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DESIGN AND CALIBRATION OF A NEUTRON MOISTURE METER

BY

K. N. BURN

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DESIGN AND CALIBRATION OF A NEUTRON MOISTURE METER

By K. N. BURN¹

SYNOPSIS

A probe using an actinium-beryllium neutron source and a scintillation-type detector was designed and built for use in determining the moisture density in soils below the surface of the ground. A portable battery-operated scaler was selected to make it possible to use the equipment in places where there is no available a-c supply.

Since the principle of the neutron moisture meter is based upon the physical interaction of neutrons with hydrogen atoms, considerable thought was given to the idea of calibrating the instrument in media other than actual soils; the advantages of using substitute materials are better uniformity and better control of both bulk density and distribution of hydrogen atoms.

Requirements of the artificial media are discussed and those adopted for calibration are described.

A number of techniques employing radioactivity have been developed for determining the density of moisture in soils and other similar materials. In principle, these utilize the scattering of neutrons from a radioactive source and the detection of the moderating effect of the moisture in the material on the neutron flux. The various techniques differ only in the means used to detect the moderated neutron flux.

THE PRINCIPLE OF THE NEUTRON METER

The predominant process that occurs when high-energy neutrons are scattered into a medium is a loss of velocity of each neutron as it collides with the nuclei of the atoms of the medium. Eventually, through loss of kinetic energy, the neutron velocities are reduced to those encountered in gases at normal tempera-

tures and pressures, and finally the neutrons are captured by the nuclei of the medium. The rate at which this slowing down process occurs depends upon: (1) the mass of the nucleus in collision with the neutron, and (2) the probability that the two will collide.

The mass of the hydrogen nucleus is nearly equal to the neutron mass. Collision with hydrogen atoms therefore reduces the kinetic energy and hence the velocity of neutrons more quickly than would collision with heavier nuclei. The large difference between the masses of hydrogen atoms and those normally encountered in soils means that the relative effectiveness of hydrogen atoms in slowing down neutrons is very pronounced.

The probability that a neutron will collide with the nucleus of an atom is dependent on the atom's scattering cross-section. For most elements, this value is low, usually increasing with decrease in neutron energy. The scattering

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cross-section of the hydrogen atom for high-energy neutrons, however, is larger than that for most of the other atoms present in soils.

These two features in combination make hydrogen the most effective medium for reducing the velocity of emission neutrons. If a detector of thermal (slow) neutrons is placed near a neutron

have had limited success because of one or two unknown parameters (1-3).²

In order to present the readings in the usual form (per cent water by weight), it is necessary to know the bulk density of the medium. A companion density meter, which operates on modification of flux from a gamma source, is nearly always used.

FIG. 1.—Neutron Probe and Portable Scaler.

source in a medium containing hydrogen, the activity registered is due almost entirely to neutrons that have been slowed down by hydrogen atoms. Other atoms present in a medium such as soil play a negligible part in this process. In natural soils, hydrogen may be present in several forms, but, with some exceptions, it occurs principally in the water held by the soil particles. Therefore, the "slow neutron" activity registered by a suitable detector can be related to the concentration of water in a soil medium by calibration. Attempts to calculate the relation between activity and moisture density

DESCRIPTION OF THE NEUTRON METER

The neutron moisture meter used by the Division of Building Research consists of a probe using a neutron source and a scintillation-type detector connected to a portable battery-operated scaler (Fig. 1). The probe was designed to slip freely through an aluminum access tube which has an outside diameter of $1\frac{1}{2}$ in. and a wall thickness of $\frac{1}{16}$ in. (Fig. 2). At the lower end is a source of

² The boldface numbers in parentheses refer to the list of references appended to this paper.

fast neutrons in the form of a mixture of actinium and beryllium which emits approximately 10^5 neutrons per sec. Direct radiation from the source to the detector crystal is prevented by a small shield of Mallory 1000 metal—an alloy of tungsten, nickel, and copper having a specific gravity of 16.9. A scintillation crystal with as large a diameter as possible was needed, and of three types that were tried a Li^6I (europium-activated) crystal was chosen because of its superior counting characteristics (Fig. 3). The largest multiplier phototube available that could be fitted into the shell of the probe was

one with a window diameter of $\frac{3}{4}$ in. It was necessary therefore to place between the scintillation crystal and the window of the photomultiplier tube a Lucite light pipe enclosed in a polished metal section to funnel the light activity from the one to the other. A preamplifier was added to obtain a suitable signal to drive this scaler through a 27-ft cable.

A retaining spring was provided to hold the components in contact with one another. Some annoying instability occurred before it was found that even the slightest movements of the probe would cause relative shifting of the parts, thus

FIG. 2.—Neutron Moisture Probe (Scintillator Type).

FIG. 3.—Probe Counting Characteristics for Neutron Meter with a Li^6I (Europium-Activated) Crystal.

affecting the readings significantly. After two or three trials a suitable spring was found.

The nuclear physics involved when slow neutrons strike a crystal is described in detail by Roy and Winterkorn (4). It is sufficient to say that the incident neutron reacts with lithium-6 to form an alpha particle and an atom of tritium, with a combined energy of 4.8 Mev. Each event produces a light pulse which is detected by the photomultiplier and converted to an electrical impulse. These are amplified and counted by the scaler.

The portable scaler, either a-c or battery-operated, was manufactured by Nuclear-Chicago Corp. (Model 2800). The automatic timing device was found to be less accurate than was required, so a stop watch was used to time the period of counting. An electrostatic voltmeter

FIG. 4.—Determination of Activity in the Wax Standard.

FIG. 5.—Determination of Center of Activity of Neutron Moisture Meter.

was added to make it possible to set and measure the input voltage to the neutron probe and the density probe. It was found that adjustment of voltage using the coarse indicator on the voltage control alone was not sufficiently accurate for calibration (Fig. 4).

To determine the center of maximum activity of the probe, a small drum of dried sand was prepared in which a $1\frac{1}{2}$ -in. thick slab of petroleum wax was placed at a known depth below the rim of the drum. By varying the vertical position

of the probe and plotting this against measured activity, a curve was obtained that indicated a rapid increase in readings toward a certain point followed by a rapid drop. This curve was symmetrical in shape so it was assumed that the point of maximum activity was reached when the probe was centered at the mid-depth of the layer of wax (Fig. 5).

THE CALIBRATION PROBLEM

Although the ultimate aim in constructing a neutron moisture meter is to measure the water contents of natural

soils, calibration *in situ* presents many difficulties. The determination of moisture content from field borings is not sufficiently accurate to use as a standard against which to calibrate. There is no control over moisture content in the field, and it is impossible, therefore, to reproduce results. Moisture contents of natural soils vary so randomly with depth that it would be extremely difficult to state what moisture content was being "sampled" by a neutron moisture meter. Since complete control of all im-

portant parameters is necessary for accurate calibration, it was decided that this must be undertaken in the laboratory.

Boundary Conditions for Laboratory Calibration:

Workers in this field have been able to calibrate the neutron meter in the laboratory by using a suitable medium in oil drums of standard 55-gal (U.S.) capacity, which measure about 23 in. in diameter and 36 in. in height. Experiments have shown that for the usual range of bulk densities of soils, the diameter of sample actually involved in the action described depends only upon the hydrogen density. The maximum diameter "sampled" by a neutron-measuring device at the lowest moisture contents generally encountered in soils is about 2 ft. This diminishes rapidly to 15 in. with only a slight increase in moisture content (5). Thus, an oil drum of this size filled with soil serves as a sample of infinite extent so far as this measuring technique is concerned. All of the drums used in this calibration were first coated on the inside with a zinc suspension to reduce the formation of rust.

Use of Natural Soil Material:

Natural soil was the first medium to be considered since it is so readily available. For calibration purposes, however, it has several disadvantages. Uniform mixing of soil and water is very difficult, especially with clays. The use of sand makes mixing easier, but very high moisture contents cannot be attained without movement of water under gravity. Even if the mixture of soil and water could be accomplished in a satisfactory manner, difficulties in placing and in control of density would occur. Total weights and volumes might be reproduced within satisfactory limits, but densities of layers may vary appreciably.

Requirements of Artificial Materials:

Fortunately, the neutron technique measures the relative occurrence of hydrogen atoms, not of water molecules; therefore, for calibration purposes hydrogen atoms in some other form can be substituted for hydrogen atoms in water. In fact, even the solid portion of the soil and the oxygen atoms associated with the hydrogen atoms in the water molecules can be replaced by any other material provided the scatter geometry of neutrons is not seriously affected. This will not occur so long as the simulated mass of the medium does not differ significantly from that of natural soils with the only variation being introduced by the addition of hydrogen atoms (6) (Fig. 5).

Some elements not only act as targets for the elastic rebound of neutrons but also capture and absorb neutrons to become induced radioactive isotopes. The presence of such elements takes a certain percentage of neutrons out of play, thus causing an erroneously low reading of thermal-neutron activity. Among the elements to be avoided are boron, iron, and the abundant halogen group.

Since atoms of small mass are more effective in slowing down neutrons than those of large mass, their use should also be avoided. Elements whose atomic weights range between that of hydrogen and those of the lightest elements usually encountered in soils (that is, oxygen, aluminum, and silicon) should not be used. Finally, the inherent accuracy of the neutron technique is much higher than the oven-drying method of determining water contents; therefore to calibrate the first against the second simply adds errors. If the hydrogen density of the material can be calculated from its specific gravity and its chemical formula, provided this is accurately known, a much more reliable calibration point can be obtained.

Use of Gaseous Mixtures:

After initial consideration a mixture of hydrogenous and nonhydrogenous gases appeared to be an ideal way of providing uniform and adjustable hydrogen densities. The problems of placing, homogeneity, and density control would not exist. Further consideration revealed

(or one containing only a very small percentage of hydrogen atoms) with a density of about 2 g per cu cm. To this could be added a hydrogenous liquid with a density of about 1 g per cu cm. With this combination, it would have been possible to follow the normal density to moisture content relationship of

FIG. 6.—Moisture Content - Bulk Density Relationship for Natural Soils and Substitute Materials.

that for the simulated moisture content range required, gases were unacceptable because of their low bulk densities. Calculations also showed that for even the low end of the range the required hydrogen density could not be obtained by the use of gaseous compounds without resorting to excessively high pressures.

Use of Liquids:

Recognition of the density requirement led next to consideration of the possibility of finding a nonhydrogenous liquid

natural soils (Fig. 6) by selecting two liquids and mixing them in various proportions.

Heavy liquids such as those used in laboratory studies to separate ores by flotation were investigated but were found to be prohibitive in cost. Also their densities were too high and they had the added disadvantages of containing some halogen element or of being toxic (Fig. 6) (thallium formate). The tables of physical constants of organic and inorganic compounds in the Handbook of Chem-

istry and Physics were searched but it was found that the liquids with the required high density contained either a halogen or some other unsuitable element, and a combination with the appropriate density range could not be found.

Fluids comprised only of organic compounds, such as petroleum products, were also considered, but their low bulk density and high concentration of hydrogen made them unsuitable for the desired range.

Use of Solids:

Solids with low melting temperatures would be suitable as calibration media since the material could be poured into place before solidifying. This type of medium would be ideal in that it could be kept indefinitely as a calibration standard without loss of hydrogen content or change in bulk density. The bulk densities of these materials, however, are about 60 lb per cu ft and the hydrogen concentration about three fourths that of water, giving relationships considerably removed from those of natural soils.

Use of Laminated Solids:

Interlayering of hydrogenous and nonhydrogenous films was considered. The cutting and stacking of thin disks in different combinations to produce various hydrogen densities seemed a tedious arrangement that might better be accomplished by rolling different film combinations onto an access tube. Many plastic films are now available, and metal foils, such as aluminum, could be used to provide the required bulk density. This would probably set up a directional geometry in the medium, however, and the idea was dismissed since the neutron flux might be distorted and therefore not representative of natural soils.

Use of Solid Mixtures:

The next step was to consider mixtures of hydrogenous and nonhydrogenous solids, even though this introduced the accompanying problems of mixing, placing, and density control. Uniform mixing of different particles is possible only through prolonged agitation of a mix, but this presents no great difficulty, requiring only time and suitable equipment. When a material comprised of particles of different density and size is removed from where it was mixed and is then weighed and placed in some other container, segregation will always occur to some degree. Spherical particles of uniform size could be used to minimize this effect, and from this the maximum range in densities could be calculated.

Several possibilities of using sand-size particles were considered. Large sizes were thought to be unsuitable because they might have the effect of concentrating hydrogenous and nonhydrogenous particles in distinctly separate pockets, and thus introduce a scatter geometry different from that in natural soils. With this size stipulation the following combinations were considered:

1. Mixtures of hydrogenous spheres with nonhydrogenous spheres to give a fairly complete range of hydrogen densities.

2. A medium consisting of nonhydrogenous spheres coated uniformly with some hydrogenous material. Film thickness could be modified to vary hydrogen density over the lower range of natural soil moisture densities.

3. Spheres of material of lower hydrogen "concentration" than that of water, at various compacted densities for the higher range of natural soil moisture densities.

4. A combination of the above methods to give required equivalent moisture densities.

Nonhydrogenous spheres (approximately) of the correct density are available in the form of sorted Ottawa sand. (Glass spheres are also available but because the material generally contains small proportions of boron it is unsuitable.) Suitable hydrogenous spheres are not readily available and coating spheres with a hydrogenous material might require the development of elaborate equipment and techniques. The first two possible combinations were therefore discarded.

The third technique was used by the author in a previous calibration of a foil-type probe. Various mixtures of well-graded sand and powdered dextrose were used with very small quantities of water. Considerable difficulty was experienced in mixing and placing, and uniformity of density was probably not attained since compaction was done with a hand tamper.

Use of Solid-Liquid Suspensions:

Another possible way to prepare a suitable calibration medium is to combine a solid and a liquid. With proper agitation and circulation, the problem of homogeneity would be solved, but this operation would involve ancillary equipment so this idea was discarded.

Two suspensions used in commercial separation of ores by flotation are known. One is unsuitable because it contains iron which has a high absorption cross-section for neutrons; the other contains galena (lead sulfide) and gives a range of densities much higher than those in natural soils for the same moisture contents.

Use of Solid-Liquid Solutions:

Several possibilities of mixing soluble solids and liquids were investigated. No combination was found that gave a complete range of equivalent moisture contents, and many compounds of the right

specific gravity were soluble only in very small proportions. The most suitable solute that could be found was sodium hexametaphosphate $(\text{NaPO}_3)_x$, commercially available as Calgon, which permits a maximum concentration equivalent to a minimum moisture content of 50 per cent, a point at about the center of the required range (Fig. 6).

Use of Saturated Solid-Liquid Mixtures:

An obvious combination for the lower end of the range was a saturated mixture of well-defined sand and water. This is equivalent to a natural soil medium below the groundwater table. Difficulties of placing and of achieving homogeneity were problems which it was hoped could be overcome by sedimenting sand into water in a suitable manner and then by vibrating to make the medium more dense, for further points at lower water contents.

The void ratios for the maximum and minimum packing are known for spherical particles of uniform size. These would be equivalent to water contents of 13.2 and 33.3 per cent respectively for saturated mixtures of Ottawa sand, but the practical possibility of obtaining the two extreme conditions is small, and the range of water contents would be considerably reduced within these theoretical limits.

PREPARATION OF CALIBRATION MEDIA

Because of the difficulties already discussed, it was necessary to calibrate with other than the ideal media mentioned. Four different mixtures were used.

Ottawa Sand and Water:

The sedimentation of wet sand in water proved to be extremely difficult. Hand sprinkling it into a barrel of water produced erratic surfaces and densities. To overcome this a brass screen with

openings slightly larger than the sand particles was made to fit across the top of the drum on to which wet sand was spread and troweled through. This was an extremely slow process, and the idea of using pre-wetted sand was abandoned although there were two advantages to the method, which were incorporated in the next step: (1) more uniform distribu-

FIG. 7.—Apparatus for Preparation of Sedimented Sand Samples.

tion of sand, and (2) more uniform level of energy of particles entering the surface of the water.

Figure 7 shows the apparatus that was finally used which produced quite uniform results. It consists of a small trough divided into five sections, pivoted on the access tube in the center of the drum and riding around the rim of the drum on a ring made from sheet metal. The access tube was centered by screws on a hub connected by four spokes to the outside track. The entrant velocity of the particles was reduced as much as possible by lowering the distributing ap-

paratus until the opening at the bottom of the trough was no more than $\frac{1}{4}$ in. above the water surface. Distribution was by hand; one person rotated the equipment slowly while the other fed sand into the various compartments in the required proportions. Scoopfuls of dry sand were kept as uniform in weight as possible by using a straightedge to strike off excess from an even rim. Weights of sand proportional to the area covered by each compartment produced a sand surface that was fairly uniform, but it was always necessary to add a little more in certain compartments to raise the sand beneath them to the same level as the rest.

Thickness of the layers was determined by measuring the distance from the rim of the drum to the surface of the sand at various locations using a wooden dowel fitted with a small foot at the lower end. All readings were made to $\frac{1}{32}$ in., and distribution was continued until these agreed within 0.4 in. for any one layer. The total volume of each layer was then calculated, and from the weight of the sand added and its specific gravity the volume and weight of water were determined.

Two such samples were prepared; the result of these calculations for one of the samples is shown in Fig. 8. The water density appears to increase with depth a little over 1 lb per cu ft from top to bottom. Determination of activity with the neutron meter at various depths, however, indicated more uniform distribution than that calculated.

After obtaining initial measurements on loose sand, the samples were compacted using a gasoline-driven immersion-type concrete vibrator. It was first applied to the exterior of each drum following a uniform pattern both in position and time interval. During the beginning of this stage it became obvious that air had been entrapped during the sedi-

menting of the dry sand. The sample in one of the drums released only a very small amount of air, but the amount that escaped from the second drum was appreciable. Although volume reduction was slight after vibration, further calibration points were determined for these samples. Further compaction was accomplished by immersing the vibrator directly in the mixtures and, assuming

Calgon and Water:

Commercially available Calgon used as water softener is about 94 per cent sodium hexametaphosphate (NaPO_3)_x. The remainder is sodium carbonate which is added to adjust the pH. Because this additive causes some effervescence when combined with water, producing suds, the pure unadjusted sodium hexametaphosphate was used. To facilitate mixing,

FIG. 8.—Sedimented Sand and Water.

uniform densities, two more calibration points were obtained. Unfortunately, this treatment resulted in water densities of about 20 lb per cu ft when less than 15 lb per cu ft had been expected.

Another point on the curve was obtained by hand compacting damp sand into the drum. Sand and water were well mixed on a concrete floor and then weighed into a drum in carefully compacted layers. The thickness of each layer was measured as before and the water density determined by the oven drying method.

it was obtained in fine powdered form. Mixing became increasingly difficult as the concentration increased. Three electric stirrers were used to keep the mix well agitated and to draw the powder down from the surface without causing it to lump or to draw in air. Several density determinations were made for each sample and they agreed very closely.

Actual water contents were determined by chemical analysis. This involved diluting the mixture in a solvent and then measuring by titration the amount of phosphorus in the solution. From this, the amount of sodium hexametaphos-

phate by weight was determined, the balance being water.

Although two points were obtained which appear to be in practically the right positions with the calibration curve, the mixture was not completely satisfactory. In the first place, the mixtures were not perfect solutions but were in fact partly suspensions. In the specimens taken from the first mixture, a settling out of this suspended material became evident in three or four days.

Bentonite, Water and Ottawa Sand:

The search for some other material for calibration led to the mixing of bentonite and water, at a moisture content of about 1000 per cent by weight, to which Ottawa sand was added in increasing amounts. The liquid limit of bentonite is about 640 per cent and its stiffness limit well above 1000 per cent. The mixture was therefore in the thixotropic range. This property made it possible to agitate

FIG. 9.—Profiles of Slow Neutron Activity for Calibration Samples.

Readings with the neutron meter were obtained on the day of mixing and the day following, but settling out of undissolved Calgon might have been significant. In the second mixture, this probably was not very important because of its very high viscosity. It had also been impossible to avoid trapping some air while stirring, and when mixing was complete the air bubbles slowly made their way to the surface. This condition would cause a small error in the determination of water densities, erring on the high side.

the gelled material, mix in a quantity of sand, and then allow the whole mixture to set up again. These proportions of bentonite and water permitted no settlement of sand even in the thinnest mixes and, because the mixture was a liquid when agitated, it was possible to pour it into glass dishes to determine the densities in the same way as that used for the Calgon-water mixtures. This combination of materials made it possible to prepare samples with water densities from 58.2 to 30 lb per cu ft. The water contents were determined by oven-dry-

ing the sample and correcting for the small proportion of chemically held water in the bentonite, the amount of which was determined by subjecting oven-dried samples to further drying at 1000 C.

The correction for this series of calibration points was about $\frac{1}{4}$ lb per cu ft throughout since the ratio of bentonite to water remained fairly constant.

FIG. 10.—Calibration Curve for Neutron Moisture Meter.

Dextrose and Ottawa Sand:

To obtain other points on the calibration curve between 5 and 20 lb per cu ft, two samples were prepared by mixing together, in a small mechanical mixer, quantities of damp Ottawa sand (water content about 2 per cent) and powdered dextrose. Dextrose contains fewer hydrogen atoms on a weight basis than water in the ratio of 1 to 1.665, so equivalent water contents are easily determined. It was necessary, of course, to resort to placing the mixture in layers and compacting to obtain a required density.

PROCEDURE AND RESULTS OF CALIBRATION

A $2\frac{1}{2}$ -gal pail was prepared as a standard by filling it with petroleum wax, in the center of which was placed a short section of aluminum access tube. Readings of activity in this standard were obtained both before and after taking readings in each calibration sample.

A profile of activity was obtained for each sample by taking readings at least at every 2 in. of depth, for a period of 5 min or more (Fig. 9). Any readings that appeared to be erratic were repeated. Those that fall along the straight section of the curve—that is, those not influenced by the upper and lower limits of the sample—were averaged. The calibration curve shows the ratio of the average reading in the medium to the average reading in the standard in relation to the moisture density in pounds per cubic foot (Fig. 10). One point was obtained for a moisture density of zero in a drum filled with dry powdered Calgon.

STABILITY OF THE INSTRUMENT

The probe was placed in the drum of Calgon and water on six different days and profiles of activity determined. From these were determined the ratios of counts in the medium to counts in the standard. The first was taken in mid-December, 1959, the ratio for which was 0.807. Further ratios determined on five days in early January, 1960, gave the following values: 0.812, 0.808, 0.804, 0.804, and 0.810. The smooth calibration curve would also appear to substantiate the fact that the instrument is stable.

CONCLUSIONS

1. The deviation of points from the mean position on the calibration curve is not large, indicating stability in the instrument. This is further substantiated by the results of readings obtained on the same sample on different days.

2. The bentonite-water-sand mixtures partly satisfy the conditions for an ideal substitute medium in that they were liquids which eliminated the problem of density control. The actual water content, however, had still to be determined by the oven-drying method.

3. The Calgon-water mixtures more nearly met the requirements for an ideal medium by eliminating the need for density control, in spite of the fact that the mixtures were partly suspensions. A small error is introduced in determining the water content by dilution and titration and probably a somewhat larger error in determining the densities because of small quantities of entrapped air.

4. The third group of points for samples prepared by sedimenting sorted sand into water are quite scattered. Several sources of error exist and uniformity of density was not attained. Compaction by vibration was not very effective and no doubt resulted in an even greater variation in density than existed in the initial samples.

5. The Ottawa sand and dextrose samples are also prone to the errors involved in placing and compaction, but the points determined for them appear close to the mean curve.

6. The accuracy of the instrument would appear from the calibration curve

to be at least within 1 lb per cu ft. Only one point has a larger error, which is believed to have been caused by an erroneously high calculated density for a sample that contained an appreciable amount of entrapped air.

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