

NUCLEAR SOIL-MOISTURE CORRELATION

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Nuclear soil-moisture gauges are commonly used to control the compaction of soil. The nuclear moisture test depends on the moderation, or slowing down, of fast neutrons (emitted from a radioisotope contained in the gauge) by the hydrogen in the soil water. The free or easily evaporable soil water must be controlled in earth construction. However, different soils have varying amounts of other forms of water such as hydrates, interlayer water, and hydroxyl water. Those are not easily evaporable but are detected by the gauge. Also, some soils contain elements that absorb the slow neutrons. This reaction may affect the nuclear moisture test. Consequently, a method must be found of adjusting a gauge calibration determined in the laboratory so that it may be used with the gauge in the field to test a variety of soils. The experimental data were obtained by testing (with 2 nuclear moisture gauges) 4 soil-water systems compacted in large molds. They were prepared at various water contents and included a silica sand, a kaolin clay, a clayey field soil, and an expanded shale. The results of this research indicate that it is satisfactory to assume that the free water calibration curves for various soils differ by a constant that depends on the sum of the effects of structural water and absorbing elements present. The slope of the nuclear moisture calibration curves is satisfactorily established by testing on reference standards of silica sand and water.

•IN THE FALL of 1967, research directed at development of calibration standards for nuclear moisture-density gauges was undertaken by the California Division of Highways. 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 were 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 that had no further water added was used as a low-moisture standard. Subsequent weighing of that sample indicated little change in moisture content. When it was torn down, the top 1 to 2 in. was found to be slightly drier than the greater depths. However, that had a negligible effect on the nuclear moisture tests taken on that sample.

When a calibration curve established by those standards was compared with one (for the same gauge) developed by the use of "oven-dry" field moistures, the two were displaced by about 3 lb/ft³ of water. That is, the indicated water content of a site was lower when the oven-dry calibration was used than when the sand-water calibration was used. It was very evident, however, that the estimated regression through the field data points was essentially parallel to the laboratory calibration. Because these were developed independently, the conclusion was that the relation between the 2 methods of calibration should be investigated further.

"STRUCTURAL" SOIL WATER

The conclusion was 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 gauge response is proportional to the hydrogen contained in any form of soil water. In addition to the free water, that may include interlayer water, hydrates, and hydroxyl water. That all of those forms of "structural" soil water are not evaporated at temperatures of 105 to 110 C is easily demonstrated. 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 half that of the corresponding quantity of free water molecules. Consequently, the nuclear moisture gauge would respond to the free water equivalent of the hydroxyl water in kaolin (or about 7 percent by weight).

Typical data (estimated from Grim, 8) for some clay minerals are shown in Figure 1. The values are approximate average values and show the trend toward additional water loss at temperatures above 100 C. For the portion of that loss considered to be hydroxyl water, the equivalent free water (half) is given.

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

THERMAL NEUTRON ABSORPTION

Figure 2 shows moisture correlation data for a nuclear gauge used on Leda clay (3). Nuclear readings versus oven-dry moisture contents of the soil are nearly 6 lb/ft³ to the right of the reference curve. This is 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; those standards generally correspond to the temporary California highway standards previously described. Burn speculated that the discrepancy between the 2 calibrations was due to the presence in the clay of iron, which absorbed the neutron flux. He observed, as is apparent in the figure, that the displacement between the 2 curves appears to be a constant for that soil.

DISCUSSION OF CALIBRATION PROBLEM

It has been shown that, if the nuclear gauge is calibrated by oven-dry moisture determinations, it can indicate a water content of field soils as much as ± 6 lb/ft³ of water from that based on an inert system such as silica sand and water. However, free soil moisture must be controlled in earth construction, and that has been shown to be a variable percentage of the total soil water to which the nuclear gauge responds. Consequently, unless it can be demonstrated that a consistent relation exists between a calibration based on standards of known water content and free water content of individual soils, the users of the gauges are forced to calibrate by field correlation. That is difficult and, in some cases, impossible to do because 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 gauges of 4 soil-water systems in large molds: commercial silica sand, commercial kaolin clay, typical clayey field soil, and 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, the exercise of careful control of the amount of water added made it possible to closely estimate both the free water and total (equivalent) water content of the individual samples.

The physical data concerning the dimensions, volumes, densities, and water contents of all samples tested with the nuclear moisture gauges in the experimental program are given in Table 1. The volumes of the samples were checked to ensure that they were essentially infinite for testing with the gauges.

The top surfaces of most of the samples were protected by a layer of polyethylene plastic. Readings taken with and without the covering indicated that the plastic had a negligible effect on the testing.

Silica Sand Samples

Silica sands obtained from 2 sources were 99.7 to 99.9 percent pure SiO_2 . The approximate grading of the sands used is given in Table 2. Four samples were fabricated:

1. Sample A-70 was compacted dry in approximately 2-in. lifts with an electric impact hammer; a plywood disk was used to confine the sand. The sample was provided with a piezometer and a perforated plastic tube so that it could be saturated from within.

2. Sample E-25 was prepared similarly, except that No. 25 sand was used.

3. For sample A-70-200, a ground silica sand (No. 200 flour) was uniformly mixed with No. 70 sand to reduce the void space so that a lower saturated water content was obtained.

4. In sample E-25, the dry silica sand standard was fabricated with No. 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 as much as 0.2 percent moisture.

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, sufficient water movement occurred in the top layer to affect the gauge readings and 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, according to the producer, are given in Table 3.

An independent analysis of a sample of this clay was made by X-ray diffraction and DTA at the Materials and Research Laboratory. The analysis confirmed that the clay was a typical kaolin and gave 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. The samples lost 0.1 percent by weight between 105 and 400 C and 12.2 percent by weight between 400 and 600 C. The 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 similarly prepared in large molds for testing, except that different amounts of free water were added to each. The intent was to obtain as high and as low 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 they were dismantled after the testing was completed. One of the samples (kaolin high) was dried in a 105 to 110 C oven and then retested with the nuclear moisture gauges.

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 Table 4.

Figure 1. Approximate water loss of various minerals.

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

Figure 2. Calibration curve for Leda clay.

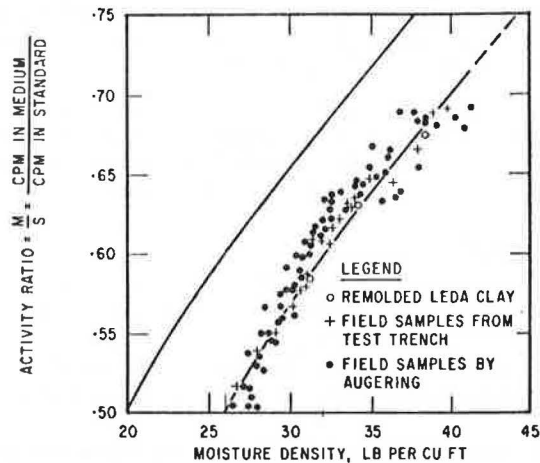


Table 1. Physical data for experimental soil-water samples.

Soil-Water System	Sample	Shape	Least Top Surface Dimension (ft)	Depth (ft)	Volume (ft ³)	Density (lb/ft ³)		Water Content (lb/ft ³)		
						Wet	Dry	Free	Struct.*	Total
Silica sand	A-70 wet	Cylindrical	1.87	0.94	2.63	125.4	100.9	24.5	—	24.5
	E-25 wet	Cylindrical	1.87	0.92	2.55	130.2	110.9	19.2	—	19.2
	A-70-200 wet	Cylindrical	1.87	0.87	2.43	123.9	109.6	14.3	—	14.3
	E-25 dry	Cylindrical	1.87	1.19	3.31	—	111.7	—	—	—
Kaolin clay	High	Octagon	1.50	1.17	2.31	97.1	77.9	19.2	4.7	23.9
	Low	Rectangular	1.00	1.50	2.25	80.1	70.7	9.4	4.2	13.6
	High, dry	Octagon	1.5	1.17	2.31	—	77.9	—	4.7	4.7
Field soil	High	Octagon	1.50	1.17	2.31	146.0	131.9	14.1	2.0	16.1
	Low	Rectangular	1.00	1.50	2.25	137.4	129.8	7.6	2.0	9.6
	Dry	Cylindrical	1.87	1.22	3.29	89.9	89.9	—	1.4	1.4
Expanded shale	ES-20.6	Cylindrical	1.87	0.95	2.76	109.2	87.0	22.2	—	22.2
	ES-17.4	Cylindrical	1.87	0.94	2.64	94.4	77.0	17.4	—	17.4
	ES-12.0	Cylindrical	1.87	0.94	2.62	93.8	81.8	12.0	—	12.0
	ES-6.0	Cylindrical	1.87	1.14	3.27	88.2	82.1	6.1	—	6.1
	ES dry	Cylindrical	1.87	1.19	3.29	88.6	88.2	0.4	—	0.4

*Equivalent water content.

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 500 C was assumed to indicate an equivalent water content of 1.5 percent by weight.

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 in a rotary kiln heated to temperatures of approximately 2,000 F. The grading of the expanded shale-sand and the manufacturer's chemical analysis are given in Table 5.

Sample ES-20.6 was prepared by compacting and then flooding until it was saturated. The ES-dry sample was left in a dry or "as-compacted" state. The remaining 3 samples were prepared by moistening the shale-sand to intermediate water contents and then compacting. Because of the absorbent nature of the calcined shale, the procedure appeared to work satisfactorily, and little migration of water was discerned 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 gauges in the order presented, nor was it possible to retain all samples so that they could be tested concurrently. That was compensated for by reading all available samples as each new sample was being tested. The average of those readings was then plotted against sample free water (Fig. 3) and against the estimated total water content (Fig. 4).

Two nuclear soil gauges were used during this study. Gauge A is an older unit that has been found to be relatively stable during an extended period of time. The source contained in this gauge is 4.5-mC radium 226-beryllium. Gauge B is of more recent manufacture and contains a 50-mC americium 241-beryllium fast neutron source.

Free Water Correlation Data

Figure 3 shows the response of gauges A and B plotted against the free water content of the samples. The curves are drawn by visual "best fit." For gauge A, the calibration curves for the 4 soil-water systems are essentially parallel, except for the expanded shale at the lower water contents. The readings taken with gauge B indicate a greater variation in the slope of the individual calibration lines.

The data for the expanded shale-water system reflect a high degree of neutron flux absorption. A report by the Oklahoma Department of Highways showed a similar datum point for an expanded shale-water sample tested (4, p. 89). The chemical analysis of the shale-sand indicates the presence of about 6 percent of iron and potassium in the material; that amount does 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) (Fig. 3). 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 that 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.

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 oversimplification. However, it is concluded that the slope of a calibration curve determined by using standards of silica sand and water should be satisfactory for nuclear gauge moisture determinations on most soils. An exception may have to be made for those containing highly absorptive elements, especially at water contents below 6 lb/ft³ of water. It is not known to what degree such materials may be encountered in soils used in highway construction.

Table 2. Grading for silica sand.

Item	E-25	E-85	No. 200	A-70
Fineness	25.88	84.42	—	65
Sieve, percent retained				
20	2.2			
30	38.6			
40	57.6	0.0		06
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	

Table 3. Physical and chemical data for lone kaolin.

Property	Value
Physical	
Specific gravity	2.60
Moisture (max), percent	0.5 to 1.0
Particle size, percent	
-2 μm	50 to 55
+5 μm	20 to 25
pH	4.5 to 5.5
Water of plasticity, percent	31.9
Average particle size, μm	2.2
Chemical	
Silica, percent	45.0 to 47.0
Alumina, percent	37.5 to 39.0
Iron oxide, percent	0.3 to 0.5
Titanium dioxide, percent	1.5 to 2.0
Ignition loss, percent	13.0 to 14.0

Table 4. Grading and chemical data for field soil.

Item	Value ^a	Item	Value ^a
Sieve, percent passing		Mineral, percent	
3/4	100	Quartz	25 to 30
1/2	84	Feldspar	10 to 15
3/4	73	Chlorite	10 to 15
4	52	Mica	5
8	41	Hornblende	5
16	31	Miscellaneous clay	5 to 10
30	23	Talc	2 to 3
50	18	Augite	2 to 3
100	15	Other crystalline	5
200	13	Amorphous	15
5 μm	8		
1 μm	6		

^aEstimated from X-ray diffraction data.

Table 5. Grading and chemical data for expanded shale-sand.

Item	Value
Sieve, percent passing	
4	99
8	82
12	52.6
16	56
30	36
50	23
100	12
200	6.5
Chemical	
	Basalt Rock Co.
	M and R
	Raw Shale
	Calcined Shale
SiO ₂	59.2
Al ₂ O ₃	20.0
CaO	2.6
Fe ₂ O ₃	3.3
MgO	2.7
Na ₂ O	2.6
K ₂ O	1.6
H ₂ O	8.0
Ignition loss	Trace

Figure 3. Nuclear free moisture correlation test data.

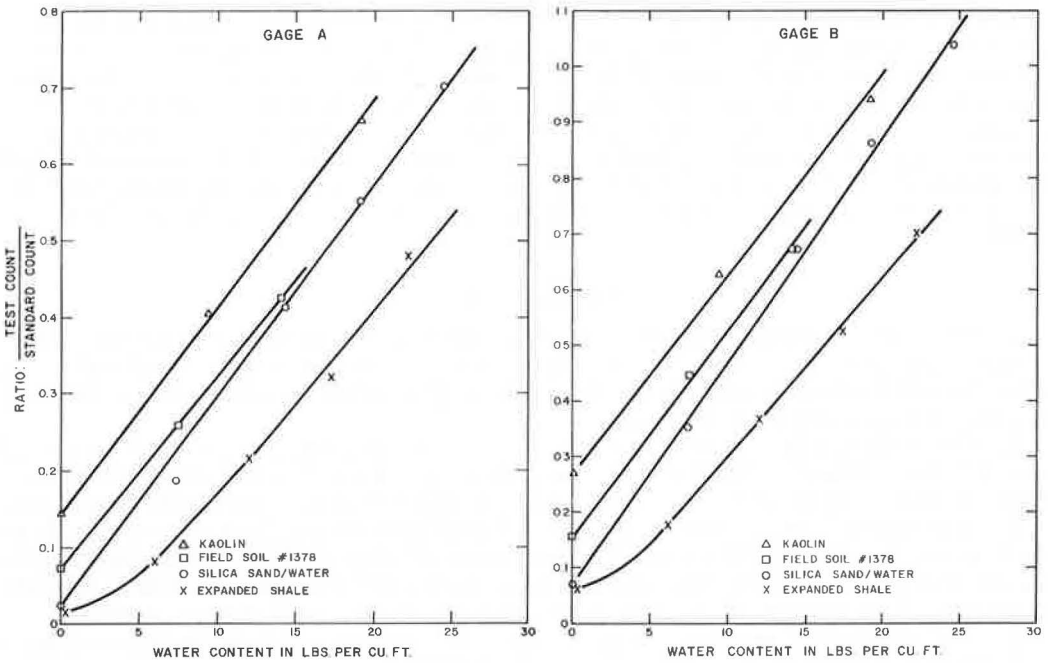
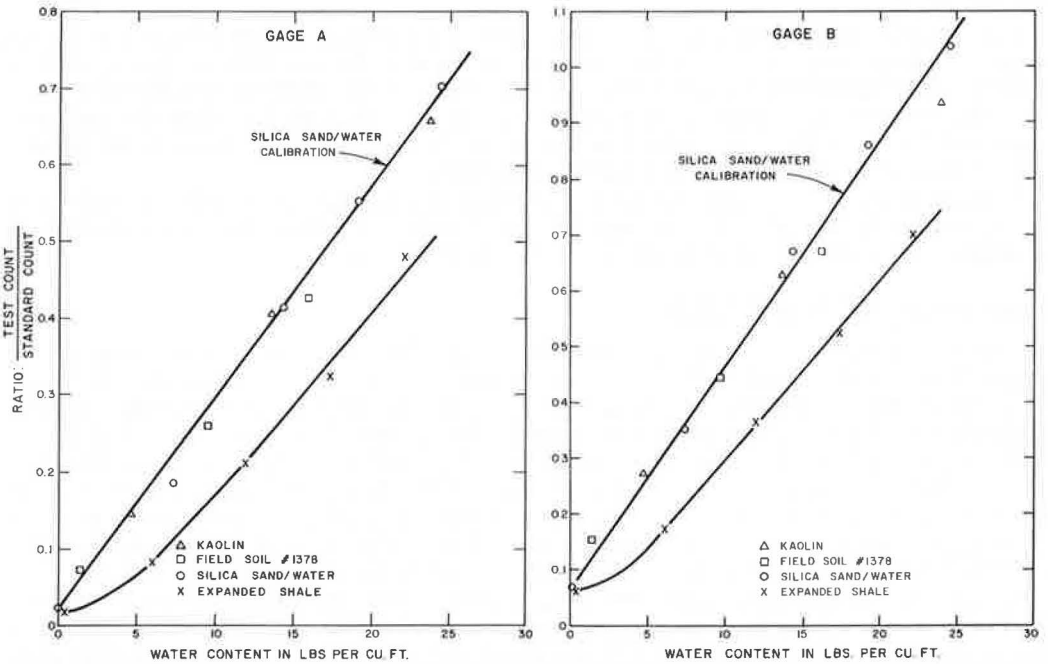


Figure 4. Nuclear total moisture correlation test data.



Total Water Correlation Data

Figure 4 shows the average nuclear gauge 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 instruments for measuring moisture 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 because 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 gauge (5, p. 548; 6, p. 347).

CONCLUSIONS

1. The nuclear moisture gauges respond to the total water content of a soil. That includes bound or adsorbed water, interlayer 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.
2. For a given water content, the presence of slow neutron-absorbing elements will decrease the response of the nuclear moisture gauge. Except at low water contents, however, the presence of the absorbing elements appears to be a calibration constant.
3. The change in soil water during normal construction activities occurs almost entirely in the 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.
4. 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 gauge 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 lb/ft^3 of water is calculated and plotted against the nuclear gauge moisture reading. A calibration curve for that soil is drawn through the point and parallel to the sand-water reference calibration.

This procedure has 2 disadvantages: (a) It is only good for the particular soil, and (b) any errors in the estimates of moisture or density are reflected in the calculated moisture. An alternate and preferred method is discussed.

"Approximate Wet-Weight" Method

A practical method of compaction control is possible; it uses 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 "wet-weight" method of compaction control used by California (7, 216-F) and others. This procedure is based on expressing the wet density of a soil and the laboratory control density in terms of the same moisture content. The following example contains a slight approximation, but the percentage of relative compaction is usually within 0.5 percent of that calculated on the basis of the "exact" wet- or dry-weight method.

Suppose that it has been established that a certain soil should have a wet density of 135 lb/ft^3 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. Suppose also that a nuclear moisture gauge had been used to test the location where the soil was obtained at the time it was removed, and the water content was x . At a later time, a site with similar soil is tested and found to have a wet density of 140 lb/ft^3 and water content of $x + 3$. The wet density of the site in terms of the water content at

which the control had been established is 140 - 3 or 137 lb/ft³. This equivalent wet density is greater than the 135 lb/ft³ 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 its actual water content being known.

ACKNOWLEDGMENTS

This work was accomplished in cooperation with the Federal Highway Administration. Appreciation is extended to Dale Sathre and Frank Champion, who performed the many and sometimes laborious technical operations with care and precision. The contents of this report reflect the views of the author who is responsible for the facts and the accuracy of the data presented. The contents do not necessarily reflect the official views or policies of the California Division of Highways or of the Federal Highway Administration; nor does the report constitute a standard, specification, or regulation.

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