

A COMPARATIVE ENGINEERING STUDY OF THE HYDRATED (4H₂O)
AND DEHYDRATED (2H₂O) FORMS OF HALLOYSITE

by

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24 May 1954

Professor L. F. Hamilton
Secretary of the Faculty
Massachusetts Institute of Technology
Cambridge, Massachusetts

Dear Sir:

In partial fulfillment of the requirements for the degree of Master of Science in Civil Engineering at the Massachusetts Institute of Technology, we submit herewith a thesis entitled, "A Comparative Engineering Study of the Hydrated ($4H_2O$) and Dehydrated ($2H_2O$) Forms of Halloysite".

Yours very respectfully,

Thomas T. Jones

John S. Egbert

ACKNOWLEDGMENT

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The authors also wish to thank the personnel of the Arctic Frost Effects Laboratory of the New England District of the Corps of Engineers, for conducting the frost tests used in this investigation.

A COMPARATIVE ENGINEERING STUDY OF THE HYDRATED (4H₂O)
AND DEHYDRATED (2H₂O) FORMS OF HALLOYSITE

Abstract

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Submitted to the Department of Civil and Sanitary Engineering on 24 May, 1954, in partial fulfillment of the requirements for the degree of Master of Science.

The existence of the clay mineral halloysite in a hydrated and dehydrated form has been recognized by clay mineralogists for about twenty years. During this period, research has established certain facts of interest to the engineer. These facts are as follows:

1. Under certain conditions the hydrated form of halloysite will dehydrate irreversibly to the dehydrated or 2H₂O form.
2. Dehydration will occur at a temperature of 105°C.
3. Partial dehydration will result from air drying hydrated halloysite.

Halloysite has been encountered on several engineering projects. On such occasions its unusual engineering characteristics presented problems which required additional time and money to solve. A knowledge of halloysite on such occasions would probably have obviated most difficulties.

The objective of this investigation is to consider the engineering problems posed by halloysite and to provide needed information relative to the comparative engineering properties of the hydrated (4H₂O) and dehydrated (2H₂O) forms.

Hydrated halloysite was subjected to a series of drying tests and both hydrated and dehydrated were given routine engineering tests from which the following conclusions are drawn:

1. Hydrated halloysite will dehydrate to a partially dehydrated form under certain field conditions. If dehydration should occur the engineer must be prepared for the change in soil behavior.
2. Certain routine laboratory tests will indicate the possible presence of halloysite. These indications should suggest a complete mineral analysis for positive identification.
3. Certain routine laboratory tests must be corrected to obtain accurate results. The correction is necessary to account for the interlayer water lost in heating hydrated halloysite to 105°C.
4. Each form of halloysite has certain engineering advantages. Conclusion (1) may be utilized by the engineer working with hydrated halloysite to gain more desirable soil behavior.

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I. Introduction

A. Objectives

The objectives of this study are twofold:

1. To determine and compare the engineering properties of the clay mineral, halloysite, in its hydrated or unstable form, and in its dehydrated or stable form.
2. Based upon a comparison of the engineering properties, to draw conclusions which will aid engineers who must work with similar soils under field conditions.

B. Background

1. Definitions

In the literature, several different terms are associated with the structural composition of halloysite; it is therefore necessary to define the terms used throughout this research paper. Most of the terms used are identical to those recommended by Grim. (1953)

a. Halloysite

This name identifies the mineral as a class to differentiate it from the other sub-divisions of the kaolinite group.

b. Hydrated Halloysite

The chemical formula for this form is $Al_2O_3 \cdot 2SiO_2(kH_2O)$ when (k) has an average value of about 4 and the x-ray basal spacing is 10.1 Angstroms. (Brindley, et al., 1948) This will be referred to as the hydrated or

or $4\text{H}_2\text{O}$ form. In some publications, this form is referred to as endellite or halloysite.

c. Dehydrated Halloysite

The chemical formula for this form is $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2(k\text{H}_2\text{O})$ when (k) has an average value of about 2 and the x-ray basal spacing is 7.2 Angstroms. This will be referred to as the dehydrated or $2\text{H}_2\text{O}$ form. Some publications refer to this form as metahalloysite.

d. Partially Dehydrated Halloysite

This form occurs between the two limits of hydrated and dehydrated halloysite. The coefficient (k) as noted in the chemical formulas above, does not occur in the range of 2.7 to 3.5 since the interlayer water seems to emerge so fast from 3.5 to 2.7 that no measurements can be made. (Bates, et al, 1950, and Brindley, et al, 1948)

e. Interlayer Water

This refers to the layers of water molecules held in chemical bond between the silica sheets as indicated by the above formulas for both forms of halloysite.

f. Adsorbed Water

This is non-pore water that is attached to the soil grain and actually contributes to the soil grain size. It is differentiated from the free or pore-water which can move through the soil under application of hydrostatic pressure. The application of heat can, of course, remove the adsorbed water. (Lambe, Dec. 1949)

g. Tubular Water

This is the water held by capillary forces within the completely closed tubes of hydrated halloysite.

2. History

Halloysite was first discovered near Liege, Belgium, in 1826 and was named in honor of Omalius D'Halloy. Practically all of the published results to date have been from experiments of a very technical nature conducted by mineralogists investigating the chemical composition, the dehydration process in relation to basal spacings, and the structural arrangement of the molecular layers of both forms of halloysite. The known and generally accepted conclusions obtained from these experiments are summarized below.

a. Hydrated halloysite is developed in nature by the weathering of plagioclase feldspars in a slightly acid environment. (Bates, et al, 1950 and Lambe, 1950, #9)

b. The hydrated form is composed of overlapping curved sheets of the kaolin type, i.e. silica sheets, with molecular sheets of water between the silica sheets, forming cylindrical tubes. (Bates, et al, 1950)

c. The dehydration process which changes the $4\text{H}_2\text{O}$ form is non-reversible. (Hendricks, 1938)

d. The hydrated halloysite has been observed to undergo partial dehydration by air drying for one week and also by being heated in an oven at 50°C . for 24 hours. (Hendricks, 1938 and Mehmel, 1935)

e. The value of (k) in the formula previously given for $4\text{H}_2\text{O}$ actually varies between 3.5 and 4.5, with 4

used as an average value. (Grim, 1953)

f. When air dried, the value of (k) in the formula for dehydrated halloysite varies from 2.2 to 2.4. This means that the dehydrated form retains about 1 water layer per 6 silicate layers when air dried. (Brindley, et al, 1948)

g. Electron micrographs show that the dehydrated form exists either as ~~beam~~-collapsed, split, or partially unrolled tubes, the latter being similar to a spatula in shape. The above shapes of $2H_2O$ are caused by strains within the tubular grains that are induced by the dehydration process. Double concentric tubes have also been observed, with an "empty" space apparent between the inner and outer tubes. (Bates, et al, 1950)

h. The average outside diameter of the hydrated tubular crystals is 0.07 microns and the average inside diameter is 0.04 microns. (Bates, et al, 1950)

i. The difference in the basal spacings already noted for the two forms is 2.9 Angstroms which is the width of a single molecular sheet of water. (Brindley, et al, 1948)

j. Partial dehydration will occur at a pressure of 5000 Kg/cm² acting over a period of about 12 hours and such high pressures could contribute to the dehydration process in nature. (Brindley, et al, 1948)

k. There is a great disorderliness in the arrangement of the molecular layers, but a partial orientation was noticed in the $2H_2O$ form when subjected to high pressures.

Heating this form to 450°C. does not change the disorderliness of the layers. (Hendricks, S.B., 1938 and Brindley, et al, 1948)

Very little appears to be known about the engineering properties of the two forms of halloysite. Grim (1953) states that the value of specific gravity for pure $4H_2O$ is 2.0 to 2.2 and the specific gravity of $2H_2O$ is 2.55. Lambe (1952) states that the maximum dry density is about 70 lbs/cu. ft. and trouble in compacting and working the soil should be expected. The remainder of the engineering data found available is contained in a series of private communications (Lambe, T.W., #9) regarding the Sasumua dam. However, the latter data does not differentiate between the two forms of halloysite.

C. Statement of the Problem

As already noted, an outstanding feature of the previous work with halloysite is the absence of research and study to determine those characteristics which would be of practical value to the engineer.

In 1950, engineers working on the Sasumua dam in Kenya, Africa, encountered a soil whose unusual characteristics presented serious problems. The soil being used in the dam construction was not known to consist mainly of hydrated halloysite until an extensive testing program positively identified it. On this occasion, so little was known about halloysite in either form, that much time and money was spent in gaining enough knowledge and confidence in the soil's behavior to continue construction.

This experience illustrates the problems which in the future will confront other engineers. An investigation

designed to solve such problems must seek to provide reasonable answers to the following questions:

1. Can routine laboratory test results indicate to the engineer the possible presence of hydrated or dehydrated halloysite in significant quantities?

2. Recognizing the presence of hydrated halloysite, what precautions, if any, should be observed in interpreting test data and handling the soil in the field?

3. What are the relative engineering characteristics of two soils, one containing hydrated halloysite in large quantities and one containing a large fraction of dehydrated halloysite?

4. Can the engineer take advantage of the instability of hydrated halloysite in order to utilize the possible superior engineering characteristics of dehydrated halloysite?

II. Discussion of Theory

Previous investigations (see background) have determined that hydrated halloysite would break down to dehydrated halloysite when heated to 105°C. Knowing this, it is readily apparent that routine water content determinations will give results quite different from those which would be obtained if the hydrated halloysite retained its interlayer water. This presents the problem of determining a correction factor which might present test results in a form consistent with the known structural makeup of hydrated halloysite. Such a correction factor could be obtained by determining the interlayer water driven off at 105°C.

Expressed in equation form:

$$w_c = \frac{Ww - W_{iw}}{W_s + W_{iw}} \text{ where } w_c = \text{corrected water content}$$

$W_{iw} =$ interlayer water lost at 105° C.

$Ww =$ wt. water driven off at 105° C.

$W_s =$ wt. solids at 105° C.

This expression is easily put in more workable form with W_{iw} as a fraction of W_s . Let $\frac{W_{iw}}{W_s} = C$ and the expression becomes:

$$w_c = \frac{W_s - CW_s}{W_s + CW_s} = \frac{Ww/W_s - C}{1 + C} = \frac{w - C}{1 + C} \text{ where } w = \text{uncorrected water content as normally defined (expressed as a decimal)}$$

It follows that experimentation should seek to determine C , in order to correct normal moisture content determinations.

Assuming a soil is composed exclusively of hydrated halloysite, C may be determined by considering the known chemical compositions of hydrated and dehydrated halloysite as follows:

1. $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}^*$ = dehydrated halloysite.
2. $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 4\text{H}_2\text{O}$ = hydrated halloysite.
3. Molecular weight of dehydrated halloysite

Al_2	$= 2 \times 26.97$	$=$	53.94
90	$= 9 \times 16.00$	$=$	144.00
2Si	$= 2 \times 28.06$	$=$	56.12
4H	$= 4 \times 1.008$	$=$	4.03
	Total	$=$	258.09

4. Molecular wt. of hydrated halloysite

$$258.09 + 2\text{H}_2\text{O} = 258.09 + 36.03 = 294.12$$

* Brindley and Goodyear, et al, 1948, have shown that interlayer water is substantially but not completely removed below 400° C. The interlayer water lost above 100° C. however, is very small and hence the value given is correct enough for practical purposes.

$$5. C = \frac{W_{iw}}{W_s} = \frac{36.03}{258.09} = .139 \text{ or } .14$$

If the hydrated halloysite fraction of any soil being considered is known, the above value of C would be modified by multiplying it by the known fraction. For example, if hydrated halloysite comprises 80% of the soil, C becomes $(.80)(.1394) = .11$.

III. Experimental Procedure

A. Preparation of Soil for Testing

The soil used for testing was prepared from five sub-samples sent from the Sasumua dam in Kenya, East Africa, in 1952. The sub-samples were taken from the four borrow pits and the treatment works site. All sub-samples were tested by x-ray diffraction to (1) insure that the halloysite in the soil was of the hydrated form and (2) to identify samples which contained the most hydrated halloysite. The x-ray patterns were obtained with an air evacuated power camera of 114.6mm diameter and unfiltered chromium radiation. Treatments used in conjunction with the x-rays for positive identification of mineral species were those of Brindley (1951). The data from all x-ray diffraction tests are tabulated in Annex A.

Those samples which indicated the highest hydrated halloysite fraction were mixed to obtain the soil on which all tests were performed.

Dehydrated halloysite samples were obtained by heating hydrated halloysite samples to constant weight at 105°C.

B. Determination of the Hydrated Halloysite Fraction

The hydrated halloysite fraction was required to obtain a value for the correction factor (C). This value would serve as a check on any other correction factor determined from drying tests. It was felt that in the event no positive results could be obtained from the drying, the correction factor determined by this method would furnish the most accurate determination of the amount of interlayer water lost from hydrated halloysite at 105°C.

A hydrated halloysite sample was first sieved through a #60 and a #200 sieve. The two fractions obtained (all soil was passed through the #60 sieve) were then tested by Differential Thermal Analysis, employing the procedure set forth by Lambe (#9, 1951). The reference thermogram (API #H-12) shown in Annex B, was from Bedford, Indiana halloysite.

Using the results of the D.T.A. and the fractions retained on and passing the #200 sieve, the percent of hydrated halloysite in the sample was computed. (Annex B)

C. Drying Tests

This series of tests was designed to determine experimentally the correction factor for interlayer water. Simultaneously, the tests were expected to give an indication of the structural stability of hydrated halloysite under possible field conditions.

The tests consisted of observing prepared samples under various conditions of temperature and relative humidity. At various time intervals, a sample was weighed and x-ray diffraction tests were performed on a second sample undergoing

the same test. All test conditions are self explanatory. Fifty percent relative humidity was obtained in a desiccator containing a saturated solution of $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ in contact with an excess of salt. The x-ray diffraction test was used to determine if the tested hydrated halloysite was breaking down to the dehydrated form. Samples were heated to equilibrium at 105°C . at the end of each test in order to express the water remaining in the sample as a fraction or percentage of the dry weight (W_g) at 105°C .

D. Standard Engineering Tests

The following tests were selected as routine laboratory tests which, within the limits of time and soil availability, would yield a reasonably complete picture of the engineering characteristics of the two soils tested. The procedures recommended by Lambe (1951) were followed whenever possible; exceptions to this general rule have been noted.

1. Specific gravity
2. Atterberg Limits

The liquid limit device used was of the wood base type.

3. Grain size analysis

The combined type of analysis was used for both forms of halloysite. The outlined procedure was modified by separating the samples into those fractions which passed or were retained on the #200 sieve, by the test described in Section A.

Because of the large percentage of clay size

particles, both samples were washed through the #200 sieve for several minutes to insure as little error as possible.

4. Compaction

The static testing procedure outlined by Wilson (1950) was followed. The compactive effort was twenty five blows per layer in three layers, using a 40 lb. load per blow. All prepared samples were aged 24 hours at prepared water contents before compaction.

5. Unconfined compression

Samples used were those extruded from the Wilson Miniature compaction device. The apparatus illustrated in Figure XII - 4, reference (17) was used.

6. Permeability

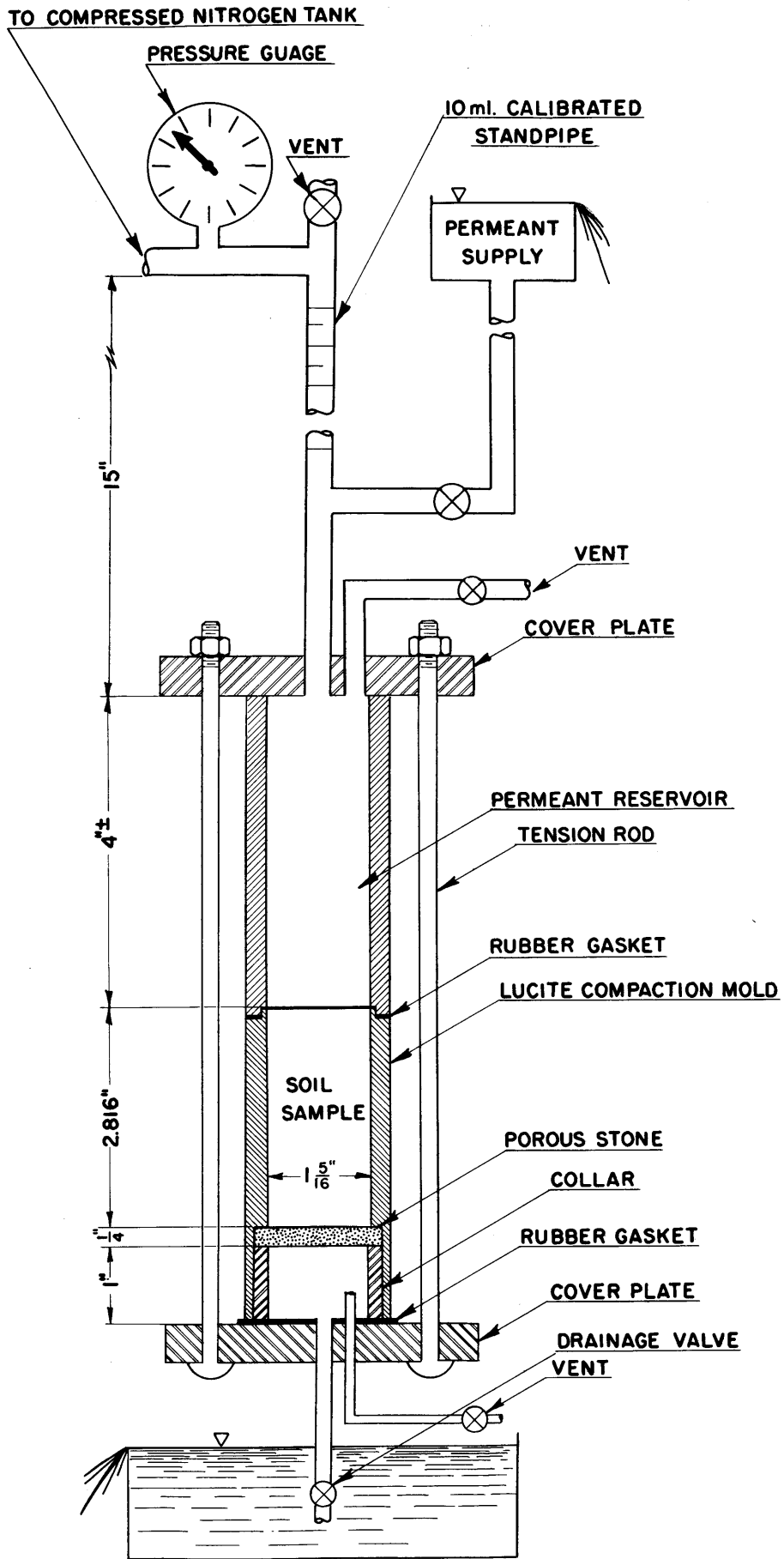
Permeability tests were performed on the constant head permeability apparatus illustrated in Figure 1. (Lambe, 1951, 12) The soil was first compacted in the sample mold using the compactive effort described for the compaction tests. The mold was then mounted below the permeant reservoir and the following steps performed in order.

a. Water was drawn from the tailwater up against the bottom porous stone while the vent and drainage valve were closed.

b. The permeant reservoir and standpipe were filled with water.

c. The air vent at the top of the standpipe was closed.

d. Pressure was applied to the system using compressed nitrogen. For all the tests, a pressure of 10 psi



PERMEABILITY TEST SET-UP

FIG. 2*1

was applied.

e. Time was allowed for the water to penetrate the sample.

f. The system was checked for leakage and the drainage vent opened.

During the progress of each test, two sets of readings were taken, the first as a check on the second. The second readings were used for the computation of permeability.

7. Consolidation

Load increments in each test were those suggested by Lambe. (1951, 11) A fixed ring type container with a diameter of approximately 4.3 inches was used exclusively. The square root of time fitting method was employed. (Taylor, 1948)

E. Frost Susceptability Tests

The Frost Laboratory of the New England Division, U.S. Corps of Engineers, performed a frost susceptability test on both forms of the soil being tested.

The adapted procedure is set forth by the Frost Laboratory as follows:

1. Test Procedures

The two forms of halloysite ($2H_2O$ and $4H_2O$) were prepared for freezing tests in 3.915-inch inside diameter lucite cylinders to a 4.84-inch and 4.89-inch height, respectively. The inside walls of the cylinders were lined with a liner of 0.007-inch thick sheet cellulose acetate and the surfaces of the acetate were lubricated with silicone.*

*Silicone is a non-melting, translucent material that retains the consistency of petroleum jelly at temperatures ranging from $-40^{\circ}F.$ to over $+400^{\circ}F.$ It is heat stable, oxidation resistant, inert to metals, plastics and most organic materials, and has

A receptacle, composed of filter paper, a 3/8-inch thick porous stone, and a brass cap with a 3/8-inch nipple, was fastened to the base of the lucite cylinders and sealed against air and water leakage with glyptol. The samples were molded at approximately optimum water content by means of the Standard Proctor Density method as described in ASTM Standard Designation D698-42T. A receptacle, as described above, was then fitted to the top of the cylinders using rubber sleeves and bends to seal against leakage. The specimens were next evacuated from the top and bottom and saturated from the bottom with de-aired water in the cold room at a temperature of -35°F . to -38°F . The degree of saturation for each sample was computed from weights of sample and container before and after saturation. Thermocouples were inserted at one-inch intervals along the longitudinal axis of the samples, including top and bottom, to observe temperatures within the samples and the progress of freezing temperature. The thermocouples were inserted through the side of the specimen and the entrance points were sealed with sealing wax.

After preparation, the samples were placed in a freezing cabinet, and granulated cork was placed around the sides for the full height of the samples. The top receptacle was removed from the samples, and the bottom receptacles were connected to a constant water level device which maintained a free water surface approximately 1/8-inch above the porous stone at the bottom of the samples. The samples were allowed

other useful characteristics, i.e., waterproof and water repellent.

to temper in the freezing cabinet until all thermocouples indicated a temperature of -35°F .

The samples were frozen by lowering the temperature in the test cabinet to -20°F . and artificially instigating crystallization on the surface of the samples by seeding with ice crystals. The cabinet temperature was measured by means of a thermocouple inserted in a glycerin-filled glass vial, $3/8$ -inch in diameter and $1-1/2$ -inches long, suspended near the top of the samples. At this time, steel base plates and lead weights with a surcharge intensity of 0.5 psi (previously tempered to 28°F .) were placed on top of the samples, and the cabinet temperature was raised to -28°F . The specimens were then frozen from the top by gradually decreasing the temperature in the freezing cabinet while the bottoms of the samples were exposed to the cold room temperature which was maintained between -35°F . and -38°F . Temperatures within the soil samples were read by means of the thermocouples, and the cabinet temperature was adjusted to maintain a rate of penetration of the 32°F . temperature into the samples at $1/4$ -inch per day. Heave measurements were taken daily throughout the freezing test with a meter stick placed on a designated point on the surcharge weights over the samples.

Upon completion of the test, the samples were removed from the freezing cabinet, measured, split longitudinally, examined for ice segregation and broken up to determine the water content distribution.

2. Frost Susceptibility Classification

The freezing tests performed in the manner described

above are not intended to simulate average field conditions, but are intended to determine the intensity of ice segregation in soils under extremely severe field conditions in which an unlimited supply of water is available to the soil during the freezing process. The following tentative scale for classification of the degree of frost susceptibility of soils tested by this procedure, based on average rate of heave, has been adopted for rates of freezing between 1/4 and 3/4 inches per day:

<u>Average Rate of Heave</u> <u>mm/day</u>	<u>Frost Susceptibility</u> <u>Classification</u>
0 - 0.5	Negligible
0.5 - 1.0	Very Low
1.0 - 2.0	Low
2.0 - 4.0	Medium
4.0 - 8.0	High
8.0	Very High

3. Test Results

The results of the freezing tests performed on the two forms of Halloysite soil are summarized in Annex K. The detailed heave data, the penetration of the 32°F. temperature and test cabinet temperature, plotted versus time, are presented on Figure #8. Photographs of the test specimens after freezing are Figures #6 and 7. The water content distribution and observations from examination of each test specimen after freezing are given in Annex K.

IV. Presentation of Results

The results obtained from all phases of testing are set forth in Table 1 and Figures 2 through 8 which follow.

Table #1

SUMMARY OF RESULTS

Specific Gravity

(2H ₂ O) Mixture	2H ₂ O	4H ₂ O		4H ₂ O	Passing #200 Sieve	
	Passing #200 Sieve	C = 0	C=0.10	C = 0	C=0.10	C=0.10
2.95	2.81	3.03	2.55	3.07	2.48	

ATTERBURG LIMITS

	2H ₂ O	4H ₂ O	
		C = 0	C=0.10
Liquid Limit	57.7	70.4	54.6
Plastic Limit	47.3	55.7	41.6
Plasticity Index	10.4	14.7	13.0

RESULTS OF TESTS ON HYDRATED HALLOYSITE TO DETERMINE INTERLAYER WATER AND STRUCTURAL STABILITY

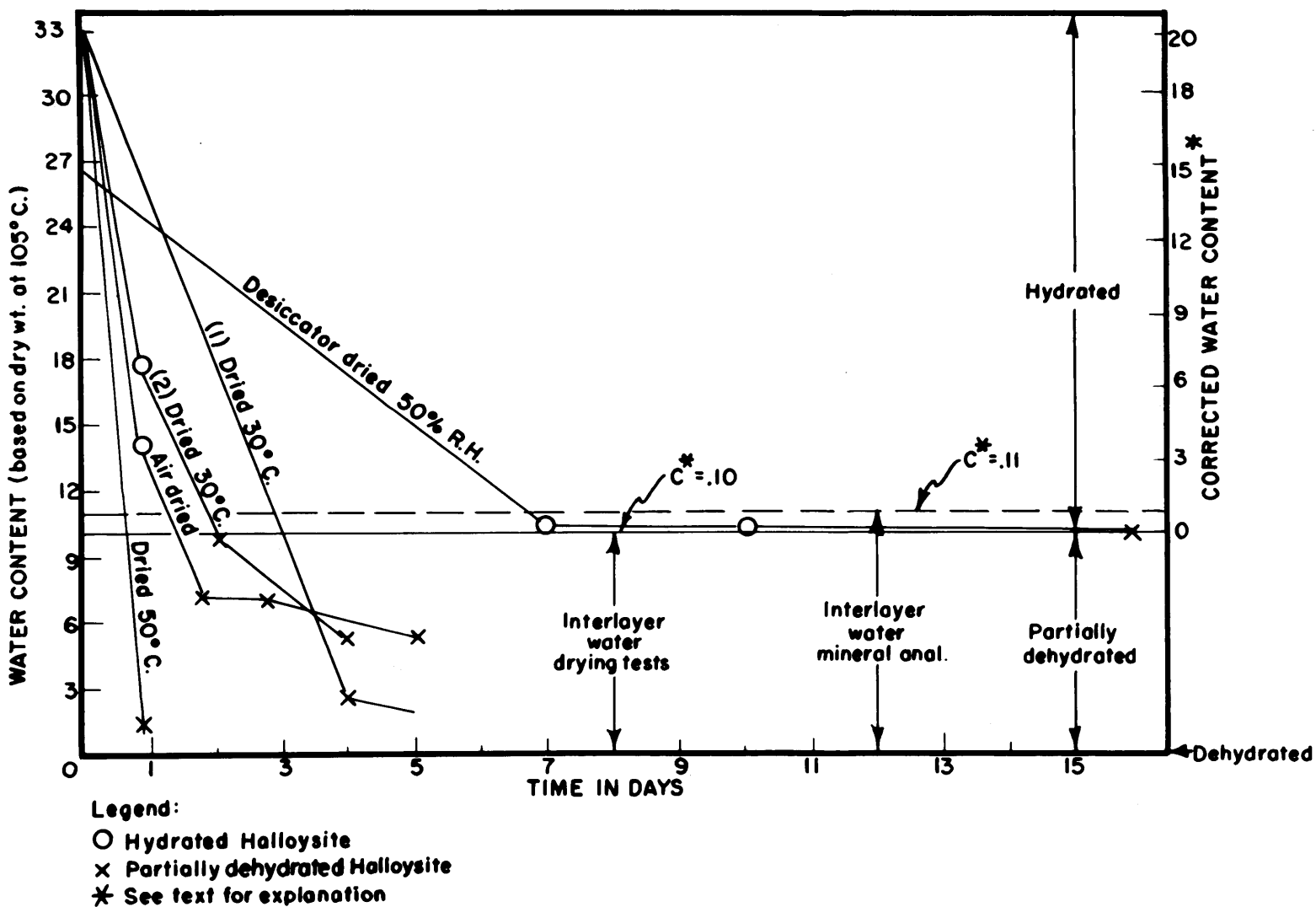


Fig. #2

-15b-

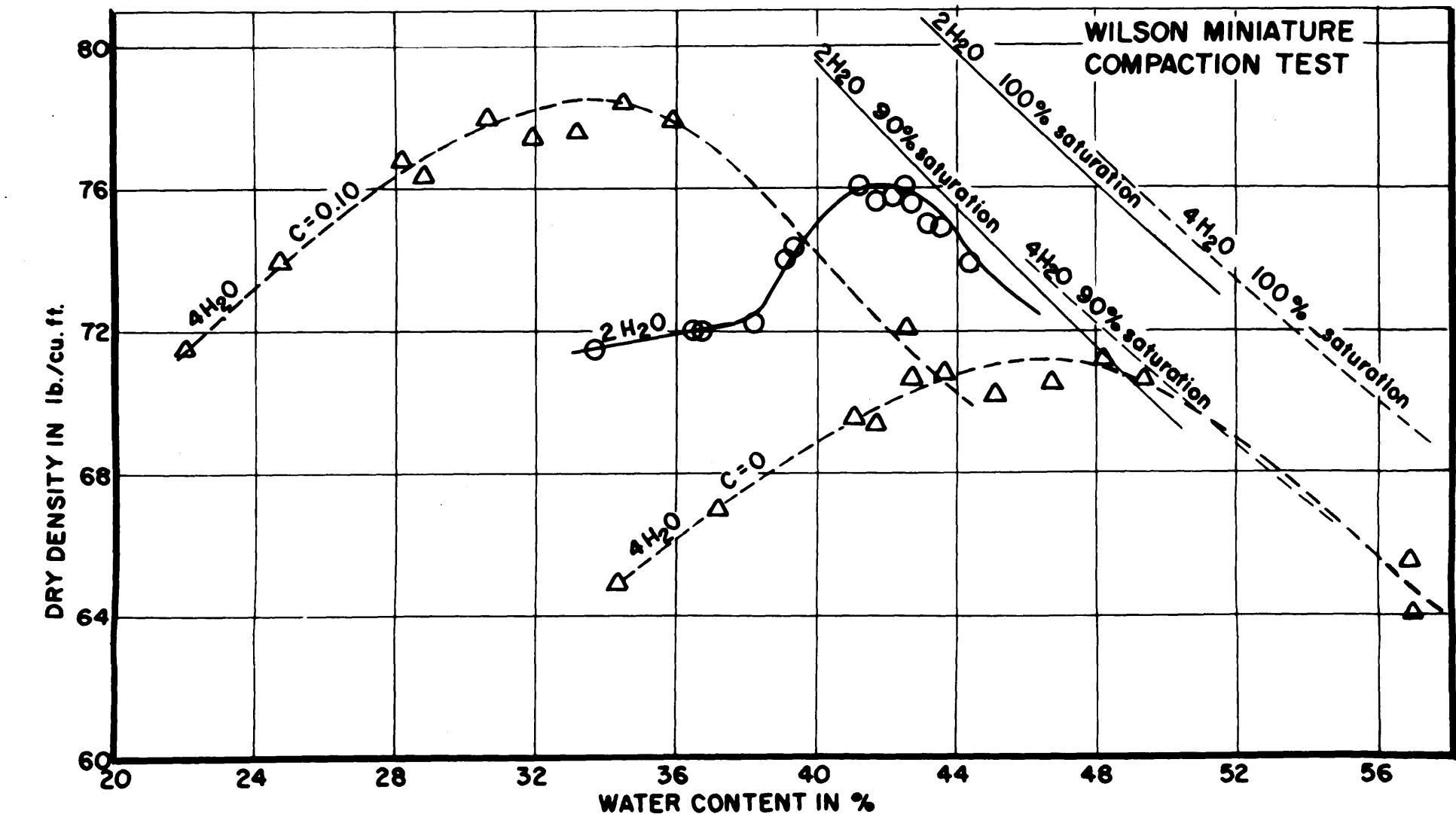
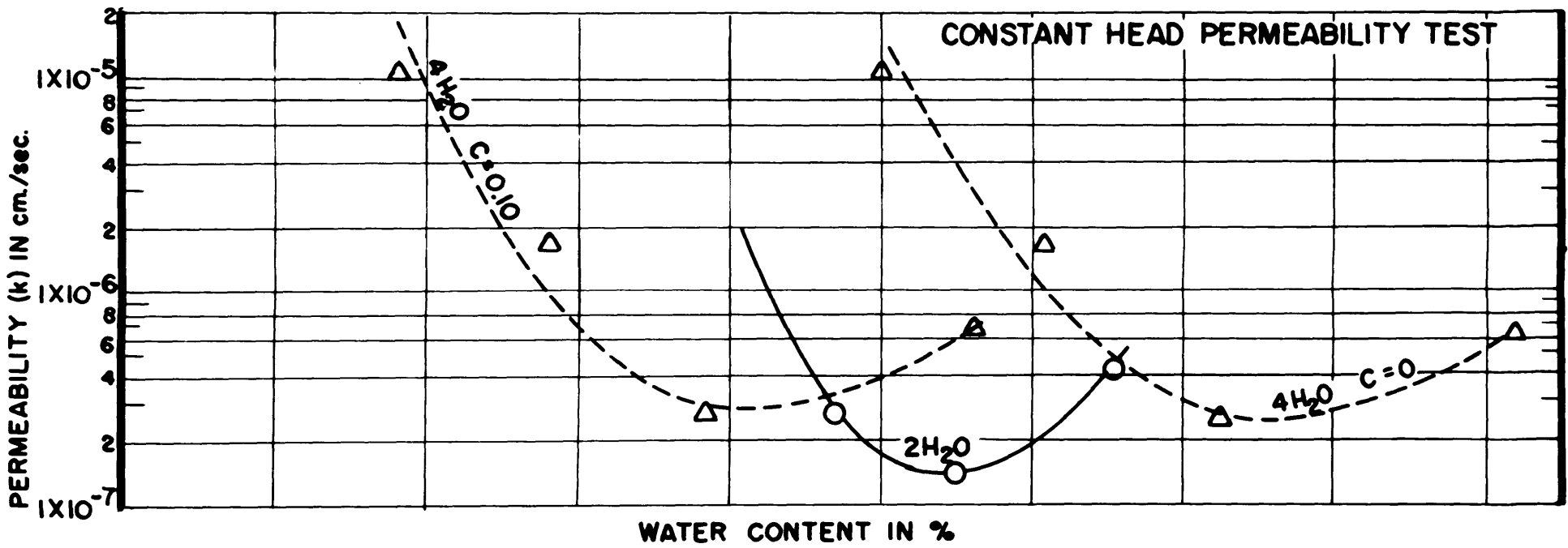
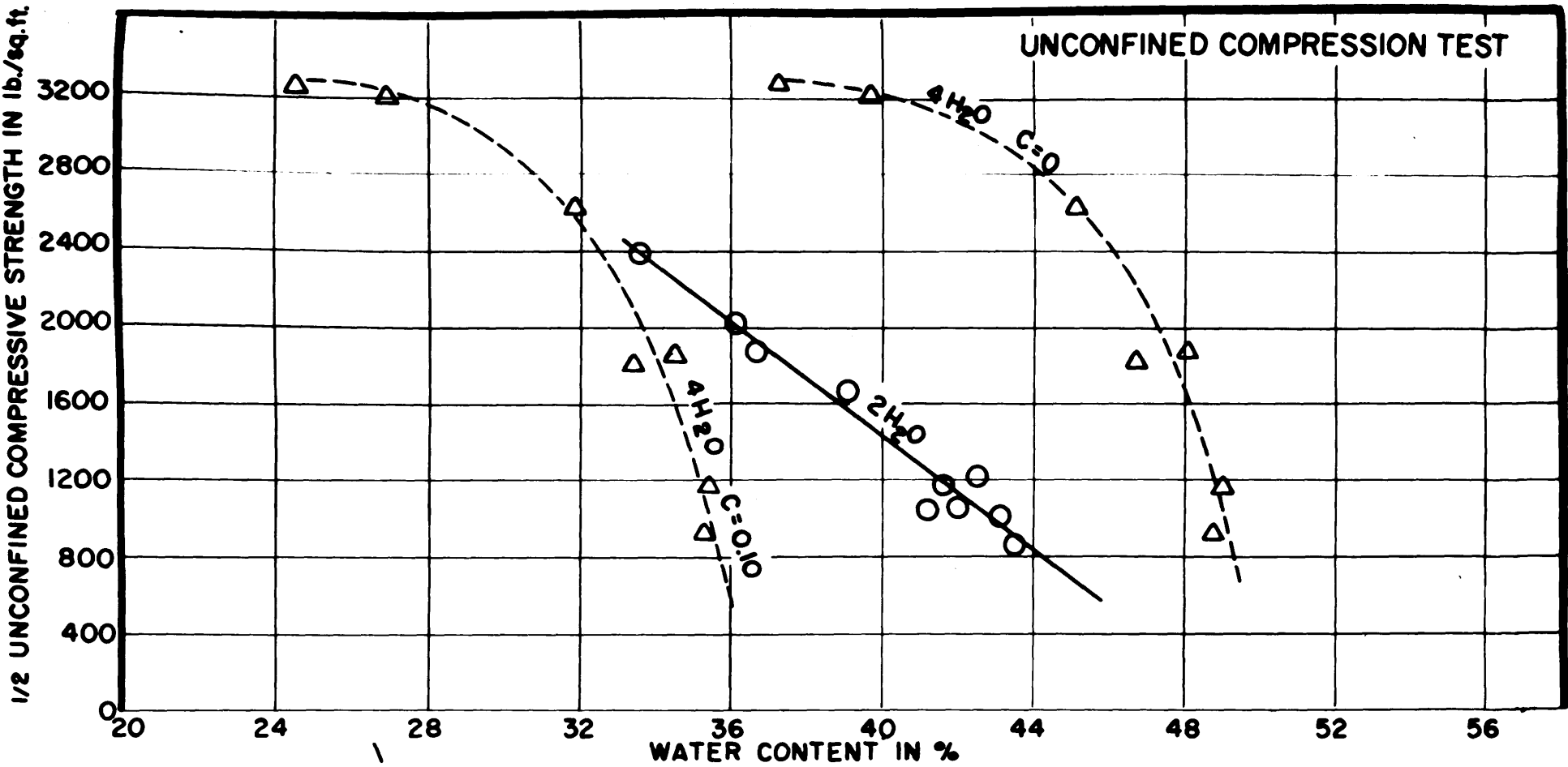
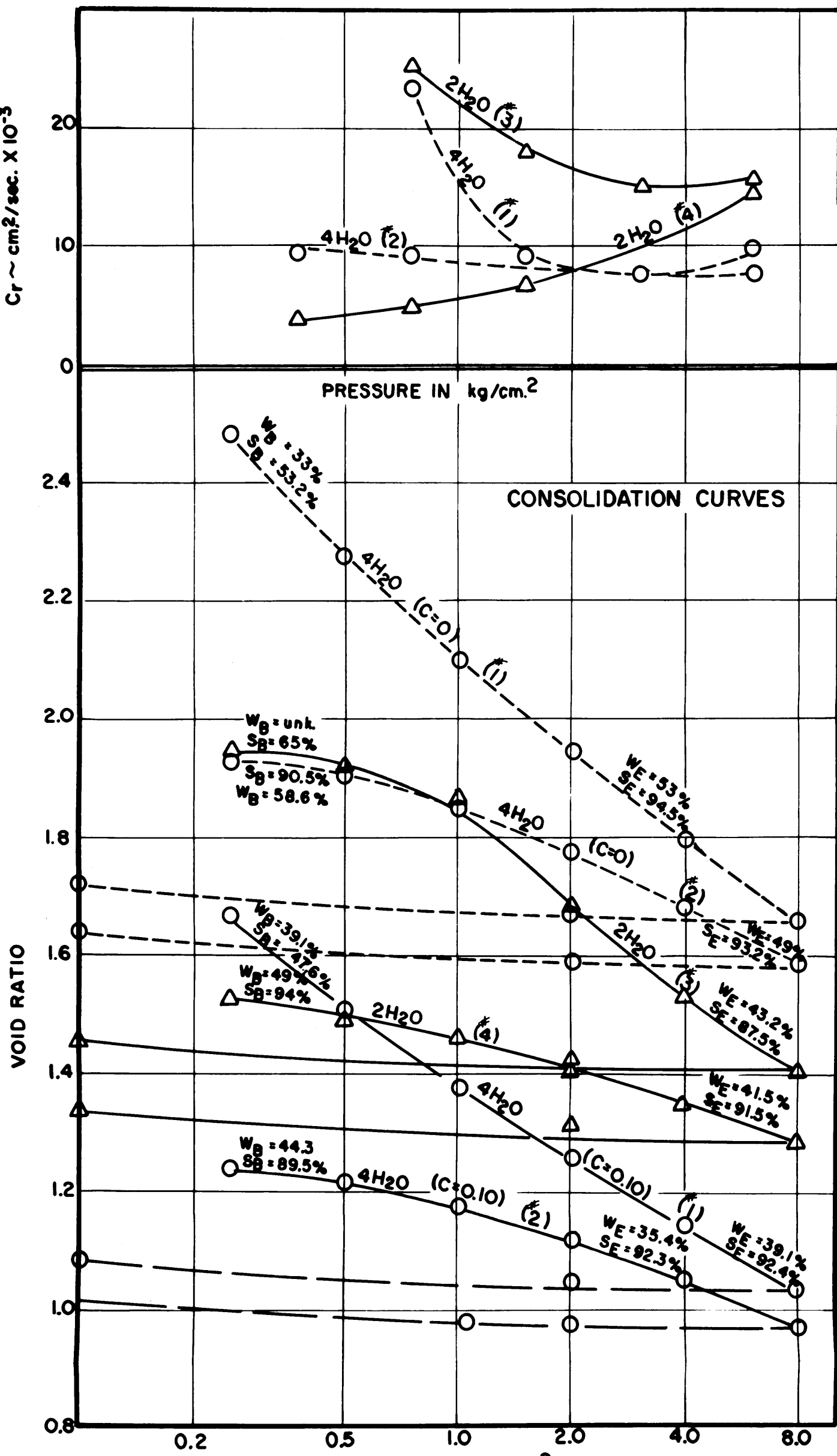


Fig. #3

COEFFICIENT OF CONSOLIDATION
 $C_r \sim \text{cm}^2/\text{sec.} \times 10^{-3}$

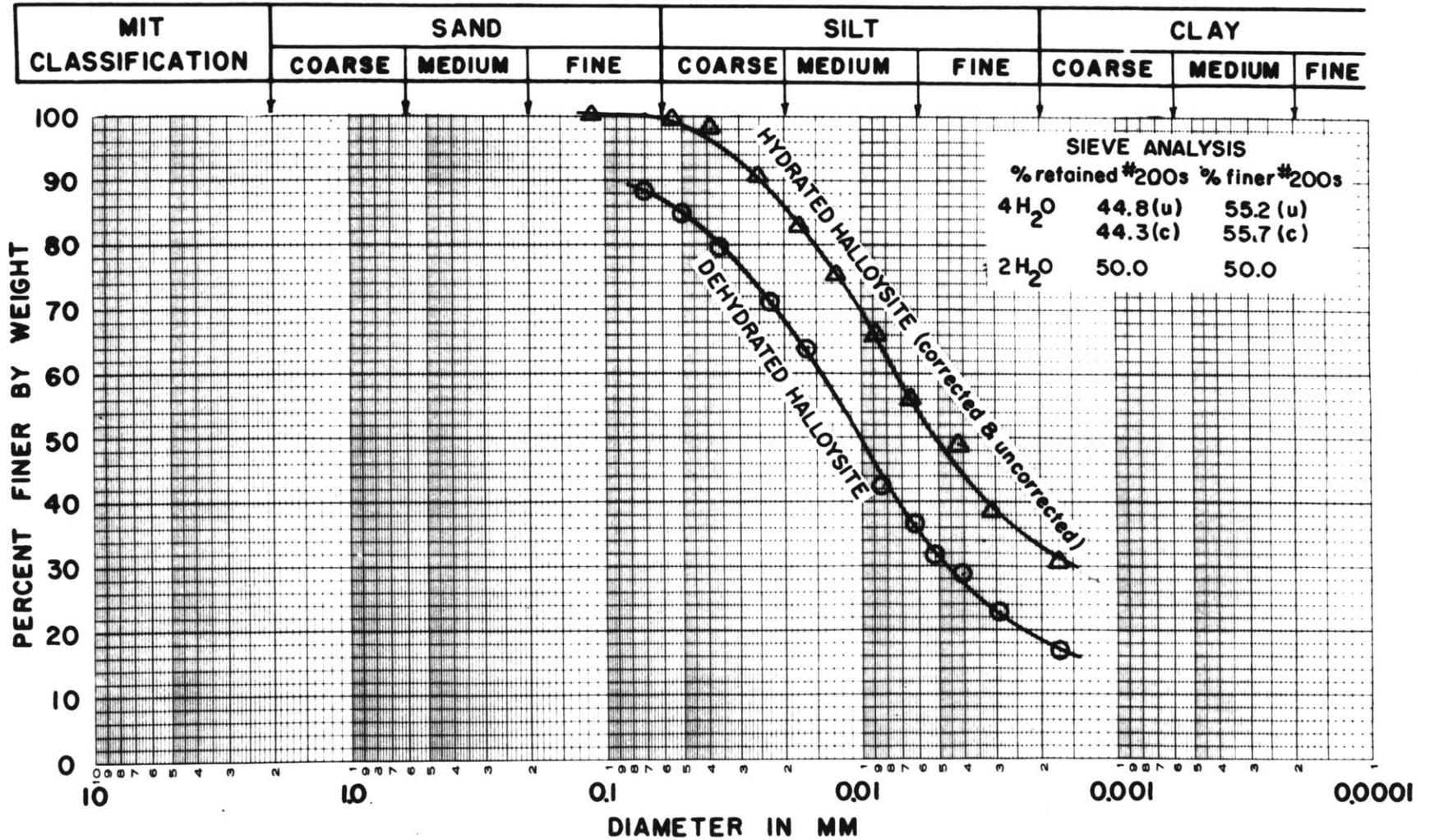


LEGEND:
 S_B - % saturation at start
 S_E - " " " end
 W_B - water content at start
 W_E - " " " end

The curves in both graphs are numbered 1 to 4 for cross reference.

Fig. #4

GRAIN SIZE DISTRIBUTION FRACTION PASSING #200 SIEVE



154

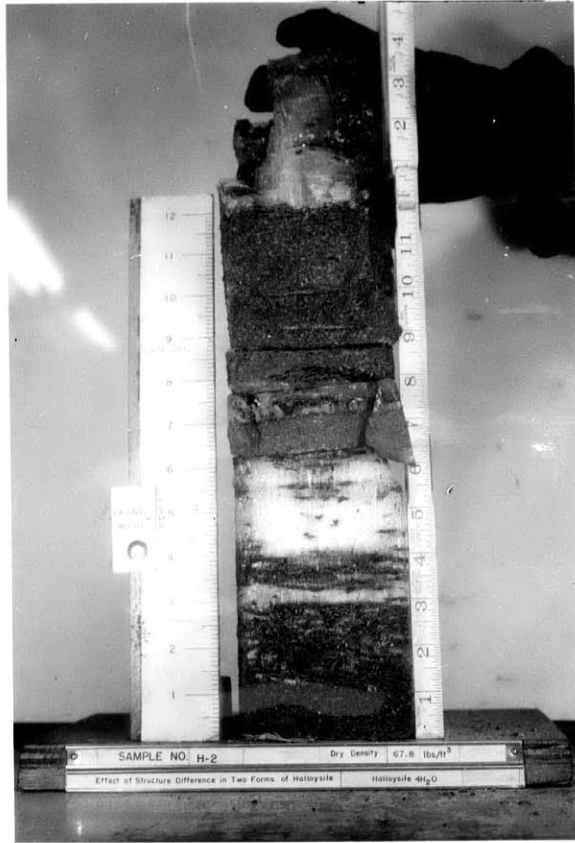
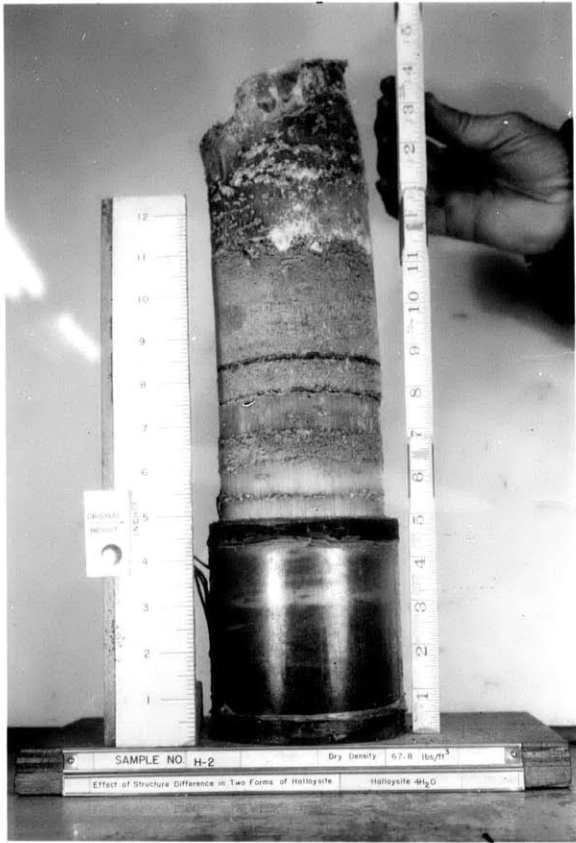


Fig. 6

159

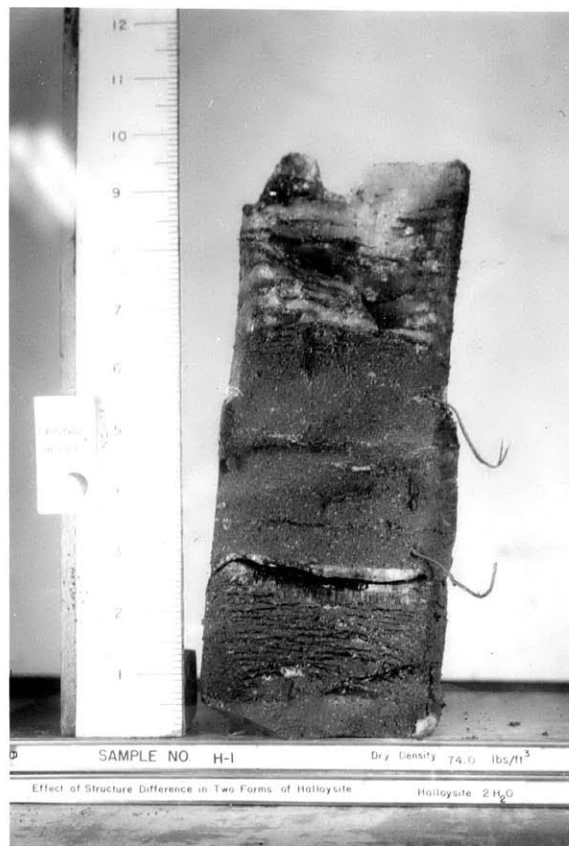
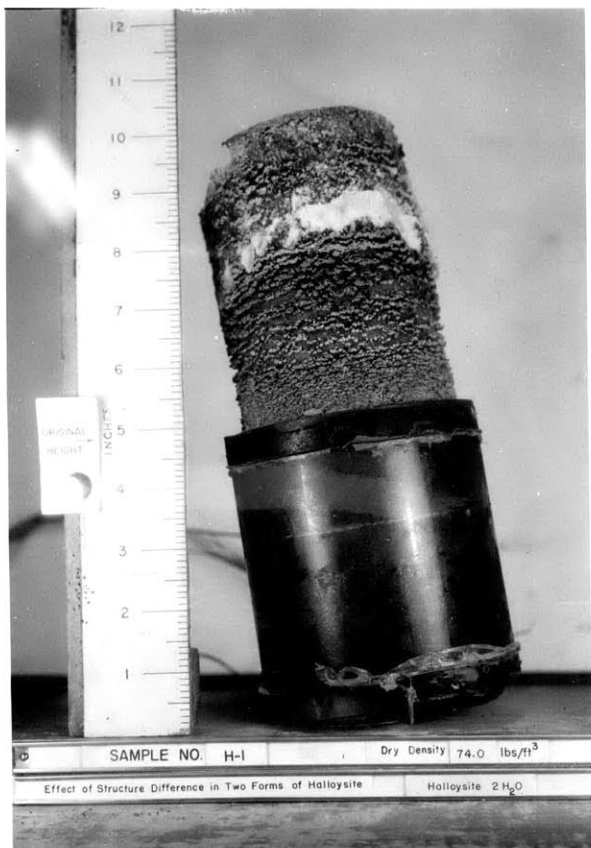
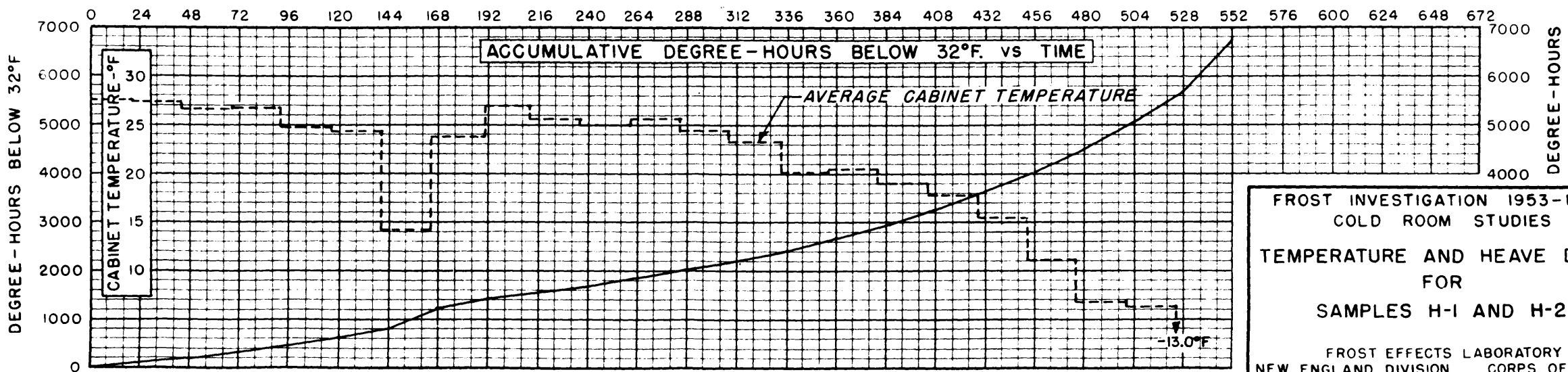
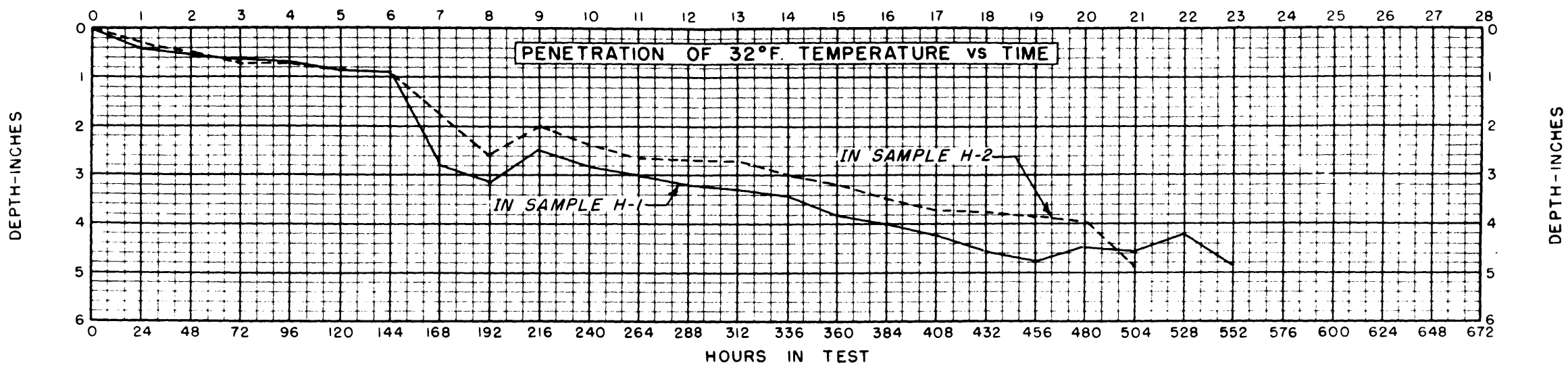
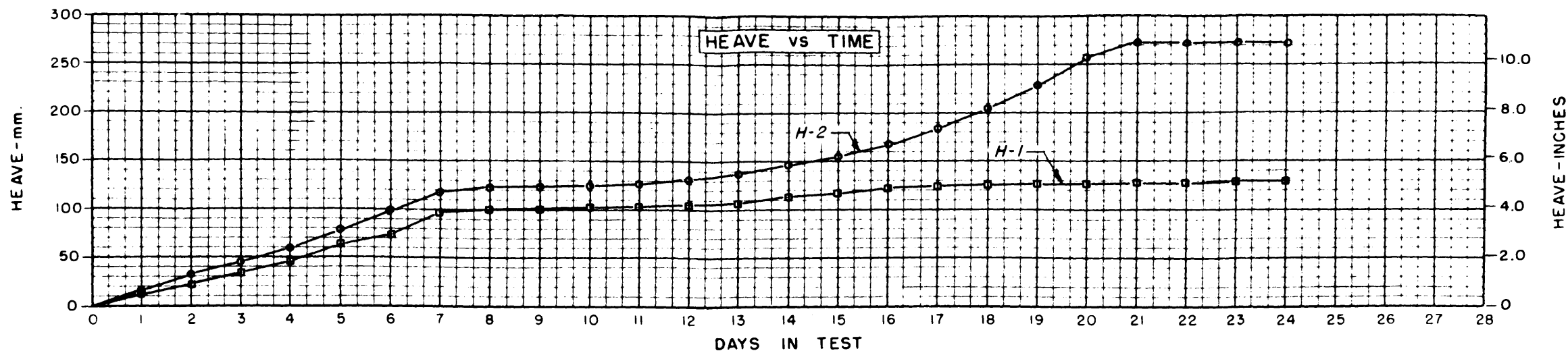


Fig. 7



FROST INVESTIGATION 1953-1954
 COLD ROOM STUDIES
 TEMPERATURE AND HEAVE DATA
 FOR
 SAMPLES H-1 AND H-2
 FROST EFFECTS LABORATORY
 NEW ENGLAND DIVISION CORPS OF ENGR.
 BOSTON, MASS.

FIGURE #8

V. Discussion:

A. Correction Factor and Drying Tests

The correction factors determined by the two different methods show excellent agreement. (Figure 2) A difference of 10% is considered to be well within the range of expected error. Of the two values for C, 0.11 determined by the sieve analysis and DTA is probably the least accurate. Use of $3.5\text{H}_2\text{O}$ (a lower limit) instead of $4\text{H}_2\text{O}$ as an expression for the interlayer water in the hydrated halloysite reduces C from 0.11 to 0.098. The assumption that all interlayer water lost in the dehydration process of halloysite is lost at 105°C . tends to make the .11 value too high. The clay fraction determined by DTA is about 15% higher than the 64% suggested by Skempton. (Lambe, 1950, #9) Using the 64% clay fraction in computing C gives a value of 0.089 for C. This particular difference may easily be due to sample or testing differences, though it presents a lower limit for C. The 0.10 value for C is considered to be the most accurate expression for the lost interlayer water and hence was used throughout in determining corrected water contents. This conclusion is based on two facts; (1) the determination of C as a result of the drying tests depended only upon the qualitative interpretation of x-ray diffraction tests; (2) the desiccator dried (50% relative humidity) sample started to break down at a well defined point. This reasoning assumes that all pore and adsorbed water was lost before any interlayer water was drawn off.

A second fact clearly shown in Figure #2 is that hydrated halloysite will definitely start to dehydrate to the

$2H_2O$ form under conditions quite easily duplicated in the field. Under most conditions, samples reached the breakdown point in a relatively short period of time. This suggests the possibility of changing borrow pit material on a job from the hydrated to the dehydrated form simply by air-drying. The time for complete dehydration to occur is not so easy to determine. In fact, the experimental evidence would indicate that soil being air dried at about $20^{\circ}C$. for example, would never completely reach the dehydrated form. At the end of five days, the air dried sample shown in Figure #2 had reached a condition roughly equivalent to $3H_2O$. Dehydration at this point was still continuing, hence it is difficult to determine where the dehydrating process would stop. Working with air dried samples, Brindley and Goodyear (1951) found that all interlayer water was not removed in dehydration and that the $4H_2O$ factor changed to the form ranging from $2.25H_2O$ to $2.75H_2O$. We may then state that the suggested partial dehydration under field conditions could easily occur in a few days. This reveals practical possibilities but raises the question of whether or not the partially dehydrated halloysite resulting from such a treatment would exhibit the same engineering characteristics as determined for the $2H_2O$ form. On the basis of structure change and the fact that such partially dehydrated forms are very close to the dehydrated form, one can conclude that differences would be minor. This should of course be checked and offers a field for further investigation.

B. Specific Gravity

Grim (1953) states that hydrated halloysite has a G_s varying from 2.0 to 2.2 as compared to a value of 2.55 for the dehydrated form. Logical consideration of this evidence leads to the conclusion that the $4H_2O$ samples would have a smaller G_s than the $2H_2O$ samples. Noting the uncorrected values of G_s in table #1, we find the relative values reversed. The explanation of why the normal testing procedure gives these results is as follows:

1. The laboratory formula for obtaining G_s is

$$G_s = \frac{G_T W_s}{W_s + W_2 - W_1}$$

2. If tests on $4H_2O$ and $2H_2O$ are conducted at the same temperature and equal values of W_s are obtained at the end of each test by oven drying to $105^\circ C.$, the following relationships for the two tests would hold:

- a. G_T is equal for both tests
- b. W_s is equal for both tests
- c. W_2 is equal for both tests

3. The only values that can differ are W_1 , since if the W_1 values were equal, the values of G_s would be the same.

4. $(W_1)_{2H_2O} = W(\text{pyc.}) + W(\text{water}) + (W_s)$

$$(W_1)_{4H_2O} = W(\text{pyc.}) + W(\text{water}) + (W_s) + W_{1w}$$

Where W_{1w} = interlayer water that is driven off by heating to $105^\circ C.$

The above leads to the conclusion that if $W(\text{water})_{4H_2O} + W_{1w} = W(\text{water})_{2H_2O}$ the values of G_s would be

the same, but $(Gs)_{4H_2O} > (Gs)_{2H_2O}$, so the equation must be written:

$W_{(water)4H_2O} + W_{1w} > W_{(water)2H_2O}$, hence $(W_1)_{4H_2O} > (W_1)_{2H_2O}$ so $(Gs)_{4H_2O} > (Gs)_{2H_2O}$. This also leads to the conclusion that the interlayer water has a greater density than the pore water. Some investigators (Alexander, L.T., et al, 1943), have obtained a density for the interlayer water as high as 1.5.

An important result of the specific gravity test is its possible use to indicate the presence of hydrated halloysite. If other information, such as low dry density leads one to suspect that halloysite might be present, then the specific gravity tests might be valuable as a further check. A normal specific gravity test can be run on the soil before it has been dried, then a test can be run on the same sample after it has been oven dried to approximately 105°C. If the first test gives a higher Gs than the second test, this might indicate the presence of hydrated halloysite.

The correction factor of 0.10 gives a Gs of 2.55 for $4H_2O$ instead of the value of 3.03 obtained by the normal testing procedures. The error is about 19%, based on the corrected value. The effect of the corrected Gs will be indicated and discussed in each experiment wherever applicable.

It should be noted that no simple formula for conversion of a known Gs for $4H_2O$, to a corrected Gs, as determined by standard testing procedures is possible, since it is necessary to correct the individual components of the raw data at each temperature and weight reading.

C. Atterberg Limits

As one would expect, the corrected limits for hydrated halloysite are in better agreement with the soil's engineering properties than the uncorrected values. On this basis, the hydrated and dehydrated forms have approximately the same liquid limit, but different P.I.'s. Applying some of the relationships given by Cassagrande, the compressibility should be about the same; the permeability of the $4H_2O$ form should be less and the dry strength of the $4H_2O$ form, greater. Observations and tests generally agree with these predictions, although the permeability of the dehydrated form at maximum dry density is slightly less than the corresponding permeability for the hydrated form. The accuracy of the test data and the relatively small difference in the P.I.'s makes this deviation quite possible. Comparative strength and plasticity characteristics are in agreement with predicted results.

The unusual limits of both halloysite forms might offer additional indications of the possible presence of halloysite in the field, though the inexact nature of the test makes the value of the test questionable in this respect.

The only major deviation from expected results occurred in the frost test. One would expect the $2H_2O$ form to be the more susceptible to frost action but the opposite is true. This deviation is in the same direction as the permeability results but to a far greater extent.

D. Compaction, Permeability and Unconfined Compression Test

Since these three tests give engineering results

which should be considered in relation to each other, an integrated discussion of all three is presented in one section.

The following characteristics can be noted from the three curves on Figure #3.

1. Using standard testing procedures, the maximum dry density for the $2H_2O$ form appears to be about 7% higher than the $4H_2O$ form with the corresponding optimum water contents at 42% and 47% respectively. Correcting the $4H_2O$ water contents increases the apparent maximum dry density to 78.4#/cu. ft. and decreases the water content at optimum to a corrected water content of 34%.

2. Permeability tests at water contents wet and dry of optimum aid in verifying the maximum dry density points. The $2H_2O$ peaks agreed very well. The slight deviation between the $4H_2O$ peaks is entirely possible since the permeability curve is based upon only one point, in the optimum range. The permeabilities are of the same order of magnitude with the $2H_2O$ form having the lower value.

3. At maximum dry density the shear strength of $4H_2O$ is approximately twice that of $2H_2O$.

The compaction tests offer one of the easiest obtainable indications of the presence of either form of halloysite. The test results would indicate that any maximum dry density values in the vicinity of 80#/CF or lower, would indicate the possible presence of halloysite in either form. If standard compaction tests are run (using no correction factor) on an undried sample and on an oven dried (105°C.) sample from the same test boring, a difference in the maximum dry

densities is an indication that $4H_2O$ exists.

The test results also show that the use of $C = 0.10$ has no effect on the direct measurement of permeability and shear strength. The use of $C = 0.10$ only shifts the curves to the left to apparently lower water contents that are based on the definition of $(W_s)_{4H_2O}$. Since the method of determining water contents by heating to $105^\circ C$. is standard procedure, the use of a correction factor for direct permeability determinations and for all shear strength data is not necessary. Tabular values of dry density are definitely affected by a correction factor. Although the application of the correction factor definitely changes the dry density curve, its use is not considered necessary. This follows from the fact that the dry density curve has significance only in locating the point of optimum density, at which point one can expect to get maximum strength, minimum permeability and minimum settlement. This, as already stated, has the advantage of allowing routine test procedures to be used.

As already indicated, the test results show the strength of the $4H_2O$ form to be approximately twice that of the $2H_2O$ form at optimum. These results must be tempered, however, by a consideration of the number of tests run and the type of test. Obviously, more investigation is desirable. Such an investigation should include triaxial compression or direct shear tests to verify the above results and to determine accurately the comparative friction angles.

The difference in shear strength of the magnitude shown on Figure #3 has considerable significance. Hydrated

halloysite would appear to be much preferable for use in fills, dams, and embankments in which strength is of fundamental importance. For example, a slope stability analysis using the slices method would probably show the $4H_2O$ form to be preferable. Such an analysis based upon unconfined compression tests is of course, not practical, but qualitatively, we may say that the actuating forces would be about equal in both cases (the wet densities at optimum differed by about 3%) and hence the stability in each case is generally dependent upon comparative strength.

As a foundation material, the $4H_2O$ form would also be preferable because of its higher strength. Most relationships use shearing strength of cohesive soils as a fundamental factor in determining q_a although it can be argued that in practice, allowable settlements usually are the governing factor in foundation design and hence compressibility must also be considered important. (Taylor, 1948)

The above discussion poses an interesting problem in all cases where the engineer has the $4H_2O$ form available. For reasons already noted, surface sections of embankments, fills, etc., will likely dehydrate to the partially dehydrated form. Moreover, buildings containing heating apparatus would probably cause the top portion of the soil foundation to dehydrate.

In the first case, the best procedure would probably be to utilize a more conservative value for shear strength or a larger factor of safety since the depth of partial dehydration is questionable. The important fact is to recognize

the possibility of change. The second case could result in dehydration to a depth of several feet. Since the $4H_2O$ form appears to be slightly more compressible (Figure #4), dehydration might actually result in less settlement. On the other hand, the bearing capacity of the soil would be definitely decreased. Recognizing these factors, the safest procedure would be to insure that any bearing test results conducted on the soil before construction are considerably modified to anticipate the change.

The lower permeability of the $2H_2O$ form makes it more desirable under circumstance where this factor might outweigh strength considerations. A possible example is its use in the core of an earth dam. The difference here does not appear to be great, however, so that either form might be used depending on availability.

E. Consolidation

A comparison of the consolidation test results (Figure #4) for the two forms of halloysite, shows the following points:

1. The slopes of the (e) vs. $(\log P)$ curves are approximately parallel for the corrected $4H_2O$ and $2H_2O$ tests that had about the same degree of saturation at the start. The compression indexes (C_c) for the 4 to 8 Kg/cm^2 loading increments are of the same order of magnitude when the tests are compared as noted; the (C_c) values being more nearly equal for tests having the closest initial degrees of saturation.

2. The average coefficient of consolidation (C_v) ,

for $2H_2O$ is 56% greater than the average (c_v) for $4H_2O$ in the 4 to 8 Kg/cm² range.

3. It has already been indicated how the use of $C = 0.10$ changes the $4H_2O$ values of (G_s), (w) and (W_s) $_{4H_2O}$. The combined effect of these three changes on consolidation test results can be summarized as follows:

- a. Void Ratio (e) at constant pressure-----Decreases
- b. Saturation (S)----- " Slightly
- c. Coefficient of Compressibility (a_v)-----Decreases
- d. Compression Index (C_c)-----Decreases
- e. Permeability (k)-----No Change
- f. Coefficient of Consolidation (c_v)-----No Change
- g. Primary Compression Ratio (r)--No Change

4. The small amount of expansion on the rebound loads indicates negligible swelling characteristics for both forms of halloysite.

5. The $4H_2O$ form is more compressible than $2H_2O$.

6. The uncorrected $4H_2O$ curves appear to parallel the corrected $4H_2O$ curves, but slope calculations show C_c for the uncorrected curves in the straight line portion is greater than for the corrected curves.

The consolidation test can also be used to identify the $4H_2O$ form. The same process of testing one sample without drying it, then drying a sample from the same boring and testing it applies, but the initial saturation of the two forms should be the same to provide the best basis for a comparison.

Since the correction factor changes the value of (S) relatively little, standard calculations to obtain (S) by using $(S = \frac{Gw}{e})$ would give satisfactory initial degrees of saturation.

An estimate of the experimental error in the consolidation tests is difficult because of the small number of tests run. It is believed they are within the allowable experimental errors since the points for the (e) vs. (log P) curves follow the general pattern of standard consolidation tests. The permeability obtained by the consolidation tests agrees closely with the results obtained from the separate permeability tests. The greatest source of error would be in the values for (c_v) since (c_v) is very sensitive to the value used for (t_{90}) . Some of the compression curves gave very accurate values of (t_{90}) but on others, due to the dial sticking or moving too fast in the first 60 - 90 seconds, the initial points were more scattered, therefore requiring a lot of personal judgement in drawing the straight line part of the compression curve.

The main use of consolidation test data is to predict the amount and rate of settlement under various loads. Using the following formula, (Taylor, 1948) all analysis will be made to show the error, if any, that would result in using standard laboratory data for $4H_2O$ without applying the correction factor.

$$a. \quad e_{u1} = \frac{2H_1}{1-e_1} \frac{P_2-P_1}{\frac{1}{2}(P_2+P_1)} (0.435 Cc)_1 \text{ for } C=0 (4H_2O)$$

$$b. \quad e_{u2} = \frac{2H_2}{1-e_2} \frac{P_2-P_1}{\frac{1}{2}(P_2+P_1)} (0.435 Cc)_2 \text{ for } C=0.10 (4H_2O)$$

c. Pressures and strata thicknesses are equal.

$$d. \therefore \frac{e_{u1}}{e_{u2}} = \frac{1-e_2}{1-e_1} \frac{Cc_1}{Cc_2}$$

Using data from the two (4H₂O) curves and their corresponding corrected curves, the results are:

$$e_{u1} = 1.01e_{u2} \text{ -- curve \#1}$$

$$e_{u1} = 0.96e_{u2} \text{ -- curve \#2}$$

The above results indicate that standard consolidation test results can be used to predict ultimate settlements for 4H₂O without using a correction factor. More tests on the 4H₂O form at varying initial degrees of saturation should be made to further verify this point.

The time prediction for a given amount of settlement to occur in a 4H₂O strata does not depend on the correction factor since (c_v) in the formula: $t(\text{years}) = \frac{TH^2}{(c_v)}$ does not depend on the correction factor.

A comparison of the ultimate settlement between 2H₂O and 4H₂O gives the following results when the 2H₂O and 4H₂O tests having approximately the same initial degree of saturation are compared. (See Figure #4)

$$a. (e_u)_{4H_2O} = (1.2)(e_u)_{2H_2O}$$

$$b. (e_u)_{4H_2O} = 1.69(e_u)_{2H_2O}$$

F. Hydrometer Analysis

The results of this test do not agree with the DTA results for the percentage of clay sizes in the soil mixture. The reason for this is explained very clearly in reference #10. Robertson (#9) points out, in this reference, that the

mineral aggregates are firmly bound together and the normal hydrometer analysis results refer to the aggregates. To obtain a curve representing more nearly the individual grain sizes, it would be necessary to add intensive dispersing agents and to grind the soil to break down the aggregates.

The $2H_2O$ curve logically falls to the left of the $4H_2O$ curve thus indicating greater effective particle diameter since the loss of the interlayer water from the $4H_2O$ aggregates increases the specific gravity of the grains, hence they settle out faster.

G. Frost Susceptability

Both soil samples demonstrated marked susceptibility to frost action. The degree to which each sample heaved under the test conditions is shown in Figure #8. The difference in heave has definite practical significance, but an explanation of this difference is considerably less obvious. The percentage finer than .02mm size for the hydrated halloysite is 47% as compared to 39% for the dehydrated halloysite. (Figure #5) In accordance with accepted frost susceptibility criteria, this difference could explain some variation in the heave, but probably not to the degree obtained. Since the criteria was developed primarily by tests on gravels, sands, and silts, (Haley, 1950), there is also doubt as to whether the 0.02mm size is significant in clays.

Another factor which probably contributes to the difference is the higher permeability of the $4H_2O$ form. This conclusion is consistent with the results of tests by the Frost Effects Laboratory, New England Div., Corps of Engineers,

in which the frost susceptibility in finer grained plastic soils appeared to increase with increase in permeability. (Haley, 1950) This effect cannot accurately be estimated but the difference in permeabilities does not appear large enough to justify a difference in heave of the magnitude encountered.

Haley (1950) suggests other factors affecting frost susceptibility, none of which appear applicable in the present discussion. The best explanation in this particular case seems to be the difference in soil structure of the two clays. The additional interlayer water in the hydrated halloysite may also be a significant factor in its very high susceptibility to frost.

A second factor apparent from the test results is that the hydrated halloysite, when subjected to the severe frost conditions shown on Figure #7, retained its structure. This was determined by x-ray diffraction (Annex A, Sample 28A) at the completion of the frost test. It follows that halloysite encountered in regions subject to severe winters may be in either form, dependent upon other factors.

Although both soils are undesirable from the standpoint of frost susceptibility, a forced choice between the two is obvious. If faced with the possible use of halloysite, the engineer should make every effort to air dry any fill material which may be in the hydrated form.

VI. Conclusions

The facts and conclusions derived from the previous discussion provide certain definite and constructive answers to

the questions already posed. These answers are as follows:

1. Routine laboratory test results may provide indications of the possible presence of halloysite. The most significant results in this respect are low dry density at characteristically high water contents and the unusual Atterberg Limits. If the halloysite is in the hydrated form, several different tests may be used to indicate the characteristic change in structure. In the presence of some or all of these indications, a mineral analysis should be run for positive identification.

2. Hydrated halloysite is extremely sensitive to dehydration and may be expected to change to a partially dehydrated form if air dried for a few days. It is definitely stable under mechanical stresses of the order found in consolidation and compaction tests. Frost action in the test performed, did not cause it to change form. (See Annex A, Sample 28A)

3. Certain routine laboratory test results must be corrected to account for the amount of interlayer water lost in heating hydrated halloysite to 105°C. The correction is recommended for use only in the Atterberg Limits tests where it will more nearly reflect the true characteristics of the soil. In other tests its use will not substantially affect routine engineering calculations as already discussed for each test.

4. Hydrated halloysite appears to have the higher shear strength. It is definitely not preferable to dehydrated halloysite if frost action is expected. In other respects there is no definite advantage to seeking a particular form

of halloysite.

5. The instability of hydrated halloysite may be utilized to obtain dehydrated halloysite by air drying if desired. This procedure is not recommended unless frost action is expected.

6. In situ changes from hydrated to partially dehydrated or dehydrated halloysite may occur under certain conditions. When such a possibility exists, designs should be modified to account for probable losses in shearing strength.

VII. Recommendations

As a result of questions raised by this investigation, the following subjects are recommended for further study:

A. A determination of the engineering characteristics of partially dehydrated halloysite.

B. A study of the effect of halloysite in either form when added in given quantities to other soils.

C. An investigation of the effect of curing on the strength of hydrated and dehydrated halloysite. (This subject was suggested by the chance observation of a considerable strength increase in cured samples of hydrated halloysite.)

D. A further study of the stability of hydrated halloysite under field conditions and the time required for equilibrium to be reached.

E. Using triaxial shear tests, a more complete investigation of the comparative shearing strengths of the two forms of halloysite should be conducted.

Annex A

X-RAY DIFFRACTION TEST RESULTS FOR SAMPLES UNDER
VARIOUS TEST CONDITIONS

Test #	MIT Lab #	Line #	d(Å)	Intensity
6	392	1	7.24	5
		2	4.85	2
		3	4.42	7
		4	3.66	3
		5	3.27	1
		6	2.57	2
11	399	1	11.09	8
		2	4.90	2
		3	4.44	5
		4	4.04	1
		5	3.74	4
		6	2.57	1
12	400	1	7.24	2
		2	4.95	1
		3	4.43	3
		4	3.63	2
		5	3.32	2
		6	2.57	0
16	404	1	7.20	2
		2	4.84	1
		3	4.45	4
		4	3.65	3
		5	3.26	0
		6	2.56	1
19	413	1	11.3	4
		2	4.86	0
		3	4.46	3
		4	3.72	3
		5	3.36	1
		6	2.58	0
22	418	1	11.0	4
		2	4.92	1
		3	4.46	4
		4	3.73	3
		5	3.34	0
		6	2.60	0

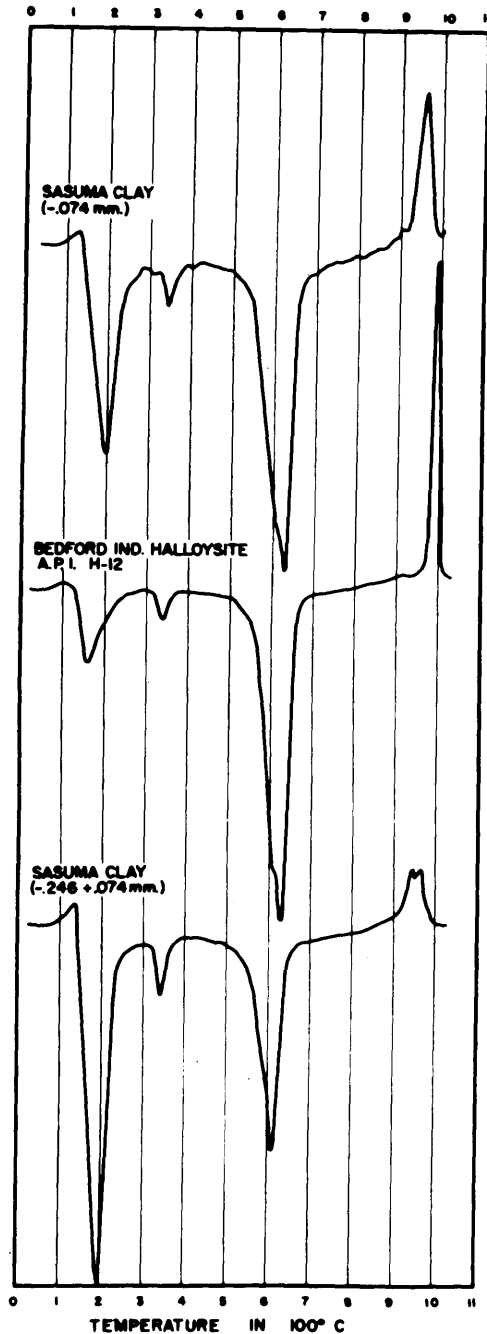
Annex A (Cont'd)

Test #	MIT Lab #	Line #	d(Å)	Intensity
34	484	1	11.7	1
		2	7.30	2
		3	4.93	2
		4	4.45	5
		5	3.70	3
		6	3.21	0
		7	2.58	1
35	485	1	11.9	1
		2	7.18	2
		3	4.93	2
		4	4.45	4
		5	3.70	3
		6	3.57	2
		7	2.58	2
36	486	1	7.14	1
		2	4.95	2
		3	4.46	3
		4	3.68	2
		5	3.34	0
		6	2.58	0
37	487	1	7.28	2
		2	4.90	1
		3	4.45	4
		4	3.65	2
		5	3.29	0
		6	2.57	1
28A Frost Specimen	459	1	11.4	4
		2	7.18	1
		3	4.92	1
		4	4.44	3
		5	4.05	1
		6	3.73	2
		7	2.58	0
24	420	1	7.16	2
		2	4.94	1
		3	4.47	3
		4	3.61	1
		5	3.23	0
		6	2.58	1

Annex A (Cont'd)

Test #	MIT Lab #	Line #	d(Å)	Intensity
25	426	1	11.1	4
		2	4.85	1
		3	4.45	3
		4	4.07	0
		5	3.71	1
		6	3.37	0
		7	2.58	1
26	429	1	7.18	2
		2	4.85	2
		3	4.45	4
		4	3.65	2
		5	3.33	1
		6	2.56	1
31	481	1	11.4	5
		2	4.89	1
		3	4.46	4
		4	3.75	2
		5	2.58	1
32	482	1	7.11	2(B)
		2	4.89	2
		3	4.45	4
		4	3.68	3
		5	3.56	0
		6	2.58	1
33	483	1	11.2	4
		2	4.89	1
		3	4.44	4
		4	3.75	3
		5	3.33	0
		6	2.57	1

**ANNEX B
DETERMINATION OF CORRECTION FACTOR C
(Method I)**



SIEVE ANALYSIS

A. Fraction retained on #200 sieve

- (1) Wt. sample = 67.2 gr.
- (2) Uncorrected m./c. = 26.6
- (3) Dry wt. sample = 53.1 gr.

B. Fraction passing #200 sieve

- (1) Wt. sample = 77.0 gr.
- (2) Uncorrected m./c. = 24.3
- (3) Actual dry wt. sample = 62.0 gr.

C. Percent of sample passing and retained on #200 sieve

- (1) Total dry wt. sample = 115.1 gr.
- (2) % retained on #200 sieve = 46.2
- (3) % passing #200 sieve = 53.8

D.T.A. RESULTS

A. Reference D.T.A. - Bedford Indiana Halloysite

B. Halloysite in fraction retained on #200 sieve ≈ 65%

C. Halloysite in fraction passing 200 sieve ≈ 93%

COMPUTATION OF C

A. Halloysite in entire sample

- (1) Contribution fraction retained #200 sieve = 29.9%
- (2) Contribution fraction passing 200 sieve = $\frac{50.1}{80}$ %
- Total = 80 %

B. Theoretical % interlayer water = 13.9

C. $C = (.80)(13.9) \approx .11$

Annex C

SELECTED DATA FROM DRYING TESTS ON HYDRATED HALLOYSITE

Date	Time	Temp. °C.	Wt. Dish - Soil	Wt. Soil	X-Ray # Results	$\frac{W_L}{W_w - W_T}$ *	$\frac{W_w - W_T}{W_s}$ %
<u>Test Conditions: Heated 50°C.</u>							
2/10	1600	Room**	118.927	30.150	-----	-----	-----
2/11	1125	50	111.780	23.003	#6	.314	1.38
					2H ₂ O	-----	-----
2/15	1320	91.3	111.466	22.689	-----	-----	-----

<u>Test Conditions: Heated 30°C.</u>							
2/15	1615	Room	118.829	30.060	-----	-----	-----
2/16	1103	30.5	113.830	25.061	-----	-----	-----
2/17	0850	30.2	112.284	23.515	-----	-----	-----
2/19	1115	30.2	111.898	23.129	#12	.577	2.56
					Diffused	-----	-----
2/20	1100	30.2	111.849	23.080	-----	.528	1.76
2/25	1110	105	111.321	22.552	-----	-----	-----

<u>Test Conditions: Heated 30°C.</u>							
2/22	1300	Room	92.968	40.686	-----	-----	-----
2/23	1115	29.8	86.192	33.910	-----	-----	-----
2/24	0826	30.1	84.302	32.020	#16	1.604	5.26
					Diffused	-----	-----
3/16	1130	105	82.698	30.416	-----	-----	-----

* W_L is wt. of water left at time (T)

** Room temperature = 22°C.

Annex C (Cont'd)

SELECTED DATA FROM DRYING TESTS OF HYDRATED HALLOYSITE

Date	Time	Temp. °C.	Wt. Dish - Soil	Wt. Soil	X-Ray # Results	$\frac{W_L}{W_w - W_T}$ *	$\frac{W_w - W_T}{W_s}$ %
<u>Test Conditions: Heated 30°C.</u>							
4/15	1030	Room	163.553	65.613	-----	-----	-----
4/16	0850	30.1	155.746	57.806	#33 4H ₂ O	8.752	17.85
4/17	0920	30.1	151.940	54.000	#35 Diffused	4.946	9.92
4/19	1000	29.0	149.568	51.655	#37 Diffused	2.601	5.30
4/21	920	105	146.994	49.054	-----	-----	-----
<u>Test Conditions: Air Dried - Room Temperature 25°C.</u>							
4/14	1340	Room	121.042	32.864	-----	-----	-----
4/15	1015	Room	116.412	28.234	#31 4H ₂ O	3.509	14.2
4/16	0845	Room	114.683	26.505	#32 Diffused	1.780	7.21
4/17	0915	Room	114.630	26.452	#34 Diffused	1.727	6.98
4/19	0955	Room	114.308	26.130	#36 Diffused	1.405	5.69
4/21	0915	105	112.903	24.725	-----	-----	-----
<u>Test Conditions: Dried in Desiccator Controlled 50% RH</u>							
3/1	1045	----	52.711	4.059	-----	-----	-----
3/4	1030	----	52.215	3.563	-----	-----	-----
3/6	1050	----	52.190	3.538	-----	-----	-----
3/8	1050	----	52.189	3.537	#22 4H ₂ O	.332	10.36
3/11	1110	----	52.180	3.527	#25 4H ₂ O	.322	10.03
3/17	0930	----	52.181	3.528	#26 Diffused	.323	10.07
3/18	1003	105	51.857	3.205	-----	-----	-----

Annex D

SUMMARY OF SPECIFIC GRAVITY TESTS

Test No.	(Gs)	
	(4H ₂ O) C = 0	Mixture C=0.10
1	2.94	2.5
1	3.04	2.56
1	3.02	2.54
2	3.05	2.56
2	3.06	2.57
2	3.06	2.58
8	3.04	2.56
8	3.02	2.55
8	3.04	2.56
	Av.=3.03	Av.=2.55

Test No.	(Gs)	
	(4H ₂ O) C = 0	Mixture C=0.10
13	3.07	2.48
13	3.06	2.47
13	3.08	2.48
13	3.08	2.48
	Av.=3.07	Av.=2.48

Test No.	(2H ₂ O) Gs
10	2.94
10	2.96
10	2.96
11	2.94
11	2.95
11	2.94
	Av.=2.95

Test No.	(2H ₂ O) Passed #200 Sieve Gs
5	2.77
5	2.80
5	2.78
7	2.83
7	2.82
7	2.85
	Av.=2.83

The maximum deviation based on the lowest values obtained is as follows:

	% Deviation
4H ₂ O - Mixture (C=0)	4.08 %
4H ₂ O - Mixture (C=0.10)	3.2 %
4H ₂ O - Passing #200 sieve (C=0)	0.654%
2H ₂ O - Mixture =	0.68 %
2H ₂ O - Passing #200 sieve =	2.88 %

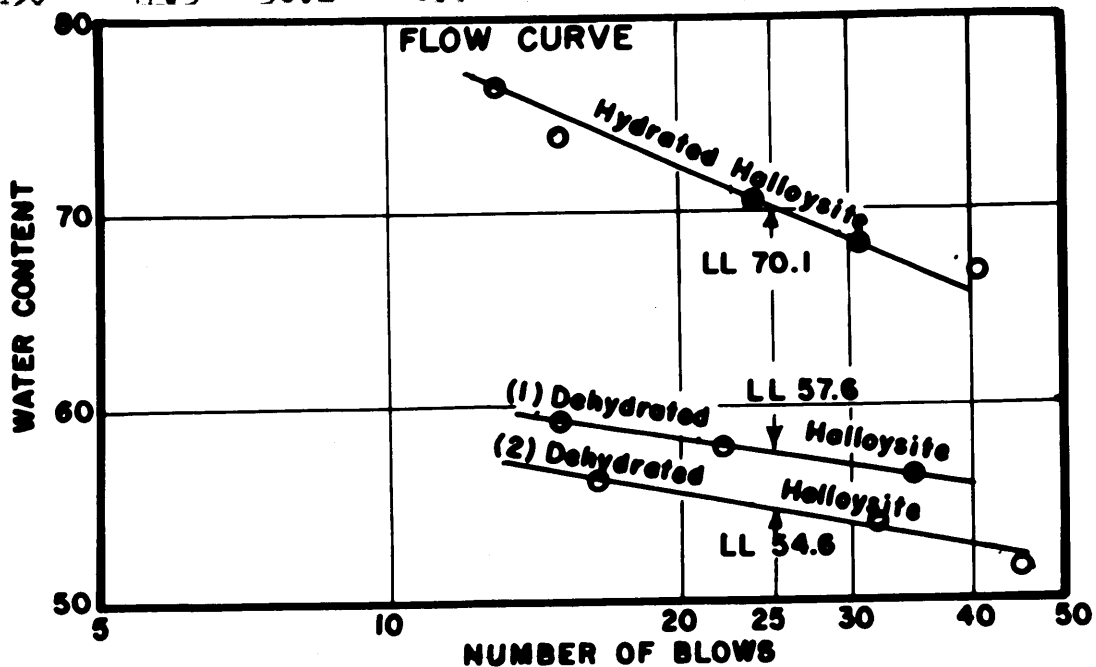
Annex E

SUMMARY OF ATTERBURG LIMITS DATA

Soil Sample	Plastic Limit		Liquid Limits		
	Det. No.	w _p	Det. No.	No. Blows	w
4H ₂ O 2/22/54	1	58.3	1	24	70.6
	2	55.4	2	41	66.8
	3	53.2	3	15	73.9
	4	55.9	4	13	76.5
				5	31
2H ₂ O 2/24/54	1	48.1	1	15	59.4
	2	46.8	2	35	56.8
	3	47.6	3	22	57.8
	4	46.6			
2H ₂ O 2/26/54	-	----	1	18	56.5
	-	----	2	32	54.1
	-	----	3	45	51.7

Result Summary:

	P.L.	L.L.	P.I.	C = 0.10			Shrinkage Limit
				P.L.	L.L.	P.I.	
4H ₂ O	55.7	70.1	14.4	41.6	54.6	13.0	37.2
2H ₂ O	47.3	56.1	8.8	----	----	----	38.1



COMMENTS

1. Shrinkage limits are based on only one determination; results are not valid comparisons due to the collapse of the hydrated halloysite during the drying of the soil pat.
2. Results are based on average values; they are considered accurate within 10%. Such an estimate considers the nature of the test and the judgement of the individual performing the test.

Annex F

RESULTS OF WILSON MINIATURE COMPACTION TESTS

2H ₂ O				
Test No.	Wet Density #/CF	Water Content %	Void Ratio	Dry Density #/CF
1	107.66	43.5	1.46	74.9
2	107.06	41.6	1.43	75.6
3	99.97	38.2	1.55	72.2
4	103.77	39.3	1.48	74.3
5	98.37	36.65	1.56	72.0
6	102.27	39.1	1.5	74.0
7	98.37	36.6	1.56	72.0
8	107.47	43.1	1.46	75.0
9	107.47	41.2	1.42	76.1
10	95.89	33.65	1.57	71.6
11	107.8	42.65	1.43	75.6
12	108.44	42.57	1.42	76.1
13	107.66	42.05	1.43	75.7

4H ₂ O								
Test No.	C=0	C=0.10	C=0	C=0.10	C=0	C=0.10	C=0	C=0.10
1	92.06		37.2	24.7	1.82	1.15	67.0	74.0
2	93.65	N	39.83	26.9	1.82	1.155	67.0	73.8
3	98.37	O	41.05	28.2	1.72	1.07	69.6	76.8
4	105.3	*	48.1	34.5	1.66	1.03	71.1	78.4
5	87.23	C	34.3	22.0	1.90	1.23	64.9	71.5
6	101.99	H	45.1	31.9	1.695	1.06	70.2	77.4
7	103.33	A	46.7	33.2	1.68	1.05	70.5	77.6
8	105.81	N	49.35	35.8	1.68	1.04	70.6	77.9
9	100.86	G	57.0	42.7	1.96	1.1	64.0	70.6
10	98.3	E	41.75	28.8			69.4	76.4
11	101.9		43.6	30.6			70.9	78.0
12	102.8		56.8	42.5			65.5	72.0

Annex C

TABULATED PEAK POINTS FROM UNCONFINED COMPRESSION TEST
HYDRATED HALLOYSITE (4H₂O)

I. Initial Data:

Proving Ring Calibration - 1.05#/Div.
Circumference of Samples - 4.125 in.
Initial Area of Samples (A₀) - 1.352 sq. in.
Initial Length of Samples (L₀) - 2.81 in.

Test #	Date*	Water Content		Initial Void Ratio	
		C = 0	C=0.10	C = 0	C=0.10
C-1-2	3/3	37.2	24.6	1.82	1.15
C-1-3	3/3	39.7	26.9	1.82	1.155
C-1-4	3/3	40.8	28.0	1.72	1.07
C-1-5	3/3	48.1	34.5	1.66	1.03
C-1-6	3/3	34.3	22.0	1.90	1.23
C-3-1	6/3	45.1	31.9	1.695	1.06
C-3-2	6/3	46.7	33.2	1.68	1.05
C-3-3	6/3	49.0	35.2	1.68	1.04
C-3-4	10/3	48.8	35.1	---Rejected---	

Test #	Strain %	Area A sq. ft.	Prov. Ring in .0001"	Axial Load P, in lbs.	P/2A lbs/ft ²
C-1-2	1.07	.00951	159	62.0	3260
C-1-3	1.42	.00953	158	60.9	3200
C-1-4	1.96	.00959	157	59.8	3120
C-1-5	4.84	.00987	135	36.8	1863
C-1-6	1.07	.00951	132	33.6	1768
C-3-1	1.85	.00957	148	50.4	2630
C-3-2	1.92	.00958	133	34.7	1810
C-3-3	5.06	.00992	122	23.1	1165
C-3-4	2.73	.00968	117.5	18.4	951

*All dates are in 1954.

Annex G (Cont'd)

DEHYDRATED HALLOYSITE (2H₂O)

Test #	Date*	Water Content	Initial Void Ratio
C-2-1	5/3	33.6	1.57
C-2-2	5/3	42.65	1.43
C-2-3	6/3	42.4	1.42
C-2-4	6/3	42.0	1.43
C-4-1	10/3	43.5	1.46
C-4-2	10/3	37.8	1.60
C-4-3	10/3	41.6	1.43
C-5-1	15/3	38.2	1.545
C-5-2	15/3	39.1	1.48
C-5-3	15/3	36.1	1.55
C-7-1	20/3	39.1	1.50
C-7-2	20/3	36.6	1.56
C-7-3	20/3	43.1	1.46
C-7-4	20/3	41.2	1.42

Test #	Strain %	Area A sq. ft.	Prov. Ring in .0001 ⁿ	Axial Load P, in lbs.	P/2A lbs/ft ²
C-2-1	1.96	.00949	143.0	45.2	2385
C-2-2	4.38	.00983	124.8	26.0	1322
C-2-3	4.98	.00990	123.0	24.2	1222
C-2-4	2.81	.00967	119.5	20.5	1060
C-4-1	8.96	.01031	116.9	17.8	862
C-4-2	1.49	.00955	126.0	27.3	1430
C-4-3	4.31	.00983	122.0	23.1	1176
C-5-1	1.71	.00956	142.0	44.1	2305
C-5-2	2.92	.00968	131.0	32.5	1679
C-5-3	1.28	.00952	133.0	34.6	1819
C-7-1	1.03	.00950	117.0	17.9	942
C-7-2	1.39	.00954	134.0	35.7	1873
C-7-3	9.15	.01037	119.8	20.8	1003
C-7-4	4.31	.0983	120.0	21.0	1068

*All dates are in 1954.

Annex H

DIRECT PERMEABILITY TEST

	2H ₂ O			4H ₂ O - (C=O)*				
	1	2	3	4	5	6	7	8
Burette (i)	2.54	2.61	2.86	3.02	2.63	1.80	3.54	3.84
Burette (f)	3.00	2.86	3.52	8.05	5.30	2.28	1.28	1.98
Q _{cc}	0.46	0.25	0.66	5.03	2.73	0.48	0.74	1.14
Time (Min.)	30	30	30	9	30	34	31	35
k - cm/sec	$\frac{2.99}{107}$	$\frac{1.62}{107}$	$\frac{4.28}{107}$	$\frac{1.09}{107}$	$\frac{1.77}{107}$	$\frac{2.75}{107}$	$\frac{4.65}{107}$	$\frac{6.36}{107}$
Swelling in Inches	Neg.	Neg.	Neg.	1/4"	3/16"	Neg.	1/32"	1/32"

*Correction factor does not apply to this test.

Annex I

CONSOLIDATION TESTS

Primary Compression Ratio

Load in Kg/cm ²	2H ₂ O 3/15/54*	2H ₂ O 3/22/54	4H ₂ O 2/12/54	4H ₂ O 3/22/54
$\frac{1}{4}$ to $\frac{1}{2}$	-----	-----	-----	0.290
$\frac{1}{2}$ to 1	-----	-----	-----	0.378
1 to 2	0.1185	0.233	0.804	0.264
2 to 4	0.181	0.244	0.506	0.336
4 to 8	0.252	0.260	0.527	0.346

Coefficient of Consolidation

Load in Kg/cm ²	2 H ₂ O				4 H ₂ O			
	t ₉₀ sec.	c _v cm ² /sec	t ₉₀ sec	c _v cm ² /sec	t ₉₀ sec	c _v cm ² /sec	t ₉₀ sec	c _v cm ² /sec
0 to $\frac{1}{4}$	---	-----	427.8	-----	331	6.78x10 ⁻³	28.56	48x10 ⁻³
$\frac{1}{4}$ to $\frac{1}{2}$	---	-----	519	3.77x10 ⁻³	240	9.32x10 ⁻³	26.16	54.8x10 ⁻³
$\frac{1}{2}$ to 1	79.22	25.2x10 ⁻³	390	4.92x10 ⁻³	228	9.32x10 ⁻³	54.18	23.4x10 ⁻³
1 to 2	101.4	18x10 ⁻³	282.6	6.54x10 ⁻³	216.6	9.13x10 ⁻³	126	9.15x10 ⁻³
2 to 4	107.7	15x10 ⁻³	145.8	12.0x10 ⁻³	254.4	7.52x10 ⁻³	135	7.7x10 ⁻³
4 to 8	93.6	15.6x10 ⁻³	115.8	14.4x10 ⁻³	228	7.79x10 ⁻³	93.6	9.9x10 ⁻³

* The date of the test is given so a cross reference to the data can be made.

Annex I (Cont'd)

CONSOLIDATION TEST

Tabulated Void Ratios vs. Pressures

Load in Kg/cm ²	2H ₂ O		4H ₂ O (2/12/54)		4H ₂ O (3/22/54)	
	3/15/24*	3/22/54	C = 0	C=0.10	C = 0	C=0.10
0	-----	1.552	1.965	1.262	3.04	2.09
$\frac{1}{4}$	1.944	1.526	1.93	1.238	2.48	1.669
$\frac{1}{2}$	1.92	1.50	1.90	1.215	2.278	1.508
1	1.85	1.461	1.85	1.173	2.10	1.375
2	1.68	1.407	1.77	1.113	1.945	1.255
4	1.53	1.348	1.68	1.046	1.80	1.14
8	1.40	1.281	1.58	0.967	1.658	1.032
2**	1.41	1.31	1.59	0.974	1.672	1.044
1.077	-----	-----	1.592	0.977	-----	-----
0.1	1.452	1.334	-----	-----	1.718	1.08

* The date the test was started is given so a cross reference to the actual test can be made.

** The last three loads are rebound loads.

Annex I (Cont'd)

CONSOLIDATION TESTS

	2H ₂ O	2H ₂ O	4H ₂ O (2/12/54)		4H ₂ O (3/22/54)	
	3/15/54	3/22/54	C = 0	C = 0.10	C = 0	C = 0.10
* Com- pression Index-C _c	.432	.222	0.332	0.262	0.472	.358
* Coeff. of Compres- sibility a _v -cm ² /kg	.0313	.0161	0.024	0.019	.0342	.026
* Perme- ability k-cm/sec	3x10 ⁻⁷	1x10 ⁻⁷	7.1x10 ⁻⁷	7.36x10 ⁻⁷	1.24x10 ⁻⁷	1.24x10 ⁻⁷
% Sat- uration End - S _e	65	94	90.5	89.5	53.2	47.6
% Sat- uration End - S _e	87.5	91.5	93.2	92.3	94.5	92.4
Water Content Start-w _g	----	49	58.6	44.3	53.0	39.1
Water Content End - w _e	43.2	41.5	49.0	35.4	53.0	39.1

* These values computed for the 4 to 8 Kg/cm² load increment

Annex J

GRAIN SIZE ANALYSIS

Date: 3/20/54

SOIL: HYDRATED HALLOYSITE (4H₂O)

Part A: Sieve Analysis

Sample Weight

Weight wet sample: 205.3 gr.
Water content: 30.2
Actual dry wt. soil (105°C): 143.3
Corrected water content: 18.4
Corrected dry weight: 167.7

Wt. Soil Passing #200 Sieve

Wt. soil retained on sieve

After drying to 105°C: 70.7 gr.
Corrected weight soil: 77.0 gr.

Results

Sieve No.	Sieve Openings	Wt. Soil Ret.	% Retained	% Finer
200	.074 mm	70.7 grs	49.3	50.7
200	.074 mm	77.0 grs	46	54

Part B: Hydrometer Analysis: See Figure 5.

* * * * *

SOIL: DEHYDRATED HALLOYSITE (2H₂O)

Part A: Sieve Analysis

Sample Weight: 109 gr.

Wt. Soil Passing #200 Sieve: 54.5 grs.

Results

Sieve No.	Sieve Openings	Wt. Soil Ret.	% Retained	% Finer
200	.074 mm	54.5 grs	50.0	50.0

Part B: Hydrometer Analysis: See Figure 5.

Annex K

SUMMARY OF COLD ROOM TEST DATA

<u>LABORATORY SAMPLE NUMBER (1)</u>	<u>DRY UNIT WEIGHT pcf</u>	<u>SPECIFIC GRAVITY</u>	<u>VOID RATIO e</u>	<u>INITIAL DEGREE OF SATURATION IN PER CENT</u>
H-1	73.9	2.95	1.492	98.2
H-2	67.8	3.03	1.790	99.8

<u>LABORATORY SAMPLE NUMBER (1)</u>	<u>AVERAGE WATER CONTENT IN PER CENT</u>		<u>HEAVE IN PER CENT (2)</u>	<u>AVERAGE RATE OF HEAVE mm/day</u>	<u>FROST SUSCEPTIBILITY CLASSIFICATION</u>
	<u>BEFORE TEST</u>	<u>AFTER TEST</u>			
H-1	49.7	117.3	111.0	6.4	High
H-2	58.9	212.7	221.3	10.7	Very High

NOTES:

- (1) Sample H-1 designated as 2H₂O
- () Sample H-2 designated as 4H₂O
- (2) Samples were completely frozen at end of freezing test.

Annex K (Cont'd)

OBSERVATIONS FROM EXAMINATION
OF SPECIMENS AFTER FREEZING

WATER CONTENT DISTRIBUTION

SAMPLE NUMBER H-1
Open System

Original Height = 4.84"
Final Height = 10.21"
Original Water Content = 49.7%

<u>Depth"</u>	<u>W.C. %</u>
0.0 - 2.0	602.2
2.0 - 3.7	320.2
3.7 - 4.7	80.3
4.7 - 6.0	61.7
6.0 - 7.0	63.4
7.0 - 8.0	117.1
8.0 - 9.5	86.1
9.5 -10.21	61.4

SAMPLE NUMBER H-2
Open System

Original Height = 4.89"
Final Height = 15.71"
Original Water Content = 58.9%

<u>Depth"</u>	<u>W.C. %</u>
0.0 - 2.0	1046.5
2.0 - 4.2	93.8
4.2 - 6.0	84.6
6.0 - 7.0	73.2
7.0 - 8.0	91.3
8.0 - 9.0	685.0
9.0 - 9.7	108.6
9.7 -10.5	1819.5
10.5 -11.5	1358.5
11.5 -13.0	360.0
13.0 -14.0	310.6
14.0 -15.71	92.2

ICE LENS DISTRIBUTION

SAMPLE H-1: Top 3.75 inches (*) is an ice mass containing soil lenses. Short soil lenses 0.06 inches in thickness spaced 0.06 to 0.35 inches apart near the top, becoming longer and more closely spaced with depth. Hairline lenses spaced 0.06 inches apart from 3.75 to 6.75 inches. Few scattered hairline lenses from 6.75 to 7.75 inches. Lenses 0.25 inches in thickness at 7.75 and 9.5 inches. Lenses 0.03 inches in thickness spaced 0.06 inches apart from 8.0 to 9.5 inches. No visible ice segregation below 9.75 inches. Sample completely frozen. Total height of sample at end of test 10.21 inches.

SAMPLE H-2: Top 4.25 inches is an ice mass containing a few
* Measurements are distances measured from top of frozen sample.

Annex K (Cont'd)

SAMPLE H-2:
(Cont'd)

soil lenses, 0.12 inches in thickness and up to 1.0 inches in length. Hairline lenses spaced 0.06 inches apart from 4.25 to 8.0 inches with one ice lens 0.12 inches in thickness at 7.1 inches. Ice lens from 8.0 to 8.75 inches containing a few soil lenses 0.06 inches in thickness and 0.5 inches in length. Hairline lenses spaced 0.06 inches apart from 8.75 to 9.5 inches. Ice lens from 9.5 to 11.5 inches containing one layer of soil from 0.12 to 0.25 inches in thickness. Alternate soil and ice lenses 0.12 inches in thickness from 11.5 to 12.1 inches. Ice lens from 12.1 to 12.6 inches. Ice lenses 0.06 inches in thickness spaced 0.06 inches apart from 12.6 to 14.0 inches. Hairline lenses spaced 0.06 inches apart from 14.0 inches to bottom of sample. Sample completely frozen. Total height of sample at end of test 15.71 inches.

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