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16. ABSTRACT

Control of today's earth construction requires an increased capacity for testing field densities. The use of nuclear moisture and density gages, despite certain deficiencies, offers a potential in this direction. For example, the California Division of Highways has about 140 of these units in use in construction control testing at the present time (1, Calif. 231-D).

However, there appears to exist a considerable difference of opinion concerning the calibration of the gages for soil moisture. The Oklahoma Department of Highways has indicated that a single calibration curve, based on laboratory standards fabricated of various aggregates and water, is satisfactory (2, 1968). The Indiana State Highway Commission, in cooperation with Purdue University, also reported that a single calibration is suitable for most soils (3, 1967), and suggested that calibration be done in the field.

Ballard and Gardner on the other hand, imply that individual calibrations may be required for different soils (4, 1965). They propose that non-soil laboratory standards be employed in conjunction with a mathematical regression using soil parameters determined experimentally. Burn (5,6,7, 1960-64) reports the use of laboratory silica sand-water standards (and other materials), for the calibration of the nuclear gages. He shows that if the silica sand-water calibration curve were used with a nuclear moisture gage to test a certain clay; the indicated water content would be 6 lbs of water per cu ft less than that determined on the basis of the oven-dry moisture test. 1 In a preliminary investigation by the California Division of Highways, it was found that if a similar sand-water calibration were used on a different clay, the indicated water content could be as much as 6 lbs per cu ft greater than that calculated on the basis of the oven-dry moisture test.

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Nuclear Soil Moisture Correlation

Robert E. Smith

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NUCLEAR SOIL MOISTURE CORRELATION

Robert E. Smith

Robert E. Smith
B.C.E. University of Florida, Gainesville, 1960

THESIS

Submitted in partial satisfaction of
the requirements for the degree of

MASTER OF SCIENCE

in

Civil Engineering

AT THE

SACRAMENTO STATE COLLEGE

Approved:

_____, Chairman

_____
Advisory Committee

Date_____

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The opinions, findings, and conclusions expressed in this paper are those of the writer; and not necessarily those of the California Division of Highways, the Bureau of Public Roads, or Sacramento State College

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INTRODUCTION

Control of today's earth construction requires an increased capacity for testing field densities. The use of nuclear moisture and density gages, despite certain deficiencies, offers a potential in this direction. For example, the California Division of Highways has about 140 of these units in use in construction control testing at the present time (1, Calif. 231-D).

However, there appears to exist a considerable difference of opinion concerning the calibration of the gages for soil moisture. The Oklahoma Department of Highways has indicated that a single calibration curve, based on laboratory standards fabricated of various aggregates and water, is satisfactory (2, 1968). The Indiana State Highway Commission, in cooperation with Purdue University, also reported that a single calibration is suitable for most soils (3, 1967), and suggested that calibration be done in the field.

Ballard and Gardner on the other hand, imply that individual calibrations may be required for different soils (4, 1965). They propose that nonsoil laboratory

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standards be employed in conjunction with a mathematical regression using soil parameters determined experimentally. Burn (5, 6, 7, 1960-64) reports the use of laboratory silica sand-water standards (and other materials), for the calibration of the nuclear gages. He shows that if the silica sand-water calibration curve were used with a nuclear moisture gage to test a certain clay; the indicated water content would be 6 lbs of water per cu ft less than that determined on the basis of the oven-dry moisture test.[1] In a preliminary investigation by the California Division of Highways, it was found that if a similar sand-water calibration were used on a different clay, the indicated water content could be as much as 6 lbs per cu ft greater than that calculated on the basis of the oven-dry moisture test.

It is the free, or easily evaporable, soil moisture that is of interest in highway construction. At the present time it appears that individual calibrations for this water content may vary as much as 12 lbs of water per cu ft from one another. This poses a serious problem inasmuch as it is often necessary to estimate, or adjust for change in, the free water content when controlling the compaction of soil.

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[1] In nuclear testing, the water content, or water "density" is directly determined in lbs of water per cu ft of soil. The "oven-dry water content" is calculated using the percent moisture (ASTM: D 2216-66) and the in-place wet density of a soil.

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A current project which is entitled "Calibration Standards for Nuclear Gages" is underway, under the direction of the writer, at the California Division of Highways Materials and Research Laboratory. This is a federally financed project in cooperation with the United States Department of Transportation, Bureau of Public Roads. The research is for the development of density standards, as well as moisture standards. Investigation of the density standards is nearing completion at the time of submission of this paper. A report has been prepared (8), and has received the approval of the Bureau of Public Roads.

Being involved in this work, the writer has become aware of the overall moisture calibration problem and, thus, independently developed the hypothesis presented herein. Briefly stated, the free moisture calibration curves for different soils should all have essentially the same slope. Because of the pertinence of this concept to the calibration problem, a supplemental test program was proposed to the Materials and Research Department, and Sacramento State College Engineering Department as a suitable topic for research. This proposal was accepted by both departments.

The physical research was confined to testing in the laboratory, with several nuclear moisture gages, the water contents of large soil samples. These soil samples were

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prepared by compacting materials with known mineral composition and water contents, in large molds. The work was performed using state facilities and equipment as part of a Federally-sponsored Division of Highways research project (F-4-22). All work was planned and directly supervised by the writer as part of this project.

The literature review, analysis of data, and all of the written portions of the thesis were conducted as an independent effort, largely on the writer's own time. Much of the thesis material will be included or cited in the final report on the moisture standards portion of the above-mentioned Calibration Standards for Nuclear Gages research project.

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I

PRINCIPLE OF THE NUCLEAR MOISTURE GAGE

The basis for determining soil moisture by nuclear methods has been more than adequately dealt with by others (4, p. 23, 5, p. 14, 9, p. 64). The phenomenon depends upon the thermalization, or slowing down, of fast neutrons (emitted from a suitable radioisotope) in elastic collisions with the hydrogen in the soil water. Since a neutron and a hydrogen nucleus have nearly the same mass, about one-half of the fast neutron's initial kinetic energy is expended when the two collide. In contrast, when the fast neutrons strike the larger nuclei of most of the remaining constituents of soil, much less energy is lost. Water is therefore an efficient moderator of fast neutrons because of its high hydrogen content.

The neutron has no electrical charge, and is not greatly affected by the electron fields surrounding the various atoms which make up a soil mass. As a consequence, it is well established that the numbers of slow neutrons formed when a soil is irradiated with fast neutrons, are proportional to the hydrogen content (4, p. 23, 5, p. 15). The hydrogen in compounds such as ammonium nitrate (NH_4NO_3), and organic hydrocarbons, can affect the nuclear moisture

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determination. However, it has been generally presumed that these materials are not common in most soils used in engineering construction.

An abridged table is presented below showing the capacity of various elements for slowing down fast neutrons. It is taken from a training manual supplied by one of the larger manufacturers of nuclear gages, Troxler Electronics Laboratories, Raleigh, North Carolina (10).

TABLE 1.

Relative Effectiveness of Elements in Slowing Down Fast Neutrons

Element	Average number of collisons required for thermalization	Element	Average Number of collisions required for thermalization
Hydrogen	18.2	Silicon	262
Boron	104.5	Phosphorus	288
Carbon	115.4	Sulfur	298
Nitrogen	133.5	Chlorine	329
Oxygen	152	Potassium	362
Sodium	215	Calcium	371
Magnesium	227	Iron	514
Aluminum	251	Cadmium	1028
		Uranium	2169

Some elements are capable of absorbing thermal neutrons, and affecting the determination of soil moisture. The following table, also abridged from Troxler, is helpful in estimating the relative importance of this factor. It is seen that the only elements which should ordinarily have to be considered would be chlorine and perhaps lithium and

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and boron, although some research indicates that the nitrogen-potassium-iron group may be important. (7, pp. 100-102)

TABLE 2.

RELATIVE ABSORPTION CAPABILITY OF SOME ELEMENTS FOR THERMAL NEUTRONS (0.025 ev)

Element	*Barns	Element	*Barns
Cadmium	2450	Sodium	0.505
Boron	755	Calcium	0.44
Indium	196	Hydrogen	0.332
Gold	98.8	Aluminum	0.230
Lithium	71.0	Magnesium	0.063
Silver	63.0	Carbon	0.0034
Chlorine	33.6	Sulfur	0.00052
Iron	2.53	Oxygen	0.0002
Potassium	2.07	Phosphorus	0.0002
Nitrogen	1.88	Silicon	0.00016

*A "barn" or "Fermi" is a measure of the probable cross-sectional target area.

The nuclear moisture gage itself contains a radioisotope which emits fast neutrons, a detector sensitive to slow neutrons, and a counting device (scaler). The water content of a soil sample is estimated by irradiating it with the fast neutrons, and then measuring the slow neutrons produced by the hydrogen contained in the soil moisture. Because the radiation from a radioisotope is a random process, it is necessary that the count of thermalized neutrons be sufficiently large for statistical acceptability.

There are many other factors regarding the relative geometry of source and detector, type of radioisotope used,

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shielding, etc., that must be reconciled in the gage design, which are not considered in this discussion.

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II

THE CALIBRATION PROBLEM

Field Moisture Correlation

In the fall of 1967, the writer was assigned responsibility for conducting research on the development of calibration standards for the nuclear moisture-density gages being used by the California Division of Highways. This work was in progress at the Materials and Research Laboratory, 5900 Folsom Blvd., Sacramento, California.

At that time, temporary standards having high and low water contents were prepared for calibration of the moisture gages. These were fabricated by compacting a local river sand obtained near Perkins, California, in molds cut from steel drums. Before compaction, the sand was allowed to come to air-dry equilibrium moisture. The air-dry sand was compacted similarly in each of two tubs, and the bulk densities of both were calculated.

One tub had a plastic tube leading to the bottom so that it could be saturated under a positive head of water. A piezometer was provided to show the saturated water level. This sample was then flooded so that the free water surface

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was at the sand surface, and all voids were essentially full of water. It was therefore assumed that no further change could occur in the distribution of water. The amount of water added to this tub was measured, and checked by weighing the tub and contents before and after the operation. The initial water content of the air-dry sand was estimated from the oven-dry moisture contents of small samples taken during compaction of the sand. 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 the low moisture standard. Subsequent weighing of this sample indicated little change in moisture content. When the tub containing the air-dry sand was torn down it was found that the top one to two inches was slightly drier than at greater depth. However, this seemed to have a negligible effect on the tests taken on that sample with the nuclear moisture gages. Water was added to the tub of saturated sand as necessary. The surfaces of both the high and low water content tubs were protected by a plastic cover.

Figure 1 shows plotted field nuclear moisture test data, gathered for a variety of soils by a sales representative of one of the nuclear gage manufacturers. The points show water content in lbs of water per cu ft plotted with respect to the nuclear reading. This reading is in terms of

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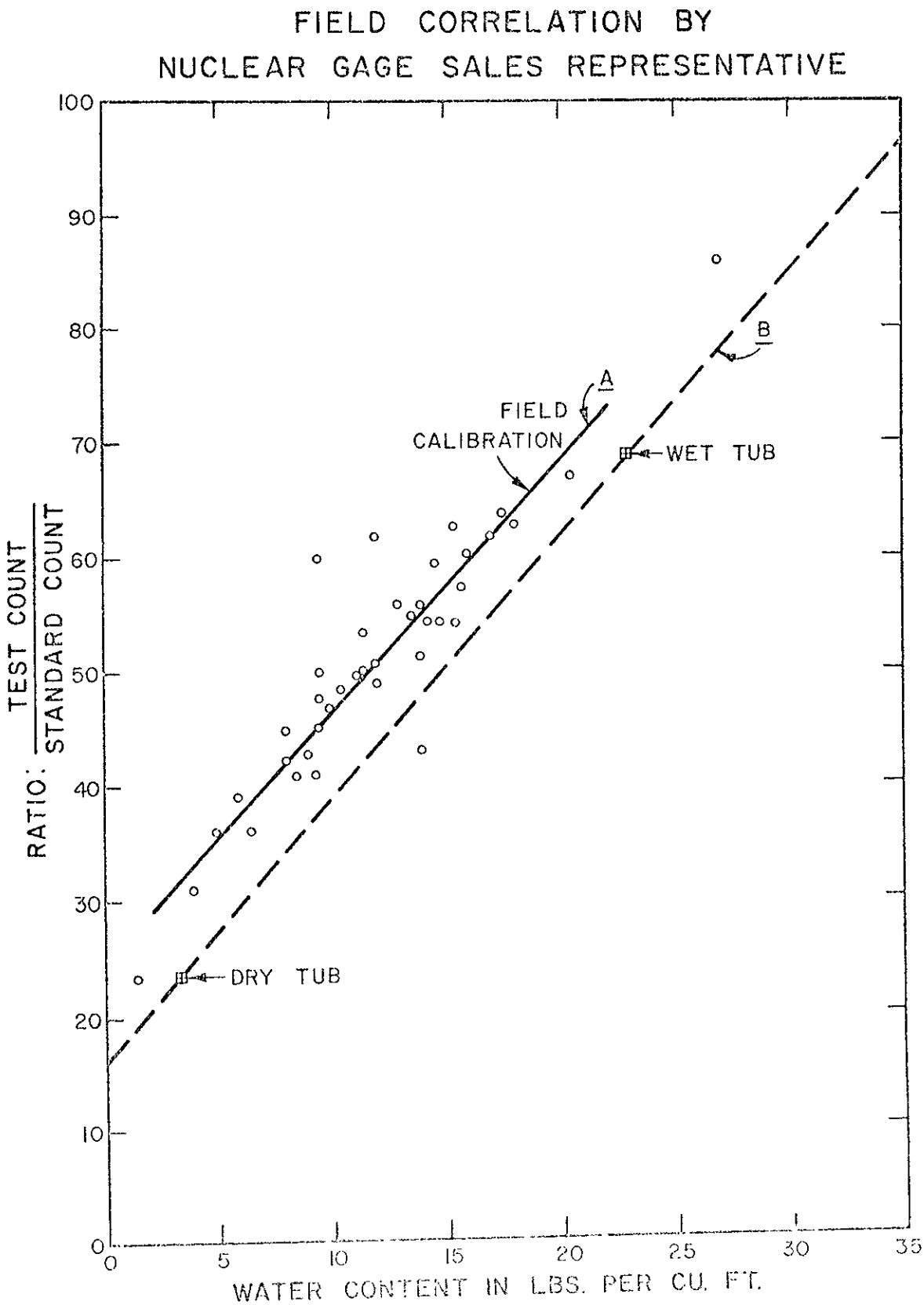
Figure 1



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"count-ratio," which is the average of one or more one-minute test counts on the soil, divided by the average of several one-minute test counts on an arbitrary non-soil standard provided with the gage. This practice tends to compensate for drift or fluctuation of the gage over a period of time. The water content of the field soils was computed using oven-dry moistures and field wet densities. Two curves are shown in Figure 1. Curve A is a visual regression line through the plotted data. Curve B is drawn through the two data points derived from nuclear readings taken (with the same gage used to take the field data) on the temporary standards with a known high and low water content. Curve B was superimposed <u>after</u> Curve A was estimated.

The field data points tend to lie to the left of the reference curve determined on the temporary moisture standards. The displacement of the estimated field regression curve from the reference curve is equivalent to about 3 lb per cu ft of water. In other words, the average water content of the soils indicated by the nuclear gage was greater than that by the oven-dry method. However, it is apparent that Curve A and Curve B are very nearly parallel.

It should be noted when examining Figure 1 that the scatter of data is not [illegible] taken as a measure of

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the accuracy of the nuclear method. When soil water content per unit volume is computed by the oven-dry test method, the resultant value is subject not only to error in the sampling and measuring of the percentage of moisture, but also to error in the determination of wet density. Further, the sample of soil taken for the oven dry moisture determination may not be representative of that volume of soil tested by the nuclear gage. Thus, there is a strong possibility of including several errors in such a correlation.

In this particular example, the information was derived from different soils at several locations. This, undoubtedly also contributed to the scatter of data in Figure 1. Similar data gathered for the Materials and Research Department showed that various soils seemed to have individual displacements from the reference curve of 1, to as much as 6 lbs of water per cu ft.

Total Soil Moisture

In the section "Field Moisture Correlation" it was demonstrated that the nuclear moisture gages appeared to indicate more water in some soils than was determined by the oven-dry moisture test. It was believed that this was due to the probable presence of moisture, especially in the clay minerals, which [illegible] not removed in the oven-dry test. The nuclear moisture gage responds to total soil

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moisture, which may include in addition to the free water: inter-layer water, hydrates, and hydroxyl water.[1,2] It is easily demonstrated that all of these forms of water are not evaporated at temperatures of 105-110°C. For example, Nutting reports that the kaolin minerals loose an additional 14 percent by weight (approximately) of hydroxyl water between 400 and 800°C (11, 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 will respond to the free water equivalent of the hydroxyl water in kaolin; or, about 7 percent by weight.

The montmorillonite - smectite clay minerals loose significant amounts of interlayer water at temperatures around 100-200°C, and hydroxyl water at between 300-800°C (12, pp. 314-315). Typical data for some clay minerals are summarized in Table 3. These are approximate average values showing the trend toward additional water loss at temperatures above 100°C. For the portion of this loss

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[1]Free water is defined in this paper as pore water which evaporates at a temperature of 105-110°C. This may include a few materials such as gypsum which loose all or portions of their hydrated water below this temperature.

[2]The term "hydroxyl water" is commonly used to indicate the presence of the hydroxyl ion (OH^-) in clay minerals, such as Kaolin $(OH)_8Si_4Al_4O_{10}$.

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TABLE 3

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

Mineral	Temperature °C (100 – 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 (12)

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considered to be hydroxyl water, the equivalent free water loss (1/2) 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. Many soils have appreciable quantities of both the clay minerals and hydrates in them. Also, Lambe reported that a diatomaceous earth retained significant amounts of water at temperatures above 105°C (13). 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.

The data for Figures 2 and 3, which follow, are from information relevant to soil samples secured by others within the Materials and Research Department, who were conducting a specific investigation regarding nuclear moisture determinations. The nuclear and oven-dry water contents of several in-place soils were determined. Samples of the same soils were also compacted in a large mold, and tested for water contents by the two methods. The mold and in-place field data are undifferentiated, although in some cases there were differences between them.

Representative data from two of the soils tested are shown on Figures 2 and 3. [illegible] illustrate the [illegible]

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Figure 2

NUCLEAR MOISTURE CORRELATION
TEST DATA

SOIL A

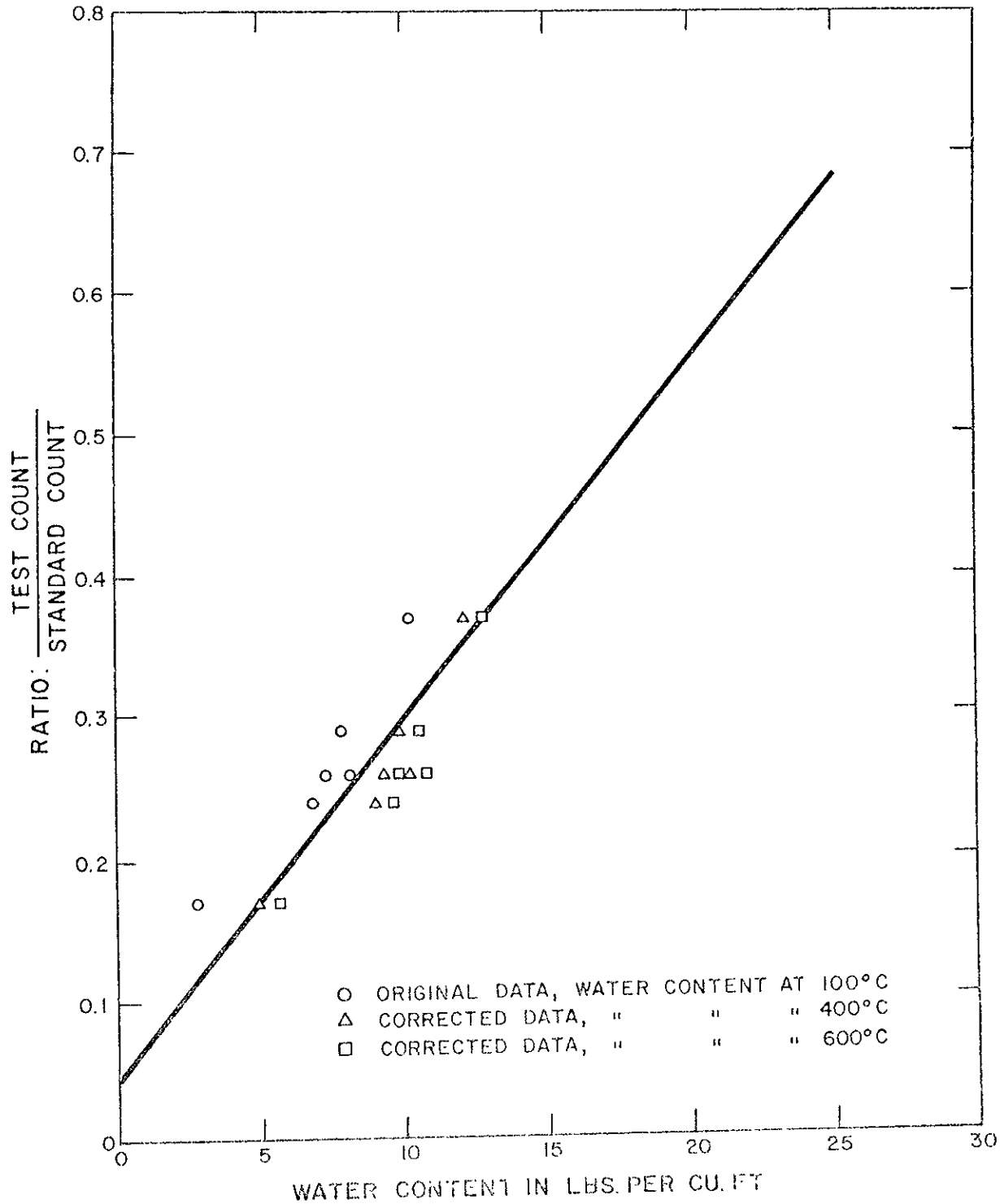

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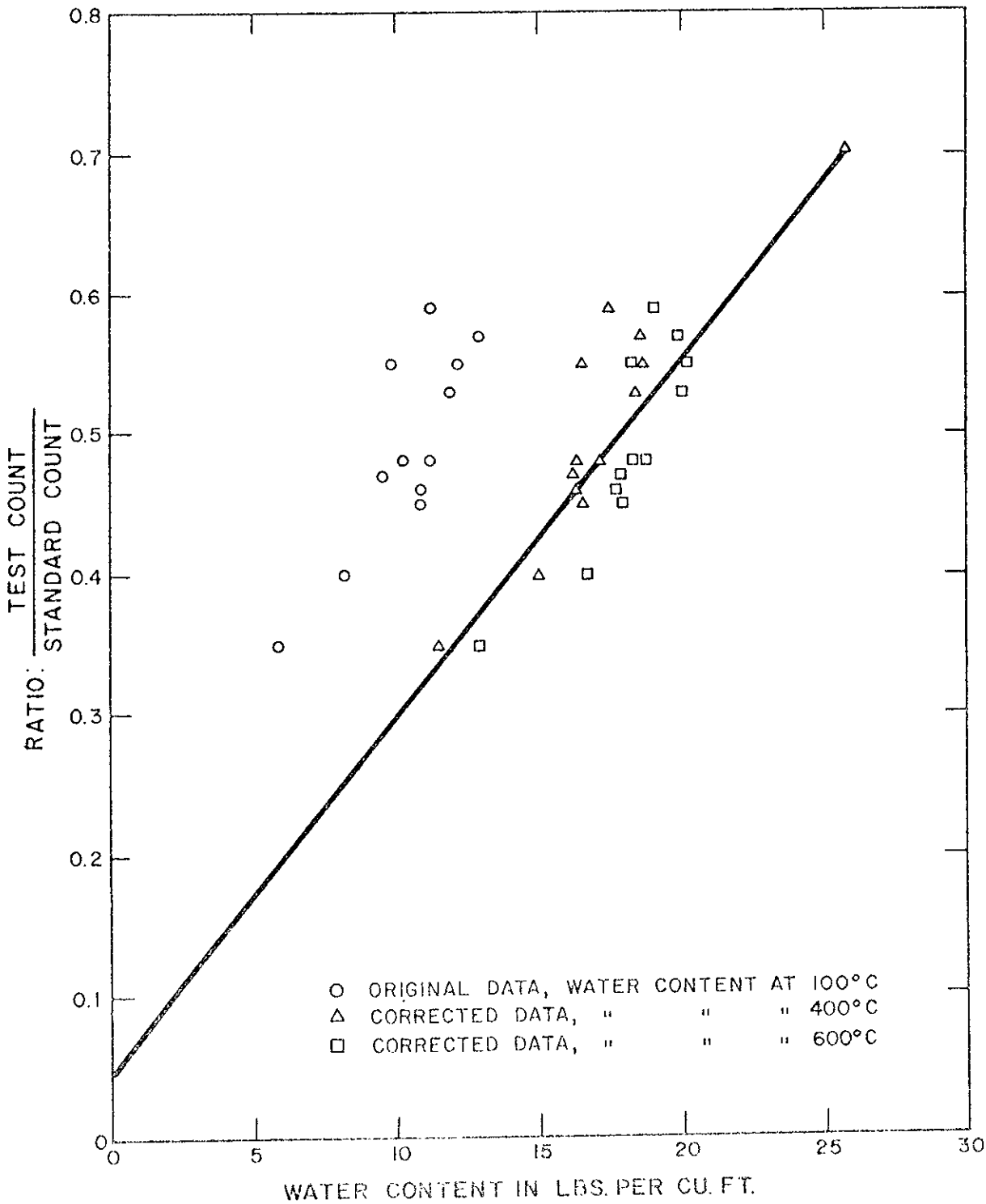
Figure 3

NUCLEAR MOISTURE CORRELATION
TEST DATA
SOIL B



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of 100°C to insure a similar base moisture content. These

correlations between the two test methods which may be encountered on typical projects. The circular points represent the original field correlation data. The linear curve is a reference calibration established using the temporary sand-water standards previously discussed. Soil A data are displaced from the reference calibration line approximately 1 lb per cu ft of water, while the data for soil B have an offset of some 6 lbs per cu ft of water. Soil A is a semi-granular material with less than 3 percent of the soil passing the #200 sieve. Soil B was a highly weathered soil with a much higher percentage of clay size minerals.

Samples, generally representative of the materials tested in the field correlation study, were returned to the laboratory. From these, small samples were obtained for further testing. High temperature evaporation tests were made to 400 and 600°C and the equivalent structural water content determined. The original field correlation data were then corrected for this additional water content. This is also shown on Figures 2 and 3. As the loss in weight between 400 and 600°C is arbitrarily taken to be hydroxyl water, only one-half of this loss by weight is plotted as additional water content.[1] This corresponds to

[1]The corrected water contents are based on the field dry densities as determined for the original field correlation study. Small samples of the soils were first dried at 100°C to insure a similar base moisture content. These were then dried to 400 and 600°C, and the percent loss of the weight at 110°C computed.

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the assumption made for the data in Table 3.

The important thing to note in Figures 2 and 3 is that the data groups tend to converge on the reference calibration curve, when the additional water content is taken into account. It was concluded that the presence of structural water[1] in the field soil caused the discrepancy between the original field correlation data, and the reference calibration curve.

It should be remembered, while examining these plots, that in each case the correction for additional moisture loss is applied to the original data. Since the correction is a fixed amount of water for each temperature increase, any scatter in the original points is reproduced in replotting at the additional water contents.

Thermal Neutron Absorption

In 1964, Burn published a report, "Calibration of a Neutron Moisture Meter in Leda Clay," (7). Figure 4 which follows is a reproduction of a graph from that paper showing moisture correlation data for a nuclear gage used on

[1]"Structural Water" is defined to be the total of the hydroxyl water, and the inter-layer water and hydrates which are not evaporated at temperatures below 105-110°C.

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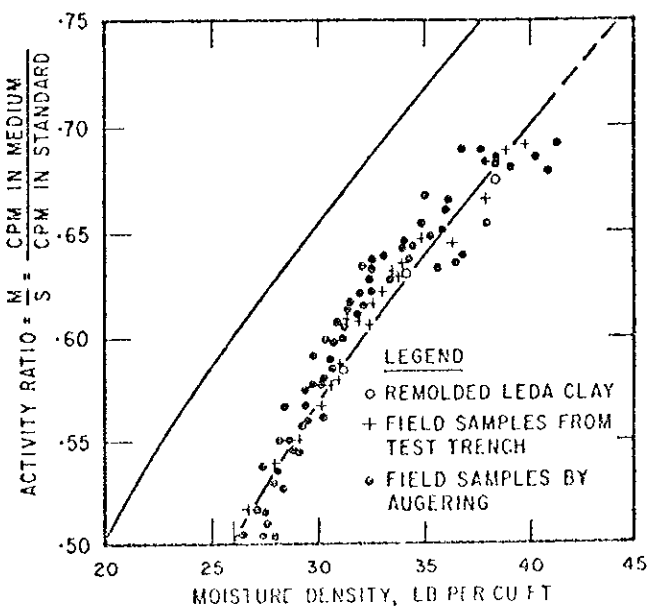
the Leda clay. Here, it is shown that the plot of nuclear reading versus oven-dry moisture content of the soil is nearly 6 lbs per cu ft to the right of the reference curve. This is to the opposite extreme from the data shown in Figures 1, 2, and 3. Burn's reference curve is based (in part) on silica sand and water standards having known total water contents. These calibration standards generally correspond to the temporary California Highway standards previously described. It is noted that the calibration by Burn is linear to approximately 20 lbs of water per cu ft (5, 6, 7) and curvilinear thereafter, as shown in Figure 4. The California standards reflect only the linear portion of the relationship, which extends to approximately 25 lbs of water per cu ft.

Figure 4.

CALIBRATION CURVE FOR LEDA CLAY



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Burn speculated that the discrepancy between the two calibrations was due to the presence of iron in the clay which absorbed the neutron flux. In his discussion, it was acknowledged that the presence of the hydroxyl water would make an even greater difference between the laboratory and field calibration curves. He observed, as is apparent in the figure, that the displacement appears to be constant for that soil.

In summary then, it has been shown that if the nuclear gage is calibrated on an inert system such as silica sand and water, 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 oven-dry moisture determination from the same soil.

Current Practice

The obvious question must be answered, "What are the instrument users doing now?" There are several answers. First, the preceding examples may be extreme; so that the users are able to utilize the calibration supplied with the gage until such time as it requires recalibration. This may be caused by adjustment, or replacement of a major component of the instrument. Second, on the projects where there is little change in field moisture, the inspectors may use uncorrected field wet densities. The nuclear

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moisture circuit in the gage is simply not used. Third, (and most common) the operators calibrate by correlating the units directly against field soil water contents as determined by the oven-dry method.

Field calibration may be satisfactory for a particular soil if a sufficient range of moistures is obtainable, and the data not too scattered. Too frequently though, the normal variance in correlation data, and an inadequate available moisture range; make it impossible to obtain a statistically acceptable calibration by this method. At best it is a laborious, impermanent, and generally inaccurate procedure. Moreover, the process must be repeated each time a new soil is encountered, or the gage suspected of drift or malfunction.

To conclude, it is the free soil moisture that must be controlled in earth construction. As has been shown, this is 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 with any measure of accuracy.

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Selected Literature

The basic reference material regarding the construction of calibration standards for nuclear moisture gages are reports published by Burn in 1960 and 1962 (5, 6). He describes the use of silica sand and water as being a satisfactory medium, augmented with such materials as calgon and dextrose. He briefly mentioned the discrepancy in field moisture correlation with respect to the Leda clay in reference 6. This subject was further discussed by him (7) as previously cited in the section, "Thermal Neutron Absorption."

The references by Preiss & Grant (14), and Preiss (15), suggest that the chemical effects (absorption and bonding of hydrogen atoms), such as noted by Burn (7), can be alleviated by the use of a detector tube having a thin metallic shield to absorb very low energy neutrons. However, it is difficult to see how this can help the gage distinguish between free and structural soil water.

Ballard and Gardner (4, p. 21-27) state that, while there is debate concerning the accuracy of nuclear moistures, the determination is not as critical as density; and

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therefore has not received the same attention.[1] They conclude that measurements with the nuclear gages are as reproducible as any existing non-nuclear method. They propose the use of non-soil standards with a regression analysis embodying corrections for chemical effects, etc., based on a suggested mathematical model.

Williamson and Witczak suggest that moisture calibration be done in the field (3, p. 131). They also state that one calibration can be used for the materials commonly found in highway construction, although they do indicate that this is open to further investigation. This conclusion is contrary to the evidence presented herein, which definitely shows that calibration for free water content varies with the individual soil.

Anday and Hughes illustrate the use of the nuclear gages with a specially constructed test strip as a control. Field nuclear readings of both moisture and density are taken and compared to those of the test strip (16). They state that any calibration is satisfactory, as long as it is sufficiently sensitive, since the field and control

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[1]They stated (4, p. 21, 26) that the moisture gage does not have to be as accurate percentage-wise as a density gage because of the lesser amount of water in the total. Actually, the required accuracy is by weight of water to total wet weight of soil; therefore the moisture gage must equal the density gage in its performance.

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tests are performed on the same soil. However, it can be easily shown that the slope of the moisture calibration curve must be valid. Consequently, even with the test strip technique, the fundamental problem still exists of providing appropriate standards for calibration and checking of the gages.

In another paper, Hughes and Anday report their findings at the July 1965 "Correlation and Conference" (use of nuclear soil gages) held in Charlottesville, Virginia (17). Moisture standards were prepared of chemicals containing "bound" moisture, for measurement with the various gages brought to the conference. A representative calibration curve is shown in their report. It was reported that specially prepared test sections were measured with the gages to determine the water content. Hughes and Anday acknowledge that the instruments reflect total moisture. However, they believed that the water contents determined with the nuclear gages on test sections known to contain a considerable amount of chemically bound water were not affected (p. 254). For some unexplained reason the moisture contents of the compacted test sections were not directly determined, and comparison of results with actual field moisture contents could not be made (p. 252).

The report by LeFevre and Hanke (2, 1968) illustrates the use of various base materials such as gravel, crushed

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limestone, and a red clay, to establish a calibration curve. They show that the moisture calibration using the natural materials differs from the curve supplied by the manufacturer of their gage. It was indicated that a single calibration curve should be satisfactory for field use. However, two samples tried by them, a clay and an expanded shale, did not fit their calibration curve. They concluded that these data points were in error.[1] A minor technical error is made when they state that their calibration curve should have a zero intercept when testing a soil with no water (p. 88). Some thermalization occurs in the complete absence of water, and will be recorded by the instrument. Thus, the intercept will be necessarily greater than zero, see Figure 1, 2, or 3.

The ASTM publication (18) listed in the references contains a survey of world wide research and practice with regard to the use of the nuclear gages. The bibliography in the report is comprehensive, with 265 entries.

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[1]The expanded shale data point is to the right of their calibration curve (2, Figure 10), about 8 lbs of water per cu ft. Research for the thesis has produced similar data which is discussed in the section, "Experimental Procedure."

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III

HYPOTHESIS

In summary form, the hypothesis can be stated:

1) The nuclear moisture gage responds to the total water in a soil, including both the free water and the structural water.

2) The variations in total water content in a soil, such as added compaction water, evaporation, etc., occur almost entirely in the free water.

3) The free water calibration curves for different soils should be parallel. That is, they should be offset from one another by a constant amount according to the differences in structural water and the presence of neutron absorbing elements.

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IV

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 testing of these samples with the nuclear moisture gages should permit evaluation of the hypothesis concerning the nature of the gage measurements, and the correlation between calibrations based on total and free water content of soils.

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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 the following Table 4. The volumes of the samples were checked to insure that they were essentially infinite for testing with the gages. It was found that the dimensions were satisfactory. A slightly higher reading could be obtained from the dry samples by increasing the volume, but the practical effect was determined to be negligible insofar as this research program was concerned.

The top surfaces of most of the samples were protected during the period of testing 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 a nuclear gage on several samples with and without the plastic.

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TABLE 4

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-85-7*	"	"	1.15	3.27	110.5	103.1	7.4	-	7.4	○
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

* 5 layers with plastic between to prevent migration of H_2O.
** Equivalent water content.
*** As used in Figures 6, 7, 8, and 9.
NOTE: The dimension "D" is the least top surface dimension.

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Silica Sand Samples

Silica sands from two sources were obtained from the Amador Mineral Co., Ione, California. One is an eastern sand imported from the Wedron Silica Sand Co., Chicago, Illinois, which has rounded grains. The other is a local sand, crushed and processed by the Amador Mineral Co. These sands are 99.7 to 99.9 percent pure SiO_2. The approximate grading of the sands used is given in Table 5.

TABLE 5.

SILICA SAND GRADING

Ident.	E-25	E-85	#200	A-70
Source	Wedron	Wedron	Wedron	Amador
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	

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Five samples were fabricated from these sands. They were:

1) A-70-W. This sample was fabricated of Amador #70 sand, compacted in approximately 2 in. lifts with an electric impact hammer. A plywood disc was used to confine the sand during compaction. The sample was provided with a perforated plastic tube so that it could be saturated from within. A piezometer was attached so that the water level could be monitored. The (W) in the notation indicates a "wet" or saturated sample. After completion, the water content and dry density of the sample were as shown in the preceding Table 4.

2) E-25-W. This sample was prepared similarly, except that the eastern sand #25 was used.

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

4) E-85-P. In order to obtain an "in-between" water content, it was necessary to attempt to fabricate a sample which would be only partially saturated. This is indicated by the (P) in the sample notation.

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To prevent migration of water during the period of testing, the moistened sand was compacted in increments using sheets of 6-mil polyethylene plastic to separate the layers. A trial sample was prepared with the plastic sheets and dry sand. This sample was tested with a moisture gage, and the reading compared to a reading on a similar dry sample prepared without the plastic sheets. No significant difference in the readings on the two samples was observed, and it was concluded that the effect of the plastic was negligible.

However, in spite of the precautions taken to prevent migration of water in the partially saturated sample (E-85-P), it was found that the water did migrate. This movement of water, especially in the critical top layer, affected the testing of the sample with the nuclear moisture gages. Consequently, this method of preparing a sample was not considered to be satisfactory.

5) E-25-D. The dry silica sand standard (D) was fabricated with the eastern #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.

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Kaolin Samples

A kaolin clay of high purity was obtained from the Amador Mineral Co. The physical properties and chemical analysis given by the company are shown in Table 6.

TABLE 6.

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 technique 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.

A differential thermal analysis (DTA) was made using an oven-dry sample of this material. Typical kaolin dehydration chacteristics are shown, with little loss of water

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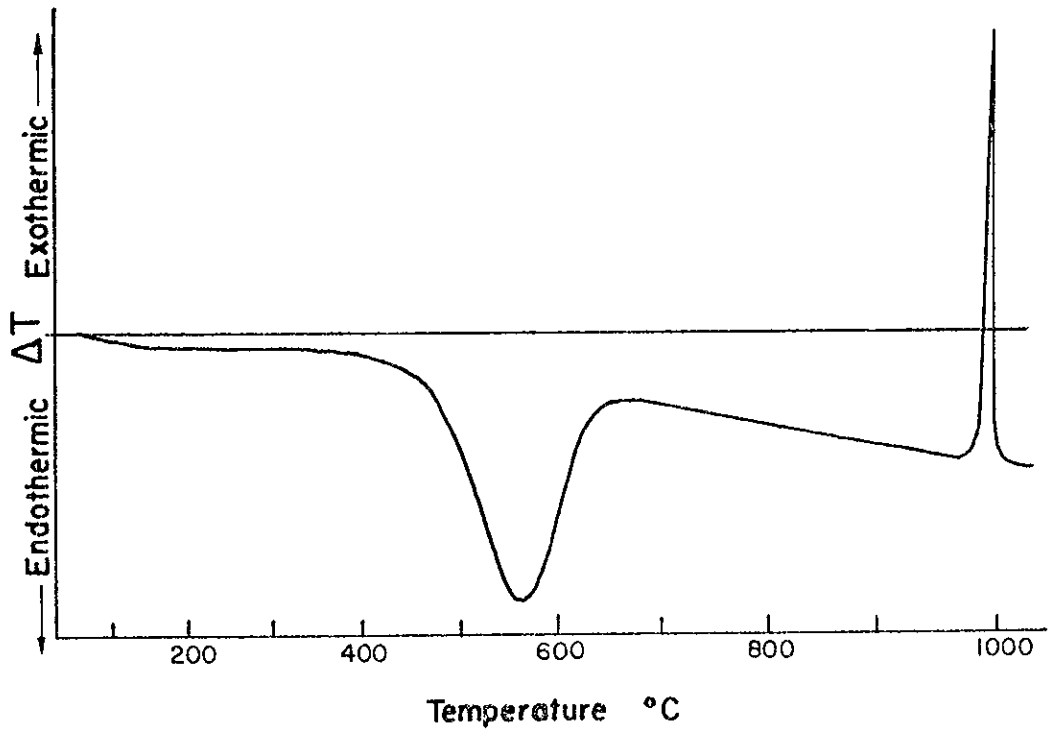
between 100 and 400°C. The sharp endothermic reaction between 450 and 650°C indicates loss of hydroxyl water, (12, p. 299).

Figure 5.

IONE KAOLIN, DTA CURVE



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

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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 were given in the preceding Table 4. 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 clay was compacted in approximately 15 to 19 equal increments with a 10-lb drop hammer. This impact device had a 4- by 4-in. foot, and a total weight of 18 lbs. The surface of each lift was compacted until there was no further apparent densification.

The amounts of water to be added, by weight of dry kaolin, were estimated on the basis of preliminary testing. Due to the very loose or "fluffy" nature of the kaolin, some loss of the batched material was unavoidable during compaction. Consequently, it was not possible to check the batched quantities of soil and water directly against the total weight of the completed sample. The amount of free water added was estimated on the basis of representative oven-dry samples of the clay taken during compaction. The water contents of the samples were also checked when

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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. The pertinent moisture-density data for the Ione kaolin are as shown in the preceding Table 4.

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 in a mold. Table 4 may be referred to for the moisture and density information, and sample dimensions. The grading and chemical data for the soil are given in the following Table 7.

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.

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TABLE 7.

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 "	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. This light-weight product is produced at Napa, California by the Basalt Rock Co. It is marketed under the trade name, Basalite Light-weight Expanded Shale Aggregate; and is used to make light-weight masonry products. The grading of the expanded shale sand is given in the following Table 8, and its chemical analysis as given by the Basalt Rock Co. in Table 9.

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TABLE 8.

EXPANDED SHALE-SAND, GRADING

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

TABLE 9.

EXPANDED SHALE-SAND, 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 4) was prepared by compacting, and then flooding it until it was saturated. The (ES-dry) sample was similarly compacted, but was left in a dry or "as compacted" state. The remaining three expanded shale-sand samples were prepared by moistening the shale-sand to the desired water contents, and then compacting the material in the molds. Due to the absorbent nature of the calcined shale, the procedure appeared to work satisfactorily, with little

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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 increment.

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IV

NUCLEAR MOISTURE DATA

The soil samples were not necessarily fabricated or tested in the order presented. Furthermore, it was not possible to retain all samples so that they could be tested concurrently. Consequently, there is some possibility of variation in the apparent relationship of the testing of the samples. This could have been caused by instrument drift, or slight changes in test conditions and procedure. To compensate, as each new sample was being tested, all available samples were read. At these times, the individual samples were tested with the nuclear moisture gages by taking the average of three or more one-minute test counts. These average one-minute readings were then divided by the gage reading taken on an arbitrary standard to obtain the "count-ratio" as discussed earlier. Therefore, for some of the individual samples, several of these "count-ratios" were accumulated over the period of time the samples were undergoing testing. These were then averaged and plotted against the sample free water in Fibure 6, 7 and against total water content in Figures 8, 9.

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Figure 6

NUCLEAR FREE MOISTURE CORRELATION
TEST DATA
GAGE A

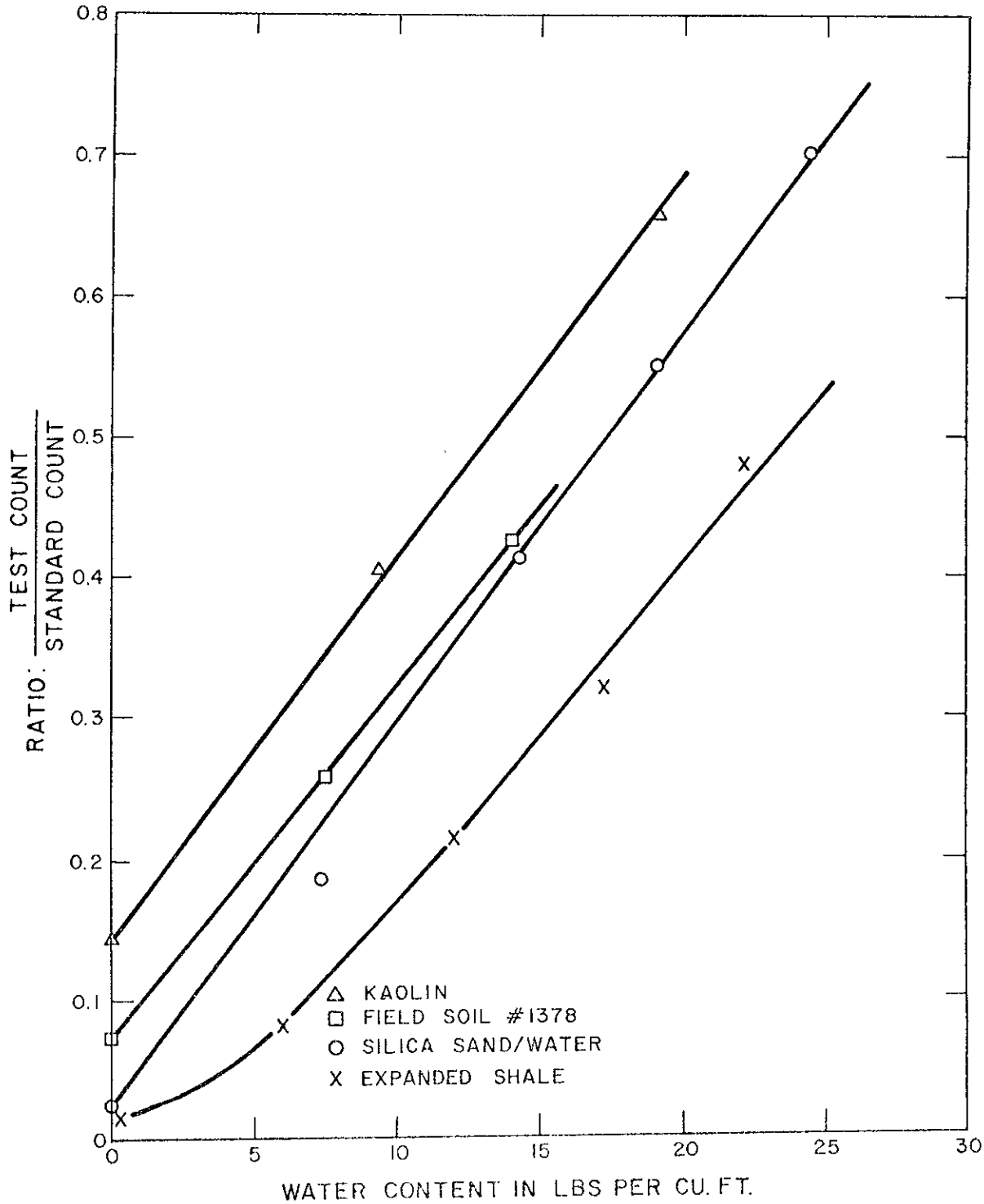

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Figure 7

NUCLEAR FREE MOISTURE CORRELATION
TEST DATA
GAGE B

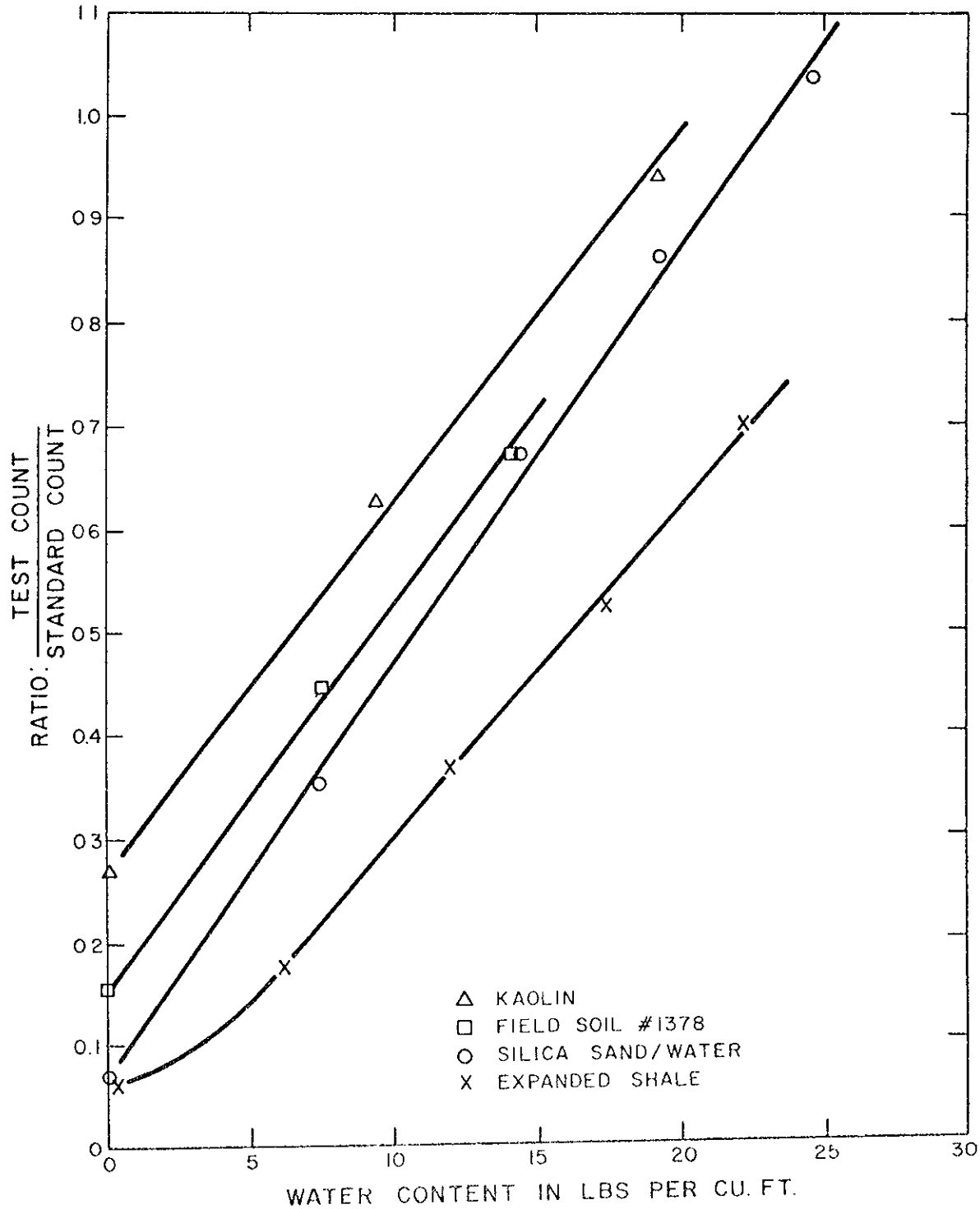

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Figure 8

NUCLEAR TOTAL MOISTURE CORRELATION
TEST DATA
GAGE A

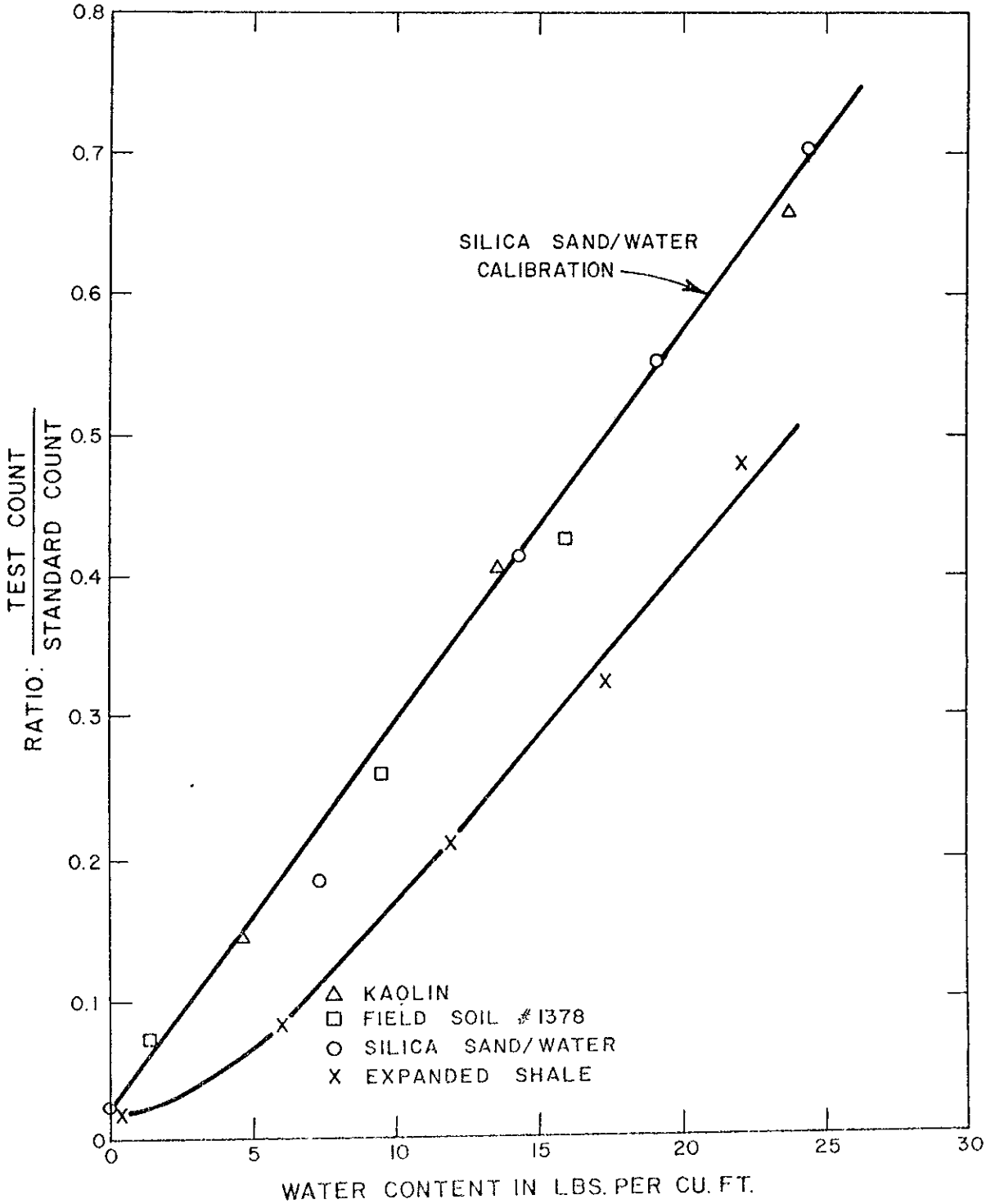

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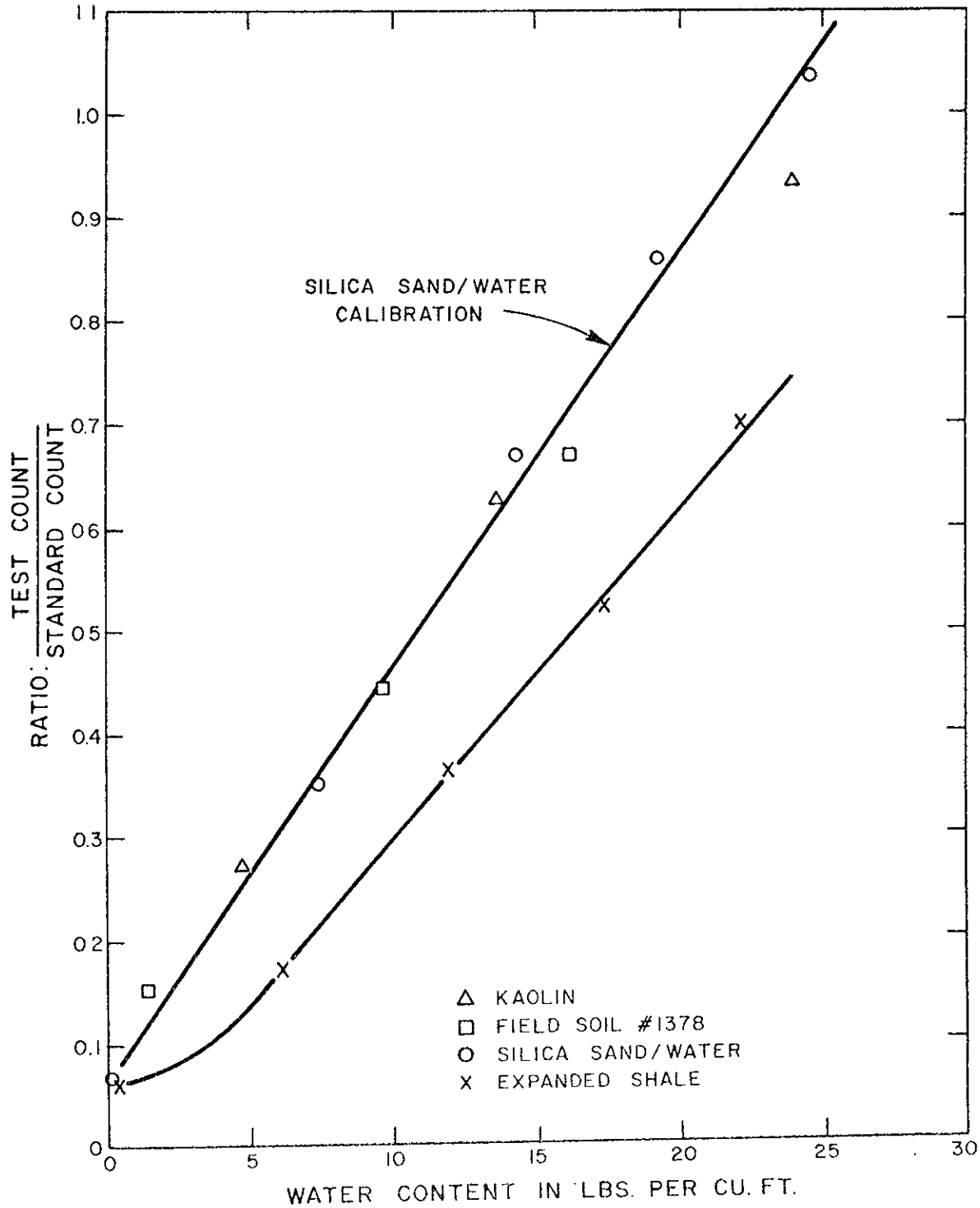
Figure 9

NUCLEAR TOTAL MOISTURE CORRELATION
TEST DATA
GAGE B



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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 is not considered to be as consistent in performance as Gage A. Gage B contains a 50 mc Americium$_{241}$-Beryllium fast neutron source.

Free Water Correlation Data

Figure 6 illustrates the response of Gage A plotted against the free water content of the samples. With this gage, the calibration curves for the four soil-water systems are essentially parallel, except for the expanded shale at the lower water contents. The datum point for the partially saturated silica sand (E-85-P) is not on the silica sand calibration line, apparently due to migration of water within the top layer of the sample.

The readings taken on the samples with Gage B plotted with respect to the free water content, are presented in Figure 7. A greater variation in the slope of the individual calibration lines of the four soil-water systems is exhibited by this instrument. In particular, the kaolin and field soil calibration curves tended to converge with the silica sand calibration curve, while the expanded shale calibration curve diverges somewhat from the silica sand

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curve. It is felt this discrepancy may be attributed to a property of Gage B, after consideration of the similar data from Gage A (Figure 6), and the information presented by Burn (Figure 4).

The response of Gage B to the partially saturated silica sand sample (E-85-P) is closer to the average bulk water content than was Gage A. It appears that Gage B is less affected by the apparent migration of water in the top layer of that sample.

The reason for the displacement of the data for the expanded shale-water system is not positively known. As has been commented upon previously, the report on the study undertaken by the Oklahoma Department of Highways showed a similar datum point for an expanded shale-water sample tested by them (2, p. 89). The chemical analysis of the shale-sand (Table 9) used in this research indicates that there is a total of about 6 percent of iron and potassium present in the material, which would not seem sufficient to cause the deviation shown. However, Burn (7, p. 100) concluded that the presence of between 6 and 7 percent of iron could account for the displacement of the calibration curve in Leda clay. The identification of the source of the apparent absorption of neutron flux in the expanded shale should be the object of further study.

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The oven-dry expanded shale sample (ES-dry) plotted very closely to the dry silica sand sample (E-25-D) in Figures 6-9. This apparently means that a certain level of activity is required before the capacity of the soil to absorb thermal neutrons becomes saturated. In other words, the dry expanded shale sample produces a small number of thermalized neutrons, in a manner comparable to that of the dry silica sand.[1] The initial increments of water are accompanied by an increased production of thermal neutrons; but a very high percentage are absorbed before detection. Then, as the water content is further increased, a smaller and smaller percentage of the production of thermal neutrons is absorbed. At some water content, this absorptive capacity of the soil begins to be satisfied. The production of thermal neutrons, thereafter, 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 are therefore nearly 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

[1]Almost any material would produce some thermalized neutrons. In Figures 6-9, if the straight line portion of the expanded shale curve were extrapolated to its interception of the zero water ordinate, the thermal neutron activity would be less than zero; which is impossible.

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soil, is a calibration constant; is undoubtedly an oversimplification. However, on the basis of this research, 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 soils 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. Consequently, further research is needed to assess the possible importance of this factor.

Total Water Correlation Data

Figures 8 and 9 showed the 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 (14, p. 548; 15, p. 347).

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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 very 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.

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

V. Further research is required to determine the most common absorbing elements which may occur in the soils used in highway construction, and the effect they may have on nuclear moisture gage calibration. It is also necessary to identify the gage design parameters that may be sensitive to this absorption factor.

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IMPLEMENTATION

Implementation by Adjustment of the Calibration Curves for Specific Soils

In this test procedure, the nuclear gage would first be calibrated on reference standards having known total water contents, such as the silica sand-water samples. For a specific soil, the operator would then take readings with the nuclear moisture gage at several field sites. He would also determine the wet densities of these locations with the density portion of his nuclear gage. At these same sites he would take representative soil samples, and ascertain the oven-dry moisture contents. He would then compute the water content of the sites on the basis of the oven-dry moistures and field wet densities.

The average of the several nuclear moisture readings would then be plotted with respect to the average water content (oven-dry) in lbs of water per cu ft. A curve would be drawn through the point and parallel to the calibration previously determined on the reference standards. This parallel curve would then be used to test for the free moisture content of the soil using the nuclear gage. This

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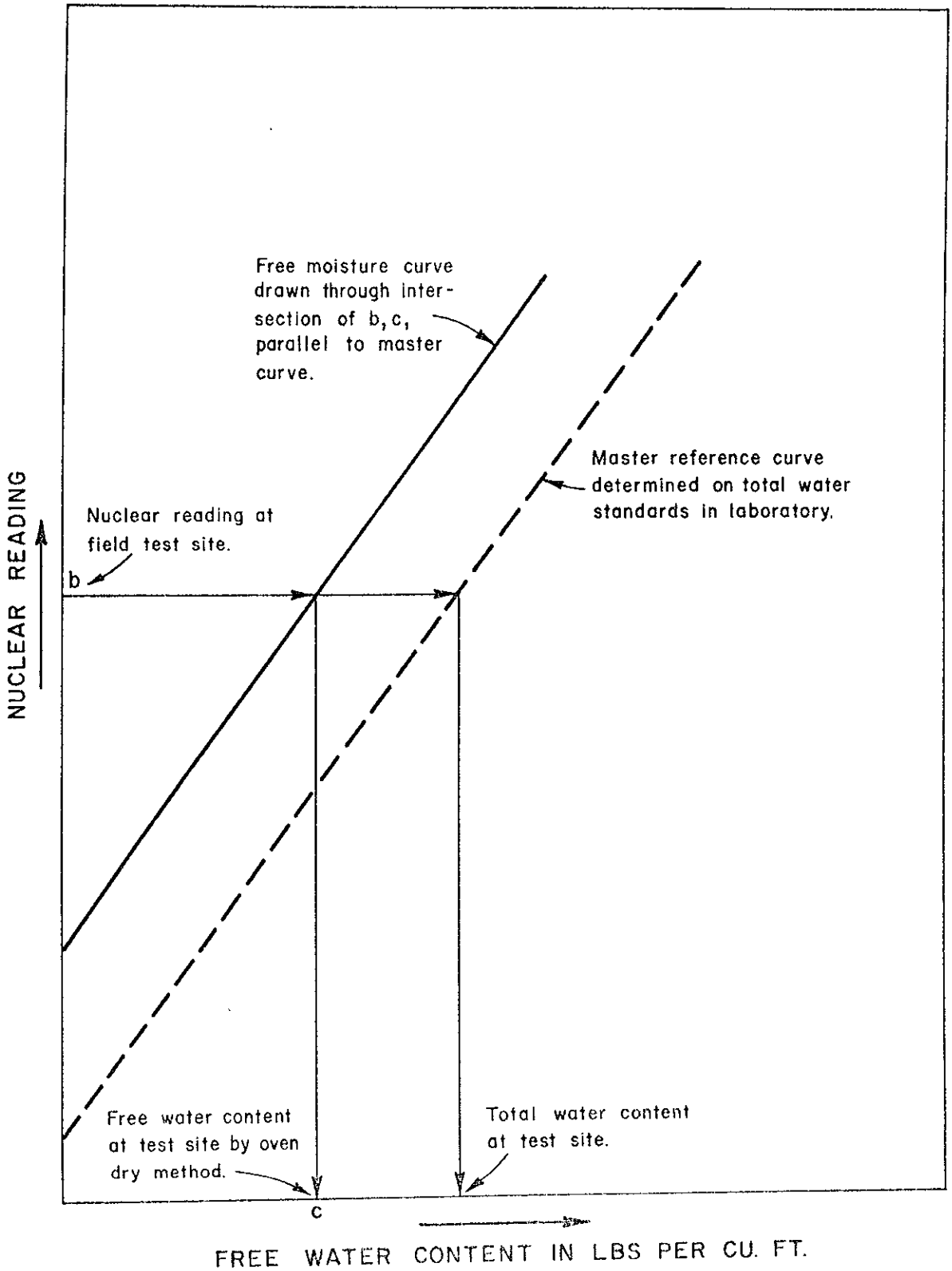
Figure 10

FREE WATER CALIBRATION BY OFFSET FROM TOTAL WATER CALIBRATION CURVE



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is illustrated in Figure 10. The process can be repeated for different soils used on the project.

"Wet Weight" Method of Implementation

An alternate, and probably more desirable form of implementation exists. It can be shown that a practical method of compaction control is possible, utilizing only the slope of the moisture calibration curve. The so-called "wet weight" (1, Calif. 216-F) method of compaction control depends on the ability to express the wet density of a soil, and the laboratory control density, in terms of the same moisture content. While a detailed discussion is not included in this paper, the following example will serve to show that a nuclear gage having a moisture calibration curve with a correctly established slope, can be used to determine the relative compaction of a soil; without knowing its absolute water content. The percent relative compaction computed on the basis of this method contains a slight approximation, but is usually within one-half of one percent of that calculated on the basis of the dry weight method.

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.

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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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