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Publisher's version / Version de l'éditeur:

Canadian Geotechnical Journal, 1, 2, pp. 94-103, 1964-03

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by

K. N. BURN

Reprinted from
CANADIAN GEOTECHNICAL JOURNAL
Vol. I, No. 2, March 1964
pp. 94-103

RESEARCH PAPER No. 212
OF THE
DIVISION OF BUILDING RESEARCH

19632

Price 25 cents

Ottawa
March 1964

NRC 7847

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CALIBRATION OF A NEUTRON MOISTURE METER IN LEDA CLAY

K. N. BURN*

ABSTRACT

A neutron moisture meter using a scintillation detector for slow neutrons was previously calibrated in materials carefully selected to avoid inaccuracies resulting from neutron absorption. Preliminary readings of moisture density in the field using this calibration, however, were not in agreement with values obtained by auger borings. Consequently the instrument was recalibrated; in the laboratory using samples of remolded Leda clay; and in the field, by taking readings throughout a summer in the natural material. Details of moisture content and density control are described. The new calibration curve is parallel to the original one, but considerably displaced from it by what appears to be a neutron absorption effect. Chemical analyses of the clay and pore water failed to detect any elements whose capture cross-sections for neutrons are large. They did however indicate a high percentage of iron whose capture cross-section is a little larger than those for elements occurring naturally in mineral soils. Although further research is required for confirmation of this observation, results of preliminary experiments point primarily to the presence of iron in quantities large enough to explain the discrepancy between the two calibration curves.

SOMMAIRE

Un appareil à neutron employé pour la mesure de la teneur en eau et faisant usage d'un détecteur de scintillation pour les neutrons lents a été tout d'abord calibré dans des matériaux choisis avec soin pour éviter les imprécisions dues à l'absorption de neutrons. Les lectures préliminaires de l'humidité et de la densité obtenues sur le terrain au moyen de cette calibration ont été bien différentes de celles obtenues dans les trous de tarière. Conséquemment, l'instrument a été recalibré en laboratoire en employant des échantillons remaniés d'argile Léda, et sur le terrain en prenant des lectures au cours de l'été dans le dépôt naturel. Les détails relatifs au contrôle de la densité et de la teneur en eau sont décrits. La nouvelle courbe de calibration est parallèle à la première mais considérablement décalée, dû probablement à un effet d'absorption de neutrons. Les analyses chimiques de l'argile et de l'eau interstitielle n'ont pas révélé la présence d'éléments dont les aires efficaces de capture sont grandes. Elles ont indiquées cependant une forte teneur en fer dont l'aire efficace de capture est légèrement plus grande que celles des éléments habituellement rencontrés dans les sols minéraux. Quoiqu'une étude plus approfondie sera nécessaire pour confirmer cette observation, les résultats des expériences préliminaires indiquent que le fer est présent en quantité suffisamment grande pour expliquer la différence entre les deux courbes de calibration.

Leda clay is a deposit of fine-grained marine sediment occurring extensively in the valleys of the Ottawa and St. Lawrence Rivers, which once formed the basin of the post-glacial Champlain Sea. Although it was an arm of the Atlantic Ocean the salinity of the water in the Champlain Sea was modified by the enormous quantities of melt water that carried the silt and clay particles into it from the retreating ice sheet.

The physical properties of Leda clay are closely linked with its water content. Significant moisture changes occur near the surface of the soil with seasonal variation in climate and result in appreciable volume changes. These can lead to damage of light structures constructed on shallow foundations on

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such soil, a consequence that is increased by the presence of trees (Bozozuk and Burn, 1960).

In order to augment facilities for the study of these properties the Division of Building Research sought a method of *in situ* testing that would make possible repetitive measurement of moisture content in exact locations. The neutron method of moisture detection was selected and a moisture meter of the scintillation type was designed, constructed and calibrated in the laboratory, using artificial media. This work was reported previously (Burn, 1960) and resulted in the calibration curve shown in Figure 1.

Based upon the experience of others, it was hoped, although with some reserve, that this curve would be valid for most soils in the field. Preliminary field tests, however, indicated substantially lower moisture contents, using

FIGURE 1. Calibration curve of neutron moisture meter

this relationship, than those obtained by oven-drying samples. As the differences were quite large, it was considered necessary to check the laboratory calibration of the instrument in Leda clay to seek the reason for the discrepancy.

CALIBRATION

Laboratory Controlled Points

Two large samples of Leda clay at different moisture contents were obtained. Each was remoulded separately in small quantities to a liquid consistency and stored in a humid room to prevent evaporation. When sufficiently large quantities had been prepared the samples were transferred to 45 gallon drums. A small pencil-type concrete vibrator was used to break down lumps of re-gelled clay as the material was added to each drum and vibrating had the added effect of releasing trapped air from the liquefied material.

In order to obtain a third point on the calibration curve, the drier of the two samples was spread on a large table top for further drying. Its moisture content was kept as uniform as possible by turning the sample every few minutes with a small electric cake mixer. Although the clay at this reduced moisture content was much stiffer than it had been originally, it was still possible to make it flow slowly in the drum by vibrating.

From about every 2 in. of depth three small samples of clay were obtained for determining moisture content by oven-drying. At about every 6 in. of depth quantities of the clay were obtained for measurement of density. The average thus obtained was compared with the density obtained by weighing the drum and its contents and measuring the volume of the clay. The difference between the two methods was less than 0.5 per cent. Moisture content distribution in the three drums in terms of moisture density is shown in Figure 2(a).

Thermal neutron activity was recorded for each inch of depth, and ratios of these readings to readings in a wax standard were calculated (Figure 2(b)). Mean values of those activity ratios unaffected by the upper and lower limits of the sample were determined and plotted against moisture density on an enlarged section of the original calibration curve in Figure 3. It was immediately apparent that the calibration points fell on a line parallel to the original calibration curve but considerably displaced from it.

Field Trials

In advance of the work in the laboratory an access tube had been placed in the field in order that readings could be taken periodically during the summer of 1960 for later correlation. The access tube was located 20 ft. from the base of an elm tree, near enough to have accelerated moisture changes without running the risk of coming too close to the main roots. With a drill rig to maintain vertical alignment a special Jamaica spiral auger, ground to a diameter slightly less than the outside diameter of the access tubes, was used to bore the hole to a depth of 12 ft. The aluminum access tube, to which a conical tip had been welded, was pushed into the hole with the aid of the

FIGURE 2. Profiles of activity and moisture density for laboratory samples of Leda clay

FIGURE 3. Calibration curve for Leda clay

hydraulic feed. Activity ratios were determined for every foot of depth at intervals of time chosen to coincide with certain stages of dryness of the soil. These stages were estimated on the basis of weather records as described by Bozozuk and Burn (1960).

On each day that neutron moisture meter readings were made, water content samples were obtained by hand augering from the soil adjacent to the access tube. The holes were all bored within a radius of 2.5 ft. of the access tube, but no closer than 1 ft. Measurements were made periodically from June to September, when a test trench was dug adjacent to the tube and block samples for density measurements and for moisture content determinations were procured. Three samples for each were taken from each 6 in. of depth.

Density measurements. Wet densities of the block samples were determined by two methods. The first, used for most of the samples, consisted of computing the density from weight in air and the volume by displacement of oil in a graduated tank. The second, used for a few samples representing the whole profile, especially the more friable and porous near the surface, consisted of computing the density from weights in air and in water after the sample had been coated with a measured amount of wax of known specific gravity.

Results of the two methods agreed fairly well, but those of the second consistently gave values slightly lower than those obtained by the oil displacement method. All values at any one depth agreed within 3 lb./cu. ft., with the exception of two or three at the bottom of the profile that varied by as much as 5 lb./cu. ft. The standard deviation was 0.4 lb./cu. ft. Mean

values of these wet densities were used in determining the dry densities for the clay.

The neutron meter actually averages conditions over a small increment of depth. Consequently the profile of dry density was established by taking the average of a value at any one depth and those from immediately above and below it. The best curve through these points is shown in Figure 4.

Moisture density. Moisture density profiles were established using the values of dry density, obtained as just described, and the values of moisture content, obtained by oven-drying samples from the borings made periodically near the access tube. Again, because the neutron probe "samples" a volume of soil larger than a specimen procured by hand boring, three successive values of moisture content were averaged for each depth: the moisture content at that depth and those from immediately above and below.

FIGURE 4. Dry density *versus* depth

Individual values of moisture density thus determined for each depth in every profile are plotted against the appropriate activity ratio found at the time of the boring. These relationships are shown as dots in Figure 3. They are distributed on both sides of the new curve drawn through the three laboratory points, but most of them fall on the upper side.

Points marked with crosses represent the results of tests on samples taken from the trench in the immediate vicinity of the access tube and are therefore more reliable than those from the boring operations. General distribution of the points is the same as that from the auger borings. The average departure from the new curve is 0.6 lb./cu. ft., which is considered good agreement. The scatter, though appreciable, is probably just the reflection of natural variations in moisture content over short horizontal distances. For example two borings, made on the same day $3\frac{1}{2}$ ft. apart, yielded water content profiles that agreed within 1 per cent to a depth of 7 ft., but below this the differences increased rapidly to 10 per cent.

The general trend between 30 and 35 lb./cu. ft., which roughly delineates depths between 6 and 10 ft., may be the reflection to a slightly lesser degree of the process that caused a general shift of this calibration curve for Leda clay to the right of that for inert materials. This over-all shift in position from the original calibration curve to the new one represents a reduction in detected neutron activity of about 11 per cent.

Possible Causes of Differences in the Calibration Curves

If it is agreed that the initial calibration of the instrument in inert materials is substantially correct, and that the instrument is stable with regard to time as indicated by readings of activity in the wax standard, only two possible explanations for the shift in position of the calibration curve appear to remain. Either moisture contents are not determined correctly by oven drying or there exists in Leda clay some other condition that reduces the flux of slow neutrons in the vicinity of the detector.

Adsorbed water and OH lattice water account for approximately 4 per cent of the dry weight so that the true value of moisture density is a little higher than that calculated from moisture contents based on oven-drying of samples at 105° C. This, however, increases the difference between the two calibration curves; some other factor or factors therefore must be involved in this discrepancy.

Analyses of the pore water from samples of Leda clay indicated the presence of salts in varying amounts. Samples of the pore water involved in this calibration were subjected to spectrographic examination, which showed that Ca, Mg, Si, Na were the main constituents of the salts, and that they contained small quantities of chlorine. This element has a high capture cross-section for neutrons; further testing therefore was carried out to find the concentration of chloride ions present in the pore water. The results showed less than 0.1 g/litre for the two original laboratory samples. Such minute quantities could have very little effect on the neutron flux, and according to J. W. Holmes (1956) would not reduce the counting rate by any more than

2 per cent. High salt concentrations of 15 g/litre are not common in Leda clay, but at depths greater than 10 ft. they do often exceed the values of these two samples (Crawford, 1961) and must therefore be taken into consideration at each location where the neutron meter is used as a tool to study changes in water content.

A second possible reason for reduction in neutron flux would be the presence in the clay minerals of some element or elements of high capture cross-section. Mineral analyses show that the particles are primarily feldspars, micas, quartz, and amphiboles, with some chlorite, montmorillonite, and vermiculite, which are not always present in detectable amounts. None of the chemical formulae of these minerals indicated the presence of elements of very high capture cross-section for neutrons. Both potassium and iron, however, have relatively higher capture cross-sections than other elements present in the soil, and if they occur in sufficiently large quantities they could produce an absorption effect equivalent to small amounts of elements which have high capture cross-sections for slow neutrons.

From analyses of the mineral composition (Brydon and Patry, 1961) and the chemical formulae of these minerals a rough estimate was made of the quantity of potassium occurring in Leda clay. It was necessary to make some broad assumptions, some of which proved later to be incorrect, but on the strength of this estimate some preliminary experiments to determine the absorption effect of potassium in solutions of potassium carbonate were conducted. The results showed an appreciable decrease in counting rate, roughly linear with concentration; a reduction of 4 per cent was measured in a solution whose concentration of potassium was equivalent to 2 per cent of the dry weight of the soil at a water content of 50 per cent. The estimated proportion of potassium in the clay was 5 per cent, which, if correct, would have caused a reduction in counting rate of about 10 per cent. The results of partial chemical analyses of the samples used in the laboratory and field calibration showed potassium concentrations of only 0.5 to 1.0 per cent of dry weight, and this could not account for the discrepancy between the curves.

Further chemical analyses, however, revealed a relatively high percentage of iron—between 6 and 7 per cent of dry weight. Theoretically the absorption effect of this quantity of iron should be equivalent to the absorption effect of a little less than 5 per cent potassium, in which case it would account for a reduction of counting rate of about 10 per cent. This calculation is based upon the relative occurrence of atoms of the two elements and the relative magnitudes of their capture cross-sections. Iron is a little heavier than potassium and, therefore, has fewer atoms for a given weight, but this is largely offset by a slightly larger capture cross-section. When these two factors are considered it requires an extra 25 per cent by weight of iron over potassium to accomplish the same reduction in counting rate. By coincidence this is the approximate ratio of iron actually present in the soil to the calculated amount of potassium.

The combined absorption effect of slow neutrons, caused by the presence of iron and a small quantity of potassium, would then appear to explain the

total shift in position of the calibration curve for inert artificial soil media to that for Leda clay. This may limit the usefulness of the neutron method of measuring moisture densities in Leda clay, but fortunately analyses (Brydon and Patry, 1961) show fairly uniform mineral composition throughout. It is therefore anticipated that the corrected calibration curve can be widely used in this deposit.

Instrument Stability

Although the instrument appeared to be stable in the laboratory, it was considered desirable to check its performance in the field. Accordingly, three sets of profiles were read in one access tube on one day, four on another. The results showed an average variation between maximum and minimum readings to be 0.31 lb./cu. ft., and the maximum variation to be 1.0 lb./cu. ft. of water. The standard deviation, however, was only 0.18 lb./cu. ft.

Since reporting the initial calibration of the neutron meter, decay of the detector crystal has caused a slow but perceptible change in the counting characteristics of the system, making it necessary to use higher voltages across the photomultiplier tube to maintain the level of counting. This is done by checking the position of the voltage plateau for the wax standard and making the necessary voltage adjustment to operate on the flattest section of the curve. The accuracy of activity ratios is not affected, but it does mean that more time is involved in making readings, amounting to an extra hour every few days.

CONCLUSIONS

1. A substantial difference in counting rate by a neutron moisture meter exists between natural Leda clay and inert soil substitutes at the same moisture density.
2. A calibration curve established for Leda clay appears to be parallel to that for artificial materials, indicating a uniform absorption of slow neutron flux amounting to 11 per cent.
3. The effect of chlorine salts in the pore water of the clay soils used in this calibration was investigated and the neutron absorption was found to be negligible because of very low concentrations. This factor would be significant where salt concentrations are higher.
4. The absorption effect of potassium on the neutron flux was investigated experimentally and found to cause a significant decrease in counting rate.
5. Chemical analyses showed that potassium is present only in small quantities, but that iron is present in quantities regarded as sufficient to account for most of the reduction in the field counting rate.
6. The general usefulness of the neutron meter as an absolute moisture measuring device appears to be limited by neutron absorption. Further study of neutron absorption by iron and potassium is needed.
7. Moisture density of Leda clay at the site under study can be determined within an accuracy of ± 0.5 lb./cu. ft. This may well be extended to other

areas in this widespread deposit because of the uniform mineralogical composition of the material.

8. The instrument appears to be stable, making it possible to reproduce readings of activity ratio equivalent to moisture densities with a standard deviation of less than 0.2 lb./cu. ft. Even without absolute values of dry density this would permit the use of the instrument in following moisture density changes very closely.

ACKNOWLEDGMENTS

The writer wishes to thank R. Ironside, N.R.C., Division of Applied Chemistry, for the chemical analyses used in this report; Dr. Klaus Geiger of the N.R.C., Division of Applied Physics for many helpful discussions; and colleagues at the Division of Building Research for their continued encouragement in this project; particularly L. Boyd, who spent many hours accumulating the readings upon which this work is based. This report is a contribution from the Division of Building Research and is published with the approval of the Director of the Division.

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