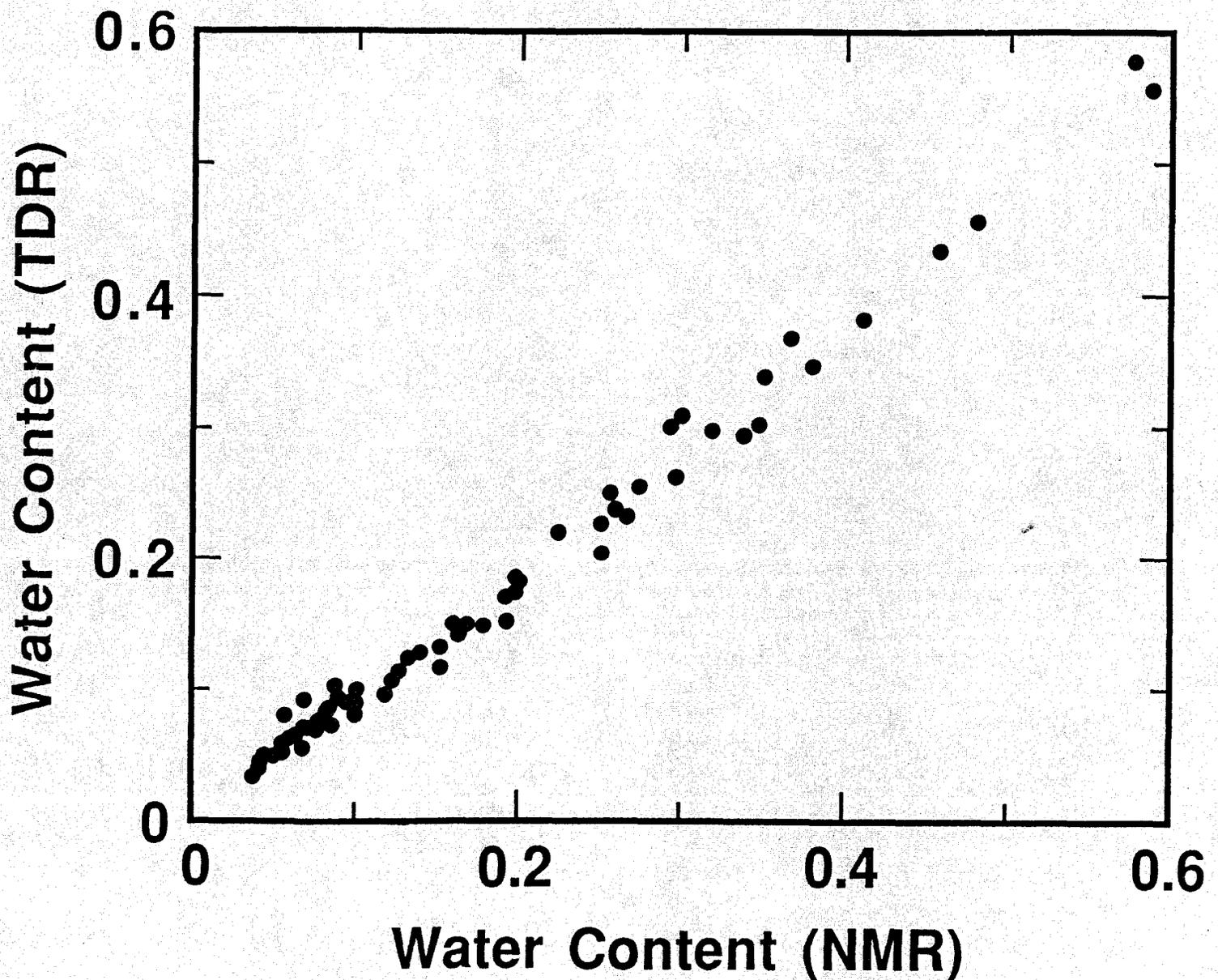


*Measurement of the  
unfrozen water content of soils  
Comparison of NMR and TDR methods*



# CRREL Report 88-18

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## *Measurement of the unfrozen water content of soils Comparison of NMR and TDR methods*

Michael W. Smith and Allen R. Tice

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19. ABSTRACT (Continue on reverse if necessary and identify by block number) The results of a laboratory testing program, carried out to compare two independent methods for determining the unfrozen water content of soils, are described. With the time domain reflectometry method, the unfrozen water content is inferred from a calibration curve of apparent dielectric constant vs volumetric water content, determined by experiment. Previously, precise calibration of the TDR technique was hindered by the lack of a reference comparison method, which nuclear magnetic resonance now offers. This has provided a much greater scope for calibration, including a wide range of soil types and temperature (unfrozen water content). The results of the testing program yielded a relationship between dielectric constant and volumetric unfrozen water content that is largely unaffected by soil type, although a subtle but apparent dependency on the texture of the soil was noted. It is suggested that this effect originates from the lower valued dielectric constant for adsorbed soil water. In spite of this, the general equation presented may be considered adequate for most practical purposes. The standard error of estimate is $0.015 \text{ cm}^3 \text{ cm}^{-3}$ , although this may be reduced by calibrating for individual soils. Brief guidelines on system and probe design are offered to help ensure that use of the TDR method will give results consistent with the relationship presented.					
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## PREFACE

This report was prepared by Dr. Michael W. Smith, of the Geotechnical Science Laboratories, Department of Geography, Carleton University, Ottawa, Canada, and Allen R. Tice, Physical Science Technician, of the Geochemical Sciences Branch, Research Division, U.S. Army Cold Regions Research and Engineering Laboratory. Funding for this project 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 Soils*.

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# Measurement of the Unfrozen Water Content of Soils

## Comparison of NMR and TDR Methods

MICHAEL W. SMITH AND ALLEN R. TICE

### INTRODUCTION

The two methods that are the subject of this paper—pulsed nuclear magnetic resonance (NMR) and time domain reflectometry (TDR)—offer complementary attributes in the measurement of soil water content. NMR provides a fast and accurate method for use with laboratory samples, while TDR can be used for both in-situ measurements in the field and laboratory experiments.

Time domain reflectometry (TDR) measures the travel time of a megahertz pulse through the soil, from which an apparent dielectric constant is determined. The unfrozen water content is inferred from a calibration curve of the apparent dielectric constant vs volumetric water content, determined by experiment. Previously, precise calibration of the technique for determining water content was hindered by the lack of a reference comparison method, which NMR now offers. The calibration procedure involves simultaneous measurements of unfrozen water content (NMR) and dielectric constant (TDR) for a variety of soil specimens. Before describing the experiment, we will briefly review the two methods.

#### Nuclear magnetic resonance (NMR)

All atomic nuclei have magnetic moments but the quantum energy levels are characteristic of the particular nuclear species. When radio frequencies are applied, atoms absorb a certain amount of energy to realign to another stable position within the magnetic field. If a soil-water mixture is placed in a pulsed NMR analyzer and a single radio frequency pulse is applied, a voltage (which corresponds to the number of atoms absorbing energy) is induced in a receiver coil that surrounds the specimen. This voltage is detected by the NMR analyzer; its magnitude (minus the background) is directly proportional to the amount of water (hydrogen) in the mixture. Therefore, the NMR can

be used as a soil water detector (subject to certain considerations, e.g. see Tice and Oliphant 1984).

A drop in signal intensity is observed as soil water freezes, since the NMR used (Praxis PR-103) was tuned to the hydrogen proton associated with liquid water (10.72 MHz). The signal associated with the protons of the solid ice and soil constituents is not recorded. Tice et al. (1982) describe the NMR technique for determining the unfrozen water content of soils, and demonstrate the accuracy of the technique by the agreement of NMR results with physical desorption data.

#### Time domain reflectometry (TDR)

TDR is a common method for obtaining frequency-dependent permittivity (dielectric constant) values in the VHF through microwave frequency range, utilizing Fourier transformations of picosecond-duration pulses incident and reflected from a test material contained in a suitable waveguide (e.g. Delaney and Arcone 1984). In a different application, Topp et al. (1980) proposed obtaining single (frequency-independent) permittivity values for soils using a non-Fourier approach, in which the travel time of an electromagnetic pulse, launched along a coaxial line of known length  $L$  containing a soil specimen, is measured. (Alternatively, a pair of parallel metallic rods may be embedded in the soil specimen.) The reflections originating at the front and back (beginning and end) of a sample (or in-situ sample volume) are recorded and the time delay  $t$  between these reflections is measured (Fig. 1).

Electromagnetic theory shows that the velocity of propagation  $v$  can be expressed in terms of an apparent dielectric constant  $K_a$  for the test medium and the velocity of electromagnetic radiation in free space,  $c$  ( $3 \times 10^8$  m s<sup>-1</sup>):

$$v = c/\sqrt{K_a}. \quad (1)$$

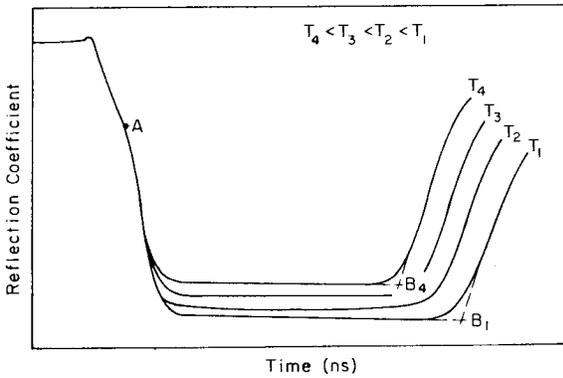


Figure 1. Typical TDR traces at a variety of freezing temperatures.

Since

$$v = L/t \quad (2)$$

then

$$K_a = (ct/L)^2. \quad (3)$$

However, since wet soils are generally dispersive at frequencies in the low gigahertz range (Hoekstra and Delaney 1974, Delaney and Arcone 1984), the value of  $K_a$  determined by the travel time method will approach the true value of  $K$  only if the TDR pulse contains no such high frequencies by the time it returns from the end of the transmission line (see also Arcone and Wills 1986). Two features of the TDR measurement system used in our experiments contribute to this condition:

1. The TDR model used (Tektronix 1502) has a relatively slow rise time (about 140 ps), and thus the incident pulse has virtually no frequency content above about 800 MHz.
2. When transmission lines of about 20 cm or larger are used, most of the high frequency content of the signal is dispersed along the line and the reflection at the end of the line is dominated by the lower frequencies in the launched signal.

## EXPERIMENTAL RATIONALE

Topp et al. (1980) determined an empirical relationship between  $K_a$  and the volumetric water content ( $\theta_u$ ) that is nearly independent of soil type, density, temperature and salinity. As the water content increases, the time delay  $t$  increases (i.e.  $K_a$  increases); thus a relationship exists as follows:

$$\theta_u = f(K_a).$$

Patterson and Smith (1981) argued that Topp's relationship could be applied as a first approximation to the determination of the volumetric unfrozen water content  $\theta_u$  of frozen soils, since the low frequency dielectric constant of ice (3.2) is close to the value for soil minerals (3 to 4). This hypothesis was confirmed by combined TDR and dilatometer measurements (Patterson and Smith 1981, Smith and Patterson 1984), although results were restricted to temperatures above  $-3^\circ$  or  $-4^\circ\text{C}$ , because dilatometer errors are cumulative with cooling. These authors expected, however, that the relationship for frozen soils would depart from the Topp curve at lower temperatures (lower unfrozen water contents and higher ice contents), as ice is "substituted" for air ( $K = 1$ ) in the soil. In addition, Topp et al. (1980) passed their calibration curve through a value of 80 for bulk water, although their maximum soil water contents were below about 50%. Our measurement system yielded a value for the apparent dielectric constant of water that was consistently lower than 80 (about 72). Thus a departure from the Topp curve is also to be expected at high soil water contents.

These considerations pointed to the need for a calibration dedicated to frozen soils, and the NMR technique provides a suitable reference method for this. The TDR/NMR approach offers considerable scope for comparison, accommodating a wide range of soil types and temperature (unfrozen water content).

Further details concerning the NMR and TDR methods can be found in Tice et al. (1978, 1982, 1984), Topp et al. (1980) and Patterson and Smith (1981).

## EXPERIMENTAL METHODS

Separate specimens, from the same prepared soil sample, were subjected to NMR and TDR measurements. To facilitate filling of the TDR coaxial specimen container, all the samples were saturated with distilled water to the point of dilatancy. The containers were from 12 to 20 cm long, and 1.35 cm in diameter. The specimen volume was from 16 to 25  $\text{cm}^3$ , and the specimen mass was typically 25 to 40 g. The NMR specimens were compacted in test tubes to the same density as in the TDR tests; the specimen was typically 12 to 15 g.

The two sets of specimens were placed in precision temperature baths containing an ethylene

**Table 1. Values of specific surface area, gravimetric and volumetric water contents and dry density for soils used in this study. Line separates original 17 soils from those used to confirm the  $K_a$  vs  $\theta_u$  relationship.**

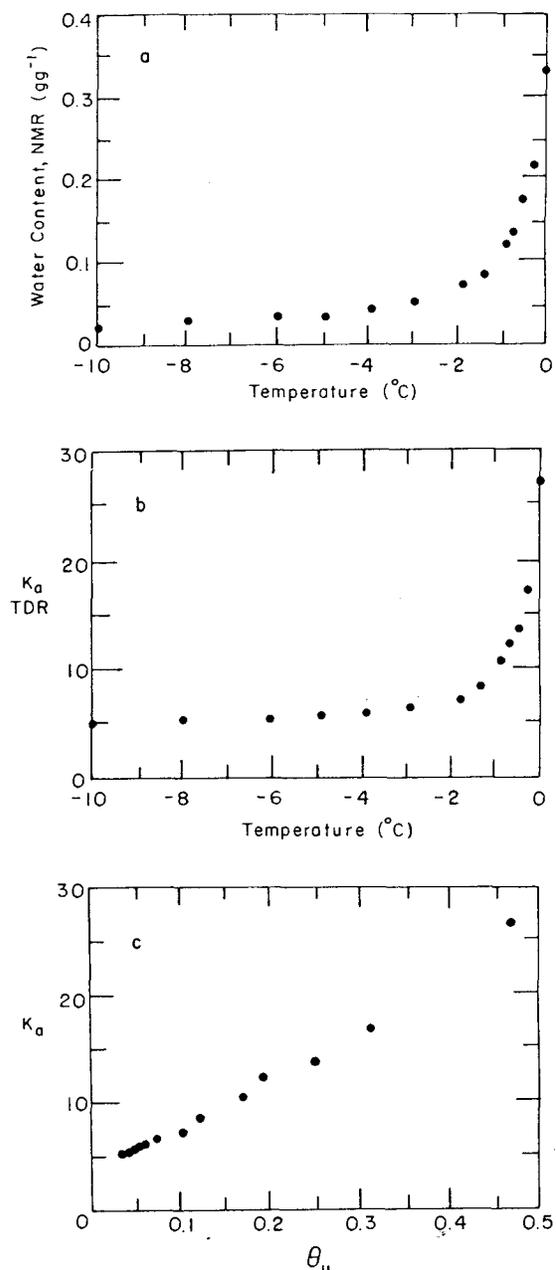
Soil	SSA	w	$\theta_v$	$\rho_d$
West Lebanon gravel	15	0.240	0.375	1.563
Castor sandy loam	—	0.261	0.385	1.475
Manchester silt	18	0.303	0.432	1.425
Kaolinite (KGa-1)	23	0.396	0.500	1.262
Chena Hot Springs silt	40	0.284	0.414	1.456
Leda clay	58	0.333	0.470	1.412
Mayo silty clay	—	0.378	0.495	1.311
Morin clay	60	0.362	0.472	1.305
O'Brien clay	61	0.381	0.515	1.352
Goodrich clay	68	0.362	0.467	1.289
Tuto clay	78	0.733	0.674	0.920
Sweden VFB 478 clay	113	0.491	0.548	1.116
Suffield silty clay	148	0.340	0.455	1.339
Frederick clay	159	0.426	0.492	1.209
Ellsworth clay	184	0.372	0.450	1.210
Regina clay	291	0.595	0.572	0.961
Umiat bentonite	714	2.25	0.821	0.365
Lanzhou silt (saline, China)	34	0.220	0.364	1.655
Niagara silt	37	0.222	3.365	1.645
Norway LE-1 clay	52	0.337	0.477	1.415
Kaolinite no. 7	72	0.570	0.587	1.029
Athena silt loam	83	0.352	0.456	1.296
Sweden CTH 201 clay	106	0.508	0.573	1.128
Hectorite	419	1.023	0.722	0.706
Volcanic ash	474	0.668	0.602	0.901

glycol-water mixture. They were cooled to between  $-10^\circ$  and  $-15^\circ\text{C}$  and progressively warmed to  $0^\circ\text{C}$ . NMR and TDR readings were taken at predetermined temperatures on the warming cycle. Readings were also taken for the completely thawed specimens. The TDR traces were recorded on a Hewlett-Packard 7045B X-Y recorder; this allowed the travel time to be determined to within  $\pm 1\%$ .

Anderson and Tice (1972) demonstrated that the unfrozen water content of frozen soils depends not only on the temperature but also the *specific surface area* (SSA,  $\text{m}^2 \text{g}^{-1}$ ) of the soil. Thus for the present experiments, a variety of soils (17 in number) were selected, which covered a representative range in specific surface area (Table 1). An additional eight soils (Table 1), which were not part of the calibration set, were also tested as a means of verifying the relationship determined for  $K_a$  vs  $\theta_u$ .

## RESULTS AND DISCUSSION

With the dielectric constant at each temperature determined by the pulse reflection method (TDR)



**Figure 2. Water content, NMR and TDR relationships for Leda clay.**

and the unfrozen water content determined by the NMR technique, a typical set of results is shown in Figure 2. Correlation of the dielectric constant with the amount of unfrozen water present was achieved by matching the two curves on the basis of temperature. Since the NMR values are on a gravimetric basis, the *volumetric* unfrozen water content in the TDR specimen at any temperature was obtained by multiplying the NMR value by the dry density  $\rho_d$  of the TDR specimen:

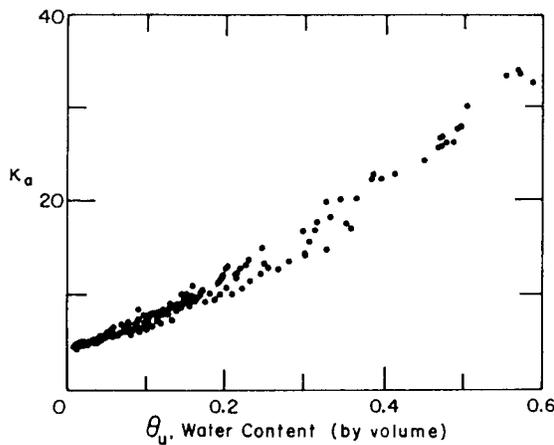


Figure 3. Water content vs  $K_a$  (data for all soils).

$$\theta_u = \rho_d w_u / \rho_w$$

where  $\rho_w$  is the density of water. The gravimetric water content and soil density were determined at the completion of each test.

The comparison procedure resulted in a plot of  $K_a$  vs  $\theta_u$  (Fig. 2c), providing a calibration of the TDR technique for unfrozen water content determination. In this case, a cubic polynomial fits the data with a standard error (SE) of 0.5% in  $\theta_u$ , which is indicative of the accuracy possible with the TDR technique when it is calibrated for a single soil. However, the real purpose of the experiment was to examine the dependence or otherwise of the TDR technique on soil type.

To this end, the comparison procedure was repeated for all 17 test soils, yielding over 220 points for  $K_a$  vs  $\theta_u$  (Fig. 3). A third-degree polynomial

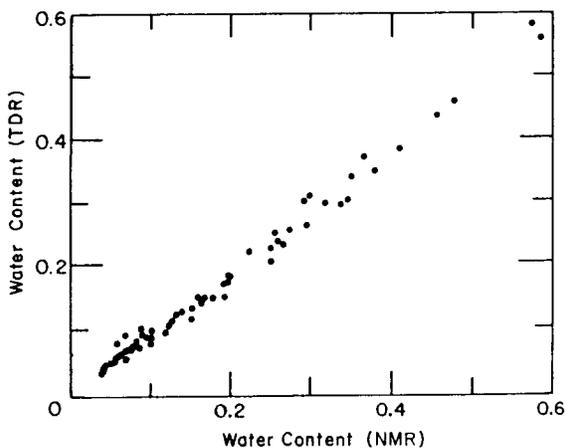


Figure 5. Results of verification tests of eq 4.

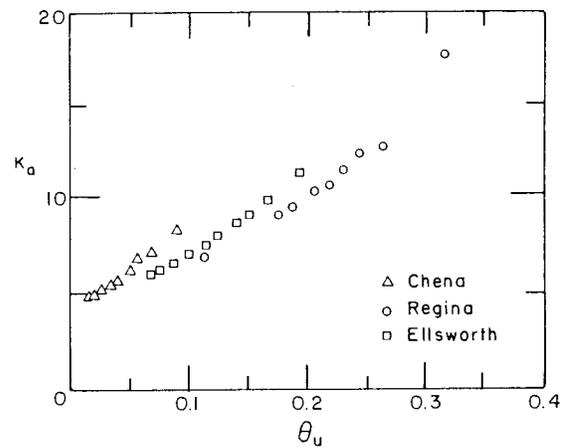


Figure 4. Water content vs  $K_a$  (data for three soils).

fitted to these data gives the following general relationship:

$$\theta_u = -1.458E-1 + 3.868E-2K_a - 8.502E-4K_a^2 + 9.920E-6K_a^3 \quad (4)$$

with a standard error of estimate of  $\pm 1.55\%$  in  $\theta_u$ . The main reason for the scatter in Figure 3 is a subtle but apparent dependency on the texture (specific surface area?) of the soil. This is demonstrated in Figure 4, where data for three soils are illustrated; it is evident that, for a given water content, the apparent dielectric constant decreases with the fineness of the soil. It is suggested that, since the dielectric constant of adsorbed water must be (much) lower than that of bulk water, the large proportion of adsorbed water in materials with high SSA lowers the overall dielectric constant (travel time) of the soil system at a given water content.

For most practical purposes, however, eq 4, which does not distinguish between soil types, may be used to determine  $\theta_u$  from the measurement of  $K_a$  to within  $\pm 0.03 \text{ m}^3 \text{ m}^{-3}$  (2 SEs), with a 95% level of confidence. As a verification of this relationship, a further eight soils, which *did not* form part of the calibration set, were tested. These results form an independent assessment of eq 4 for determining the unfrozen water content of soils. The comparison, which is shown in Figure 5, indicates that the general relationship is satisfactory, and that the TDR method can be used without regard to soil type. The slight bias noticeable in Figure 5 is probably due to the phenomenon illustrated in Figure 4.

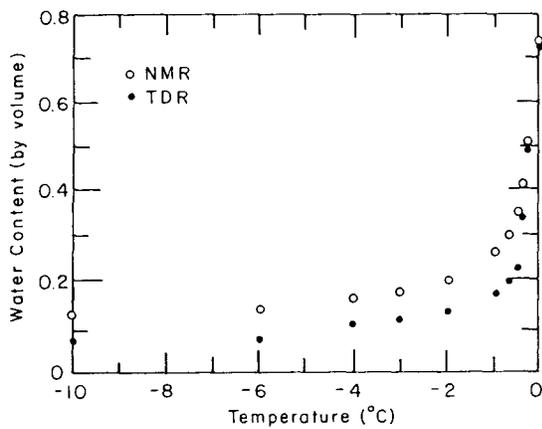


Figure 6. Comparison of NMR and TDR data for hectorite.

The most notable departure from the general relationship described in eq 4 was observed in the results for two colloidal soils—hectorite (SSA = 419) and volcanic ash (SSA = 474). In both cases, the unfrozen water contents determined with eq 4 were consistently lower than the NMR data, except very near 0°C (Fig. 6). These soils presumably represent an extreme case of the phenomenon discussed above. Further investigations of this are warranted since it now appears that a separate calibration would be necessary when using the TDR with such materials.

## ERRORS

In light of the comments in the introductory section of the paper about  $K_a$  and  $K$ , it appears that use of a measurement system distinctly different from that used in the calibration experiments—such as TDR equipment with faster rise times, or the use of very short transmission lines—could lead to a larger error than suggested by eq 4.

Finally, one other possible source of error is derived from observed variations in the time-base calibration of different 1502 TDR units. Using different machines, we have measured  $K_a$  values for distilled water ranging from 69 to 77. The unit that was used in determining eq 4 gave a value of 72 for water at 20°C, in a 20-cm coaxial line (500  $\Omega$  in air). If one were to obtain a value of 75, say, on another unit, then all measurements using that unit should be reduced by 4% in order to use eq 4 without additional error.

## CONCLUSIONS

It appears that the TDR technique, which is appropriate for use in undisturbed soil conditions (in the field or laboratory), does not require special calibration for most practical purposes. Overall, the results obtained from the combined NMR and TDR experiments, based on 25 soils covering a wide range of SSAs likely to be normally encountered in the field, yield a relationship between apparent dielectric constant and unfrozen water content that is only very slightly dependent on soil type. Use of this relationship implies a likely error of about  $\pm 0.03 \text{ cm}^3 \text{ cm}^{-3}$  in water content with the TDR method. The only notable exceptions observed so far are the results for two soils with very high SSAs, for which the predicted water contents were markedly lower than the corresponding NMR values.

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West Lebanon Gravel

Temp	w <sub>u</sub>
-0.1	.081
-0.2	.043
-0.4	.029
-0.6	.024
-0.9	.021
-1.4	.018
-2.2	.015
-2.6	.014
-3.6	.012
-4.7	.012
-6.7	.011
-9.9	.009

Castor Sandy Loam

Temp	w <sub>u</sub>
-0.1	.089
-0.2	.042
-0.4	.026
-0.6	.020
-0.9	.016
-1.4	.015
-2.2	.012
-2.6	.011
-3.6	.007
-4.7	.007
-6.7	.005
-9.9	.005

Manchester Silt

Temp	w <sub>u</sub>
-0.22	.082
-0.38	.037
-0.46	.032
-0.64	.029
-0.88	.027
-1.45	.023
-1.92	.020
-3.07	.015
-4.91	.012
-6.83	.010
-8.45	.009
-9.89	.007
-11.94	.006

Temp	K	$\theta$
0.0	22.43	.375
-0.3	6.11	.054
-0.5	5.62	.044
-0.7	5.44	.040
-0.9	5.36	.035
-1.4	5.15	.029
-1.9	5.10	.026
-3.9	5.02	.019
-6.2	4.98	.016
-10.1	4.94	.013

Temp	K	$\theta$
0.6	22.96	.385
-0.3	6.07	.053
-0.5	5.35	.039
-0.65	5.19	.034
-0.9	5.10	.028
-1.4	4.90	.022
-1.9	4.78	.018
-3.9	4.73	.012
-6.2	4.69	.009
-10.1	4.65	.007

Temp	K	$\theta$
0.6	28.10	.471
-0.3	6.39	.065
-0.5	5.80	.050
-0.7	5.53	.044
-0.9	5.44	.037
-1.35	5.02	.030
-1.95	4.90	.025
-2.95	4.86	.021
-3.9	4.78	.018
-6.0	4.69	.014
-8.0	4.59	.013
-10.1	4.48	.011
-12.0	4.41	.009

Kaolinite (KGa 1) (Sample #1)

Temp	w <sub>u</sub>
-0.1	.294
-0.2	.196
-0.4	.124
-0.6	.091
-0.9	.066
-1.4	.045
-2.2	.032
-3.6	.020
-4.7	.015
-5.8	.013
-6.7	.011
-7.7	.010
-9.9	.007

Temp	K	$\theta$
0.6	30.10	.503
-0.3	13.02	.203
-0.5	8.70	.135
-0.7	7.61	.104
-0.9	6.72	.083
-1.35	5.72	.054
-1.85	5.37	.041
-2.9	5.14	.029
-3.9	4.99	.022
-4.9	4.91	.019
-6.0	4.84	.016
-8.0	4.77	.012
-10.1	4.73	.010

Kaolinite (KGa 1) (Sample #2)

No data collected

Temp	K	$\theta$
0.6	33.53	.555
-0.1	17.36	.359
-0.2	14.87	.248
-0.3	12.67	.202
-0.4	10.83	.158
-0.5	10.01	.146
-0.7	7.98	.110
-0.85	7.28	.088
-2.0	5.90	.044
-5.0	5.43	.019
-10.0	5.06	.010

Chena Hot Springs Silt

Temp	w <sub>u</sub>
-0.1	.173
-0.2	.077
-0.4	.050
-0.6	.041
-0.9	.031
-1.4	.027
-2.2	.019
-3.6	.015
-4.7	.014
-5.8	.013
-6.7	.012
-8.9	.011
-9.9	.009

Temp	K	$\theta$
0.6	22.96	.414
-0.3	8.36	.090
-0.5	7.16	.069
-0.7	6.89	.057
-0.9	6.17	.050
-1.35	5.64	.040
-1.85	5.44	.034
-2.9	5.18	.027
-3.9	5.06	.023
-4.9	4.95	.020
-6.0	4.91	.018
-8.0	4.88	.016
-10.1	4.77	.014

Leda Clay

Temp	w	Temp	K	$\theta$
-0.1	.259	0.5	26.89	.471
-0.2	.240	-0.3	16.81	.313
-0.3	.218	-0.5	13.38	.251
-0.4	.187	-0.7	12.13	.195
-0.6	.163	-0.9	10.56	.171
-0.7	.136	-1.35	8.36	.124
-1.0	.111	-1.85	7.16	.102
-1.7	.074	-2.9	6.50	.073
-2.5	.059	-3.9	6.13	.062
-3.7	.044	-4.9	5.80	.052
-5.8	.034	-6.0	5.60	.047
-7.7	.029	-8.0	5.44	.042
-9.7	.024	-10.0	5.29	.034

O'Brien Clay

Temp	w	Temp	K	$\theta$
-0.1	.247	0.5	29.88	.515
-0.2	.201	-0.2	17.13	.272
-0.4	.161	-0.3	14.54	.245
-0.6	.139	-0.4	13.69	.218
-0.8	.121	-0.5	12.57	.203
-1.1	.107	-0.85	10.92	.164
-1.7	.085	-1.05	9.83	.150
-2.4	.071	-1.5	8.71	.123
-3.3	.059	-2.1	7.98	.105
-4.5	.048	-5.0	6.75	.062
-6.2	.040	-10.0	6.18	.042
-8.0	.036			
-10.0	.031			

Mayo Silty Clay (Sample #1)

Temp	w	Temp	K	$\theta$
-0.2	.228	0.5	27.13	.516
-0.35	.164	-0.35	12.37	.215
-0.46	.148	-0.6	10.45	.171
-0.64	.126	-0.9	8.95	.141
-0.82	.109	-1.4	7.79	.114
-1.42	.088	-1.95	7.02	.097
-1.95	.079	-2.95	6.42	.080
-3.02	.064	-3.9	6.08	.070
-3.67	.056	-4.9	5.76	.063
-4.99	.050	-5.95	5.60	.057
-6.86	.041	-8.0	5.29	.050
-8.42	.038	-10.1	5.21	.044
-9.94	.035	-12.0	5.10	.041
-12.02	.031	-15.0	4.88	.037
-15.11	.027			

Mayo Silty Clay (Sample #2)

Temp	K	$\theta$
0.6	26.41	.486
-0.1	17.51	.354
-0.2	14.41	.299
-0.4	11.74	.211
-0.5	11.11	.193
-0.7	9.62	.157
-0.85	8.89	.139
-2.0	7.11	.098
-5.0	6.02	.064
-10.0	5.53	.045

Morin Clay

Temp	w	Temp	K	$\theta$
-0.17	.179	0.5	26.41	.478
-0.35	.136	-0.35	11.56	.197
-0.41	.126	-0.6	10.08	.162
-0.61	.115	-0.9	9.05	.133
-0.80	.098	-1.4	8.17	.107
-1.42	.082	-1.95	7.52	.091
-1.95	.074	-2.95	7.02	.075
-2.99	.058	-3.9	6.67	.065
-3.67	.052	-4.9	6.33	.058
-4.93	.045	-5.95	6.13	.053
-5.72	.038	-8.0	5.68	.046
-6.81	.036	-10.1	5.44	.041
-8.40	.033	-12.0	5.33	.037
-9.81	.031	-15.0	4.99	.034
-12.00	.025			
-15.09	.020			

Goodrich Clay

Temp	w	Temp	K	$\theta$
-0.1	.209	0.6	25.84	.467
-0.2	.168	-0.3	11.79	.193
-0.4	.132	-0.5	9.87	.153
-0.6	.113	-0.7	9.10	.131
-0.9	.096	-0.9	8.22	.117
-1.4	.081	-1.35	7.20	.097
-2.2	.065	-1.85	6.76	.084
-3.6	.047	-2.9	6.25	.068
-4.7	.041	-3.9	5.88	.060
-5.8	.038	-4.9	5.64	.054
-6.7	.034	-6.0	5.44	.049
-8.9	.029	-8.0	5.25	.043
-9.9	.028	-10.1	5.14	.039

Tuto Clay

Temp	w
-0.22	.402
-0.33	.243
-0.41	.213
-0.56	.166
-0.80	.137
-1.37	.099
-1.87	.086
-2.97	.070
-3.65	.060
-4.91	.052
-6.78	.044
-8.37	.037
-9.89	.033

Sweden VFB 478 Clay

Temp	w <sub>u</sub>
-0.3	.491
-0.4	.402
-0.6	.335
-0.7	.294
-1.0	.270
-1.3	.246
-1.7	.227
-2.2	.203
-2.5	.189
-2.9	.169
-3.7	.152
-4.7	.129
-5.8	.117
-6.8	.105
-7.7	.100
-8.7	.093
-9.7	.089

Suffield Silty Clay

Temp	w
-0.1	.222
-0.2	.167
-0.4	.135
-0.6	.116
-0.9	.110
-1.4	.095
-1.6	.091
-2.2	.085
-3.6	.071
-4.7	.067
-5.8	.064
-7.7	.058
-9.9	.055

Temp	K	$\theta$
0.6	35.19	.674
-0.25	14.76	.327
-0.4	11.88	.196
-0.6	8.97	.148
-0.8	7.98	.126
-1.0	7.66	.113
-1.5	6.54	.084
-2.0	6.04	.075
-3.0	5.83	.063
-5.0	5.37	.048
-10.0	5.04	.030

Temp	K <sub>a</sub>	$\theta_u$
0.5	33.86	.576
-0.3	33.21	.548
-0.4	28.50	.449
-0.5	24.72	.412
-0.8	17.95	.319
-0.95	15.53	.306
-1.45	13.28	.264
-2.0	11.78	.238
-3.0	9.83	.189
-5.0	8.14	.141
-7.0	7.59	.117
-10.0	7.05	.098

Temp	K	$\theta$
0.5	27.90	.494
0.6	26.89	.469*
-0.1	18.32	.322
-0.25	12.87	.217
-0.35	11.40	.191
-0.5	10.46	.169
-0.6	9.66	.155
-0.8	9.14	.149
-1.0	8.71	.142
-1.5	8.30	.125
-2.0	7.98	.116
-10.0	6.75	.100

\* Second specimen

Frederick Clay (Results from two runs combined)

Temp	w	Temp	K	$\theta$
-0.1	.286	0.5	27.61	.492
-0.2	.231	-0.1	20.17	.346
-0.3	.186	-0.2	13.58	.279
-0.4	.169	-0.3	13.18	.225
-0.6	.154	-0.4	12.25	.214
-0.7	.149	-0.5	11.24	.196
-1.0	.140	-0.7	10.99	.189
-1.3	.130	-0.9	10.20	.181
-1.7	.120	-1.4	9.68	.161
-2.2	.111	-1.9	9.34	.148
-2.9	.101	-2.9	8.67	.128
-3.7	.093	-4.0	8.35	.115
-4.7	.085	-5.1	8.03	.105
-5.8	.078	-6.0	7.72	.098
-6.8	.074	-8.0	7.46	.090
-7.7	.072	-10.1	7.26	.080
-8.7	.069			
-9.7	.064			

Ellsworth Clay

Temp	w	Temp	K	$\theta$
-0.1	.224	0.6	24.38	.450
-0.2	.173	-0.3	11.28	.194
-0.4	.145	-0.5	9.87	.167
-0.6	.133	-0.7	9.15	.151
-0.9	.117	-0.9	8.70	.141
-1.4	.106	-1.35	7.93	.125
-2.2	.092	-1.85	7.47	.114
-2.6	.087	-2.9	7.07	.100
-3.6	.077	-3.9	6.67	.092
-4.7	.071	-4.9	6.50	.086
-5.8	.068	-6.0	6.29	.081
-6.7	.065	-8.0	6.17	.074
-7.7	.061	-10.1	6.00	.069
-8.9	.058			
-9.9	.057			

Regina Clay

Temp	w <sub>u</sub>	Temp	K	$\theta$
-0.1	.385	0.5	37 <sup>a</sup> .40	.603
-0.2	.301	-0.15	17.72	.316
-0.4	.257	-0.3	12.67	.265
-0.6	.242	-0.4	12.37	.244
-0.9	.226	-0.6	11.40	.230
-1.4	.202	-0.8	10.65	.220
-2.2	.179	-1.0	10.28	.207
-2.6	.167	-1.5	9.48	.188
-3.6	.151	-2.0	9.22	.175
-4.7	.143	-10.0	7.35	.113
-5.8	.137			
-6.7	.129			
-7.7	.125			
-8.9	.120			
-9.9	.119			

Niagara Silt

Temp	w <sub>u</sub>	Temp	K	$\theta$
-0.3	.108	0.5	20 <sup>a</sup> .33	.365
-0.54	.084	-0.2	10.74	.197
-0.88	.076	-0.3	9.45	.175
-1.32	.063	-0.4	9.11	.162
-2.44	.048	-0.5	8.56	.138
-3.52	.042	-0.85	7.87	.120
-5.78	.031	-1.05	7.31	.116
-7.84	.027	-1.5	6.82	.098
-10.02	.025	-2.1	6.53	.084
		-5.0	5.66	.052
		-10.0	5.44	.041

Lanzhou Silt (China)

Temp	w <sub>u</sub>	Temp	K	$\theta$
-1.55	.178	-1.5	16 <sup>a</sup> .66	.298
-2.91	.127	-2.05	12.97	.255
-3.38	.102	-3.1	10.19	.194
-3.94	.087	-4.1	8.38	.137
-4.28	.079	-6.05	7.51	.089
-4.8	.069	-8.0	7.05	.068
-5.72	.056	-10.0	6.61	.056
-7.47	.044			
-9.49	.035			
-11.59	.029			

Umiat Bentonite (Sample #1)

Temp	w <sub>u</sub>	Temp	K	$\theta$
-0.1	1.903	-0.25	27 <sup>a</sup> .90	.493
-0.2	1.714	-0.35	22.39	.384
-0.3	1.273	-0.5	19.79	.326
-0.4	1.053	-0.6	15.64	.305
-0.6	.923	-0.7	13.69	.230
-0.7	.698	-0.95	10.65	.200
-1.0	.588	-1.6	8.80	.153
-1.3	.494	-10.0	6.20	.073
-1.7	.451			
-2.2	.406			
-2.9	.368			

Norway LE-1 Clay

Temp	w <sub>u</sub>	Temp	K	$\theta$
-0.1	.259	0.6	25 <sup>a</sup> .93	.477
-0.2	.205	-0.2	16.22	.290
-0.4	.157	-0.3	13.65	.256
-0.6	.134	-0.4	12.25	.222
-0.8	.118	-0.6	10.03	.190
-1.1	.100	-0.8	9.22	.167
-1.7	.082	-1.0	8.56	.150
-2.4	.064	-1.5	7.56	.122
-3.3	.052	-2.0	7.06	.100
-4.5	.044	-3.05	6.53	.079
-6.2	.035	-5.2	5.98	.058
-8.0	.031	-8.2	5.62	.044
-10.0	.028	-10.1	5.44	.040

Umiat Bentonite (Sample #2)

No data collected

Temp	K	$\theta$
-0.7	14 <sup>a</sup> .84	.258
-1.0	10.50	.218
-1.5	9.00	.175
-2.0	8.13	.157
-3.1	7.31	.132
-4.0	7.01	.119
-5.1	6.72	.109
-6.0	6.39	.101
-8.0	6.16	.090
-10.1	5.93	.081

Athens silty loam

Temp  $w_u$

No data collected

Temp K  $\theta_u$

0.6 24.54 .456  
 -0.4 7.41 .102  
 -0.6 7.06 .088  
 -0.95 6.72 .080  
 -2.0 6.20 .065  
 -3.05 6.07 .057  
 -4.0 5.80 .052  
 -8.0 5.57 .041  
 -10.0 5.53 .038

Kaolinite #7

Temp  $w_u$

-0.1 .359  
 -0.2 .344  
 -0.4 .327  
 -0.6 .287  
 -0.8 .258  
 -1.1 .225  
 -1.7 .175  
 -2.4 .124  
 -3.3 .093  
 -4.5 .072  
 -6.2 .052  
 -8.0 .044  
 -10.0 .036

Temp K  $\theta_u$

0.6 32.69 .587  
 -0.3 16.22 .346  
 -0.4 15.71 .336  
 -0.6 14.13 .295  
 -0.8 12.71 .265  
 -1.0 11.48 .249  
 -1.5 9.34 .192  
 -2.0 8.03 .150  
 -5.0 5.89 .068  
 -10.0 5.10 .031

Sweden CTH-201 Clay

Temp  $w_u$

-0.1 .424  
 -0.2 .362  
 -0.4 .309  
 -0.6 .281  
 -0.8 .243  
 -1.1 .210  
 -1.7 .165  
 -2.4 .128  
 -3.3 .107  
 -4.5 .088  
 -6.2 .074  
 -8.0 .067  
 -10.0 .060

Temp K  $\theta_u$

0.5 33.95 .573  
 -0.2 21.20 .408  
 -0.3 18.80 .379  
 -0.4 18.19 .349  
 -0.6 15.86 .317  
 -0.8 13.90 .274  
 -1.0 12.47 .249  
 -1.5 10.55 .200  
 -2.0 9.31 .161  
 -3.05 8.14 .129  
 -5.2 7.05 .094  
 -8.2 6.39 .073  
 -10.1 6.32 .068

Hectorite

Temp  $w_u$

-0.25 .715  
 -0.35 .579  
 -0.45 .491  
 -0.65 .420  
 -0.95 .367  
 -1.95 .280  
 -3.0 .244  
 -4.0 .230  
 -6.0 .198  
 -10.0 .177

Temp K  $\theta_u$

0.5 41.30  
 -0.25 28.05  
 -0.35 18.07  
 -0.45 12.47  
 -0.65 11.11  
 -0.95 10.19  
 -1.95 8.47  
 -3.0 7.98  
 -4.0 7.51  
 -6.0 6.46  
 -10.0 6.32

Volcanic Ash

Temp  $w_u$

-0.25 .358  
 -0.35 .347  
 -0.45 .338  
 -0.65 .325  
 -0.95 .317  
 -1.95 .290  
 -3.0 .282  
 -4.0 .273  
 -6.0 .258  
 -10.0 .255

Temp K  $\theta_u$

0.5 34.03  
 -0.25 17.91  
 -0.35 13.99  
 -0.45 12.25  
 -0.65 11.74  
 -0.95 11.42  
 -1.95 10.50  
 -3.0 10.09  
 -4.0 9.79  
 -6.0 9.34  
 -10.0 9.28