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16. ABSTRACT

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Experimental data were obtained by testing four soil-water systems with two nuclear moisture gages. The results indicate that it is satisfactory to assume that free water calibration curves differ by a constant, which depends on the sum of the effects of structural water and absorbing elements present. The slope of the nuclear moisture calibration curve is satisfactorily established by testing on reference standards of silica sand and water.

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THE CALIBRATION PROBLEM

Field Moisture Correlation

In the fall of 1967, research directed at development of calibration standards for nuclear moisture-density gages was undertaken by the California Division of Highways at the Materials and Research Laboratory, 5900 Folsom Blvd., Sacramento, California. Temporary moisture standards having high and low water contents were prepared by compacting a local river sand in molds cut from steel drums. Before compaction, the sand was allowed to come to air-dry equilibrium moisture. It was then compacted similarly in each of the tubs, and the bulk densities of both calculated.

One tub was constructed with a piezometer and a plastic tube leading to the bottom so that it could be saturated. This sample was flooded till the free water surface was at the sand surface, and all voids were essentially full of water. The amount of water added was measured, and checked by weighing the tub and contents before and after. The initial water content of the sand was estimated from oven-dry moisture samples and as a result, the total water content of the saturated moisture standard was known.

The tub containing air-dry sand with no further water added was used as a low moisture standard. Subsequent weighing of this sample indicated little change in moisture content. When it was torn down it was found that the top one to two inches was slightly drier than at greater depth. However, this had a negligible effect on the nuclear moisture tests taken on that sample.

When a calibration curve established on these standards was compared with one (for the same gage) developed using "oven-dry" field moistures, it was found that the two were displaced by about 3 lb. per cu.ft. of water. That is, the indicated water content of a site using the oven-dry calibration was lower than if the sand-water calibration were used. It was very evident however, that the estimated regression through the field data points was essentially parallel to the laboratory calibration. Since these were developed independently, it was concluded that the relationship between the two methods of calibration should be investigated further.

"Structural" Soil Water

It was concluded that the observed phenomenon was due to the presence of moisture, especially in the clay minerals, which was not removed in the oven-dry test. The nuclear moisture

gage response is proportional to the hydrogen contained in any form of soil water. This may include in addition to the free water: inter-layer water, hydrates, and hydroxyl water. It is easily demonstrated that all of these forms of "structural" soil water are not evaporated at temperatures of 105-110°C. For example, Nutting reports that the kaolin minerals lose an additional 14 percent by weight (approximately) of hydroxyl water between 400 and 800°C [1, p. 206], with the ensuing destruction of the kaolinite. The number of hydrogen atoms in hydroxyl water is one-half that of the corresponding quantity of free water molecules. Consequently, the nuclear moisture gage would respond to the free water equivalent of the hydroxyl water in kaolin; or, about 7 percent by weight.

Typical data for some clay minerals are summarized in Table 1.

TABLE 1

APPROXIMATE WATER LOSS OF VARIOUS MINERALS, 100 TO 800°C

Mineral	Temperature °C (100 – 200 – 300 – 400 – 500 – 600 – 700 – 800)	Structural Water Total (equiv) H_2O loss
Kaolin Halloysite	Little loss; Approx. 14% by wt. OH^- (equiv. H_2O loss = 7%)	7%
Montmorillonite Smectite	Approx 3% loss inter-layer H_2O; Approx 5% loss OH^- (equiv. H_2O = 2½%)	5½%
Vermiculite	Approx. 8% loss inter-layer, cation hydrates; Approx. 3% OH^- (equiv. H_2O = 1½%)	9½%
Illite (Mica group)	Gradual loss 2% (presumed H_2O); OH^- loss est. 4½% (equiv. H_2O approx. 2%)	4%
Chlorite	Little loss; OH^- loss approx. 12% (equiv. H_2O = 6%)	6%
Sepolite	Gradual loss to 10% (presumed H_2O); OH^-, gradual loss to 6% (equiv. H_2O = 3%)	13%

Clay mineral data estimated from Grim (8)

These are approximate average values showing the trend toward additional water loss at temperatures above 100°C. For the portion of this loss considered to be hydroxyl water, the equivalent free water (one-half) is tabulated.

Hydrates such as the zeolites and serpentines also contain significant amounts of water which are not driven off by temperatures of 105-110°C. Also, Lambe reported that a diatomaceous earth retained significant amounts of water at temperatures above 105°C[2]. This is apparently caused by the attractive forces of the very large internal surface area of this soil. This water should be similar in behavior to the bound water of the clay minerals.

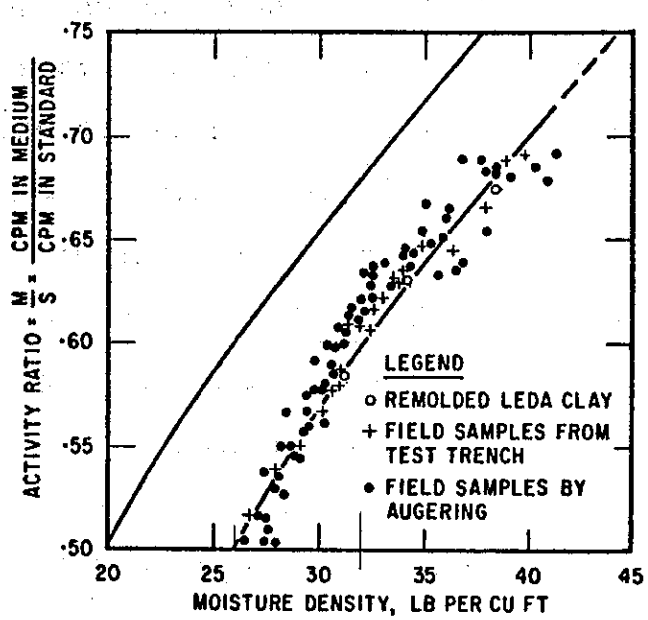
Thermal Neutron Absorption

In 1964, Burn published a report, "Calibration of a Neutron Moisture Meter in Leda Clay"[3]. Figure 1 is a reproduction of a graph from that paper showing moisture correlation data for a nuclear gage used on the Leda clay. Here, it is shown that the plot of nuclear readings versus oven-dry moisture contents of the soil is nearly 6 lbs. per cu.ft. to the right of the reference curve. This is to the opposite extreme from the California field calibration data. Burn's reference curve is based (in part) on silica sand and water standards having known

total water content which generally correspond to the temporary California Highway standards previously described.

Figure 1

CALIBRATION CURVE FOR LEDA CLAY



(BURN 3, 1964)

Burn speculated that the discrepancy between the two calibrations was due to the presence of iron in the clay which absorbed the neutron flux. He observed, as is apparent in the figure, that the displacement between the two curves appears to be a constant for that soil.

Summary

It has been shown that if the nuclear gage is calibrated by oven-dry moisture determinations, it can indicate a water content of field soils as much as plus or minus 6 lbs. per cu.ft. of water from that based on an inert system such as silica sand and water. However, it is the free soil moisture that must be controlled in earth construction. This has been shown to be a variable percentage of the total soil water to which the nuclear gage responds. Consequently, unless it can be demonstrated that a consistent relationship exists between a calibration based on standards of known water content, and the free water content of individual soils, the users of the gages are forced to calibrate by field correlation. This is difficult, and in some cases impossible to do, since a sufficient range of data is often not available to establish the slope of the curve with any degree of confidence.

EXPERIMENTAL PROCEDURE

The experimental procedure involved the careful fabrication, and testing with nuclear moisture gages, of four soil-water systems in large molds. They were:

1) A commercial silica sand
2) A commercial kaolin clay
3) A typical clayey field soil
4) An expanded shale

The approximate structural water content of the soils was determined on the basis of high temperature evaporative tests, mineral identification, and differential thermal analysis (DTA). Therefore, by exercising careful control of the amount of water added, both the free water and total (equivalent) water content of the individual samples were closely estimated.

The physical data concerning the dimensions, volumes, densities, and water contents of all samples tested with the nuclear moisture gages in the experimental program are presented in Table 2. The volumes of the samples were checked to insure that they were essentially infinite for testing with the gages.

TABLE 2

PHYSICAL DATA FOR THE EXPERIMENTAL SOIL-WATER SAMPLES

Sample	Physical Description				Density (lbs cu ft)		Water Content (lbs H_2O cu ft)			** Legend
	Shape	"D" (ft)	Depth (ft)	Volume (cu ft)	Wet	Dry	Free	Struct.*	Total	
A-70-W	Cylindrical	1.87	0.94	2.63	125.4	100.9	24.5	-	24.5	○
E-25-W	"	"	0.92	2.55	130.2	110.9	19.2	-	19.2	○
A-70-200-W	"	"	0.87	2.43	123.9	109.6	14.3	-	14.3	○
E-25-D	"	"	1.19	3.31	-	111.7	-	-	-	○
Kaolin-H	Octagon	1.50	1.17	2.31	97.1	77.9	19.2	4.7	23.9	△
Kaolin-L	Rectangular	1.00	1.50	2.25	80.1	70.7	9.4	4.2	13.6	△
Kaolin-H(dry)	Octagon	1.5	1.17	2.31	-	77.9	-	4.7	4.7	△
Field Soil-H	Octagon	1.50	1.17	2.31	146.0	131.9	14.1	2.0	16.1	□
Field Soil-L	Rectangular	1.00	1.50	2.25	137.4	129.8	7.6	2.0	9.6	□
Field Soil(dry)	Cylindrical	1.87	1.22	3.29	89.9	89.9	-	1.4	1.4	□
ES-20.6	Cylindrical	1.87	0.95	2.76	109.2	87.0	22.2	-	22.2	x
ES-17.4	"	"	0.94	2.64	94.4	77.0	17.4	-	17.4	x
ES-12.0	"	"	0.94	2.62	93.8	81.8	12.0	-	12.0	x
ES-6.0	"	"	1.14	3.27	88.2	82.1	6.1	-	6.1	x
ES-(dry)	"	"	1.19	3.29	88.6	88.2	0.4	-	0.4	x

* Equivalent water content.
** As used in Figures 2 and 3.
NOTE: The dimension "D" is the least top surface dimension.

The top surfaces of most of the samples were protected by covering them with a layer of polyethylene plastic. It was determined that the plastic had a negligible effect on the testing by taking readings with and without the covering.

Silica Sand Samples

Silica sands from two sources were obtained, which were 99.7 to 99.9 percent pure SiO_2. The approximate grading of the sands

used is given in Table 3.

TABLE 3

SILICA SAND GRADING

Ident.	E-25	E-85	#200	A-70
Fineness	25.88	84.42	---	65
Sieve	(Percent Retained)			
20	2.2			
30	38.6			
40	57.6	0.2		0.6
50	1.4	2.4		3.9
60				9.3
70	0.2	10.0		20.5
80				22.1
100		45.0		31.2
140		20.0	1.0	11.6
200		11.0	4.0	0.7
270		2.4	6.0	
325		1.0	9.0	

Four samples were fabricated from these sands. They were:

1) A-70-W. This sample was compacted dry in approximately 2 in. lifts with an electric impact hammer using a plywood disc to confine the sand. The sample was provided with a piezometer and a perforated plastic tube so that it could be saturated from within. The (W) in the notation indicates a "wet" or saturated sample.

2) E-25-W. This sample was prepared similarly, except

that the #25 sand was used.

3) A-70-200-W. A ground silica sand (#200 flour) was uniformly mixed with the #70 sand to reduce the void space, so that a lower saturated water content was obtained.

4) E-25-D. The dry silica sand standard (D) was fabricated with the #25 sand. The moisture content of this sand when initially compacted was hardly detectable. After exposure to the atmosphere in the test area for a period of several months, it gained moisture of up to 0.2 percent.

An attempt was also made to fabricate a partially saturated sample, by compacting the moistened sand in increments. Polyethylene plastic was used to prevent migrations of water. However, it was found that sufficient water movement occurred in the top layer to affect the gage readings, which made this type of specimen unsatisfactory.

Kaolin Samples

A kaolin clay of high purity was obtained from Ione, California. The physical properties and chemical analysis given by the

producer are shown in Table 4.

TABLE 4

IONE KAOLIN, PHYSICAL AND CHEMICAL DATA

Physical Properties:	
Specific Gravity	2.60
Moisture (max.) %	0.5 - 1.0
Particle Size, %	
Minus 2 microns	50 - 55
Plus 5 microns	20 - 25
pH	4.5 - 5.5
Water of Plasticity, %	31.9
Average Particle Size	2.2 microns
Chemical Analysis:	
Silica, %	45.0 - 47.0
Alumina, %	37.5 - 39.0
Iron Oxide, %	0.3 - 0.5
Titanium Dioxide, %	1.5 - 2.0
Ignition Loss, %	13.0 - 14.0

An independent analysis of a sample of this clay using X-ray diffraction and differential thermal analysis (DTA), was conducted at the Materials and Research Laboratory. It was confirmed that the clay was a typical kaolin, with little or no indication of the presence of other clay minerals.

High-temperature evaporation tests were conducted with oven-dry samples of the Ione kaolin. These samples lost 0.1 percent by weight between 105 and 400°C, and 12.2 percent by weight between 400 and 600°C. This loss in weight was considered to be primarily hydroxyl water, and an equivalent

water loss between 105 and 600°C of 6 percent was presumed for the purposes of the study.

Two samples of the kaolin were then prepared in large molds for testing. The dimensions and volumes of the samples are given in Table 2. These two samples were prepared similarly, except that different amounts of free water were added to each. The intent was to obtain as high (H) and low (L) a water content as possible, with uniform compaction, and not too great a difference in the dry density of the two. The amount of free water added was estimated on the basis of preliminary testing and representative oven-dry samples of the clay taken during compaction. The water contents of the samples were also checked when dismantling them after completion of the testing. One of the kaolin samples (the Kaolin-H) was dried in a 105-110°C oven, and then retested with the nuclear moisture gages.

Field Soil Samples

Two samples of a typical field soil were prepared and tested similarly at a high and low free water content. A third field soil sample was fabricated by compacting the material in an oven-dry condition. The grading and chemical data for the soil are given in the following Table 5.

High-temperature evaporation tests were also conducted on oven-dry samples of the field soil. A loss of 0.5 percent by weight was recorded between 105 and 400°C, and 2.2 percent between 400 and 600°C. The loss in weight between 105 and 600°C was assumed to indicate an equivalent water content of 1.5 percent by weight.

TABLE 5

FIELD SOIL, GRADING AND CHEMICAL DATA

Grading		Mineral Identification	
Size	% Passing	Mineral	*Approx. %
3/4	100	Quartz	25 - 30
1/2	84	Feldspar	10 - 15
3/8	73	Chlorite	10 - 15
#4	52	Mica	5
8	41	Hornblende	5
16	31	Misc. Clay	5 - 10
30	23	Talc	2 - 3
50	18	Augite	2 - 3
100	15	Other Xtaline	5
200	13	Amorphous	15
5 micron	8		
1 micron	6		

*These percentages are estimated from X-ray diffraction data.

Expanded Shale-Sand Samples

Five samples were prepared of an expanded shale-sand. This material is manufactured by expanding sand-sized particles of montmorillonite shale by heating in a rotary kiln to temperatures of approximately 2,000°F. The grading of the expanded shale-

sand, and the manufacturer's chemical analysis is given in Table 6.

TABLE 6

EXPANDED SHALE-SAND, GRADING & CHEMICAL ANALYSIS

Sieve	Percent Passing (Basalt Rock Co.)	(M. & R.)
#4	99	
8	82	82.6
12		52.6
16	56	
30	36	33.3
50	23	22.1
100	12	13.5
200		6.5

	Chemical Analysis "Raw" Shale	Calcined Shale
SiO_2	59.2	62.8
Al_2O_3	20.0	21.7
CaO	2.6	3.2
Fe_2O_3	3.3	4.4
MgO	2.7	3.3
Na_2O	2.6	2.6
K_2O	1.6	1.6
H_2O	8.0	0.0
Ignition Loss	Trace	0.4

One of the expanded shale-sand samples (ES-20.6, Table 2) was prepared by compacting, and then flooding it until it was saturated. The (ES-dry) sample was left in a dry or "as compacted" state. The remaining three expanded shale-sand samples were prepared by moistening the shale-sand to intermediate water contents, and then compacting. Due to the absorbent nature of the

calcined shale, the procedure appeared to work satisfactorily, with little discernible migration of water during the period of testing. The water contents of several of the shale-sand samples were checked by removing the material in layers, and oven drying the entire increments.

NUCLEAR MOISTURE DATA

The soil samples were not necessarily fabricated or tested with the nuclear gages in the order presented, nor was it possible to retain all samples so that they could be tested concurrently.

To compensate, as each new sample was being tested, all available samples were read. The average of these readings was then plotted against sample free water in Figure 2 and against the estimated total water content in Figure 3.

Two nuclear soil gages were used during this study. Gage A is an older unit that has been found to be relatively stable over an extended period of time. The source contained in this gage is 4.5 mc Radium 226-Beryllium. Gage B is of more recent manufacture, and contains a 50 mc Americium 241-Beryllium fast neutron source.

Free Water Correlation Data

Figure 2 illustrates the response of Gages A and B plotted against the free water content of the samples. The curves are drawn by visual "best fit". For Gage A, the calibration curves for the four soil-water systems are essentially parallel, except for the expanded shale at the lower water contents. The readings taken with Gage B indicate a greater variation

FIGURE 2

NUCLEAR FREE MOISTURE CORRELATION

TEST DATA

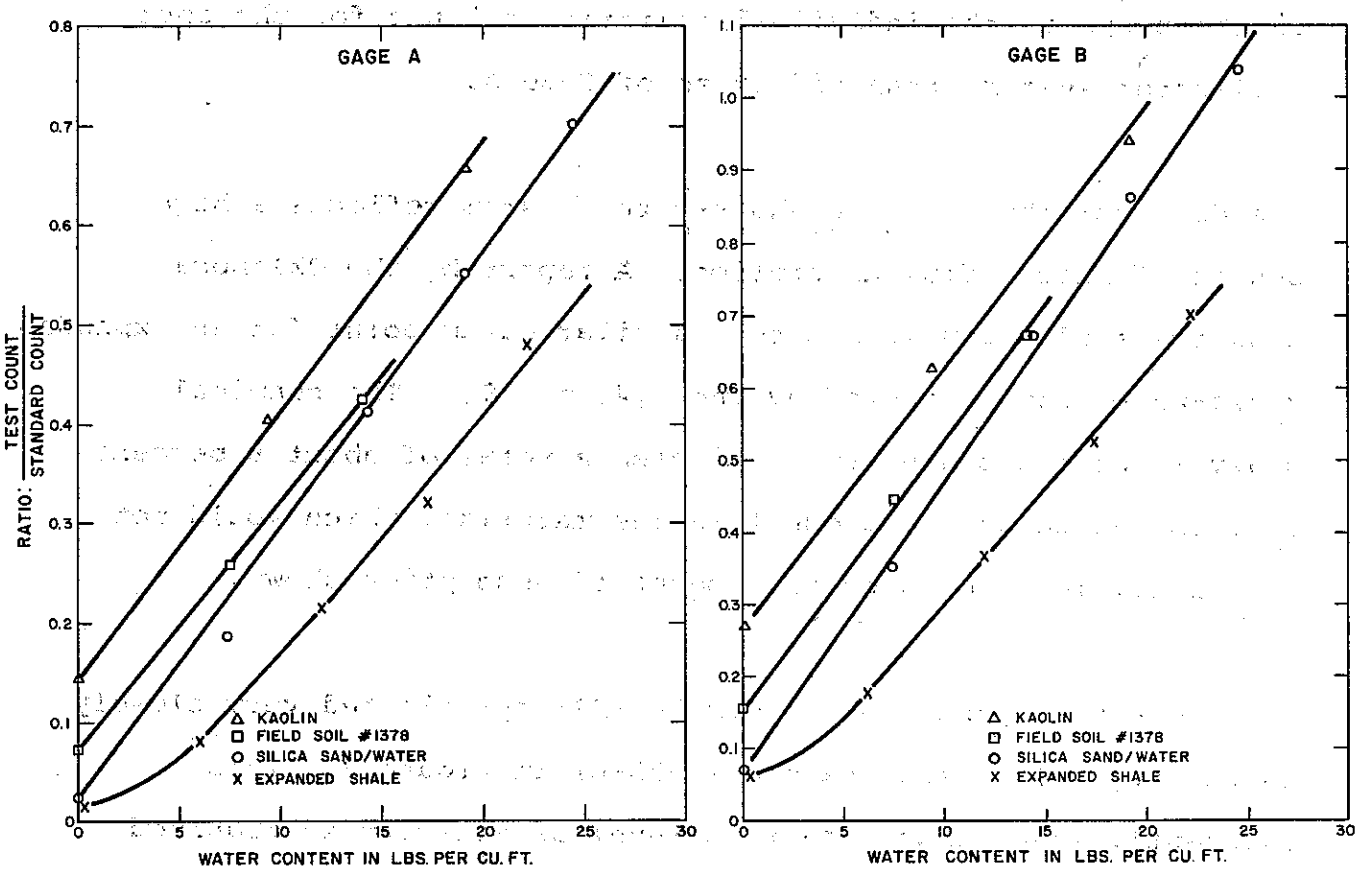
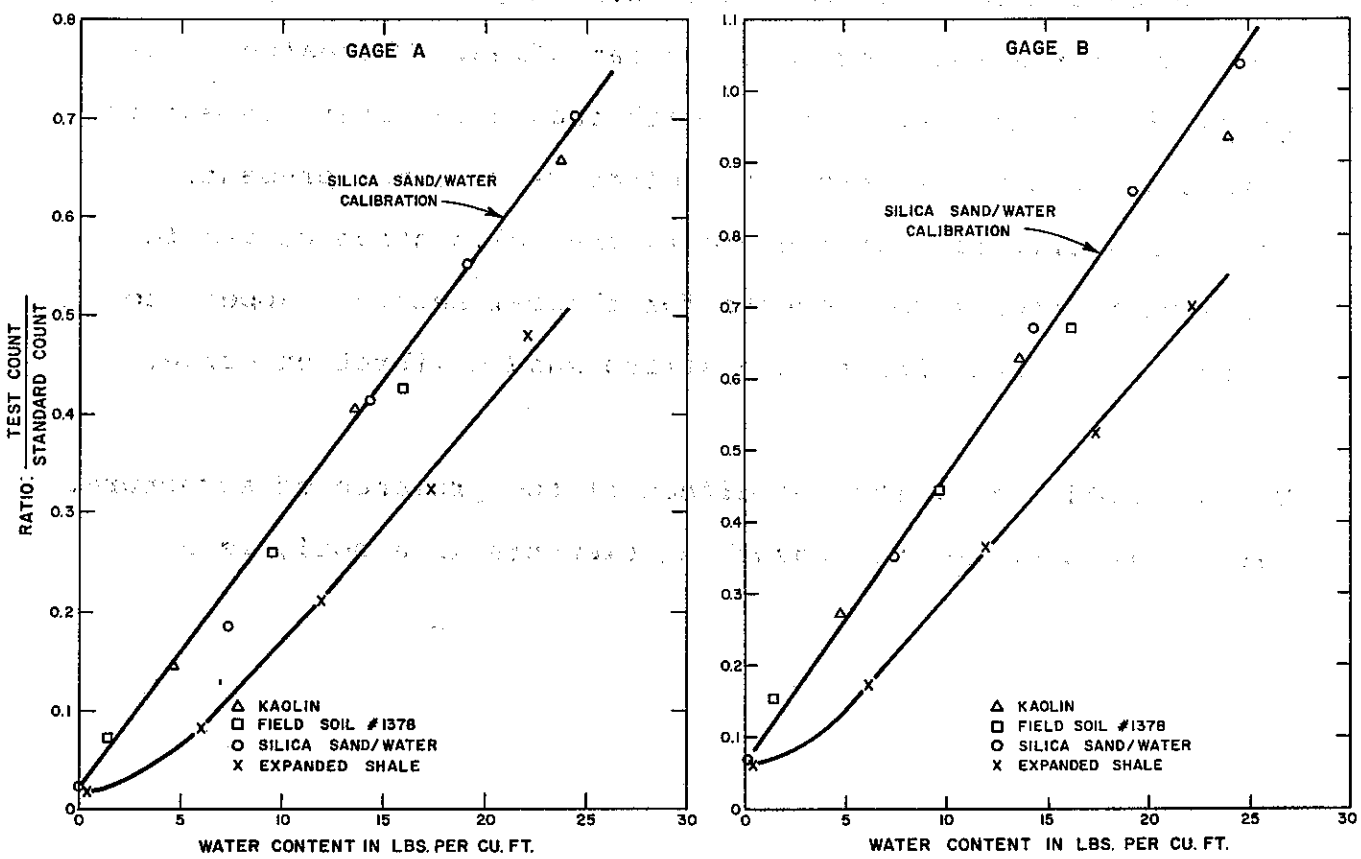


FIGURE 3

NUCLEAR TOTAL MOISTURE CORRELATION

TEST DATA



in the slope of the individual calibration lines for the four soil-water systems than did those of Gage A.

The data for the expanded shale-water system reflects a high degree of nutron flux absorption. A report by the Oklahoma Department of Highways showed a similar datum point for an expanded shale-water sample tested by them [4, p. 89]. The chemical analysis of the shale-sand indicates a total of about 6 percent of iron and potassium present in the material, which would not seem sufficient to cause the amount of absorption shown.

The oven-dry expanded shale sample (ES-dry) plotted very closely to the dry silica sand sample (E-25-D) in Figure 2. This apparently means that a certain level of activity is required before the capacity of the material to absorb thermal neutrons becomes saturated. At some water content, this absorptive capacity of the soil begins to be satisfied. Thereafter, the production of thermal neutrons with change in water content is almost as though the absorptive elements were not present. Above this critical water content, the calibration curves for soils with absorptive elements for thermal neutrons appear to be reasonably parallel to the silica sand calibration curve.

The assumption that the net effect of the presence of structural water, and slow neutron absorbing elements in a soil, is a

calibration constant; is undoubtedly an over-simplification. However, it is concluded that the slope of a calibration curve determined using standards of silica sand and water should be satisfactory for nuclear gage moisture determinations on most soils. An exception may have to be made for those containing highly absorbtive elements, especially at water contents below 6 lbs. of water per cu. ft. It is not known to what degree such materials may be encountered in soils used in highway construction.

Total Water Correlation Data

Figure 3 shows the average nuclear gage readings plotted with respect to the estimated total water content of the samples. Most of the moisture data for the clay soils now plot either on, or to the right of the silica sand calibration curve. It is concluded that nuclear moisture measuring instruments do respond to the structural water, as well as to the free water. However, the effect of the hydroxyl water does not appear to be directly proportional to the hydrogen content. This is to be expected, since the bonding of the hydrogen atom to the soil minerals probably reduces its capacity to thermalize the fast neutrons emitted by the radioisotope in the gage [5, p. 548; 6, p. 347].

CONCLUSIONS

I. The nuclear moisture gages respond to the total water content of a soil. This includes bound or adsorbed water, inter-layer water, hydrates, and hydroxyl water. A water content estimated on the basis of an oven-dry moisture determination reflects only the free, or easily evaporable water.

II. For a given water content, the presence of slow neutron absorbing elements will decrease the response of the nuclear moisture gage. Except at low water contents however, the presence of the absorbing elements appears to be a calibration constant.

III. The change in soil water during normal construction activities occurs almost entirely in the soil free water. The results of this research indicate that it is satisfactory to assume that the free water calibration curves differ only by a constant, which depends on the sum of the effects of the structural water and absorbing elements present.

IV. The slope of the nuclear moisture calibration curves is satisfactorily established by testing on reference standards of silica sand and water.

IMPLEMENTATION

"Offset" Method

For specific soils, where an equivalent oven-dry moisture calibration curve for the nuclear gage is desired, a moisture sample is taken at the site of a nuclear moisture reading. The density of the location must be determined by nuclear or other means. The oven-dry moisture in lbs. per cu. ft. of water is calculated, and plotted against the nuclear gage moisture reading. A calibration curve for that soil is drawn through the point and parallel to the sand-water reference calibration.

This procedure has two disadvantages: 1) It is only good for the particular soil and 2) any errors in the estimates of moisture or density are reflected in the calculated moisture. An alternate and prefered method is discussed as follows.

"Approximate Wet-Weight" Method

It can be shown that a practical method of compaction control is possible, utilizing only a wet density correction based on the slope of the moisture calibration curve. The procedure is especially applicable to structural section materials, for which it is often possible to pre-establish a satisfactory

compaction standard. The concept is similar to that of the so-called "wet-weight" method of compaction control used by California [7, Calif. 216-F] and others. This procedure is predicated on expressing the wet density of a soil, and the laboratory control density, in terms of the same moisture content. The following contains a slight approximation, but the percent relative compaction is usually within one-half of one percent of that calculated on the basis of the "exact" wet or dry weight methods.

As an example, suppose that it has been established that a certain soil should have a wet density of 135 lbs. per cu. ft. at some definite (but unknown) water content. This unknown water content is that contained in the material when it was obtained for the laboratory compaction testing. Assume also that a nuclear moisture gage had been used to test the location where the soil was obtained, at the time of removal, and it was found to have a water content of (x). At a later time, a site with similar soil is tested, and found to have a wet density of 140 lbs. per cu. ft. with (x+3) water content. The wet density of the site in terms of the water content at which the control had been established is (140 - 3), or 137 lbs. per cu. ft. This equivalent wet density is greater than the 135 lbs. per cu. ft. previously established as a minimum control density. Thus, if the slope of the nuclear moisture calibration curve is correct, it has been found that the density of the site is satisfactory without knowing its actual water content.

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