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GRADE POUR LEQUEL CETTE THÈSE FUT PRÉSENTÉE Ph.D.YEAR THIS DEGREE CONFERRED/ANNÉE D'OBTENTION DE CE DEGRÉ 1976NAME OF SUPERVISOR/NOM DU DIRECTEUR DE THÈSE D.A. Kasianchuk, R.S. Williams

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VOLUME CHANGE OF FROZEN SOILS

by

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A Thesis

Presented to the Faculty of Graduate Studies
of Carleton University in Partial
Fulfillment of the Degree of
DOCTOR OF PHILOSOPHY IN CIVIL ENGINEERING

Faculty of Engineering

Carleton University

Ottawa, Canada

June, 1975

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"Volume Change of Frozen Soils"
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ABSTRACT

Volume Change of Frozen Soils

A temperature and pressure-controlled dilatometer was developed for the accurate measurement of volume changes of frozen soils induced by change of temperature or applied external pressure. The soil sample in the dilatometer is completely saturated and there is no exchange of mass between the soil sample and the surroundings. Four different soils were investigated ranging from a sandy-silty clay to a clayey soil. The results of the observed volume change are analyzed from the points of view of thermodynamics and soil mechanics.

Based on the thermodynamic relations governing the ice-water phase composition the results of the experimental work demonstrate that changes in volume of a soil on freezing, resulting from isobaric temperature change, can be predicted from the free energy-moisture content relationship of the unfrozen soil, down to -5°C . The results also show that the effect of applied external pressure on volume changes of a frozen soil can be predicted using the thermodynamic relation between the freezing-point depression of the soil water and the applied pressure. It is further shown that the mineralogical composition and the pore-size structure of a soil influence the magnitude of the soil volume change on freezing.

Based on the interpretation of test results it is suggested that "clay-packets" are formed during soil freezing. The ice intrudes through the soil matrix with the ice-water interfaces representing the boundary between the formed packets and the ice. At substantially lower temperatures however, the ice intrudes through the soil pores within the individual packets. It is further suggested that the process of volume changes in a frozen soil, induced by lowering the temperature, is analogous to the consolidation of the unfrozen soil under applied pressures.

Thanks:

To My Father!

For he gave me skill, stamina and
the knowledge of my past.

To My Mother!

For she gave me the love for life
and taught me to respect it.

"My Heart Soars"

By Chief Dan George

ACKNOWLEDGEMENTS

The experimental work was carried out in the Geotechnical Science laboratories, Department of Geography.

The author is indebted to his research supervisor Dr. P.J. Williams, of that Department for his inspiring challenges and helpful guidance throughout the work. Dr. D.A. Kasianchuk acted as coordinating supervisor on behalf of the Faculty of Engineering.

Numerous discussions with Dr. J.K. Torrance are gratefully acknowledged. The technical assistance of Mr. A. Pendlington, Head, Research and Instrumental Facilities, Geography Department in developing the apparatus is much appreciated.

The generous sponsorship provided by the National Research Council is also acknowledged.

Gratitude is also extended to Ms. S. MacLaren for kindly typing the manuscript.

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DEFINITIONS

Matric suction: The negative gauge pressure relative to the external gas pressure on the soil water, to which a solution identical in composition with the soil water must be subjected in order to be in equilibrium through a porous permeable wall with the soil water.

Osmotic suction: The negative gauge pressure to which a pool of water must be subjected in order to be in equilibrium through a semi-permeable (i.e. permeable to water molecules only) membrane with a pool containing a solution identical in composition with the soil water.

Total suction: The negative gauge pressure relative to the external gas pressure on the soil water to which a pool of pure water must be subjected in order to be in equilibrium through a semi-permeable membrane with the soil water. Total suction is thus equal to the sum of matric suction and osmotic suction.

The absolute Gibbs free energy f : An arbitrary chosen function from the mathematical analysis of energy problems, defined by the equation $f = h - Ts$, useful because of its

property that at equilibrium, the change in free energy, $df = 0$ for all possible infinitesimal changes.

The relative free energy of soil water Δf : The free energy of soil water relative to the free energy of pure free water at 0°C . For simplicity, the relative free energy may be referred to as the "free energy".

Relative Humidity: Is the ratio of the vapour pressure of the soil water to the vapour pressure of pure free water, both at the same temperature.

Unfrozen water: That part of the soil water that remains unfrozen at temperatures below 0°C , and is in equilibrium with ice.

SYMBOLS

e	Internal energy of a system.
f	Absolute Gibbs free energy.
Δf	Relative free energy, or free energy of soil water.
g	Gravitational acceleration.
h	Heat content or enthalpy.
H	Soil water suction expressed as height of a column. of water in cm.
L	Latent heat of fusion of ice.
pF	Logarithm of the soil water suction H .
P_a	Pressure of air in the soil.
P_i	Pressure of ice in the soil.
P_w	Pressure of water in the soil.
dq	The amount of heat absorbed by the system from the surroundings.
r_{iw}	Radius or equivalent radius of ice-water interface.
r_{aw}	Radius or equivalent radius of air-water interface.
s	Specific entropy.
ds	Change in entropy.
T	Absolute temperature $^{\circ}\text{K}$.
ΔT	Freezing-point depression of the soil water.
v	Specific volume.

dw Total work performed by a system on its surroundings.

dw_m Mechanical work performed by a system on its surroundings.

σ_{iw} Interfacial energy (surface tension) ice-water.

σ_{aw} Interfacial energy (surface tension) air-water.

INTRODUCTION

As the development of the North becomes more important for its vast natural resources, the study of the physical properties and engineering behaviour of frozen ground becomes very important in exploration for oil fields and other minerals, in the design of foundations, and in the construction of roads and pipelines.

The change in volume of soils on freezing or thawing results from alteration of the soil thermal regime imposed by engineering activities as well as changes in climatic conditions. The resulting heaving on freezing or consolidation on thawing leads to disruption of engineering construction and environmental effects. Consequently, much attention has been paid to the problems of frost heaving and the consolidation of thawing soils.

In frozen soils part of the pore water remains unfrozen at temperatures below 0°C and it is acknowledged that the change in the ice-water phase composition has a substantial influence on the mechanical, thermal, and hydrological properties of frozen soils. Nevertheless the basic concepts governing the ice-water phase composition have received little attention among workers in this field. A study of changes in volume of frozen soils resulting from changes of insitu ice-water phase composition will provide a deeper understanding

of these basic concepts and will result in the better evaluation of many of the phenomena observed in the field by engineers.

Until the 1950's it was generally assumed that in frozen soils the moisture content of soil occurred more or less as ice." A few earlier workers in the field of soil physics had discussed depressions of freezing-point of water in saturated soils but these were generally thought to be only of the order of a few tenths of a degree and due to dissolved salts in soil water. However, Nersessova (1953-1957) and Lovell (1957) conducted a series of simple calorimetric measurements and showed that the quantities of heat required to be extracted to cool a soil to below 0°C were insufficient to assume freezing of all of the soil moisture content. This drew attention to the fact that a relatively large portion of the soil water remains unfrozen at temperatures below the freezing-point of free water. This stems from the fact that the state of water in the soil is different from that of free bulk water; the soil water being under the influence of capillarity, surface adsorption, and osmotic effects.

In the absence of an external water supply, the main factor governing the proportion of ice and water is the

freezing temperature. The applied external pressure is a less but still significant factor. Other factors which influence the ice-water phase composition of soils are the mineralogical composition and the pore size structure of the soil matrix.

To correctly interpret the behaviour of soils on freezing and the associated change in volume, it is essential to consider the thermodynamic relationships relating to phase equilibria, and the manner in which temperatures, applied external pressures and soil type affect the ice-water phase composition and the behaviour of the two phases.

CHAPTER I

Review of Earlier Work

I-1 The State of Water in Soils

I-1-A The Concept of Suction in Soils

The concept of suction may be considered as expressing, either qualitatively or quantitatively the affinity of soil for water due to a complex interaction of several components. The liquid phase of the soil shares a large amount of interfacial area with the solid phase on the one hand and the gas phase on the other. The interactions between phases exert a profound influence on the movement and retention of water by the soil which in turn affects the distribution of moisture in the soil.

There are two distinct components of suction, matric suction and osmotic suction.

Matric Suction: It results from the presence of capillary forces and the adhesion forces between the liquid and solid phases.

a. Capillarity forces: The phenomenon known as capillarity results from a combination of surface tension of the air-water interface, pore geometry and the tendency of some liquids to wet surfaces they come into contact with; this wetting causes the curvature of the liquid surface. The surface of the liquid meets that of the solid at a definite angle known as the contact angle, θ .

In partly saturated soils, the interface between air and water lies within the soil pores. The soil water at the air-water interfaces will, thus, be under reduced pressure. The pressure difference that exists at the air-water interface is given by the capillary equation

$$P_a - P_w = \frac{2\sigma}{r} \cos \theta$$

In soils, it is generally assumed that the contact angle is zero. Thus, the equation reduces to

$$P_a - P_w = \frac{2\sigma}{r}$$

where, P_a and P_w are the air pressure and water pressure respectively, σ is the interfacial energy, and r is the radius of curvature of the interface which is equivalent to the radius of the pore in which the interface lies.

Since the hydrostatic pressure immediately above the air-water interface does not differ appreciably from that at the free water surface, the water at the interface will be under negative pressure.

b. Forces of adsorption: In clays, the clay lattice carries a net negative charge as a result of the isomorphous substitutions within the interior of the crystal lattice. The net negative charge is compensated by the accumulation of an equivalent amount of ions of opposite sign in the liquid immediately surrounding the particle. These compensating cations have a tendency to diffuse away from the lattice surface.

On the other hand, they are electrostatically attracted to the charged surface of the lattice. The results of these opposing trends is the creation of an atmospheric distribution of the cations in a diffuse electrical double layer. The water next to the particles' surface is strongly bonded to the clay particles and it is generally acknowledged that its properties differ from ordinary water.

Osmotic Suction: Solutions have an affinity for pure solvents. When a pure solvent is placed in contact with a solution through a semipermeable membrane, the solvent will then flow towards the solution. To stop this flow, the solution must be placed under a hydrostatic pressure ΔP_o , called the osmotic pressure. In other words, the osmotic pressure expresses quantitatively the affinity of solutions for pure solvents. For dilute solutions, the osmotic pressure ΔP_o , by Van't Hoff's law, is given to a first approximation by

$$(\Delta P_o)v = mRT$$

where m is the mol fraction of solute in the solution and is equal to $\frac{m_s}{m_l + m_s}$, where m_s is moles solute, m_l , moles solvent, v specific volume of solution, R gas constant, and T absolute temperature $^{\circ}K$.

In soils, dissolved salts are present in the pore water with the result that a certain amount of suction, numerically equal to the osmotic pressure, is developed in the soil water.

This augments that due to capillary or adsorption effects. The osmotic suction can be estimated by the analysis of the pore solution extracted from the soil and by making use of the Van't Hoff's law mentioned above.

I-1-B The Soil Water Potential

According to the above discussion, water in soil is subject to several force fields originating from the presence of the soil-solid phase and dissolved salts. These effects may be quantitatively expressed by assigning potentials to the soil water. The sum of these potentials is designated the total potential of soil water and may be identified with the partial specific Gibb's free energy of the soil water relative to free pure water at the same temperature (Inter. Soc. Soil Sci. No. 23, 1963). The total potential of soil water may be defined as the amount of work that must be done per unit quantity of pure water in order to transport reversibly and isothermally an infinitesimal quantity of water from a pool of pure water at some elevation at atmospheric pressure to the soil water at the same elevation. The total potential is divided into two parts; osmotic potential, and matric potential.

I-2 Thermodynamic Considerations: Ice-Water Phase Equilibrium

From a thermodynamic point of view, if two phases, or more, of a substance are in equilibrium with each other, their specific free energies must be the same.

The absolute Gibbs free energy f of a substance is defined by the two equations.

$$f = e + P.v - T.s = h - T.s \quad (1)$$

These are equivalent because $h = e + P.v$. Here e represents the internal energy, v the specific volume, h the heat content, and s the entropy, of the substance under consideration.

Now the total work dw performed by a system on its surroundings may, in general, be separated into two parts, the work of expansion Pdv against a pressure P and any other mechanical work dw_m that the system might perform. In general, therefore, the total work dw may be represented by

$$dw = Pdv + dw_m \quad (2)$$

Differentiating the first part of equation 1, we have

$$df = de + Pdv + vdP - Tds - sdT \quad (3)$$

according to the First Law of Thermodynamics, we have

$$de = dq - dw \quad (4)$$

Here dq is the amount of heat absorbed by a system from its surroundings. Also from the definition of entropy

$$ds = \frac{dq}{T}, \quad T \text{ is the temp. } ^\circ K \quad (5)$$

combining equations 2 and 5, and substituting into equation 4, we have

$$de = Tds - Pdv - dw_m \quad (6)$$

Substituting (6) into (3), we have

$$df = vdP - sdT - dw_m \quad (7)$$

If the pressure remains constant and if no mechanical work is obtained, equation 7 reduces to

$$df = - s dT \quad (8)$$

or

$$\left(\frac{\delta f}{\delta T} \right)_p = - s \quad (9)$$

Thus, the rate of change of the absolute free energy with respect to change of temperature equals the negative of the specific entropy.

The specific entropy of ice is less than the specific entropy of free water. So, suppose we have bulk ice and water at equilibrium and then the temperature is raised. Equilibrium will be disrupted and, according to equation (9), the free energy of ice would be greater. Equilibrium condition necessitates that all of the ice changes into water. Likewise, when the temperature is lowered, all of the water changes into ice.

I-3 Ice-Water Phase Equilibrium in Freezing Soils

The earlier discussion of the state of water in soils shows that the soil water is influenced by forces of capillarity, surface adsorption and osmotic forces. The free energy of soil water decreases with decreasing water content due to the increasing effect of one or more of the above mentioned forces. So, in contrast to pure free water, the free energy of soil water will be less than the free energy of bulk ice at 0°C.

The soil water, therefore will not freeze at 0°C .

On lowering the temperature below 0°C , ice formation generally commences on the surface and at temperature close to 0°C (provided that the sample is saturated). The part of the soil water filling the larger pores (whose freezing-point is higher than the soil temperature) is supercooled and its free energy is less than the free energy of the formed ice. Accordingly it is in a state of metastable equilibrium. The water in these larger pores freezes as a result of the intrusion of ice formed at the surface, and the heat liberated on freezing raises the temperature of the soil. An equilibrium temperature below 0°C is reached where ice and water co-exist.

On lowering the temperature further, under constant confining pressure, equilibrium will be disturbed. The free energy of the ice decreases according to the equation $\left(\frac{\delta f}{\delta T}\right)_p = -s$, (equation 9). The free energy of soil water also decreases due to two factors; (a) lowering the temperature according to equation (9), and (b) the increasing effect of one or more of the above mentioned forces. However, the free energy of the soil water decreases at a slower rate than that of ice. Equilibrium will be re-established if a portion of the soil water changes into ice until their free energies are equal. Thus, a new equilibrium temperature is reached where ice and water still co-exist. If we continue to lower the temperature, the ice phase will progressively increase at the

expense of the unfrozen water, but it will continue to be in equilibrium with the remaining unfrozen water at each successive equilibrium temperature. So in contrast to pure free water, water in freezing soils progressively freezes at lower freezing temperatures. In other words, we are referring to a depression in the freezing point of soil water.

I-4 The Relation Between the Freezing-Point Depression and Free Energy of Soil Water

Equations relating the change in free energy of soil water and freezing-point depression have been derived through different approaches.

Schofield (1935) thought that in moist soils most of the pore water in the soil is under tension or reduced pressure and the ice first formed is under atmospheric pressure. He gives an equation for calculating the freezing-point depression of soil moisture. He does not give any derivation for it, but mentions that pF^* values he has plotted in his curve were calculated by means of it. This relationship is given by

$$H = \frac{L}{T \cdot g} \Delta T \quad (11A)$$

where, H is the suction expressed as height of a column of water, L is the latent heat of fusion of ice, T is absolute temperature in degrees Kelvin, g gravitational acceleration, and ΔT is freezing-point depression of soil water. The soil

* pF is the logarithm of the suction (expressed in cm of water)

water suction P is given by

$$P = \rho \cdot H \cdot g, \rho \text{ is the density of water}$$

Thus from equation 11A

$$\frac{P}{\rho g} = \frac{L}{T \cdot g} \Delta T$$

But the change in free energy of soil water under isothermal pressure change is given by

$\Delta f = v \Delta P$, v is the specific volume of water and is equal to $\frac{1}{\rho}$. Therefore,

$$\Delta f = \frac{\Delta P}{\rho}$$

or

$$\Delta f = \frac{L}{T} \cdot \Delta T \quad (11)$$

Edlefsen and Anderson (1943) considered the generalized Clausius-Clapeyron equation to find the effect of pressure on the freezing-point of the pore water. They considered the case where the hydrostatic pressure exerted uniformly on ice differs from that exerted uniformly on water. They suggested that for the case of moist, but not saturated soils, the freezing point is changed by changing the pressure on water while the pressure on the ice remains constant. They concluded that if the ice separates from the soil moisture to a pressure of 1

atmosphere, then the relation between the pressure deficiency and the freezing-point depression in the pore water is given by

$$dP_w = \frac{LdT}{Tv_w} \quad (11B)$$

which is identical with equation (11). Edlefsen and Anderson (1943), however, later on suggested that an appreciable amount of the soil moisture is not under tension, rather it is under the surface adsorption forces which entail a hydrostatic pressure. The hydrostatic pressure increases as the surface of the soil particle is approached. They related the observed freezing-point depression in soil moisture to the presence of dissolved salt in pore water and the compression of the water under the influence of the surface adsorption forces. They obtained a formula giving the relationship of the free energy of soil moisture and the corresponding freezing-point

$$\Delta f = RT \ln \frac{P_D}{P_o} - \frac{L\Delta T}{T\Delta v} \quad (12)$$

where

Δf = change in free energy of soil moisture (at the ice-water interface)

R = Gas constant

T = Absolute temperature $^{\circ}K$

P_D = Vapour pressure of ice at temperature T

P_o = Vapour pressure of free water at temperature T

L = Latent heat of fusion

Δv = Difference in specific volumes of ice and water

ΔT_p = The part of the total freezing-point depression T
due to hydrostatic pressure acting on both phases

Williams (1967) considered the case of partly saturated frozen soils. He gives the relation between the freezing temperature and the size of the soil pores

$$L \ln \frac{T}{T_0} = - \frac{v_w \cdot 2\sigma_{iw}}{r_{iw}} + \frac{(v_w - v_i) 2\sigma_{ia}}{r_{ia}} \quad (13)$$

where

L = Latent heat of fusion

v_w & v_i = Molar volumes of water and ice

T = Absolute temperature $^{\circ}K$

T_0 = Freezing-point of free water

σ_{iw} & σ_{ia} = Interfacial energy, ice-water and ice-air

r_{iw} & r_{ia} = Radius of curvature of interface, ice-water
and ice-air

For the case of completely saturated soil, the interface between ice and air is essentially plane, lying at the surface, and $r_{ia} = \infty$. Equation (13), thus, reduces to

$$L \ln \frac{T}{T_0} = - \frac{v_w \cdot 2\sigma_{iw}}{r_{iw}}$$

But

$$P_i - P_w = \frac{2\sigma_{iw}}{r_{iw}} = \Delta P, \text{ the capillary equation}$$

Thus

$$P = \frac{L}{v_w} \ln \frac{T}{T_o} \quad (11C)$$

Equation (11C) can be transformed into equation (11).

Low et al (1968) obtained an equation relating the relative humidity of the soil moisture to the freezing temperature

$$\left(\ln \frac{P}{P_o}\right)_{T=T_a} - \left(\ln \frac{P}{P_o}\right)_{T=T_f} = \frac{(H_1^o - \bar{H}_1)}{R} \left[\frac{T_a - T_f}{T_a \cdot T_f} \right] \quad (14)$$

where $\frac{P}{P_o}$ is the ratio of the vapour pressure of liquid water in the soil to the vapour pressure of pure free water at the same temperature, T_a is any temperature of interest, T_f is the freezing temperature, H_1^o is the molar heat content of pure free water (the standard state), $\bar{H}_1$ is the partial molar heat content of the water in the liquid phase in the soil, and R is the molar gas content. Hence, if $\frac{P}{P_o}$ and $(H_1^o - \bar{H}_1)$ are known at $T = T_a$, $\frac{P}{P_o}$ can be calculated for $T = T_f$.

I-5 Effect of Externally Applied Pressure on Freezing-Point

As mentioned before, if two phases of a substance are in equilibrium with each other, their free energies must be the same. Suppose the two phases are ice and water at 0°C and at atmospheric pressure. If the pressure on the system is changed, the equilibrium is disturbed, and the free energies will no longer remain equal unless other changes are made in the system.

If we recall equation (7):

$$df = v dP - s dT - dw_m \quad (7)$$

If the temperature remains constant and if no work is done on the surroundings, equation (7) reduces to

$$df = v dP \quad (15)$$

or

$$\left(\frac{\delta f}{\delta p} \right)_T = v \quad (15)$$

Since for water, at 0°C , $v = 1.00$, and for ice $v = 1.090$, we have the rate of increase of the free energy of water with respect to pressure is

$$\left(\frac{\delta f}{\delta p} \right)_T = 1.00 \quad (15A)$$

and we have for ice

$$\left(\frac{\delta f}{\delta p} \right)_T = 1.090 \quad (15B)$$

It is evident from equations (15) that an isothermal rise of pressure causes a greater increase in free energy of the ice than it does in water. Equilibrium will, thus, be restored if the ice passes into water. By similar reasoning, since a given decrease in pressure causes a greater decrease of the free energy of ice than it does of water (equation 15) equilibrium will be restored if water passes into ice. Equations (15) predict, therefore, that an increase of pressure dP on the system causes a decrease dT of the freezing-point of water, and vice-versa.

I-6 The Relation Between the Applied External Pressure ΔP and the Freezing-Point Depression ΔT of Soil Water

Edlefsen and Anderson (1943) considered the Clausius-Clapeyron equation to find the relation between the change in pressure dP applied to the soil system and the change in freezing-point of the soil water. They obtained the following relation

$$dp = \frac{L \cdot dT}{T \Delta v} \quad (16)$$

where, L is the latent heat of fusion of ice, T the freezing-point of pure free water, and Δv the change in the specific volume of the water in going from the liquid phase to the solid phase.

For finite changes, equation (16) takes the form

$$\Delta p = \frac{L \cdot \Delta T}{T \cdot \Delta v}$$

Substituting the proper values for the specific volumes of water and ice, and the latent heat of fusion, we find that a total increase in pressure ΔP equal to 1.0 atmosphere lowers the freezing-point of the soil water by 0.00748°C .

Low et al (1968) considered the Kelvin equation

$$F_s - F_l = RT \ln \frac{P}{p_0} \quad (17)$$

to obtain an equation that gives the relation between the externally applied confining pressure and the change in free energy of the pore water, in restoring equilibrium under isothermal conditions. This equation is given by

$$RT \left[\left(\ln \frac{P^\Delta}{P^0} \right)_{P=P} - \left(\ln \frac{P^\Delta}{P^0} \right)_{P=P_a} \right] = (V_s - \bar{V}_1^0) (P - P_a) \quad (18)$$

where, $\bar{F}_s$ is the partial molar free energy of solid water in the soil, $\bar{F}_1^0$ is the molar free energy of water in the standard state, T is any temperature of interest, $\frac{P^\Delta}{P^0}$ is the ratio of the vapour pressure of ice in the soil to the vapour pressure of pure liquid water at the same temperature, V_s and $\bar{V}_1^0$ are the partial molar volume of the ice in the soil and of pure liquid water, respectively, P_a is atmospheric pressure and P is the applied external pressure.

The term on the left-hand side of equation (18) represents the change in free energy of the soil water as a result of the application of an external pressure, P . According to equation (11)

$$\Delta T = \frac{T}{L} \Delta f \quad (11)$$

Substituting the value of f into equation (18) gives

$$\frac{L \Delta T}{T} = (V_s - \bar{V}_1^0) (P - P_a)$$

or

$$\Delta P = \frac{L \cdot \Delta T}{T \Delta v}$$

which is identical to equation (16) derived by Edlefsen and Anderson.

Williams and Mitchell (1969) derived an equation to determine the effect, at a given temperature, of the application of an isotropic external pressure, ΔP , on the change in

free energy, Δf , of the soil water

$$\Delta f = \Delta p (v_i - v_w) \quad (19)$$

where v_i and v_w are the specific volume of ice and water in the soil. The same operation can be performed on equation (19) to show that it is identical to equation (16).

CHAPTER II

Present Research Work

II-1 Objectives

This research work was carried out to investigate the change in volume of freezing soils at different freezing temperatures (isobaric) and under different applied pressures (isothermal). The investigation involved:

1. Determination of changes in the proportion of ice and water with temperature from the experimental measurements of changes in volume of the frozen soil, induced by isobaric temperature changes. The results are compared with those predicted from the free energy-moisture content relationship of the unfrozen soil. This will allow the prediction of changes in volume of frozen soils under isobaric temperature changes.

2. Determination of the effect of applied external pressure on the ice-water phase composition from the experimental measurements of changes in volume of the frozen soil under isothermal pressure changes. The results are compared with those predicted from the free energy-moisture content relationship of the unfrozen soil. This will allow the prediction of the effect of applied external pressure on the isothermal volume change of frozen soils.

II-2 Techniques

II-2-1 Determination of Volume Change of Freezing Soils Using the Dilatometer

A temperature and pressure-controlled dilatometer has been developed for the purpose of the accurate determination of change in volume of soils on freezing. A full description of the dilatometer and auxiliary equipment together with the test procedure are presented in the next chapter.

II-2-2 Prediction of Ice-Water Phase Composition Under Isobaric Temperature Changes

Both the free energy/moisture content relation of unfrozen soil and the free energy/unfrozen water content relation of the same soil on freezing are a characteristic of that soil. The first relation is commonly referred to as the soil water characteristic, and the latter one is known as the soil freezing characteristic.

It has been shown (Miller, 1963 and Williams 1967) that analogies can be drawn between the soil water characteristic and the soil freezing characteristic of a soil. In doing so two extreme types of soils are to be recognized, incompressible soils and completely compressible soils. In incompressible soils, soil particles are in direct solid-to-solid contact. The particles are more or less fixed in place, being wedged

between their neighbours. A change in water content of the soil is accomplished when water present in the pores is displaced by air, or ice, with little or no change in bulk volume. In completely compressible soils, on the other hand, a change in water content is accompanied by a corresponding change in the bulk volume rather than by intrusion of a foreign phase (air or ice) into the pores. They came to the conclusion that, in incompressible soils, for a given water content (water content of the unfrozen soil and the unfrozen water content of the soil on freezing) the air-water or ice-water interface lies in the same pores, i.e. $r_{iw} = r_{aw}$. According to the capillary equation

$$P_a - P_w = \frac{2\sigma_{aw}}{r_{aw}}, \text{ and } P_i - P_w = \frac{2\sigma_{iw}}{r_{iw}} \quad (20)$$

where, P_a is air pressure, P_i ice pressure, P_w water pressure, r pore size, and σ is the interfacial energy. It follows that at a given water content

$$\frac{P_a - P_w}{\sigma_{aw}} = \frac{P_i - P_w}{\sigma_{iw}}, \text{ or } (P_i - P_w) = \frac{\sigma_{iw}}{\sigma_{aw}} (P_a - P_w), \text{ or}$$

$$\Delta f_{\text{unfrozen}} = \frac{\sigma_{aw}}{\sigma_{iw}} \Delta f_{\text{frozen}}^* \quad (21)$$

Thus, for a given incompressible soil, the soil water characteristic and the soil freezing characteristic curves will superimpose if water content is plotted as a function of

$$\frac{P_a - P_w}{\sigma_{aw}} \text{ or } \frac{P_i - P_w}{\sigma_{iw}}.$$

* $\Delta f_{\text{unfrozen}}$ is numerically equal to $(P_a - P_w)$ in C.G.S.

Δf_{frozen} is numerically equal to $(P_i - P_w)$ in C.G.S.

In completely compressible soils, the water content varies as $(P_x - P_w)$ varies and it is immaterial whether x is air or ice. In other words, the soil water characteristic and soil freezing characteristic curves should be identical if water content is plotted as a function of $(P_a - P_w)$ or $(P_i - P_w)$.

A typical soil encountered in the field is likely to be a composite of the two extreme types mentioned. According to Williams (1967), the air intrusion value of the soil would define a boundary. At suctions less than the air intrusion value the soil is regarded as completely compressible. Beyond this value the soil cannot be any more regarded as completely compressible. However, entry of air or ice into the pores does not necessarily prevent further compression of the soil.

Technique

The unfrozen water content-freezing temperature relationship is determined using two different techniques.

a. The Dilatometric technique: In the dilatometer the soil sample is enclosed in a rubber membrane. Accordingly there is no movement of soil water into or out of the dilatometer, the soil sample itself is completely de-aired in the dilatometer. So any observed volume changes will be mainly attributed to water-ice phase change. The observed volume changes are converted into an equivalent weight of ice based on the fact that when water changes into ice its volume increases by 9.05%.

The weight of the ice is subtracted from the total weight of water in the soil sample to find the weight of water remaining unfrozen. Observing the change in volume of the freezing soil at different freezing temperature thus enabled us to obtain the unfrozen water content-freezing temperature relation.

b. The free energy-water content relation technique: The free energy-water content relation for the soils investigated is obtained from the suction plate test, the pressure membrane test, or the high pressure membrane test (a description of the different tests will be presented in a later chapter). If the free energy scale is converted to freezing temperature using the equation

$$\Delta T = \frac{T}{L} \cdot \Delta f \quad (11)$$

the free energy-water content relation can be converted to freezing temperature unfrozen water content relation. These results are to be compared with the results of the first technique.

II-2-3 Prediction of Changes in the Ice-Water Phase Composition Under Isothermal Pressure Changes

As mentioned in Chapter I, article I-5, under isothermal conditions the rate of increase of the free energy of a substance df with respect to pressure, due to the application of additional external pressure dp is given by

$$\left(\frac{\delta f}{\delta p}\right)_T = v \quad \text{equation 15}$$

and for finite change

$$\Delta f = v \Delta P$$

On application of pressure ΔP to a system of frozen soil initially at equilibrium, the free energy of the soil water increases by the amount

$$\Delta f_w = v_w \Delta P_w = 1.0 \cdot \Delta P_w$$

and the free energy of the ice increases by the amount

$$\Delta f_i = v_i \Delta P_i = 1.090 \cdot \Delta P_i$$

Restoration of equilibrium necessitates that $\Delta f_w = \Delta f_i$. In other words the free energy change in the soil water Δf_w is equivalent to

$$\Delta P_w = 1.09 \cdot \Delta P_i$$

that is, a change of pressure of the water greater by 9%, than that of the ice. This is accomplished by melting of part of the ice as discussed in article I-5. On application of pressure ΔP to the frozen soil, both phases experience this pressure change initially. Restoration of equilibrium then requires an additional free energy change $\Delta(\Delta f)$ of the water equals to $0.09 \Delta P_i$. Bearing in mind that $\Delta P = \Delta P_i$, then $\Delta(\Delta f)$ is equal to

$$\Delta(\Delta f) = 0.09 \Delta P \quad (22)$$

Technique: Comparison of observed and predicted effects of pressure change ΔP .

A set of dilatometric measurements are carried out at constant freezing temperature and the applied pressure is changed. The tests are repeated at different freezing temperatures and the soil freezing characteristic curves under different applied pressures are obtained.

The unfrozen water content at a temperature T and under pressure ΔP is predicted as follows. The free energy of soil water at temperature T is calculated from equation 11. This is the free energy $(\Delta f)_{a,T}$ of the soil water at temperature T and under atmospheric pressure. The free energy of the soil water $(\Delta f)_{\Delta P,T}$ under pressure ΔP and at the same temperature T can be obtained by making use of equation 22, and is given by

$$(\Delta f)_{\Delta P,T} = (\Delta f)_{a,T} - 0.09 \Delta P \quad (23)$$

Following the preceding discussion in article II-2-2, the unfrozen water content of the frozen soil at temperature T is the same as the water content of the unfrozen soil at

$\Delta f_{\text{unfrozen}} = K * (\Delta f)_{\text{frozen}}$. Thus the unfrozen water content under pressure ΔP and at temperature T can be obtained from the free energy-moisture content relationship. The predicted unfrozen water content is compared to the unfrozen water content, at the same temperature and under the same pressure, calculated from the dilatometer test results.

* K is a factor equal to unity for completely compressible soils and to $\frac{\sigma_{aw}}{\sigma_{1w}}$ for incompressible soils.

CHAPTER III

The Dilatometer

BACKGROUND

One of the early dilatometric measurements was carried out by Bouyoucos (Bouyoucos 1917). Soil at the desired water content was placed in the dilatometer under an immiscible liquid that would not freeze above about -10°C . The assembled apparatus was then undercooled to a desired temperature. After thermal equilibration, ice nucleation was initiated. The volume change during freezing was observed from the position of the meniscus of the immiscible liquid in the calibrated capillary extension of the dilatometer. This method was subsequently utilized by Buehrer and Aldrich (1946) and Hemwall and Low (1955). A critical assumption involved in this procedure involves the extent to which the immiscible liquid remains completely inert when it covers the sample and fills all the interstices. If the interface so formed is large enough and if partial solubilization or some other interaction occurs, then a potentially large error can be involved (Anderson and Tice, 1970).

INTRODUCTION

A pressure and temperature controlled dilatometer was specially built for the accurate determination of the change

in volume of freezing soils. The soil sample is enclosed in a rubber membrane and placed inside a stainless steel cylinder. The space between the soil sample and the walls of the cylinder is filled with mercury. This arrangement eliminates the above mentioned potential error involved in early dilatometric measurements.

In the early stages of this research programme, experiments were carried out using a brass cylinder. Silicon oil was used instead of mercury. Although the oil was de-aired, large volume changes were observed on the application of low pressure to the dilatometer. Many attempts were made and failed to avoid such sponginess. Only on using mercury and accordingly rebuilding the dilatometer using stainless steel, were volume changes on application of pressure greatly reduced.

This chapter presents a full description of the dilatometer and auxiliary equipment for temperature control, together with test procedure.

III-1 The Dilatometer

It consists of two parts, the cell and the volume measuring assembly.

III-1-1 The Cell

It consists of a stainless steel cylinder - 2.2 in. inner diameter, 4 in. outer diameter, and 4. in. in height - together with two stainless steel end plates, see Fig. 1. The

lower end plate has a drainage port. Two thermoelectric heat pumps (Peltier modules) are attached to the two end plates for cooling the cell, to the desired temperature. The power supply to these modules is controlled by a thermoelectric cooling control unit (Williams, 1968) so that the cell temperature can be maintained constant for suitable periods of time. Nine thermistors are fixed to the wall of the cell at various points to determine the average cell temperature and to check the uniformity of temperature throughout the cell. Another two thermistors are fixed to the two end plates to determine their temperature and thus to adjust their temperature to the desired value, by adjusting the thermoelectric cooling control unit settings. The thermistors were calibrated through a temperature range of 0 to -6°C (accuracy $\pm 0.005^{\circ}\text{C}$), see Appendix VII.

The cell is connected to the volume measuring assembly through a stainless steel tube. It is also connected to the mercury reservoir through a stainless steel valve (valve no. 4), see Figure 2.

III-1-2 The Volume Measuring Assembly

This consists of two pipette tubes, 10 ml. and 1.0 ml., see Figure 2. The two tubes are connected to each other and to the cell by means of stainless steel tubes. A set of valves (valves no. 1, 2, 3) are arranged in such a manner as to allow for the flow of mercury from one pipette tube to the

other and from one pipette tube or both to the cell and vice versa. The 1.0 ml. pipette tube is used for the accurate determination of the displacement of the mercury level inside the pipette resulting from the change in volume of the mercury inside the cell (reading accuracy ± 0.001 c.c.). The 10.0 ml. pipette serves as a reservoir for the flow of mercury to or from the 1.0 ml. pipette. It may be used, however for a rough estimate of the change in volume of mercury inside the cell. The top end of each pipette is connected, through a stainless steel valve (valves 5 and 6) to a nitrogen supply under pressure. The desired pressure is applied as a gas pressure at the top of the 1.0 ml. pipette through the mercury, to the soil sample inside the cell. Each pipette tube is located inside a perspex cylinder filled with silicon oil. The bursting pressure against the wall of the pipette is reduced by applying gas pressure to the oil inside the perspex cylinder. Furthermore, the oil and the glass cylinder provide a safeguard against the explosion of the pipette when high pressure is applied.

III-2 Control of Cell Temperature

Extreme temperature control is necessary to secure a uniform temperature all over the cell and that the cell temperature remains stable for long periods of time. Specifically, it is of extreme necessity to keep the temperature stable when

investigating the effect of changing the applied pressure on the ice-water phase composition.

In order to eliminate any heat exchange from the cell to the surroundings, the cell is wrapped in an insulating material, foam rubber, and placed inside an insulated box, see Figure 2. The air temperature inside the box is brought down to the cell temperature. This is accomplished by circulating a liquid (methanol) at a controlled constant temperature, through a coil of copper tubing placed adjacent to the inside walls of the box. A thermostat is also placed inside the water reservoir to keep the temperature of the cooling water line, passing through the heat sinks, constant.

The recording thermistor's signals are amplified and recorded continuously on a strip chart recorder giving 0.3°C full scale; temperature changes are readable to 0.001°C , see Figure 3. Changes in cell temperature can thus be inspected during the test.

The arrangements mentioned resulted in a uniform temperature all over the cell ($\pm 0.01^{\circ}\text{C}$ deviation from average value). Also, the recorded cell temperature showed a stability better than 0.001°C , for periods up to several hours. It was observed however that the recorded temperature on occasion drifted irregularly within a range of $\pm 0.02^{\circ}\text{C}$. Spot observations made of the other thermistors showed no change in cell temperature. Also it was noticed that there was no effect on observed volume

change. The drift was found to be due to changes in room temperature, draughts, etc. which affected the performance of the recording system.

Preparation of the soil sample and the test procedure are described in Appendix VIII.

CHAPTER IV

Determination of the Soil Water Characteristic

There are a number of techniques for the determination of the soil suction*/moisture content relationship. These are listed in the following table together with the range of suctions which each covers. The different techniques have been fully described by Croney et al (1952).

Technique	pF Range
Suction plate or pressure plate	0 - 3.3
Pressure membrane	0 - 6.2
Centrifuge	3 - 4.5
Freezing-point depression	3 - 4
Vacuum desiccator and Sorption balance	5 - 7
Calibrated electrical absorption gauges	3 - 7

In the research programme we were interested to determine the soil water characteristic curve of the various soils investigated, in the range of pF 2.85 (10psi) to pF 5.15 (2000psi). It was decided to use the pressure plate and pressure membrane techniques.

* The suction is essentially numerically equal to the free energy of the soil water.

IV-1 The Pressure Membrane Technique

In this technique, the soil sample is placed in contact with a cellulose membrane, which is itself in contact with water at atmospheric pressure, see Figure 5. The air pressure surrounding the sample is increased to produce a pressure differential between the soil water and the water in the membrane. Water leaves the sample until equilibrium is reached and the suction of the soil is numerically equal to the applied pressure. The suction/moisture content relationship can then be determined by carrying out tests at increasing applied pressures.

Two types of pressure membrane apparatus were used:

IV-1-1 The Conventional Pressure Membrane Apparatus

The apparatus was originally devised by Richards (1941). It is operated at pressures up to 500psi (pF.4.55), see Figure 6.

In carrying out a test, the soil sample was allowed to dry at room temperature, crushed, and mixed with enough distilled water to bring it to slurry. The rubber diaphragm keeps the soil in contact with the membrane by the application of higher gas pressure above the diaphragm than that surrounding the soil sample. The water leaving the sample therefore moves in the liquid phase rather than the vapour phase and moisture equilibrium is usually reached after 3-4 days.

IV-1-2 The High Pressure Membrane Apparatus

The apparatus was developed at the Road Research Laboratory (U.K.) (Coleman and Marsh, 1961) and is operated at pressures up to 20000psi (pF.6.15). A similar apparatus was constructed at the Division of Building Research, National Research Council of Canada. A full description of the apparatus and its operation is given by Williams (1964). The apparatus is shown in Figure 7.

In carrying out a test, the soil sample is prepared in the same manner as in the conventional pressure membrane test. A teflon tube $\frac{1}{2}$ " diameter by $\frac{1}{2}$ " high is glued to the membrane while it is dry. The membrane is soaked in water, then placed symmetrically on the porous disc. The soil sample is poured to fill the teflon tube. This arrangement prevents any soil particles lying between the membrane and the bottom of the cylinder, which would cause the rupture of the membrane. The cylinder is then bolted in position and the apparatus is mounted in a compression machine with the piston so arranged that the Bridgeman seal is above the side inlet port. Compressed air is then introduced into the cylinder through the port. When the piston is pushed down, the compressed air supply is automatically isolated from the pressure cylinder as the piston passes the port. Thereafter any further downward movement of the piston intensifies the working pressure of the machine.

It was found that the time for moisture equilibrium to be established increases with increasing the applied pressure. It seems that on drying, the soil sample tends to hang to the wall of the teflon tube and to loose contact with the membrane. Periods of 7 days under 500psi applied pressure, 10 days under 1000psi, and 12 days under 2000psi were found to be necessary for establishing moisture equilibrium.

IV-2 The Pressure Plate Technique

It is basically similar to the pressure membrane technique. It is used to determine the soil suction/moisture content relationship at low suction where the pressure membrane technique is not accurate enough. The only difference between the two techniques is that, in the pressure plate technique the soil sample is placed in contact with a porous plate. Moisture equilibrium in the soil is usually reached after 3-4 days. The apparatus is shown in Figure 8.

IV-3 Evaluation of the Contribution of Osmotic Suction to the Free Energy of Soil Water

The total suction or the relative free energy of the soil water includes both the matric suction and the osmotic suction. It is generally agreed that in the pressure plate and the pressure membrane techniques relatively unrestricted passage of dissolved salts in the soil water through the plate or the

membrane occurs. Therefore the contribution of the osmotic forces to the free energy of the soil water is not measured.

The free energy-moisture content relationship was to be determined for the purpose of predicting the unfrozen water content-temperature relationship. The osmotic effects should therefore be considered since they influence the freezing-point depression of soil water and unfrozen water content-temperature relationship.

To account for the osmotic effects the salt concentration in water for the soils investigated was determined and the implied freezing-point depression was obtained, see appendices IV, V.

CHAPTER V

Presentation of Test Results

V-1 Description of Soils Investigated

Four soils were investigated ranging from a sandy-silty-clay to clayey soils. The properties of the different soils are given in the following table. The Oneida clay, $\phi < 5\mu$ was obtained by decanting the Oneida clay soil.

	Sand %	Silt %	Clay %	Clay Mineral	Organic Matter %
Oneida clay	20.5	54.0	25.5	mainly hy- drous mica	not significant
Oneida clay $\phi < 5\mu$	0.0	15.0 v. fine	85.0	mainly hy- drous mica	not significant
Fithian illite	4.5	64.2	31.3	mainly hy- drous mica	not significant
Tanzania clay	5.0	16.0	79.0	montmorill- onite > 60% mica < 20% kaolinite > 20%	6.7

V-2 Dilatometer Test Results

V-2-1 Volume Change of Frozen Soils with Temperature, Under Constant Pressure

The results of the observed volume change of the different soils on freezing under isobaric conditions are given in Tables 1 through 7, see Appendix I. On lowering the temperature of the cell the volume of mercury inside the cell, the volume of soil sample, the volume of air entrapped in the system, and the

walls of the cell undergo a thermal contraction. So the observed volume changes are not the actual volume changes of the soil sample, and a correction has to be introduced to the test results.

Correction for thermal contraction:

The displacement of the mercury level in the pipette of the volume measuring assembly over the range of temperature (from room temperature to initial freezing temperature) before initiation of freezing, was recorded. Considering the thermal contraction to be linear with temperature, a correction factor C was obtained and is given by

$$C = \frac{\text{Displacement of mercury level (CM}^3\text{)}}{\text{Room temperature} - \text{initial freezing temperature, } T_{iF} \text{ } ^\circ\text{C}}$$

The actual volume change of the soil sample at any freezing temperature T_F is equal therefore to

$$\text{observed volume change} + C (T_F - T_{iF}).$$

The corrected volume change of the soil sample (excluding the thermal contraction of ice) and the percent volume change are given in Tables 1 through 7 (Appendix I).

The percent volume changes as a function of temperature for the different soils are shown in Figure 9. The time allowed for reaching thermodynamic equilibrium was 24 hours for each point in the graph.

V-2-2 Effect of Change of Pressure on Volume Changes, at
Constant Temperature

This was investigated by, first, freezing the soil sample (the Tanzania clay) under 10 Kg/cm^2 applied pressure, to a specific temperature, and recording the change in volume. Then the applied pressure was lowered to 5 Kg/cm^2 and volume change was observed again when equilibrium was reached. The pressure was then lowered to atmospheric and volume change observed. The sample was then thawed and refrozen to a lower temperature under 10 Kg/cm^2 and the entire procedure repeated. The results of these tests are given in Table 8, see Appendix I.

A correction has to be introduced to the observed volume change due to the expansion in the volume of air entrapped in the system on releasing the pressure. Table 8, column 4 (Appendix I), gives the observed displacement of mercury level, ΔV , in the pipette of the volume measuring assembly, on application of pressure P at room temperature T . The corresponding displacement of mercury level ΔV_F at the freezing temperature T_F was calculated and is given by

$$\Delta V_F = \frac{T_F}{T} \cdot \Delta V$$
, on the assumption that the ideal gas law is obeyed. The actual volume change of the soil sample was calculated and is given by

Sample volume change = observed volume change - ΔV_F

The results of these tests are given in Tables 8 and 9 (Appendix I) and are plotted in Figure 21.

V-3 Interpretation of the Soil Freezing Characteristic From
Dilatometer Test Results

As outlined in Chapter II, article II-2-2, the unfrozen water content of freezing soil as function of freezing temperature can be calculated from the dilatometer test results. The results of these calculation for the different soils are given in Tables 9 through 16 (Appendices I, II), and are shown in Figures 15 to 18.

V-4 Soil Suction-Moisture Content Test Results

Results of the pressure plate, pressure membrane and high pressure membrane tests for the four soils investigated are given in Tables 17 to 20 (Appendix III). The soil suction-moisture content relations for the unfrozen soils (also known as the free energy-moisture content relation or the soil water characteristic) are shown in Figure 14.

V-5 Interpretation of the Soil Freezing Characteristic From
the Soil Water Characteristic

As outlined in Chapter II, article II-2-2, the soil water characteristic can be converted into the soil freezing characteristic by making use of the two relations

$$(P_1 - P_w) = \frac{\sigma_{1w}}{\sigma_{2w}} (P_a - P_w) \quad (\text{equation 21})$$

$$\Delta T = \frac{T}{L} (P_1 - P_w) \quad (\text{equation 11})$$

It was also mentioned in article IV-3 that the pressure membrane and pressure plate techniques measure only the matric component of the total soil suction. The freezing-point depression ΔT_m resulting from the matric component of the soil suction was calculated using the above two relations. The salt concentration in the soil water was determined and the corresponding freezing-point depression was obtained (see Appendices IV, V).

The results of these calculations are given in Tables 21 to 24 (Appendix V). The unfrozen water content/freezing temperature relations for the different soils are shown in Figures 15 to 18.

CHAPTER VI

Discussion of Test Results

VI-1 Volume Change of Frozen Soils

Figure 9 shows percent volume changes of different soils investigated as function of temperature, under atmospheric pressure (zero gauge pressure). Figure 10 shows the associated unfrozen water content calculated from observed volume changes.

Reference to Tables 13 and 16 (Appendix II), we note that the initial water contents for the Oneida clay, $\phi < 5\mu$ and the Tanzania clay are 85.24% and 109.41% respectively. The Oneida clay, $\phi < 5\mu$ exercised, however, larger volume changes on freezing than did the Tanzania clay, see Figure 9. This is not unusual since the clay mineral of the Oneida clay is mainly mica while the predominant clay mineral in the Tanzania clay is montmorillonite, article V-1; and accordingly the Tanzania clay would have finer pore-sizes, and thus greater unfrozen water content, than the Oneida clay, see Figure 10. This is in agreement with earlier findings (e.g., Nersesova and Tsytovich, 1963) which demonstrated that the mineralogical nature of the soil matrix influences the amount of soil water that remains unfrozen.

The effect of the pore size on volume changes and the associated unfrozen water content is clearly demonstrated by the volume change curves of the Oneida clay and the Oneida clay,

$\phi < 5\mu$ (prepared by decantation), Figure 9. The two soils have basically the same mineralogical and chemical formation, yet they exhibit different amounts of volume changes on freezing.

VI-2 Effect of Temperature on Volume Change of Frozen Soils

In Figure 9, the volume changes observed in the dilatometer, under atmospheric pressure, as a function of temperature are shown for the different soils investigated. The associated unfrozen water contents at different temperatures were calculated from the observed volume changes as outlined earlier in article II-2-2. Such information was plotted as the unfrozen water content versus temperature, Figures 15 to 18.

To examine the relationship between the free energy of soil water and freezing temperatures, predictions of the unfrozen water content as a function of freezing temperature have been made, using determinations of free-energy-moisture content relationship, as outlined in article II-2-2. The unfrozen water content-temperature relationship predicted by this procedure is compared to that determined by the dilatometer observations in Figures 15 to 18.

The agreement between the predicted unfrozen water contents and those implied by the measured volume changes over the whole temperature range (0°C to -5°C) is good, Figures 15 to 18. The predicted values were obtained with the use of the factor $\frac{\sigma_{1w}}{\sigma_{aw}} = 0.42$ in equation 21 for each point. In predicting

the unfrozen water content-temperature relationship from the free energy-moisture content relationship, the contribution of dissolved salts to the freezing-point depression of soil water was evaluated on the assumption that the formed ice does not contain any significant amount of dissolved salts (see Appendix V). It is not impossible however that the formed ice contains occluded pockets of salt solution, and the actual salt concentration associated with each quantity of unfrozen water is less than the calculated value. It is also likely that in the pressure membrane apparatus some of the dissolved salt is restricted from passing through the membrane; and therefore part of the osmotic suction is measured in the apparatus. One expects therefore that the agreement between the predicted and calculated unfrozen water content will even be closer.

In earlier work (Miller 1963, Koopmans and Miller 1966, Williams 1967), it was shown that if the suction developed in the unfrozen water is less than the air-intrusion value of the soil the relevant factor will be unity. At suctions higher than the air-intrusion value a factor in the range of 0.42 to unity is required.

The air-intrusion values were determined for two of the soils investigated (see Appendix VI) and were found to be in the order of 8, and 16 Kg/cm² for the Fithian Illite and Oneida clay, $\phi < 5\mu$, respectively. It was not experimentally possible to determine the air-intrusion value for the Tanzania clay.

However, the results of the experimental work suggest that it

is higher than 16 Kg/cm^2 , see Appendix VI. When the air-intrusion values were converted to the temperature scale (equations 21 and 11), these values would correspond to -0.27°C , -0.54°C , and lower than -0.54°C , for the Fithian Illite, Oneida clay, $\phi < 5\mu$, and Tanzania clay, respectively. In contrast to what was expected, the agreement between the predicted and observed unfrozen water content, at temperatures higher than those values, was obtained using the factor 0.42.

The agreement between the predicted and observed values of the unfrozen water content with the use of the factor 0.42 suggests that the ice formed at freezing temperatures very near to 0°C intruded through the soil matrix as temperature was lowered regardless of attainment of the air (or ice) intrusion value of the soil. It further suggests that, on freezing a clayey soil behaves in a manner similar to a granular soil.

It is relevant at that point to introduce the concept of clay-packet formation on drying since it is recognized that soil freezing is a drying process (i.e., as water is converted to ice, the remaining water is under the influence of increasing capillary, adsorption, and osmotic forces, just as is the case during drying).

Clay-packet formation in clays on drying

Michaels (1959) has suggested that as a clay dries and the air-water menisci retreat into the soil matrix the clay tends to form into "packets". This idea is confirmed by the

well known phenomenon of "micro-shattering" encountered in most desiccated clays (Burland 1961(b)). A clay which is micro-shattered consists of numerous small grains which are easily mistaken for sand grains but which, when rubbed in the palm of the hand with water, break down into clay.

Some electron micrographs obtained by Burland (1961(a)) have indicated this packet formation. They have shown further that many "levels" of soil structure can exist in which larger packets are subdivided into smaller packets presumably down to the micro size. The process is probably a continuous one right through from large blocks to the lowest level where the clay particles are found to exist as domains.

Microscopic examination of the soil structure of compacted Kaolin and West Water clay carried out by Barden and Sides (1970) revealed a marked difference in structure wet and dry of optimum moisture content. Observations with the naked eye revealed an apparently homogeneous structure wet of optimum; but revealed 0.3 - 0.6 cm diameter pellet-like macropeds dry of optimum. Mitchell, Hooper, and Campanella (1965) earlier reported that similar macropeds were formed during compaction even after carefully controlled hand mixing, and at increasing moisture contents they reported the disappearance of such peds.

Interpretation of Test Results

In the discussion of article I-3 it was mentioned that in a soil at freezing temperature very near to 0°C, ice formation

commences on the surface. The soil water which fills the larger pores on the other hand is supercooled and in a state of metastable equilibrium. Formation of ice takes place only in the larger pores where ice nuclei will be stable. Two types of forces are then developed:

- a) compressive forces resulting from the expansion of water on freezing (the water is confined into the pores), and
- b) tensile forces resulting from the negative pore water pressure developed at the ice-water interfaces.

These forces would tend to cause the clay matrix to shatter into "packets", as described above. The ice will then intrude in between the formed packets. The process of clay shattering and the formation of clay-packets will continue as the soil temperature is lowered. It is reasonable to assume that the first formed packets will be of considerable size, but will continue to subdivide into smaller packets as the temperature is lowered. Not until the soil temperature is substantially lowered will the ice intrude through the soil pores within the individual packets. Augmenting the effect of this process, the negative pore water pressure developed at the ice-water interfaces creates a soil water suction which pulls the soil particles within each clay-packet into a closer packing; accordingly extracting the pore water out of smaller pores within the packet toward the ice which has intruded between the clay-packets. The extracted water freezes.

Observations with the naked eye of a sample of frozen Tanzania clay revealed the formation of cubes of frozen soil of varying sizes separated by layers of ice lenses, see Figure 19. These observations confirm the formation of macroscopic clay-packets during freezing of this soil.

In conclusion, packets are formed when clay soils freeze. The ice intrudes through the soil matrix with the ice-water interfaces representing the boundary between the clay-packets and the ice in the larger pores. At substantially lower temperatures, the ice intrudes through the soil pores within the individual packets.

Based on the data obtained, the thermodynamic relation between the free energy of the soil water and freezing temperature (equation 11) governs the proportion of ice and water in a system of frozen soil, down to -5°C .

It is further concluded that the unfrozen water content-temperature relationship is related to the free energy-moisture content relationship by the relation $\Delta f_{\text{frozen}} = 0.42 \Delta f_{\text{unfrozen}}$ down to -5°C .

These findings enable the interpretation of the unfrozen water content of a soil as function of temperature from the free energy-moisture content relationship, down to -5°C . They further enable the evaluation of the change in volume of a soil on freezing induced by isobaric temperature changes.

VI-3 Assumptions and Limitations in the Application of the
Thermodynamic Analysis

The equations used in the thermodynamic analysis namely

$v_i = 1.09 v_w$, v_i and v_w are specific volumes of ice
and water

and

$$\Delta f = \frac{L}{T} \cdot \Delta T \quad (\text{equation 11})$$

are quite general. In applying them to frozen soil systems it is necessary to consider the manner in which the quantities in the equations may differ from those usually accepted for water and ice. Such differences will arise, in certain cases, as an effect of the proximity of the water (ice) to the soil mineral particles.

Anderson and Low (1958 a,b) investigated the density of water adsorbed on clay minerals and found the density to be lower than normal as the particle surface was approached. Later studies by Jorgenson (1967), however, showed the quantity of water so affected to be very small. It is, therefore, reasonable to assume that the density of soil water at temperatures down to -5°C does not differ from that of free water and correction need not be made to the values of v_i and v_w .

Similar reasoning may be applied to the question of the latent heat of fusion (equation 11). Investigation by Williams (1963), and Low, Anderson and Hoekstra (1968), based on heat associated with wetting showed that at temperatures

below -5°C the unfrozen water has a significantly and continually reduced latent heat. Water freezing at -4.5°C however can be regarded as having a latent heat of fusion essentially equal to that of normal free water.

6

VI-4 Accommodation of Volume Changes in a System of Frozen Soil

Williams (1976) mentioned the possibility that in small samples volume changes would be accommodated by movement of water to or from ice layers on the sample surface, thus avoiding significant changes in the soil mineral matrix.

In order to investigate this possibility, a test was carried out on a sample of Tanzania clay in the dilatometer. The soil sample was initially frozen at a temperature very near to zero. After equilibrium was reached, the temperature was lowered in steps allowing for equilibrium to be reached between each temperature change. After termination of the test, the soil sample while still frozen was inspected. Observations with the naked eye revealed that the majority of the frozen water existed as layers of ice-lenses distributed all through the soil sample. These observations indicate that it is unlikely the change in volume of frozen sample is accommodated by movement of water to ice layers on the sample surface. Recent experiments by Williams and Burt (1974) have given values for hydraulic conductivity of the unfrozen water. The obtained values indicate that it is unlikely for the flow of

water to occur of sufficient magnitude to transfer the water, displaced by the volume change to the surface of the sample, in the time to reach thermodynamic equilibrium (less than 24 hours).

The question of the accommodation of volume changes by the soil system was further investigated. A test was carried out on a sample of Tanzania clay in the dilatometer. The sample was initially frozen. After equilibrium was reached the cell temperature was lowered in steps. At each temperature change, the rate of volume change with time was observed and recorded until equilibrium was reached. The soil temperature was continuously recorded, using a thermistor inserted into the soil sample and coupled to the temperature recording equipment. A sample of the recorded rate of volume change together with the rate of change of soil temperature with time is shown in Figure 20.

In Figure 20, we note that in lowering the temperature from -0.627°C to -0.824°C , 93.5% of the total change in volume was achieved after 11.5 hours from the time of lowering the cell temperature. The equalization of soil temperature (thermal equilibrium) was achieved after 16 hours. These results suggest that the time taken to reach thermodynamic equilibrium (cessation of volume change) is more or less the same as that for thermal equilibrium.

It is relevant at that point to present the concept of

"grain contact stability" introduced by Burland (1961 b). In unfrozen soil, application of external loads under fully saturated drained conditions causes both shear and normal forces to develop at each grain contact point. Under the action of these forces the grains take up a closer pack by rolling and sliding. The process of drying the soil has a totally different effect on the soil structure. During drying, a clayey soil may be visualized as consisting of sand and silt grains together with "grains" of clay packets. The formed air-water menisci at the grain contact points tend to bond the grains together thereby increasing the rigidity of the soil mass. On the other hand, the clay particles of the individual packets will be forced into closer pack under the influence of the developed negative pore water pressure. If the soil mass is wetted the air-water menisci disappear. Volume change takes place not only as a result of grain contact slippage but also by the swelling of the clay packets. The extent to which either effect predominates will depend on the clay content and the clay mineralogy.

In the freezing experiment described above, the cell temperature was brought down to below 0°C without freezing taking place. The soil temperature was then allowed to equalize all through the soil sample. Freezing was then initiated. On freezing, packets of clay were formed. The early formed ice intruded between the formed packets as well

as through larger pores. The soil grains (sand and silt particles and clay packets) will be bonded together by the high curvature menisci formed at the grain contact points, thus, restricting grain slippage. However, the highly developed negative pore water pressure at the ice-water interface would force the clay particles of the individual packets into closer pack extracting the water out of smaller pores within the packet toward the ice which has intruded between the clay-packets. The extracted water froze. This process continued until thermodynamic equilibrium was reached.

The next step in the dilatometer test was to lower the cell temperature. The outer surface of the soil sample felt the temperature change earlier than the core. Accordingly a thermal gradient was set up between the core of the sample and the outer surface. The free energy of the soil water at the outer surface will be less than that at the core (according to equation 9). The water would therefore tend to migrate towards the outer surface. In doing so it would come in contact with the ice formed in larger pores and/or between the clay-packets, and freezes before reaching the outer surface of the sample. In the mean time, the early formed clay packets will continue to subdivide into smaller packets and the ice-water interface will come to lie between the newly formed packets and in smaller pores. On the other hand, the clay particles of the individual packets will continue to compress

into closer pack and the extracted water freezes.

The process of migration of water and water freezing would continue until the soil temperature equalizes inside the sample, and then ceases. Also the intrusion of ice into smaller pores and/or between newly formed clay packets would cease when thermal equilibrium is reached. Volume changes, however might continue at a very slow rate presumably over a period of days due to the further compression of clay packets and the resulting freezing of the extracted water.

The above discussion of the manner in which the volume changes are accommodated by the soil system can be compared to the consolidation process of unfrozen soils. The cessation of volume change (volume changes resulting from the intrusion of ice together with migration of water) on the equalization of soil temperature is analogous to the termination of primary consolidation on the dissipation of excess pore-water pressure. The continued compression of the clay particles within the individual packets and the resulting freezing of the extracted water is analogous to the secondary consolidation phase.

VI-5 Effect of Pressure on Volume Changes

Figure 21 shows dilatometric observations of change in volume of frozen Tanzania clay due to isothermal pressure changes, and Figure 22 shows the associated changes in unfrozen water content. These results are compared with those predicted

from the free energy-moisture content relationship of the unfrozen soil as outlined in article II-2-3.

To illustrate the procedure for comparing the predicted and observed unfrozen water content under a certain applied pressure, consider the soil temperature T is -0.5°C . From equation 11, the free energy* of the soil water at -0.5°C and under atmospheric pressure relative to pure free water at 0°C and 1 atmosphere, is equal to 6.25 Kg/cm^2 . So the free energy of the soil water at -0.5°C and under 5 Kg/cm^2 applied pressure is given by

$$(\Delta f)_{5, -0.5} = 6.25 - 0.09 \times 5 = 5.8 \text{ Kg/cm}^2$$

From equation 21 we note that the unfrozen water content of the frozen Tanzania clay at -0.5°C and under 5 Kg/cm^2 applied pressure is the same as the water content of the unfrozen soil at $\Delta f_{\text{unfrozen}} = 2.4 \times 5.8 = 13.92 \text{ Kg/cm}^2$. Reference to Figure 23 shows for Tanzania clay, a water content accordingly of 42.8%. This is the predicted unfrozen water content at -0.5°C for 5 Kg/cm^2 applied pressure. Reference to Figure 22 we find that the unfrozen water content, calculated from the observed volume change of the soil sample in the dilatometer, at -0.5°C for 5 Kg/cm^2 applied pressure is 42.8%.

Results of the predicted unfrozen water content at different temperatures and under 5, and 10 Kg/cm^2 applied pressure

* It is common practice to express Δf in pressure units in C.G.S. since Δf is numerically equal to change in pressure under isothermal conditions.

calculated by the procedure just described, are compared with those calculated from the observed volume changes, in the following table.

Temperature °C	Unfrozen Water Content under 5 Kg/cm ² Applied Pressure		Unfrozen Water Content under 10 Kg/cm ² Applied Pressure	
	Predicted	Observed	Predicted	Observed
	%	%	%	%
	Dry Wt.	Dry Wt.	Dry Wt.	Dry Wt.
-0.2	51.2	53.2		56.0
-0.3	48.5	47.4	50.0	48.2
-0.4	44.7	44.4	45.1	45.0
-0.5	42.8	42.8	43.4	43.4
-0.6	41.6	41.6	42.0	42.0
-0.7	40.6	40.5	41.0	41.0
-0.8	39.8	39.7	40.1	40.0

As shown in the table, the agreement between the observed values and those predicted, from the thermodynamic analysis, is very good at lower freezing temperatures (-0.4°C and lower). At -0.2°C and -0.3°C the agreement is not as good. Bearing in mind that in that range of temperature, 0°C to -0.3°C, the change in volume of the frozen system is extremely sensitive to any temperature changes and, hence high precision of temperature measurement and control is required; the agreement with the predicted values is still reasonable.

Note on Figure 23

The suction-moisture content curve for the Tanzania clay that appears in Figure 14 is the drying curve associated with

initial freezing and therefore is not appropriate since the freezing curves in Figure 22 are those associated with second and subsequent freezings of the soil sample (the effect of initial and subsequent freezings of soils on volume changes is discussed in article VI-6). The free energy-moisture content curve itself (Figure 23) was therefore determined from the unfrozen water content-temperature relationship, the atmospheric pressure curve in Figure 22, using equations 11 and 21 (based on the conclusion that the unfrozen water content-temperature relationship is related to the free energy-moisture content relationship, article VI-2). It has the advantage of removing the uncertainty arising in evaluating the contribution of dissolved salts to the freezing-point depression of soil water, discussed earlier in article VI-2.

Alternative Procedure for Comparison

Figure 21 shows volume changes of frozen Tanzania clay under 0, 5, and 10 Kg/cm² applied pressure and Figure 22 shows the implied unfrozen water contents. Reference to the two figures, we note that the three curves are similar in form but each one is displaced from the other by an approximately constant temperature difference, ΔT , generally ranging from 0.03°C to 0.04°C.

The value of ΔT can be predicted. If we recall equation 11, we note that the free energy of the soil water Δf , is given as a function of the freezing-point depression ΔT . ΔT in equation 11 is the depression in the freezing-point of the

soil water relative to the freezing-point of pure free water at atmospheric pressure; that is

$$\Delta T = T_0 - T$$

where T_0 is the freezing point of pure free water at atmospheric pressure, and T is the temperature of the frozen soil.

Equation 11 therefore refers to the case where the ice phase is pure and at atmospheric pressure. Consider now the case of frozen soil under an applied pressure, e.g., 5 Kg/cm^2 .

Clearly the ice is not at atmospheric pressure, and in applying equation 11, T_0 and accordingly ΔT must be modified. As noted earlier in article I-5, the application of an additional external pressure ΔP lowers the freezing point of the water by an amount $(\Delta T)_p$ given by

$$(\Delta T)_p = \frac{T}{L} \cdot \Delta P \cdot \Delta v \quad (\text{equation 16})$$

The value of $(\Delta T)_p$ is 0.0374°C for 5 Kg/cm^2 applied pressure.

Consider the commencement of freezing of a saturated sample at atmospheric pressure, and then instead, at 5 Kg/cm^2 . In the latter case freezing will commence at a temperature lower by 0.037°C . Furthermore, as freezing continues the fall in unfrozen water content will continue to lag behind that occurring in the atmospheric pressure case. The course of the water content versus temperature curves will be entirely similar (it is the same soil and the same factors governing Δf) but the 5 Kg/cm^2 curve will be consistently displaced by 0.037°C .

In Figures 21 and 22, consider the two curves for applied pressures of 5 Kg/cm^2 and 0 Kg/cm^2 , or 10 Kg/cm^2 and 5 Kg/cm^2 . It is predicted they will differ by $\Delta T = 0.037^\circ\text{C}$. The observed value ranges from 0.03°C to 0.04°C . Bearing in mind the high precision of temperature measurement and control required, the agreement is good.

VI-6 Initial and Subsequent Freezing of Soils

A series of tests were carried out on samples of Fithian Illite and Oneida clay, $\phi < 5\mu$. The soil sample was first frozen in the dilatometer under atmospheric pressure (zero gauge pressure) and the temperature was lowered in steps down to about -5.5°C . Volume changes associated with temperature changes were observed and recorded when equilibrium for each step was reached, Tables 2 and 4 (Appendix I), Figures 11 and 12. The soil sample was then allowed to thaw. The same sample was refrozen under 5 Kg/cm^2 applied pressure and again the temperature was lowered in steps and the corresponding volume changes were recorded, Tables 3 and 5 (Appendix I), and Figures 11 and 12. The same test sequence was repeated for the Oneida clay, $\phi < 5\mu$ under 10 Kg/cm^2 applied pressure, Table 6 (Appendix I), Figure 12.

Contrary to what is theoretically predicted (articles I-5 and I-6), the application of additional pressure did not result in decreasing the observed volume changes relative to

these observed under atmospheric pressure. The fact that the curves of volume changes under atmospheric pressure corresponded to the initial freezing of the soil sample and those under higher pressure were associated with subsequent freezings explains the unexpected results obtained.

It is recognized that soil freezing is analogous to drying of the unfrozen soil and thawing is analogous to a wetting process. In Figure 13 (after Croney et al, 1958), curve D refers to a soil drying to oven-dryness from an initially slurried condition, curve B is the rewetting from oven-dryness, and curve C is the second drying curve. The figure shows that for a given soil suction the moisture content of the soil drying from an initially slurried condition is higher than the moisture content of the same soil undergoing second drying. This is attributed to the preconsolidation of the soil associated with initial drying due to the influence of increasing capillary, adsorption, and osmotic forces in the soil on drying. The increasing suction developed forces the soil particles into closer packing, and the original arrangement is never completely restored on rewetting.

In the freezing experiment described above, the soil sample was initially slurried and allowed to freeze under atmospheric pressure. As the soil temperature was lowered below freezing and ice nuclei developed, the increasing suction developed in the soil extracted soil water from the smaller

pores towards the growing ice nuclei in the larger pores and forced the soil particles into closer packing. This process continued until the lowest temperature was reached. When the soil sample was thawed and then refrozen under 5 Kg/cm^2 , the following considerations apply:

- a) the externally applied pressure will lower the freezing-point of the soil water (according to equation 16) thus causing less water to freeze and accordingly decreasing the observed volume change relative to the atmospheric pressure case, and
- b) the soil particles were again forced into a closer packing and the freezing of the extracted water added to the volume change. The effect of soil compression overweighed the freezing-point depression effect with the net result that the observed volume changes during second and subsequent freezings (under higher pressures) were greater than those observed during the initial freezing (under atmospheric pressure).

The effect of soil compression associated with freezing on volume change is more pronounced in the case of Tanzania clay. In Figure 21, the dashed curve represents volume change of the frozen Tanzania clay associated with initial freezing, and the open circles represent the volume change of the same soil sample associated with subsequent freezings, both under atmospheric pressure. The two curves are almost parallel with the initial freezing curve lying below.

The results suggest that because of the compression of

the soil produced by the negative pore pressures developed on freezing, and the associated irreversible change of pore structure, second and subsequent freezings of a soil sample tend to increase the observed volume changes relative to those associated with initial freezing. This process can not go on indefinitely. After a number of cycles of freezing and thawing, the soil should cease to compress and subsequent freezing would exhibit the same volume changes.

The test sequences described above were carried out for the purpose of investigating the effect of external pressure on volume change of frozen soils. The results did not reveal that aspect and another technique was adopted.

VI-7 Significance of the Research Work from the Engineering Standpoint

VI-7-1 Significance of the Unfrozen Water Content-Temperature Relationship

From the engineering point of view, a knowledge of the change in the unfrozen water content with the change in freezing temperature is of great importance. During freezing or thawing of soils, the latent heat associated with ice-water phase change is evolved or absorbed progressively as freezing or thawing proceeds. One of the major problems in performing calculations of frost penetration depth and, the thawing of permafrost due to heat transferred from foundations, wells,

pipe lines ... etc., has been a lack of understanding of how to treat the latent heat term. The engineering approach to deal with the latent heat term is to consider that the total amount of latent heat is evolved or absorbed instantaneously on the assumption that the soil water is completely frozen at 0°C , e.g., the Stefan and Neumann approaches. Bearing in mind that the latent heat term is eighty times the specific heat of water, it becomes clear that the assumption while it might be reasonable in case of granular soils, it is absolutely not true in case of fine grained soils.

Calculations of frost penetration depth or thawing of permafrost require a knowledge of the heat capacity of frozen soils. The heat capacity is expressed as the sum of the heat capacities of the soil components plus a term to account for the latent heat associated with the ice-water phase change. This term is given by

$$\int_{T_1}^{T_2} L \cdot \frac{\delta W_u}{\delta T} \cdot dT$$

where, L is the latent heat of fusion, T_1 and T_2 are freezing temperatures and $\frac{\delta W_u}{\delta T}$ is the rate of change of the amount of unfrozen water with respect to freezing temperature. It is worth noting that the term $\frac{\delta W_u}{\delta T}$ represents the slope of the unfrozen water content versus temperature curve. In other words, it is the rate of change of the amount of unfrozen

water, and not the water content of the unfrozen soil, which governs the rate of frost penetration or rate of thawing.

VI-7-2 Determination of the Unfrozen Water Content-Temperature Relationship

This has been determined using various techniques (Anderson and Tice, 1970). The majority of these techniques suffer from difficulties and complexities associated with their performance. Other techniques, e.g., surface area measurements technique devised by Anderson and Tice (1972), are not applicable within the 0°C to -5°C temperature range which is of major geotechnical interest.

It was established by Williams (1964 b, 1967 a) that the thermodynamic relation $\Delta f = \frac{L}{T} \Delta T$ (equation 11) is valid for a system of frozen soil, down to -1°C . The results of this investigation showed that the thermodynamic relation (equation 11) is still applicable down to -5°C .

Validation of the thermodynamic relation for a system of frozen soil provides a new technique for determination of the unfrozen water content-temperature relationship from the free energy-moisture content relationship, to -5°C . Such a technique is simple enough and economic in operation.

VI-7-3 The Pressure State of Water and Ice in Frozen Soils

The thermodynamic relationship between the free energy of soil water and temperature (equation 11) facilitates evaluation

of the water pressure at different freezing temperatures. Also, the effect of the application of external pressure to the frozen soil matrix in changing the free energy of the soil water is explained by the thermodynamic relation represented by the equation $\Delta f = v\Delta P$. Qualitatively speaking, the application of external pressure would have the same effect as raising the soil temperature, both effects would increase the free energy of the soil water, and vice versa.

Williams (1976) suggests that the ice of the frozen soil is more correctly to be identified with the pore fluid, than with the solid matrix. He argues that although the pressure state of the ice is different from that of the water at temperatures below 0°C , it is not possible to change the pressure on either ice or water without producing an equivalent change (in free energy) on the other. So although ice masses (larger than pore size) in frozen ground carry the overburden pressure and thus might be thought to be comparable to solid bodies in an unfrozen soil, the situation is not an equilibrium one if there are neighbouring ice bodies under lower stresses. Migration of ice will occur towards zones under lower stresses.

The determination of the pressure state of soil water is of prime importance in the evaluation of the mechanical properties (volume changes, permeability, and strength) of a frozen soil system. It is also of prime importance in the evaluation of frost heaving in soils (Williams, 1966).

VI-7-4 The Creep Phenomenon in Frozen Soils

The application of an external load to frozen soil will result in increasing the free energy of the soil water within the stressed zone. The establishment of a pressure gradient between the stressed and the unstressed zone will result in movement of unfrozen water towards the unstressed zone. As the water leaves the stressed area, the stressed ice will have a higher free energy than the decreasing amount of water. Thus additional amounts of ice will melt. Outside the stressed zone, on the other hand, the influx of water will tend to increase the free energy of water, such that water builds up into additional ice. In other words there will be a transference of ice masses from the stressed to the unstressed zones. This is identified as a creep phenomenon and was demonstrated experimentally by Vialov (1959), and others.

Changes of temperature will have similar effects. The establishment of a temperature gradient in frozen ground, because of the different free energies of the water associated with different temperatures (equation 11), will result in migration of ice towards the colder parts.

The migration of ice will occur only over long periods of time since the hydraulic conductivity (permeability) of frozen soils is very low (Williams and Burt, 1974). Harlan (1974) has pointed out the unstable nature of permafrost particularly under natural conditions where pressure and temperature

gradients occur universally.

The creep phenomenon has a great implication in field considerations. The effect of disturbing the thermal regime of permafrost imposed by pipelines, foundations, and oil wells will produce slow but ultimately extremely significant volume changes of the adjacent frozen soil.

LIST OF REFERENCES

- Anderson, D.M. and Low, P.F. (1958 a) The Density of Water Adsorbed by Lithium-, and Potassium-Bentonite. Soil Science Society of America Proc. 22, pp. 99-103.
- Anderson, D.M. and Low, P.F. (1958 b) The Partial Specific Volume of Water in Bentonite Suspension. Soil Science Society of America Proc. 22, pp. 22-24.
- Anderson, D.M. and Tice, A.R. (1970) The Unfrozen Interfacial Phase in Frozen Soil Water System. Proc. Inter. Symp. Soil Water Physics and Technol., Rehovoth, Israel.
- Anderson, D.M. and Tice, A.R. (1972) Predicting Unfrozen Water Contents in Frozen Soils From Surface Area Measurements. H.R.B., H.R.R. No. 393, pp. 12-18.
- Barden, L. and Sides, G.R. (1970) Engineering Behaviour and Structure of Compacted Clay. Proc. ASCE, 96, SM4, pp. 1171-1200.
- Bouyoucos, G.J. (1917) Classification and Measurement of the Different Forms of Water in Soil by Means of the Dilatometer Method. Mich. Agric. Coll. Exp. Stat., Tech. Bull. No. 36, 48 p.
- Buehrer, T.F. and Aldrich, D.G. (1946) Studies in Soil Structure, VI. Water Bound by Individual Soil Constituents as Influenced by Puddling. University of Arizona, Tech. Bull. No. 110.
- Burland, J.B. (1961 a) Discussion, Sec. I, Proc. 5th Inter. Conf. Soil Mech. and Fdn. Engng., Vol. III, p. 120.
- Burland, J.B. (1961 b) Some Aspects of the Mechanical Behaviour of Partly Saturated Soils. Symposium on Moisture Equilibrium and Moisture Changes in Soils Beneath Covered Areas, Butterworths, Australia, p.p. 270-278.
- Coleman, J.D. and Marsh, A.D. (1961) An Investigation of the Pressure-Membrane Method for Measuring the Suction Properties of Soil. Journal of Soil Science, Vol. 12, No. 2, pp. 343-362.

- Croney, D., Coleman, J.D., and Black, W.P.M. (1958) Movement and Distribution of Water in Soil in Relation to Highway Design and Performance. H.R.B. Special Report No. 40, p.p. 226-252.
- Croney, D., Coleman, J.D., and Bridge, P.M. (1952) The Suction of Moisture Held in Soil and Other Porous Materials. Road Research Tech. Paper No. 24, London, H.M.S.O.
- Edlefsen, N.E. and Anderson, A.B.C. (1943) Thermodynamics of Soil Moisture. Hilgardia, Vol. 15, No. 2, pp. 31-156.
- Harlan, R.L. (1974) Dynamics of Water Movement in Permafrost: A Review. Proc. Workshop Seminar, Permafrost Hydrology, Canadian Nat. Com., Inter. Hydrol., pp. 69-77.
- Hemwall, J.B. and Low, P.F. (1955) The Hydrostatic Repulsive Force in Clay Swelling. Soil Science, Vol. 82, pp. 135-145.
- International Society for Soil Science (1963), Bull 23, pp. 7-10.
- Jorgenson, P. (1967) Density, Conductance and Infrared Studies of Clay-Water Systems. Unpublished Ph.D. Thesis, Purdue University.
- Koopmans, R.W.R. and Miller, R.D. (1966) Soil Freezing and Soil Water Characteristic Curves. Soil Science Society of America Proc., Vol. 30, No. 6, pp. 680-685.
- Low, P.F., Anderson, D.M., and Hoekstra, P. (1968) Some Thermodynamic Relationships for Soils at or Below Freezing Point: Freezing Point Depression and Heat Capacity. Water Resource Research, Vol. 4, No. 2, pp. 379-394.
- Michaels, A.S. (1959) Discussion on Paper by Rosenquist. Proc. ASCE, 85, SM2, pp. 91-102.
- Miller, R.D. (1963) Phase Equilibria and Soil Freezing. Inter. Conf. Permafrost Proc., pp. 193-197.
- Mitchell, J.K., Hooper, D.R., and Campanella, R.G. (1965) Permeability of Compacted Clay. Proc. ASCE, 91, No. SM4, pp. 41-65.

Nersesova, Z.A. and Tsytovich, N.A. (1963) Unfrozen Water in Frozen Soils. Proc. of the Permafrost Inter. Conf., National Academy of Science - National Research Council, Publ. 1287.

Richards, L.A. (1941) A Pressure-Membrane Extraction Apparatus for Soil Solution. Soil Science, 51, pp. 377-386.

Schofield, R.K. (1935) The pF of the Water in Soils. 3rd Inter. Cong. Soil Science, Vol. 2, pp. 37-48.

Vialov, S.S. (1959) Rheological Properties and Bearing Capacity of Frozen Soils. Translation 74, U.S. Army, CRREL, Hanover N.H.

Williams, P.J. (1963 a) Specific Heats and Unfrozen Water Content of Frozen Soils. Proc. 1st Canadian Conf. Permafrost, Tech. memo. No. 76, Assoc. Comm. Soil and Snow Mechanics, N.R.C., pp. 109-126.

Williams, P.J. (1963 b) Suction and Its Effects in Unfrozen Water of Frozen Soils. Permafrost Inter. Conf. pp. 225-229.

Williams, P.J. (1964 a) Pressure-Membrane Apparatus for the Investigation of High Suctions in Soils. Building Research Note No. 45, Division of Building Research, National Research Council, Ottawa, Canada.

Williams, P.J. (1964 b) Unfrozen Water Content of Frozen Soils and Soil Moisture Suction. Geotechnique, Vol. 14, No. 3, pp. 231-246.

Williams, P.J. (1966) Pore Pressure at a Penetrating Frost Line and Their Prediction. Geotechnique, Vol. 16, No. 3, pp. 187-208.

Williams, P.J. (1967 a) Unfrozen Water in Frozen Soils: Pore Size-Freezing Temperature-Pressure Relationships. Norwegian Geotechnical Institute, No. 72, Oslo, pp. 37-48.

Williams, P.J. (1967 b) Replacement of Water by Air in Soil Pores. The Engineer, Vol. 223, No. 5796, pp. 293-298.

Williams, P.J. (1968) Thermoelectric Cooling for Precise Temperature Control of Frozen and Unfrozen Soils. Canadian Geotechnical Journal, Vol. 5, No. 4.

- Williams, P.J. and Mitchell, R.J. (1969) Isothermal Volume Change of Frozen Soils, 22nd Canadian Soil Mechanics Conf., Queen's University at Kingston, pp. 1-19.
- Williams, P.J. and Burt, T.P. (1974) Measurement of Hydraulic Conductivity of Frozen Soils. Canadian Geotechnical Journal, Vol. 11, No. 4, pp. 647-650.
- Williams, P.J. (1976) Volume Change in Frozen Soil. Lauritz Bjerrum Memorial Volume. Norwegian Geotechnical Institute (in press).
- Lovell, C.W. (1957) Temperature Effects on Phase Composition and Strength of Partially Frozen Soil. H.R.B. Bull. 168, pp. 74-95.

Fig.1 The Cell

Fig.2 Apparatus and Auxilliary Equipment

Fig.3 Temperature Recording Equipments

Fig.4 Preparation of Soil Sample

Fig. 5 Principle of "pressure plate" and "pressure membrane apparatus, as used to determine the soil water suction.

Fig. 6 Pressure Membrane Apparatus

FIGURE 7
PRESSURE MEMBRANE APPARATUS FOR USE AT HIGH PRESSURES
(WORKING DRAWINGS AVAILABLE FROM DIVISION OF BUILDING RESEARCH,
NATIONAL RESEARCH COUNCIL.)

Fig. 8 Pressure Plate Apparatus

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Fig. 9 Dilatometer volume change observations as function of temperature for the different soils investigated.

Fig. 10. Unfrozen water content of frozen soils, calculated from the dilatometer volume change observations.

Fig. 11 Volume change of frozen Fithian Illite,
under different applied external pressures.

Fig. 12 Volume change of frozen Oneida clay, $\phi < 5\mu$, under different applied external pressures.

Fig. 13 Suction-moisture content relationship for a heavy clay soil (after Gronney et al, 1958)

Fig. 14 The suction-moisture content relationships of unfrozen soils determined by pressure plate and pressure membrane tests.

Fig. 15 Unfrozen water content of frozen Oneida clay predicted from free energy-moisture content relationship as compared to those calculated from dilatometer volume change observations.

Fig. 16 Unfrozen water content of frozen Fithian Illite predicted from free energy-moisture content relationship as compared to those calculated from dilatometer volume change observations..

Fig. 17 Unfrozen water content of frozen Oneida clay $\phi < 5\mu$ predicted from free energy-moisture content relationship as compared to those calculated from dilatometer volume change observations.

Fig. 18 Unfrozen water content of frozen Tanzania clay predicted from free energy-moisture content relationship as compared to those calculated from dilatometer volume change

SCALE 1:1

19 a

SCALE 1:1

19 b

SCALE 1:1

19 c

Fig. 19 Clay-packet formation in a sample of frozen Tanzania clay

Fig. 19a Completely frozen sample

Fig. 19b Same sample after partial thawing (clay packets are still observed)

Fig. 19c Close-up view of the sample at stage b

Fig. 20 Volume change and temperature change with time, for a sample of frozen Tanzania clay.

Fig. 21 Volume change of frozen Tanzania clay, due to pressure changes.

Fig. 22 Changes in unfrozen water content from volume changes of frozen Tanzania clay, due to pressure changes.

Fig. 23 Free energy-moisture content relationship for unfrozen Tanzania clay predicted from unfrozen water content-temperature relationship (c.f. Figure 22).

APPENDIX I

The Dilatometer Test Results

Table 1 Dilatometer Test Results of Volume Change of Frozen Oneida Clay Under Atmospheric Pressure (Zero Gauge Pressure)

Temperature °C	Observed Volume Change cm ³	Correction for Thermal Contraction cm ³	Sample Volume Change cm ³	Percent Volume Change %
0.000	0.000			
-0.328	4.361	0.000	4.361	3.028
-0.370	4.434	0.001	4.435	3.080
-0.580	4.703	0.005	4.708	3.269
-1.097	4.941	0.017	4.958	3.443
-1.943	5.109	0.035	5.144	3.572
-3.009	5.202	0.058	5.260	3.653
-3.994	5.251	0.079	5.330	3.701
-5.199	5.273	0.103	5.376	3.733

Correction factor $c = 0.0215 \text{ cm}^3/\text{°C}$

Table 2 Dilatometer Test Results of Volume Change of Frozen Fithian Illite Under Atmospheric Pressure (Zero Gauge Pressure)

Temperature °C	Observed Volume Change cm ³	Correction for Thermal Contraction cm ³	Sample Volume Change cm ³	Percent Volume Change %
0.000	0.000		0.000	0.000
-0.128	2.372	0.000	2.372	1.825
-0.248	2.858	0.003	2.861	2.201
-0.586	3.286	0.012	3.298	2.537
-1.015	3.525	0.023	3.548	2.729
-1.993	3.798	0.049	3.847	2.959
-3.006	3.932	0.076	4.008	3.083
-4.006	4.018	0.102	4.120	3.169
-4.992	4.066	0.128	4.194	3.226
-5.705	4.096	0.147	4.243	3.264

Correction factor $c = 0.0264 \text{ cm}^3/\text{°C}$

Table 3 Dilatometer Test Results of Volume Change of Frozen Fithian Illite Under 5 Kg/cm²

Temperature °C	Observed Volume Change cm ³	Correction for Thermal Contraction cm ³	Sample Volume Change cm ³	Percent Volume Change %
0.000	0.000		0.000	0.000
-0.262	2.770	0.000	2.770	2.131
-0.423	3.192	0.004	3.196	2.458
-0.961	3.577	0.017	3.594	2.765
-2.048	3.884	0.045	3.929	3.022
-3.108	4.026	0.071	4.097	3.152
-3.993	4.098	0.094	4.192	3.225
-4.985	4.152	0.119	4.271	3.285
-5.598	4.174	0.134	4.308	3.314

Correction factor $c = 0.0251 \text{ cm}^3/\text{°C}$

Table 4 Dilatometer Test Results of Volume Change of Frozen Oneida Clay, $\phi < 5\mu$ Under Atmospheric Pressure (Zero Gauge Pressure)

Temperature $^{\circ}\text{C}$	Observed Volume Change cm^3	Correction for Thermal Contraction cm^3	Sample Volume Change cm^3	Percent Volume Change %
0.000	0.000		0.000	0.000
-0.067	3.401	0.000	3.401	3.149
-0.180	3.862	0.000	3.862	3.576
-0.212	3.979	0.004	3.983	3.688
-0.301	4.109	0.007	4.116	3.811
-0.383	4.211	0.009	4.220	3.907
-0.460	4.396	0.011	4.407	4.081
-0.555	4.714	0.014	4.728	4.378
-0.628	4.877	0.016	4.893	4.531
-0.658	4.976	0.015	4.991	4.621
-1.064	5.394	0.027	5.421	5.019
-1.950	5.725	0.055	5.780	5.352
-2.993	5.854	0.087	5.941	5.500
-4.014	5.907	0.118	6.025	5.579
-4.979	5.934	0.148	6.082	5.631
-5.563	5.941	0.166	6.107	5.655

Correction factor $c = 0.0290 \text{ cm}^3/^{\circ}\text{C}$

Table 5 Dilatometer Test Results of Volume Change of Frozen Oneida Clay, $\phi < 5\mu$ Under 5 Kg/cm²

Temperature °C	Observed Volume Change cm ³	Correction for Thermal Contraction cm ³	Sample Volume Change cm ³	Percent Volume Change %
0.000	0.000		0.000	0.000
-0.194	3.894	0.000	3.894	3.606
-0.290	4.063	0.002	4.065	3.764
-0.403	4.245	0.005	4.250	3.935
-0.516	4.576	0.007	4.583	4.243
-0.631	4.924	0.010	4.934	4.568
-0.760	5.110	0.013	5.123	4.743
-0.868	5.233	0.016	5.249	4.860
-1.063	5.388	0.020	5.408	5.007
-1.870	5.716	0.039	5.755	5.329
-2.837	5.867	0.062	5.929	5.490
-3.764	5.937	0.083	6.020	5.574
-5.016	5.981	0.112	6.093	5.642
-5.548	5.990	0.125	6.115	5.662

Correction factor $c = 0.0233 \text{ cm}^3/\text{°C}$

Table 6 Dilatometer Test Results of Volume Change of Frozen Oneida Clay, $\phi < 5\mu$ Under 10 Kg/cm²

Temperature °C	Observed Volume Change cm ³	Correction for Thermal Contraction cm ³	Sample Volume Change cm ³	Percent Volume Change %
0.000	0.000			0.000
-0.139	3.475	0.000	3.475	3.218
-0.224	3.841	0.002	3.843	3.558
-0.324	4.033	0.005	4.038	3.739
-0.426	4.242	0.008	4.250	3.936
-0.527	4.479	0.010	4.489	4.156
-0.645	4.845	0.014	4.859	4.499
-0.741	5.070	0.016	5.086	4.709
-0.977	5.323	0.023	5.346	4.950
-1.847	5.756	0.046	5.802	5.372
-2.838	5.935	0.073	6.008	5.563
-3.764	6.009	0.099	6.108	5.656
-4.684	6.054	0.124	6.178	5.720
-5.485	6.079	0.145	6.224	5.763

Correction factor $c = 0.0272 \text{ cm}^3/\text{°C}$

Table 7 Dilatometer Test Results of Volume Change of Frozen Tanzania Clay Under Atmospheric Pressure (Zero Gauge Pressure), Initial Freezing

Temperature $^{\circ}\text{C}$	Observed Volume Change cm^3	Correction for Thermal Contraction cm^3	Sample Volume Change cm^3	Percent Volume Change %
0.000	0.000		0.000	0.000
-0.111	3.721	0.000	3.721	3.235
-0.248	4.255	0.004	4.259	3.703
-0.367	4.485	0.007	4.492	3.906
-0.472	4.601	0.010	4.611	4.010
-0.577	4.705	0.013	4.718	4.103
-0.931	4.930	0.023	4.953	4.307
-1.945	5.252	0.051	5.303	4.611
-2.918	5.396	0.078	5.474	4.760
-3.868	5.479	0.104	5.583	4.855
-4.755	5.536	0.129	5.665	4.926
-5.571	5.579	0.152	5.731	4.983

Correction factor $c = 0.0278 \text{ cm}^3/^{\circ}\text{C}$

Table 8 Correction of the Observed Volume Change of Frozen Samples Due to Expansion of Air Entrapped in the System.

Applied Pressure Kg/cm ²	Room Temp. °C T	Freezing Temp. °C T _f	Observed Displacement of Mercury Under Press. P, at Temp. T, (Δv)cm ³	$\frac{T_f}{T}$ $\frac{^{\circ}K}{^{\circ}K}$	Calculated Displacement in Mercury Level on Release of Press., at Freezing Temp. (Δv _f)cm ³	Observed Volume Change at Temp. T _f Under Press. P cm ³	Sample Volume Change at Temp. T _f , Under Press. P cm ³
					(1)		(2)
10		-0.158	0.000		0.000	3.231	3.231
5	15.5	-0.138	0.065	0.946	0.061	3.673	3.612
0.0		-0.171	0.267	0.946	0.252	4.430	4.178
10		-0.274	0.000		0.000	4.267	4.267
5	15.5	-0.270	0.063	0.945	0.060	4.387	4.327
0.0		-0.290	0.274	0.945	0.259	4.717	4.458
10		-0.379	0.000		0.000	4.557	4.557
5	17.0	-0.373	0.064	0.940	0.060	4.652	4.592
0.0		-0.366	0.270	0.940	0.254	4.937	4.683
10		-0.481	0.000		0.000	4.693	4.693
5	18.0	-0.484	0.054	0.936	0.051	4.776	4.725
0.0		-0.492	0.269	0.936	0.252	5.023	4.771
10		-0.685	0.000		0.000	4.868	4.868
5	17.5	-0.679	0.059	0.937	0.055	4.942	4.887
0.0		-0.719	0.254	0.937	0.238	5.165	4.927
10		-1.204	0.000		0.000	5.193	5.193
5	18.0	-1.210	0.051	0.934	0.048	5.244	5.196
0.0		-1.219	0.235	0.934	0.220	5.423	5.203

$$(1) \Delta v_f = \frac{T_f}{T} \cdot \Delta v$$

$$(2) \text{ Sample Volume Change} = \text{Observed Volume Change} - \Delta v_f$$

Table 9 Corrected Volume Change of Frozen Tanzania Clay Due to Pressure Change and the Implied Changes in the Unfrozen Water Content

Temp. °C T_f	Applied Pressure at Temperature T_f P_T , Kg/cm ²	Sample Volume Change Under Pressure P_T	Wt. of Frozen Water, gm.	Wt. of Unfrozen Water, gm.	Unfrozen Water Content % Dry Weight	Percent Volume Change %
-0.158	10	3.231	35.70	50.46	64.08	2.809
-0.138	5	3.612	39.91	46.25	58.73	3.141
-0.171	0	4.178	46.16	40.00	50.79	3.633
-0.274	10	4.267	47.15	39.01	49.54	3.710
-0.270	5	4.327	47.81	38.35	48.70	3.763
-0.290	0	4.458	49.26	36.90	46.86	3.877
-0.379	10	4.557	50.35	35.81	45.47	3.963
-0.373	5	4.592	50.74	35.42	44.98	3.993
-0.366	0	4.683	51.75	34.41	43.70	4.072
-0.481	10	4.693	51.86	34.30	43.56	4.081
-0.484	5	4.725	52.21	33.95	43.11	4.109
-0.492	0	4.771	52.72	33.44	42.46	4.149
-0.685	10	4.868	53.79	32.37	41.10	4.233
-0.679	5	4.887	54.00	32.16	40.84	4.250
-0.719	10	4.927	54.44	31.72	40.28	4.284
-1.204	10	5.193	57.38	28.78	36.55	4.516
-1.210	5	5.196	57.41	28.75	36.51	4.518
-1.219	0	5.203	57.49	28.67	36.41	4.524

APPENDIX II

Calculation of the Unfrozen Water Content
from the Dilatometer Volume Change Observations

Table 10 Unfrozen Water Content of Frozen Oneida Clay Calculated from the Dilatometer Volume Change Observations Under Atmospheric Pressure (Zero Gauge Pressure)

Temperature °C	Sample Volume Change cm ³	Weight of Frozen Water gm.	Weight of Unfrozen Water gm.	Unfrozen Water Content % Dry Weight
0.000	0.000	0.00	73.02	36.96
-0.328	4.361	48.19	24.83	12.48
-0.370	4.435	49.00	24.02	12.07
-0.580	4.708	52.02	21.00	10.55
-1.097	4.958	54.78	18.24	9.17
-1.943	5.144	56.84	16.18	8.13
-3.009	5.260	58.12	14.90	7.49
-3.994	5.330	58.90	14.12	7.10
-5.119	5.376	59.40	13.62	6.84

Table 11 Unfrozen Water Content of Frozen Fithian Illite Calculated from the Dilatometer Volume Change Observations Under Atmospheric Pressure (Zero Gauge Pressure)

Temperature °C	Sample Volume Change cm ³	Weight of Frozen Water gm.	Weight of Unfrozen Water gm.	Unfrozen Water Content % Dry Weight
0.000	0.000	0.00	63.17	35.05
-0.128	2.372	26.21	36.96	20.50
-0.248	2.861	31.61	31.56	17.51
-0.586	3.298	36.44	26.73	14.83
-1.015	3.548	39.20	23.97	13.30
-1.993	3.847	42.51	20.66	11.46
-3.006	4.008	44.29	18.88	10.47
-4.006	4.120	45.52	17.65	9.79
-4.992	4.194	46.34	16.83	9.34
-5.705	4.243	46.88	16.29	9.04

Table 12 Unfrozen Water Content of Frozen Fithian Illite Calculated from the Dilatometer Volume Change Observations Under 5 Kg/cm²

Temperature °C	Sample Volume Change cm ³	Weight of Frozen Water gm.	Weight of Unfrozen Water gm.	Unfrozen Water Content % Dry Weight
0.000	0.000	0.00	63.17	35.05
-0.262	2.770	30.61	32.56	18.06
-0.423	3.196	35.31	27.86	15.46
-0.961	3.594	39.71	23.46	13.02
-2.048	3.929	43.41	19.76	10.96
-3.108	4.097	45.27	17.90	9.93
-3.993	4.192	46.32	16.85	9.35
-4.985	4.271	47.19	15.98	8.87
-5.598	4.308	47.60	15.57	8.64

Table 13 Unfrozen Water Content of Frozen Oneida Clay, $\phi < 5\mu$ Calculated from the Dilatometer Volume Change Observations Under Atmospheric Pressure (Zero Gauge Pressure)

Temperature °C	Sample Volume Change cm ³	Weight of Frozen Water gm.	Weight of Unfrozen Water gm.	Unfrozen Water Content % Dry Weight
0.000	0.000	0.00	77.73	85.24
-0.067	3.401	37.58	40.15	44.03
-0.180	3.862	42.67	35.06	38.45
-0.212	3.983	44.01	33.72	36.98
-0.301	4.116	45.48	32.25	35.37
-0.383	4.220	46.63	31.10	34.10
-0.460	4.407	48.70	29.03	31.83
-0.555	4.728	52.24	25.49	27.95
-0.628	4.893	54.07	23.66	25.95
-0.658	4.991	55.15	22.58	24.76
-1.064	5.421	59.90	17.83	19.55
-1.950	5.780	63.87	13.86	15.20
-2.993	5.941	65.65	12.08	13.25
-4.014	6.025	66.57	11.16	12.24
-4.979	6.082	67.20	10.53	11.55
-5.563	6.107	67.48	10.25	11.24

Table 14 Unfrozen Water Content of Frozen Oneida Clay, $\phi < 5\mu$ Calculated from the Dilatometer Volume Change Observations Under 5 Kg/cm²

Temperature °C	Sample Volume Change cm ³	Weight of Frozen Water gm.	Weight of Unfrozen Water gm.	Unfrozen Water Content % Dry Weight
0.000	0.000	0.00	77.73	85.24
-0.194	3.894	43.03	34.70	38.05
-0.290	4.065	44.92	32.81	35.98
-0.403	4.250	46.96	30.77	33.74
-0.516	4.583	50.64	27.09	29.71
-0.631	4.934	54.52	23.21	25.45
-0.760	5.123	56.61	21.12	33.16
-0.868	5.249	58.00	19.73	21.64
-1.063	5.408	59.76	17.97	19.71
-1.870	5.755	63.59	14.14	15.51
-2.837	5.929	65.51	12.22	13.40
-3.764	6.020	66.52	11.21	12.29
-5.016	6.093	67.33	10.40	11.40
-5.548	6.115	67.57	10.16	11.14

Table 15 Unfrozen Water Content of Frozen Oneida Clay $\phi < 5\mu$ Calculated from the Dilatometer Volume Change Observations Under 10 Kg/cm²

Temperature °C	Sample Volume Change cm ³	Weight of Frozen Water gm.	Weight of Unfrozen Water gm.	Unfrozen Water Content % Dry Weight
0.000	0.000	0.00	77.73	85.24
-0.139	3.475	38.40	39.33	43.13
-0.224	3.843	42.46	35.27	38.68
-0.324	4.038	44.62	33.11	36.31
-0.426	4.250	46.96	30.77	33.74
-0.527	4.489	49.60	28.13	30.85
-0.645	4.859	53.69	24.04	26.36
-0.741	5.086	56.20	21.53	23.61
-0.977	5.346	59.07	18.66	20.46
-1.847	5.802	64.11	13.62	14.94
-2.838	6.008	66.39	11.34	12.44
-3.764	6.108	67.49	10.24	11.23
-4.684	6.178	68.26	9.47	10.38
-5.485	6.224	68.77	8.96	9.83

Table 16. Unfrozen Water Content of Frozen Tanzania Clay Calculated from the Dilatometer Volume Change Observations Under Atmospheric Pressure (Zero Gauge Pressure), Initial Freezing

Temperature °C	Sample Volume Change cm ³	Weight of Frozen Water gm.	Weight of Unfrozen Water gm.	Unfrozen Water Content % Dry Weight
0.000	0.000	0.00	86.16	109.41
-0.111	3.721	41.12	45.04	57.19
-0.248	4.259	47.06	39.10	49.65
-0.367	4.492	49.64	36.52	46.37
-0.472	4.611	50.95	35.21	44.71
-0.577	4.718	52.13	34.03	43.21
-0.931	4.953	54.73	31.43	39.91
-1.945	5.303	58.60	27.56	35.00
-2.918	5.474	60.49	25.67	32.60
-3.868	5.583	61.69	24.47	31.07
-4.755	5.665	62.60	23.56	29.92
-5.571	5.731	63.33	22.83	28.99

APPENDIX III

The Suction-Moisture Content Test Results

Table 17 Results of the Suction-Moisture Content Test for the Unfrozen Oneida Clay

Applied Air Pressure		Soil Water Content % Dry Weight		Average Water Content %	Technique Used
PSI	Kg/cm ²	Sample 1	Sample 2		
25	1.7	21.12	20.65	20.88	Pressure Membrane
50	3.5	15.83	16.02	15.92	" "
75	5.2	15.53	15.08	15.30	" "
100	7.0	13.31	13.47	13.39	" "
125	8.7	12.65	13.23	12.94	" "
150	10.5	11.88	11.63	11.76	" "
170	11.9	11.70	11.57	11.63	" "

Table 18 Results of the Suction-Moisture Content Test for the Unfrozen Fithian Illite

Applied Air Pressure		Soil Water Content % Dry Weight			Average Water Content %	Technique Used
PSI	Kg/cm ²	Sample 1	Sample 2	Sample 3		
10	0.7	31.11	30.97	31.25	31.11	Pressure Plate
15	1.0	30.11	28.94	29.72	29.59	Pressure Membrane
20	1.4	25.32	25.59	25.45	25.45	Pressure Plate
25	1.7	23.46	23.81	23.49	23.59	Pressure Membrane
50	3.5	20.86	21.87	20.83	21.19	" "
75	5.2	20.35	20.95	20.21	20.50	" "
100	7.0	19.19	19.85	19.25	19.43	" "
125	8.7	17.22	17.71	17.30	17.41 ¹	" "
150	10.5	16.84	16.92	16.84	16.87	" "
200	14.0	15.28	15.38	15.49	15.38	" "
250	17.5	14.39	14.75	14.55	14.56	" "
300	21.0	14.26	14.55	14.64	14.48	" "
350	24.6	13.49	13.87	13.67	13.68	" "
400	28.1	12.24	12.57	12.43	12.41	" "
450	31.6	11.81	11.98	12.11	11.97	" "
509	35.7	12.04	12.19		12.11	High Pressure Membrane
1018	71.5	10.84	11.23		11.04	" "
2036	143.1	9.96	9.98		9.97	" "

Table 19 Results of the Suction-Moisture Content Test for the Unfrozen Oneida Clay, $\phi < 5\mu$

Applied Air Pressure		Soil Water Content % Dry Weight			Average Water Content %	Technique Used
PSI	Kg/cm ²	Sample 1	Sample 2	Sample 3		
10	0.7	49.61	49.43	49.94	49.66	Pressure Plate
20	1.4	45.36	45.07	45.13	45.19	" "
30	2.1	42.50	42.37	42.54	42.47	" "
75	5.2		39.60	39.59	39.60	Pressure Membrane
100	7.0	38.60	39.23	37.17	38.33	" "
125	8.7	37.39	37.50	37.81	37.57	" "
150	10.5	35.67	35.63	35.73	35.68	" "
205	14.4	32.26	33.56	33.66	33.49	" "
250	17.5	28.68	28.97	29.30	28.98	" "
300	21.0	25.75	25.91	26.07	25.91	" "
350	24.6	24.10		23.91	24.00	" "
400	28.1	22.10	22.19	22.46	22.25	" "
455	31.9	19.39	19.84	20.70	19.98	" "
509	35.7	18.30	19.13		18.71	High Pressure Membrane
1018	71.5	13.63	13.22		13.42	" " "
2036	143.1	11.19	11.23		11.21	" " "

Table 20 Results of the Suction-Moisture Content Test for the Unfrozen Tanzania Clay, Initial Drying

Applied Air Pressure		Soil Water Content % Dry Weight			Average Water Content	Technique Used
PSI	Kg/cm ²	Sample 1	Sample 2	Sample 3		
10	0.7	71.70	71.91	71.85	71.82	Pressure Plate
20	1.4	66.60	66.57	66.35	66.51	" "
30	2.1	62.30	62.58	61.99	62.29	" "
50	3.5	56.56	56.51	56.46	56.51	Pressure Membrane
75	5.2	52.38	52.98	52.25	52.54	" "
100	7.0		49.62	49.63	49.62	" "
125	8.7	49.00	48.67	48.53	48.73	" "
150	10.5	46.63	46.42	46.54	46.53	" "
190	13.3	44.54	44.62	44.62	44.59	" "
255	17.9	43.29	43.97	43.56	43.61	" "
300	21.0	41.63	41.42	41.53	41.53	" "
350	24.6	40.50	40.63	40.46	40.53	" "
395	27.7	39.67	39.76	39.71	39.71	" "
455	31.9	38.66	38.44	38.58	38.56	" "
509	35.7	37.78	37.84		37.81	High Pressure Membrane
1018	71.5	33.50	33.74		33.62	" " "
2036	143.1	29.34	29.21		29.27	" " "

APPENDIX IV

Determination of Salt Concentration in Soil Water and the Associated Freezing-Point Depression

1. Solutions of sodium chloride at different concentrations were prepared. The electrical conductivity for the different concentrations was measured, see table below. The calibration chart, a plot of concentration against the electrical conductivity, is shown in Figure IV-1.

Concentration gm./lit.	Electrical Conductivity milli MHO
1/16	0.072
1/8	0.124
1/4	0.246
1/2	0.480
1.0	0.930
2.0	1.840
4.0	3.560

2. The soil sample was placed in the pressure plate apparatus and subjected to 0.5 Kg/cm^2 pressure. The drained water was collected and its electrical conductivity was determined, see table below.

3. The salt concentrations in the soil water for the different soils were obtained from the calibration chart of Figure

IV-1, and were found to be 0.25, 1.05, 0.70, and 0.90 gm./lit. for the Tanzania clay, the Oneida clay, Oneida clay, $\phi < 5\mu$, and the Fithian Illite, respectively.

Soil Type	Initial Water Content % dry weight	Electrical Conductivity milli MHO
Tanzania clay	97.46	0.27
Oneida clay	28.57	0.97
Oneida clay, $\phi < \mu$	53.34	0.66
Fithian Illite	36.19	0.86

4. From the Handbook of Chemistry and Physics, Table D236, the freezing-point depression at different concentrations for a solution of sodium chloride is shown in Figure IV-2.

5. The freezing-point depression in the soil water for the different soils was obtained from Figure IV-2, and was found to be 0.017, 0.065, 0.044, and 0.056°C for the Tanzania clay, the Oneida clay, the Oneida clay, $\phi < 5\mu$, and the Fithian Illite, respectively.

Fig. IV-1 Salt concentration in a solution of NaCl as function of the electrical conductivity.

Fig. IV-2 Salt concentration in a solution of NaCl and the associated freezing-point depression.

APPENDIX V

Prediction of the Soil Freezing Characteristic from the Soil Water Characteristic

The results of the suction-moisture content tests for the different soils investigated are given in Tables 17 to 20, see Appendix III. The implied unfrozen water contents at different freezing temperatures, ΔT_s , were predicted from the suction-moisture content test results by making use of the two relations

$$\Delta f_{\text{frozen}} = \frac{\sigma_{1w}}{\sigma_{aw}}, \Delta f_{\text{unfrozen}} \quad (\text{equation 21})$$

and

$$\Delta f_{\text{frozen}} = \frac{L}{T} \cdot \Delta T_s \quad (\text{equation 11})$$

The results of these calculations for the different soils are given in the following tables (Tables 21-24).

In the suction-moisture content test, it is generally agreed that the osmotic component of the total soil suction is not measured since the membrane (in the pressure membrane apparatus) is permeable to salts dissolved in the soil solution. The dissolved salts are however partly responsible for depression of the temperature of freezing. The above calculated freezing temperatures, ΔT_s , corresponding to each value of the unfrozen water content must therefore be lowered by an amount ΔT_o to account for the effect of dissolved salts.

The freezing-point depression of the water, in the unfrozen soil, associated with the dissolved salts was obtained, see Appendix IV. As ice is formed during freezing of the soil, the salts however became increasingly concentrated in the remaining unfrozen water (on the assumption that the formed ice does not contain any significant amount of dissolved salts). Williams (1964 b) suggests that since the increase in freezing-point depression due to salts in solution is approximately proportional to the concentration, the effects of dissolved salts on the freezing temperature associated with each quantity of unfrozen water are calculated using the formula

$$\Delta T_o = \frac{T_s \times \text{total moisture content}}{\text{unfrozen water content}}$$

where

ΔT_s = The freezing-point depression of the soil water in the unfrozen soil

ΔT_o = The calculated freezing-point depression associated with the unfrozen water content under consideration.

The results of these calculations for the different soils are given in the following tables (Tables 21-24).

Table 21 Unfrozen Water Content of Frozen Oneida Clay Predicted from Free Energy-Moisture Content Relationship

(1)		(2)		Total Freezing Point Depression $\Delta T = \Delta T_m + \Delta T_o$ °C	Water Content % Dry Weight
Soil Suction $P_a - P_w$ Kg/cm ²	Soil Suction $P_i - P_w$ Kg/cm ²	Freezing-Point Depression			
		ΔT_m °C	ΔT_o °C		
1.757	0.7	0.059	0.089	0.148	20.88
3.515	1.4	0.119	0.117	0.236	15.92
5.272	2.2	0.178	0.121	0.299	15.30
7.030	2.5	0.237	0.139	0.376	13.39
8.787	3.6	0.296	0.143	0.439	12.94
10.545	4.4	0.356	0.158	0.514	11.76
11.951	5.0	0.403	0.160	0.563	11.63

$$(1) (P_i - P_w) = \frac{\sigma_{iw}}{\sigma_{aw}} (P_a - P_w) = 0.42 (P_a - P_w)$$

$$(2) \Delta T_m = \frac{T}{L} \cdot \Delta f \text{ (equation 11)}$$

$$\Delta T_m = \frac{T}{L} (P_i - P_w)$$

Table 22 Unfrozen Water Content of Frozen Fithian Illite Predicted from Free Energy-Moisture Content Relationship

(1)		(2)		Total Freezing Point Depression	Water Content % Dry Weight
Soil Suction $P_a - P_w$ Kg/cm ²	Soil Suction $P_i - P_w$ Kg/cm ²	Freezing-Point Depression			
		$\Delta T_m^{\circ}C$	$\Delta T_o^{\circ}C$	$\Delta T = \Delta T_m + \Delta T_o$ $^{\circ}C$	
0.703	0.2	0.029	0.065	0.094	31.11
1.054	0.4	0.036	0.068	0.104	29.59
1.406	0.5	0.047	0.080	0.127	25.45
1.757	0.7	0.059	0.086	0.145	23.59
3.515	1.4	0.119	0.096	0.215	21.19
5.272	2.2	0.178	0.099	0.277	20.50
7.030	2.9	0.237	0.104	0.341	19.43
8.787	3.6	0.296	0.116	0.412	17.41
10.545	4.4	0.356	0.120	0.476	16.87
14.060	5.9	0.474	0.132	0.606	15.38
17.575	7.3	0.593	0.139	0.732	14.56
21.090	8.8	0.711	0.140	0.851	14.48
24.605	10.3	0.830	0.148	0.978	13.68
28.120	11.8	0.948	0.163	1.111	12.41
31.635	13.2	1.067	0.169	1.236	11.97
35.783	15.0	1.207	0.167	1.374	12.11
71.565	30.0	2.414	0.184	2.598	11.04
143.131	60.1	4.828	0.204	5.032	9.97

$$(1) (P_i - P_w) = \frac{\sigma_{iw}}{\sigma_{aw}} \cdot (P_a - P_w) = 0.42 (P_a - P_w)$$

$$(2) \Delta T_m = \frac{T}{L} \cdot \Delta f = \frac{T}{L} (P_i - P_w) \quad (\text{equation 11})$$

Table 23 Unfrozen Water Content of Frozen Oneida Clay, $\phi < 5\mu$
Predicted from Free Energy-Moisture Content Relationship

(1)

(2)

Soil Suction $P_a - P_w$ Kg/cm ²	Soil Suction $P_i - P_w$ Kg/cm ²	Freezing-Point Depression		Total Freezing Point Depression $\Delta T = \Delta T_m + \Delta T_o$ °C	Water Content % Dry Weight
		ΔT_m °C	ΔT_o °C		
0.703	0.2	0.029	0.047	0.076	49.66
1.406	0.5	0.047	0.052	0.099	45.19
2.109	0.8	0.071	0.055	0.126	42.47
5.272	2.2	0.178	0.059	0.237	39.60
7.030	2.9	0.237	0.061	0.298	38.33
8.787	3.6	0.296	0.062	0.358	37.57
10.545	4.4	0.356	0.066	0.422	35.68
14.411	6.0	0.486	0.070	0.556	33.49
17.575	7.3	0.593	0.081	0.674	28.98
21.090	8.8	0.711	0.091	0.802	25.91
24.605	10.3	0.830	0.098	0.928	24.00
28.120	11.8	0.948	0.105	1.053	22.25
31.986	13.4	1.079	0.117	1.196	19.98
35.783	15.0	1.207	0.125	1.332	18.71
71.565	30.0	2.414	0.175	2.589	13.42
143.131	60.1	4.828	0.209	5.037	11.21

$$(1) (P_i - P_w) = \frac{\sigma_{iw}}{\sigma_{aw}} \cdot (P_a - P_w) = 0.42 (P_a - P_w)$$

$$(2) \Delta T_m = \frac{T}{L} \cdot \Delta f = \frac{T}{L} (P_i - P_w) \quad (\text{equation 11})$$

Table 24 Unfrozen Water Content of Frozen Tanzania Clay Predicted from Free Energy-Moisture Content Relationship

(1)		(2)			
Soil Suction	Soil Suction	Freezing-Point Depression		Total Freezing Point Depression	Water Content
$P_a - P_w$	$P_i - P_w$	$\Delta T_m^{\circ}\text{C}$	$\Delta T_o^{\circ}\text{C}$	$\Delta T = \Delta T_m + \Delta T_o$	% Dry Weight
Kg/cm ²	Kg/cm ²			^o C	
0.703	0.2	0.029	0.023	0.052	71.82
1.406	0.5	0.047	0.025	0.072	66.51
2.109	0.8	0.071	0.027	0.098	62.29
3.515	1.4	0.119	0.029	0.148	56.51
5.272	2.2	0.178	0.032	0.210	52.54
7.030	2.9	0.237	0.033	0.270	49.62
8.787	3.6	0.296	0.034	0.330	48.73
10.545	4.4	0.356	0.036	0.392	46.53
13.357	5.6	0.450	0.037	0.487	44.59
17.926	7.5	0.605	0.038	0.643	43.61
21.090	8.8	0.711	0.040	0.751	41.53
24.605	10.3	0.830	0.041	0.871	40.53
27.768	11.6	0.937	0.042	0.979	39.71
31.986	13.4	1.079	0.043	1.122	38.56
35.783	15.0	1.207	0.044	1.251	37.81
71.565	30.0	2.414	0.049	0.463	33.56
143.131	60.1	4.828	0.057	4.885	29.27

$$(1) (P_i - P_w) = \frac{\sigma_{iw}}{\sigma_{aw}} \cdot (P_a - P_w) = 0.42 (P_a - P_w)$$

$$(2) \Delta T_m = \frac{T}{L} \cdot \Delta f = \frac{T}{L} (P_i - P_w) \quad (\text{equation 11})$$

APPENDIX VI

Determination of the Air-Intrusion Value

Definition

In a partially saturated soil, the capillary equation, $\Delta P = \frac{2\sigma_{aw}}{r_{aw}}$, expresses the pressure differences ΔP at the air-water interface as a function of the interfacial energy σ_{aw} , and the radius of curvature of the interface r_{aw} (which is assumed to be equal to the radius of the soil pore in which the interface takes place). Under a pressure difference ΔP , the air-water menisci will be confined only to pores of sizes equal to r_{aw} .

Consider a sample of saturated soil. When the sample is subjected to a relatively low air pressure, the air-water menisci lie at the surface of the sample and in larger pores. On increasing the applied air pressure, pores of sizes greater than r_{aw} (in the capillary equation) will be emptied of water. Strings of inter-connected air-filled pores extend a little deeper into the sample, but their penetration is hindered by occurrence of constrictions smaller than r_{aw} . There will be a critical applied pressure ΔP_c at which continuous air-filled channels spread through the sample. This value of ΔP_c is referred to as the air-intrusion value of the soil (Williams, 1967 b) and is associated with a substantial drainage of water.

Technique Used

Two identical samples of the soil were slurried and de-aired. A consolidation test was performed on one of the samples, and the suction-moisture content test, in the pressure membrane apparatus, was carried out using the other sample. The results of the two tests are shown in Figures VI-1, and VI-2 for the Oneida, $\phi < 5\mu$, and the Fithian Illite, where the water content for samples in consolidation and suction tests is shown as a function of effective stress (equal to the applied load, and applied suction respectively). So long as the soil is saturated the two tests give, more or less, similar results. At a certain effective stress the two sets of results diverge with the suction test results lying below the consolidation test results. This is the shrinkage limit (Williams, 1963 b), and desaturation occurs in the suction test. This value for the effective stress (or suction), roughly speaking is considered as the air-intrusion value.

Reference to Figures VI-1 and VI-2, the air-intrusion values for the Oneida, $\phi < 5\mu$ and Fithian Illite are found to be of the order 16 Kg/cm^2 and 8 Kg/cm^2 , respectively.

The same technique was performed on samples of Tanzania clay. The two sets of results indicated that up to 16 Kg/cm^2 applied load (the maximum workable load in the consolidation test) the air-intrusion value was not reached, see Figure VI-3. This implied that the air-intrusion value for the Tanzania clay is higher than 16 Kg/cm^2 .

Fig. VI-1. Comparison of observations from suction-moisture content tests and from consolidation tests (Oneida clay, $\phi < 5\mu$)

Fig. VI-2 Comparison of observations from suction-moisture content tests and from consolidation tests (Pithian Illite).

Fig. VI-3 Comparison of observations from suction-moisture content tests and from consolidation tests (Tan-zania clay).

Another technique was therefore used to determine the shrinkage limit for a sample of the Tanzania clay. A sample was prepared from a slurry and was de-aired. It was then allowed to dry at a very slow rate by containing it into a jar opened to the atmosphere through a small opening. The volume of the soil sample was determined every day, by immersing it into a container filled with mercury and determining the volume of the displaced mercury, together with its weight. The test results are shown in Figure VI-4, where the water content is shown as function of the volume of the sample. In Figure VI-4, the shrinkage curves for the two soil samples change their slope at 23% and 30% moisture content; and the implied soil water suction, from the suction-moisture content relationship in Figure 14, is higher than 140 Kg/cm^2 . The test results, therefore, suggest that the shrinkage limit for the Tanzania clay lies between 23% and 30%, and its air-intrusion value is higher than 140 Kg/cm^2 . This is an exceptionally high value compared to those reported in the literature.

Fig. VI-4 Shrinkage curves for two samples of Tanzania clay.

APPENDIX VII

Stabilization and Calibration of Thermistors

Introduction

This manual describes a procedure for stabilizing and calibrating a new thermistor, to $\pm .005^{\circ}\text{C}$.

Section I deals with the stabilization of thermistors. Section II deals with the calibration of thermistors.

Section I

Stabilization of Thermistors

Apparatus

1. Boiling water bath.
2. Dry ice.
3. Ice bath.
4. Wheatstone bridge for resistance measurements up to 1000 ohms and accurate to 0.1 ohm.

(Potentiometric voltmeter-bridge set model 300, available at the Geography laboratory, was used and found to be satisfactory).

5. Glass tube filled with silicone oil. The glass tube is placed into the ice bath to provide a constant temperature bath. Such an arrangement is used since it is much easier and safer to insert the thermistor inside the glass tube than fitting it into the ice bath. The size of the tube

is chosen such that there will be enough space for the thermistor to slip easily inside the tube without stripping the thermistor leads. The length of the tube is chosen such that enough portion of the tube is placed inside the ice bath so that any transfer of heat, that might take place from the outside through the oil, will be damped; thus, the oil at the bottom of the tube will assume constant temperature.

Test Procedure

The thermistor to be stabilized is insulated. The stabilization process is carried as follows:

1. The thermistor is dipped into the boiling water bath for 10-15 seconds.
2. The thermistor is taken out of the bath and immediately placed in contact with dry ice. This completes one cycle of the stabilization process.
3. The thermistor is allowed to go through two cycles. This would complete the stabilization process.
4. In order to obtain the degree of reproducibility of the stabilized thermistor, the thermistor is fitted into the glass tube down to its bottom. Care must be taken not to strip the thermistor leads.
5. The thermistor reading is taken and recorded using the wheatstone bridge (2 minutes are usually found to be enough for the thermistor to indicate constant reading).

6. The thermistor is taken out of the glass tube, dipped into the boiling water bath for 10-15 seconds and then taken out.
7. The thermistor is allowed to dry by waving it in the air.
8. The thermistor is placed again into the glass tube down to the bottom.
9. With the wheatstone bridge dials set up at the initial reading, the deviation of the final reading from the initial reading is observed by the fall of the wheatstone bridge needle.
10. The final reading of the thermistor is taken and recorded. The difference between the initial and the final reading would give the degree of reproducibility of the stabilized thermistor.

Section II

Calibration of Thermistors

Apparatus

1. Metal cup, 250 ml.
2. Foam rubber, one-inch thick.
3. Anti-freeze liquid.
4. Electrical stirring device.
5. Thermoelectric cooling control unit.
6. Cooling plate (with thermoelectric modules).
7. Thermometer accurate to 0.01°C .
8. Thermocouple.

1"
Scale

9. Amplifier and recorder. The amplifier will have an input range of 50, 100, 200, and 500 μ v and input of 10 mv.
10. Wheatstone bridge for resistance measurements up to 1000 ohms and accurate to 0.1 ohm.

Test Procedure

1. The amplifier is switched on: allow 30 minutes to warm up.
2. The base of the cup is greased with a heat-conductive grease; the cup is placed on the cold plate.
3. The sides of the cup are wrapped with foam rubber (approx. 2" thick). The cup is filled with the anti-freeze liquid (methanol).
4. The thermistor, or thermistors, to be calibrated together with the control (for thermoelectric unit) thermistor and the thermocouple are attached to the bulb of the thermometer by an elastic string; then the thermometer is lowered inside the cup.
5. The thermocouple is connected to the amplifier and the control thermistor is connected to the cooling control unit.
6. The stirring rod is lowered inside the cup and switched on. Care must be taken so as the stirring rod will not touch the thermometer when rotating.
7. A circulation of cold water is allowed to flow into and out of the heat sink.
8. The cooling control unit is switched on and the dial is set up to obtain a certain desired temperature.

9. The recorder is switched on.
10. Wait (approx. 20 mins) until the temperature of the liquid inside the cup assumes a constant value ($\pm 0.005^{\circ}\text{C}$), as indicated by the temperature recording chart.
11. Take the reading of the thermistor using the wheatstone bridge; record the corresponding temperature as indicated by the thermometer (use magnifying glass for reading the thermometer).
12. By varying the cooling control unit dial settings, a set of readings is obtained over the desired ranges of temperature.
13. Draw the calibration chart.

A computer program is available for fitting an equation for the calibration curve. This will allow the calculation of the temperature corresponding to any given thermistor reading with a high degree of accuracy.

APPENDIX VIII

Sample Preparation and Test Procedure

Sample Preparation

For the purpose of determination of the ice-water phase composition, the soil sample inside the cell must be completely de-aired. The displacement of the mercury level in the volume measuring assembly will be accordingly attributed to ice-water phase change on freezing.

De-airing the Soil Sample

1. The soil sample is allowed to dry at room temperature, crushed, and mixed with enough distilled water to bring it to a slurry.
2. Place the soil in a dish.
3. Place the dish inside a desiccator jar.
4. Connect the jar to a vacuum pump and switch on the pump.
5. Continue operation of the vacuum pump until the air bubbles cease to come out of the soil.
6. Fit the rubber membrane into the cell in such a manner that enough space inside the cell is provided for the expansion of the soil sample.
7. Place the cell inside the desiccator jar.
8. The jar is placed on an electric shaker which slowly oscillates in a horizontal direction.

9. The soil should be placed inside the rubber membrane in layers. Place the first layer, one inch thick, inside the membrane, switch on the electric shaker and the vacuum pump. Continue operation of the vacuum pump until the air bubbles cease to come out of the soil. Turn off the electric shaker and the vacuum pump.
10. Repeat step No. 9 until the rubber membrane is filled with the soil.
11. Immerse a millipore membrane filter (pore size 0.2μ) in boiling water until its pores are saturated.
12. Place the membrane filter on the surface of the soil sample.
13. Mount the end plate. Care must be taken, when placing the end plate, that no air bubbles are trapped between the inside surface of the end plate and the soil sample. The bolts are tightened by means of a torque wrench (applied torque 140 Kg-cm).
14. Mount the other end plate.
15. Connect the cell to the volume measuring assembly and the mercury reservoir.
16. Open the valve leading to the mercury reservoir. Also open the valves of the volume measuring assembly. The mercury is allowed to flow into the cell at a slow rate and to rise inside the pipettes to the desired level. Close the valve leading to the 10.0 ml. pipette.

17. Record the initial mercury level in the 1.0 ml. pipette.
18. Apply pressure to the cell at the top of the 1.0 ml. pipette, about 100 psi. Record the corresponding displacement of the mercury level in the pipette. If the displacement is great, more than 0.4 cm^3 , this implies that the soil has not been properly de-aired and more treatment is needed (as described).
19. Open the valve leading to the mercury reservoir and drain the mercury.
20. Disconnect the cell from the mercury reservoir and the volume measuring assembly.

Setting the Dilatometer for Testing

1. The outside surface of the cell is completely covered with one layer of foam rubber $\frac{3}{4}$ " thick. Place the cell inside the insulated box.
2. Connect the cell to the volume measuring assembly and the mercury reservoir.
3. Open the valve leading to the mercury reservoir. Also open the valves of the volume-measuring assembly. The mercury is allowed to flow into the cell at a slow rate, and to rise inside the pipettes to the desired level. Close the valve leading to the mercury reservoir.
4. The soil sample is allowed to consolidate under the weight of the mercury and the excess soil water drains

through the drainage port.

5. Wait until the mercury level in the pipettes ceases to change, i.e., the consolidation process is completed. The drainage port is sealed off.
6. Close the valve leading to the 10 ml. pipette.
7. Record the initial mercury level in the 1.0 ml. pipette.
8. Apply pressure to the cell at the top of the 1.0 ml. pipette. The applied pressure is increased in steps and the corresponding mercury level in the 1.0 ml. pipette is recorded in each increment. This is continued until the highest pressure to be applied in the course of the experiment, is reached. Release the applied pressure.
9. Disconnect the cell and the mercury reservoir.

Test Procedure

1. Open the valve leading to the 10 ml. pipette. Record the mercury level in both pipettes. Close the valve leading to the 10 ml. pipette.
2. Record the thermistor readings at room temperature.
3. Connect the cooling water line to the heat sinks and the water is allowed to flow.
4. Connect the thermoelectric heat pump power leads to the thermoelectric cooling control unit. The thermistor leads are plugged in.

5. Apply the desired pressure to the soil sample at the top of both pipettes.
6. Switch on the thermoelectric cooling control unit, and adjust the settings to give the desired cooling temperature at the two end plates. The two end plate thermistors' readings will indicate when the desired cooling temperature has been reached.
7. Allow the methanol to flow through the copper coil. Adjust its temperature to bring down the air temperature inside the insulated box to the desired temperature.
8. Check the temperature of the two end plates and readjust it to the desired cooling temperature by readjusting the thermoelectric cooling control unit settings.
9. Switch on the recording equipment for recording the variation in cell temperature.
10. Leave the whole assembly overnight to allow the soil sample to cool to the desired temperature.
11. Observe the mercury level in the 1.0 ml. pipette at consecutive intervals of time until it becomes constant. Record the final mercury level.
12. Due to supercooling, the soil water will not freeze at freezing temperatures near 0°C . To initiate freezing, the temperature of the lower end plate is lowered substantially, approximately -4°C ., until the mercury level in the 1.0 ml. pipette starts to rise. The end plate

temperature is then set back to its original value.

13. Observe the mercury level in the 1.0 ml. pipette which is rising due to the expansion of the soil sample on freezing. At the moment it reaches the top of the pipette, close valve no. 3 and open valve no. 2, see Figure 2. Apply additional gas pressure at the top of the 1.0 ml. pipette in order to force the mercury to flow into the 10.0 ml. pipette until it is brought down to its lowest level in the 1.0 ml. pipette. Readjust the gas pressure in the 1.0 ml. pipette. Close valve no. 2, record the mercury level in the 1.0 ml. pipette, and open valve no. 3. This procedure is repeated all through the expansion of the soil sample.
14. Observe the mercury level in the 1.0 ml. pipette until it ceases to change. Record the mercury level.
15. Check the stability of cell temperature on the recorder. Record the thermistor readings and find the average cell temperature.
16. Adjust the thermoelectric cooling control unit settings to the next cooling temperature and proceed as before.
17. After completion of the experiment, turn off the thermoelectric cooling control unit, the cooling water flow, the methanol flow, and release the applied pressure. Allow the soil sample to thaw.

18. Wait until the cell temperature is brought up to room temperature. Open the valve leading to the 10 ml. pipette and wait for the mercury level in both pipettes to stabilize. Record the mercury level in both pipettes. Compare the mercury level readings to those obtained in step no. 1. This is to investigate any mercury leakage that might have taken place in the course of the experiment.
19. Open valve no. 4 and collect the mercury into a graduated cylinder for volume determination of the mercury inside the cell. Calculate the volume of the soil sample.
20. Disconnect the cell from the volume measuring assembly and dismount the cell.
21. Place the soil sample in a dish for water content determination.

END

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