SHRINK-SWELL CHARACTERISTICS OF SOIL:

RESPONSE TO VARYING CONCENTRATIONS

OF NaCl AND OH/A1 RATIOS

By

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Submitted to the Faculty of the Graduate College of the Oklahoma State University in partial fulfillment of the requirements for the Degree of DOCTOR OF PHILOSOPHY July, 1972



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ACKNOWLEDGEMENTS

It is the author's wish to express his sincere appreciation to Dr. Lester W. Reed, Professor of Agronomy and the author's major adviser, for his assistance, guidance, encouragement and thoughtful suggestions throughout this study.

To Drs. J. Q. Lynd, F. Gray, R. Matlock and Eddie Basler for serving on the author's advisory committee and for reviewing this manuscript, sincere thanks.

Deep appreciation is extended to the Department of Agronomy, Oklahoma State University, for the financial support received throughout the period of this study. Recognition is also due to Mr. Orion Schmidt, Agronomy Department, who was most unselfish in helping the author in some aspects of the study.

This work was supported by Regional Research Funds obtained through the Oklahoma State University Agricultural Experiment Station, and is a contribution to Regional Project S-60. Influence of Mineralogy of Southern Soils on Certain Chemical and Physical Reactions.

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*Ethylene glycol monoethyl ether

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CHAPTER I

INTRODUCTION

The phenomenon of shrinking and swelling which takes place in most soils as the water-content increases or decreases, has attracted considerable attention over the past two decades. This change in apparent specific volume which accompanies wetting and drying is an important property of soils. It is by this process, primarily, that soils, particularly clay soils, are physically reconditioned. The effects may extend far beyond the range of purely agricultural interest, as for example, the shifting of structural foundations due to the shrinkswell behavior of subsoils. The character of shrinkage as it occurs progressively throughout the moisture range may or may not be immediately applicable. As it further characterizes the properties of the soil, the shrinkage behavior may furnish a basis for differentiating soils, with greater percision. It may also offer a partial explanation for the different behavior of soils comprising differences in texture, mineralogy and sorbed ions.

In general, the great interest which attaches to the subject lies in the fact that the constant fluctuations in moisture to which most arable soils are subjected, constitute a leading factor in the loosening of the structure of the soil and the formation of a good tilth. The non-expanding lattice of kaolin and the expanding type lattice of montmorillonite may offer a better explanation than the colloidal coating theory for the differences in shrinkage characteristics of soils and clays studied by several investigators.

The total surface area of soils is a characteristic property and has been used as a measure of identifying certain silicate minerals. The methods adapted vary, but have been based on the principle that solid materials will adsorb a monomolecular layer of a gas or vaporized substance at controlled pressures.

The following are the major objectives of this study:

(1) To establish a relationship between the swelling of soils and mixed salt solutions, including varying OH/A1 ratios.

(2) To relate experimental swelling values to soil-clay mineralogy.

(3) To correlate observed macroscopic swelling values with internal surface of soil clays.

CHAPTER II

LITERATURE REVIEW

The shrink-swell of clay soils was recognized by Haines (1923) to have practical implications. He defined two stages in the shrinkage process:

(1) Residual shrinkage, that stage at which volume change of the soil is less than the volume of water withdrawn.

(2) Normal shrinkage, a volume change of the soil equal to the water content change.

Stirk (1954), Chang and Warkentin (1968), refer to four stages in shrinkage:

(1) Structural shrinkage

(2) Normal shrinkage

(3) Residual shrinkage

(4) No shrinkage

Upon close observation, it may be seen that these four divisions are sub-divisions of the two previously outlined by Haines (1923).

The process of swelling and shrinkage which accompanies wetting and drying is an important property of some soils. It largely contributes to structural formation in clay soils, and also appears to be associated with the genesis of gilgai formation, Stirk (1954). The cracking which results from shrinkage, makes soils more permeable to water, and also assists in the aeration of the deeper horizons of the profile.

The extent of interlayer swelling of a clay is related to its mineralogical characteristics (Fink and Thomas, 1964). Present knowledge indicates that for any clay to expand, it must possess an interlayer charge resulting from an imbalance of lattice charges. If such an interlayer charge exists, the amount of swelling will be greatly influenced by the nature of the saturating cation, which at high concentrations will reduce the amount of water held by the clay minerals (Falconer and Mattson, 1933). The present knowledge of the effect of interlayer cations on clay swelling has been the result of contributions from several investigators. The initial hydration is a stepwise process, regardless of the saturating cation and exchange sites (Hendricks, Nelson and Alexander 1940). Swelling, or increase of volume upon wetting, results from forces of hydration (Grim 1968). For the first increments of water taken up, the forces are associated with hydration of exchangeable ions and the clay mineral surface. Large volume increases are believed by Bolt (1956) to be due to osmotic forces associated with the exchangeable ions. Marshall (1936) has ascribed certain swelling differences to the location of the charge in montmorillonite. The degree of cation dissociation was greater for a montmorillonite (octahedral charge) than for a beidellite (tetrahedral charge). Harward and Brindley (1966) found that synthetic montmorillonites having octahedral substitutions tend to swell more than synthetic beidellites having tetrahedral substitutions. It may, therefore, be assumed that the location of the site of charge does have an effect. Presumably, isomorphic substitutions in tetrahedral coordination permits less cation dissociation than substitution in

octahedral coordination. This results in fewer osmotically active cations to produce swelling (Low 1968). Foster (1955) also reported an inverse relationship between swelling and the polarizability of the lattice cations. Norrish (1954) reported that montmorillonite in various concentrations of salt solutions exhibited an initial swelling that is crystalline and dependent upon the hydration energy of the interlayer cation. The evidence supports the view that the charge imbalance in the crystal lattice, hence the mineralogical composition, affects shrink-swell profoundly (Fink and Thomas, 1964). Barshad (1950), Brindley and MacEwan (1953), White (1958), found that the ability of a clay to swell, is determined by the degree of charge-imbalance which is due to the surface-charge density. As the surfacecharge density decreases, the ability to swell increases (Brindley and MacEwan 1953). At relatively high surface-charge densities, the attractive energy due to interaction between the negative surfaces and interlamellar cations exceeds the repulsive energy due to the shortrange hydration (Low, 1968). On the basis of the theories of Quirk and Aylmore (1960), and Quirk (1963) one would expect the swelling of montmorillonite to depend on the cation exchange capacity due to the constant planar surface area of montmorillonite. Foster (1955) believed there is no relationship between the swelling of different Na-montmorillonites and their cation exchange capacities. Instead, it has been pointed out that swelling depends more on octahedral substitution, thus it may be dependent upon the mineralogical composition but not as prescribed by the double-layer theory. As further evidence of this, Foster (1953) found that swelling decreased as Fe^{+3} or Mg⁺² replaced $A1^{+3}$ in octahedral positions without otherwise changing the

composition.

Studies on montmorillonite saturated with sodium ions showed that the swelling curves may be predicted from the diffuse double layer theory (Warkentin, Bolt and Miller, 1957). Conversely, the Gouy theory could not account for all the forces involved in a calcium saturated system (Aylmore and Quirk, 1959; Blackmore and Miller, 1961). It has been postulated that a calcium-montmorillonite system consists of packets of particles, or tactoids within which, several [4-9] clay platelets are in parallel array, with interplatelet distance of $9A^{\circ}$. The swelling of sodium-montmorillonite may be predicted from the Gouy theory whereas that of calcium-clay may be predicted only by assuming the tactoid model and diffuse ion layers on the external surfaces of the packets (Shainberg, Bresler and Klausner, 1971).

Haines (1923) investigated the shrinkage and swelling process from the point of view of comparing the progressive volume changes of the soil with the volume of water involved in the change. If the volume of water entering the soil is represented by \triangle w and the corresponding volume--change of the soil is \triangle v, then

In any soil to which an increment of water of Δ w "inches per unit volume" is added or removed (evaporation) the normal volume change is in three dimensional process (Aitchison and Holmes, 1953) and may be represented as: -

$$w = \left[(L + \Delta L)^3 - L^3 \right] / L^2$$

where L is the original unit dimension, and \triangle L the change of each unit dimension.

Since \triangle L/L is small, then

The evidence of three dimensional volume-change in clay soils is supported by the data of Ward (1948). If the volume is one dimensional then $[\Delta w = L^2(L + \Delta L_1) - L^3]/L^2$ where ΔL_1 is the change in vertical dimension only.

In an initially saturated clay-soil subjected to drying, the process of shrinking will commence as normal ($\Delta v / \Delta w = 1$), and change to residual ($\Delta v / \Delta w < 1$) and finally change to ($\Delta v / \Delta w = 0$) at some low water content.

Ferguson and Hosking (1954) have suggested that given the same clay mineralogy, there is a general parallelism between particle size distribution and shrinkage rate. During the "constant-rate period" of drying, the rate of water loss depends only slightly on the clay content and the mineralogy of the clay. The differences in the amount and type of clay minerals begin to affect water loss only in the "decliningrate period". It is in this period that air-filled as well as waterfilled capillaries, and the size of these capillaries, is affected considerably by the clay content and its mineralogy. However, shrinkage was found to be highest in the mixed-layer mineral clays (Ferguson, 1954), although those with nonplastic ingredients had reduced shrinkage.

The swelling observed when an electrolyte is introduced into a soil, may be controlled or influenced by the type and amount of clay present, the exchangeable cations on the clay, as well as the free salts which may be present in the soil. The concentration and composition of the electrolyte as well as the presence of other materials in association with the clay should also be considered. These may include the oxides of iron and aluminum in addition to organic matter. In this respect, Rowell (1963) debated whether or not there are two ranges of electrolyte concentrations, the higher range causing swelling only, and the lower range causing both swelling and dispersion.

Anions and Swelling

The retentive capacity of sulfate in the subsoil has been found to be generally higher than in the surface layers (Buckman and Brady, 1969). This is probably due to the high sulfate retentive capacity of certain compounds which tend to accumulate in the lower soil horizons. Buckman and Brady (1969), believe these include the hydrous oxides of iron and aluminum as well as the silicate clays. The mechanism of absorbing sulfate is thought to involve OH⁻ groups in the hydrous oxides and silicate clays where hydroxyl groups held by aluminum ions are replaced by sulfate ions. A generalized equation illustrates how this may occur:



The addition of OH⁻ ions (increasing pH) will tend to drive the reaction above to the left, releasing the sorbed sulfate.

There is little experimental evidence for stoichiometric anion exchange in soils, although anion exchange probably does take place to some degree. In soils, the material most reactive with anions are amorphous aluminum and iron in the hydrous oxide or hydroxide form (Rich, 1968). As discussed by Jackson (1960, 1963), the terminal edges consist of formerly shared OH⁻ groups, now OH₂ groups due to protonation, as for example, the following:

-A1-OH-A1-
$$\longrightarrow$$
 -A1-OH²
-A1-OH ^{$-\frac{1}{2}$} + H⁺ \longrightarrow -A1-OH₂ ^{$+\frac{1}{2}$}

Small polymers or colloidal particles containing these positively charged edge groups can react directly with anions (Rich, 1968). Some evidence for direct OH^- , SO_4^{-2} exchange has been presented by Mehlich (1964), and Chao, Harward and Fang (1965). As discussed, the positively charged groups may be attached to negatively charged surfaces. These surfaces may include external surfaces of layer silicate (de Villiers and Jackson, 1967), as well as the internal surfaces of expansible layer silicates. In a proposed behavior in soils, Chang and Thomas (1963) have suggested that basically, the mechanism combines the theory that OH^- ions are replaced by other anions in the light of an increased hydrolysis of Al^{+3} in salt solutions.

Aluminum Compounds and Swelling

Sawhney (1958) has pointed out that aluminum may exist in different forms when naturally fixed or under laboratory conditions. In soil clays, aluminum interlayers resist the expansion of montmorillonite spacings beyond $14A^{\circ}$, whereas greater expansion has been observed under experimental conditions. Tamura (1957), has suggested that the presence of $Al(OH)_2^{+}$ ions and polymerized basic alumina in the interlayer spaces may be responsible for the non-collapse of the soil clay minerals which have undergone varying degrees of chloritization. At very low pH

values, $A1^{+3}$ or $A1 (OH)^{+2}$ ions may be fixed on either side of the Montmorillonite layer. The formation of aluminum polymers, e.g. $[A1_6(OH)_{12}(OH_2)_{12}]^{6+}$ may preferably occur (Jackson, 1963) giving rise to the formation of neutral crystalline "gibbsite-like" units on the negatively charged clay surfaces. The occurrence of such reactions may result in the reduction of the overall net negative charge of the layer. The consequent increase in negative charge results in an increased lattice expansion.

Laboratory synthesis of aluminum interlayers in different montmorillonites showed that the stability and composition of interlayers are controlled by both the OH/A1 ratio and the mineral structure (Sawhney, 1963). Montmorillonites did not fix aluminum ions, but fixed appreciable amounts of hydroxy-aluminum ions (Kidder and Reed, 1969). The forces responsible for retention of water and for swelling in clay soils are considered to be particle repulsive due to the interaction of diffuse layers of cations (Bolt and Miller, 1955; Childs, 1957). These forces may be due to bound water held by the attraction and by the orientating influence of the clay surface (Low, 1958). Experimental results have been interpreted both ways, and both forces probably are involved (Warkentin, 1962). The measured swelling pressure in a soil system is greater for parallel orientation than for random orientation (Warkentin, 1962). It may therefore be concluded that the measured swelling pressure on a soil or clay system is also dependent upon the drying history of the sample. The opposite effect has been reported by Seed and Chan (1959), Warkentin and Bozozuk (1961) for low swelling clays.

From an energy perspective in tensile strength determination,

Dowdy and Larson (1971) show that more energy is required to rupture monovalent specimen than divalent counterparts, while Al-clays exhibit intermediate energy demands. The data shows that saturating cations played a dominant role in the strength-energy characteristics of mono and divalent cation saturated montmorillonite clay. For the first increments of water taken up, the forces are associated with hydration of exchangeable ions as well as the surfaces of the clay minerals. Large volume increases are primarily due to osmotic forces associated with the exchangeable ions (Bolt, 1956). As a result of their investigations, Warkentin and Bozozuk (1961) have shown that there is a direct relationship between increased swelling and increased surface area of clay. Similarly, swelling increases as the salt concentration in the pore water decreases and is further increased if accompanied by increasing valence of the exchangeable cation. Horizontal and vertical shrinkage for samples of soils have been measured by Sen and Wooltorton (1942). Swelling and shrinkage of naturally oriented soils and that of laboratory samples are given for several Australian soils by Aitchison and Holmes (1953). Results by Warkentin and Bozozuk (1961) show that the shrinkage limit decreased from 27% for the undisturbed clay to 20% for a remolded sample. This indicates a probable breakdown of the edge-to-face particle arrangement over small distances, resulting in closer packing of the particles. Oven dry densities determined on representative samples corroborated this closer packing.

Specific Surface Area

Clay mineral complexes with polyhydric alcohols have been shown to be of great value in determining the 'c'-axis spacing in the mont-

morillonite group (Marshall, 1936). Ethlene glycol and glycerol have been shown to be held in two layers on the basal plane of montmorillonites (MacEwan, 1948), resulting in a 'c'-spacing of 17.1A° to 17.74° respectively. Vermiculite, on the other hand, adsorbs only one layer of these polar molecules giving a $14A^{\circ}$ spacing. The fact that ethylene glycol will form a monolayer on clay surfaces has been utilized in a method for the estimation of specific surface area (Dyal and Hendricks, 1950). The measurement of this property provides an estimate of the proportion of expandable layer silicates in soils. This method (Dyal and Hendricks, 1950), has been modified and adapted by Bower and Gschwend (1952). A further modification by Martin (1955), includes a source of free ethylene glycol in an evacuated dessicator to control the vapor pressure of ethylene glycol at the surfaces of the silicate minerals. An equilibrium method incorporating this improved method, has been developed by Bower and Goertzen (1959), and has been widely adopted. Sor and Kemper (1959) introduced a similar but somewhat more complex method. Other methods include the application of the B.E.T. theory (1938), using ethylene dibromide as well as the adsorption of organic molecules from solutions (Bower, 1963; Bradley, 1945; Hoffman and Brindley, 1961; McKenzie, 1948; Tettenhorst, Beck and Bruton, 1962; and Lawrie, 1961).

More recently, Carter, Heilman and Gonzalez (1965) introduced a more rapid method for the determination of surface area in layer silicates, in which the adsorbed phase is ethylene glycol monoethyl ether (E.G.M.E.). The results obtained from such determinations have been in full agreement with those obtained with ethylene glycol as the adsorbed phase.

Subsequently, Heilman, Carter and Gonzalez (1965), adapted the E.G.M.E. method to soils, and obtained similar specific surface area as was determined with ethylene glycol.

Comparison studies of several surface area measurements carried out by Puri and Murari (1963) have produced values which are in good agreement in all categories of soils and clays:

- (1) glycol retention
- (2) mechanical analysis
- (3) water adsorption isotherms using Harvey's calculations (1943)

Charge Variations

X-ray diffraction, petrographic microscope counts, infrared absorption and alpha-beta thermal inversion of quartz have been used by Kiely and Jackson (1965) to determine quantitatively, the occurrence of Quartz, Mica and Feldspar minerals. Identifying the silicate minerals present in a given soil may be a very logical step towards understanding the soil's physical and chemical behavior. Differences in interlayer charge may cause the same type of silicate mineral to behave differently under certain conditions. Rich (1960), showed that increasing amounts of interlayer aluminum caused vermiculite to collapse at higher temperatures and to greater (001) spacings.

Tamura (1958) and Kidder (1969*) showed that a vermiculite-like mineral could be made to expand by removing aluminum from interlayer positions. However, with clays from the sassafras soil, Douglas (1965), showed that interlayered extracted vermiculite would not expand when

^{*}Gerald Kidder Ph.D. Thesis 1969, Oklahoma State University, Agronomy Dept.

treated with ethylene glycol. The variations between these minerals may be explained by layer charge and interlayer aluminum. The minerals with the largest interlayered-aluminum possess a layer charge great enough to prevent expansion after the interlayer-aluminum has been removed (Douglas, 1965).

Aluminum interlayering may be detected by sodium citrate extraction Frink (1965). The conditions favoring the formation of Aluminum minerals or compounds seem to promote accumulation of other aluminumminerals or compounds which are similarly citrate-soluble (Frink, 1965).

Numerous investigators have been successful in synthesizing intergrade minerals in the laboratory, Sawhney (1960), Rich (1960), Slaughter and Milne (1960); Shen and Rich (1962). The varying stability of the 14A^o spacing in soil clays is an indication of the different degrees of chloritization (Sawhney 1960).

Wurman (1960) noted that the extent of interstratification in the clays of certain Wisconsin soils increases as the surface is approached and as the diffraction peaks become broader and less sharp. This could have been the result of smaller particle size as well as a loss of potassium.

The clay mineral montmorillonite has attracted wide interest because of its swelling properties in water. Hendricks, Nelson and Alexander (1940) and Mooney, Kenan and Wood (1952) have noted that swelling occurred along the c-axis of the unit crystal. Norrish and Quirk (1954) and Norrish (1954) studied these changes in basal spacing using homoionic montmorillonite immersed in electrolyte solutions over a range of water activities. Norrish and Quirk (1954) made a distinction between the initial stepwise swelling (crystalline) and the macroscopic swelling. This occurs at higher water activities, demonstrated by a type of swelling which can be controlled by a choice of salt concentrations. The crystalline swelling was further studied for the alkali and alkali-earth saturated clays and was related to size and hydration energy of the cation.

The low-angle diffraction studies of Norrish (1954), Foster, Savins and Waite (1955) have demonstrated that Na-montmorillonite swells to form a gel. It does so by water entering between the individual silicate sheets. These authors have measured the silicate layer separations as a function of H_2^0 content and salt concentration. Electrolytes restrict the swelling in such a way that the interlayer separation is proportional to $C^{-\frac{1}{2}}$ (for $C \leq 0.3N$), C being the concentration of the solution. At concentrations greater than 0.3N, the basal spacing is small resulting in reduced swelling.

There has been some evidence from measurements of swelling pressure (Bolt, 1956; Warkentin and Schofield, 1960; Aylmore and Quirk, 1962), to indicate that where the clay behaves as a gel (interlayer separations greater than $30A^{\circ}$), there are repulsive forces between opposing silicate sheets. Such repulsion is due to the development of electrical diffuse Gouy layers. Norrish (1954), compared this repulsive force with the attraction to be expected from Van der Waal's forces. The agreement was poor, the repulsive force being many times greater than the attractive force.

CHAPTER III

EXPERIMENTAL PROCEDURES AND MATERIALS

Soils Studied

Three sites selected in different parts of the State of Oklahoma were designated as suitable for soil sample collection. These soils differed sufficiently in morphological characteristics that their mineralogical compositions would provide great contrasts. <u>Dwight Series</u>: Typic Natrustalls, fine, montmorillonitic, mesic. Samples were taken approximately 100 feet south and 2035 feet west of the N.E. ½ corner of section 31, T11N; R18E, McIntosh County. The samples collected as blocks, were from the following depths; 0-7.6; 7.6-16.5; 16.5-22.9; 22.9-35.6; 35.6-49.5; 49.5-63.5; 63.5-73.7 cm,

The series consists of deep, nearly level soils. These soils formed under grass in material weathered from shale, were under small grain at the time of sample collection.

Brewer Silty Clay Loam: 0-1 per cent slopes. Pachic Argiustalls, fine, mixed, thermic. Soil samples were collected on the Fort Reno Livestock Research Station (Agronomic Research Area), El Reno. Samples were taken approximately 200 feet south, 50 feet E. of the Agronomic Field Headquarters (in a turf grass plot) 100 feet N. of the Experiment Station water well. This was in the S. W. ½ section 26; T13N; R8W. used for grass seed nursery.

These soils were developed in Pliestocene Mantles bordering the flood-plain of the North Canadian river. On the extensive low terraces bordering the floodplain are soils such as Brewer. Samples were collected from the following depths; 20,3-30.5; 30.5-40.6; 40.6-58.4; 58.4-89.0; 91.4-121.9; 121.9-152.4 cm.

San Saba: Udic Pellusterts, fine, montmorillonitic, thermic. Samples were taken approximately 1056 feet east and 980 feet north of the S. W. corner of the S. W. $\frac{1}{4}$ of section 7, T6S; R5E, Marshall County.

The San Saba series consists of soils that are deep, nearly black, and fine textured. In most places the surface layer is very dark, gray to black clay. It is very firm when moist and very hard when dry. In about 70 per cent of the acreage, the surface layer is calcareous. In some places where cracks occur, the surface layer is 3 to 4 feet deep, but in other places where the underlying material has been pushed up, it is only about 8 inches thick.

Preparation for X-ray Studies

The whole soil was air-dried, passed through a 1 m.m. sieve and stored until used. Soil preparation for x-ray diffraction was carried out by the method of Hope and Kittrick (1963).

Pilot experiments were run with a few samples to study the effect of the presence of organic matter and free iron oxides on the x-ray diffraction properties of the crystalline clay fraction. It was observed that the removal of organic matter and free iron oxides reduced the background scatter and resulted in better orientation of the clay platelets to give enhanced diffraction intensities, Iron oxides and organic matter were therefore removed from all samples as a routine procedure. X-ray diffraction patterns were obtained of the clay samples oriented by deposition on porous ceramic slides and removal of the excess moisture by vacuum. A General Electric X-ray diffractometer with a Cu target was used for analysis of all samples. Diffraction patterns were obtained on the samples after the following consecutive treatments:

- (a) Mg-saturated and air-dried.
- (b) Ethylene glycol solvation.
- (c) K-saturated and air-dried.
- (d) Heating at 200° for 4 hours.
- (e) Heating at 500° for 4 hours.

Preparation for Swelling Studies

Selected natural aggregates of soils were trimmed from large blocks, retaining the vertical and horizontal orientation which existed naturally. These blocks were brought to equilibrium in an evacuated dessicator over saturated NaCl solution. Volume changes were measured in three directions. The undisturbed equilibrated samples were trimmed to form cylindrical-like blocks so that two, or multiples of two, 1 cm³ blocks were obtained from each cylinder. The dimensions of the cubes were measured with a pair of precision calipers.

To determine swelling, cube number 1 is placed on the pedestal, beneath which is a strip of filter paper leading down into a small beaker. For convenience, this first orientation of the cube is such that lateral swellings are obtained on both axes. When saturation has been attained, cube number 1 is removed and placed in the dessicator for shrinkage. Cube number 2 is then placed on the pedestal in the same manner as cube Number 1, the device is zeroed, the solution is added to the beaker by pipette, the chamber lid is put in place, then the recorders turned on. Cube number 2 is so placed that the vertical swelling is obtained along with one of the previously obtained lateral measurements.

Description of the Apparatus

For saturation a pedestal was made to support the cube above the solution level. A filter paper (Whatman #3) wick was placed between the sample and the platform of the pedestal down into a small beaker containing the solution.

An expansion chamber made of quarter-inch plexiglass was constructed to encase the pedestal which arose from the center of the chamber. A removable lid allowed access to the pedestal and settingup of samples. The purpose of the enclosed chamber was to reduce moisture evaporation, exclude possible air currents and reduce any significant temperature variations. The complete shrink-swell investigations were conducted in a room of 22°C constant temperature. Holes in the sides of the chamber allowed the transducer rods (Hewlett-Packard model 7DCDT-500) to enter for sample contact; Figure 2. A brass plate was screwed on to the threaded transducer rod, to give larger surface contact between rod and sample. The transducer was supported on a block rising from the base erected three inches in, from chamber walls. For a complete illustration, see Figure 2.

Measuring

The change in cube size due to inhibition of the saturating solution created an increase in the voltage passing through the transducers. The voltage was amplified in a specially designed amplification system. There were two series of resistors and the swelling pressures were recorded on Rustrak recorders; Figure 2 and Appendix Figure 27.

Adjustment of the resistors was needed from time to time depending on the amount of swelling obtained in different samples. The greater the degree of swelling the greater the resistance employed. This was important since the recordings had to be confined to a chart of a range 0-100 m.v. calibration: A very satisfactory, accurate and repeatable calibration method was devised using a #24 screw. A hole, slightly smaller than the screw was tapped into a 3/4" plexiglass upright figure. Following this, the screw was slowly turned into this hole until it fit "snugly" having no play. Directly opposite the tip of the screw on another plexiglass upright, a large enough hole was made to accommodate the transducer. The probe of the transducer and the screw were so adjusted until the needle on the Rustrak chart read zero. By gradually applying varying pressures through several turns of the screw or fractions thereof, the deflection of the Rustrak's needle was varied through 100 m.v. from zero.

Since each turn of the screw is the equivalent of 1/24 inch = 1.058 m.m. any degree of swelling obtained, can be accurately inter-polated.

Specific Chemical and Analytical Analyses

Interlayer Aluminum Compounds

Interlayer material was extracted with 100 ml portions of 1M sodium citrate from approximately 10 grams soil samples heated in a water bath at 80-90°C. The solution was renewed after every two hours being preceeded by cooling, centrifugation and decantation. At the end of the extraction period, there were six separate extractants. The presence of aluminum in each periodic extract was determined colori-metrically on a Bausch and Lamb Spectronic 20 by the Aluminon method (McLean 1965).

<u>Total Cation</u>: The total cations (Ca⁺², Mg⁺², K⁺, Na⁺, A1⁺³) present were determined on a Perkin Elmer Atomic Absorption 303 machine after digestion with Perchloric and Hydrofluoric acids (Pratt, 1965).

Surface Area

Surface area determinations of whole-soil samples were carried out after the E.G.M.E.* method of Heilman, Carter and Gonzalez (1965).

Organic Matter and pH

Organic matter was determined by the modified Walkley and Black procedure, Chapman and Pratt (1961). pH determinations were by (1) IN KC1-Soil 1:1 ratio (2) Soil-water 1:1 ratio Peech (1965).

*Ethylene glycol monoethyl ether.







CHAPTER IV

RESULTS AND DISCUSSION

The results suggest that the structure of the soil aggregates had some effect upon both swelling and shrinkage. The relations obtained, indicate that better structure is associated with particular shrinkage conditions. These have the effect of reducing shrinkage for the soil. Evidence for this behavior is presented in Table X where less shrinkage has been obtained in the Brewer than in the San Saba, which exhibited greater shrinkage than the Dwight. The influence of the interlayered materials which occur naturally, is seen to be important in the degree of shrinkage. Although the San Saba, with a higher per cent clay, exhibited greater swelling than the Brewer, the amount of citrate-extractable Al-compounds was greater for the Brewer series. The question therefore arises as to what was the more important in the swelling of the San Saba, the high clay (montmorillonite) content or the lack of significant amounts of interlayered Al-compounds.

Dwight

X-ray diffraction analysis indicated poor crystallinity of all the minerals in both the coarse (0.2-2.0 μ) and fine ($< 0.2 \mu$) fractions. The expansion of the glycol-saturated samples to 22-23.8A^o bears this out. The presence of a 14-15A^o Mg-saturated diffraction peak occurred only in the fine clay fractions. The presence of

<u>^</u> /

TABLE I

	P	Ϋ́H	· • • • • • • • • • • • • • •	CEC WI	ole Soil	— <u> </u>	·		
	Soil	Soil	ОМ	meq/	'100 gms		Total (ppm)	
Depth (cm)	H ₂ 0	Kcl	%	With	Without				
	1:1	1:1		OM	OM	Ca	Mg	К	Na
				DWI	GHT				
0.0 - 7.6	6.5	6.1	2.08	6.19	2.87	693.75	2.95	33.00	55.00
7.6 - 16.5	6.8	6.3	2.03	7.63	2.90	690.65	6.95	34.05	49.00
16.5 - 22.9	6.9	6.2	1.61	6.86	3.21	784.25	11.55	34.30	59.50
22.9 - 35.6	6.8	6.1	1.65	12.56	5.98	943.75	11.55	34.80	57.50
35.6 - 49.5	6.6	6.0	1.58	13.75	6.48	943.75	13.50	36.50	60.75
49.5 - 63.5	6.8	6.2	1.69	13.75	6.43	1037.25	14.95	37.00	43 。 50
63.5 - 73.7	6.9	6.2	1.36	6.90	3.34	1009.40	15.00	37.80	41.00
				BRI	WER				
20.3 - 30.5	7.7	7.1	3.11	27.80	23.80	50.00	24.60	138.00	54.00
30.5 - 40.6	7.7	7.0	2,99	28.60	22.40	75.00	33.60	148.00	46.00
40.6 - 58.4	7.8	7.0	2.80	30.20	22.60	43.00	37.50	165.00	79.00
48.3 - 58.4	7.8	7.0	2.51	29.20	23.20	46.00	49.50	173.00	103.00
58.4 - 89.0	7.6	7.1	2.34	17.20	15.40	35.00	54.60	162.00	104.00
91.4 - 121.9	8.0	7.3	0.55	14.00	10.60	102.00	25.20	171.00	121.00
121.9 - 152.4	8.1	7.4	0.47	12.80	10.00	99.00	23.70	165.00	128.00
				SAN S	SABA				
0.0 - 15.2	7.7	6.9	4.09	42.10	38.60	734.51	23.10	44.12	58.00
25.4 - 35.6	7.9	7.0	3.20	44.90	40.10	774.36	34.20	39.12	62.00
50.8 - 61.0	8.1	7.2	2.83	39.30	38.20	942.81	35.71	40.12	62.00
63.5 - 76.2	7.7	7.1	2.03	33.70	32.80	901.74	47.84	37.38	64.00

CHEMICAL PROPERTIES

TABLE II

DEPTH (CM)	% Clay		SILT	SAND	FREE	TITANIUM	TOTAL A]
	2.0-0.2 ù	0.2 u	%	%	Fe ₂ 0 ₃ %	%_	%
<u> </u>	·			DWIGHT	· ·		
0 - 7.6	29	3	37	31	0.03	0.01	3.42
7.6 - 16.5	33	4	32	31	0.03	0.02	3.42
16.5 - 22.9	37	4	31	28	0.03	0.02	4.04
22.9 - 35.6	36	18	21	25	0.03	0.05	5.24
35.6 - 49.5	31	26	22	21	0.03	0.02	5.74
49.5 - 63.5	24	26	29	21	0.03	0.01	5.78
63.5 - 73.7	32	29	11	28	0.04	0.01	6.00
				BREWER			
20.3 - 30.5	4.7	57.4	33.0	4.8	0.03	0.01	4.66
30.5 - 40.6	33.4	21.8	36.6	8.0	0.03	0.01	5.46
40.6 - 58.4	54.0	14.8	26.1	4.2	0.04	0.08	6.04
48.3 - 58.4	36.5	21.0	34.0	8.5	0.03	0.01	6.58
58.4 - 89.0	43.2	17.6	30.8	8.4	0.02	0.01	5.32
91.4 - 121.9	46.7	8.2	27.5	17.5	0.03	0.05	6.68
21.9 - 152.4	32.6	11.9	35.0	20.4	0.02	0.07	5.78
				SAN SABA			
0.0 - 15.2	17	43	13	27	0.09	0.01	7.58
25.4 - 35.6	19	44	13	24	0.10	0.01	8.00
50.8 - 61.0	30	29	8	33	0.10	0.01	8.08
63.5 - 76.2	25	34	8	33	0.10	0,01	7.96

PHYSICAL ANALYSIS

TABLE III

EGME RETENTION AND SURFACE AREA OF SOILS

	EGME Retained	Surface Area
Depth (cm)	mg/g	m ² /g
	DWIGHT	
0.0 - 7.6	14.30	45.18
7.6 - 16.5	18.70	59.20
16.5 - 22.9	22.42	71.51
22.9 - 35.6	28.69	89.41
35.6 - 49,5	41.34	131.54
49.5 - 63.5	37.87	119.59
63.5 - 73.7	15.21	47.33
	BREWER	
20.3 - 30.5	41.30	131.67
30.5 - 40.6	52.80	168.49
40.6 - 58.4	46.60	148.35
48.3 - 58.4	86.80	283.42
58.4 - 89.0	60.00	194.05
91.4 - 121.9	35.00	111.02
121.9 - 152.4	23.73	75.04
	SAN SABA	
0.0 - 15.2	88.75	291.71
25.4 - 35.6	95.04	317.71
50.8 - 61.0	87.62	287。48
63.5 - 76.2	76.91	252.19
		······································
TABLE IV

X-RAY DIFFRACTION d-SPACINGS IN A^O (001) DWIGHT (0.2-2.0)

Depth (cm)	Mg-Sat. 25 ⁰ C	Glycol Solv. 25 ⁰ C	K Sat. 25 ⁰ C	Heat 500 ⁰ C
0.0 - 7.6	7.248	7.248		,
······································	3.348	3.361	3.161	
7.6 - 16.5	7.248	7.189	3.610	
	3.348	3.348	3.150	
16 5 22 0	7 2/9	7 2/0		
10.5 = 22.9	7.240	/ • 2 40 2 = 7 2		
	. 3.312	2 9/9	2 208	3 208
	3,348	0409 C	3,161	3,172
		<u> </u>		,
22.9 - 35.6	14.717	Shoulder		
		10.155	10.517	
	7.189	7.189	7.248	
	5.034			
	3.587	3.572	3.587	
	3.348	3.335	3.398	
			3.161	······
356 - 495	14 717	Shoulder		
JJ • 0 = +/•J	7,189	7.189		
	3,587	3,587		
	3.348	3.348	3.139	
(0.5				•
49.5 - 63.5	15.225	7 100		
	7.190	7.189		
	4.260	2 507		
	3.58/	3.58/	2 1 5 0	2 120
	3.348	3.340	5.130	3.139
63.5 - 73.7	14.248	19,190		
	10.273	10.273		
	7.189	7.248		
	3.373	3.360		
			3.161	3.150

Depth (cm)	Mg-Sat. 25°C	Glycol Solv. 25°C	K Sat. 25°C	Heat 500°C
0 0 7 6	15 /01	22 071		
0.0 - 7.0	2 559	22.071		
	3 /80	2.00		
	5.400		3,150	
7 6 16 5	1 5 76 9	22 071		
1.0 - 10.5	15.700	22.071		
	2,000	2,557	2 150	
<u> </u>	3.340	3.130	5.150	
16.5 - 22.9	16.660	23.859	Shoulder	10.390
	3.348	3.348	3.361	3.361
		• • • · · · · · · · · · · · · · · · · ·	3.161	
22 9 . 35 6	15 768	22 071	Shoulder	
22.9 = JJ.0	2 272	22.0071	3 361	
		5,015	3.139	
35.6 - 49.5	16.054	Shoulder		
	7.368	10.273		10.155
	3.558	3,530		
	3.348	3.360	3.160	3.373
				3,150
49.5 - 63.5	16.054			
	7.180	7.189		
	3.587	3,587		
<u>_,</u>	3.348	3.348	3.160	3.150
63 5 73 7	15 005	Shouldon		
1000 - C.C.	7 360	7 180		
	2 558	1.107		
	3.360	3,335	3,150	3,139
	J.JUU	CC*C	J • T J 0	J 0 I J 7

TABLE V

X-RAY DIFFRACTION d-SPACINGS IN A^O (001) DWIGHT (0.2 u)



Figure 3. X-ray diffraction patterns and d-spacings A° Dwight (49.5 - 63.5 cm) 0.2-2.0 u and 0.2 u.



Figure 4. X-ray diffraction patterns and d-Spacings A^O Dwight (63.5 - 73.7) 0.2-2.0 u and 0.2 u.

kaolinite was evidenced by the 3.55-3.58A^O diffraction peaks. Quartz and Gypsum was evidenced by the 3.35 and 3.15-3.16A^O in every section of the profile. The presence of first order kaolinite was observed, but the 7.18-7.24 peaks which occurred were completely collapsed as a result of either glycol or K-saturation.

The silt fraction is composed largely of quartz and gypsum with occasional evidence of the presence of feldspar.

An increase in expanding minerals was directly related to an increase in E.G.M.E. retention as evidenced by an increase in specific surface area determined.

Brewer

The x-ray diffraction data indicates that even the clay from the lower horizons may have been weathered to some degree or have new materials deposited. This is shown by the presence of some expanded layers that upon dehydration increase to the intensity of the $10A^{\circ}$ maxima. Some amount of interstratification is evident in some portions of the profile. This is suggested by the peaks being rather broad and tapering to the lower angle, and the increased intensity in some instances of the $10A^{\circ}$ spacing upon heating.

The data on E.G.M.E. retention, clearly shows some increase in specific surface area in lower depths, reaching a maximum in the zone of highest clay accumulation.

X-ray diffraction studies show that the silt fraction is composed largely of quartz. Feldspars are present throughout all depths. No montmorillonite illite or kaolinite was found in the silt fraction.

The results show that the clay fraction is composed primarily of

* * .

					·····
Depth	(cm)	Mg-Sat. 25°C	Glycol Solv. 25°C	K Sat. 25 [°] C	Heat 500 ⁰ C
20.3	- 30.5	10.040	10.040		
		7.1897	7.2484		
		³ •5728	3.5728		
<u></u>	·	3.3482	3.3482	3.3482	3.3731
30.5	- 40.6	14.717	18.394		
		10.273	10.040		
		7.2484	7.1897		
		3.5728	3.5728		
		3.3482	3.3482	3.3482	3.3482
<u></u>				3.1399	3.1399
40.6	- 58.4	15,491	18.785		
		10.040	10.155		
		7.1897	7.1897		
		5.0065	3.5728		
•		3.587	3.3482	3.3482	3.3482
		3.3482		3.1617	3.1/2/
48.3	- 58.4	14.717	18.785		10.155
		10.155	10.040	10.040	
		7.2484	7.132	7.1897	
		3.5870	3.5587	3.5728	_
		3.3606	3.3482	3,3482	3.3359
·		·····		3.1508	
58.4	- 89.0	15.225			
		10.155			
		7.2484			
		5.0348			
		3.5728			
		3.3482	3.3482	3.3482	3.3482
		<u></u>		3,1508	3,1508
91.4	- 121.9	17.659	18.394		
		10.040	10.155		
		7.1897	7.2484		
		3,5870	3.5587		
		3.3482	3.3482	3.3482	3.3482
. <u> </u>	· · · · · ·			3.1508	3.1399
121.9	- 152.4	14.717	18.394		
		10.273	10.040		
		7.2484	7.1897		
		3.5728	3.5728		
		3.3482	3.3482	3.3482	3.3482
				3.1399	3.1399

X-RAY DIFFRACTION d-SPACINGS IN A° (001) BREWER (0.2-2.0 λ)

			-	
Depth (cm)	Mg-Sat. 25°C	Glycol Solv. 25°C	K Sat. 25 [°] C	Heat 500°C
20.3 - 30.5	14.967 4.9511 3.3236	19.193 10.040	10.040	10.273 3.348
30.5 - 40.6	13.798	19.193 9.8168		
	3.3482	3.3482	3.1508	3.1508
40.6 - 58.4	14.967 5.0633 3.3482	20.065 9.8168 3.3731	0.1500	. 1500
48.3 - 58.4	14.717 4.9511 3.3482	18.780 10.273 3.3482	3.3482 3.1508	10.517 3.3482
58.4 - 89.0	15.225 5.0065 3.3482	15.225 3.3731	3.3236 3.1399	3.3606
91.4 - 121.9	15.768 4.9511 3.3482	18.394 9.8168	3.1508	3.1399
121.9 - 152.4	15.491 4.9787 3.3482	19.619 10.394 3.3482	10.394 3.3482	10.394 3.3606
			3,1508	3.1508

TABLE VII

X-RAY DIFFRACTION d-SPACINGS IN A^O (001) BREWER (<0.2.41)



Figure 5. X-ray diffraction patterns and d-Spacings A^{O} Brewer (40.6 - 58.4 cm) 0.2-2.0 A1 and < 0.2 A1.

4

-20

3

montmorillonite which gave a $17-18A^{\circ}$ reflection upon ethylene glycol solvation. What has been interpreted as a first order kaolinitic peak occurred throughout most depths at $7.19-7.24A^{\circ}$ but collapsed completely upon glycol solvation. This behavior occurred only in the coarse fractions (0.2-2.0 u). Lesser amounts of kaolinite (3.57-3.59 A°) and quartz was detected, the quartz in both clay fractions and the kaolinite only in the coarse fractions. Illite was detected in both clay fractions, but there was evidence of better crystallinity in the 0.2-2.0 u fraction.

Diffraction determinations of the silt fraction indicated maxima at the $3.21-3.25A^{\circ}$ interpreted as feldspatic components. Quartz maxima at $3.35-3.67A^{\circ}$ were the most pronounced. Both coarse and fine silt fractions were quite similar throughout the profile,

San Saba

The particle size data Table II shows an approximately equal distribution of silt and clay throughout the profile. The results of the chemical analyses are reported in Table I. The p.H. ranges from 7.7 in the 0-6" depth to a maximum of 8.1 in the 51.0-61.0 cm depth. In general, the pH tends to increase with depth. The organic matter content tends to decrease with depth reaching a minimum in the 63-76 cm depth.

The high cation exchange capacities of the San Saba corroborate the results of x-ray diffraction and specific surface area studies, indicating a high montmorillonite content.

X-ray diffraction studies show that the silt fraction is composed largely of quartz. Feldspars are present throughout all depths. No

TABLE VIII

Depth	(cm)		Mg-Sat. 25°C	Glycol Solv. 25°C	K Sat. 25 [°] C	Heat 500 [°] C
0,0	- 1	5,2	14.717 7.189 3.580 3.340	19.619 7.130 3.550 3.340	3.150	
25.4	- 3	5.6	14.717 7.130 5.060 3.610 3.370	19.610 7.240 3.600	3.360	3.360
50.8	- 6	1.0	14.717 7.240 3.580 3.340	19.190 7.180 3.550 3.360	7.24 3.580 3.340 3.150	10.270 3.340
63.5	- 70	6.2	14.967 7.189 3.550 3.340	18.780 3.340	3.340	10,510 3,160

X-RAY DIFFRACTION d-SPACINGS IN A° (001) SAN SABA (0.2-2.0 A))

TABLE IX

X-RAY DIFFRACTION d-SPACINGS IN A° (001) SAN SABA (<0.2 A_{1})

Depth (cm)	Mg-Sat. 25 [°] C	Glycol Solv. 25°C	K Sat. 25 C	Heat 500°C
0.0 - 15.2	14.717 7.360 3.550	18.780 7.300 3.510	3,160	3.140
25.4 - 35.6	15.220 7.300 5.000 3.570	19.190 7.240 3.510	3.160	3.150
50.8 - 61.0	14.717 3.550	18.390 3.550	15.760 3.150	
63.5 - 76.2	16.350 7.300	18.390	· · · · · · · · · · · · · · · · · · ·	· · · · · · · ·





Figure 6. X-ray diffraction patterns and d-Spacings A^O San Saba (63.5-76.2 cm) 0.2-2.0 A and $\angle 0.2$ A.

montmorillonite, illite or kaolinite was found in the silt fractions as is seen in Figure 25. Diffraction studies of the 2.0-0.2 au and

0.2 μ fractions indicated, (Figure 9) that it is composed primarily of montmorillonite (17-18 A°) with lesser amounts of kaolinite (3.57-3.59 and 7.15-7.20 A°) quartz 3.35 A° . Illite was not detectable in the 2.0-0.2 μ fraction; its presence was nevertheless verified by x-ray diffraction analyses of the \lt 0.2 μ . The quartz content tends to be relatively uniform throughout the profile whereas kaolinite tends to increase with depth.

Most of the montmorillonite was concentrated in the $\angle 0.2$ u fraction (Figure 6). Kaolinite and quartz are the dominant minerals in the 2.0-0.2 μ fraction with lesser amounts of montmorillonite. The most noticeable feature is the lack of any large differences from one layer to another within the profile. Generally, there seems to be a gradual increase in total carbonates with depth.

The Brewer and San Saba showed evidence of aluminum compounds being the principal component of the interlayer material. No evidence was found for naturally occurring interlayers being composed of hydroxy-Fe groups.

The data obtained for citrate extractable interlayered Al-compounds from the three soils are summarized in Figures 7, 8 and 9. Highest Al-compounds were obtained for the Brewer. Lowest extractable Alcompounds in the study was from the Dwight soil. Table I gives the pH measurements for each soil and depth. It appeared that pH did not influence the amount of extractable Al-compounds as did the type of clay present. Selected x-ray diffractograms from various depths indicate a direct relationship between the presence of montmorillonite



Figure 7. Na-Citrate Extractable Al-compounds (%) San Saba. A total of six extractions carried out on all three soils; figures 3, 4, 5. Each extraction period being 2 hours. Aluminum extraction in ppm in Appendix.



Figure 8. Na-Citrate Extractable Al-Compounds (%) Dwight.





TABLE X	
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MOISTURE RELATIONSHIP AND SHRINKAGE BEHAVIOR

			Average Moisture Content at Linear Shr Saturation* (% Dry Weight				inkage %		
			of Soil Solut	Ave.	Ave. of four readings				
·····			мана та спира инстрийний у стити и солоний и на т		Horiz	ontal	Verti	cal	
Depth	(cm)	OH/A1	NaC1	OH/A1	NaC1	OH/A1	NaC1	
D				, , ,					
0.0	-	7.6	7.0	5.1	0.5	0.5	0.5	0.5	
7.6	-	16.5	7.5	5.8	0.5	0.5	0.5	0.5	
16.5	-	22.9	8.1	6.2	0.5	0.5	1.0	0.5	
22.9		35.6	10.3	8.0	0.5	1.5	1.5	0.5	
35.6	-	49.5	15.6	9.7	1.0	1.5	2.0	1.0	
49.5	-	63.5	13.2	10.1	1.5	1.1	2.0	0.5	
63.5	-	73.7	12.3	7.3	0.5	0.5	1.5	0.5	
B									
20.3	-	30.5	16.9	12.6	1.5	0.5	1.0	0.5	
30.5	-	40.6	. 17.8	14.8	2.0	1.0	1.5	0.5	
40.6	-	58.4	19.2	17.1	2,0	1.5	2.5	1.0	
48.3	-	58.4	19.9	17.5	1.5	1.5	2.5	1.0	
58.4	-	89.0	24,1	21.1	2.5	0.5	3.0	1.5	
91.4	-	121.9	18.7	12.8	1.5	0.5	1.5	0.5	
121.9	•	152.4	9.4	6.3	0.5	0.5	0.5	0.5	
SS									
0,0	-	15.2	18.6	10.7	1.5	2.0	2.5	1,5	
25.4		35.6	23.5	19.3	2.5	1.5	2.5	2.0	
50.8	-	61.0	28.1	18.9	3.5	1.0	3.5	2.5	
63.5	-	76.2	14.7	12.4	2.5	1.1	2.5	1.5	

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* Point at which detectable swelling ceases.

D Dwight

B Brewer

SS San Saba

and extractable Al-compounds.

The observation was made with respect to some samples on their x-ray diffraction, notably the Dwight and San Saba, Figure 24, that a peak produced at 7.18-7.24A^O when Mg-saturated, was completely collapsed upon K-saturation. Such a behavior seemed to suggest a 2:1 type clay mineral. However, no expansion was observed upon ethylene glycol solvation. This ruled out the initial suggestion of a second order montmorillonite.

In an effort to identify this mineral samples were boiled for $2\frac{1}{2}$ minutes in 0.1N NaOH, centrifuged and saturated with MgCl₂. Upon proceeding with successive treatments of Mg-saturation, ethylene glycol solvation and heating to 300° C and 500° C the mineral was identified as kaolinite. The assumption is that due to the presence of some amorphous material, there was a kind of masking effect prior to treatment with NaOH. Obviously the amorphous material was destroyed by the NaOH treatment.

The results of the titanium and iron determinations in the soils are recorded in Table II. The results show no relationship between titanium content and cation exchange capacity of the soils. The titanium content remains practically unchanged with depth. The present study shows that iron and titanium content is very low in all three soils and shows no noticeable mobility.

Hydroxy/cation Ratio

The OH/cation ratio of interlayer material is a question that is largely unresolved. Attempts have been made in the past to relate the change in C.E.C. with the amount of $A1^{+3}$ added or removed from the

interlayer space. A major problem is whether all the added material enters the interlayer space or whether all the extracted material comes from the interlayers.

All expansible layers in a mineral particle may not react in the same manner to hydroxy-Al (Barnhisel and Rich, 1966). To explain the larger extractable Al-compounds, and larger degree of swelling in montmorillonite, it may be postulated that the expanded interlayer space in montmorillonite provides a favorable locale for the organization of hydroxy-Al ions. The absence of montmorillonite, or the presence of small quantities of montmorillonite in some samples is associated with reduced swelling and lower extractable Al-compounds. This type of swelling behavior can be observed from Figures 14-23. Much reduced swelling pressures have been produced near to the surface. X-ray diffractograms did not indicate the presence of Montmorillonite above 9 inches in the Dwight soil. Similarly, the least amount of swelling and citrate-extractable Al-compounds was obtained above the 9 inch depth for this soil.

It has been discussed (Colombera, 1971) that the amount of aluminum which can be adsorbed increases with increasing OH/A1 ratio of the solution. The results obtained in these experiments show some agreement in regions of high clay content (montmorillonite). There were significant deviations from this behavior in samples low in swelling type clays.

Sulfate Ions in Swelling

The magnitude of sulfate adsorption by soils may follow the order of chemical valency of the saturating cations. Chao, et al.

(1963) found that saturation with divalent or trivalent cations resulted in greater sulfate adsorption than with monovalent cations. Sulfate adsorption by soil may be governed by more than one mechanism. One might be based on differences in chemical valency of the cations involved. Divalent or trivalent exchangeable cations may act as a bridge between the sulfate ions and the soil complex. This type of soil-cation-anion linkage has been suggested by Ravikovitch 1934, and Birch 1951 to explain the adsorption of phosphate ions through exchangeable calcium. Wild (1953) explained the effect of exchangeable cations on the retention of phosphate by clays on the basis of relationships of different cations to the Zeta potential.

From a purely chemical viewpoint, one may predict that in the presence of strongly co-ordinating anions (those which have a decided tendency to form complex ions with aluminum), less hydroxyl ions would be required to precipitate the aluminum. Such would be the reaction, since these strongly coordinating anions have already neutralized a part of the charge on the aluminum ion.

Sulfate is a moderately strong co-ordinator with aluminum, and as the sulfate ion concentration in the solution is increased, the amount of added hydroxyl ion needed for precipitation decreases. The suggestion here is that much of this reaction takes place at the time of solution preparation. However, the introduction of this solution into the soil provides the needed surface for deposition of the precipitate. The assumption is that this precipitate is primarily A1 (OH)₂.

To further understand the reactions that might have taken place in the saturated samples, Comargo Bentonite was aged in 1:5 OH/Al ratio

solutions from August 5, 1971 to May 22, 1972. Results of x-ray diffractions are presented in Figure 26. When the aged samples were washed three times with distilled water the montmorillonitic peak was produced at 15.768A^o. Upon being solvated with ethylene glycol, there was an expansion to 18.785A^o.

When this sample was Mg-saturated, a diffraction spacing of 14.717A° was obtained. Upon being solvated with ethylene glycol there was an expansion to 16.66A° The difference between the 14.717A° and 15.768A° outlined above, and the expansion of the 14.717A° to 16.66A° seem to indicate a removal of some excess Al-interlayered material. This material contains some sulfate ions some of which should be in the easily exchangeable form. This is supported by the reduced d-spacing upon Mg-saturation. Solvation with ethylene glycol resulted in expansion to 16.66A° suggesting the presence of some interlayered material which resisted further expansion. This induced interlayered material must have been the hydroxy-Aluminum or hydroxy-Aluminum-sulfate complex formed as a result of aging. The above monitored interlayered formation indicates to some extent the expansion effects of the hydroxy-aluminum solutions upon the samples during the process of swelling. When the ethylene glycol solvated samples were K-saturated, the result was a collapse and broadening of the $18A^{\circ}$ peak. Such a response has been regarded as a definite indication of interlayering. Heating of the K-saturated sample to 5000° resulted in a collapse and total disappearance of the broadened K-saturated, air-dried sample. The implication is that the high temperature

destroyed the induced interlayering material.

It is believed that upon Mg-saturation the excess hydroxy-Aluminum complex is removed from the interlayered region. The effect of such a removal would be to increase the intensity reflections. The saturation with magnesium may also have resulted in loosening of exchangeable aluminum-hydroxy complexes from the interlayer spaces.

The above discussion may explain the improved peak intensity when magnesium saturated, over the sample saturated with the OH/Al solution. The presence of sodium in the complex from the sodium hydroxide, most likely will result in the neutralizing of most of the negative charges while the hydroxy-aluminum polymers would be positively charged.

Some Mechanics of Shrinking and Swelling

There are several aspects of the process of volume change in the soil as a result of water content change. First, the soil volume may change by uniform swelling or shrinking in three dimensions. Such a process (three-dimensional volume change) results in cracking during drying and a closing of the cracks during wetting. Such behavior is most obvious in the San Saba clay. Alternatively, the soil volume may change in the vertical dimension only. This is a process (one dimensional volume change) in which plastic flow of the soil and greater rearrangement of the particle configuration is involved. Within the soil profile this type of volume change is likely to occur more at depths within the soil when water contents are high and weight of overburden is considerable.

The data of figures 14 to 23 support the interpretation that the process of shrinking and swelling in the clay horizons of the - 47

selected soil profiles approximates quite closely the condition of three dimensional normal volume change.

The dominant soil characteristics which might logically be expected to affect swelling and shrinkage are texture, structure and type of clay mineral. The marked influence of texture has been demonstrated in the different behavior and characteristics of the clay (montmorillonite) and non-clay horizons. The true effect of structure has not been fully assessed, but the hypothesis is advanced, based on qualitative evidence, that soils composed of small structural units or aggregates exhibit a greater range of shrinkage and swelling than coarsely structured or massive soils.

Because of the possible complex inter-relationship of the clay mineral component of these soils it is difficult to relate differences of soil swelling behavior to differences of clay type alone. The further interaction of saturating solution has to be considered. Some of the measured swelling might in fact be due to readjustments of the crystal lattice as well as the process of normal volume change.

Observations made on the changes in the form of the soil cubes during swelling have been made. When Sodium Chloride was the saturating solution, the cubes were swollen to a smooth homogeneous appearance. Samples saturated with the OH/Al solutions of the higher ratios, 1:2 and 1:3 OH/Al saturated, underwent cracking before attaining saturation. These large cracks undoubtedly contributed to the total measured swelling. How much of this total swelling was due to these cracks is unknown. It may be argued, however, that these cracks were a part of the total swelling. This hypothesis may be supported by the fact that upon shrinking, these cracks did not close. Such re-

sponses were more pronounced in the samples of high montmorillonite clay content (San Saba and Brewer), and agrees with the shrink-swell behavior of these soils, particularly the San Saba, under natural conditions.

The large degree of swelling obtained with the OH/Al solutions over that of the sodium chloride solutions may be ascribed to the introduction of interlayered material into the interlayered regions of the montmorillonitic clays present. The occurrence of significant degrees of swelling only in samples in which montmorillonite was determined by x-ray diffractions to be high seems to suggest this point. A significant observation made, was that in samples high in montmorillonite, the OH/A1 ratios of 1:2 and 1:3 invariably resulted in the greatest degree of swelling. However, in samples low in montmorillonite, the OH/Al ratios which induced the greatest degree of swelling were the 1:5 and 1:4 respectively. The interpretation of this may be that at high clay (montmorillonite) content, ratios 1:2 and 1:3 resulted in the greatest degree of interlayering. On the other hand at lower clay content, it required ratios of 1:5 and 1:4 to deposit a certain amount of crystalline material for large swelling. The effect of the sulphate ions in these reactions so far as is known may be significant.

The higher degree of interlayering in samples high in montmorillonite, San Saba and Brewer, may be attributed to the differences in the amount and location of charge between the minerals.

The likelihood of cation demixing Fink et al. (1971), being responsible for a major portion of the swelling cannot be overlooked. In the past, it was largely assumed that all exchangeable cations

would be homogeneous in their distribution on the clay mineral surfaces. Experimental evidences continue to accumulate which seem to suggest that some exchangeable cations do indeed demix on some clay minerals. This is particularly so for montmorillonite. Such demixing could influence water movement, swelling and dispersion of soil materials.

The significant differences obtained at different depths and between different soils, was directly related to the clay (montmorillonite) content. For this reason, curves showing degree swelling in millivolts against time are shown from selected depths. Further information on this relationship may be observed from the data in the Appendix.

Samples which attain saturation at a rapid rate are usually those in which montmorillonitic clay is almost absent. It may be argued that in such cases, it is the pore spaces primarily in which the solution enters that is responsible for much of the recorded expansion. Once the pore spaces are saturated, no more solution can be taken up, the curves straighten out and become horizontal. From this point on, no more expansion of the sample takes place.

Contrary to absorption where only physical forces are operating, adsorption is due to physico-chemical energies whereby a certain amount of the solution is taken up and fixed in the molecular structures of the clay minerals. Thus, deposits from the various solutions may be fixed at the broken bands and eventually the surface of the clay mineral particles. Some of the materials may enter the lattice itself as some form of hydroxy-Al compound causing expansion of the c-axis. This type of expansion to a lesser extent may also take place in the case of sodium saturating systems. No expansion will take place in quartz, very little or none may be observed in Kaolinite and Halloysite but an appreciable amount of adsorption will be noticed in Montmorillonite. An expansion of the soil that contains montmorillonite clay is to be expected. As is shown in the x-ray diffraction curves, Figures 6 to 9 and recorded data, Tables IV to IX, expansion will be directly related to the amount of montmorillonite present in the soil. It will be noticed also, that whatever the adsorption, definite and characteristic swelling curves are obtained for each sample as well as for the different solutions.

As a rule, soils are not pure mixtures of clay minerals. They contain impurities of many kinds and these impurities exert a more or less appreciable influence on the sorption characteristics of the soil. The important point however, is not the exact identification of the clay mineral of the soil, but the behavior of the soil when in contact with water or any given solution.

The concept of edge-to-face bands resisting swelling may be a successful explanation of most of the observed features of the swelling of clays. Figure 13 shows the probable structure of a swollen oriented clay sample, with edge-to-face contacts between the elementary sheets in the positions marked 'A'. As swelling takes place, sheets will move in relation to one another, and new ones will be formed. With this concept in mind, the more random the orientation then the greater the extent to which the structure will oppose change, either swelling or shrinking. This may adequately explain the higher degrees of swelling obtained in samples high in clay (montmorillonite). The edge-to-face bands have the same effect as an applied load. With no external load, swelling is opposed only by these frictional forces,











Figure 12. Typical rate of swelling; San Saba (63.5-76.2 cm) 1:5 OH/A1.



Figure 13. Schematic diagram of a swollen oriented aggregate of Na-montmorillonite A, edge-to-face bands.

and it is easy to see that an asymetrical distribution of interlayer distances may result.

In view of the excellent agreement between theory and experimental results with Na⁺ and Cl⁻ ions, the large differences when the saturating solutions comprised Na⁺, Al⁺³, SO₄⁻², OH⁻ raises interesting questions. As regards the behavior of soils when sodium and chloride are the predominating ions present, no mechanism operative under such circumstances has been adequately explained. Van der Waals forces have been frequently mentioned in this connection, but the magnitude of such forces acting between relatively large particles at relatively great distances has not been well understood, it seems. Since these forces seem to be negligible in Na-Montmorillonite systems, the assumption might be that their presence in an environment of A1(OH)₃ is absent.

The data show that when NaCl is the saturating solution, the samples have a smaller water holding capacity than when varying OH/Al ratio concentrations are the saturating solutions. Results show that the water holding capability of a soil due to swelling pressure forces depend on its mineralogical composition, the amount of salt present as well as the saturating ionic species. A close examination of the degree of swelling exhibited by the Dwight as compared to the Brewer or San Saba will bear this out. The data reported for mixed-ion montmorillonite, suggest that the degree to which water-holding capability changes is dependent upon the relative proportions of the cations occupying the exchange sites. A linear dependence is assumed. Probably certain physical properties of a clay or soil-water system change gradually in a manner proportional to the predominant electro-

lyte present. Some amount of linearity can be observed in certain regions of soil swelling, Figures 10 to 12.

An important feature of the illustrations as typified in Figures 20 to 22 has been the general close relationship and appearance of the curves representing lateral swelling. There are a few exceptions as are illustrated in total swellings, Figures 14 to 23. Initially, the rate of swelling is fairly uniform, then at a certain point, probably determined by salt accumulation, the rate of lateral swelling increases rapidly. This behavior seems to support the hypothesis that swelling controlled the changes in permeability over a range of electrolyte accumulation. Beyond a certain saturation level, the soil particles undergo some amount of dispersion activity and movement.

Solutions of electrolytes induce the formation of spontaneous structures under the influence of molecular forces which tend to lead to the formation of an ion envelope. This is especially true when the particles have a lamella shape, as in the case of clay. Salt solutions exert an influence only on irregularly unstably grouped particles. This concept agrees well with earlier experiments with soil swelling in electrolyte solutions.

Solution absorption and swelling of soil, are determined by the structure of the particle orientation. Particular importance is attached to the compactness of the particles at the interface of solid-phase-air.

To really understand the type of swelling obtained, one needs to understand that

(1) The process of swelling is in its essence characterized by the change in properties inside the micelle which accompanies the



Figure 14. Dwight (7.6-16.5 cm) three dimensional swelling in response to varying concentrations of NaCl and OH/A1 ratios.



Figure 15. Dwight (16.5-22.9 cm) three dimensional swelling in response to varying concentrations of NaCl and OH/Al ratios.



Figure 16. Dwight (22.9-35.6 cm) three dimensional swelling in response to varying concentrations of NaCl and OH/Al ratios.



Figure 17. Dwight (35.6-49.5 cm) three dimensional swelling in response to varying concentrations of NaCl and OH/Al ratios.



Figure 18. Dwight (49.5-63.5 cm) three dimensional swelling in response to varying concentrations of NaCl and OH/Al ratios.



Figure 19. Brewer (30.5-40.6 cm) three dimensional swelling in response to varying concentrations of NaCl and OH/Al ratios.


Figure 20. San Saba (0-15.2 cm) three dimensional swelling in response to varying concentrations of NaCl and OH/Al ratios.



Figure 21. San Saba (25.4-35.6 cm) three dimensional swelling in response to varying concentrations of NaCl and OH/Al ratios.



Figure 22. San Saba (50.8-61.0 cm) three dimensional swelling in response to varying concentrations of NaCl and OH/Al ratios.



Figure 23. San Saba (63.5-76.2 cm) three dimensional swelling in response to varying concentrations of NaCl and OH/Al ratios.

volume increase (intramicellary swelling). Utilizing this notion, the following types of swelling can be distinguished.

- (a) The swollen micelle retains its molecular bond intact. This type of swelling is explained by the osmotic action of the solution within the micelle.
- (b) The swelling is characterized by a complete reconstruction of the inner structure of the micelle. This exerts a complicated influence on swelling.
- (2) The differences in structure of the soils have proven to be quite important in the swelling of argillaceous minerals. An example of this is to recognize that the crystalline network of kaolinite is less sensitive to the action of water than that of montmorillonite. Proceeding from this, it is easily understood that the destruction or unsettling of the natural aggregates or disposition of the soil will influence swelling to a high degree.

Comparison of Results with Previous Investigations

Many investigations have conducted swelling pressure and/or swelling potential tests on compacted soils. In other cases, confined and unconfined swelling pressures have been investigated on extracted soil clays and pure bentonite. Over the last decade no one has looked at the swelling of soils in their natural orientations in multiple directions. These swelling investigations have been conducted on preferentially oriented clays, therefore very little information is available for samples investigated in random orientation.

Because of the marked effects of interlayers on swelling and shrinking of clays, many attempts have been made to determine the

nature of interlayers and the processes by which they are formed. These studies have involved studies of soil profiles and laboratory synthesis of interlayers in smectites. Work in this area has progressed to the point of removal and analysis of naturally occurring interlayers.

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Some methods employed for removal of Al-interlayers have been based on the complexing ability of citrate for Aluminum. Other proposed methods have been based on the selective destruction of hydroxygroups upon heating at different temperatures. This has been the method adopted in this investigation. However, it should be appreciated that there is the possibility that in the process of dissolving the interlayers, a portion of the silicate structure may also be attacked. Nevertheless, such techniques have proven useful in determining, within limits, the composition and nature of the interlayer material.

Certain generalizations can be made from these studies.

(a) In soil clays, the degree of filling of the interlayer region is generally small.

(b) Artificially produced hydroxy-Al interlayers are more easily removed than those naturally occurring.

(c) No single method of extraction is suitable for all types of interlayers. The severity of the extraction treatment depends on the degree of development (artificial or natural), and composition of the interlayer material.

Much evidence has indicated that the occurrence of Al-interlayers in smectite is very common in soils. Viewed at with respect to soil clay mineralogy, it seems informative to investigate the properties of these minerals as they are in soils and also to disclose the struc-

ture of their original minerals.

The results obtained indicate that the interlayer Aluminum compounds did not have a uniform extractability, some of it being difficultly extracted with N sodium citrate. The mechanisms by which such a phenomena operate may include

(1) A portion of the aluminum compounds occur in the insoluble form and is therefore difficultly exchanged with sodium citrate.

(2) Some of the interlayered compounds occur as polymers and are held tightly by the clay.

(3) The exchange sites of the montmorillonitic clay are not homogeneous in their strength in holding the interlayer aluminum.

The first two mechanisms are obvious from the results presented, but are not adequate to interpret all the results. As a result, the importance of the third mechanism listed above, is being emphasized. A most important factor in studying clay, hydroxy-aluminum interactions is the species composition of hydroxy-aluminum solutions. The composition of these solutions used in this investigation is known but it may be assumed that a range of polynuclear hydrolysis products is present. Thus, the expansion characteristic of the soil will be determined by both the OH/A1 ratio concentration as well as the degree of hydrolysis which is unknown. At the aluminum ratio concentrations used in this study the OH/A1 ratios is considered to be the more important factor in the shrink-swell characteristics exhibited by the various samples.

Some of the observed swelling may actually be that of repulsive reactions between negatively charged clay surfaces and negatively charged sulfate ions. The extent of this repulsion may be due to the type of clay which predominates. Such an assumption may help to explain the larger swelling obtained in samples high in smectite at high OH/Al ratio concentrations as against lower swelling in samples of low clay content.

The decrease in lateral dimensions may probably be due to the breaking up of face to face bonds. An example of this may be in the case of coplanar platelets which do not touch each other but are held together by another parallel platelet overlapping the others.

The smaller swellings of the samples when sodium chloride is the saturating solution may be partially explained by the state of aggregation of sodium-montmorillonite in aqueous solution. From the results obtained and the results of other investigators of swelling behavior it has been revaled that the following factors influence behavior of soils above all others.

- (I) Type and amount of clay
- (II) Stress history
- (III) Temperature of experimental conditions
- (IV) Volume change permitted during swelling, that is, whether the nature of the swelling is confined or unconfined.
- (V) Shape and size of the sample
- (VI) Type of, and concentration of saturating solution

If the experimental method is standardized, the type and amount of clay along with the type of the saturating solution and its concentration may be the basic parameters influencing swelling behavior of soils.



Figure 24. Kaolin identification; NaOH treatment San Saba (63.5-76.2 cm) top; Dwight (16.5-22.9 cm) bottom, 0.2-2.0 A fractions.



Figure 25. Typical Silt X-ray Diffraction d-Spacings (001).

a, b, c Dwight, Miller, San Saba, respectively 0.2 u a_1 , b_1 , c_1 Dwight, Miller, San Saba, respectively 0.2-2.0 u.



Figure 26. Comargo Bentonite 2 u, Aged in 1:5 OH/Al solution. (a) OH/Al-Mg Sat (b) OH/Al-Mg Sat, Gly. Solv. (c) OH/Al-No.Mg. Sat (d) OH/Al. no Mg. Sat; Gly. Solv. (e) OH/Al no. Mg. Sat; Gly Solv., K Sat. (f) Comargo Bentonite-Mg. Sat. no aging.

CHAPTER V

SUMMARY AND CONCLUSION

The most significant contribution of this investigation is believed to be the possibility which the data suggests for a quantitative measurement of the shrink-swell status of soils. The size distribution of the pore space in soil, aggregate analysis, and total porosity determinations have all been used as an index of soil structure. None of these, however, measures the structural level in terms of the degree to which structural development has progressed or deteriorated.

The results of these investigations, provide information concerning the type of clay mineral present in certain soils. It constitutes a measurement of the expansion that may take place under certain conditions and will allow, in some cases, a better interpretation of other soil properties determined by routine tests.

The experiments on swelling in solutions of electrolyte were designed to look at the possibility of regulating the process of swelling in soils by means of adding salt. It was also desired to observe the swelling response of natural aggregates to hydroxy-aluminum compounds. The study of the phenomena of the swelling of soil that retains its natural structural conditions allows investigations of soil structure from a new point of view.

The preparation of hydroxy-aluminum solutions of differing OH/Al ratio concentrations have usually been accomplished by adding NaOH to

7:5

AlCl₃ in solution. The use of Aluminum Sulfate $[Al_2(SO_4)_3]$ has substituted AlCl₃ in this investigation primarily because SO_4^{-2} ions are more common than Cl⁻ ions in the soil.

The identified clay minerals by x-ray diffraction, type of swelling, particle size distribution and interlayer extractable aluminum compounds were all in agreement. The primary clay mineral in both the Brewer and San Saba was montmorillonite. Some amount of mica (illite) was evidenced in the Brewer. Except for a few sample depths the Dwight was void of any montmorillonite. Quartz and a second order kaolinite along with feldspar, were the chief identifiable clay minerals for the Dwight.

In these experiments, varying amounts of interlayer material were probably present in different samples of the same soil prior to treatments. This may complicate the interpretation with respect to degree of swelling. It has been assumed, therefore, that the degree of swelling obtained, is to some extent related the amount or nature of interlayer material present. In predicting interlayer swelling of soil clays in response to mixed-salt solutions (Al-sulfate-NaOH) it has been assumed that each species of exchangeable cation is distributed over all the mineral surfaces in proportion to the exchangeablecation percentage.

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APPENDIX

Profile Description of the Dwight Series

The Dwight series consists of deep, nearly level soils. These soils have been formed under grass, in material weathered from shale. The A horizon ranges from 3 to 8 inches in thickness and from grayish brown to light gray in color. In places it is massive and strongly vesicular when dry. In bare or disturbed areas, a hard crust 1/2 - 1 inch thick forms as the surface dries.

- A₁-0-6" grayish brown (10YR5/2) silt loam; dark brown (10YR4/2) when moist; weak, fine, granular structure; friable when moist and hard when dry; pH 6.0, abrupt, wavy boundary.
- B2lt-6-25" dark grayish-brown (10YR 4/2) clay; very dark grayish brown (10YR 3/2) when moist; few to common, medium, yellowish-brown mottles that are distrinct when moist but faint when dry; strong coarse, blocky structure; very firm when moist and extremely hard when dry; thin, faint clay films are more common on horizontal faces than on vertical faces; many faces of vertical cracks have coatings of pale-brown very fine sandy loam; on horizontal faces these coatings of pale-brown very fine sandy loam; on horizontal faces these coatings are thinner and less common; pH 7.0; graduate boundary.
- B22t-25-39" variegated grayish brown (10YR 5/2) and yellowishbrown (10YR 5/8) clay; coarse blocky structure; very firm when moist and extremely hard when dry; light-gray, very fine sandy loam coatings on faces of some peds, more common on vertical faces than on horizontal ones; small nests and fine seams of salt crystals are common, gradual boundary, pH 8.0.
- B₃-39-60"+ yellowish-brown (10YR 5/4) clay, faint yellowish brown (10YR 4/4) when moist; few, faint, medium mottles of light grayish brown; massive; extremely firm when moist and extremely hard when dry; a few nests or clumps of salt crystals that are coarser than the crystals in horizon B22t; few medium and small concretions; pH 8.5.

Profile Description of Brewer Silty Clay Loam

- Al 0-8" Dark grayish-brown (10YR 4/1.5; 3/1 when moist*) silty clay loam massive to weak coarse blocky very firm; very hard when dry; slowly permeable; pH 7.0, has a weak tendency to coarse platiness and shears readily in the horizontal plane, then breaks further into medium subangular blocks; common pores; roots most abundant in the horizontal cleavage planes; lower portion less compact and somewhat more crumbly; grades shortly to the layer below.
- B2 8-22" Dark grayish brown (10YR 4/1.5; 3/1 when moist) clay, moderate fine subangular blocky; very firm; crumbly when moist; hard when dry; slowly permeable; pH 7.0; sides of peds have very dark gray, somewhat shiny coatings; many pores; common worm casts; occasional old root channels filled with clay loam material from above; occasional brown streaks; grades through a four-inch transition which is more reddish than the main horizon to the layer below.

Sample 54-OK-9-20-3

C1 22-32" Dark brown (7.5 YR 4/2; 3/2, when moist) clay streaked with light reddish-brown (5YR 6/3) clay loam or light clay; strong medium and fine subangular blocky; crumbly when moist; very hard when dry; slowly permeable; pH 7.5; sides of peds weak shiny when moist; occasional pores and very find concretions of CaCO₃; grades through a two to four inch transition to the layer below.

Sample 54-OK-9-20-4

C2 32-52"+ Brown (7.5YR 4/2; 3/2, when moist) crumbly clay much like the layer above but contains a few fine concretions and many threads of CaCO₃ disseminated in the mass; pH 8.0; calcareous in seams; there are only occasional concretions in the material below about 42 inches.

Not Sampled.

52-70"+ Brown (7.5YR 4/3; 3/3 when moist) crumbly clay which is like the layer above and has few to occasional CaCO₃ concretions.

Variations: Surface soils vary in color from dark grayish-brown

to very dark gray and in texture from clay loam to heavy silty clay loam. Thickness of surface clay loam varies from six to eighteen inches in the grass nursery (due to leveling) and from six to twelve inches elsewhere. Subsurface horizons vary in color from dark grayish brown to black and in structure from subangular blocky to weak blocky. Most layers are of crumbly clays with a weak shine on the peds. Cl layers range from brown to reddish brown, while the substrata range from brown through strong brown to reddish brown in color and from clay to clay loam in texture. Subsoils have prismatic breakage in dry cuts and sides of prisms have weak dusty coverings which are probably of CaCO₃.

Profile Description of the San Saba Series

The San Saba series consists of soils that are deep, nearly black, and fine textured. In most places the surface layer is very dark, gray to black clay. It is very firm when moist and very hard when dry. In about 70 per cent of the acreage, the surface layer is 3 to 4 feet deep, but in other places where the underlying material has been pushed up, it is only about 8 inches thick.

0-18" Very dark gray (2-5Y 3/1; black 2-5Y 2/1, moist) clay; strong medium granular; crumbly and friable; very sticky and very plastic; calcareous, grades into horizon below.

18-40" Very dark gray clay; weak coarse blocky or compound coarse granular, very firm; very ticky and stiff; strongly calcareous; contains small subrounded particles of hard CaCO₃; grades into horizon below.

40-54" Olive gray (5Y 4/2; olive 4/3, moist) clay; of same structure and consistence as layer above; contains small hard particles of CaCO₂.

54"+ Hard limestone or partly weathered limestone interbedded with thin seams of marl.

TABLE XI

Vertical		Lat	eral 1	Lateral 2		
mv	Time (mins.)	mv Time (mins.)		mv	Time (mins.)	
	n haan oo too dhaa dhaa dhaa ah					
		N	aCl (1N)			
4	60	2	60	3	60	
9	120	7	120	9	120	
14	180	11	180	15	180	
22	240	12	2 40	21	240	
23	300	15	300	27	300	
24	360	16	360	29	360	
		17	420			
		NaC	1 (0.1N)			
3	60	2	60	5	60	
9	120	6	120	8	120	
13	180	11	180	14	180	
27	240	17	240	19	240	
31	300	26	300	20	300	
32	360	29	360	21	360	
		30	420			
		NaC	1 (0.01N)			
5	60	4	60	3	60	
8	120	7	120	7	120	
15	180	11	180	11	180	
19	240	17	240	14	240	
23	300	19	300	15	300	
26	360	21	360	17	360	
29	420	25	420			
		28	480			
		NaC1	(0.001N)			
3	60	3	60	6	60	
9	120	11	120	13	120	
13	180	16	180	19	180	
19	240	21	240	2.4	2.40	
21	300	24	300	30	300	
24	360	26	360	31	360	
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THREE DIMENSIONAL SWELLING RELATIONSHIPS (DATA) SAN SABA SOIL, 10-14" DEPTH, NaCl SOLUTIONS

TABLE XII

Vertical mv Time (mins.)		Lat mv	eral 1 Time (mins.)	Late mv	Lateral 2 mv Time (mins.)			
OH/A1 (1:2)								
6	45	2	15	5	60			
9	60	6	30	12	120			
15	120	9	45	18	180			
38	180	12	60	27	240			
46	240	31	120	37	300			
57	300	39	180	49	360			
65	360	47	240	57	420			
69	420	56	300	61	480			
76	480	67	360	63	540			
77	540	70	420					
		<u>OH</u>	/A1 (1:3)					
5	45	4	60	3	60			
8	60	12	120	9	120			
15	120	19	180	17	180			
34	180	39	240	30	240			
48	240	47	300	52	300			
57	300	56	360	59	360			
69	360	63	420	63	420			
72	420	65	480					
73	480							
		<u>OH</u>	/A1 (1:4)					
6	30	5	30	2	60			
11	60	11	60	11	120			
26	120	21	120	22	180			
38	180	41	180	37	240			
47	240	53	240	43	300			
56	300	61	300	51	360			
67	360	71	360	60	420			
74	420	72	420	69	480			
77	480			71	540			
		<u>OH</u>	<u>/A1 (1:5)</u>					
6	60	3	45	4	60			
9	120	7	60	8	120			
17	180	16	120	13	180			
25	240	21	180	19	240			
33	300	29	240	22	300			
36	360	30	300	24	360			
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THREE DIMENSIONAL SWELLING RELATIONSHIPS (DATA) SAN SABA SOIL, 10-14" DEPTH, OH/A1 SOLUTIONS

TABLE XIII

							
Ve	rtical	Lateral 1		Lat	Lateral 2		
mv	Time (mins.)	mv	Time (mins.)	mv	Time (mins.)		
		NaC	1 (1N)				
4	60	1	15	3	60		
12	120	11	30	9	120		
21	180	15	45	12	180		
27	240	19	60	18	240		
34	300	28	120	26	300		
42	360	32	180	29	360		
50	420	32	240	30	420		
50	480	34	300	31	480		
		34	360				
		NaC1	(0.1N)				
0	60	2	60	6	60		
12	120	8	120	15	120		
27	180	11	180	19	180		
34	240	24	240	22	240		
38	300	38	300	29	300		
43	360	39	360	32	360		
44	420	40	420	34	420		
		NaCl (0.01N)				
5	60	2	60	0	60		
8	120	9	120	5	120		
18	180	11	180	13	180		
23	240	19	240	14	240		
31	300	24	300	15	300		
32	360	26	360	16	360		
33	420	26	420				
		NaCl (0.001N)				
0	15	4	60	6	60		
6	30	13	120	8	120		
18	60	21	180	19	180		
27	120	24	240	21	240		
36	180	27	300	21	240		
49	240	28	360	23	300		
56	300						
63	360						
64	420						
	·			90 12			

THREE DIMENSIONAL SWELLING RELATIONSHIPS (DATA) DWIGHT SOIL, 3-6¹/₂" DEPTH, NaCl SOLUTIONS

TABLE XIV

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Vertical		Lateral 1		Lateral 2		
mv	Time (mins.)	mv T:	ime (mins.)	mv	Time (mins.)	
		OH/A	1 (1:2)	<u></u>		
2	30	4	45	2	30	
8	60	10	60	16	45	
16	120	15	120	20	60	
20	180	17	180	27	120	
27	240	19	240	30	180	
32	300	19	300	31	240	
39	360			31	300	
40	420			32	360	
		OH/A	1 (1:3)			
8	30	5	30	2	15	
20	45	11	45	12	30	
27	60	16	60	17	45	
39	120	28	120	21	60	
41	18 0	29	180	25	120	
43	240	29	240	26	180	
44	300	29	300	27	2 40	
				28	300	
				29	360	
		아버/ል	1 (1.4)	29	420	
				_		
5	30	0	15	0	60	
15	45	0	30	1	75	
20	60	2	45	3	90	
31	120	9	60	6	105	
36	180	18	75	9	120	
40	240	24	90	12	180	
43	300	24	120	13	240	
43	360	26	180	14	300	
45	420	28	240			
		OH/A	1 (1:5)			
4	45	1	60	0	60	
12	60	3	120	5	120	
20	120	14	135	12	180	
43	180	20	150	17	2,40	
52	240	24	165	21	300	
64	300	28	180	28	360	
68	360	34	240	29	420	
70	420	39	300			
72	48 0	41	360			

THREE DIMENSIONAL SWELLING RELATIONSHIPS (DATA) DWIGHT SOIL, 3-6¹/₂" DEPTH, OH/A1 SOLUTIONS

V	Vertical	····	Lateral 1	-Lateral 2
mv	Time (mins.)	mv	Time (mins.) mv Time (mins.)
		43	420	•
		44	480	
	•	•	-	

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TABLE XV

	Vertical	Lat	eral l	Lateral 2		
mv	Time (mins.)	mv	Time (mins.)	mv	Time (mins.)	
		NaC1	(1N)			
~	(A		(11)	_	<i>(</i>)	
0	60	0	60	1	60	
4	120	3	120	3	120	
7	180	8	- 180	5	180	
9	240	10	240	7	240	
11	300	13	300	9	300	
13	360					
		NaC1	(0.1N)			
2	60	2	60	1	60	
5	120	4	120	4	120	
8	180	8	180	7	180	
9	240	10	240	9	240	
11	300	11	300			
14	360	12	360			
		NaC1	(0.01N)			
4	60	3	60	2	60	
7	120	7	120	6	120	
11	180	12	180	ŷ	180	
17	240	15	240	11	240	
21	300	17	300	12	300	
23	360	18	360	14	500	
24	420	10	200			
		NaC1	(0.001N)			
		<u>Madi</u>	(0.00111)			
2	60	2	60	1	60	
3	120	2	120	1	120	
5	180	5	180	2	180	
7	240	6	240	2	240	
9	300	6	300			
11	360	7	360			

THREE DIMENSIONAL SWELLING RELATIONSHIPS (DATA) DWIGHT SOIL, 9-14" DEPTH, NaCl SOLUTIONS

TABLE XVI

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46 300 45 360 35 360								
48 360 49 420 36 420								
48 360 50 480 37 480								
50 420								
OH/A1 (1:5)								
2 60 4 60 1 60								
11 120 9 120 7 120								
19 180 16 180 11 180								
26 240 21 240 <u>13</u> <u>240</u>								
31 300 23 300 15 300								
31. 360 23 360 <u>16</u> 360								
33 420 25 420								

THREE DIMENSIONAL SWELLING RELATIONSHIPS (DATA) BREWER SOIL, 12-16" DEPTH, OH/A1 SOLUTIONS

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Rl is adjusted to give desirable swing of 100 mv on Rustrak meter. RlA is then adjusted to give variations to original settings; i.e. original reading can be divided to allow for extensive swelling of material under test.

Figure 27. Electronic Soil Expansion Measuring Device

Depths (cm)	1	2	3	4	5	6
<mark>е — пер</mark> ия и и история на селото и странити. И		DWIG	HT	<u></u>		
0.0 - 7.6	460			300	300	300
7.6 - 16.5	500	340	320	330	320	300
16.5 - 22.9	390					
22.9 - 35.6	420	400				320
35.6 - 49.5	440	430	300	300	320	
49.5 - 63.5	570	380	340	370		
63.5 - 73.7	430	350	300	380		320
		MILL	ER			
20.3 - 30.5	920	860	7 40	720	660	420
30,5 - 40,6	880	740	765	660	740	525
40.6 - 58.4	880	765	765	765	720	720
48.3 - 58.4	860	860	740	765	685	820
58.4 89.0	880	820	920	765	685	660
91.4 - 121.9	740	820	660	720	685	740
121.9 - 152.4	800	720	720	740	740	685
		SAN SA	BA			
0.0 - 15.2	550	460	525	420	350	340
25.4 - 35.6	525	300	340	445	370	300
50.8 - 61.0	445	390	440		320	320
63.5 - 76.2	510	380	460	400	340	330
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TABLE XVII

Na-CITRATE EXTRACTABLE A1-COMPOUNDS (ppm) 6 EXTRACTIONS

VITA

Sedley Athlestan Williams

Candidate for the Degree of

Doctor of Philosophy

Thesis: SHRINK-SWELL CHARACTERISTICS OF SOIL: RESPONSE TO VARYING CONCENTRATIONS OF NaCl and OH/Al RATIOS

Major Field: Soil Science

Biographical:

- Personal Data: Born at St. Margaret's Bay in the Parish of Portland, Jamaica, June 22, 1937, the son of Mr. and Mrs. Leonard L. Williams.
- Education: Graduated from the Jamaica School of Agriculture June 1957; received the Bachelor of Science degree from North Carolina Agricultural and Technical State University, 1964, with a major in Agronomy; received the Master of Science degree from Tuskegee Institute with a major in Plant Science; completed the requirements for the Doctor of Philosophy Degree in July, 1972.
- Experience: Chemical Analyst, Sucrest Sugar Co., N. Y., 1965-1967; Graduate assistant, Tuskegee Institute, 1967-1969; Oklahoma State University, Agronomy Department, 1969-1972.
- Professional Organizations: American Society of Agronomy; Soil Science Society of America; International Soil Science Society.