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BY

P. J. WILLIAMS

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# EXPERIMENTAL DETERMINATION OF APPARENT SPECIFIC HEATS OF FROZEN SOILS

by P. J. WILLIAMS\*

## SYNOPSIS

A property of water in porous materials is that it freezes at temperatures below  $0^{\circ}\text{C}$ . There is no single freezing temperature for water in soils. As ice is formed the freezing point of the decreasing quantity of unfrozen water falls further below  $0^{\circ}\text{C}$ . Latent heat of fusion is thus involved in temperature changes over a range extending to several degrees below  $0^{\circ}\text{C}$ . The latent heat and specific heat together constitute an apparent specific heat.

Apparent specific heats for various silt, clay and organic soils have been measured in a calorimeter. The apparent specific heats generally rise as temperatures approach  $0^{\circ}\text{C}$ , and in a clay soil may be ten times as great at  $-1^{\circ}\text{C}$  as at  $-5^{\circ}\text{C}$ . The apparent specific heats for a given temperature depend on whether the soil is freezing or thawing, and on various other factors. The precise determination of heat quantities involved in temperature changes in soil in situ is difficult, mainly because of the several factors influencing the freezing of the soil moisture.

Une propriété de l'eau dans les corps poreux est qu'elle gèle à des températures inférieures à  $0^{\circ}\text{C}$ . Il n'y a pas de température unique de congélation de l'eau dans le sol. Au fur et à mesure que la glace se forme, le degré de congélation descend au dessous de  $0^{\circ}\text{C}$ . Une chaleur de fusion latente est donc en cause lors des changements de température couvrant plusieurs degrés au dessous de  $0^{\circ}\text{C}$ . La chaleur latente et la chaleur spécifique constituent ensemble une chaleur apparente spécifique.

Les chaleurs spécifiques apparentes pour des limons, des argiles et des sols organiques divers ont été mesurées dans un calorimètre. D'une manière générale, la chaleur spécifique apparente s'élève lorsque la température s'approche de  $0^{\circ}\text{C}$ , et dans le sol argileux elle peut être dix fois plus grande à  $-1^{\circ}\text{C}$  qu'à  $-5^{\circ}\text{C}$ . La chaleur spécifique apparente pour une température donnée dépend soit du gel, soit du dégel du sol, et d'autres facteurs divers. La détermination précise des quantités de chaleur en cause lors des changements de température dans le sol, sur place, est difficile, surtout à cause de plusieurs facteurs qui influencent la congélation de l'humidité se trouvant dans le sol.

## INTRODUCTION

When water in finely porous materials freezes, much of it does so at temperatures lower than  $0^{\circ}\text{C}$ . This phenomenon is important in the freezing and thawing of soils. Half of the water in clay soils may be unfrozen at  $-2^{\circ}\text{C}$  and generally, smaller quantities in coarser-grained soils. The equilibrium freezing point temperature is different in different parts of the soil water, so that the process of freezing or thawing of soils generally takes place over a wide range of temperature. Consequently, the quantity of heat required to raise or lower the temperature of soil within this range is made large by the latent heat of freezing (Williams, 1962). In this present Paper, the term "apparent specific heat" refers to such heat quantities to distinguish them from "true" specific heats which are not associated with change of phase.

This Paper describes determinations made with a calorimeter of the specific heats and apparent specific heats of various soils, for temperatures above  $0^{\circ}\text{C}$  and down to  $-10^{\circ}\text{C}$ . The use of such values in estimating the apparent specific heats of soils met with in field problems is discussed briefly.

## CALORIMETRIC INVESTIGATIONS

A calorimeter has been constructed (Fig. 1) that permits measurement of the amount of heat added to or removed from a specimen to raise or lower its temperature by a certain amount. During warming of a sample, the only source of heat is that supplied at a measured rate by a heating coil attached to the specimen holder. During cooling, heat is lost from the sample holder at a practically constant rate determined by the temperature of the outer container which, in this case, is maintained lower than that of the sample holder by a certain amount.

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Temperatures are measured by thermocouples at three points within the sample. Readings from each thermocouple are recorded by a Speedomax recorder with preamplifier, at intervals of 5 minutes or less. Also recorded at 3-minute intervals on the same apparatus are temperatures from four thermocouples placed on the inner side of the outer container (Fig. 1). Under normal conditions these latter readings serve merely to check the operation of the calorimetric equipment.

The calorimeter is immersed in a tank of ethylene glycol solution, cooled by a compressor. During warming tests, when the only source of heat is to be the measured quantity supplied through the heating coil, heat exchange between the sample holder and its surroundings is avoided by maintaining the outer container at substantially the same temperature as the surface of the sample holder. This is achieved by regulation of the temperature of the ethylene glycol in which the calorimeter is immersed. The temperature of the ethylene glycol

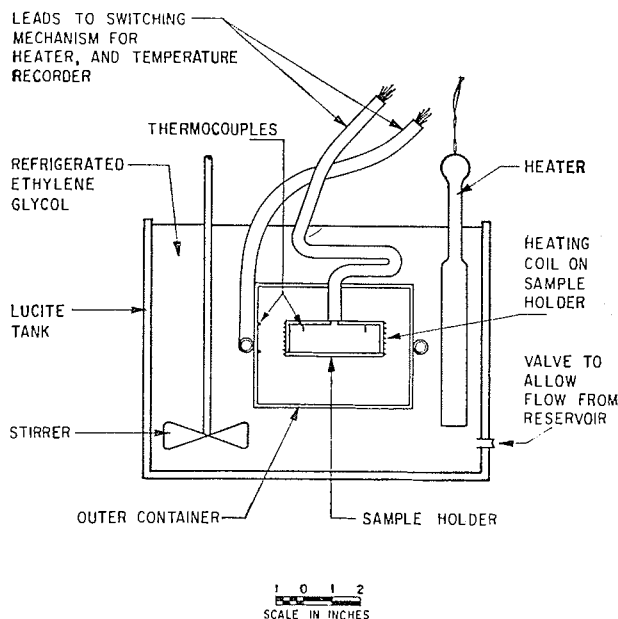


Fig. 1. The calorimeter

normally tends to fall slowly. When its temperature and hence that of the outer container falls about  $0.1^{\circ}\text{C}$  below that of the sample holder, a blade heater in the glycol is automatically switched on until the temperature of the outer container is about  $0.1^{\circ}\text{C}$  warmer. The switching mechanism is operated by the amplified signal received from two thermocouples in series, one on the sample holder and outer container.

During cooling (freezing), when heat is extracted at a nearly constant rate, the temperature of the outer container is maintained consistently lower than that of the sample holder. This is achieved by adjustment of the zero control of the amplifier, such that its output is sufficient to actuate the relay mechanism and heater only when the ethylene glycol becomes cooler than the sample holder by more than the predetermined amount.

The rates of heat extraction during cooling for various temperature differences were measured in calibration tests. In most soil tests, however, observations were made over a cycle involving cooling followed by rewarming to the initial temperature. The rate of heat loss is then calculated by dividing the total heat input in the "thawing" part of the freeze/thaw cycle, by the time taken to cool the sample in the "freezing" part of the cycle. Both

procedures involve minor errors, but the latter gives more consistent results. 3-5 days were normally required for each freeze/thaw cycle.

### *Calculation of results*

Although the method used in calculating the specific and apparent specific heats was occasionally varied in minor respects, the following was the general procedure.

The procedure is similar in most respects for both thawing (warming) tests, and for freezing (cooling) tests. On the time-temperature record on the Speedomax chart, starting from the point corresponding to 0 mV (i.e. 0°C), points are marked for every 0.25 mV, (equal to 0.13°C) change in the temperature, for one of the thermocouples in the soil sample. The heat supplied to or removed from the specimen and sample holder in the time taken for each of these intervals of temperature change ( $\Delta T$ ), is then calculated. For warming tests the relationship used is:

$$0.24 VIt = Q \text{ calories}$$

where

0.24 = conversion factor, cal/J

$V$  = voltage across heating coil on sample holder, volts

$I$  = current through heating coil on sample holder, amps

$t$  = time (seconds) for sample temperature to change through  $\Delta T$

During cooling the relationship used is:

$$Q = \frac{\text{Total heat input for warming part of cycle}}{\text{Time (sec) to complete cooling part of cycle}} \times t_f$$

where  $t_f$  = time (sec) for sample temperature to change through  $\Delta T$ .

The specific heat, or apparent specific heat, is then calculated and expressed in calories per gram of soil per °C:

$$\lambda = \frac{Q - \Delta T C_c}{W_s \Delta T}$$

where:

$W_s$  = total weight of soil sample, g

$C_c$  = calorimeter "constant", cal/°C, i.e. the heat required to change the temperature of the empty sample holder by 1°C.

Values obtained for various soils are shown in Figs 2 and 3 where they are plotted as a function of temperature.

For many soils, the large heat quantities (largely composed of latent heat) involved in temperature changes in the range 0°C to -0.5°C cannot be shown satisfactorily on this type of graph. These heat quantities are shown as a separate value beside each graph. In calculating these values, the same expressions were used, except that  $\Delta T$  has a value of 0.5°C.

### *Experimental accuracy*

The temperatures of the soil are observed with an accuracy of about  $\pm 0.05^\circ\text{C}$  for individual points, but a line drawn through several recorded points generally gives the thermocouple temperature with somewhat greater accuracy. Temperature differences within the sample are normally less than 0.1°C. When there is a very small quantity of ice present locally in the sample, the temperature of the part of the soil furthest from the ice may change more rapidly than that adjacent to it, because of the absorption of latent heat by the melting ice. Temperatures in different parts of the soil may then differ by 0.2°C, and sometimes more. This is probably the main reason for an observation frequently made during thawing: the time/temperature curves indicated that the effect of liberated latent heat is still present at

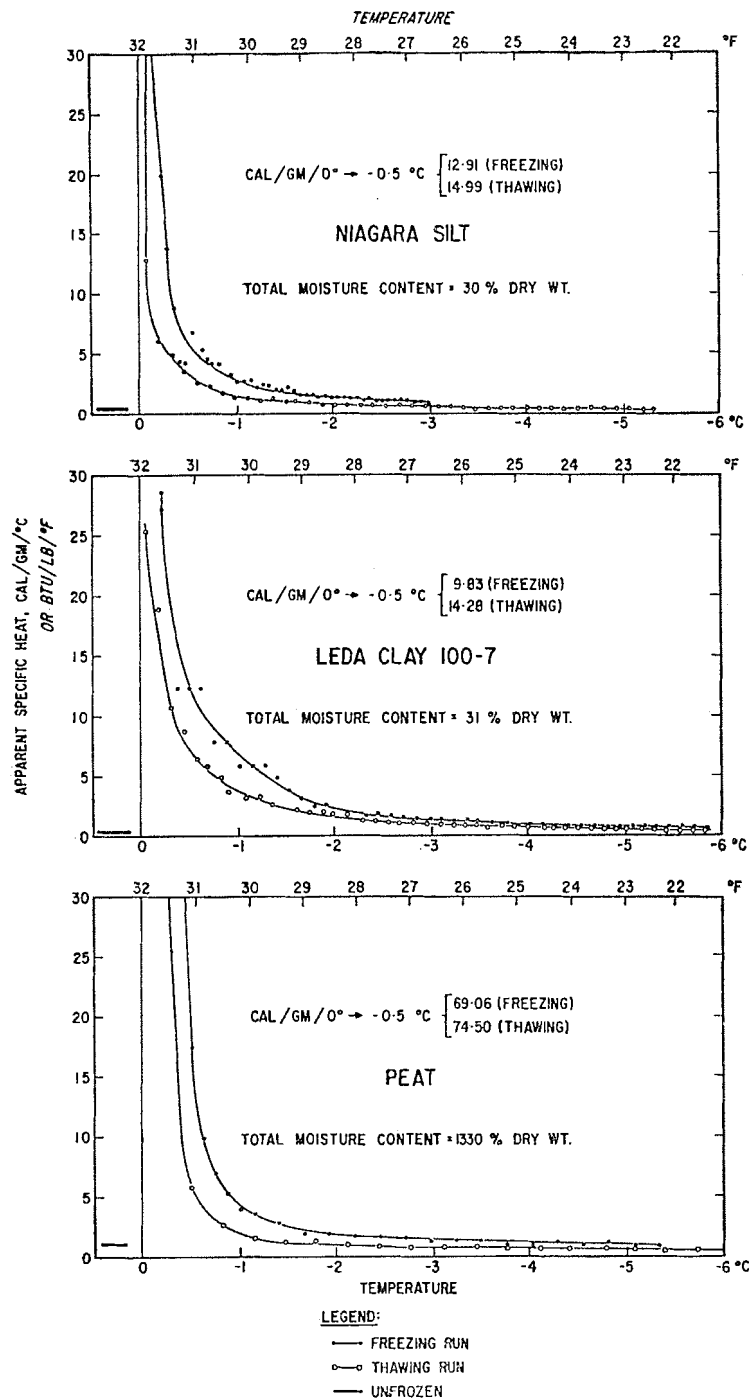


Fig. 2. Specific heats and apparent specific heats of various soils. Also shown are the heat quantities exchanged in a temperature change between 0°C and -0.5°C

recorded temperatures of  $+0.4^{\circ}\text{C}$  or even higher.\* The last remaining ice is probably remote from the thermocouples and thaws at or below  $0^{\circ}\text{C}$ , so that the latent heat of the last thawing ice has been calculated as part of the heat quantity involved in temperature change between  $0^{\circ}\text{C}$  and  $-0.5^{\circ}\text{C}$ . The specific heats above  $0^{\circ}\text{C}$  are therefore shown as a single value on the graphs.

In addition to the quality of the equipment that measures temperature and heat input, the accuracy of the determined specific heats depends substantially on the control of the temperature of the outer container of the calorimeter. If the temperature of the outer container deviates on the average from that of the sample holder, there will be a corresponding unmeasured quantity of heat added to or removed from the specimen. The amount of heat lost will be proportional to the time taken for the test. For soil samples of given weight, the percentage error in apparent specific heat due to this heat loss, will, for practical purposes, be inversely proportional to the measured rate of heat input or extraction. Conversely, for a

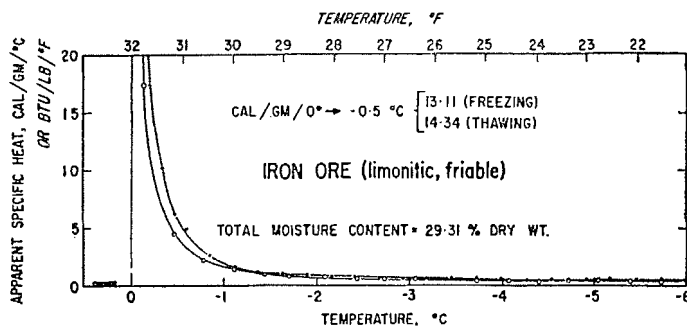


Fig. 3. Specific heats and apparent specific heats of iron ore. Also shown is the heat quantity exchanged in a temperature change between  $0^{\circ}\text{C}$  and  $-0.5^{\circ}\text{C}$

given rate of heat transfer, the error will be proportional to the weight of the sample. During tests of long duration (as in the case of most soils) errors of this type could be reduced by occasional slight readjustments of the temperature controlling mechanism.

The amount of heat involved in warming or cooling the sample holder was determined in calibration tests. The value used for this calorimeter "constant" was  $87 \text{ cal/}^{\circ}\text{C}$ . It may be in error by 1 to 2 calories in some cases, mainly because of minor changes that became necessary in the mounting of the thermocouples. Since the weight of the sample is normally about 200 g it follows that an error of perhaps 1–2% may occur in the observed specific heats, when these are very low (e.g.,  $0.2 \text{ cal/g/}^{\circ}\text{C}$ ). For higher (apparent) specific heats the error due to inaccuracy of the calorimeter constant becomes negligible.

The effects of the limitations of instrumental accuracy and of the various sources of error mentioned here, together with other minor ones, are not predictable quantitatively for every test. To some extent, in each test these effects cancel out. In a series of five tests carried out with distilled water, the observed values of the specific heat showed a standard deviation of  $0.04 \text{ cal/g/}^{\circ}\text{C}$ , or 4.0%, about the true value. The average of the deviations of the observed values from the true values was  $0.02 \text{ cal/g/}^{\circ}\text{C}$  (Table 1).

Although this gives a good indication of the accuracy that can be obtained with the calorimeter, investigations of soils involve additional complications, especially those associated with the rapid change of apparent specific heats with temperature. These are illustrated by tests on two different-sized samples from the same soil. For each soil sample the

\* This may be compared with temperatures which are recorded by a thermocouple placed in an ice bath to which heat is being slowly supplied. Temperatures of several tenths of a degree above  $0^{\circ}\text{C}$ , are observed even while ice is still present, but there is no marked increase in rate of temperature rise until all the ice has melted.

Table 1  
Control tests carried out on distilled water

| Temp. °C | Specific heat cal/g/°C |                                    | Deviation = $x$ |      |
|----------|------------------------|------------------------------------|-----------------|------|
|          | observed               | Hdbk of physics<br>and chem., 1962 | cal/g/°C        | As % |
| 12       | 0.978                  | 1.001                              | -0.023          | 2.3  |
| 3.3      | 0.958                  | 1.005                              | -0.047          | 4.7  |
| 7.6      | 0.996                  | 1.002                              | -0.006          | 0.6  |
| 17.3     | 1.032                  | 0.999                              | 0.033           | 3.3  |
| 6.5      | 1.017                  | 1.003                              | 0.014           | 1.4  |

$$\text{Standard deviation} = \sqrt{\frac{\sum x^2}{5}}$$

$$= 0.04 \text{ cal/g/°C}$$

$$= 4\%$$

$$\text{Average of deviations} = 0.02 \text{ cal/g/°C}$$

apparent specific heat was obtained for fifty-two temperatures at increments of 0.13°C, from -0.5°C to -7.3°C. With the two values thus obtained for each temperature (one from each test), a mean value was calculated. The deviations of the observed values from their respective means were expressed as a percentage of these means. The standard deviation of these percentages for the whole range was 9.4%. The smoothed curves drawn through the two sets of observed points are closely similar, however (Figs 6(a) and (b)). The freezing process (and consequently the apparent specific heats) are unlikely ever to be exactly similar in any two tests on the same soil, because natural soils are not entirely homogeneous. Small differences in the nature and distribution of voids, as well as different stresses that may arise at each freezing, result in slightly different amounts of moisture freezing or thawing at a given temperature.

These considerations, together with the fact that the temperature of soils in situ is rarely definable to within 0.1°C, show that the reproducibility of the results is quite satisfactory for practical purposes.

#### *Experimental results and their interpretation*

Figs 2 and 3 illustrate the dependence of apparent specific heat on type of soil. At least two freeze/thaw tests were made on each of five soil types and in some cases six or more. These repeated tests showed that the apparent specific heats are independent of the rate of temperature change, at least for those rates likely to occur under field conditions. The grain-size composition of the soils is shown in Fig. 4. The finer-grained soils have, in general, higher apparent specific heats. This is a result of the larger quantity of water that freezes, or thaws, at temperatures below 0.5°C in these soils. For each increment of temperature change in such soils, there is a correspondingly larger component of latent heat involved. The freezing of water in soils at these temperatures is due mainly to the state of stress (pF) produced in the soil water by capillary and other effects (see Schofield, 1935; Edlefsen and Anderson, 1943). In general the stresses developed in the water in finer-grained soils are such that larger quantities of water will freeze at temperatures below -0.5°C than in coarser-grained soils. The conspicuous peak shown in the freezing curve of the Leda clay (Fig. 5(a)) is probably due to an unusual amount of water in the soil under a state of stress resulting in freezing at about -1.1°C. This may be compared with the sharp drop in moisture content for a small change of suction, that occurs in the suction-moisture content curves (see e.g. Coleman and Croney, 1961) for some clay soils.

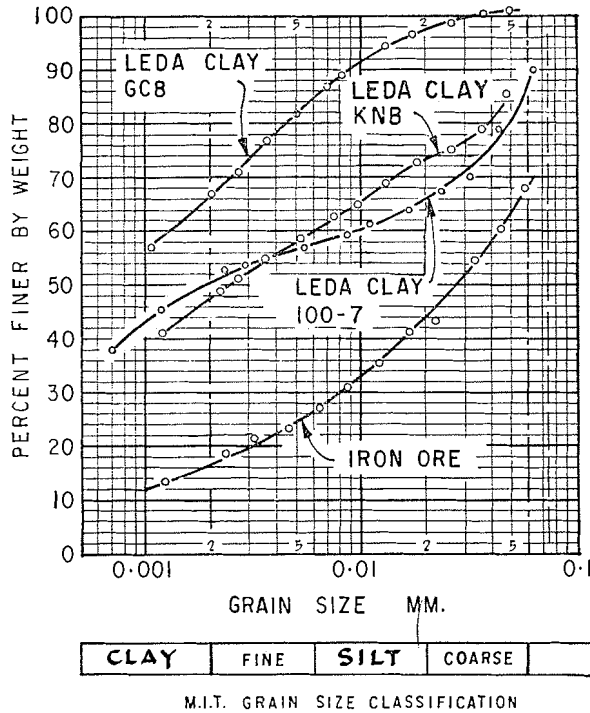


Fig. 4. Grain-size composition curves for soils investigated calorimetrically. The curve for Niagara silt was closely similar to that for iron ore, only differing by being 0 to 10% higher in the range 0.02 to 0.06 mm

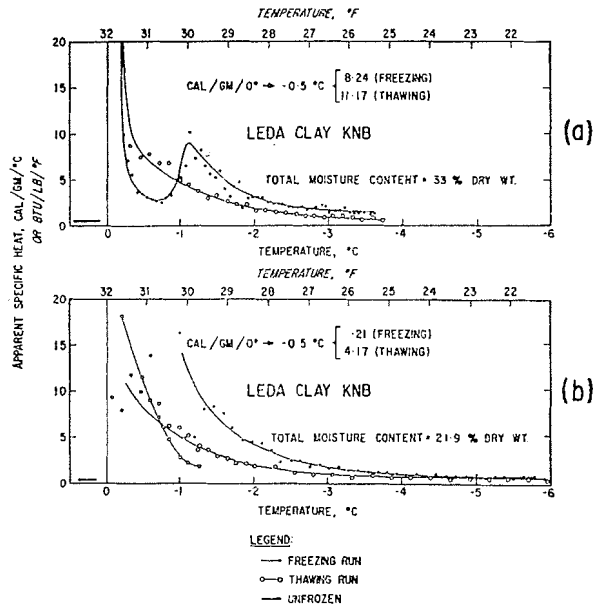


Fig. 5. Specific heats and apparent specific heats of Leda clay at natural moisture contents (a) 33% dry weight and (b) partly dried (21.9% dry weight). In the latter case, freezing began at a temperature of  $-0.99^{\circ}\text{C}$ ; two thawing curves are shown, corresponding to minimum temperatures during freezing of  $-8.3^{\circ}\text{C}$  and  $-1.4^{\circ}\text{C}$

Different values of apparent specific heat are obtained depending on whether the soil is freezing or thawing and in the latter case on the lowest temperature reached during freezing (Fig. 5(b)). This may be compared with the different moisture contents under similar stresses which exist in porous materials undergoing drying in the one case and wetting in the other.

In the case of compressible soils, a freeze/thaw cycle on previously unfrozen soils gives results different from those obtained in subsequent tests. This is illustrated by the examples in Figs 6(b) and 6(c). It will be seen that in the latter example the apparent specific heat for temperatures of  $-0.5^{\circ}\text{C}$  to  $-3.0^{\circ}\text{C}$  are smaller, whereas the heat quantities involved between  $0^{\circ}$  and  $-0.5^{\circ}\text{C}$  are larger in the second freezing than in the first. In this respect, freezing may be regarded as similar to drying, which is well known as causing consolidation (see e.g. Warkentin and Bozozuk, 1961) and a corresponding change in the stress/moisture content relationships of compressible soils. After the second freezing, subsequent freeze/thaw cycles do not increase the effect significantly unless carried out to even lower temperatures. The effect of subsequent freezing to temperatures lower than about  $-2.5^{\circ}\text{C}$  is small, in any case. It is to be expected that consolidation due to applied loads will similarly result in somewhat different apparent specific heats. It might be noted that the different apparent specific heats observed on freezing and on thawing are not solely the result of consolidation during the freezing (or "drying") process, since they occur even in the case of non-compressible soils.

Particularly important is the effect shown, for example, by comparison of Figs 5(a) and 5(b) where apparent specific heats were determined on a saturated sample and on a similar sample from which about one third of the water was removed by drying. The apparent specific heats for temperatures below  $-0.5^{\circ}\text{C}$  were rather similar in both cases. This illustrates the point that there is a considerable amount of water held in soils, which freezes in the range  $0^{\circ}\text{C}$  to perhaps  $-0.2^{\circ}\text{C}$ , and which can be varied or even totally removed with little alteration of the apparent specific heats for lower temperatures.\* Slightly lower apparent specific heats are to be expected when the total moisture content is very high. This is because, per unit weight, there will be more ice and less soil material. As the apparent specific heats are largely composed of latent heat of freezing of water under the influence of the porous structure of the soil, the latent heat quantity will be reduced if there is less soil material.

#### VALUES OF APPARENT SPECIFIC HEATS FOR APPLICATION IN FIELD PROBLEMS

##### *Considerations involved in comparison of experimental and field values*

Because of the wide variety of soil type and moisture content, and the complex relationships involved in the freezing of soil water, it is rarely possible to estimate precisely the heat quantities involved in temperature changes below  $0^{\circ}\text{C}$ , on the basis of grain-size composition. Even for soils tested calorimetrically, field conditions may be such as to give rise to values substantially different from those determined experimentally. The important factors will be summarized here.

1. In the finer-grained, so-called frost susceptible soils, substantial quantities of water migrate to the freezing soil, the amount depending, among other factors, on water availability, rate of freezing, and soil type (see e.g. Penner, 1958). The addition of water in this way cannot, of course, occur to the calorimeter sample (although internal migration will occur). Large quantities of heat are involved in the freezing of this additional water. It is not possible to predict the amount accurately but it can be allowed for by measurement of the total moisture content of the frozen soil.

2. Theoretical considerations, based on the work of Schofield, 1935; Edlefsen and Anderson, 1943; *et al.*, suggest that over-burden pressures or high pore-air pressures, will change

\* This and the ensuing remarks are more easily understood when it is remembered that most of the ice in freezing soils is in discrete (and often quite large) masses larger than pore size. Variation in size of the ice masses will not alter the pore structure and neither therefore the freezing temperatures of the remaining unfrozen water within the pores.

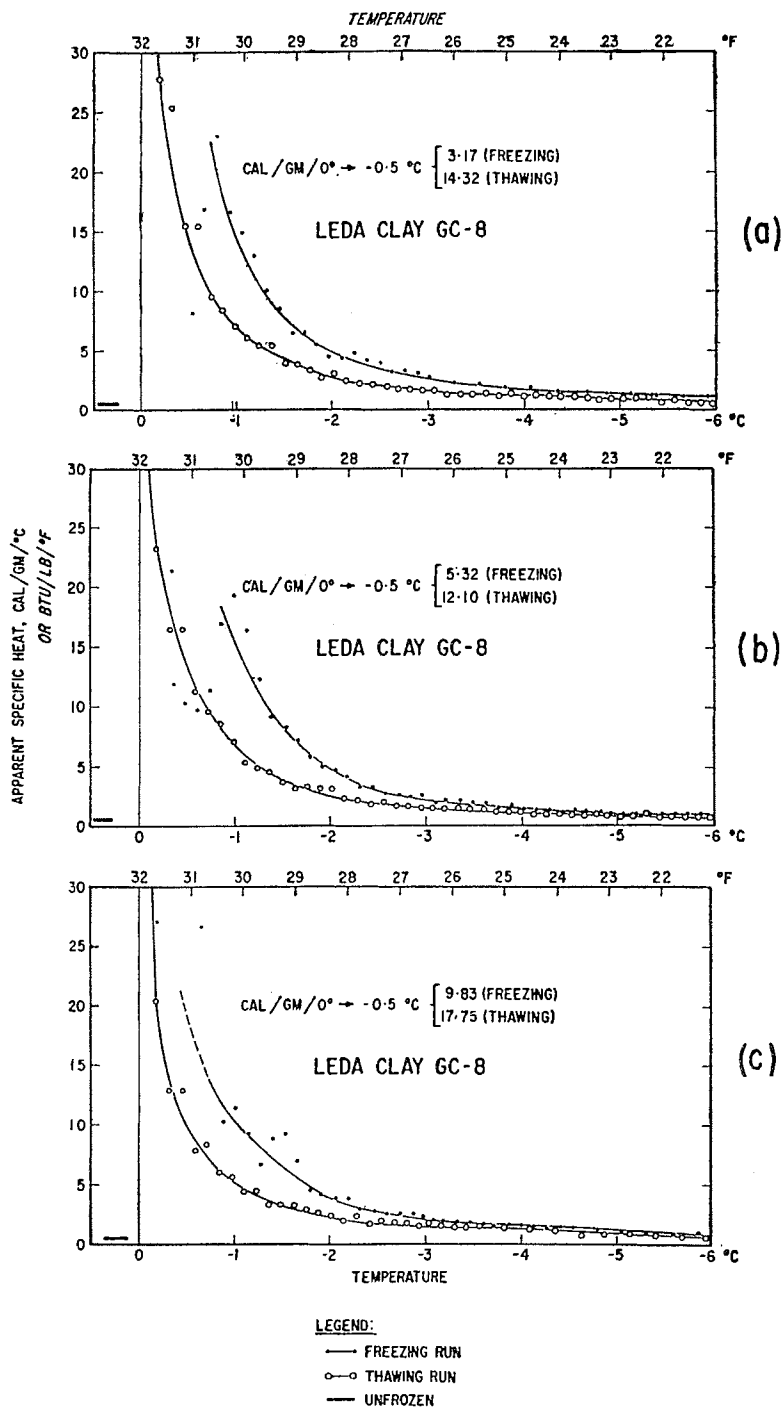


Fig. 6. Specific heats and apparent specific heats of Leda clay GC-8. (a) and (b) show the results of identical tests on identical samples, and illustrate the reproducibility of the calorimetric observations. (b) and (c) illustrate the different results obtained on first freezing (b) and second freezing (c) of a sample

the amount of water that freezes over a given temperature range. There will be corresponding changes in apparent specific heats. At present these effects are not fully understood quantitatively but they are probably of little significance at one to two metres depth in the ground, without other over-burden (compare Penner, 1959).

3. Although the freezing of water in soils at temperatures below 0°C is broadly related to the pore sizes and void ratio and thus to grain-size composition of the soil, other effects, especially those due to salts and to surface forces of the clay particles, may be significant. Thus, Leonards and Andersland (1960) give information on the freezing of a clay, which, on the basis of grain-size composition alone, does not seem compatible with the findings presented in this paper. Except possibly for obviously saline soils, the occurrence of these effects is not easily predictable at present without calorimetric tests.

Work recently completed indicates that the suction-moisture content relationship of a soil can be used to obtain a good estimation in many cases, of the apparent specific heat. This will be considered in a subsequent publication.

#### CONCLUSIONS

1. Determinations of the apparent specific heats of various soils at temperatures below about 0.5°C have been made and are presented graphically. The heat quantities involved in warming and cooling through 0° to -0.5°C are also given.

2. Apparent specific heats are dependent on soil type, being generally larger for finer-grained soils.

3. For a given soil apparent specific heats are dependent on:

- (a) The temperature, being in general larger at temperatures nearer 0°C.
- (b) Whether the soil is freezing or thawing and, in the latter case, the lowest temperature reached during freezing.
- (c) In compressible soils, whether the soil has been previously frozen and thawed, or dried and rewetted, and if so, to what extent.

Differences in total moisture content, however, within the range that might occur naturally have little effect on the apparent specific heats for temperatures below about -0.5°C.

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