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RELATIONSHIP BETWEEN THE ICE AND UNFROZEN WATER PHASES IN FROZE--ETC(U)
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*Relationship between the ice and unfrozen water phases
in frozen soil as determined by pulsed nuclear magnetic
resonance and physical desorption data*

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*Cover: Region of detection of nuclear
magnetic resonance analyzer.*

CRREL Report 82-15

June 1982



*Relationship between the ice and unfrozen water phases
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A.R. Tice, J.L. Oliphant, Y. Nakano and T.F. Jenkins

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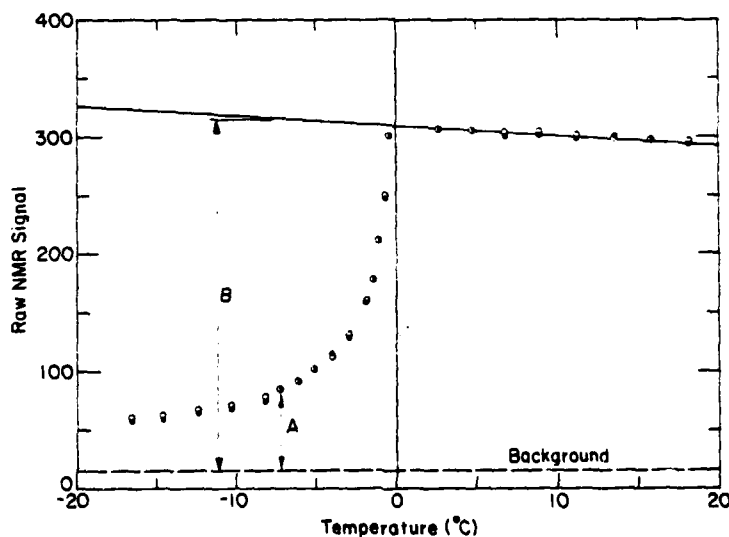
CRREL Report 82-15

The last paragraph on pg. 2 should read as follows:

The raw NMR data from the warming run are shown in Figure 1 for both samples. It can be seen from this figure that the first pulse amplitude increases as temperature decreases for the samples containing no ice, but the first pulse amplitude is reduced when water begins to freeze just below 0°C. Unfrozen water contents are calculated by extending the line drawn through the data points taken with no ice present down to low temperatures by linear regression (solid line, Fig. 1). The unfrozen water content is then calculated as the total water content (determined gravimetrically) multiplied by the distance from the experimental measurement to the background amplitude (A in the figure) and then divided by the distance from the regression line to the background amplitude reading (B in the figure).

Unfrozen water content vs temperature data are

The following figure should replace Figure 1:



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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) An experiment is described that demonstrates the balance between the ice and the unfrozen water in a frozen soil as water is removed. Nuclear magnetic resonance (NMR) is used to monitor the unfrozen water content as the soil is dehydrated by a molecular sieve material. Our results show that the unfrozen water content of a Morin clay soil remains constant until the total water content has been reduced to the point where no ice remains in the system. Once the ice is depleted, the unfrozen water content determined by NMR corresponds to the total water content of the soil determined by the weight of water removed by the molecular sieve material. Thus the validity of utilizing NMR in determining unfrozen water contents vs temperature is established.		

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PREFACE

This report was prepared by A.R. Tice, Physical Science Technician, and Dr. J.L. Oliphant, Research Physical Scientist, of the Earth Sciences Branch, Research Division, Dr. Y. Nakano, Chemical Engineer, of the Applied Research Branch, Experimental Engineering Division, and T.F. Jenkins, Research Chemist, also of the Earth Sciences Branch, Research Division, U.S. Army Cold Regions Research and Engineering Laboratory. Funding for this research was provided by DA Project 4A161102AT24, *Research in Snow, Ice and Frozen Ground*, Task A, *Properties of Cold Regions Materials*, Work Unit 002, *Properties of Frozen Soil*.

Lawrence Gatto and Frederick Crory of CRREL technically reviewed the manuscript of this report.

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RELATIONSHIP BETWEEN THE ICE AND UNFROZEN WATER PHASES IN FROZEN SOIL AS DETERMINED BY PULSED NUCLEAR MAGNETIC RESONANCE AND PHYSICAL DESORPTION DATA

A.R. Tice, J.L. Oliphant, Y. Nakano and T.F. Jenkins

INTRODUCTION

In cold regions engineering, the difficulty of building stable structures can be compounded by the migration of water to freezing surfaces, which can result in frost heaving that severely damages buildings, roads and airfield runways. Secondary frost heaves can result from the migration of water through partially frozen ground from warmer to colder sites within a frozen soil-water matrix. This water migration through frozen soil and the resulting secondary frost heave are of concern to engineers currently designing chilled gas pipelines.

Even though many different theories on the mechanics of frost heave have been proposed, there is one point of agreement: that a continuous film of unfrozen water (with a thickness highly dependent on temperature) exists in fine-grained frozen soil around 0°C and that this film is the conduit through which liquid water is supplied to a growing ice lens. The existence of liquid water in a frozen soil-water system was first demonstrated by Bouyoucos (1917) by use of a dilatometer. Bouyoucos reported that the amount of unfrozen water decreased as the temperature was progressively lowered below 0°C and that the amount of unfrozen water at a given temperature increased with decreasing soil particle size. Since the pioneering work of Bouyoucos, various types of calorimetry have successfully determined the amount of unfrozen water in frozen soils (Nersesova 1953; Lovell 1957; Kolaian and Low 1963; Williams 1963, 1964; Nersesova and Tsytovich 1966; and Anderson and Tice 1973).

More recently Tice et al. (1978a, b) have used

nuclear magnetic resonance (NMR) analyzers and reported a simple procedure for determining a soil phase composition curve (unfrozen water content vs temperature data) that is independent of the soil characteristics. They have shown that their unfrozen water curves determined by NMR agree favorably with those from isothermal calorimetry and dilatometry. However, the accuracy of any of the available techniques has not been substantiated by direct physical measurements.

This investigation was undertaken to determine the validity of making unfrozen water determinations from NMR results and to show the interdependency of the ice phase and the unfrozen water phase of a frozen soil undergoing dehydration. This investigation contributes to a two-part analysis of water transport mechanisms through frozen soils. The first part (Nakano et al. 1982) discusses the movement of water from a wet soil column to a dry soil column under isothermal conditions with no ice present. The second part (Nakano et al. in prep.) investigates the movement of water under isothermal conditions from a wet soil column containing ice to a dry soil column with no ice. A major question in this second analysis is whether the ice phase or the unfrozen water phase is depleted as a frozen soil-water system is dehydrated. This current report provides a satisfactory answer.

USE OF NUCLEAR MAGNETIC RESONANCE

The nuclei of some atoms, including hydrogen, resemble miniature bar magnets that will align

along a strong magnetic field. When radio frequencies are applied, these atoms will absorb enough energy to realign to another stable position within the magnetic field. If a thawed soil-water mixture is placed in a pulsed NMR analyzer and a single 90° radio frequency pulse is applied, a voltage (which corresponds to the amount of atoms absorbing energy) is induced in a receiver coil that surrounds the sample. It is this voltage that is detected by the NMR analyzer. The magnitude of the voltage (minus the background) is directly proportional to the amount of the water (hydrogen) in the mixture; therefore, the NMR can conveniently be used as a water detector for a given soil.

The pulsed NMR used in this investigation was a Praxis model PR-103 operated in the 90° mode, 0.2-s clock, and at fast scan. First pulse amplitudes (signal intensities) were measured for each sample starting at the first test temperature of -16.5°C.

This instrument was factory-tuned to detect only hydrogen. The above mode of operation was selected because only hydrogen atoms associated with liquid water were of interest. Other modes are available that can detect hydrogen atoms associated with ice or those which make up the soil structure.

UNFROZEN WATER CONTENT DETERMINATION

Distilled, deionized water was added to 60 g of oven-dried Morin clay to form a mixture of 20% water to clay by weight. After mixing, the soil and water were sealed for 1 week to allow for complete moisture equilibration. Then, two replicates were compacted to a wet density of 1.72 g/cm³ in glass test tubes to a volume of 9 ± 0.05 cm³. A copper-constantan thermocouple was inserted into the center of the soil of one sample to monitor temperature (number 36 gauge wire was used because it does not affect the NMR signal due to displacement of soil). Both test tubes were sealed with rubber stoppers to prevent moisture changes. A third sample was taken for a water content determination.

Following sample preparation, the test tubes with soil were placed in a precision temperature bath containing an ethylene glycol-water mixture and allowed to equilibrate at the first test temperature, -16.5°C, overnight. The following morning a background reading on the NMR spectrometer was recorded and the test tubes were sequentially removed from the bath, wiped dry, and measured

in the NMR analyzer. The sample temperature and NMR signal amplitude were recorded (elapsed time, about 4 s) and the samples were reinserted in the bath.

After both samples had been analyzed, the bath temperature was then increased by about 2°C. About 30 min was allowed for the samples to attain thermal equilibrium at the new temperature and the measurements were repeated. After being raised to approximately -3°C, the bath temperature was increased at 0.4°C intervals until the samples were completely melted. Above 0°C, NMR readings were obtained at about 2.5°C increments until a temperature of about 18°C was reached. These above-freezing measurements were made to determine how much the NMR signal intensity is affected by temperature and to establish a paramagnetic regression line. Substances that are even slightly paramagnetic, including water, yield decreasing signal amplitudes with increasing temperatures.

Following the last NMR warming determination at about 18°C, a complete cooling curve was obtained by reversing the above procedure. Unfrozen water contents were calculated in the following manner. First the paramagnetic linear regression line was calculated by fitting the thawed experimental data. All NMR signal intensities that increased with decreasing temperatures were included in the regression. If a reading was lower than 1% of the projected regression line, it was assumed to be partially frozen. (Tice et al. [1981] have shown a linear relationship between the NMR signal intensity and temperature, which may occur well below 0°C in soil-water systems until nucleation occurs.) After nucleation, a reduction in signal intensity which corresponds to the amount of water present as ice was recorded.

The raw NMR data from the warming run are shown in Figure 1 for both samples. It can be seen from this figure that the first pulse amplitude increases as temperature decreases for the samples containing no ice, but the first pulse amplitude is reduced when water begins to freeze just below 0°C. Unfrozen water contents are calculated by extending the line drawn through the data points taken with no ice present down to low temperatures by linear regression (solid line, Fig. 1). The unfrozen water content is then calculated as the total water content (determined gravimetrically) multiplied by the distance from the regression line to the first pulse amplitude reading (A in the figure) and divided by the distance from the regression line to the background amplitude reading (B in the figure).

Unfrozen water content vs temperature data are

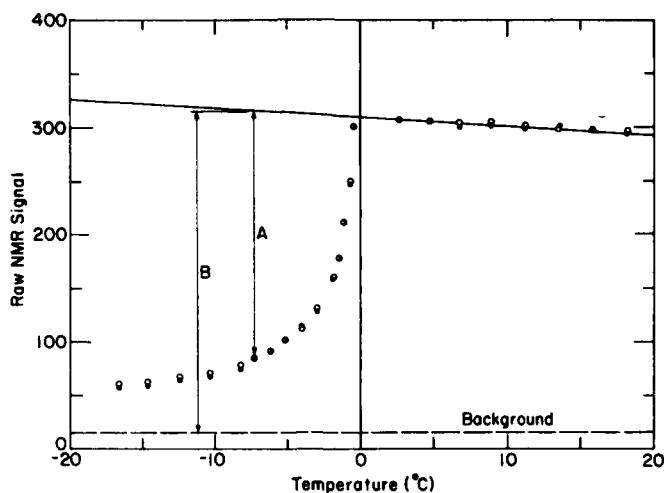


Figure 1. Raw nuclear magnetic resonance data vs temperature for two samples of Morin clay (warming run). (A and B are defined in the text.) \circ designates the sample used in the desorption experiment, \bullet designates the sample used to show the reproducibility of measurements.

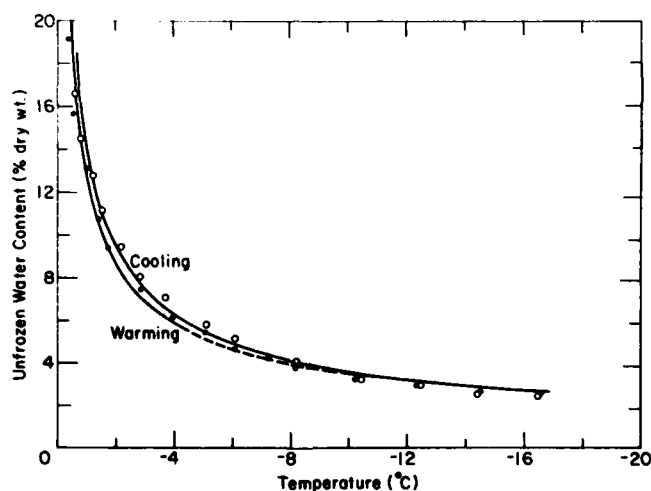


Figure 2. Unfrozen water content vs temperature curves for Morin clay.

shown in Figure 2 and Table 1 for one sample only. The second sample was run to check the reproducibility of the measurement, which proved to be excellent in all cases (Fig. 1).

DESORPTION EXPERIMENT

After completing the unfrozen water content cooling run, both frozen samples were immersed

in a second precision temperature bath preset and maintained at $-1^{\circ} \pm 0.003^{\circ}\text{C}$ throughout the 300-hr testing period. The bath contained an ethylene glycol-water mixture as a coolant. The two test tubes were allowed to thermally equilibrate overnight. A third test tube, containing 7 ± 0.004 g of oven-dried molecular sieve material (average diameter, 2 mm) and sealed with a rubber stopper, was inserted in the bath to equilibrate at the same temperature for the same period. This molecular

Table 1. Unfrozen water content vs temperature data for Morin clay.

<i>Warming curve</i>		<i>Cooling curve</i>	
<i>Temperature (°C)</i>	<i>Unfrozen water content (% dry wt)</i>	<i>Temperature (°C)</i>	<i>Unfrozen water content (% dry wt)</i>
-16.55	2.52	-0.67	16.60
-14.57	2.66	-0.85	14.53
-12.4	2.94	-1.29	12.79
-10.26	3.23	-1.58	11.13
-8.18	3.78	-2.26	9.47
-7.26	4.20	-2.91	8.08
-6.12	4.69	-3.75	7.11
-5.09	5.45	-5.12	5.79
-3.99	6.15	-6.15	5.16
-2.91	7.46	-8.24	4.05
-1.81	9.41	-10.45	3.23
-1.45	10.79	-12.42	2.94
-1.06	13.07	-14.41	2.53
-0.61	15.71	-16.49	2.45
-0.43	19.23		

sieve material, distributed by the Perkin-Elmer Corporation, is intended to be used as an absorbent for cryogenic vacuum pumps. This material was selected over other potential drying agents (i.e. salts or powdered clays) because it is insoluble, dense, and can be completely removed from the frozen soil surface simply by pouring.

Following thermal equilibration, all of the molecular sieve material was poured into one of the two test tubes containing the frozen Morin clay. Figure 3 is a schematic representation of this experimental setup. NMR readings corresponding to the amount of unfrozen water were periodically taken throughout the day on both test tubes. As was the case in the unfrozen water content determinations, the purpose of the second tube was to monitor the reproducibility of the measurements and confirm the stability of the NMR instrument throughout the experiment.

Following an elapsed time of 24 hours, the molecular sieve material was removed and immediately replaced with an identical amount of the oven-dried (at 105 °C) and precooled material. The amount of water absorbed by the molecular sieve material during each 24-hour period was determined by re-weighing this material. The process of taking periodic NMR measurements during the day and replacing the molecular sieve material at the end of each 24-hr period was continued for 12 days. The amount of water removed during the 12-day period by the molecular sieve material is shown in Table 2 and Figure 4. The unfrozen wa-

ter content measurements of the sample undergoing desorption and those determined by NMR are presented in Figure 5 and Table 3.

In the above desorption experiment, the total water loss as measured by weight gain in the molecular sieve material was compared to the NMR signal (Fig. 5). However, the NMR looks at only a portion of the sample, while the gravimetric data relate to the entire sample. Therefore, it could be that any comparisons between the two measurements would not be valid unless the NMR detected a large portion of the sample. To resolve this question it was necessary to determine the portion of the sample that was within the NMR detection region. To accomplish this, Morin clay was prepared at a water content of 20% dry weight and compacted in layers in a glass test tube. NMR readings were taken at room temperature following addition of each layer until a soil height of 46 mm was obtained, which corresponded to the soil height used in the desorption experiment. If the NMR were accurately measuring total water content, a linear relationship between the NMR signal intensity and soil height would be observed within the sample.

The height of the soil in the tube is plotted vs the NMR signal in Figure 6. As can be seen from Figure 6, the coil does not detect unfrozen water contained in small areas (C) near the bottom and the top of the soil sample. In the center part of the soil sample (A), where the relation between the height of soil and the NMR signal is almost linear, the

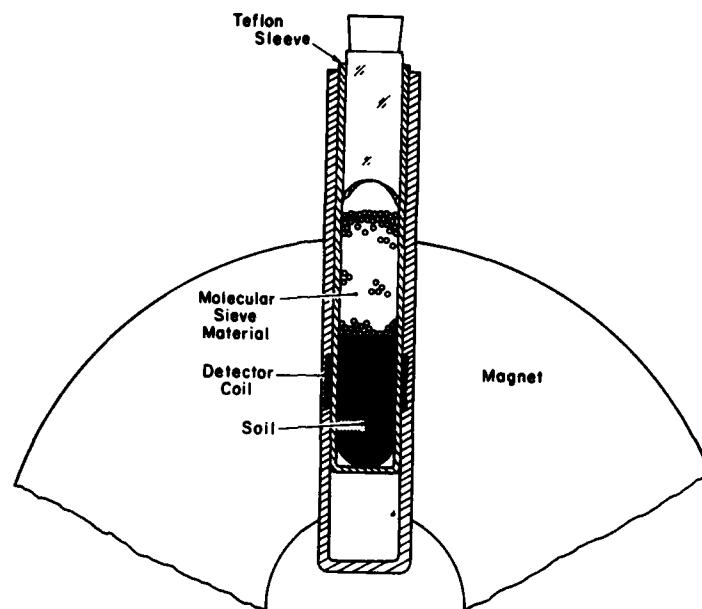


Figure 3. Nuclear magnetic resonance experimental setup.

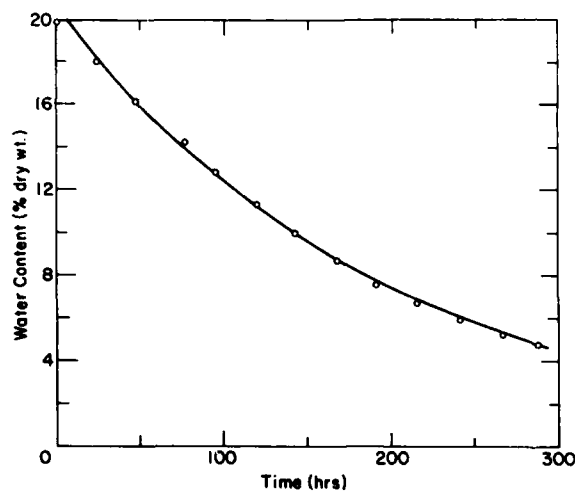


Figure 4. Desorption data for Morin clay at -1°C .

unfrozen water emits signals proportional to the amount of unfrozen water to the detective coil. This center core (A) was determined to be about 54% of the total volume of the sample. In the remaining part of the soil sample (B), which we may call the transitional region, the unfrozen water was detected, but the NMR signal was no longer proportional to the amount of unfrozen water. This transitional region (B) was determined to be

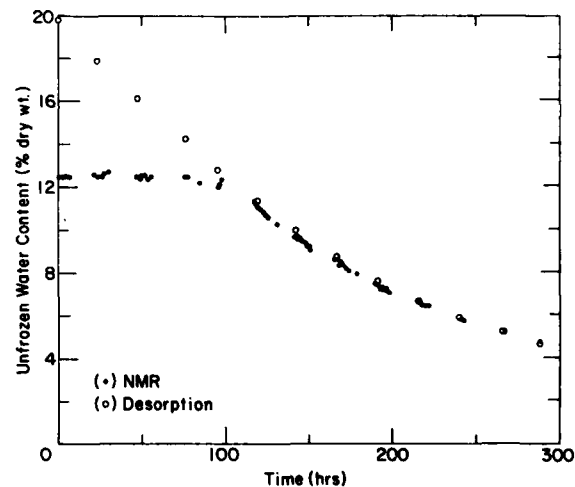


Figure 5. Simultaneous NMR (unfrozen water content) and desorption (total water content) data for Morin clay at -1°C .

about 34% of the total volume of the soil sample. Although the sensitivity of NMR detection of unfrozen water in the transitional region is low, it is reasonable to conclude that any decrease or increase of unfrozen water in the combined region (A + B) of the center core and the transitional region affects NMR readings. The volume of this combined region was 88% of the total sample soil volume.

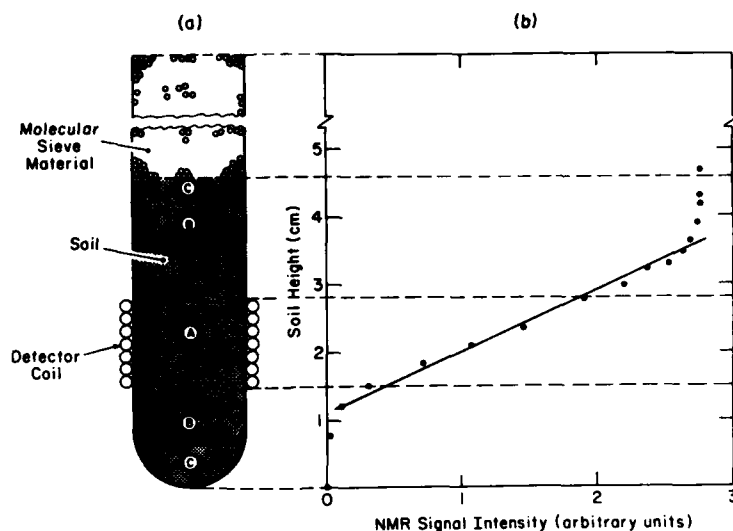


Figure 6. Region of detection of the NMR analyzer: (a) dimensions of the test sample, (b) height of the soil sample vs NMR signal intensity. (A-C are described in text.)

It is very important that a zone of nondetectability be maintained at the top of the soil column to ensure that the water adsorbed by the molecular sieve material is not being detected by the NMR. This zone of nondetectability was confirmed by a simple experiment in which an air-dried Morin clay (about 0.7% water content) was compacted in a glass test tube to a height of 46 mm. A reading of 32 was observed on this air-dried Morin clay by the NMR at room temperature. Some molecular sieve material was soaked in water and then allowed to air dry. The molecular sieve material was placed in a separate glass test tube and the NMR yielded a reading of 680 (which is considerably higher than that for the thawed Morin clay at a water content of 20% dry weight). The moistened molecular sieve material was then poured on top of the air-dried Morin clay. The reading of 32 was maintained, proving that any water adsorbed by the molecular sieve material would not be detected by the NMR.

DISCUSSION

From the unfrozen water content vs temperature curves presented in Figure 2, Morin clay should have approximately 12.8% or 14.5% unfrozen water, respectively, at -1°C depending on whether the warming or cooling curve is used. For the desorption experiment, the samples were fro-

zen at -16°C and warmed to -1°C , so that the unfrozen water content should correspond to the value predicted from the warming curve, 12.8%. When the NMR readings obtained during the first 100 hours of the desorption experiment are converted to unfrozen water contents (shown in Fig. 5), a value of about 12.7% is obtained, very close to the 12.8% found in the first test (Fig. 1).

Neglecting the vapor phase, the water in partially frozen soil can be separated into two types (Anderson et al. 1978), giving a simple equation of the form

$$W = W_u + W_i \quad (1)$$

where W = total sample water content

W_u = unfrozen water content

W_i = ice content.

Anderson et al. (1978) state that "any removal of water from the soil by evaporation or sublimation must be accomplished at the expense of W_i ," and that as W is reduced and W_i becomes equal to zero,

$$W = W_u \quad (2)$$

Let us examine this hypothesis from the data in Figure 5. At time zero we have a total sample water content of about 20% (open circles). The unfrozen water content determined by NMR is about

Table 2. Water desorption vs time for Morin clay.

Elapsed time (hr)	Weight of water removed (g)	Sample water content (% dry wt)
0	0	19.82
24	0.240	17.96
48	0.233	16.16
77	0.246	14.25
96	0.181	12.85
120	0.191	11.37
143	0.175	10.02
168	0.164	8.75
192	0.142	7.65
216	0.121	6.71
241.5	0.101	5.93
267	0.085	5.27
288	0.061	4.80

12.7% (closed circles). After 24 hours, the first molecular sieve sample was removed and weighed. From the weight gain of the molecular sieve material, the sample water content was reduced by about 2% (Table 2). The unfrozen water content as measured by the NMR signal, however, remained constant during this 24-hr period (Table 3 and Fig. 5). As each increment of water was removed, there was no change in the unfrozen water content. Thus, the decrease in total water content was at the expense of the ice content, which is consistent with the hypothesis of Anderson et al. (1978).

Following an elapsed time of about 100 hr, we find that the total water content equals the unfrozen water content (Fig. 5). Hence no frozen water should be present, the unfrozen water content having remained nearly constant over this entire time interval. During the elapsed time of 120 to 280 hr a steady reduction in the unfrozen water content is observed, which corresponds nearly exactly to the total sample water content, as determined by the amount of water removed from the soil by the molecular sieve material. This excellent agreement between the unfrozen water contents determined by NMR and the water removed by the molecular sieve material is apparent (Fig. 5).

CONCLUSIONS

This study demonstrates the balance between the ice phase and the unfrozen water phase in partially frozen soil as water is removed. The results show that as water is removed from a clay soil, the

Table 3. Unfrozen water content vs time determinations for a water-content-reduced sample of Morin clay.

Time (hr)	Unfrozen water content (% dry wt)	Time (hr)	Unfrozen water content (% dry wt)
0	12.53*	147	9.48
1.5	12.53	149	9.41
3	12.53	150.5	9.28
5.5	12.53	152	9.14
22.5	12.60	166.5	8.65
25	12.53	168	8.65
26.5	12.53	169	8.38
28.5	12.67	170	8.51
30.5	12.74	171	8.38
47	12.53	173.5	8.24
49	12.46	175	8.10
50.5	12.53	180	7.96
52	12.60	180.25	7.96
53.5	12.46	190.5	7.55
56	12.53	191.5	7.48
77	12.53	192	7.48
77.5	12.53	193	7.40
78	12.53	194	7.33
85.5	12.25	195	7.33
96	12.04	196.5	7.29
97	12.11	197.5	7.29
98	12.39	199	7.12
118.5	11.35	215	6.71
119	11.28	216	6.71
120	11.21	217.5	6.58
121	11.01	219	6.58
122	11.01	220	6.51
123	10.94	221.5	6.51
124.5	10.73	222	6.51
126.5	10.59	241.25	5.88
132	10.31	241.5	5.88
142.5	9.69	243	5.81
143	9.69	267	5.33
144	9.76	267.5	5.26
145	9.62	288	4.85
146	9.69		

* Reading taken before the addition of the molecular sieve material.

ice content is gradually reduced while the unfrozen water content remains nearly constant. Once the ice is depleted, the unfrozen water content determined by NMR corresponds to the total water content of the soil determined by the weight of water removed by the molecular sieve material.

This study also demonstrates the accuracy of the nuclear magnetic resonance technique in the determination of unfrozen water in frozen soil by the agreement of NMR results with physical desorption data. Now that the accuracy of this technique has been demonstrated, further tests will be conducted at different temperatures and water

contents and with different soil types to show the relationship between ice and unfrozen water in partially frozen soil.

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