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THE EFFECT OF IRON IN THE DETERMINATION OF MOISTURE
CONTENT BY THE NEUTRON METHOD

BY

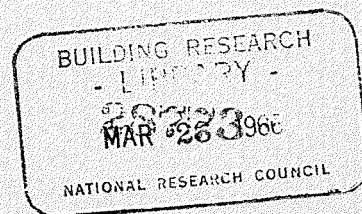
K. N. BURN

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EFFET DU FER DANS LA DETERMINATION DE LA TENEUR EN HUMIDITE PAR LA METHODE NEUTRONIQUE

SOMMAIRE

La différence constatée entre les humidités relevées au laboratoire et celles enregistrées *in situ* au moyen d'un hygromètre à neutrons s'explique par la présence, dans le sol naturel, d'un élément qui absorbe les neutrons en quantités suffisantes pour réduire de 11% le taux de comptage. Des analyses chimiques ont révélé une teneur en fer de 7% dont la section efficace de capture est légèrement plus grande que celle des autres éléments qui se trouvent dans les sols naturels. Une série d'expériences effectuées dans un sol artificiel auquel on a ajouté des quantités de fer de plus en plus importantes a confirmé le fait que le fer présent dans le sol naturel explique dans une large mesure la différence constatée.

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EFFECT OF IRON ON THE DETERMINATION OF MOISTURE CONTENT BY THE NEUTRON METHOD¹

K. N. BURN

The method of determining moisture content by measuring the moderation of high energy neutrons scattered into solid media is a very promising one, and indeed is already widely used in some industries. The rapidity with which measurements can be made, and the obviation of destructive sampling are the prime advantages of this technique. Under controlled conditions and where uniformity of manufactured test specimens is maintained within narrow limits it provides accurate results, but when applied to materials in the natural state, unknown conditions and constituents may significantly affect the precision of the method.

The physical principle upon which this technique is based is a simple one. High-energy neutrons are scattered into a test medium and they collide with the atoms of the medium in the vicinity of the source. Because the masses of hydrogen atoms and neutrons are approximately the same, and those of other elements likely to be present several times greater, the neutrons lose kinetic energy in a manner that is essentially proportional to the quantity of hydrogen atoms within a given volume. A detector of thermal neutrons indirectly measures the loss of kinetic energy. By assuming that all hydrogen atoms in the test medium are chemically associated with oxygen atoms in the form of water the technique becomes a method of measuring moisture content.

A calibration curve relating detected thermal neutron activity to the weight of moisture per unit volume is usually produced by placing a source of high-energy neutrons at a fixed distance from a thermal neutron detector on or in media whose moisture contents have been accurately determined and observing electronically, in counts per minute, the rate at which the neutrons are slowed by hydrogen atoms in the moisture. Source materials, detectors, and their relative geometry vary considerably from one instrument to another, but such measurements usually show a linear increase in activity at the detector for increasing moisture content of the media. Of course, the presence of unknown quantities of hydrogen in organic compounds or other natural materials introduces errors in this relationship.

A second basic assumption is that the loss of kinetic energy by collision is the only significant reaction taking place when neutrons are scattered into a test medium. All elements, however, have some ability to absorb neutrons over various energy levels, and this results in a reduction of detected thermal neutron activity. Unless otherwise taken into account this will be interpreted as a lower water content than actually exists. Natural soils are in large part composed of the most common rock-forming elements listed in Table I. Their abilities to absorb thermal neutrons are compared in this table.

¹A contribution from the Division of Building Research, National Research Council, Ottawa, Canada, published with the approval of the Director of the Division.
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TABLE I
Capture cross sections for thermal neutrons of the most abundant soil elements

Element	Relative abundance		Capture cross section (barns)
	By weight (%)	By atom (%)	
1. Oxygen	*46.46	60.36	0.0016
2. Silicon	27.61	20.45	0.16
3. Aluminium	8.07	6.21	0.23
4. Iron	5.06	1.89	2.5
5. Calcium	3.64	1.89	0.43
6. Sodium	2.75	2.49	0.5
7. Potassium	2.58	1.37	2.2
8. Magnesium	2.07	1.77	0.4
9. Titanium	0.62	0.27	5.2
10. Hydrogen	0.14	2.89	0.3
11. Carbon	0.09	0.17	0.0045
12. Chlorine	0.05	0.02	33.0

*From Clarke (1924).

A particular soil may, of course, have any combination of these elements, devoid of some and replete with others. None has a high capture cross section for thermal neutrons when such elements as boron (700 barns), cadmium (2 500 barns), the rare earths, and others are considered. But among these elements chlorine is relatively effective in capturing neutrons and, if present in sufficient quantities in the form of salts, will cause an appreciable error in the measurement of soil moisture by the neutron method (Holmes 1956).

In the past, manufacturers of neutron moisture meters have made the assumption that a calibration curve, determined in one soil or a substitute medium, was valid for all soils but evidence is mounting that differences in absorption effects, too large to ignore, do exist between one soil type and another.

The author's experience has shown the existence of a very large discrepancy between laboratory and field calibration curves for a scintillation-type neutron meter. This may represent an extreme case but is, nevertheless, characteristic of the problem. Substitute soil media, carefully selected to avoid the effects of neutron absorption on the results, were used in the laboratory calibration (Burn 1960). Attempts to use this relationship in a natural deposit of post-glacial clay showed a large discrepancy when indicated values of moisture content were compared with those determined in the usual way (Burn 1964). Field calibration demonstrated an absorption effect that reduced detected neutron activity at every moisture content by about 11%.

Chlorine was immediately suspected but chemical analyses for this element in both the soil and pore water indicated only small quantities that could not have had any major effect on the results. Attention was then drawn to the larger than average capture cross sections exhibited by potassium and iron and it was reasoned that, if present in sufficiently large quantities, either of these might be the cause of the observed reduction in neutron activity.

While the results of more complete chemical analyses were being obtained, a pilot experiment, based upon an estimate of the quantity of potassium

present in the natural clay from a mineralogical analysis, was conducted with solutions of potassium carbonate to observe the reduction in counting rate with increase in concentration. The results indicated that the count rate would be reduced by 4% for a concentration equivalent to 2% by weight of the dry soil. Extrapolating to an estimated 5% by weight of potassium, the relationship, if valid, would account for the larger part of the observed discrepancy.

The results of chemical analyses, however, showed less than 1% of potassium present in the clay, but it did show about 7% of iron by weight. Consequently, the following experiments were conducted to assess the absorption effect of iron.

A substitute soil of activated bentonite, silica sand, and water was prepared in the ratio of 2:4:3 to provide a basic medium having a bulk density and range of moisture contents close to that of the natural soil. In these proportions the mixture has the consistency of a soft mortar, easily workable but sufficiently stiff to prevent segregation of the components. To this basic mix, increasing quantities of soluble ferric sulphate were blended thoroughly in a 20-gal pigment mixer.

In order not to produce an artificially high flux of thermal neutrons available for capture by iron, an attempt was made to maintain the ratio of iron content to water within the limits occurring naturally in the soil. (Seasonal moisture content changes cause this ratio to vary between 10 and 35%.)

The mixtures were placed in a 15-gal drum and, to avoid the inclusion of air, were made to flow using a small immersion-type concrete vibrator. When filled, an aluminium access tube of the type used in the field and in previous laboratory calibration (outside diameter $1\frac{1}{2}$ in., inside diameter $1\frac{3}{8}$ in.), was pushed down from the top along the vertical axis of the drum and secured in place. Readings with the effective center of the neutron probe positioned at each inch of depth were taken and plotted to determine the depths at which the readings were not affected by the boundaries of the medium. In each case, readings over a 3- or 4-in. interval near the center of the sample gave uniform results, indicating adequate size.

Densities were calculated from the weight of each sample and the volume it occupied in the previously calibrated container. The moisture in the soil was composed both of free water and that of crystallization so that it was necessary to determine water content by chemical analysis. Several samples were obtained from each prepared medium and the values averaged. Small variations of $\pm 0.5\%$ were found. As expected, the moisture contents varied slightly (28.00 to 33.25 lb/ft³) from one mixture to another, but it was possible to eliminate this variable by adjusting the readings according to the initial calibration curve. The results of chemical analyses also showed a quite uniform distribution of iron in each sample and the actual range for the five media from 0.8 to 9.9% by weight. These are plotted on Fig. 1 against observed reduction in slow neutron activity.

The reduction of activity at 7% iron by weight is about 9% and therefore

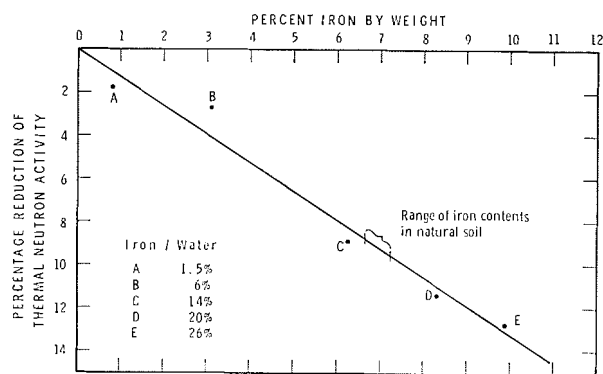


FIG. 1

accounts in large measure for the reduction previously observed in the field. The remaining 2% might easily be accounted for by the small quantities of potassium and chlorine also present in this soil.

On the basis of these experiments it is recommended that each neutron meter calibration curve be checked and, if necessary, adjusted for each soil type in which the instrument is used.

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SOIL MECHANICS SECTION,
DIVISION OF BUILDING RESEARCH,
NATIONAL RESEARCH COUNCIL,
OTTAWA, CANADA.