

NUCLEAR MAGNETIC RESONANCE SENSORS FOR MOISTURE MEASUREMENT IN ROADWAYS

G. A. Matzkanin and C. G. Gardner, Southwest Research Institute

The feasibility of practical instrumentation based on the nuclear magnetic resonance method was investigated to address the need for accurate, reliable moisture measurements in highway systems. A prototype of an implantable sensor that uses the pulsed nuclear magnetic resonance spin-echo technique was developed and subjected to laboratory verification. The sensor is 3 by 2 by 2 $\frac{1}{4}$ in. (7.6 by 5.1 by 5.7 cm) and weighs about 3 lb (1.35 kg). It directly measures the mass of moisture present; to obtain the percentage of moisture requires that soil density be determined by an auxiliary method. Experiments with bentonite, silica flour, and topsoil indicated that the sensor operated reliably, with an accuracy of approximately ± 1 percent up to 25 percent moisture, ± 2 percent up to 100 percent moisture, and ± 4.5 percent up to 200 percent moisture. A dependence of sensor response on soil type necessitated calibration for each soil type. Organic matter and dissolved salt did not affect the sensor readings for bentonite, but organic matter had an effect for silica flour. All of the results obtained were qualitatively accounted for in terms of a simple moisture adsorption and bonding model.

•SERVICE behavior of a highway system strongly depends on the moisture content of the system's components. Despite recognition of this relationship, progress in highway construction and maintenance practices related to control of moisture and its effects has been impeded by lack of adequate instrumentation and methodology for measuring moisture under conditions and constraints of highway practice. The economic significance of the problem is proved by the large annual investment aimed at removal of excess water that causes loss of supporting capacity of subgrade soils and aggregate bases, instability of embankments, and deterioration of pavements.

The techniques currently used in the highway field for measuring moisture content in situ are generally inadequate to meet researchers' needs. Instrumentation is specifically needed for remote readout of local sensing, for remote sensing of subsurface conditions, for high-precision measurements, and for long-term continuous monitoring. Devices that can be installed permanently and portable devices for sampling at random locations are needed to provide data to allow for development of new designs and use of new materials.

A moisture sensor based on the detection of hydrogen nuclei by the nuclear magnetic resonance (NMR) spin-echo method has been developed. A prototype of a sensor was fabricated and subjected to a laboratory verification program to determine its suitability for highway applications. Currently, a field evaluation program is under way, during which a number of replicate sensors will be tested for 1 year in actual highway field installations. This report describes the basic principles of operation of the NMR sensor and the results of the laboratory verification program. More complete details have been documented elsewhere (1).

PRINCIPLES OF NUCLEAR MAGNETIC RESONANCE

Many atomic nuclei possess nonzero spin angular momentum and an associated magnetic dipole moment. As a top spins in a gravitational field, the spinning nucleus precesses about the direction of an applied steady magnetic field (Fig. 1). The precession frequency is proportional to the strength of the magnetic field, and the proportionality constant (the gyromagnetic ratio) has a value characteristic of the specific nuclear species. For the hydrogen nucleus, the gyromagnetic ratio has the value $2.67 \times 10^4 \text{ S}^{-1} \text{ G}^{-1}$ ($2.67 \times 10^8 \text{ S}^{-1} \text{ T}^{-1}$). A resonance condition can be achieved by applying a second magnetic field, which alternates at a frequency corresponding to the precessional frequency of the nuclei of interest, in a direction orthogonal to that of the steady field. For appropriate values of steady and alternating fields, the spin axis of the nucleus can be reoriented; this is the phenomenon of NMR. The NMR detection of moisture involves the hydrogen nuclei (protons) in the water molecules. For fields of around 700 Oe ($56 \times 10^3 \text{ A/m}$), the NMR frequency for hydrogen nuclei is in the radio-frequency (RF) region, i.e., 3 MHz.

Although several methods of producing and detecting NMR signals are possible (2), the approach used in the moisture sensor described in this report is the pulsed NMR spin-echo technique. If the RF field is applied in a short burst, or pulse, of the proper duration, the nuclear magnetization vector can be made to rotate 90 deg into the plane orthogonal to the direction of the constant field H_0 , which is measured in Oe or A/m (Fig. 2). At the termination of the pulse, the magnetization vector precesses in the plane perpendicular to H_0 and induces a voltage in the RF coil that then serves as a sensor. This voltage is proportional to the number of nuclei within the effective volume of the RF coil. However, two effects must be taken into consideration: (a) Interactions between the nuclei in the specimen tend to diminish the amplitude of the precessing transverse magnetization, and (b) inhomogeneities in the applied static field gradually destroy the coherence among the individual nuclear spins that compose the net magnetization and cause a fanning out of the precessing nuclear magnetic moments. As a consequence of these two effects, the voltage induced by the precessing magnetization gradually decays to zero; this is called free induction decay. If, now, a second RF pulse is applied to the specimen at a suitable time following the first pulse, i.e., after the initial voltage has decayed because of the fanning out of the nuclear magnetic moments but before the amplitude of the transverse magnetization has diminished to zero because of internal interactions, then the nuclear magnetic moments can be refocused into a spin echo, which manifests itself as an induced voltage following the second applied pulse. This is shown in Figure 3.

The amplitude of the spin-echo signal is related to the magnitude of the nuclear magnetization (M_0), which in turn is proportional to the number of nuclei within the detection coil. Figure 4 shows an oscilloscope photograph of spin echoes from bentonite specimens containing different amounts of water. The echo amplitude also depends, of course, on a number of other parameters such as NMR frequency (which in turn depends on the magnetic field), temperature, and pulse spacing. These dependencies are so well-known that, once the calibration is established between a particular instrumentation and the amount of moisture present, corrections can be made for changes in these parameters.

The essential elements of an arrangement used to implement the NMR spin-echo method are shown in Figure 5. The specimen is encircled by an RF induction coil. A static magnetic field of strength H_0 is applied by a magnet. An RF oscillator supplies pulses of RF energy of the proper duration and separation to the coil to produce spin echoes. The induced voltage is increased by the amplifier, demodulated by the detector, and displayed as a function of time on a cathode ray oscilloscope. A digital readout of the amplitude of the spin echo is carried out by a sampling gate and a digital voltmeter.

Figure 1. Precession of nuclear magnetic moment in static magnetic field.

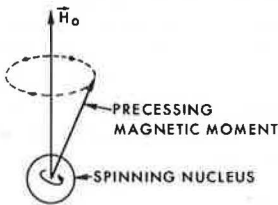


Figure 3. Free induction decay and spin echo associated with two-pulse sequence.

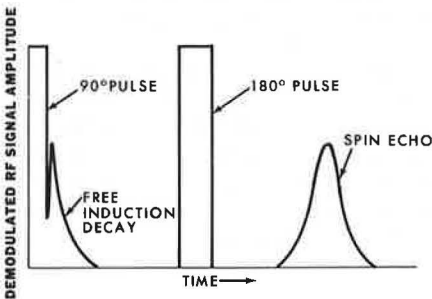


Figure 5. Essential elements of arrangement for pulsed nuclear magnetic resonance measurements.

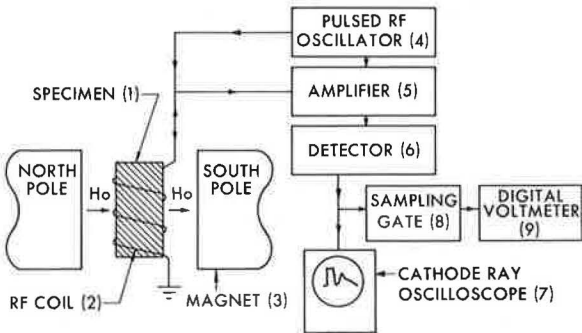


Figure 2. Rotation of nuclear magnetization by radio-frequency pulse followed by free induction decay.

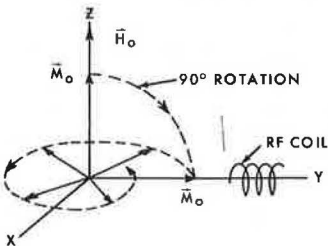


Figure 4. Pulsed nuclear magnetic resonance spin echoes from moisture in bentonite.

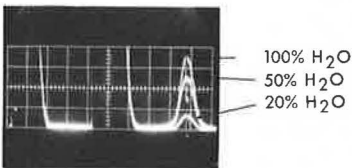
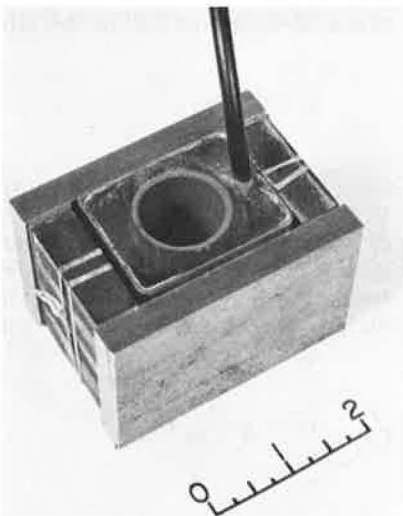


Figure 6. Prototype of implantable nuclear magnetic resonance moisture sensor.



IMPLANTABLE NUCLEAR MAGNETIC RESONANCE MOISTURE SENSOR

Most laboratory work on NMR is done by using large electromagnets to produce strong, homogeneous fields. This, of course, is unfeasible for the desired highway applications; therefore, initial design efforts centered around developing a miniaturized sensor that is made with a permanent magnet and an RF transmission and detection coil, small enough to be buried. This necessitates a smaller, weaker magnet than is ordinarily used, and a consequent loss in signal amplitude results (the NMR signal is proportional to the square of the magnetic field). However, initial tests showed that, when magnetic fields were as low as 700 Oe (56×10^3 A/m), spin-echo signals could still be measured down to a moisture density of 0.1 g/cm^3 for specimen volumes of 10 cm^3 . This corresponds to a dry weight moisture percentage of 5.5 for a soil with a dry density of 112 lb/ft^3 (1.82 g/cm^3).

One version of a buriable prototype NMR moisture sensor is shown in Figure 6. For achieving a sufficiently strong magnetic field with as little magnet material as possible and with acceptable homogeneity over the specimen volume, the Watson magnet design was used (3). In the design in Figure 6, the magnets are cast Alnico VIII, and the plates are $\frac{3}{8}$ -in.-thick (0.95-cm) mild steel. The overall dimensions of the structure are 3 by 2 by $2\frac{1}{4}$ in. (7.6 by 5.1 by 5.7 cm), and the magnet gap (separation of the plates), in which a field of approximately 600 Oe (48×10^3 A/m) is developed, is $1\frac{1}{2}$ in. (3.8 cm). The sensor, as photographed, weighs $3\frac{1}{4}$ lb (1.5 kg).

The RF transmission and detection coil is approximately 1 in. (2.54 cm) long and is wound on a plastic tube with an inside diameter of 0.875 in. (2.2 cm). This provides an active specimen volume of 0.6 in.^3 (9.8 cm^3). The coil and tube are permanently cast with an epoxy-like material into a thin-walled rectangular copper case that serves as a RF shield. A coaxial cable serves as the connection between the coil and the detection and readout instrumentation. In laboratory tests, cable lengths from 2 to 66 ft (0.6 to 20 m) have been used without serious degradation of signals.

LABORATORY VERIFICATION

Calibration

The measured NMR (spin-echo) signal amplitude, ξ (in volts), is proportional to the mass of moisture within the effective volume of the detection coil, that is

$$\xi = C m_v V \quad (1)$$

where m_v (in g/cm^3) is the moisture density, V (in cm^3) is the volume of the coil, and C (in V/g) is a proportionality factor between the measured NMR signal and the mass of water. The value of C depends on the size and geometry of the detection coil, the filling factor, the gain of the detection electronics, the strength of the applied magnetic field, the magnitude of the nuclear magnetic moments, and the temperature. Moisture density is related to the conventional dry weight moisture fraction (m) by the expression

$$m_v = \frac{m}{1 + m} D \quad (2)$$

where D is the density (in g/cm^3) of soil plus moisture. Thus the NMR signal amplitude is related to the dry weight moisture fraction by the equation

$$\frac{\xi}{D} = K \frac{m}{1 + m} \quad (3)$$

where K is a proportionality factor. Thus, for a series of specimens of various moisture content, volume, and density, ξ/D is expected to be linearly related to the quantity $m/(1 + m)$.

So that the capabilities of the prototype of a NMR sensor could be established, a set of specimens was prepared and placed in glass tubes that could be inserted into the sensor cavity. A portion of each specimen was weighed and oven-dried to determine gravimetrically the moisture percentage. The density of each specimen was determined by directly weighing and measuring the volume of each specimen. Three soil types were investigated: (a) bentonite clay, (b) silica flour, and (c) a local, organic, silty clay topsoil. Specimens were prepared at various dry weight percentages up to 213 percent for bentonite, 24 percent for silica, and 55 percent for the topsoil.

The results for bentonite are shown in Figure 7; $\mu = m/(1 + m)$ and is the wet weight moisture fraction. The data are linear within experimental error, except for the observation of a nonzero intercept. A least squares fit results in the equation

$$\xi/D = [2.288\mu - 0.291] V \cdot \text{cm}^3/\text{g} \quad (4)$$

Figure 8 shows the sensor readings for bentonite replotted against the dry weight moisture percentage, $m \times 100$ percent. The solid curve is a graph of Eq. 4 and essentially constitutes the sensor calibration curve for bentonite.

The failure of the observed data for bentonite to extrapolate through zero (in accordance with Eq. 3) may be interpreted in terms of a model based on the adsorption and binding of water molecules in the soil system. In the preceding analysis, all hydrogen nuclei in the water molecules in the sample were assumed to be sensed. However, the NMR spin-echo method is sensitive to the physical state of the detected nuclei. Atomic and molecular interactions affect the decay time of the transverse nuclear magnetization shown in Figure 2. Generally speaking, tight molecular binding, as in solids, causes a fast decay time, whereas looser molecular coupling, as in liquids, produces longer decay times. Thus, a spin-echo signal associated with the water molecules in the liquid state may be produced by appropriately choosing the time interval between the two RF pulses. If some of the water molecules present are adsorbed on the surface of soil particles, or are chemically bonded to solid constituents (e.g., water of hydration), thus effectively removing them from the liquid state, the hydrogen in the molecules so affected will generally not be detected by the NMR spin-echo technique when the instrumentation is adjusted for optimum detection of hydrogen in liquid water. If this situation exists, ξ in Eq. 1 should be modified as follows:

$$\xi = C(m_v V - \Delta_{w,c}) \quad (5)$$

where $\Delta_{w,c}$ is the mass of water (in g), within the effective volume of the detection coil, which does not contribute to the observed NMR signal. Letting m' be the dry weight moisture fraction for this bound moisture, the following modification of Eq. 3 is obtained:

$$\frac{\xi}{D} = K(1 + m') \frac{m}{1 + m} - Km' \quad (6)$$

If the adsorption (or binding) of water molecules by the solid constituents of the sample

Figure 7. Nuclear magnetic resonance moisture sensor signal versus wet weight moisture fraction for bentonite.

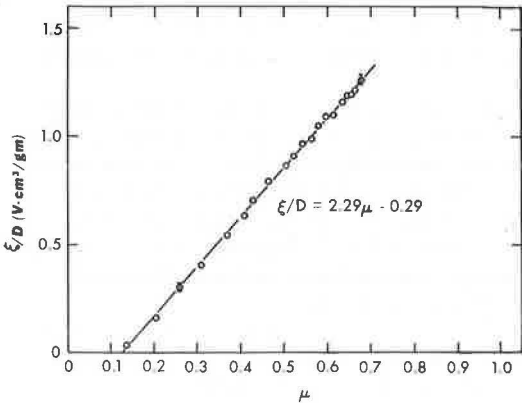


Figure 8. Nuclear magnetic resonance moisture sensor signal versus percentage of moisture for bentonite.

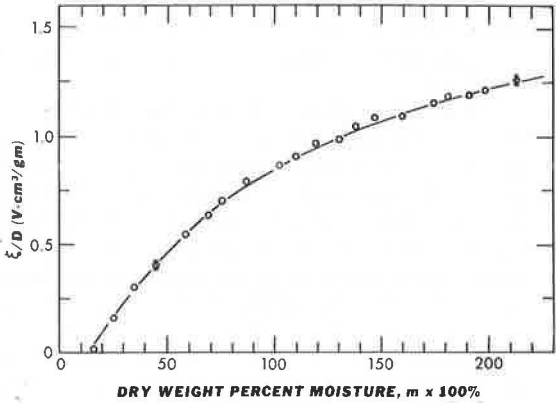


Figure 9. Theoretical dependence of pulsed nuclear magnetic resonance response on moisture content (based on adsorption model).

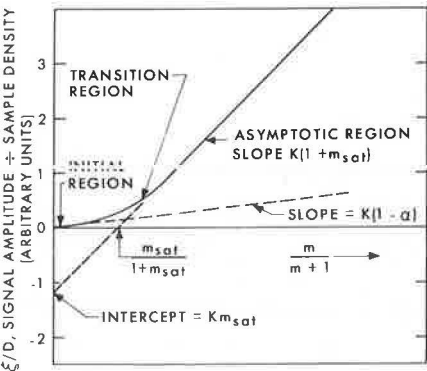


Figure 10. Nuclear magnetic resonance sensor signal versus percentage of moisture for bentonite, silica flour, and organic silty clay.

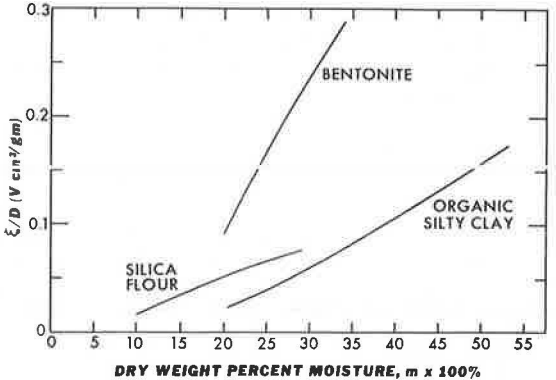


Table 1. Comparison of nuclear magnetic resonance sensor and gravimetric moisture determinations.

Specimen Number	Density (g/cm^3)	Moisture (percent)		Deviation	
		NMR Sensor	Gravimetric	Amount	Percent
1	1.184	19.8	20.4	-0.6	2.9
2	1.224	22.8	23.1	-0.3	1.3
3	1.200	22.2	23.4	-1.2	5.1
4	1.334	29.6	31.4	-1.8	5.7
5	1.405	29.8	32.3	-2.5	7.7
6	1.476	33.1	35.2	-2.1	6.0
7	1.579	42.5	45.1	-2.6	5.8
8	1.429	54.2	52.5	+1.7	3.2
9	1.231	81.3	82.6	-1.3	1.6
10	1.247	106.9	105.8	+1.1	1.0
11	1.176	160.9	158.7	+2.2	1.4
12	1.085	214.7	221.4	-6.8	3.1

eventually saturates at some moisture fraction, as seems physically plausible, then beyond that moisture fraction, m' is a constant, denoted by m_{sat} . Thus for $m > m_{sat}$, Eq. 6 predicts that ξ/D is again a linear function of the variable $m/(1+m)$ and has slope $K(1+m_{sat})$. When this straight line is extrapolated backwards ($\xi/D = 0$), it crosses the $m/(m+1)$ axis at $m_{sat}/(1+m_{sat})$, and when further extrapolated to $m/(m+1) = 0$, it intercepts the ξ/D axis at $-Km_{sat}$. This behavior is shown in Figure 9.

When the moisture content of the specimen is below the value for which the adsorption (or binding) of water molecules is fully saturated, m' , the bound-water mass fraction, is not a constant but depends on m , the total-water mass fraction. In fact, for sufficiently small values of m , m' must be directly proportional to m ; i.e., $m' = \alpha m$ as $m \rightarrow 0$, where α is the proportionality constant between the bound-water mass fraction and the total-water mass fraction. In this moisture range, Eq. 6 reduces to

$$\frac{\xi}{D} = K(1 - \alpha) \frac{m}{1 + m}, \quad m \rightarrow 0 \quad (7)$$

Thus, near the origin, ξ/D is again a linear function of $m/(1+m)$ and has slope $K(1-\alpha)$ as shown in Figure 9. If the value of K can be inferred from the $m > m_{sat}$ region of the curve, then α can, in principle, be determined from the slope near the origin.

By comparing Figures 7 and 9, one can see that results in bentonite at least qualitatively agree with the foregoing adsorption model. From Eqs. 4 and 6 one can obtain, for bentonite, the following parameters: $K = 2.0 \text{ V cm}^3/\text{g}$ and $m_{sat} = 0.145$ (14.5 percent moisture by dry weight). Parameter α was not determined because moisture levels below 14 percent were not measured.

For further verification of the reliability of the prototype sensor for measuring moisture in soil, a second, independent series of bentonite specimens was prepared at nominal moisture percentages ranging from 20 to 220 percent. These specimen materials were also loaded into glass vials for insertion into the sensor cavity. From the measured spin-echo signal amplitude, the moisture percentage was obtained by using the bentonite calibration curve shown in Figure 8. After the measurement with the prototype sensor was completed, the specimen material was removed from the glass vial, and the moisture content determined gravimetrically. A comparison of these results and the deviations and percentage of deviations between the NMR sensor and gravimetric moisture determinations are given in Table 1. As can be seen by comparing columns 3 and 4 of Table 1, the agreement between the moisture content obtained with the NMR sensor and the gravimetrically determined moisture content is quite good over the entire moisture range investigated. The average deviation in percentage of moisture between the sensor and gravimetric results is 2.0.

Dependence on Soil Type

Results of investigations with the silica and topsoil specimens indicated that the sensor response depends on soil type. Figure 10 shows the calibration curves experimentally obtained for each of the three soils studied, and individual data points are omitted for clarity. Only the initial part of the bentonite calibration curve is shown. Although the exact cause of this dependence on soil type is not understood, it may tentatively be interpreted in terms of the previously described moisture adsorption model, as follows. In bentonite, at moisture levels above 14 percent, the adsorption-bonding mechanism is saturated, and all additional moisture is sensed; in silica flour and topsoil, the adsorption mechanism is not saturated up to the investigated moisture levels, and not all added moisture is detected. Therefore, the observed calibration curves depend on the particular details of water adsorption in given soil types. This interpretation is supported by experimental results showing that larger NMR signals are obtained from pure water than are obtained from a comparable amount of water mixed with silica or topsoil.

From the practical standpoint, this dependence of calibration on soil type necessitates calibrating the sensor for each particular soil type in which it is to be used.

Influence of Organic Matter

The influence of organic matter was investigated by mixing peat, up to 20 percent by dry weight, into bentonite and silica specimens. The results for bentonite are shown in Figure 11, in which the solid curve is the sensor calibration curve for pure bentonite already shown in Figure 8. Within the limits of experimental error, the data points for the peat specimens fall on the previously established calibration curve for pure bentonite. This indicates that for bentonite there is no observable influence attributable to the added organic matter; however, for silica, there is an influence due to the organic matter as shown in Figure 12.

The results for the silica-peat specimens may be understood in terms of the adsorption model by recalling that previously not all of the moisture in the silica specimens was being detected because of an adsorption-bonding mechanism. The addition of organic matter to the silica provides capillary storage of free water, all of which is sensed. Therefore, the net fraction of water adsorbed or bound in silica flour that contains organic matter is less than that for a pure silica specimen at the same nominal moisture content. This results in a correspondingly larger signal. As the percentage of peat is increased, the observed moisture signals from the silica-peat specimens also increase (Fig. 12).

Influence of Dissolved Salts

The influence of dissolved salt was determined by preparing some bentonite specimens with water containing NaCl at concentrations up to 2,000 ppm by weight. These salt concentrations were chosen to conform to those found in runoff water along roadways (4). The results of sensor readings plotted against the gravimetrically determined moisture content are shown in Figure 13. The solid curve is the bentonite calibration curve previously shown in Figure 8. The good agreement between the sensor readings for the specimens containing dissolved salts and the calibration curve shows that the dissolved NaCl presents no significant effect on the NMR spin-echo signals from bentonite. The influence of dissolved salt was not investigated in soil other than bentonite; however, it is not expected to affect the sensor response, as long as it does not contribute to the number of hydrogen atoms present.

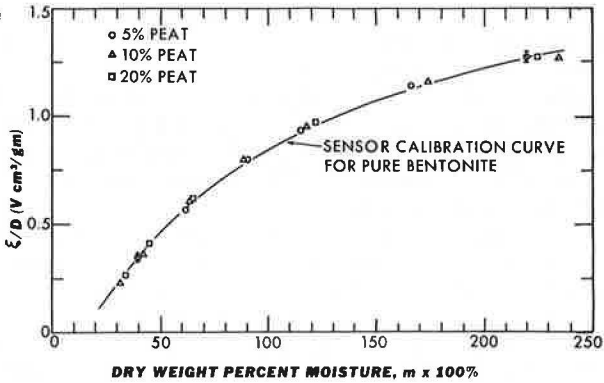
Effect on Moisture Distribution

The question of whether the presence of the buried sensor disturbs the free-field distribution of moisture in the soil was briefly addressed. This was done by selective coring and gravimetrically determining the distribution of moisture in a container of silica flour into which water was allowed to permeate, both with and without the sensor being present. These results indicated that the free-field distribution of moisture was not significantly altered by the presence of the buried sensor, at least for silica flour. Further studies of this question are planned during the field evaluation program.

SUMMARY

The basic feasibility of using the pulsed NMR spin-echo method for measuring soil moisture has been demonstrated, and a prototype of an implantable sensor has been developed. Because sensor response depends on soil type, calibration of the sensor for each soil in which it is to be used will generally be necessary. The sensor response was unaffected by the organic matter and the dissolved salt that were mixed into bentonite

Figure 11. Nuclear magnetic resonance moisture sensor signal versus percentage of moisture for organic peat with bentonite.



clay. For the silica flour, the sensor output was affected by organic matter in a qualitative agreement with a model of adsorption of water molecules in pure silica flour. The accuracy of the prototype version of the sensor was at least ± 1 percent moisture over the 6 to 25 percent moisture range, ± 2 percent moisture over the 25 to 100 percent moisture range, and ± 4.5 percent moisture over the 100 to 200 percent moisture range. The sensitivity (resolution of change in moisture percentage) was at least 0.5, 1.5, and 2 percent moisture respectively for the corresponding ranges of percentage of moisture.

A program is currently in progress to develop a fieldworthy version of the NMR moisture sensor with portable readout instrumentation and to conduct a field evaluation of the system. Plans call for the implantation of about 30 sensors in 4 or 5 test sites in various locations around the country. The system will be evaluated during 1 year for reliability, accuracy, and suitability for measuring moisture in highway-related applications. This project is expected to be completed by December 1976.

ACKNOWLEDGMENTS

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