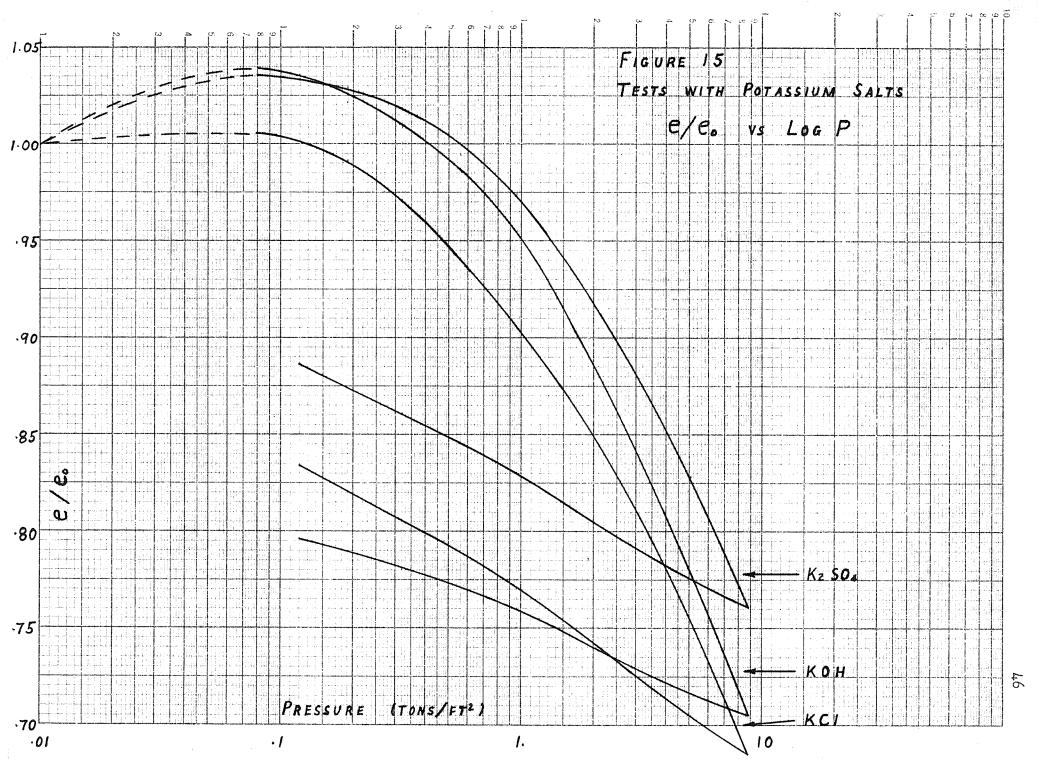


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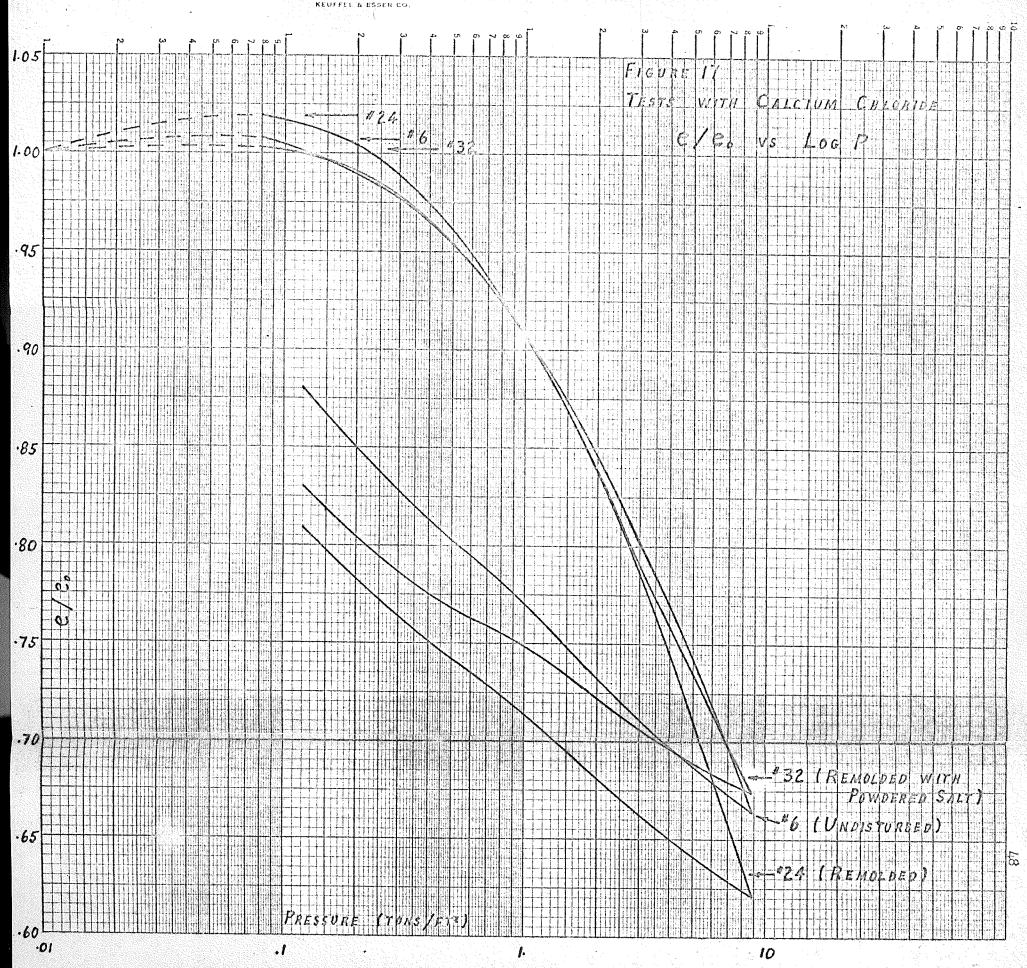
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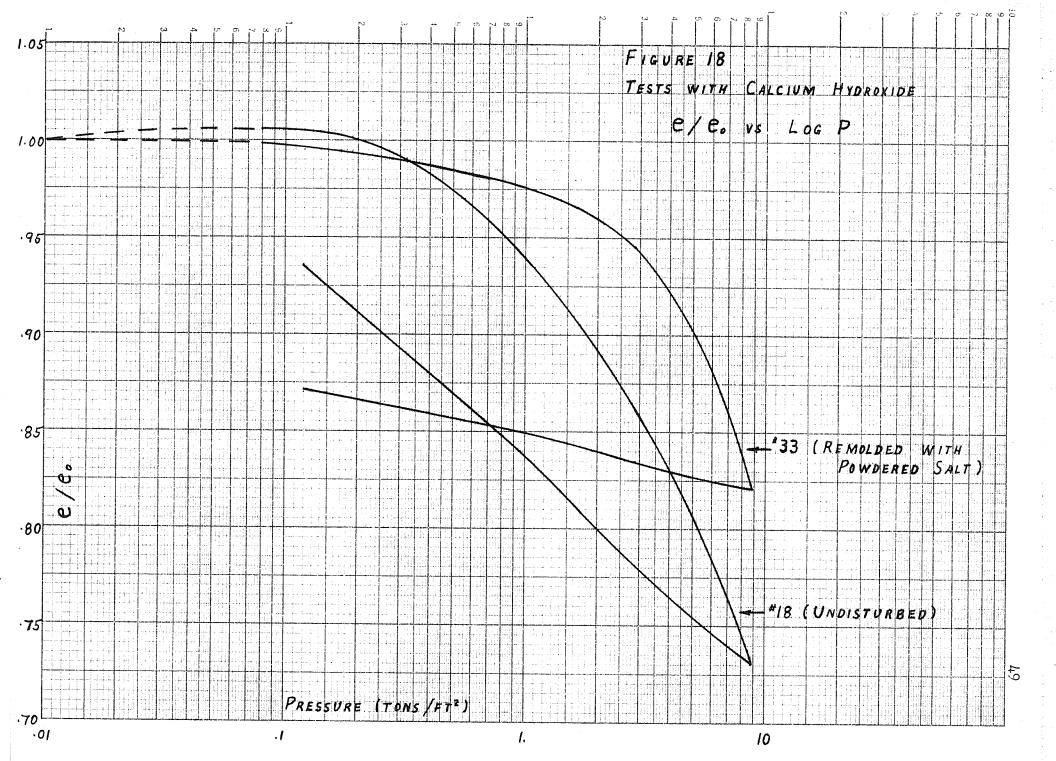




1.

10





Where the sodium salts were used in the swelling solution cation exchange was shown to have taken place. The sodium ion completely replaced the calcium in the interlayer water of the clay. This was also the case in test No. 20 where a mixture of liquid detergent and water was used for swelling. Again the sodium ion replaced the existing calcium.

In the tests where potassium salts were used, good exchange was noted especially with the chlorides and sulphates. However in the case of the potassium hydroxide the exchange was not complete. While the potassium was shown to be in the absorbed water layer some calcium or magnesium still remained.

Complete exchange also occurred when magnesium chloride and magnesium sulphate were used. Again the calcium was replaced by the magnesium cation.

When the calcium salts were used no exchange appeared to have taken place but the calcium ions in the interlayer water were intensified. This was more noticeable with the calcium hydroxide than with the calcium chloride. In test Nos. 32 and 33 the salts in powder form were mixed with the remolded clay. In these two instances, the exchangeable calcium ion was more greatly intensified than in the previous cases when the salts were applied in solution form.

The aluminum salts also produced exchange reactions with the calcium ion being replaced by the aluminum.

Ammonium chloride was used as a solution in one test. In this case, it is evident that the exchangeable calcium was replaced

by the monovalent ammonium ion.

In the above cases where the same solution was used on the soil in a relatively undisturbed state and in a remolded condition, there was no difference in the resulting mineralogical composition of the soil.

For two of the samples which were treated with sodium and potassium sulphate, separate DTA tests were done on the outside layers and on the center layer of the soil sample from the consolidation test. No difference was noted between the outside and the center layers; i.e. complete exchange occurred throughout the 0.6 inch thick sample.

On the thermograms of the samples treated with the sulphate salts, a few small unidentified peaks appeared which were not on the thermogram of the original untreated soil. These were probably due to the sulphate ions.

In general, the chloride salts appeared to produce a better exchange reaction.

The electro-chemical series gives the order in which elements will replace one another. A partial table of this series for the cations used in this testing is as follows:

> Potassium Sodium Calcium Magnesium Aluminum

Potassium, as it is at the top of the table, will replace all of the other ions below it when they are held in an exchangeable state. Likewise sodium will replace everything except potassium.

The differential thermal analyses has shown that both potassium and sodium cations have replaced the exchangeable calcium ion held in the interlayer water of the clay. However the magnesium and aluminum have also replaced the calcium which is contrary to the above table. This can be explained by the fact that the concentration of the magnesium and aluminum ions in solution is much greater than that of the calcium ion in the interlayer water. This high concentration overcomes the higher place that calcium holds in the electro-chemical series and exchange occurs.

Differential thermal analysis has an advantage over X-ray analysis in the identification of clay minerals. Both methods will provide an identification of the minerals but the DTA will also identify the exchange cations held in the interlayer water. The X-ray analysis will only show whether the ions are monovalent or divalent etc. In addition, the DTA is much faster and requires less preparation of the sample before testing. Variations in the heating rate of the DTA apparatus and drift due to an imbalance of the thermocouples do pose problems, but identification of the mineral is still possible. Both the X-ray and differential thermal analyses will provide qualitative results, but in each case quantitative results are only approximate, depending upon comparisons with standard samples.

### Chapter VI

#### Discussion of Results

The curves of e/eo vs log P provide a good method for comparing the relative amounts of swelling and consolidation for the various soil samples. Figure 9 is the e/eo - log P plots of tests 1 to 4 which were run with distilled water. These curves all lie within one fairly narrow band, but should, as there was only a small variation in the initial void ratios. Tests nos. 10 and 19, also with distilled water as the swelling solution are shown on Figure 10. The initial swelling and swell pressure of these two trials are considerably different probably due to the differences in initial void ratio, density and moisture content. However, the relative amount of consolidation for each is practically identical. Likewise the rebound curves are very similar.

Figures 11, 12 and 13 compare the effect of the various cations of the chloride, sulphate and hydroxide salts on the relative amounts of volume change of the soil.

The swelling characteristics of the soil, in undisturbed condition, for the different salt treatments are summarized in Table 2.( $\left(\frac{\Delta e}{e_0}\right)$ s is the maximum relative amount of swelling on the  $e/e_0$  - log P curve and Cs and Ps are from the e - log P curve.) From this table it is quite evident that both the cations and the anions in the interlayer water position of the clay minerals have a pronounced effect on the swelling properties of the soil.

TABLE 2
SUMMARY OF SWELLING CHARACTERISTICS OF THE TREATED SOILS

	$\frac{(\Delta e)}{(e_0)s}$	Ps	Cs
ORIGINAL UNTREATED SOIL	.026	•35	.210

	CHLORIDE			SULPHATE			HYDROXIDE		
SALT	$\frac{(\Delta e)}{(e_0)s}$	Ps	Cs	$\left(\frac{\Delta e}{e_0}\right)$ s	Ps	Cs	( <u>Ae</u> ) (e <sub>o</sub> )s	Ps	Cs
SODIUM	.027	.34	.172	.090	.98	.158	.256	1.3	.100
POTASSIUM	.011	.14	.116	•036	.56	.085	.039	.44	.082
CALCIUM	.008	.13	.141				•006	.21	.163
MAGNES IUM	.007	.14	.150	.021	•32	.158			
ALUMINUM	0	.06	.110	.046	•53	.100			
AMMONIUM	.014	.15	.118						

# (a) Effect of Cations on Swelling

Although there is a definite trend in the amount of swelling in association with each of the cations, sodium is the most
conspicuous. This cation causes a considerably higher swelling
pressure and thus more swelling than any of the others. The order
of cations for which the swelling is decreased for this particular
soil is as follows:

Sodium (most swelling)
Armonium
Potassium
Calcium, Magnesium
Aluminum (most stable)

This trend is evident with each of the chloride, sulphate, and hydroxide salts, although every cation does not appear
in each series.

In the sulphate series of tests, the aluminum treated soil swells less than the one treated with magnesium, but the aluminum sulphate has only a concentration of 0.5 molar compared to the 1 molar concentration of the other salts. It will be shown later that the ionic concentration also affects the volume changes of the soil.

From the above, it appears that the swelling characteristics of the soil vary with the valence of the exchangeable cations. Sodium, ammonium and potassium which are monovalent produce the most swelling while aluminum, a trivalent ion causes the least change. Calcium and magnesium, being divalent, have swelling characteristics between the two extremes.

This is essentially what was stated in Chapter 3. "Clays having monovalent ions such as sodium will take up more water than

those having divalent ions such as calcium".

Sodium and potassium are both monovalent ions but the relative amount of swelling produced by each is considerably different. The potassium has a fixing power upon the expanding lattice structure of montmorillonite. This gives the montmorillonite characteristics similar to that of illite.

The results of tests by Hardy<sup>(15)</sup> were different from those of this investigation. He found that treatment with calcium chloride reduced the swelling pressure from that of the original soil, and that treatment with sodium chloride produced a further reduction. This is contrary to the results of the above tests. No explanation of these conflicting results is readily apparent. In both cases, Hardy used saturated solutions, but a saturated solution of calcium chloride has a higher molar concentration than that of sodium chloride. Thus, the calcium chloride should produce a substantially lower swelling pressure than the sodium chloride, as will be shown in part d, Effect of Ion Concentration

### (b) Effect of Anions on Swelling

The anions appear to have an equally large effect upon the swelling properties of a clay soil, as do the exchangeable cations. From these tests it is obvious that chloride salts are the most effective in reducing the swelling of this soil. Even the sodium chloride, the worst of the series, produced swelling which was very close to that of the original soil. All of the other chloride salts greatly improved the swelling properties.

The sulphate salts, unlike the chlorides, tended to increase rather than decrease the amount of expansion of the soil.

Magnesium sulphate, which had the most stable effect of the series, only very slightly reduced the characteristics from that of the original soil. The sodium, potassium and aluminum sulphates noticeably increased the swelling, although aluminum would probably have been better with a concentration the same as the others.

Sodium and potassium hydroxide, like the sulphate salts, tend to increase the swelling of the soil. The effect of the potassium hydroxide is quite similar to that of the potassium sulphate, but the sodium hydroxide produced a much higher amount of swelling and swell pressure than the corresponding sulphate salt. On the other hand, calcium hydroxide had just the opposite effect. The swelling pressure was less than that of the original soil and the amount of swelling was very low. This closely ressembled the results produced by the calcium chloride.

The effect of the three anions are illustrated in Figures 14 and 15, showing the sodium and potassium salts respectively.

In the above discussions only the relative amount of swelling and the swell pressure were considered. It should be noted,
that the swelling index of each soil sample treated with salt was
reduced from that of the original soil. However there does not
appear to be any direct relationship between the various ions and
the swelling index.

Thus, in summary, the chloride salts with the exception of sodium, and calcium hydroxide all produced changes in the clay mineral composition which tended to stabilize the soil with res-

pect to swelling.

Figure 19 is a plot of the relative amount of swelling of the soil samples against the swelling pressure produced for all the consolidation tests run in this investigation. The points all lie within a fairly narrow band. For the lower range up to about 0.6 to 0.8 tons per sq. foot swelling pressure this band can be represented by a straight line. At higher pressures the line curves and the amount of swelling increases considerably with a slight increase in swelling pressure. Thus, if the swelling pressure which a soil can exert is known, the total amount of free swell can be estimated.

# (c) Effect of Ions on Compressibility

The consolidation characteristics for the soil samples treated with the various salts are listed in Table 3. ( $\frac{\Delta e}{e_0}$ ) is the maximum relative amount of compression from the  $e/e_0$  -  $\log P$  curve and Cc is from the e -  $\log P$  curve.) Generally the ions do not have as much effect on the compressibility of the soil as on the swelling. The sodium cation does cause the soil to compress the least while the calcium and magnesium ions (except for calcium hydroxide) appear to produce the greatest amount of consolidation. The effect of the potassium, ammonium and aluminum was intermediate and in no specific order.

The effect of the anions is shown in Figures 14 and 15 of the e/e\_o - log P curves of the sodium and potassium salts.

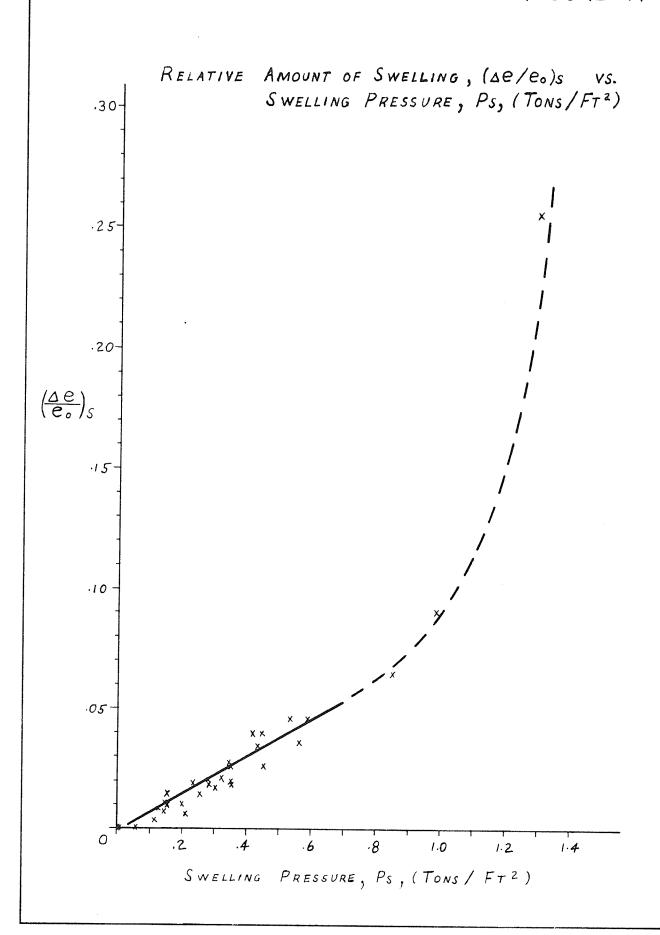


TABLE 3
SUMMARY OF CONSOLIDATION CHARACTERISTICS OF THE TREATED SOILS

	$\frac{(\Delta e)}{(e_{\Omega})c}$	Сс	
ORIGINAL UNTREATED SOIL	.261	.496	

	CHLORIDE		SULPHA	TE	HYDROX IDE		
SALT	$\frac{(\underline{\Delta} e)}{(e_{0})c}$	Cc	$\frac{(\Delta e)}{(e_0)c}$	Cc	$(\underline{\underline{A}}\underline{e})$ $(\underline{e}_{0})c$	Cc	
SODIUM	.299	.518	.236	.420	.255	.466	
POTASSIUM	.314	.380	.239	.347	.295	.458	
CALCIUM	.336	.400			.268	.448	
MAGNESIUM	.345	.403	.337	.500			
ALUMINUM	.304	.400	٠331	•500			
AMMONIUM	.306	375ء					

Sulphates induce the least compression, chlorides the most, and the hydroxides an intermediate amount.

In comparing the amount of consolidation of the salt treated soils to the original soil, in most cases the salt caused an increase in compression. However sodium and potassium sulphate and sodium hydroxide did improve the consolidation characteristics. Sodium hydroxide only slightly reduced the amount of swelling and the compressive index. The sodium and potassium sulphate reduced these values by a greater amount, but still not appreciably.

The compressive index, like the swelling index, was reduced from that of the original soil in all cases except for three salts where a very slight increase was noted. Again there is no correlation between the relative amounts of compression and the compressive index. Bolt (19) suggests that the ratio a = Cs/Cc is "indicative for the relative importance of the influence of the osmotic forces on the resistance of the clay soils against compression". In each test the "a" value was reduced from that of the untreated soil, and in some cases considerably. (See Table 1). However there is no direct relationship between "a" and either the amount of consolidation or swell.

In summary, the stabilization effect of the sulphate salts on the consolidation of the soil was small compared to the effect of the chloride salts on the swelling.

### (d) Effect of Ion Concentration

Figure 16 is a comparison of the effect of concentration of one salt on the amount of swelling and compression of the undisturbed soil. It is evident that the expansion of the soil was greatly affected by the increased concentration of the potassium chloride. The 1 molar concentration of the salt considerably lowered the amount of swelling from that of the original soil. But the 2 molar concentration further reduced the swelling potential so that no swelling was able to take place under the initial load of 0.08 tons per sq. ft.

In addition, the compressibility of the soil was also affected. The increased concentration of the salt substantially increased the amount of consolidation. This is proof of what was stated in Chapter 3: Increasing the electrolyte concentration causes a tendency toward flocculation of the clay particles.

Using van't Hoff's equation it is possible to calculate the ion concentration in the interlayer water of the clay minerals.

where P is swelling pressure (atmospheres)
R is gas constant (litre atmospheres)
(OA. Moles)

T is absolute temperature (oKelvin)

Cc is ion concentration midway between clay particles (moles/litre)

Co is ion concentration away from influence of clay particles (immersing solution) (moles/litre)

1 atmosphere pressure = 14.7 psi

therefore 1 ton/sq. ft. =  $\frac{2000 \times 1}{144}$  = 0.945 atmosphere.

Average temperature =  $72^{\circ}F$  =  $22.2^{\circ}C$  =  $295.4^{\circ}A$ 

Thus P = 24.25 (Cc - Co)

For the tests with distilled water, assuming that the ion concentration in the immersing solution is zero or negligible and using the measured swelling pressure, the ion concentration in the interlayer water can be calculated as follows:

Test #10 Ps = 0.58 tons/sq. ft.

Co = 0

 $Cc = \frac{0.945 \times .58}{24.25}$ 

- 0.0226 moles/litre (molar)

#19 Ps = 0.35 tons/sq. ft.

Co = 0

Cc = 0.0137 moles/litre

It appears that the ion concentration in the interlayer water of the natural soil is very low. This is for the position midway between the clay particles; adjacent to the particles the concentration would be higher.

Considering the tests using the undisturbed soil immersed in solutions of potassium salts:

Test #5 KCl l molar

Ps = 0.14 tons/sq. ft.

Go = 1 moles/litre

 $(0c - 1) = \underbrace{0.945 \times .14}_{24.25}$ 

Cc = 1.0055 moles/litre.

 $\#13 \text{ K}_2\text{SO}_{l_4} \text{ 1 molar}$ 

Ps = 0.56 tons/sq. ft.

Co = 1 moles/litre

Cc = 1.0219 moles/litre

#16 KOH 1 molar

Ps = 0.14 tons/sq. ft.

Co = 1 moles/litre

Cc = 1.0172 moles/litre

#21 KCl 2 molar

Ps = 0.01 tons/sq. ft.

Co = 2 moles/litre

 $(Cc - 2) = \frac{0.945 \times .01}{24.25}$ 

Cc = 2.00039 moles/litre

For the above three salts with 1 molar immersing solutions, the potassium cation concentrations in the interlayer water are similar. In these cases, as the exchangeable ion concentration increases, so does the swelling pressure. This is contrary to what was stated above but can be easily explained. This irregularity is due to the effect of the anions and/or pH as is discussed in other sections of this Chapter.

Comparing the effect of the 1 molar and 2 molar potassium chloride solutions better results are obtained. The 2 molar immersing solution produced an increase in the interlayer cation concentration over that of the 1 molar test, and as previously mentioned reduced the swelling.

The swelling pressure varies directly as the difference in the ion concentration in the interlayer water and in the swelling solution. In the above mentioned cases, the test with the 2 molar potassium chloride solution had the smallest difference and thus the lowest swelling pressure. The ion concentration in the interlayer water is always slightly greater than that of the immersing solution.

The following calculation shows that the 1 molar concentration of the swelling solutions used in this investigation was sufficiently strong to produce complete replacement of the exchangeable ions (calcium) held in the interlayer water of the clay minerals.

Average weight of soil solids = 38 grams

Average volume of swelling solution = 500 ml (0.5 litres)

Ion exchange capacity of soil = 40-50 milliequivalents

per 100 gms (use 45)

Avogadro's Number = 6.0235 x 10<sup>23</sup>

(Number of electronic charges in 1 litre of 1 molar

solution)

Ion exchange capacity of soil sample = 45 x 38 = 17

milliequivalents.

Therefore soil can exchange 17 x 10<sup>-3</sup> x 6.0235 x 10<sup>23</sup>

= 1.02 x 10<sup>22</sup> electronic charges

Available charges in solution = 0.5 x 6.0235 x 10<sup>23</sup> =

3.01 x 10<sup>23</sup>

Therefore there are 3.01 x 10<sup>23</sup> monovalent ions in solution available for exchange

or 1.505 x 10<sup>23</sup> divalent ions available

or 1.003 x 10<sup>23</sup> trivalent ions available

Thus, there are sufficient ions (electron charges) in the solution to completely replace the exchangeable ions held by the clay. The 0.5 molar concentration of aluminum sulphate is even strong enough to permit complete replacement.

# (e) Effect of Remolding the Soil

A number of tests were done by treating the remolded soil with different salts, and an extreme variation in results was found. In general the relative amounts of swelling and compression of the remolded soil was greater than that of the undisturbed soil. However, this was not the case in several tests where the opposite was noted. In addition the effect of the salts upon the remolded soil was not always the same as upon the un-

disturbed. In some instances the salt reduced the swelling, etc. for the remolded soil where it was increased with the undisturbed soil, and vice versa. This variation was probably a result of the degree of remolding and change in the physical soil structure, possibly to a more random orientation of particles.

In consolidation tests nos 32 and 33, the dry powdered calcium chloride and calcium hydroxide were mixed with the remolded soil. Figure 17 is a comparison of the e/e<sub>0</sub> - log P curves for the tests with the calcium chloride salts. When the salt was applied in solution to the remolded soil, there was a noticeable increase in both the swelling and the consolidation over that of the undisturbed soil. However, when the powdered calcium chloride was mixed with the remolded soil, both the amount of swelling and consolidation were substantially decreased, even less than that of the undisturbed soil.

Figure 18 is the  $e/e_{\rm O}$  - log P curves for the tests with the calcium hydroxide. The powdered calcium hydroxide mixed with the remolded soil had the same effect as the calcium hydroxide only to a greater extent. Here the swelling and swelling pressure was reduced to practically zero and the amount of consolidation was also appreciably lessened.

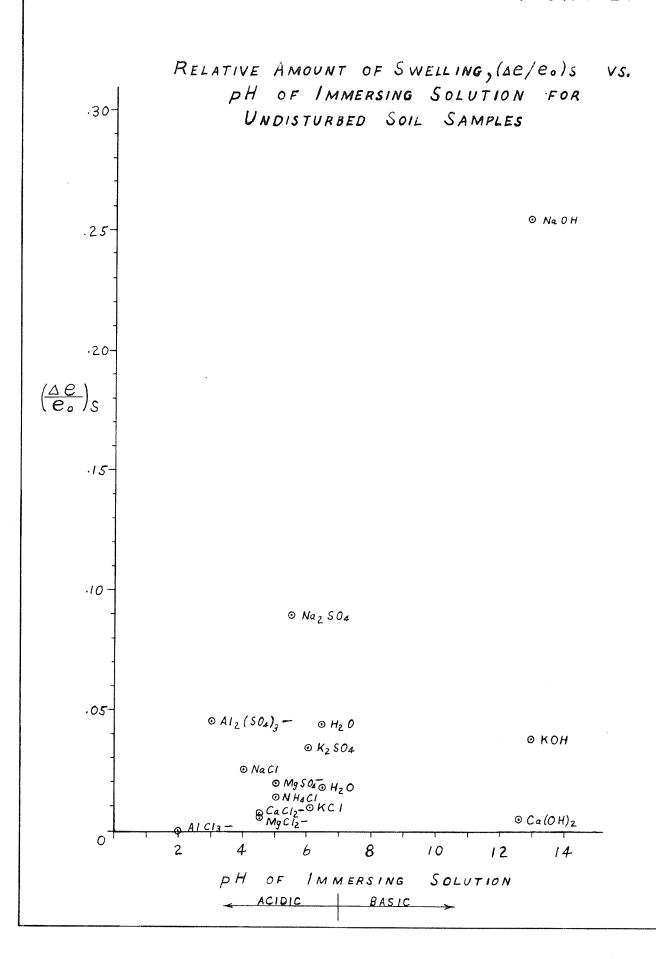
In addition, in this final test with calcium hydroxide, the deformation under the initial half of the loading cycle was considerably less than in all the other tests. This shows that the strength of the soil had increased.

The reason that the soil became more stable when mixed with the powdered calcium chloride and hydroxide was shown in the differential thermal analyses. The calcium exchange ion was greatly intensified in these two tests, whereas in the other tests with the calcium salts there was only slight intensification. This increased the ion concentration in the absorbed water, in the same way as the 2 molar concentration of potassium chloride.

### (f) Effect of pH of Immersing Solution

Figure 20 is a plot of the relative amount of swelling versus the pH of the immersing solution for the undisturbed soil samples. There is no direct correlation between the amount of swelling and the pH of the swelling solution. However one relationship is evident. For each particular cation (e.g. sodium), the chloride has the lowest pH, the sulphate slightly higher and the hydroxide the highest. (An exception is potassium chloride and potassium sulphate which have the same pH.) This is the same order in which anions affect the swelling. Chlorides produce the least swelling, sulphates more, and hydroxides generally the greatest amount. Thus it appears that for any particular cation, the salt which has the lowest pH (most acidic) will produce the least swelling in the soil.

# FIGURE 20



### Chapter VII

### Summary and Conclusions

From the results of this investigation several conclusions can be drawn.

- (1) The volume change characteristics of the clay soil in the Winnipeg area can be controlled through treatment with inorganic chemicals. It can reasonably be assumed that other montmorillonitic clay soils will similarly be affected.
- Differential thermal analyses (DTA) has definitely established that cation exchange took place in the interlayer water of the clay minerals when the soil samples were immersed in the various chemical solutions. However the DTA did not reveal anion exchange.
- Both the exchangeable cations and anions have been shown to affect the amount of swelling and compression of the soil. Chloride salts change the soil so as to reduce the swelling characteristics while the sulphates and hydroxides tend to increase the amount of swelling. The anion with the lowest pH appears to have the greatest stabilizing effect. The effect of the cations varied with their valency. Trivalent ions such as aluminum caused only a relatively small amount of swell compared to the monovalent ion like sodium. Calcium hydroxide (lime) is an exception to the rule and like the chlorides promotes stabilization of the soil, possibly through its cementing action.
- (4) The effect of the different ions on compressibility of the soil is small with respect to that on swelling.
- (5) Increasing the concentration of the exchangeable ions increases the stability of the soil.

One point that has not been discussed in this investigation is how or when does ion exchange take place. A possible explanation is that exchange occurs when there is a reduction of load on the soil. This happens twice during each test. The

first time occurs when the sample is initially immersed in the swelling solution and the effective stress on the soil grains is decreased. The tension in the pore water has a suction effect which is reduced when the solution with the ions enters the soil. Ion exchange then takes place. This would be similar to the action of a sponge.

The second load reduction is at the end of the test during the rebound cycle. The load is actually removed from the soil and swelling is allowed with the same effect as above.

However most of the exchange is thought to take place at the beginning of the test as the initial swelling characteristics of the soil are altered by the various ions.

### Chapter VIII

#### Future Work

### (a) Practical Applications

Now that it has been shown that ionic stabilization of expanding clay soils is possible, at least in the laboratory, the next problem is the application of these results in the field.

Probably the best method of treating the undisturbed soil would be through the use of vertical sand drains. The salt solutions, chlorides of aluminum, calcium, magnesium or potassium, would be pumped into the sand drains and allowed to permeate the adjacent soil or be forced through electro-osmotically. A pump system outside the area to be treated would drain the excess water. In this way the depth of stabilization would be controlled by the depth of the sand drains. The only problem would be in determining the spacing of the drains.

This method would be superior to that of spraying the surface of the soil as Winterkorn<sup>(17)</sup> discusses, as stabilization occurs only in the upper few inches of the soil by this latter method.

Holtz<sup>(7)</sup> gives an example of electro-chemical stabilization of a canal bank in which chloride salts were used. A 1:1 mixture of 7% KCl and 3% AlCl<sub>3</sub> was pumped through the anode while drainage water was removed at the cathode for a period of  $5\frac{1}{2}$  months. Calcium chloride was first introduced to open the soil, i.e, increase the permeability. The anode spacing was at 30 feet but favourable stabilization only occurred within 4 feet of the anode. It was found that potassium replaced the exchange-

able sodium ion in the soil and that aluminum aided the stabilization. Although there was a strength increase in the soil, stabilization was not considered to be adequate as heaving still occurred in the concrete lined sections of the canal.

Calcium hydroxide (lime) has been successfully used in stabilization of many soils especially for roadwork, etc. However stabilization only takes place for a depth of two or three feet where the soil has been remolded.

## (b) Laboratory Studies

There is still much work to be done in the study of ionic stabilization of clay soil. In this investigation only chloride, sulphate and hydroxide salts were used. Phosphates and nitrates are also common ions found in the soil whose effect on volume change of expansive soils is yet unknown. In addition, there are numerous cations such as lithium, iron, copper, etc. as well as organic compounds, especially those with long chain-like molecules, to be studied. However study should be limited to chemicals which are fairly common and economical to obtain.

As mentioned in the previous chapter more investigation is necessary to determine how ion exchange occurs, how much time is necessary, and through what thickness of soil exchange will take place.

Additional work is required on determining the proper concentration of salt solutions to be used. Aside from the economical aspect, the concentration of say aluminum chloride solution should be just strong enough to prevent swelling without

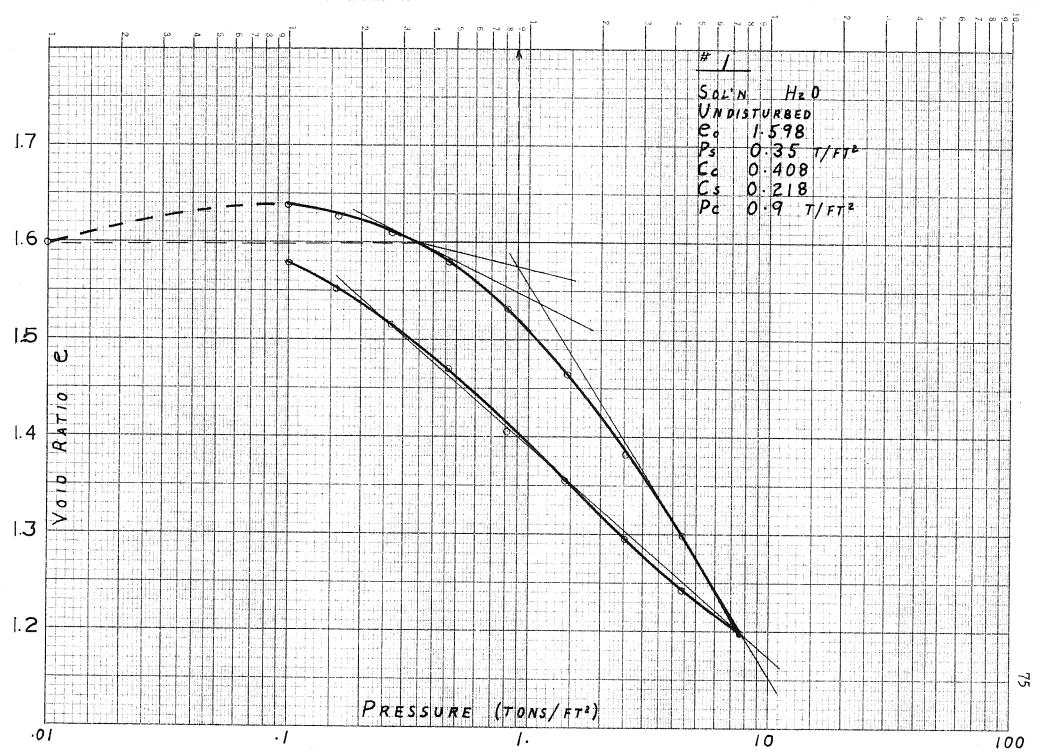
substantially increasing the amount of consolidation.

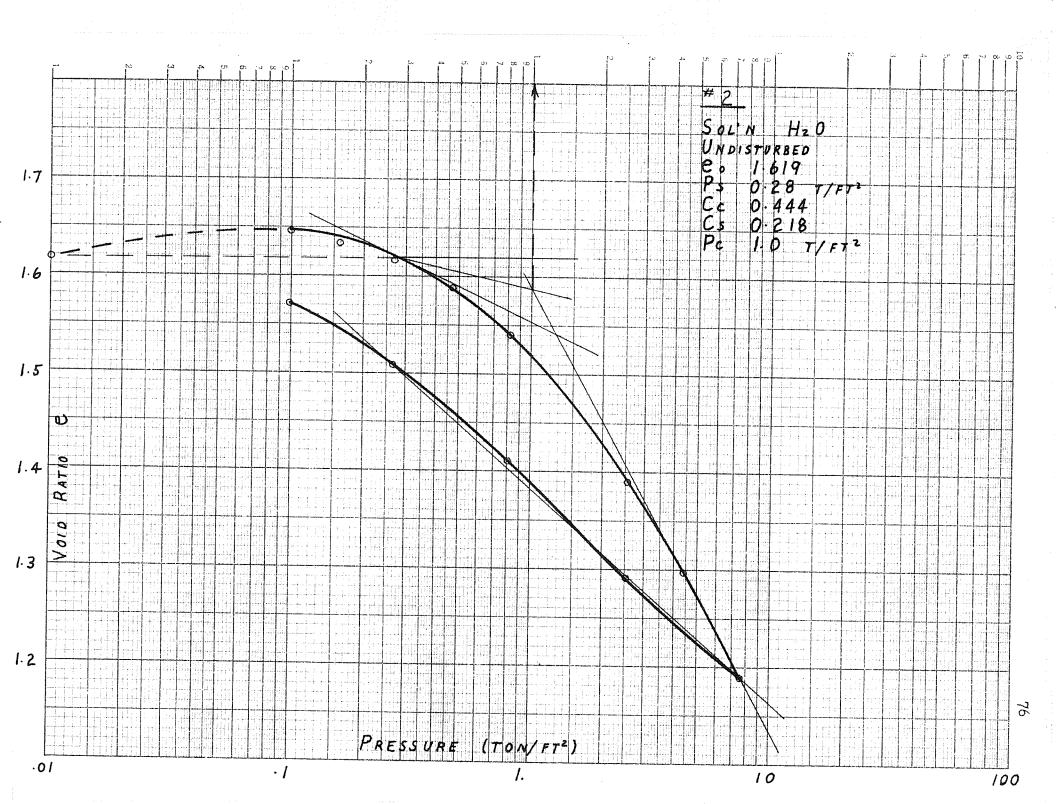
The effect of the various ions on other physical properties of the soil such as Atterberg Limits, strength and permeability is still open to study.

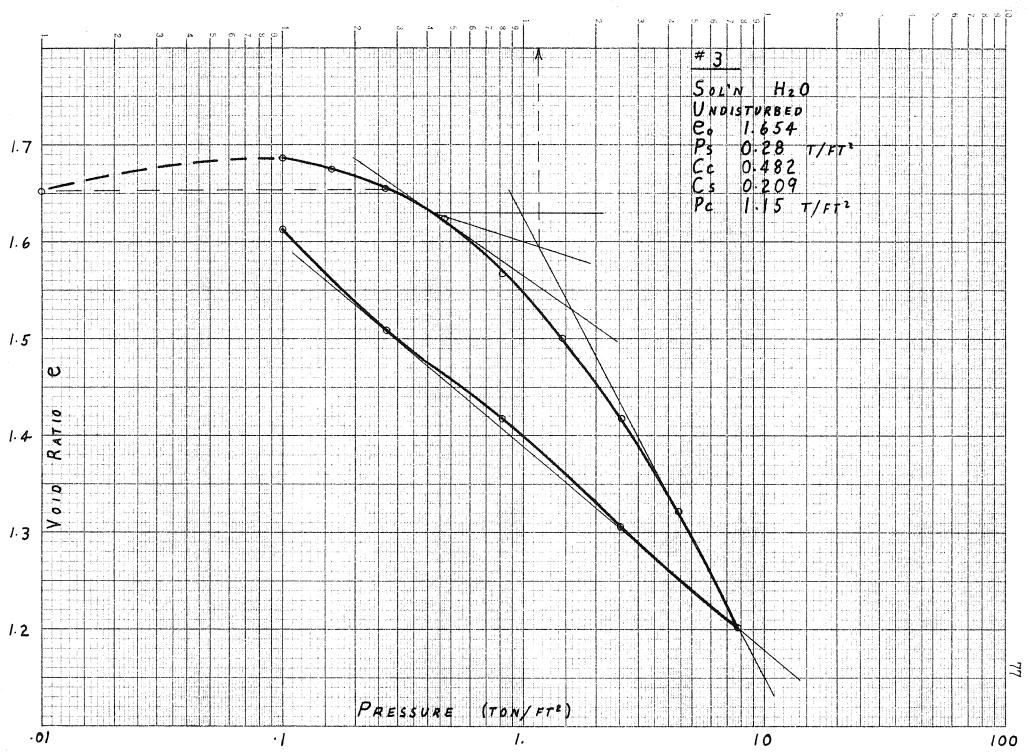
As previously mentioned X-ray analysis and differential thermal analysis provide the best methods for the identification of expanding type clay minerals. Electron microscope studies provide structure, d-spacing and particle size of minerals. However the use of relatively simple tests for finding the ion exchange capacity of the soil and determining the nature of the exchangeable cations, together with the standard consolidation test will provide a relatively good identification of troublesome clay minerals. Adequate measures can then be taken through proper design and stabilization to prevent serious detrimental ground movements.

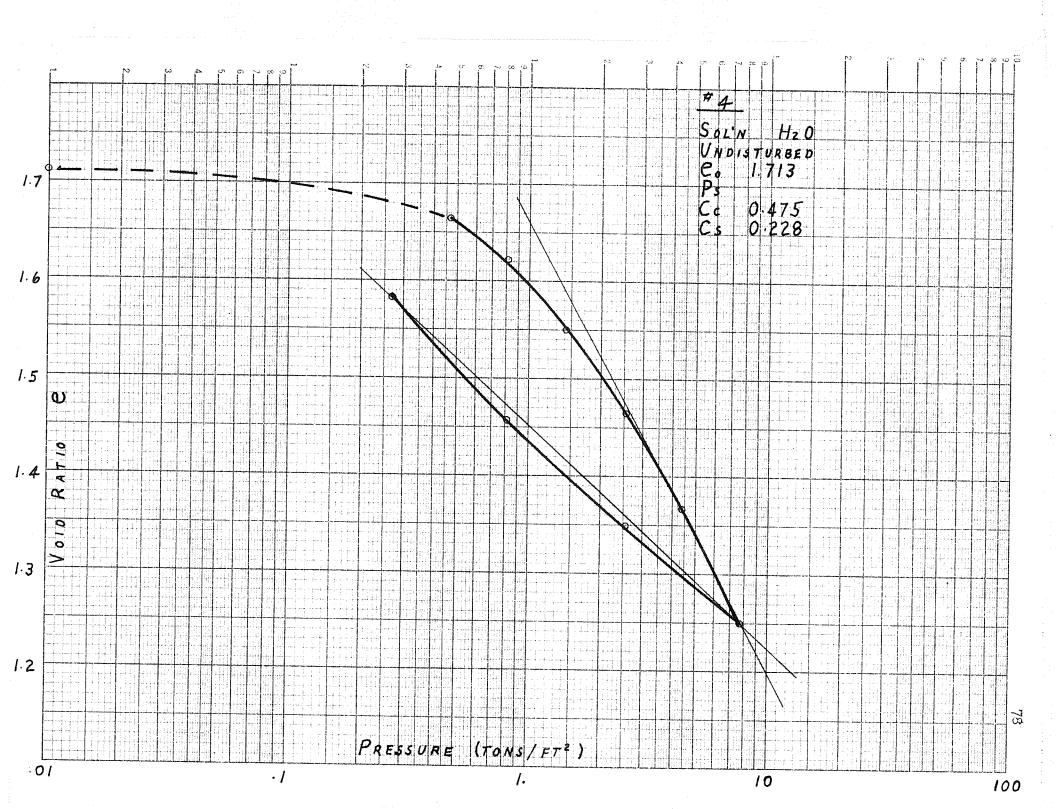
# APPENDIX A

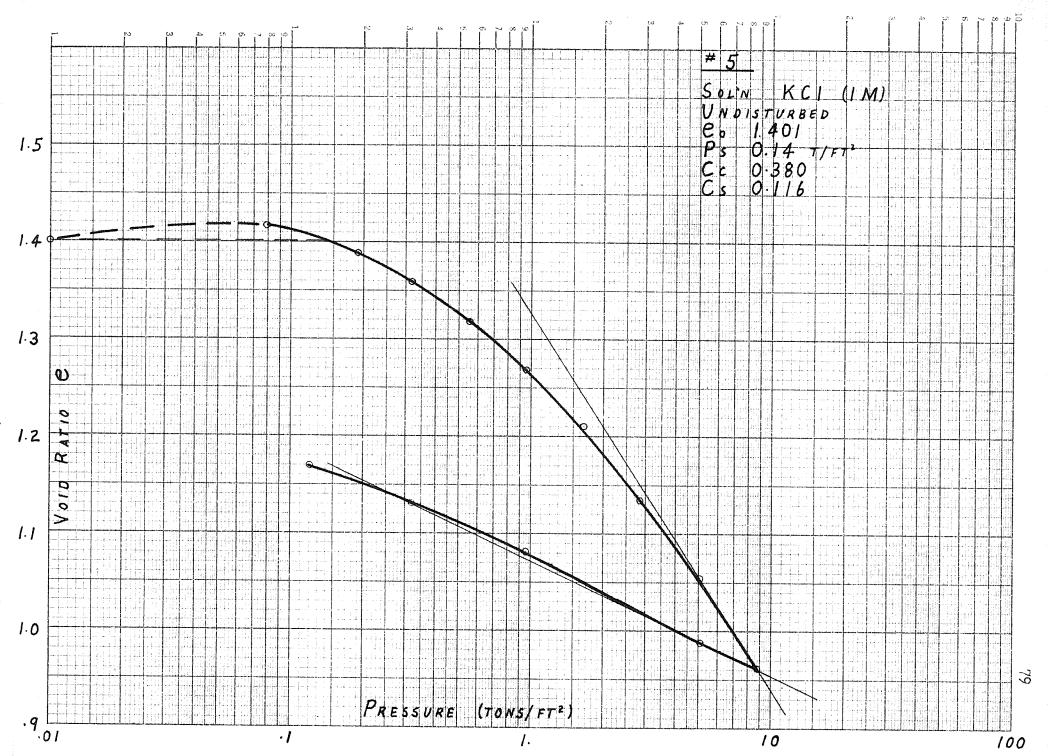
e - log P curves of the Consolidation Tests

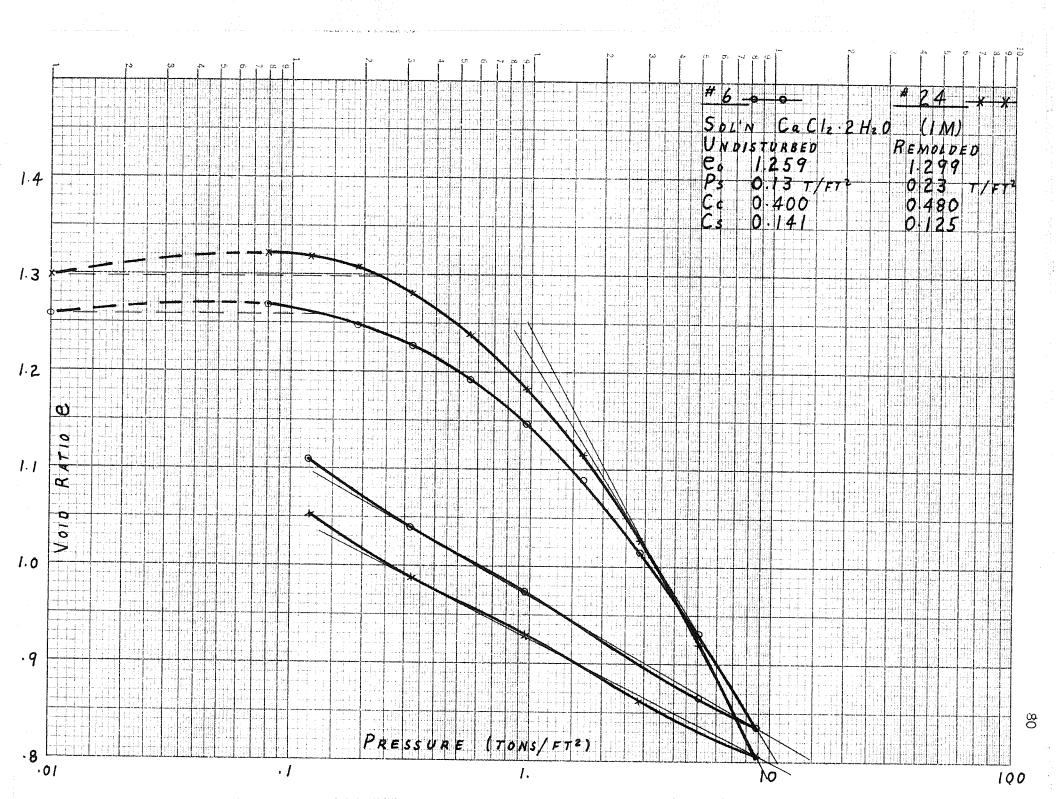


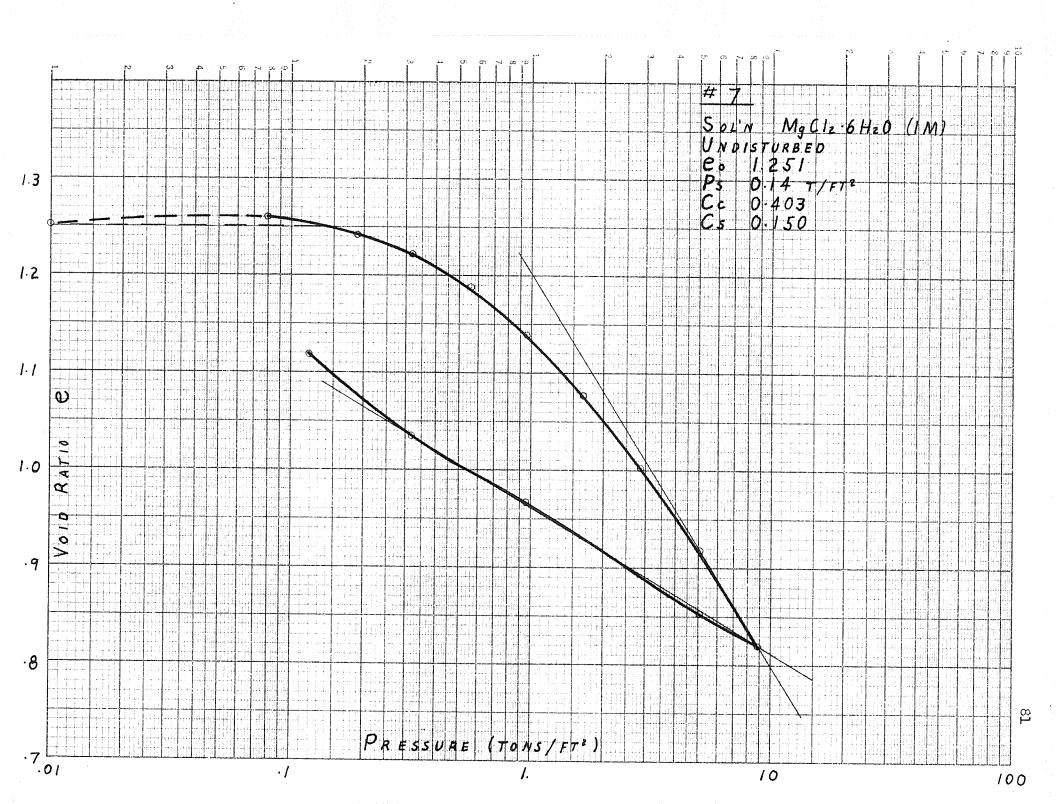


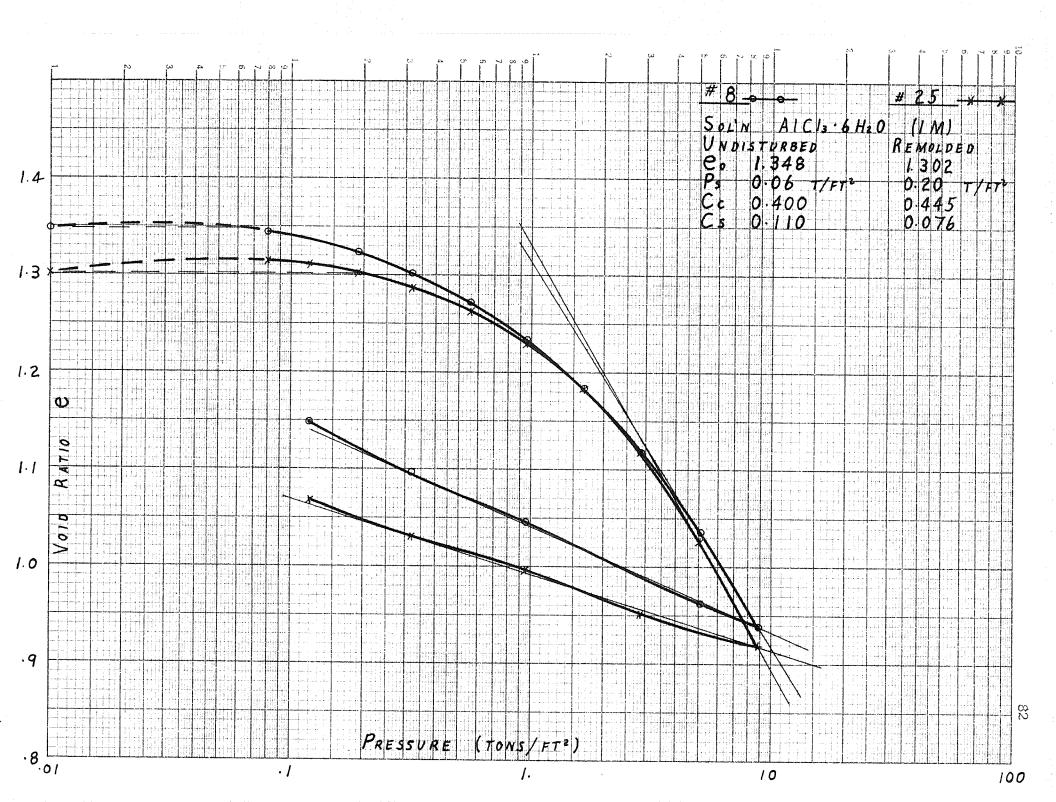


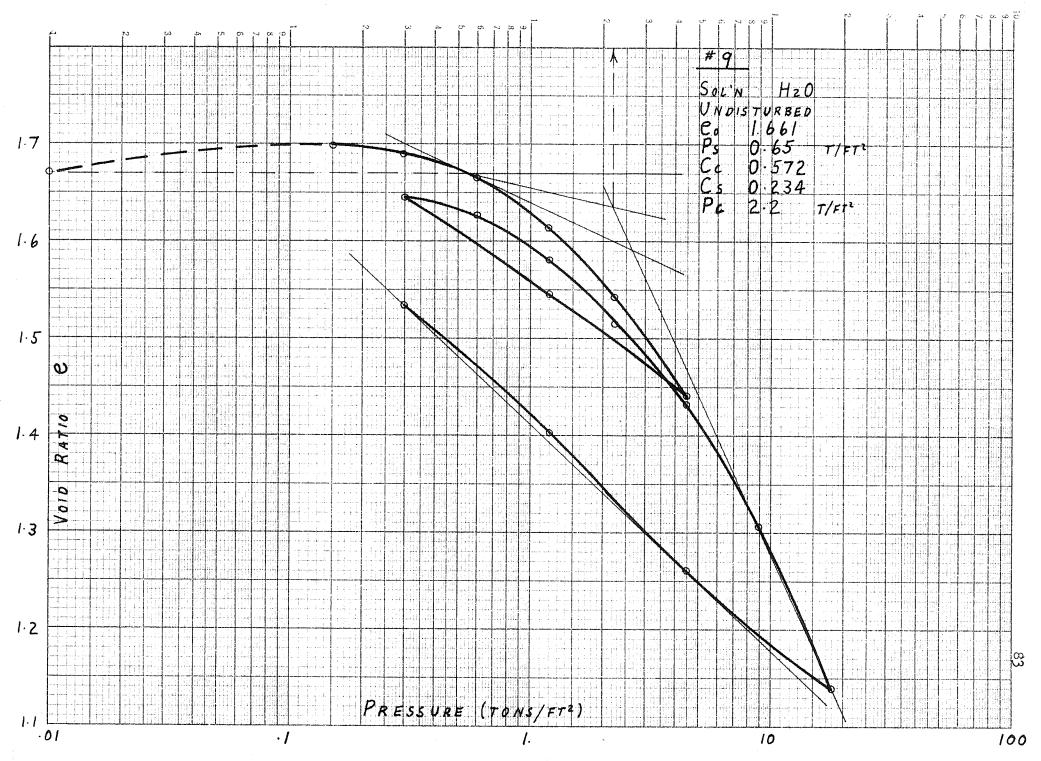


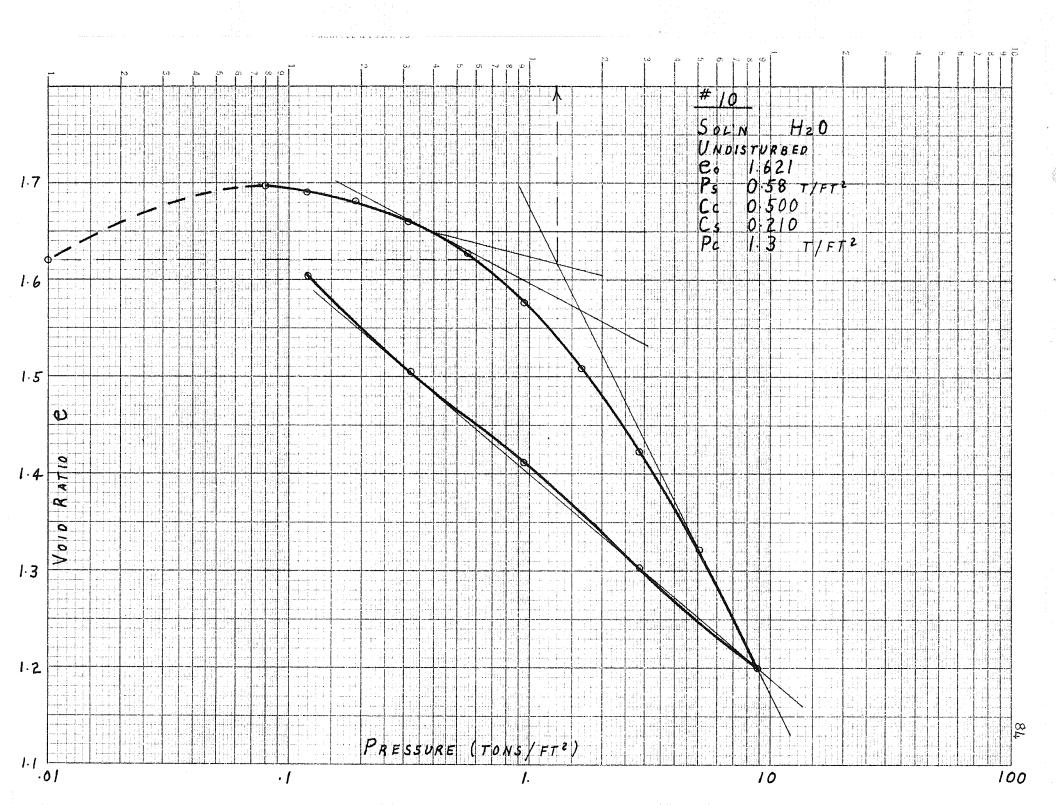


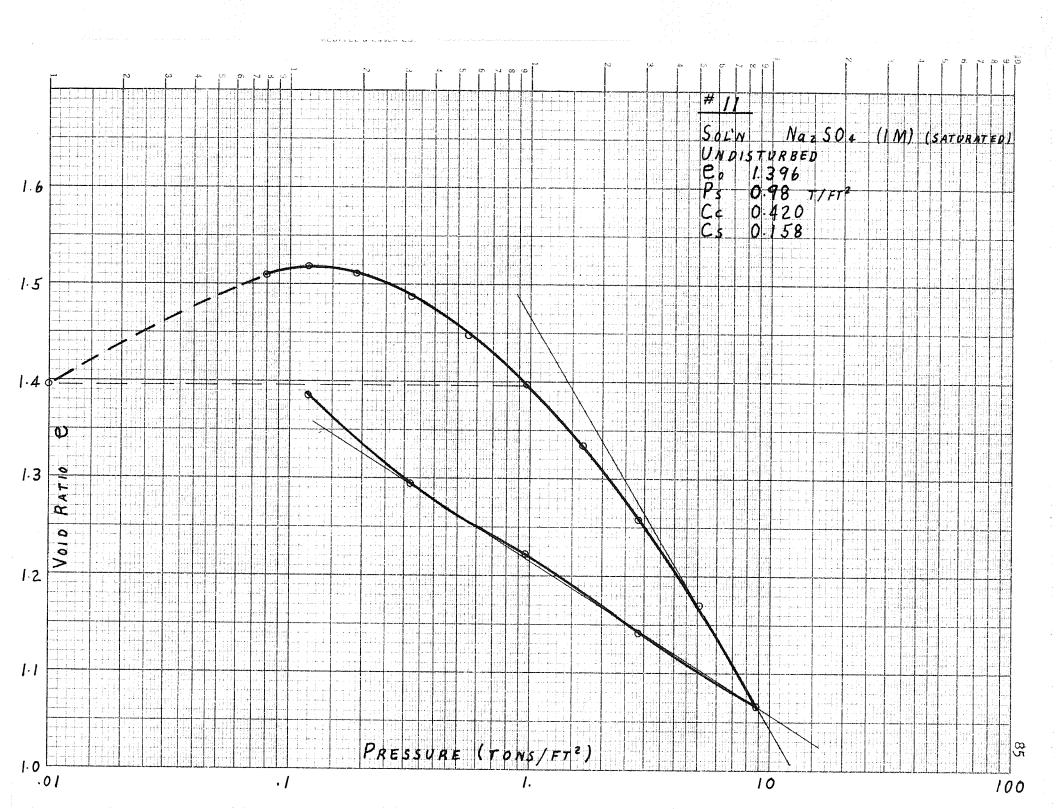


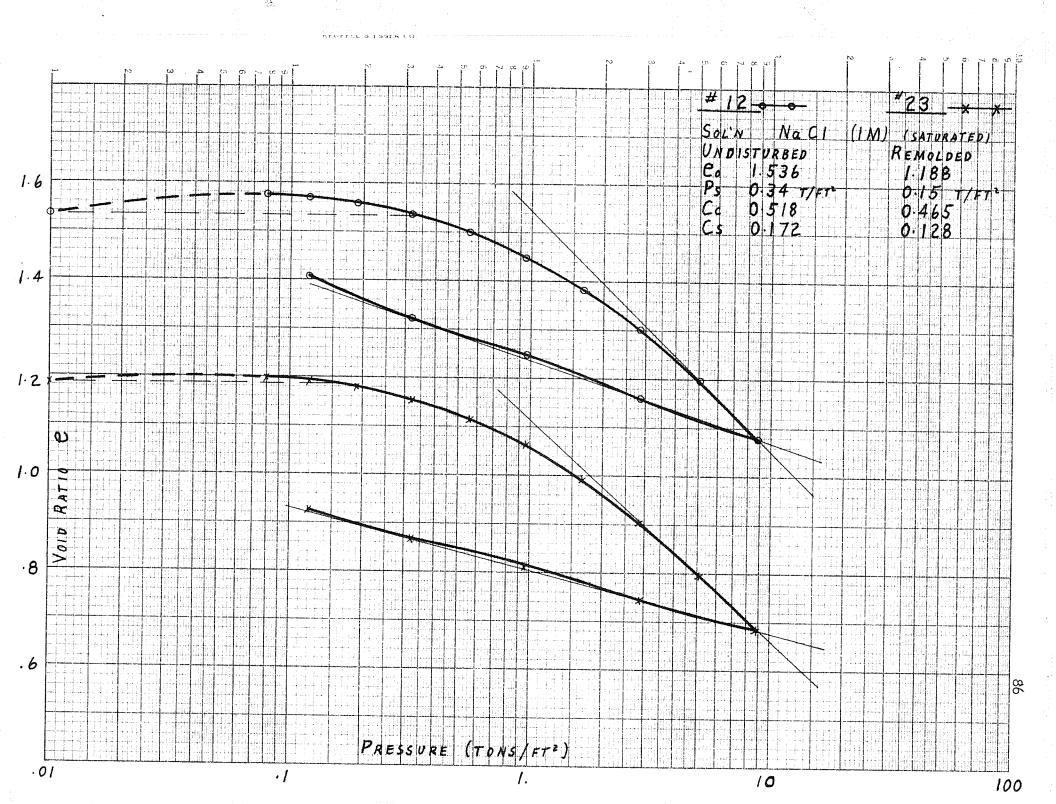


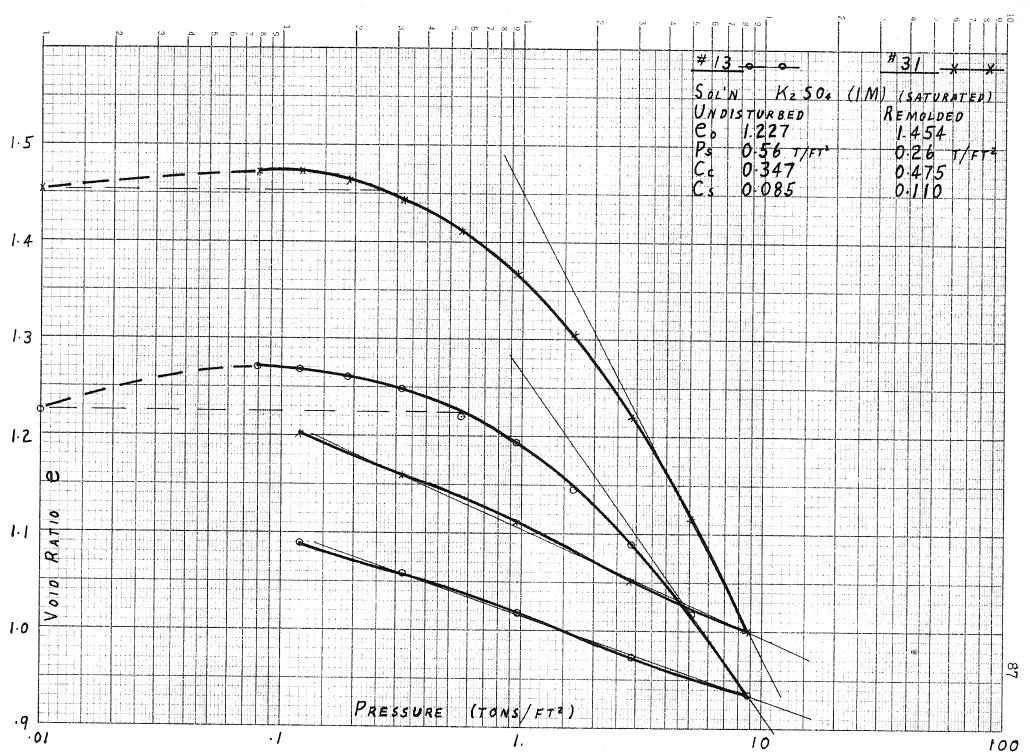


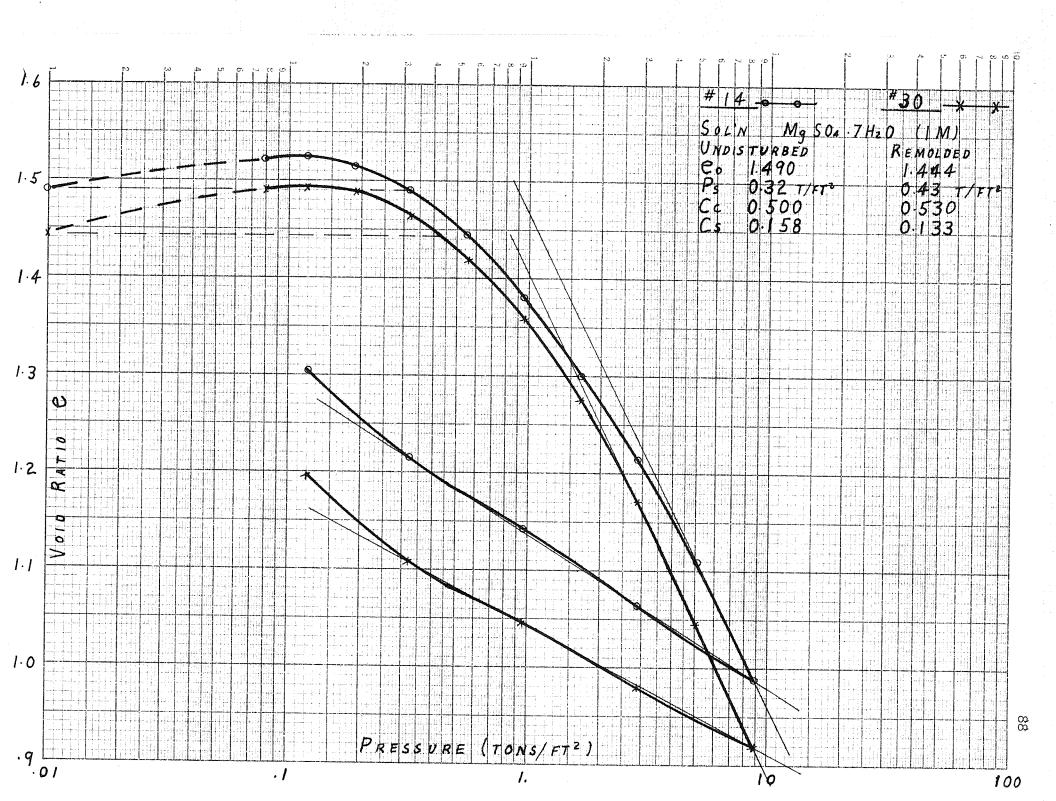


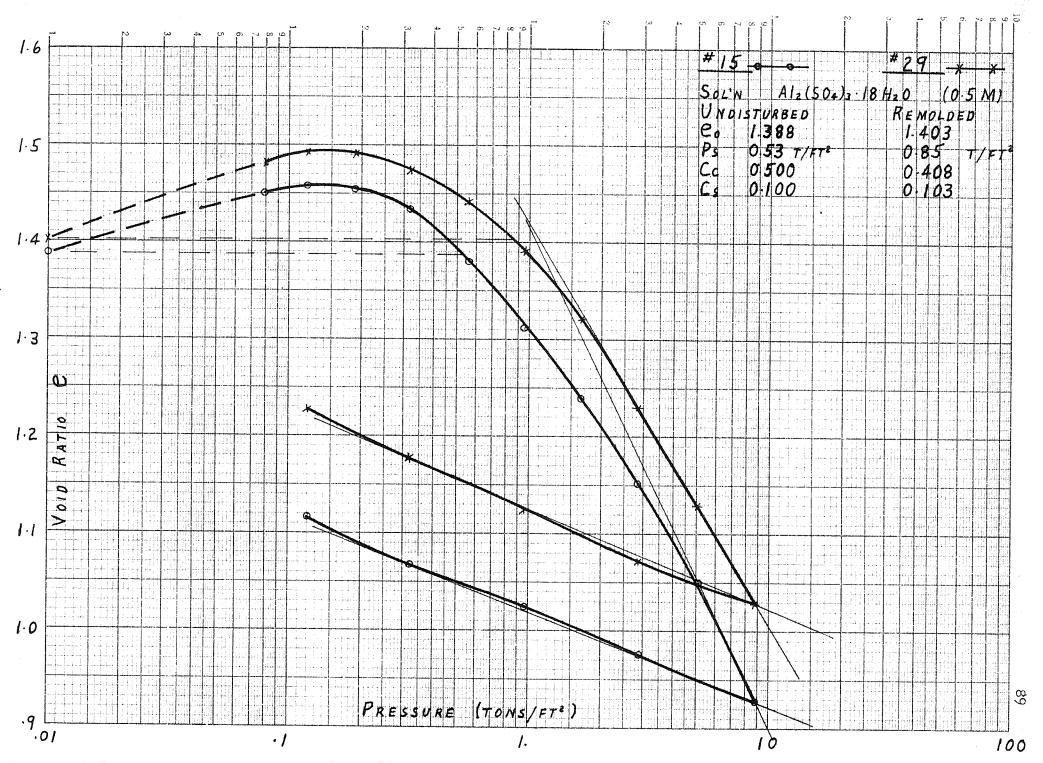


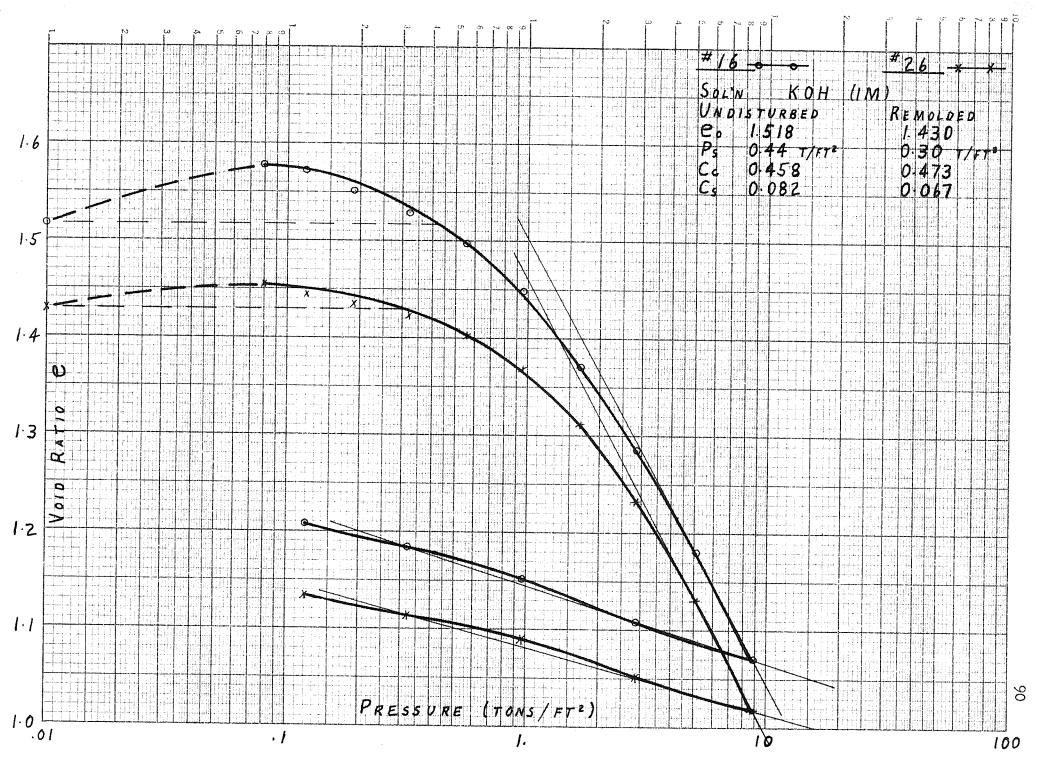


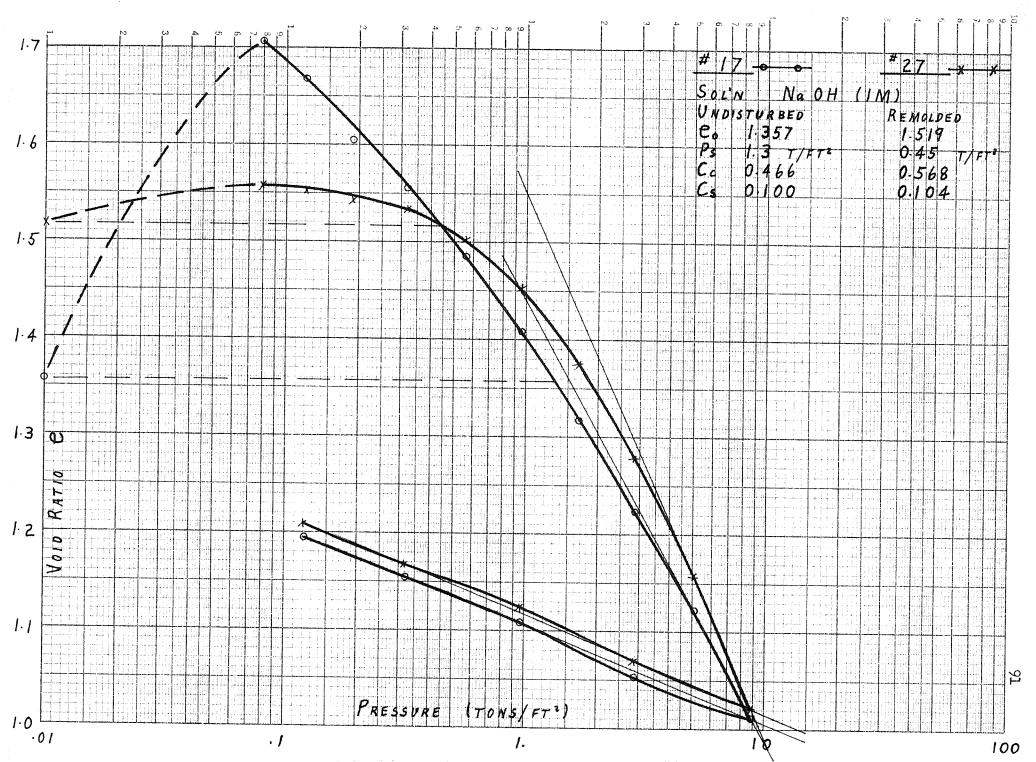


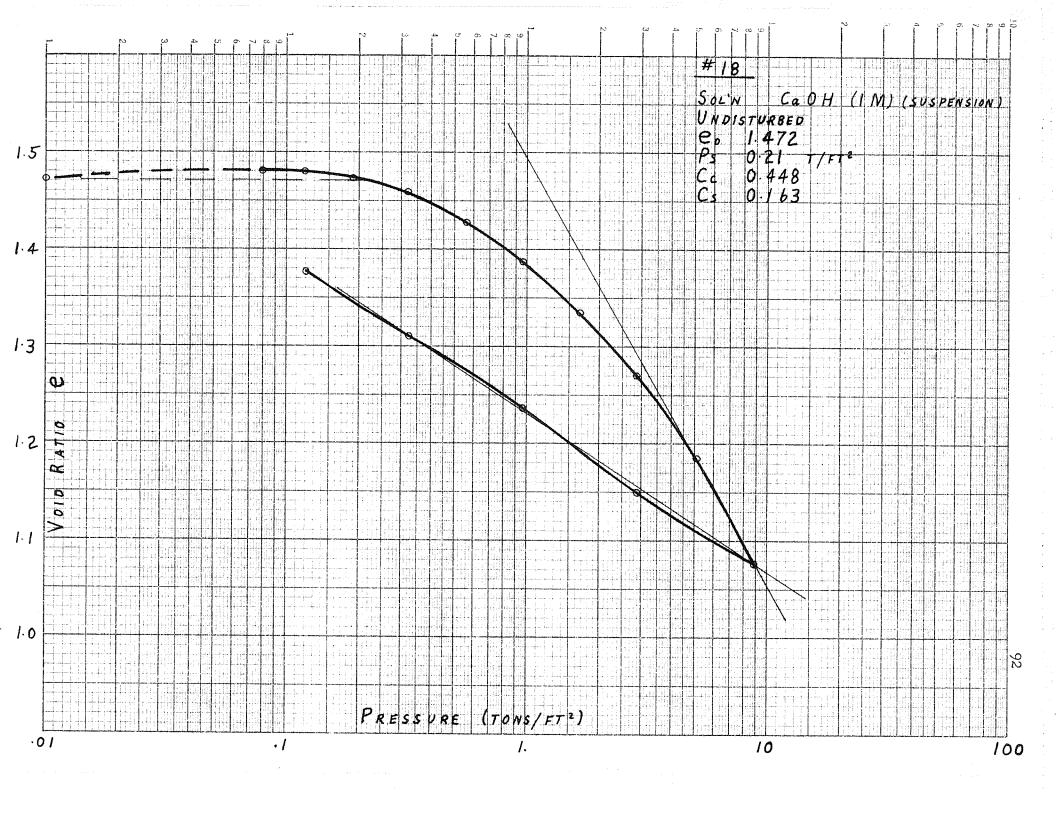


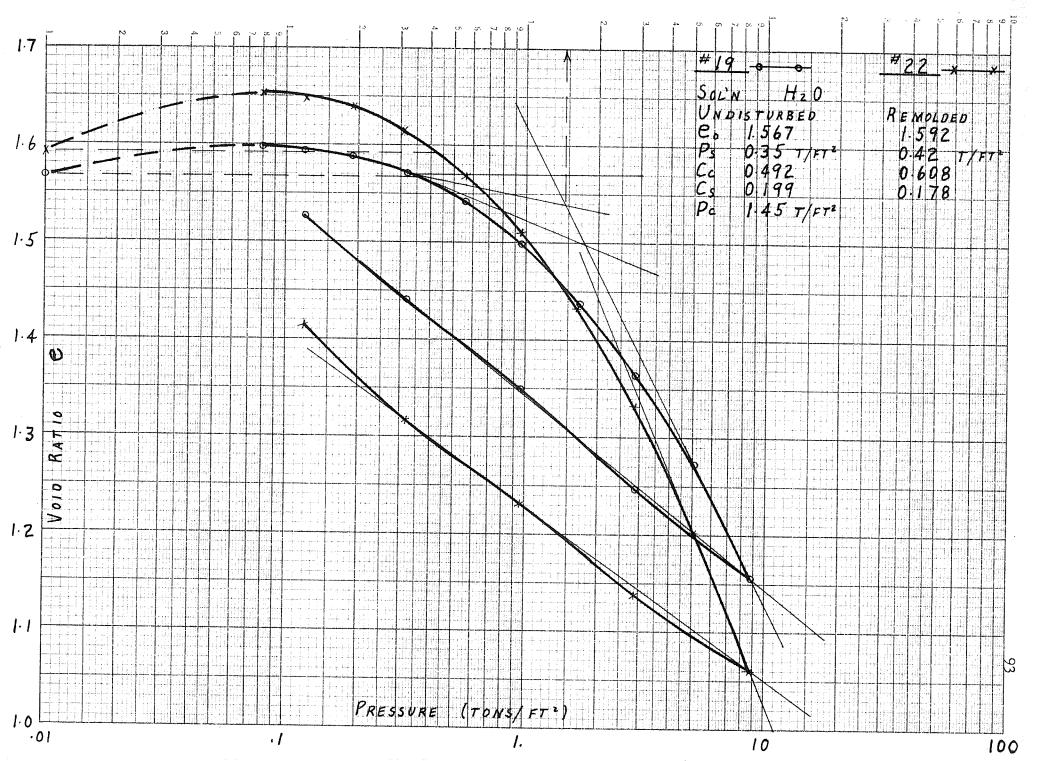


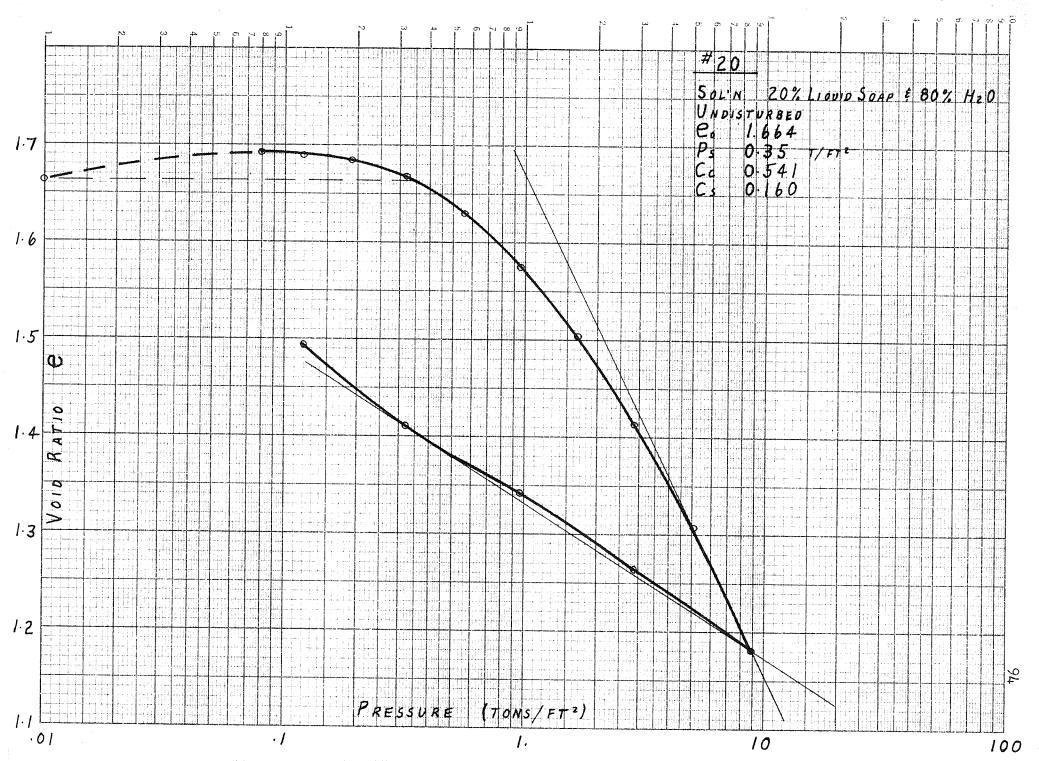


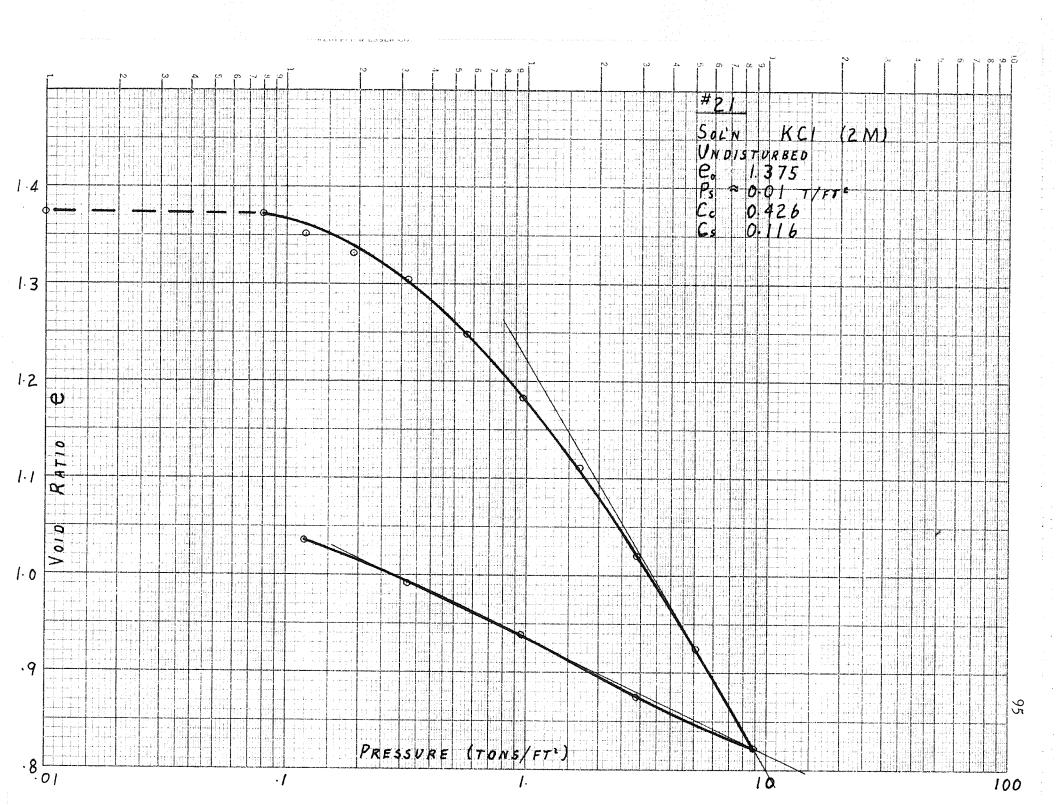


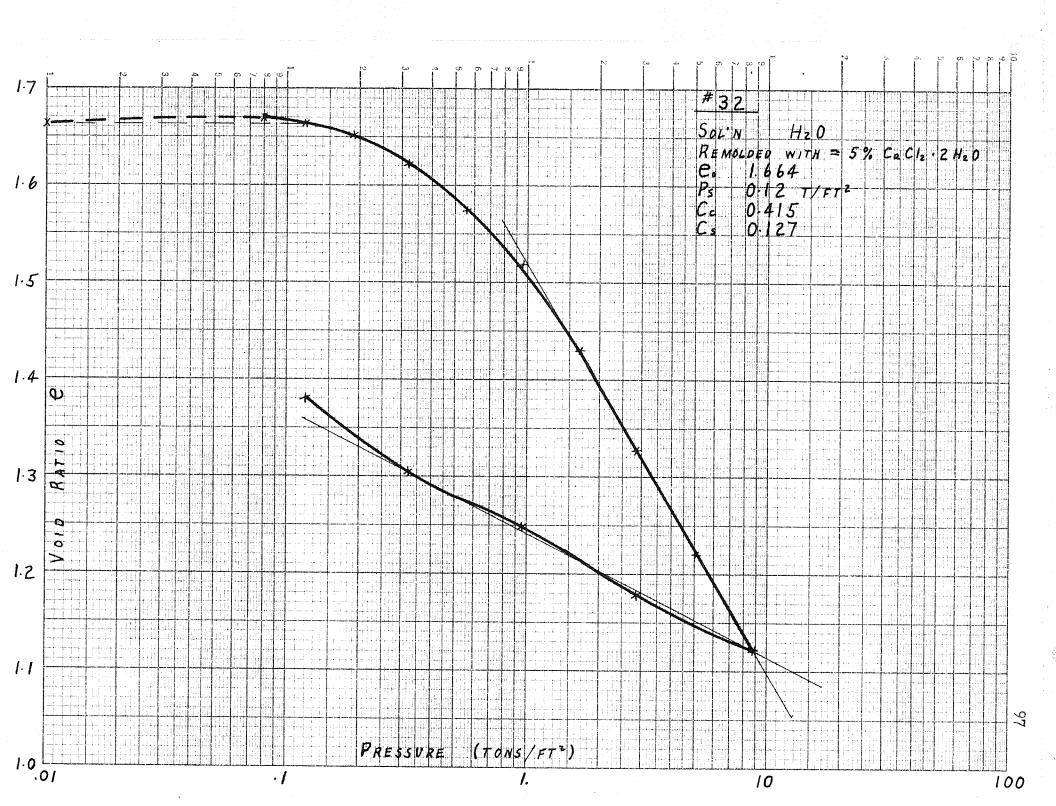


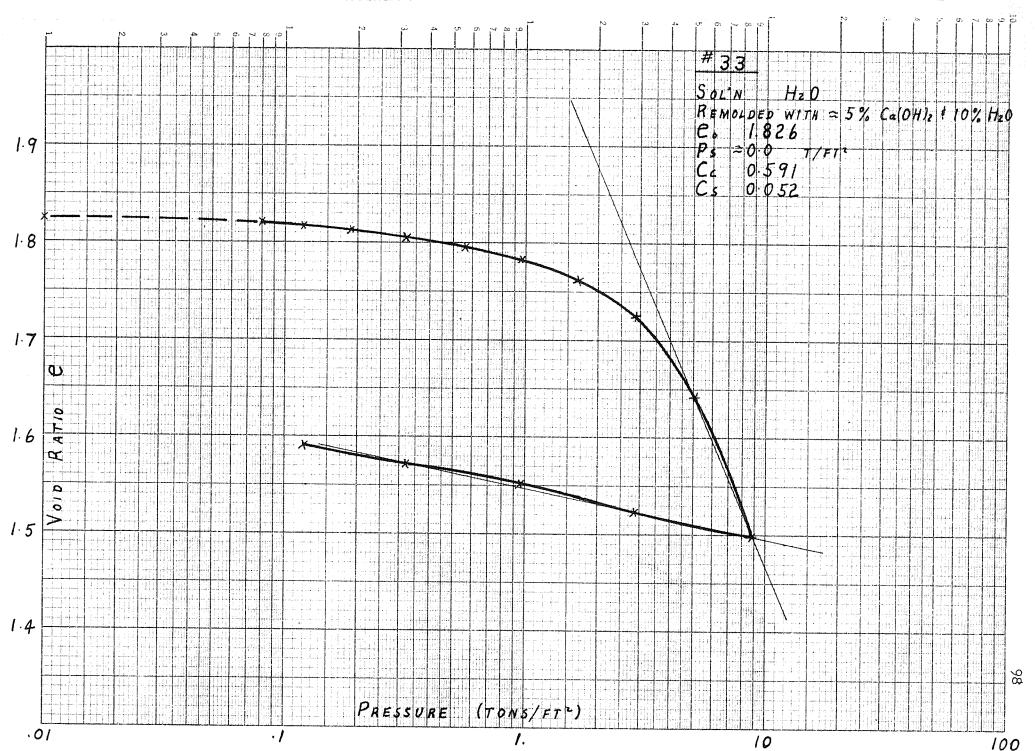












# APPENDIX B

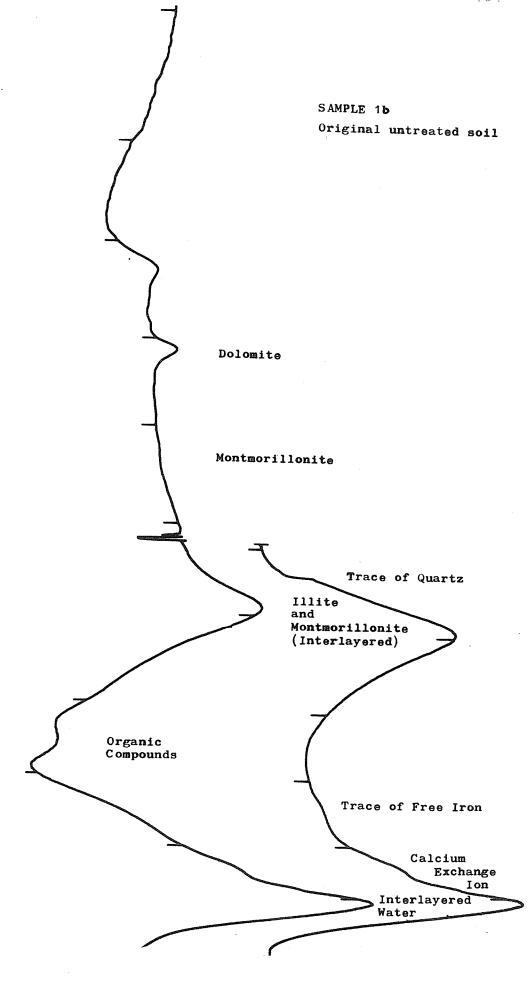
THERMOGRAMS FROM THE DIFFERENTIAL THERMAL ANALYSES

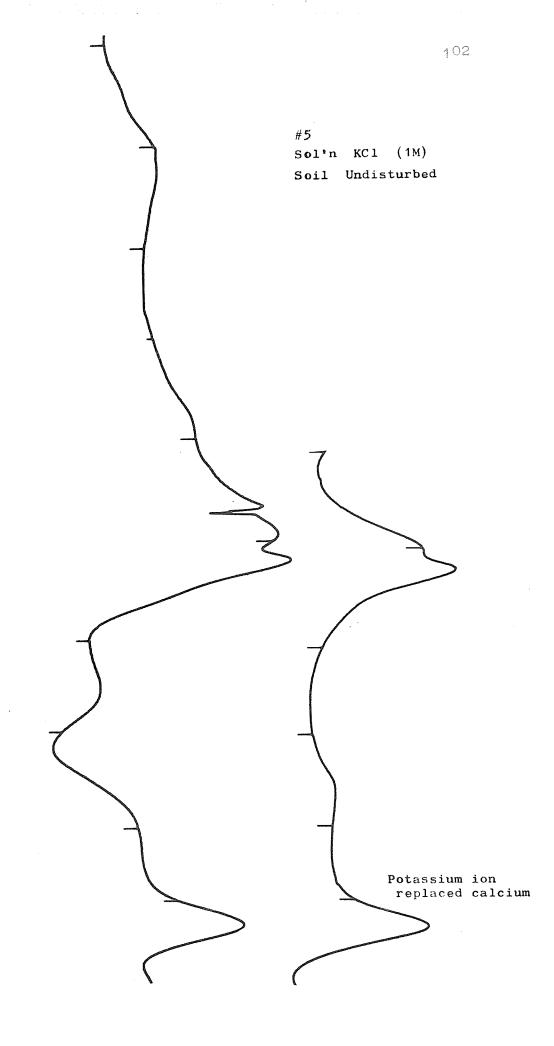
### Explanation of Thermograms

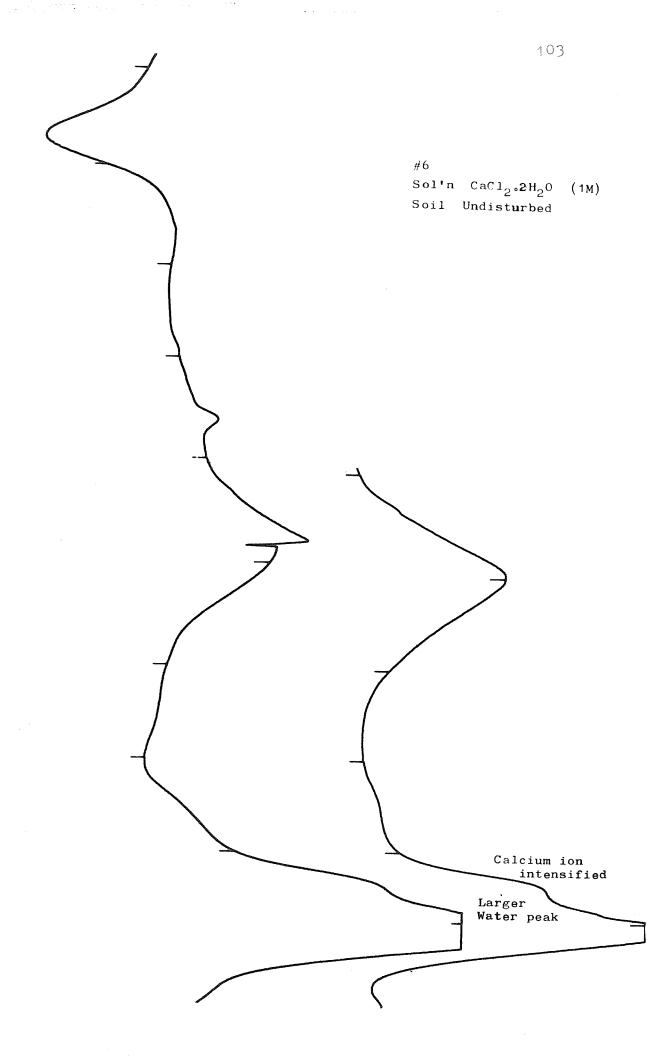
The curves on the right side of the pages were run in an atmosphere of nitrogen up to a temperature of 600°C. The curves on the left were run in an oxygen atmosphere up to between 500°C and 600°C; the atmosphere was then changed to carbon dioxide and the run continued to 1000°C.

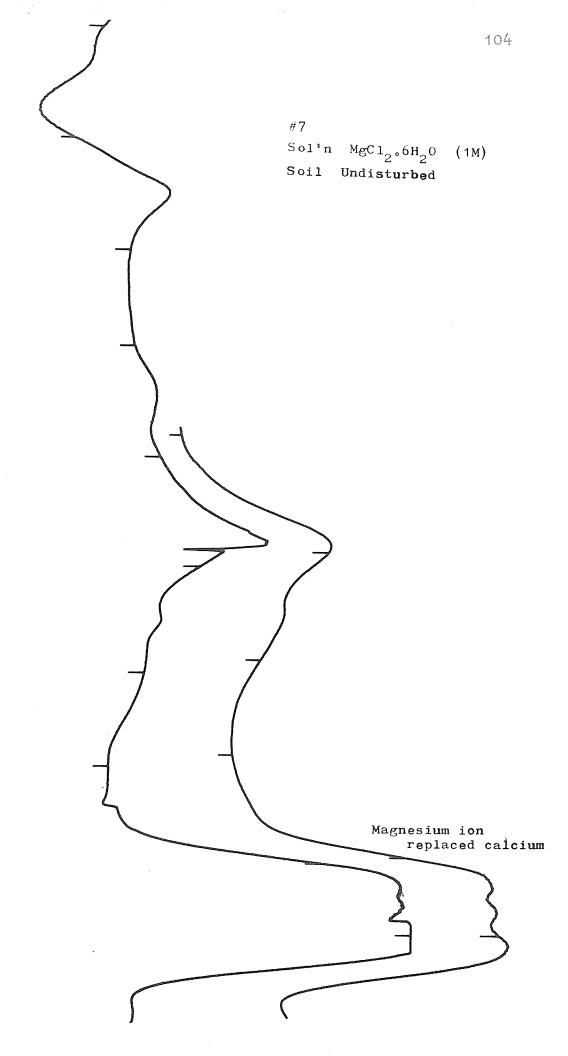
The zero temperature (of the standard) is at the bottom of the page and each small horizontal line represents a temperature increase of 100°C.

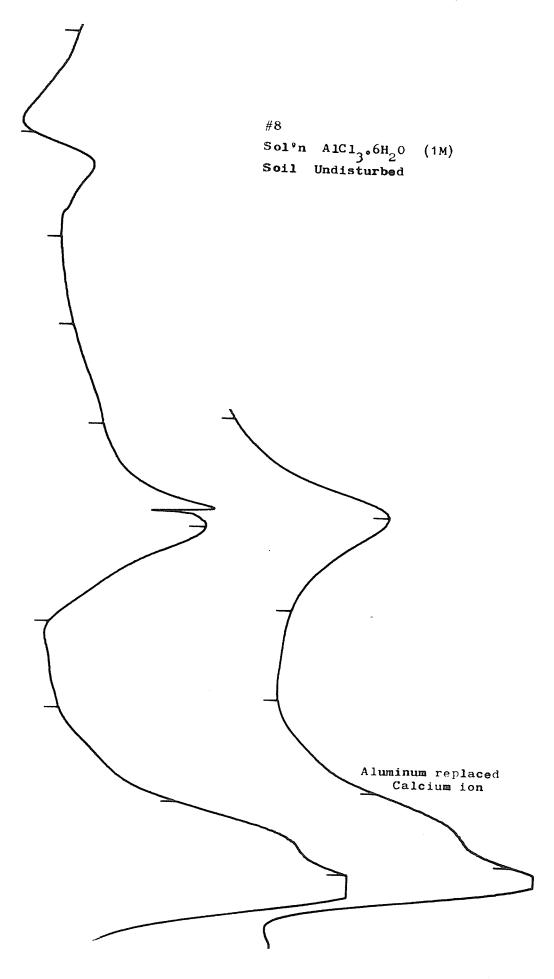
The peaks towards the right represent endothermic reactions where heat is absorbed; the ones to the left are exothermic where heat is given off.

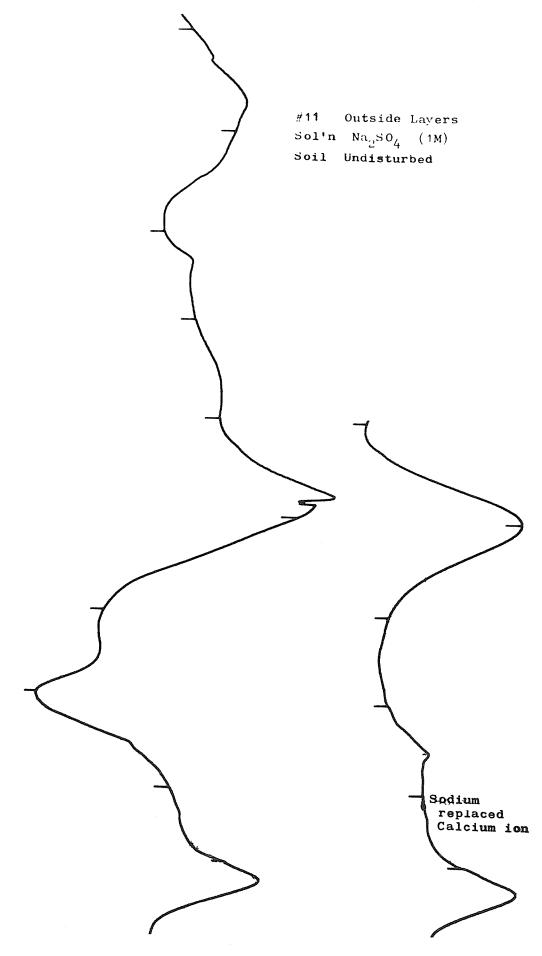


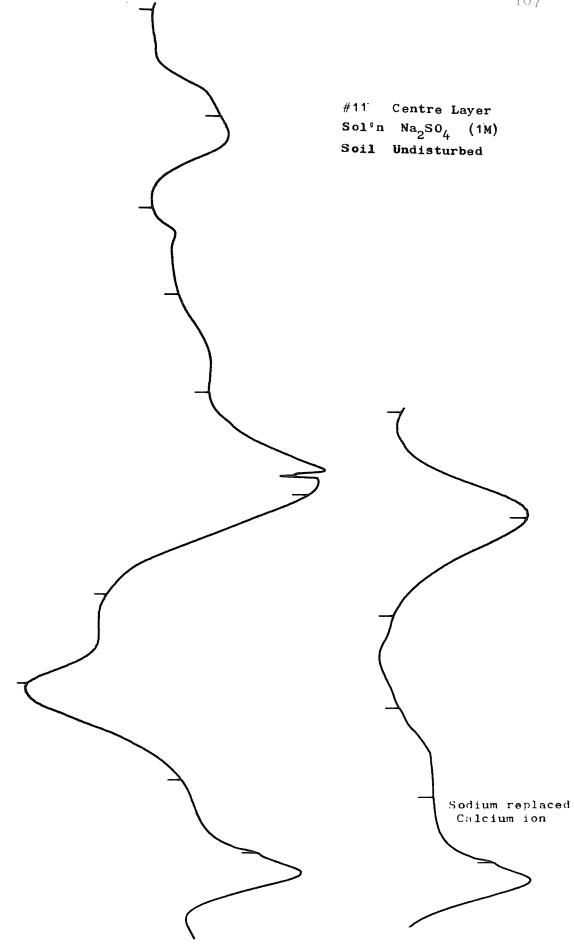


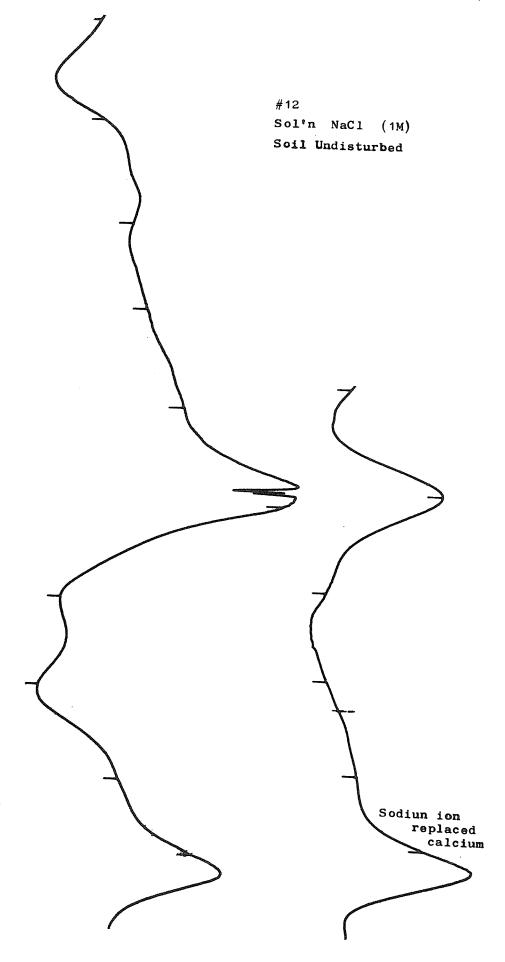


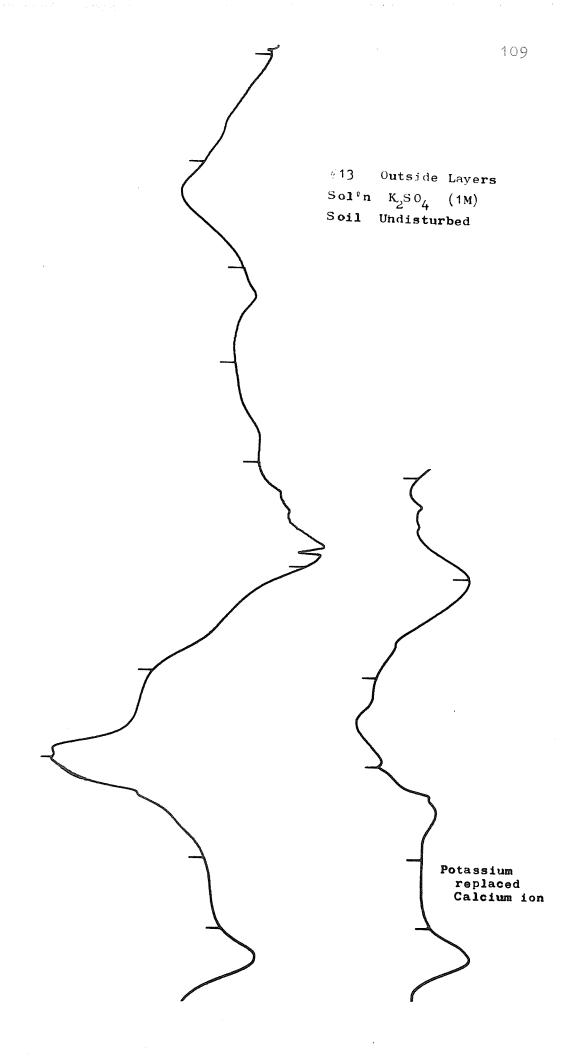


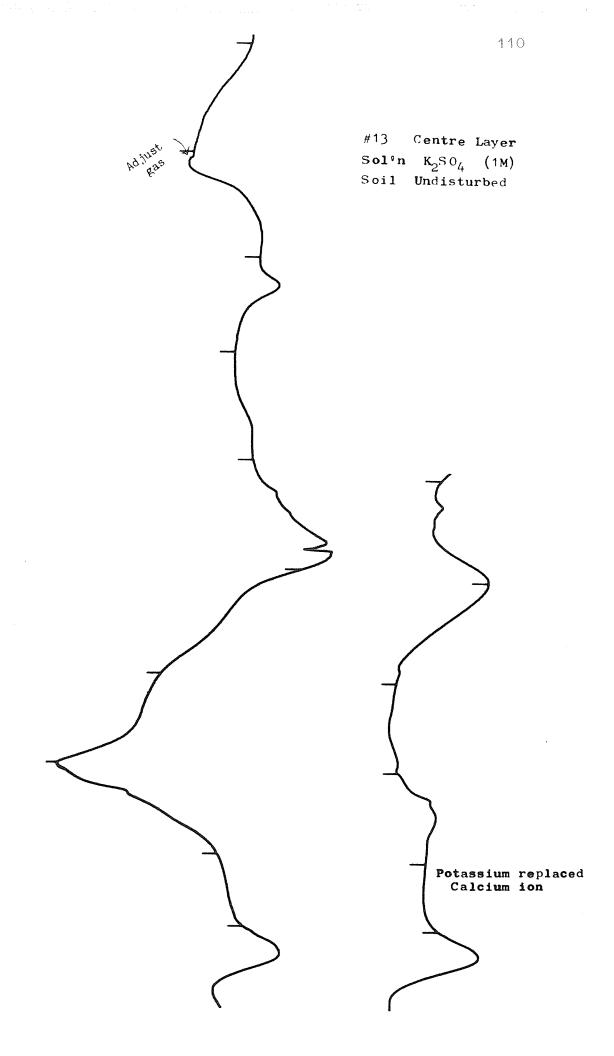


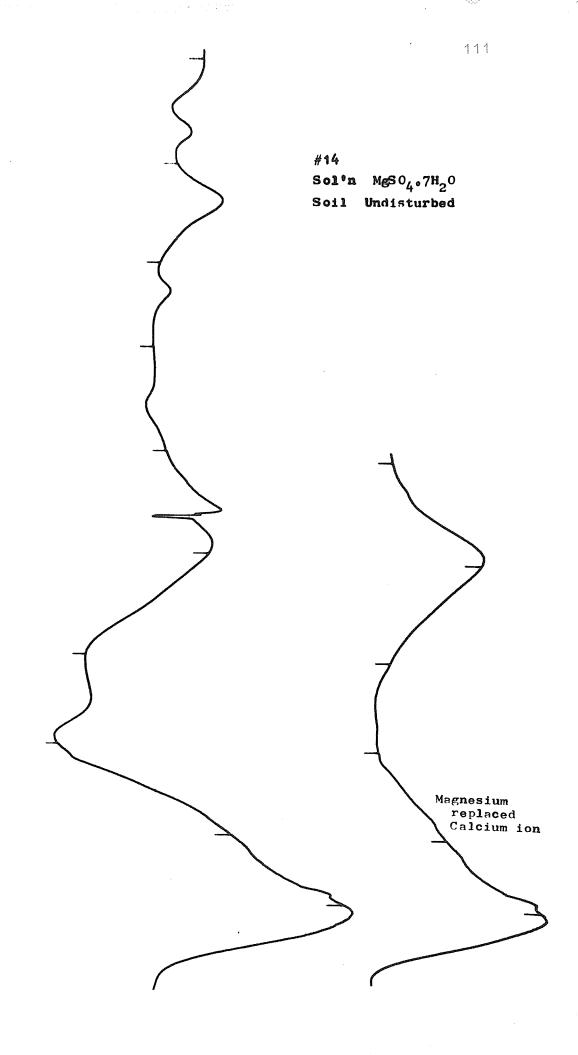


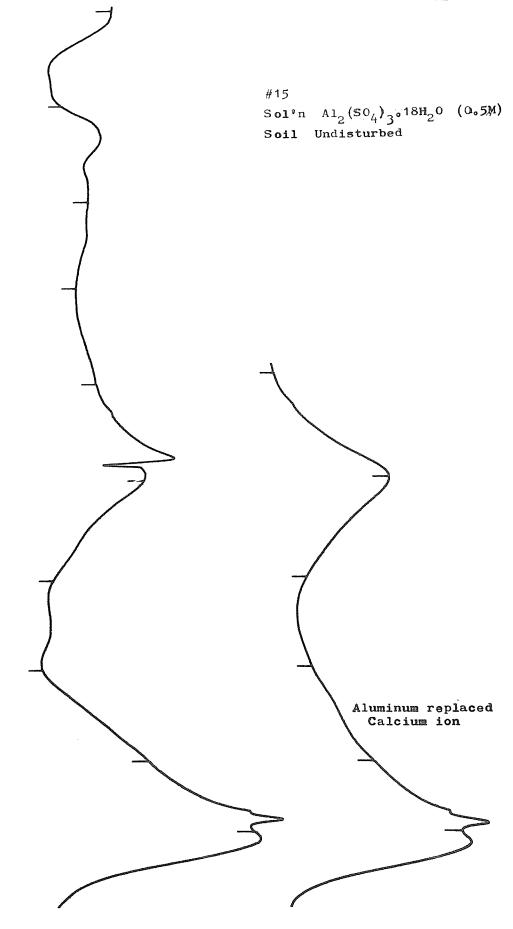


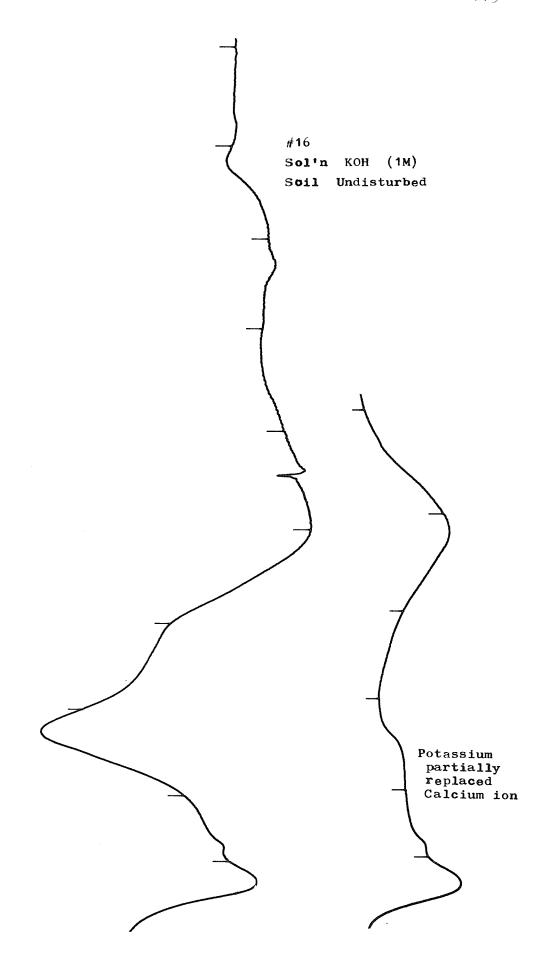


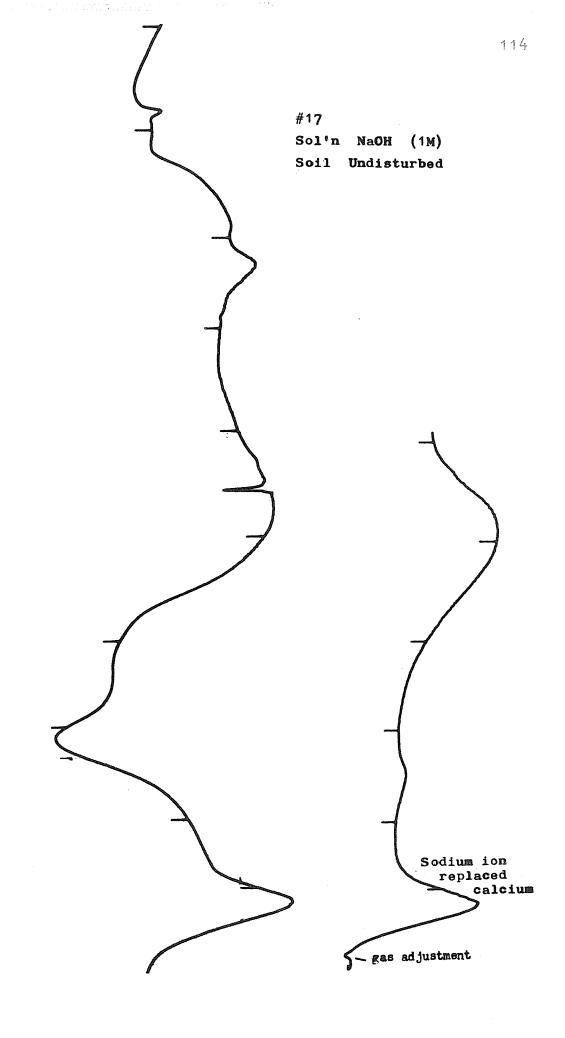


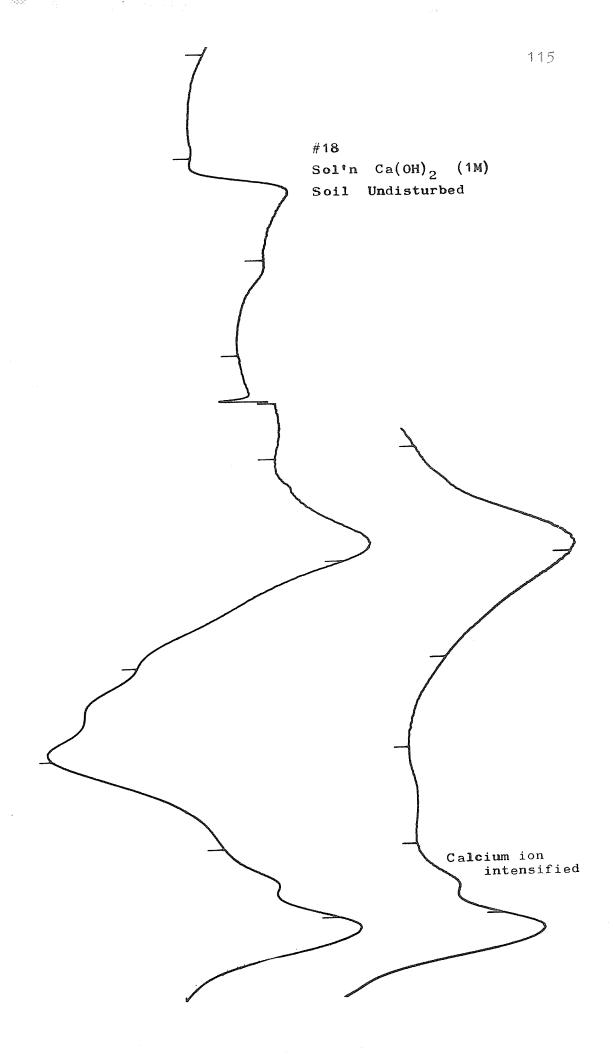


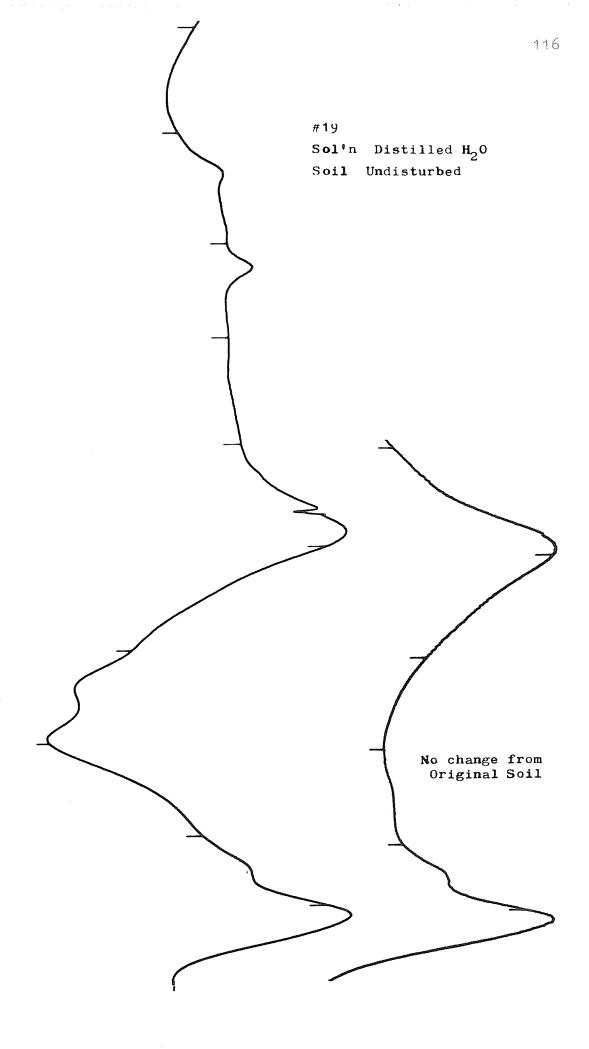


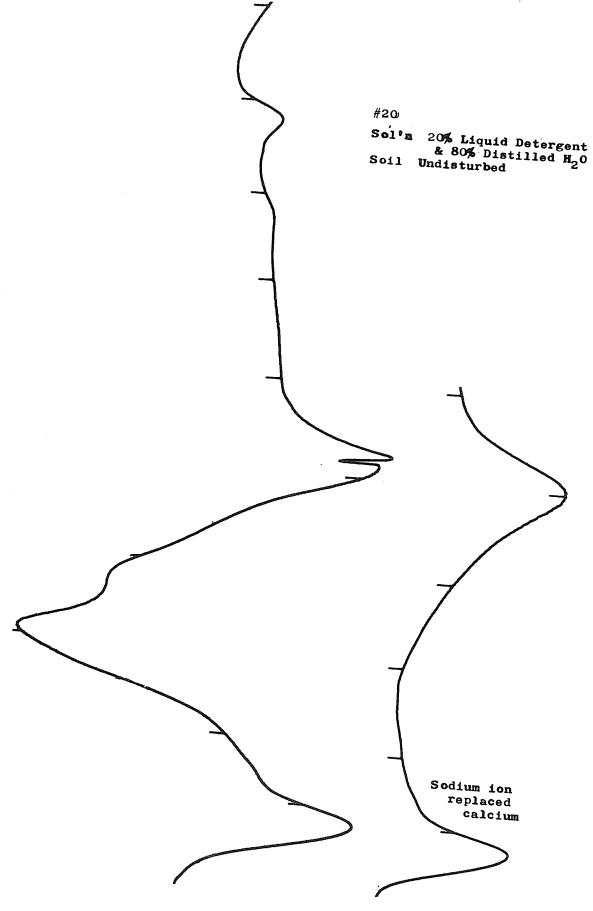


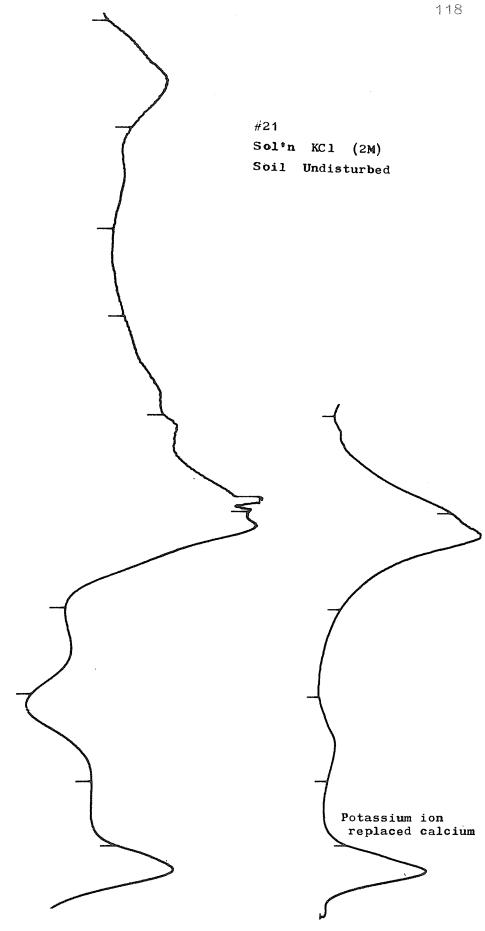


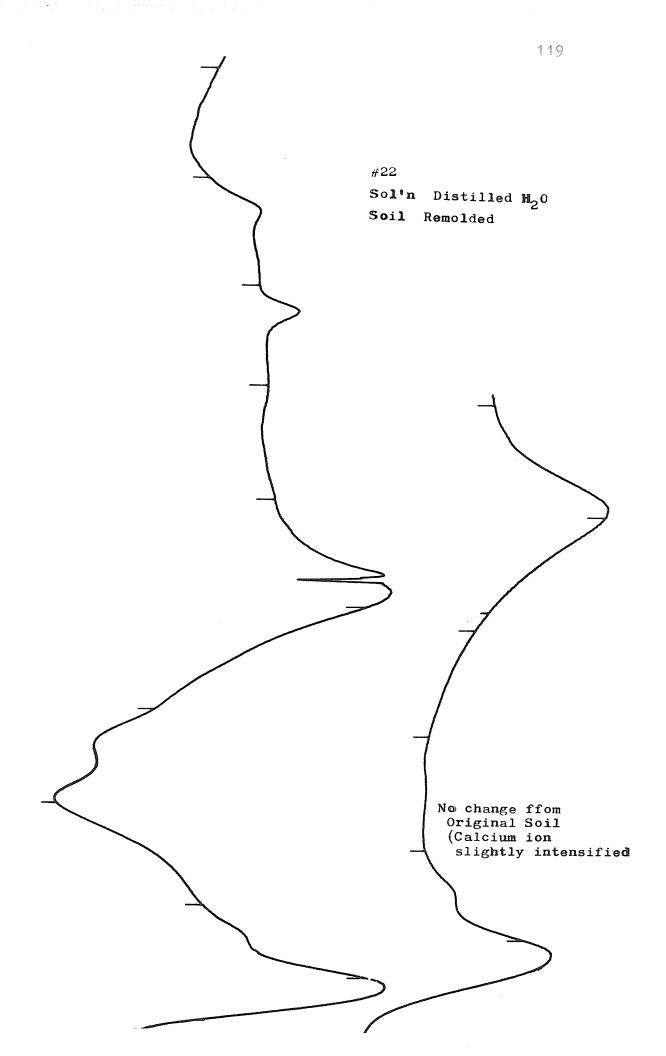


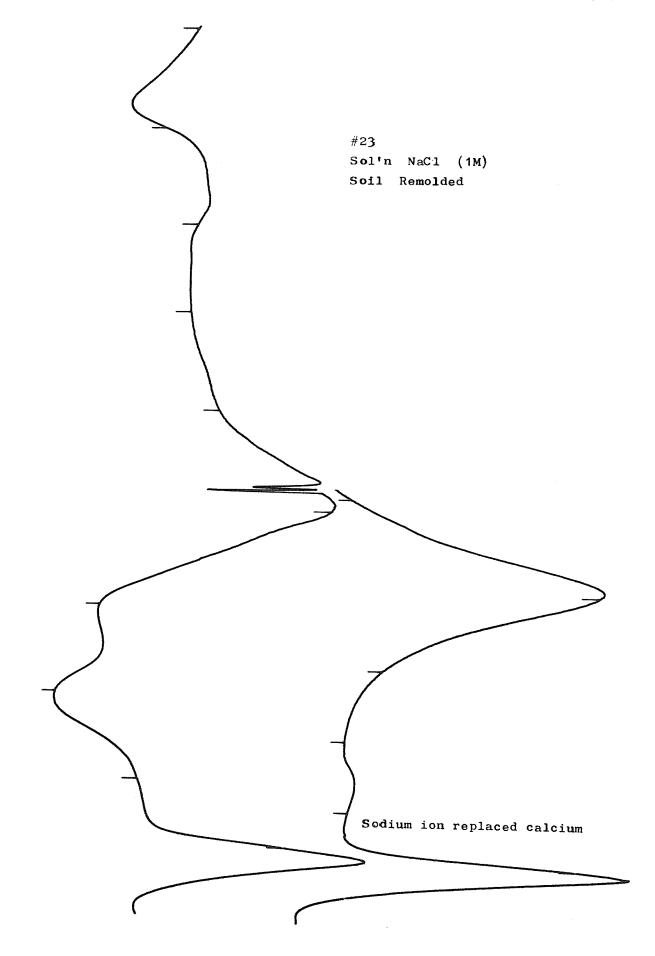


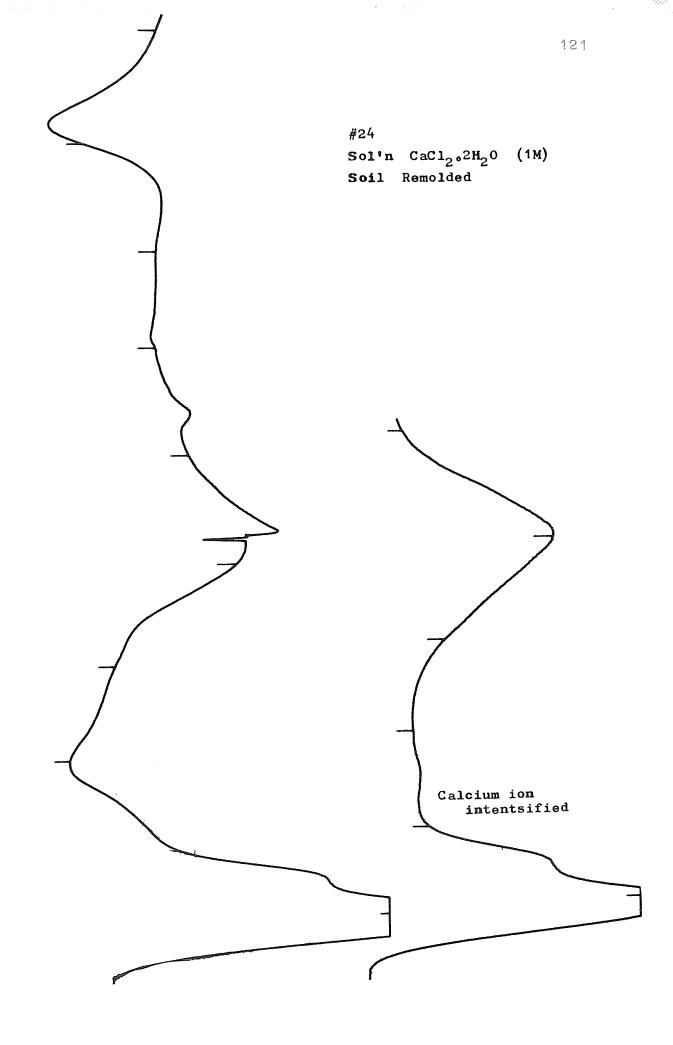


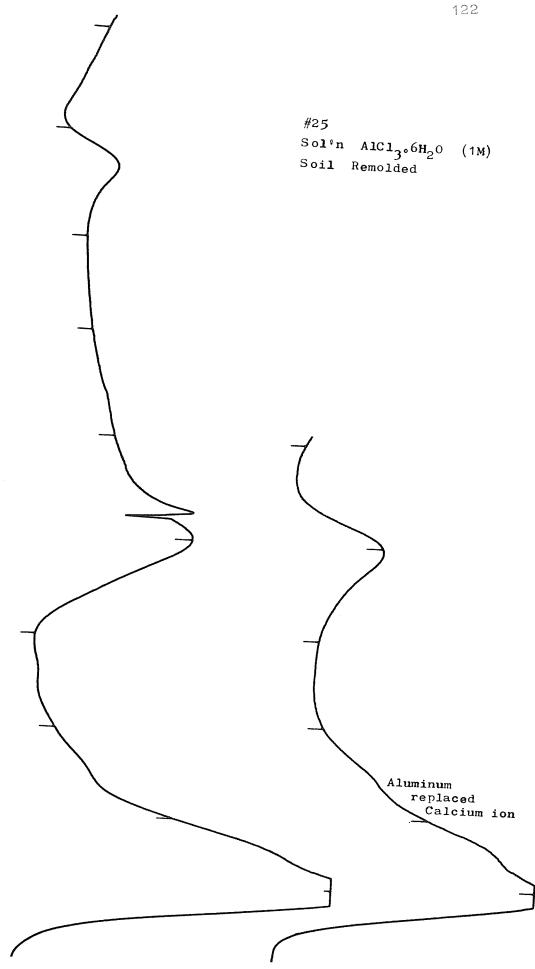


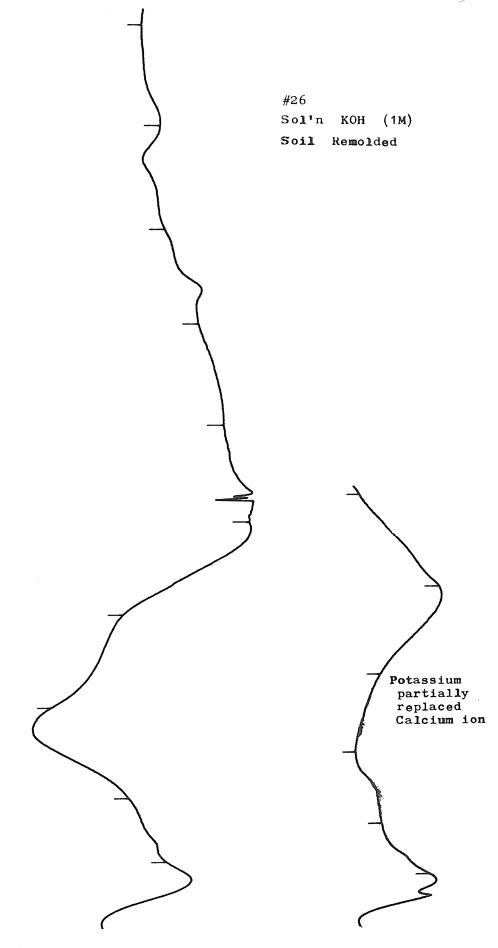


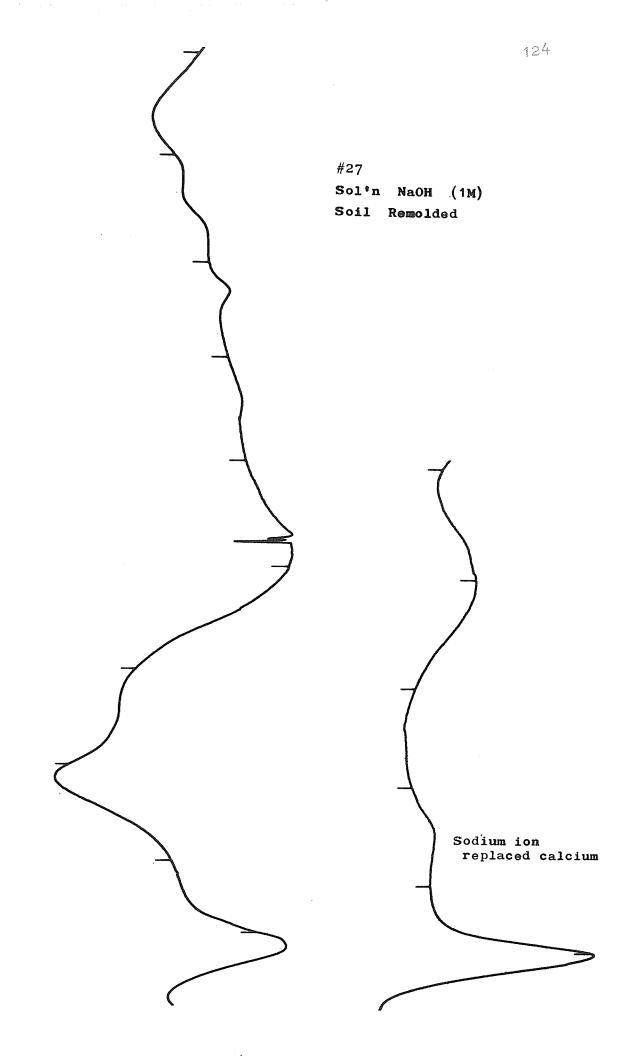


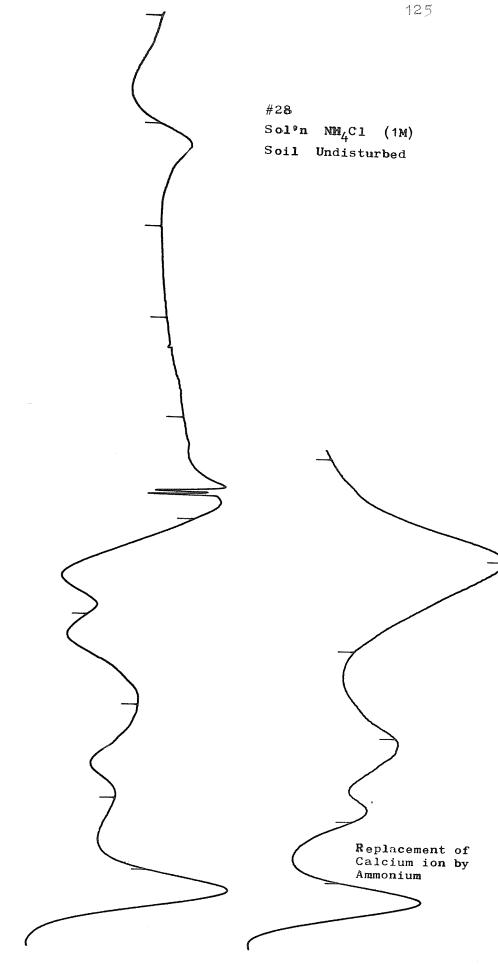


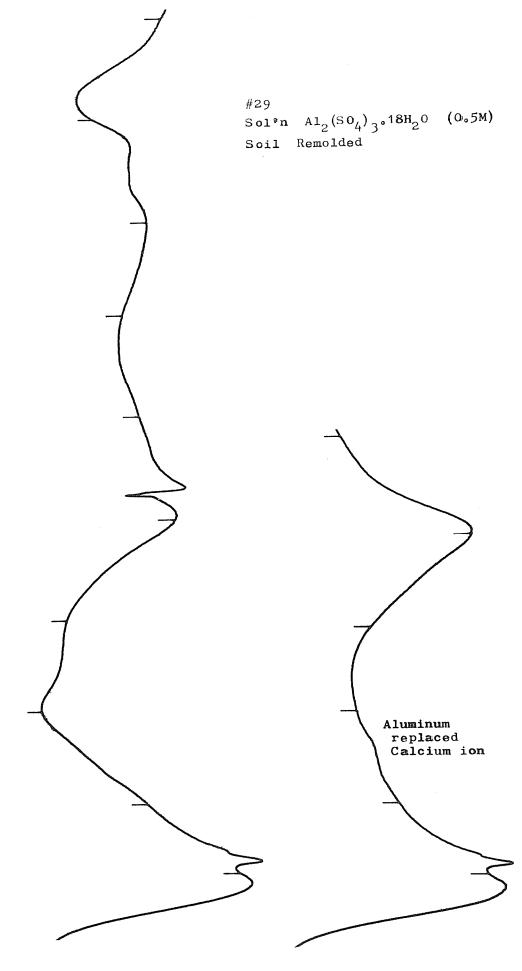


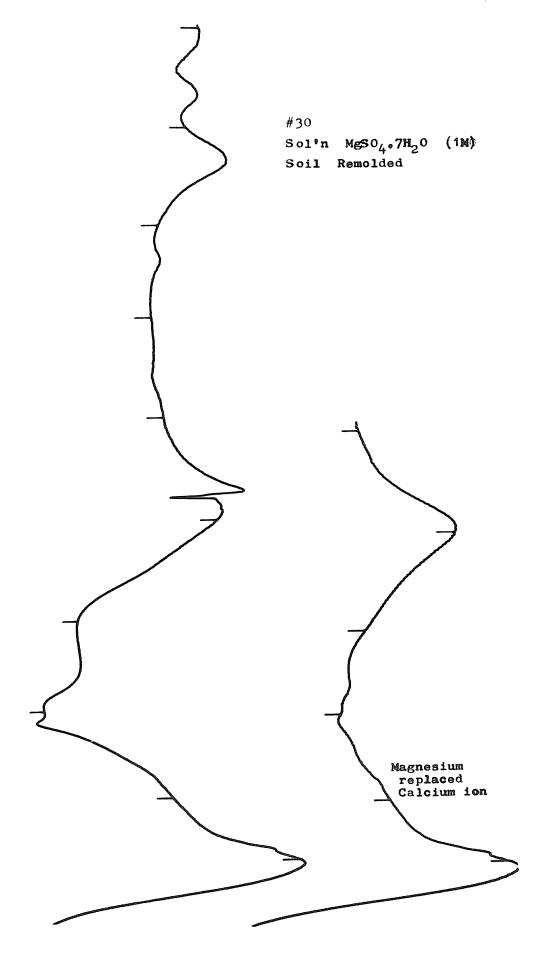


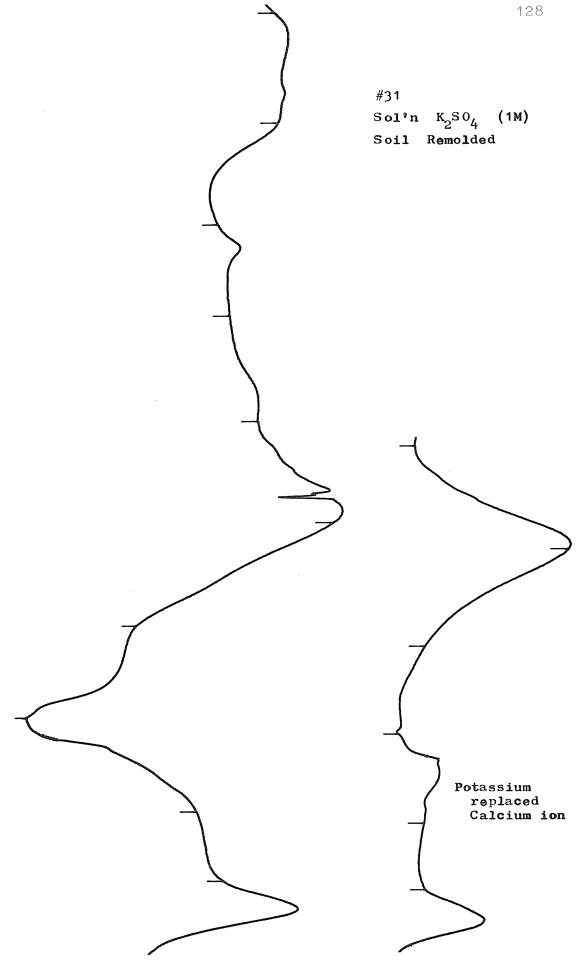


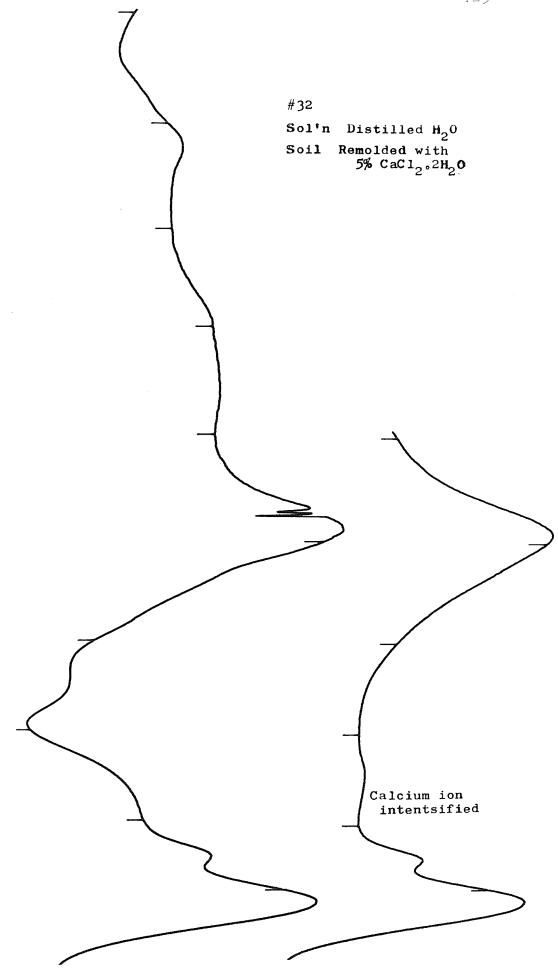


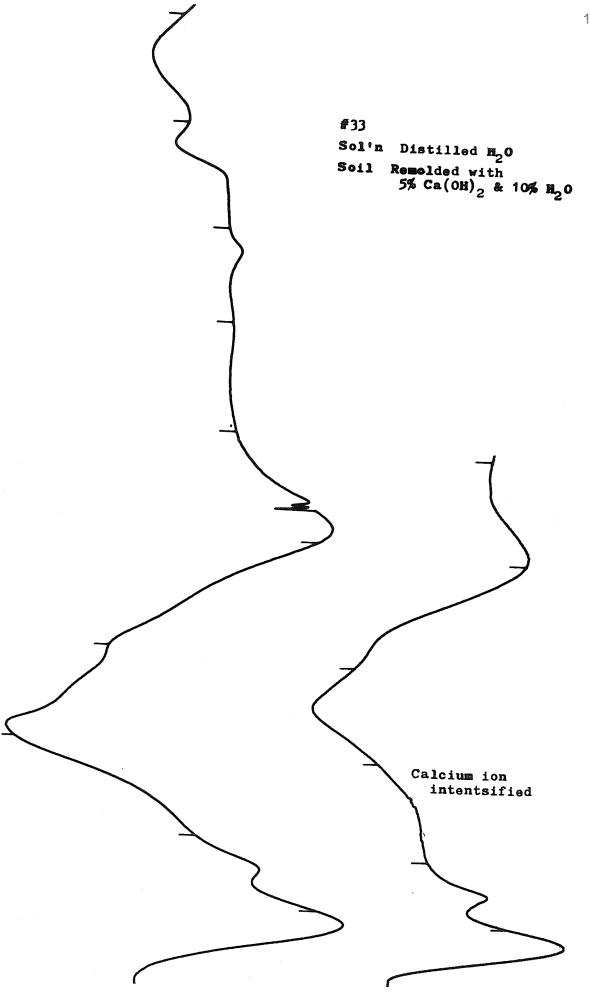








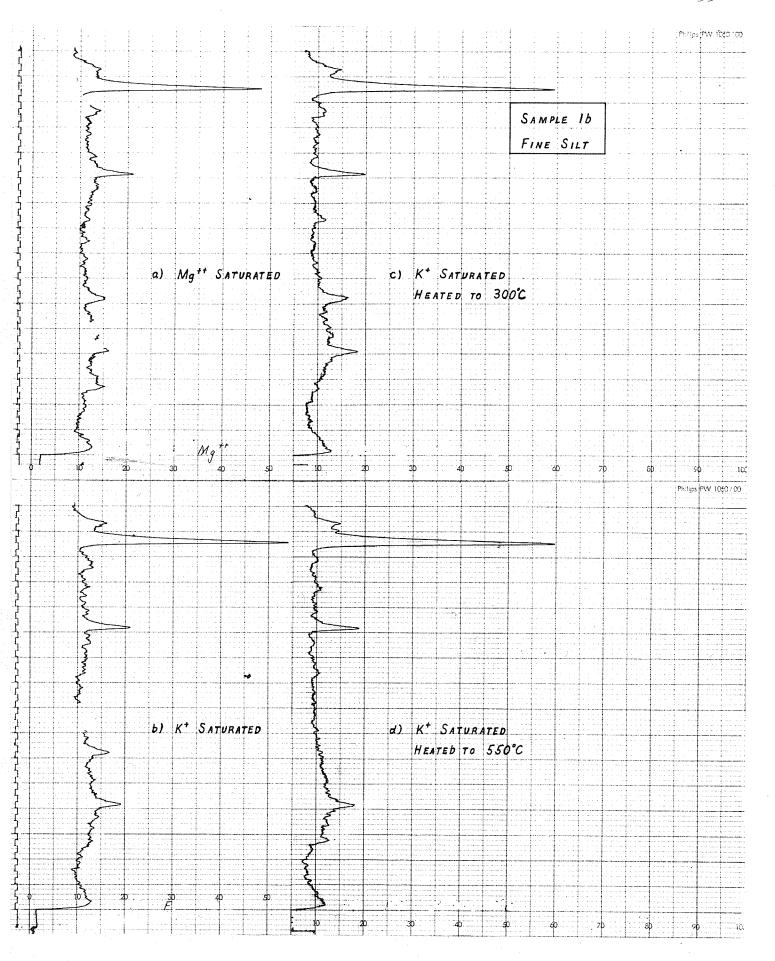


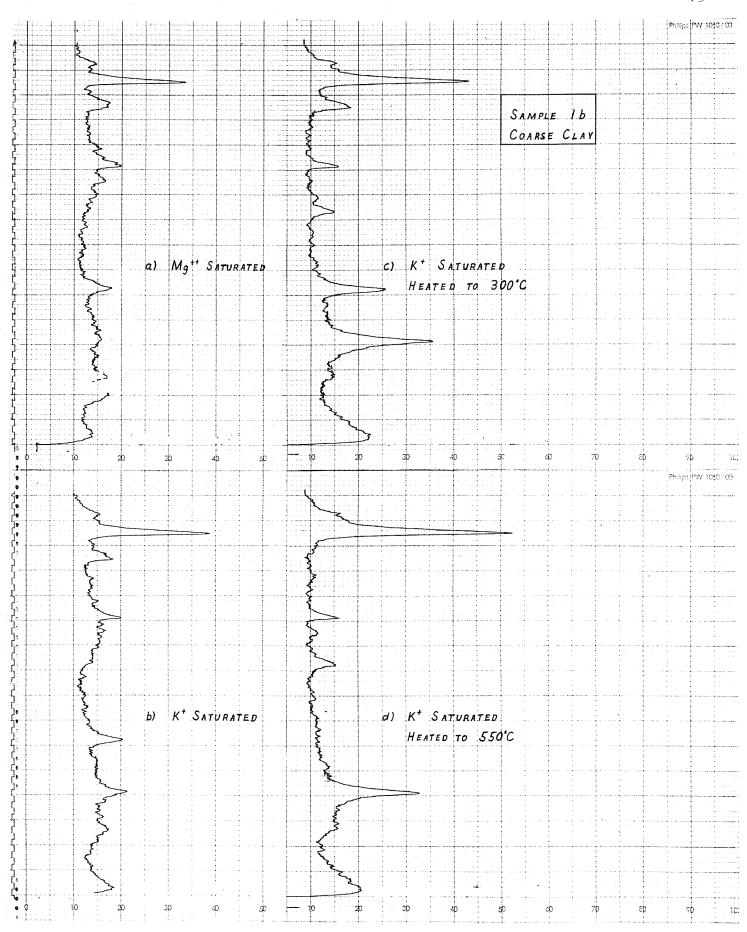


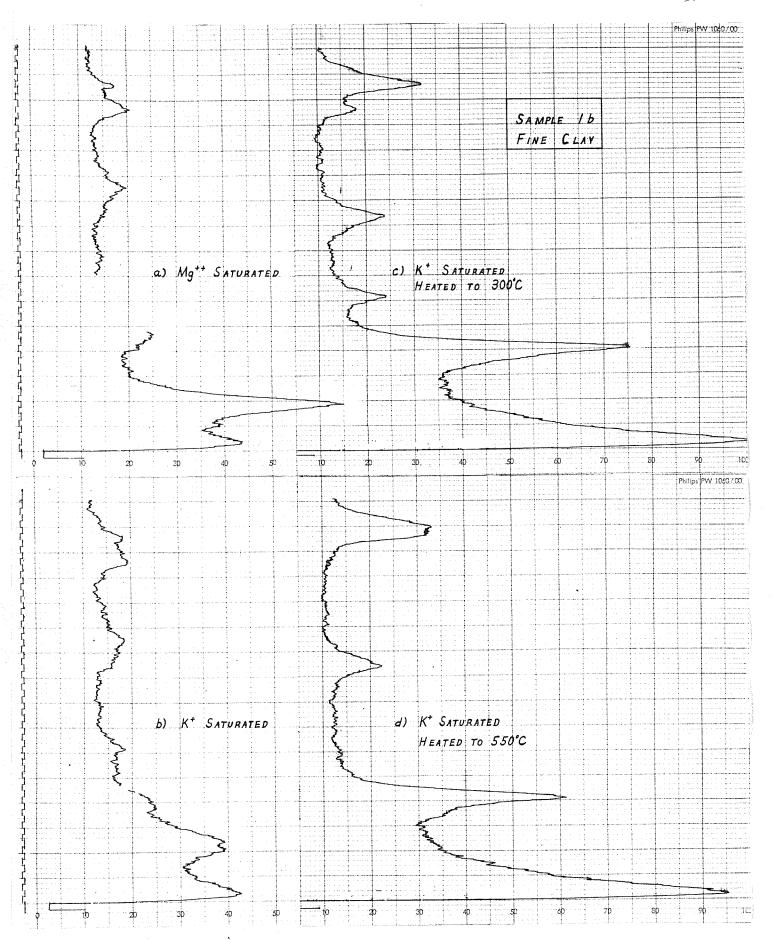
## APPENDIX C GRAPHS FROM X\_RAY DIFFRACTION ANALYSIS

## EXPLANATION OF GRAPHS

The vertical axis of the graphs is twicethe refraction angle of the X-ray (20). The test is run from  $2\theta = 2^{\circ}$  to  $2\theta = 34^{\circ}$ . The peaks at the various angles are correlated to the d-spacings providing an identification for the different minerals. The horizontal axis has little significance, but is used in quantitative analysis.







APPENDIX D

ION EXCHANGE DETERMINATIONS

## TON EXCHANGE DETERMINATIONS

To find the total ion exchange capacity of the soil and the nature of the exchangeable ions a 25 gram sample of soil in air dry condition was used. This sample was leached with a l:l mixture of ammonium acetate and ethyl alcohol. The ammonium ion replaced the exchangeable ions held in the soil.

The decantant from the leaching was used to find the various exchangeable ions. Titration revealed the amount of (1) calcium and (2) calcium plus magnesium. The difference gave the amount of magnesium. A flame comparison test was used to determine the quantities of sodium and potassium.

The ammonium ion was leached from the sample with sodium chloride. Titration of this decantant gave the total ion exchange capacity of the soil which was approximately the sum of the individual ions.

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