LABORATORY AND FIELD EVALUATION OF THE NUCLEAR MOISTURE AND DENSITY METERS

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by

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PURDUE UNIVERSITY LAFAYETTE INDIANA
To:       G. A. Leonards, Director  
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From:    H. L. Michael, Associate Director  
          Joint Highway Research Project  

February 11, 1966

FILE:  6-10-5  
PROJECT:  C-36-16B  

Attached is a report on the Nuclear Moisture-Density Project. This
report was prepared by personnel in the Joint Highway Research Project
Soils Laboratory.

The report summarizes the significant findings of the entire project.
Recommendations are given in the appendices of the report on procedures
for use of the nuclear device.

Copies of the report should be transmitted to the Bureau of Public
Roads. In particular, Mr. Preston Smith would like several copies for
review. Mr. W. T. Spencer should also receive a copy for review.

Respectfully submitted,

Harold L. Michael
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Laboratory and Field Evaluation of the Nuclear Moisture and Density Meters

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and the
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February 11, 1966
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Laboratory and Field Evaluation of the Nuclear Moisture and Density Meters

INTRODUCTION

The rapid determination of soil density and moisture content is of the utmost importance in the control of highway construction. Soil strength is determined, among other things, to a large extent by the soil's density and moisture content.

Control of moisture and density during construction is accomplished by field tests during the construction operation. Obviously, the speed, accuracy, and reliability of test methods used for control govern to a large degree, the effectiveness and cost of the control process.

In the past several years, a method of measuring soil density and moisture content has been developed through the use of radioactive attenuation. This method was developed on the theory that attenuation of gamma rays can be correlated to soil density while moderation of fast neutrons can be directly associated with the presence of water.

History of Development

In the late 1940's the Civil Aeronautics Administration (now the Federal Aviation Agency) reported the first attempt to measure soil density and moisture by nuclear methods in the United States. Results reported by Pieper (9), Krugger (6), and Delchow, Guelandoll and Sack (1) were
generally favorable. However, feasibility of nuclear techniques were limited by the lack of portable instrumentation.

Improvements in instrumentation and techniques were devised by Carlton (5) in 1953 and Roy and Winterkorn (11) in 1957. In 1956, Pocock (10), developed a mathematical analysis for portable gamma-ray surface density gages for the Michigan State Highway Department. As a result of the above advances, the first combination density-moisture surface gage was introduced in 1959 (4). Major development of the method occurred simultaneously in the United States and in South Africa (3).

Advantages of Nuclear Instruments

The uses and adaption of the gage to soil control are readily observable. The principle advantages of the nuclear moisture-density instrument lie in its portability, speed of operation, and non-destructiveness. The major shortcomings of conventional methods of density and moisture determinations are eliminated by the fact that testing at a specific spot can be accomplished in a matter of minutes. This then represents a significant step in permitting continuous control over large areas with minimum time, cost, and maximum efficiency of operation.

PURPOSE

The purposes of the study reported herein was to evaluate the nuclear moisture-density technique and to study the feasibility of using the methods in control of routine highway construction in Indiana. To accomplish this end, several commercially available instruments were tested. The variables used as the foundation of the testing program were 1) substrate material
properties, 2) instrument stability and 3) testing procedure factors. No attempt was made to correlate the performance of one instrument to another and, thus, data appearing in subsequent paragraphs were selected to illustrate the variables without regard to a specific instrument.

Extensive laboratory work was conducted in the initial phase on the project. The objective of this laboratory work was to provide a basis for establishing testing techniques that were later employed in the field study. Heavy liquids, soils, and soil-aggregate mixtures were the materials tested in the laboratory.

The test sites selected for the field testing phase included natural ground areas, compacted fills, cut areas, loose and compacted subbases, borrow pits and soil waste areas. Material types tested included silts and clays of varying plasticity, granular materials and granular-soil mixtures.

PRINCIPLES OF OPERATION

Theory

The theory of neutron moderation for soil moisture measurement is dependent upon several factors. These are: the presence of fast neutrons, the ability of hydrogen (water) to moderate or slow down the fast neutrons, and the ability of detection devices to measure these slow neutrons.

In the moisture gage, a radioactive source emits fast neutrons into the soil, where they may be either absorbed or moderated to a lower energy level. Hydrogen has the property of being both a low absorber and a highly effective moderator. Consequently, hydrogen possesses one of the highest
neutron moderation capabilities of all elements. As the slowed down neutrons backscatter in all directions throughout the substrate, some of them are transmitted to the detecting device, ionized, and sent as a pulse signal to a recording device called the scaler. Since the moderation is dependent upon the amount of hydrogen present in a volumetric zone of influence, results are calibrated against the weight of hydrogen (water) present in a certain volume.

The theory involved in the operation of a density backscatter device is extremely complex. As the nuclear particles (photons) penetrate into the soil, they can either be absorbed by the mass (photoelectric effect) or can collide with a loosely bound electron and scatter a reduced energy photon in a different direction (Compton effect).

The general equation relating the photoelectric effect can be given by: 

\[ I = I_o e^{-\mu m D m t} \]

where:  
\( I_o \) = Intensity of radiation incident to the absorber  
\( \mu m \) = Mass absorption coefficient  
\( D m \) = Substrate mass density  
\( t \) = Thickness of the absorber

For the Compton effect, a mathematical model was created and experimentally justified by Carey and Reynolds (4). The expression

\[ I = k_1 D m + k_2 D m^2 + k_3 D m^3 \]

represented the intensity recorded from a gage put on a material with an infinite number of electrons available for collisions but no absorption.

Since these relationships cannot exist independently of each other, due to a resulting absorption increase as the number of electrons increase,
the combined intensity can be stated as the product of the two effects, or:

\[ I = I_0 e^{-k_4 D_{\text{m}}} (k_1 m + k_2 D_{\text{m}}^2 + k_3 D_{\text{m}}^3) \] ............... (3)

where \( k_1, k_2, k_3, k_4 \) represent manufacturer's design constants. The latter relationship, in essence, demonstrates the feasibility of adopting physical principles to the employment of commercial gages to soil density.

**Description of a Typical Nuclear Gage**

Nuclear density units can generally be classified into two distinct types: direct transmission and surface backscatter gages. Since for this project, all instrument systems evaluated were of the latter case, discussions involving the density gage will be related to surface backscatter devices only.

A nuclear density-moisture gage, as illustrated in Figure 1, has two main components: the probe (either density, moisture, or a combination density-moisture unit) and the scaler.

The probe or surface gage houses the radioactive source, a shield of thick lead or cadmium, detectors to receive the back scattered rays and convert them to electrical impulses, and a pre-amplifier device which increases the strength of the electrical signals to levels which can be measured.

The primary functions of the scaler are to further amplify the electrical impulses and pass these impulses through a counting device, so that the number of signals received can be represented by a count per unit time.
FIG. 1. SCHEMATIC DIAGRAM OF A COMBINED SURFACE GAGE USING A SINGLE RADIOACTIVE SOURCE (FROM MICHIGAN HIGHWAY DEPARTMENT)
Methods of Expressing Nuclear Results

Results of a given nuclear reading (either density or moisture) can be plotted in two ways. The two methods are:

1. Actual counts per minute recorded on the scaler against the measured variable (either mass unit weight or weight of water per cubic foot.)

2. Nuclear count ratio versus the measured variable in question.

To use the first method, the high voltage setting is adjusted so that the gage will reproduce the same average count per minute within a reliable error for a given standard. All instrument systems contain a standard "block" for both density and moisture "self-standard" readings. Once the given standard reading has been attained, actual counts per minute of the substrate variable may then be directly plotted.

To utilize the count ratio procedure, the high voltage setting is kept constant throughout the testing period. The self-standard reading then becomes defined as the average of the self-standard readings taken for a given test period. An actual count per minute is then obtained on the substrate and the ratio of the actual count per minute to the self standard count per minute is defined as the count ratio for the measured variable.
RESULTS

Instrument Stability

Reproducibility

In order for a nuclear gage to perform satisfactorily as a field control instrument, the reproducibility of results must be consistent over a period of time for a given test variable. If the gage does not perform in this manner, its usefulness and effectiveness may be in doubt.

To periodically check test result reproducibility, use was made of voltage plateau curves for the density and moisture gages. The primary use of voltage plateau curves is to provide information concerning the selection of a proper operating high voltage. However, since these curves are self standard readings plotted as a function of high voltage, the variation of nuclear counts with time is a measure of the ability of a gage to record reproducible self standard readings at constant operating voltage.

Reproducibility of count ratio results of the density gages were obtained by determining count readings on a concrete block at various high voltages and times. Reproducibility for the moisture gages was determined by self-standard readings.

Each density instrument was assigned a specific concrete block. A permanent outline of the instrument on its block was formed by gluing a piece of weather stripping onto the block. By placing the instrument within the outline and in the same orientation each time a reading was obtained, variations due to placement and direction were eliminated. A
count ratio was obtained by dividing the reading on the concrete block by the self standard reading at the same voltage.

High voltage curves for a density and moisture gage are shown in Figures 2 and 3. From Figure 2, it can be seen that for an operating high voltage of 1000 volts, (voltage setting employed from June 1962 to completion of testing) the self standard reading decreased from 43,215 cpm (counts per minute) in January 1962 to 40,169 cpm in July 1964. This represents a decrease far out side the reliable error for the gage. In contrast, the variation of self standards in the moisture gage was within the reliable error (Figure 3).

Also, it is noted from Figure 2 that from October 1962 to July 1964 the count ratio for the standard concrete block remained practically constant, varying from 0.276 to 0.277 for a high voltage of 1000.

Figure 4 shows variation of the density gage self standard cpm, count ratio and moisture gage self standard cpm with time for constant operating voltage. The data show that the self standard readings for the density gage with Cs-137 source decreased with time for constant high voltage. However, for the test period employed, use of the count ratio procedure eliminated this effect and produced a high degree of test repeatability. For the moisture gage with a BaBe source a high degree of reproducibility is also shown. A further discussion of the techniques of expressing results based on reproducibility and aging effects are provided in the section in this text pertaining to Procedural Factors.
FIG. 2. VARIATION OF DENSITY GAGE READINGS WITH PLATEAU VOLTAGE

NOTES:
- $C_s^{137}$ SOURCE
- AVERAGE OF TWO PLATEAU CURVES FOR ONE MONTH INTERVAL
- △ JANUARY 1962
- ○ OCTOBER 1962
- × JULY 1964
FIG. 3. VARIATION OF MOISTURE GAGE READINGS WITH PLATEAU VOLTAGE
FIG. 4. VARIATION OF GAGE READINGS WITH TIME AT CONSTANT PLATEAU VOLTAGE
Aging

Radioactive materials decay at differing rates depending upon the half-life of the source. The rate at which they decay is governed by the natural radioactive decay law and; consequently, the reproducibility of self-standard readings for nuclear gages is a function of the radioactive source employed.

The half-life of the Cs$^{137}$ source illustrated in Figures 2 and 4 is 33 years in contrast to the 1620 year half-life of the RaBe source shown in Figures 3 and 4. Therefore a nuclear gage utilizing a Cs$^{137}$ source would obviously show a larger proportional decrease in counts measured by a detector tube than a nuclear gage using a RaBe source for a given time interval.

If decay ratio is defined as the nuclear activity at time zero to the activity at time \( t \), theoretical and actual decay ratios can be computed and compared for the density gage utilizing the Cs$^{137}$ source. These data appear in the lower portion of Figure 4. The general equations of radioactive decay are:

\[
N(t) = N_0 e^{-\lambda t} \quad \text{(4)}
\]

\[
A(t) = \lambda N(t) \quad \text{(5)}
\]

\[
T = \frac{\ln 2}{\lambda} \quad \text{(6)}
\]

where

\[
N(t) = \text{No. of undecayed atoms at time } (t)
\]

\[
N_0 = \text{No. of undecayed atoms at time } (t = 0)
\]

\[
A(t) = \text{Activity at time } (t)
\]

\[
A_0 = \text{Activity at time } (t = 0)
\]
\[ t = \text{time from } t = 0 \text{ (yrs)} \]
\[ \lambda = \text{proportionality constant (yrs}^{-1}) = .021 \text{ for (Cs }^{137} \text{)} \]
\[ T = \text{half-life (yrs)} = 33 \text{ yrs for (Cs }^{137} \text{)} \]

Therefore the theoretical decay ratio becomes:

\[
R_T = \frac{A_o(t=0)}{A_e(t=t)} = \frac{\lambda N_o e^{-\lambda t}}{\lambda N_o e^{-\lambda t}} = \frac{1}{e^{-\lambda t}} = e^{\lambda t}
\]

\[ \text{(7)} \]

For the actual decay ratio:

\[
R_A = \left( \frac{\text{CPM (t=0)}}{\text{CPM (t=t)}} \right)
\]

\[ \text{(8)} \]

Table 1

Comparison of Actual and Theoretical Decay Ratio

<table>
<thead>
<tr>
<th>( t ) (yrs)</th>
<th>( e^{\lambda t} )</th>
<th>( R_T )</th>
<th>( \frac{\text{CPM (t=0)}}{\text{CPM (t=t)}} )</th>
<th>( R_A )</th>
</tr>
</thead>
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<tr>
<td>0 (January 1962)</td>
<td>( e^{(.021)0} )</td>
<td>1.000</td>
<td>(43,215)</td>
<td>1.000</td>
</tr>
<tr>
<td>0.33 (October 1962)</td>
<td>( e^{(.021)(0.83)} )</td>
<td>1.017</td>
<td>(43,215)</td>
<td>1.015</td>
</tr>
<tr>
<td>1.17 (February 1963)</td>
<td>( e^{(.021)(1.17)} )</td>
<td>1.025</td>
<td>(43,215)</td>
<td>1.025</td>
</tr>
<tr>
<td>1.50 (June 1963)</td>
<td>( e^{(.021)(1.50)} )</td>
<td>1.032</td>
<td>(43,215)</td>
<td>1.032</td>
</tr>
<tr>
<td>1.67 (August 1963)</td>
<td>( e^{(.021)(1.67)} )</td>
<td>1.036</td>
<td>(43,215)</td>
<td>1.040</td>
</tr>
<tr>
<td>2.58 (July 1964)</td>
<td>( e^{(.021)(2.58)} )</td>
<td>1.056</td>
<td>(43,215)</td>
<td>1.069</td>
</tr>
<tr>
<td>3.92 (November 1965)</td>
<td>( e^{(.021)(3.92)} )</td>
<td>1.086</td>
<td>(43,215)</td>
<td>1.088</td>
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</table>
Figure 5 shows the comparison of the actual and theoretical decay ratios as a function of time for the Cs $^{137}$ source. From this, it appears that the decrease in self-standard counts for the density gage illustrated in Figure 4 can be attributed to decay of the source. For the BaBe source the theoretical decay ratio at time ($t = 4$ yrs) would be $e^{(0.00043)4} = 1.00174$. This value is also plotted in Figure 5. As pointed out previously, the decrease in self-standard counts for the moisture gage utilizing the BaBe source was almost negligible.

It has been stated that although the self-standard readings for the density gage decreased due to decay, the use of a count ratio tended to correct for the decay. It must be emphasized that Pocock (10) has shown mathematically that the use of a count ratio procedure will not completely eliminate variations due to source deterioration.

In his paper Pocock states:

"It becomes apparent that use of the count-in-soil to count-in-standard ratio will not eliminate the effect on the calibration curve of half-life in reducing source strength in practice. Yet, although use of the ratio will not eliminate the effect of half-life, it is possible that its use may reduce this effect."

He further states:

"It appears......that use of the ratio, for the purpose of lessening the effect on the calibration curve of reduction of source strength with time as a function of half-life is justifiable on theoretical grounds. It should be emphasized that use of the ratio
FIG. 5. COMPARISON OF ACTUAL AND THEORETICAL DECAY RATIOS
will not eliminate the need for recalibration, but will merely serve to reduce the required frequency of recalibration."

Interpreting the data obtained from Figure 4 along with Mr. Pocock's discussion, it can be concluded that for a testing period of approximately 2.5 yrs the use of the count ratio eliminated the need to recalibrate the nuclear gage containing the Cs $^{137}$ source. It can not, however, be concluded due to the limited test interval, that recalibration will never be required when the count ratio is utilized.

**Timer Accuracy**

The ability of a timer to consistently and accurately measure a one minute interval is undoubtedly important. However, it may be noted that if a count ratio procedure is adapted, consistency of the time interval is more important than accuracy of measurement since any deviation in time will be the same for both the self-standard cpm and cpm obtained on the measured variable. If the timer is inconsistent in measuring the time interval, both procedures; count per minute and a count ratio, will be in error.

Throughout the testing period, instruments were periodically checked with a 0.1 second calibrated stopwatch. The results showed a high degree of accuracy and repeatability for a 1 minute time interval.

**Battery Voltage**

The nuclear gages tested are equipped with an internal 6 volt battery for portable operation. Voltage variations between 5.5 volts and 6.5 volts were achieved by use of a 50 watt, 25 ohm rheostat connected to a 12 volt external battery with subsequent leads to the internal battery
terminals.

Figure 6 in an illustration of the effect of battery voltage upon readings from one of the density and moisture gages. For the density gage, variation of the self-standard between 5.5 volts and 6.5 volts was 495 cpm. Although extreme readings (5.5 volts and 6.5 volts) are within the reliable error for the self-standard count obtained at 6.0 volts, a definite trend of counts with voltage increase is noted. However, the count ratio for readings taken on the concrete calibration block were practically constant at all voltage levels. The variation of the self-standard for the moisture unit was within the reliable error and considered negligible. It should be noted that at 5.5 volts the moisture unit did not function for the fourth count reading.

Parsons and Lewis (7) state in their discussion of nuclear instruments that:

"For consistent operation of the apparatus for both density and moisture content determination a supply voltage very close to 6 volts would be required."

Temperature

If the nuclear gage is to consistently perform its functional use, repeatability of results for temperature variations become an important matter. Tests were conducted on several gages at various temperatures and time intervals in a cold room before test initiation.

The effect of temperature is summarized in Table 2. It can be seen that the effect of temperature is dependent upon the inherent stability of a particular instrument, as evidenced by the malfunction of Instrument
FIG. 6. EFFECT OF BATTERY VOLTAGE UPON NUCLEAR READINGS
2 at a test temperature Instrument 1 functioned. For Instrument 1, when the gage was placed in the cold room for 2 hours before testing, a 357 cpm decrease and 244 cpm increase existed for the density gage and moisture gage, respectively, for a temperature range of 75°F. When placed in the cold room for 5 hours before testing, a density cpm reduction of 440 cpm occurred at a temperature of 42°F. Therefore the test temperature cannot be used independently without noting the effect of length of the time the gage has been subjected to the temperature.

Subsequently, a definite period of time is needed for the gage to become equalized with the test temperature.

Table 2

Effect of Temperature Upon Counts

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Density Gage Error</th>
<th>Moisture Gage Error</th>
</tr>
</thead>
<tbody>
<tr>
<td>75°F</td>
<td>42022 +337</td>
<td>19237 +232</td>
</tr>
<tr>
<td>0°F</td>
<td>41665</td>
<td>19481</td>
</tr>
<tr>
<td>83°F</td>
<td>43215 +342</td>
<td></td>
</tr>
<tr>
<td>42°F</td>
<td>42775</td>
<td></td>
</tr>
</tbody>
</table>

Instrument No. 1
(Instrument placed in cold room 2 hours before testing)

Instrument No. 2
(Instrument placed in cold room 2 hours before testing)

75°F 19195 2240
0°F Gage Inoperative Gage Inoperative
Parsons and Lewis (7) have shown for a density gage, count variations can be considered negligible for temperatures ranging from $32^\circ F$ to $104^\circ F$. For the moisture unit, count stability was more critical; as count increases were noted for temperatures below $44^\circ F$ and a consequent count reduction for temperature in excess of $86^\circ F$.

**Procedural Factors**

**Standard Systems**

As previously mentioned, the accuracy and repeatability of a nuclear instrument is of paramount importance. To provide a technique of periodically checking gage performance, a "standard" density or moisture system must be utilized. A standard system can be thought of as a substrate material that can be either a mass system (density) or neutron moderating system (moisture) whose volume of influence for a given test variable will remain constant with time.

Once a standard system has been established, it can serve two primary functions. First, by creating several systems at variable densities or neutron moderating capabilities, a single calibration curve can be developed for the gage in question, provided the standard material has an atomic structure similar to the atomic structure of the tested material. The second function has previously been mentioned, i.e. provide a system to check the stability of the gage.

Utilization of a standard system for the purpose of obtaining a single calibration curve adaptable for field usage has many limitations. Literature concerning the development of these standards have been published by several people and scientific agencies (2) (3). Extreme care must be
taken to insure that a homogeneous material is used and, in particular, test variable gradients with depth must be minimized.

In contrast, use of the standard merely as a check of the system, is feasible so long as one condition is met. This condition is that identical volume of influences must be maintained. A standard system for checking the instrument need not be homogeneous, may contain test variable gradients in a vertical and horizontal direction, and as long as it contains mass and or a neutron moderation capability it is an adequate system. If the placement of the nuclear gage is restricted to the same position on the standard every time a standard reading is obtained, the gage will fulfill the condition of "seeing" an identical zone or volume of influence. This concept was successfully used throughout the test project.

Generally speaking, standard systems can be categorized into two types, (1) instrument self standards and (2) non-instrument standards. In this study, the use of heavy liquids was investigated to determine their feasibility as non-instrument standard systems. Carbon tetrachloride and tetrabromoethane were combined to achieve a range in density from 99.6 pcf to 161.7 pcf. Figure 16 shows the density calibration curve for the liquids tested.

The principal advantages of liquid as a standard are its homogeneity and the fact that the liquid can be brought to the same unit weight at any reasonable temperature, thus ensuring absolute control over the standard. As evidenced by the different calibration curves in Figure 16, the primary disadvantage of liquids for defining a calibration curve is
that they do not possess the same atomic structure as typical soils encountered in practice. Data from this study suggested that it is not possible to use heavy liquids to develop a calibration curve which will be applicable to soils. However, since the reproducibility and stability of the instrument are independent of the material tested, the use of heavy liquids as a suitable standard is unquestioned.

Due to their immobility and bulkiness, non-instrument standards are not readily adaptable to giving the user an immediate check on the overall stability or to serve as a reference count for the count ratio when conducting field testing. Consequently, all nuclear gages used contain a self standard system so that portability is attained or "built in" the gage itself. Self standard readings, for both density and moisture, were taken throughout the entire testing program. During the initial phase of testing in the laboratory it was noticed that extreme variances in the self standards occurred between daily tests. A wooden stand, 12 inches high, was constructed to permit an "air gap" to exist between the floor and bottom of the measuring device. Figures 7 and 8 are plots of the self standard readings versus test number. It can been seen that for tests taken with the air gap, the self standard readings for both moisture and density fell within the reliable error. It should be pointed out that for Instrument No. 1 on Figure 8 (moisture gage) the self standard without the constructed air gap also fell within the reliable error since this particular gage has an air gap built into its standard block.

Therefore, it was concluded that self standard readings obtained during testing, were influenced by the material directly under the gage.
FIG. 7 EFFECT OF STANDARD AIR GAP UNDER DENSITY GAGE
UPON SELF STANDARD READINGS
FIG. 8  EFFECT OF STANDARD AIR GAP UNDER MOISTURE GAGE UPON SELF STANDARD READINGS
To eliminate this effect, use of an air gap between the gage and substrate is recommended to provide self standard readings within the reliable error.

Count Ratio vs Counts Per Minute

It has been stated that nuclear readings can either be expressed as counts per minute (cpm) or as a count ratio (relative count). However, since the value of the nuclear reading is also a function of the operating high voltage value of the instrument, data may be reported using either a constant or variable high voltage procedure.

Figure 9 illustrates the above based on the data for Figure 2. The dotted line represents the anticipated plateau curve at a time \( t'' \) when the self standard count \( (C''_U) \) at a high voltage setting equal to the upper limit high voltage \( (HV = U) \) is identical to the initial self standard count \( (C_A) \) obtained at a high voltage setting \( HV = A \).

Three methods of expressing the results are shown in Figure 9. The procedure that utilizes results expressed as counts per minute is attained by varying the high voltage to maintain the original self standard counts. Thus, if \( C_A \) represents the initial self standard obtained at time \( (t'_0) \) and \( C'_B \) represents the same self standard reading at time \( (t') \), the high voltage would have to be varied from \( HV = A \) to \( HV = B \). The particular nuclear count obtained on any substrate material would then be the result expressed in cpm.

If a count ratio procedure utilizing variable high voltage is used, the second procedure is defined. As the voltage is varied from \( HV = A \) to \( HV = B \), the count ratio on the standard block at time \( (t') \) would change
FIG. 9. ILLUSTRATION OF DENSITY GAGE READINGS WITH PLATEAU VOLTAGE
from $R_A$ to $R_B$.

The count ratio of the standard block for the third method is obtained by keeping the operating high voltage constant. If the initial count ratio for the standard block at time $(t_0)$ is $R_A$, the count ratio at time $(t')$ would then also be $R_A$.

Based on these data, the best procedure to employ when using a nuclear gage is to express the results in the form of a count ratio obtained by keeping the operating high voltage constant at any given time. This is explained in the following manner:

If a count per minute method is used with variable voltage to achieve the initial self standard reading, three facts are noted. (1) Although the self standard reading has been kept constant ($C_A = C_B = C_U$) the reading obtained on a substrate material (in this case a standard concrete block) will increase as the high voltage is varied with time to obtain the initial self standard reading. This is shown by noting that the count ratio ($R$) curve on the standard block increases with high voltage setting but does not change as a function of time. (2) There will be a definite time period when recalibration will be necessary due to (a) a change in the standard block reading, from time $(t_0)$ to $(t')$, becoming equal to or greater than the upper reliable error for the count of the standard block reading obtained at time $(t_0)$ or (b) an increase in high voltage that results in a voltage greater than the upper limit of the plateau operating range ($HV = U$); (3) use of cpm will not eliminate any variations due to instrument instability.

For the count ratio procedure also using a varying voltage, the results are identical to the cpm procedure described above with the exception that the count ratio procedure will eliminate several effects
due to instrument stability and physical surrounding as stated in the section concerning Instrument Stability of this text. However, since the high voltage is variable, the use of the count ratio in this procedure (variable high voltage) will not eliminate any effects of time (aging). This is illustrated by the following:

When:

\[ t = t_0 : \text{HV} = A, \text{Count Per Minute} = C_A, \text{Count Ratio} = R_A \]

\[ t = t^9 : \text{HV} = B, \text{Count Per Minute} = C_B, \text{Count Ratio} = R_B \]

however, from Figure 9

\[ R_A \neq R_B \]

The operating high voltage is a function of time as the high voltage is varied with time to obtain a constant self standard reading. The time required for recalibration can be measured in terms of the high voltage necessary to produce the two cases previously mentioned knowing only the self standard and count ratio plateau curves at time \( t_0 \).

It is assumed in this example that the recalibrated curve will be parallel to the original calibration curve (ie....the slope of the count ratio curve for the standard block, a discrete density value, would be equal to slopes of all count ratio curves obtained in a similar manner at any given density). Although no data were obtained for various standard block densities, the slope of the curves are dependent only upon the electronic system used in the particular gage. Therefore, for a given nuclear gage, the assumption of parallel recalibration curves seems to be valid.
For the data obtained in Figure 2, the high voltage at which the count ratio of the standard block was equal to the initial count ratio plus the upper reliable error was $HV = B = 1110$ volts. This corresponded to a time of approximately 1.8 yrs. In other words, if the count ratio procedure with a variable voltage had been used, the gage would have had to been recalibrated 1.8 years from the date testing was initiated.

The upper limit of the plateau H.V. for the gage tested was 1200 volts. Consequently, once the operating high voltage had been varied from the initial operating voltage ($HV = A = 1000$ volts) to the upper limit high voltage ($HV = U = 1200$ volts) the gage would also have to be recalibrated. An approximate time for recalibration can be found by equating the actual decay ratio (at $HV = 1200$ volts; $C_A = C''_U = 43.215$; and $C_U = 47.500$) to the theoretical decay ratio $e^{\lambda t}$ for a Cs$^{137}$ source:

$$e^{\lambda t} = \frac{C''_U}{C_U}$$

$$e^{(.021)t} = \frac{47.500}{43.215}$$

$$e^{(.021)t} = 1.099$$

$$\ln 1.099 = \frac{t}{(.021)}$$

$$t = \frac{.0943}{.021}$$

$$t = 4.5\text{ yrs.}$$

Therefore, it can be stated that if a count ratio with a varying high voltage procedure was used to express nuclear readings, it would take approximately 4.5 yrs for the high voltage setting to reach the upper limit of the plateau operating voltage ($HV = U$) for the data.
presented in Figure 2.

For the count ratio procedure using a constant high voltage at any given time the following results are stated. The count ratio obtained on a standard concrete block remained constant over a period of approximately 2.5 years. Thus, for a period of 2.5 years the use of the count ratio not only eliminated the effects of variations in readings caused by instrument stability (instability); it also eliminated the effects of aging due to source decay. Figure 10 illustrates the effect of data scatter reduction obtained on a nuclear moisture gage employing a RaBe source. As the RaBe source has been shown to have a negligible effect on nuclear reading variations on a standard system over a period of time due to its long half-life (see Figure 3) the scatter reduction can be attributed mainly to variations of instrument stability factors.

However, as previously mentioned in this report, it cannot be conclusively stated, due to the limited time interval, that the use of a count ratio will completely eliminate the effect of source decay on nuclear readings. The important concept to be gained is that a count ratio procedure used at a constant high voltage will require recalibration at less frequent intervals than if a count ratio using a varying voltage procedure were utilized.

Consequently, if it is assumed that the life of the instrument electronic system will be more than 1.8 yrs; recalibration of nuclear instruments using a Cs$^{137}$ source, will require recalibration due to source decay (aging) provided the method used to express results is a count-ratio-varying high voltage.
FIG. 10. COMPARISON OF COUNT RATIO AND COUNT PER MINUTE PROCEDURE FOR MOISTURE CALIBRATION.

COUNT RATIO

POUNDS PER CUBIC FOOT

MOISTURE - CPM x 10^3
If a count ratio-constant high voltage procedure is used to express the data, no definite time required for recalibration can be made due to the fact that the data obtained indicate a constant count ratio on the standard block for 2.5 yrs. However, if it is assumed that the argument presented by Mr. Pocock (10) is correct concerning the fact that the count ratio will not completely eliminate the effect of aging; then the statistic of importance depends on the time required for the count ratio, at a constant high voltage \( R_{A} \), to change to \( R'_{A} \) where
\[ R'_{A} - R_{A} \geq \text{reliable error of } (R_{A}). \]
Consequently, since this difference in count ratios was negligible for a 2.5 yr period; the time required to cause a recalibration (count ratio-constant high voltage) for a nuclear gage using a Cs\(^{137} \) source might be far longer than the time required for a recalibration necessitated by an electronic failure. Therefore, it is felt the primary reason for recalibrating a nuclear gage containing a Cs\(^{137} \) source will be due primarily to electronic failure, provided a count ratio - constant high voltage procedure is used.

It should be noted that the general results and procedures stated can be adapted to any nuclear gage (density or moisture) using any nuclear source. However, it must be emphasized that the relative magnitude of these results is a function of the source (half-life) used in the nuclear gage.

Leveling Course

In use, the nuclear gage must be in firm contact with the substrate to allow a minimum of voids between the top of the soil and bottom of the gage. To investigate this effect, a 1/8 inch thick leveling course was placed on the substrate surface. All results represented in this
section were obtained in the laboratory. The density of the material represents the bulk unit weight of the material in the test container before the leveling course was placed on the surface. Conclusions are based on the assumption that surfaces free from irregularities were obtained on the substrate.

Figure 11 illustrates the effect of a leveling course on a sand-soil mix. For this material, results indicate that the use of a leveling course produced a better calibration curve in that a reduction of data scattering for the material was noticed.

Further tests were conducted using leveling courses on aggregates and soil-aggregate mixtures. These materials were graded according to the formula $p = 100\left(\frac{d}{D}\right)^n$; the open graded material had $n = 1.2$, $D = 3/4"$ (maximum aggregate size) and the soil-aggregate had $n = 0.5$ and $D = 3/8"$. Density count ratios were determined for the materials without a leveling course and with a leveling course.

Figure 12 represents the difference between density count ratios obtained with a leveling course and without a leveling course at various densities. The open graded aggregate had a larger difference in count ratios than the dense graded soil aggregate mixture. Generally, the difference in count ratios tended to decrease with an increase in density. This is more evident in Instrument 1 than is shown by Instrument 2.

It was also noted that for both the open graded aggregate and soil-aggregate mixture, no reduction in data scattering was evident when readings taken with a leveling course were compared to those taken without a leveling course.
LEGEND
--- WITHOUT LEVELING COURSE
--- WITH LEVELING COURSE

COUNT RATIO

BULK UNIT WEIGHT - POUNDS PER CUBIC FOOT

FIG 11  EFFECT OF LEVELING COURSE ON DENSITY READINGS FOR A SAND SOIL MIX
FIG. 12  DIFFERENCE OF DENSITY COUNT RATIO READINGS WITH LEVELING COURSE AND WITHOUT LEVELING COURSE FOR TWO GRADED MATERIALS
Because of the possible intrusion of the leveling course into the open graded material, the nuclear gage "sees" a different density than the true bulk density. As the density of the aggregate increases, less voids occur in the substrate, allowing less leveling course to intrude into the sample. For the dense graded material, intrusion is negligible at the surface. However, the possibility of the density of the leveling course influencing the readings in the form of a density gradient is apparent.

Although the thickness of the leveling course is kept approximately constant, the effect of a leveling course will not be the same for every density test. For a given source to detector distance, the effective depth of recorded photons, which are either absorbed or backscattered, is a function of the density of the material. This depth is commonly called the "depth of penetration" (see section of this text on Zone of Influence). The depth of penetration for a nuclear density gage decreases as density increases.

If the leveling course has a density equal to the density of the substrate, the effective depth will not change and the nuclear readings obtained without the leveling course and with the leveling course will be identical. If the leveling course has a density greater than the density of the substrate, the effective depth for the substrate with the leveling course will be reduced. Therefore, a reading obtained with the leveling course will be less than the reading without the leveling course. Conversely, if the layer density is less than the substrate density, the readings obtained with a leveling course will be greater than those
obtained without a leveling course.

The relationship for the dense graded soil aggregate mix of Figure 12 substantiates the above reasoning. Assuming a constant leveling course thickness for all tests, it can be noted that at low densities the count ratios without a leveling course was greater than the count ratio with a leveling course; at high densities the converse was true.

The results indicate that the use of a leveling course to reduce data scatter for soil aggregates can be questioned, and that actual readings recorded with a leveling course are a function of the leveling course thickness, substrate density, and gradation of the substrate. However, as field surfaces rarely exist free of irregularities, the evaluation of leveling course usage lies in the relative magnitude of errors introduced by improper seating of the gage if a leveling is not used or by the leveling course itself when used.

It is recommended that an attempt should be made to obtain as flat a surface as possible for proper gage seating to minimize the error of seating. If voids are still existant after this attempt, use of a leveling course is suggested provided the thinnest layer possible is applied to the substrate. It is believed that the optimum solution to the seating problem is to consistently produce a controlled air gap under the gage to eliminate this effect.

**Substrate Materials**

**Zone of Influence**

As radiation is emitted from a source in a backscatter gage, a limit of distance is imposed upon a radioactive particle which can
penetrate the substrate, undergo the physical reactions of the system, and yet possess sufficient energy to permit detection by the nuclear gage.

It has been known that density and moisture nuclear readings are dependent upon the distance between the source and detector tube of the nuclear gage. Carey and Reynolds (4) have investigated the possibility of measuring density at various depths by adjusting the distance between the source and detector tube. It has also been found that the depth of measurement is dependent upon the magnitude of the test variable (density or moisture). Therefore for a particular gage (i.e., constant source to detector tube distance), the volume or zone of influence is dependent only upon density (density zone of influence) or amount of moisture (moisture zone of influence) of the substrate material.

To establish the depth of penetration as a function of density, heavy liquids were studied. The liquids investigated were:

1. Sym-tetrabromoethane (Specific Gravity = 2.9638, $\frac{20^\circ C}{4^\circ C}$)
2. Carbon tetrachloride (Specific Gravity = 1.595, $\frac{20^\circ C}{4^\circ C}$)

To obtain variable densities, the liquids were blended to yield the desired intermediate densities. By recording the nuclear readings of a particular liquid (density) at various depths, the depth of penetration at a known density was obtained. Figure 13 shows the approximate depth at which the particular nuclear gage recorded consistent readings for a given heavy liquid density. Tests conducted on materials in containers of varying diameter indicated that a container diameter needed to eliminate edge effects was at least 20 inches.
FIG. 13  APPROXIMATE DEPTH OF PENETRATION VERSUS BULK UNIT WEIGHT - INSTRUMENT I (INTERPOLATED FROM BURGERS)
Depth of penetration tests for both density and moisture were also conducted on soils with two different instruments. Unfortunately, data obtained on soils with the instrument used in the heavy liquid study were inconclusive. Data for depth of penetration in soil were determined by adding successive layers of soil on top of the sample and recording nuclear readings as the depth increased. Owing to the difficulty in controlling the unit weight and weight of water for each layer, it was extremely difficult to measure precisely the depth of penetration for density and moisture. Figure 14 shows the approximate depth of penetration of soils for density and moisture. In both Figures 13 and 14, it can be seen that the depth of density penetration decreased with an increase in density. Similarly, as the quantity of moisture per unit volume in the soil was increased the moisture gage depth of penetration decreased.

The primary reason why depth of penetration curves developed on heavy liquids cannot be substituted directly for soils at the present time is the limited amount of knowledge concerning the relative effects upon nuclear gages for materials with differing atomic structures.

Because of the numerous advantages and relative ease of using heavy liquids in contrast to soils to establish density depth of penetration curves, it is recommended that further research be conducted to determine if the use of heavy liquids can be utilized in practice.

Substrate Composition

Density Gage

Material Type

By far, the most important item of conjecture in the application of nuclear density gages to field use has been that of influence of material type upon density readings. By using a single calibration
FIG. 14 APPROXIMATE DEPTH OF PENETRATION VERSUS BULK UNIT WEIGHT AND MOISTURE-INSTRUMENT 2
curve, or assuming that all material types will respond identically to
the substrate system at a given density, the assumption of equal mass
absorption coefficients, $\mu_n$, for all material types is made. However,
mass absorption coefficients, $\mu_n$, are a function of both the nuclear
particle energy and type of element. Values of mass absorption coefficients
and its dependence upon elements commonly found in soils has been given
by Parsons and Lewis (7) and is shown in Figure 15.

At energy ranges higher than about 0.3 Mev, it can be seen that
the absorption coefficients for all elements shown, with the exception
of hydrogen, are relatively constant. Conversely, wide variations
between coefficients are evident for energy ranges less than 0.3 Mev.
Figure 16 illustrates calibration curves for the heavy liquids previously
mentioned, a crushed limestone and a crushed quartzite. Both crushed
materials had identical grain size distribution curves graded to $p = 100$
$\left(\frac{-d}{D}\right)^n$ where $D = 3/8"$ and $n = 0.5$.

In July 1965, the nuclear gages were taken to Charlottesville, Virginia
for the Correlation and Conference of Portable Nuclear Density and Moisture
Systems conducted by the University of Virginia. The nuclear density gages
were calibrated on a series of calibration blocks for the Virginia study.
The chemical analysis of each of these blocks, along with a chemical analysis
of the crushed limestone and crushed quartzite studied at Purdue, is shown
in Table 3. It is seen that Blocks No. 1, 2, and 5 have an appreciable
quantity of silicon dioxide (SiO$_2$) and are similar in chemical composition
to the crushed quartzite studied. Similarly, blocks No. 3 and 4 are
similar to the crushed limestone in that the predominant chemical compound
FIG. 15 RELATIONS BETWEEN MASS ABSORPTION COEFFICIENT AND THE ENERGY OF GAMMA RADIATION FOR ELEMENTS COMMONLY FOUND IN SOIL (FROM PARSONS AND LEWIS)
LEGEND
○ QUARTZITE (PURDUE LAB., MARCH, 1963)
● CALIBRATION BLOCK - SiO₂ PREDOMINANT CHEMICAL
  (VIRGINIA NUCLEAR CONFERENCE, JULY, 1965)
△ LIMESTONE (PURDUE LAB., MARCH, 1963)
▲ CALIBRATION BLOCK - CaO PREDOMINANT CHEMICAL
  (VIRGINIA NUCLEAR CONFERENCE, JULY, 1965)
X HEAVY LIQUID (PURDUE LAB., MARCH, 1962)

NOTE: Cs¹³⁷ SOURCE

FIG. 16 EFFECT OF MATERIAL TYPE ON NUCLEAR DENSITY READINGS
is calcium oxide (CaO). It is quite obvious that for the SiO₂ blocks the nuclear readings plot near the calibration curve established for the crushed quartzite obtained in the laboratory investigation. A similar relationship between the CaO blocks and the crushed limestone curve is also noted.

Table 3

Chemical Analysis of Selected Materials

<table>
<thead>
<tr>
<th>Material</th>
<th>SiO₂ (%)</th>
<th>CaO (%)</th>
<th>Other Chemicals (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Block No 1 (Virginia)</td>
<td>100</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>2 (Virginia)</td>
<td>74.2</td>
<td>-</td>
<td>25.8</td>
</tr>
<tr>
<td>3 (Virginia)</td>
<td>-</td>
<td>54.0</td>
<td>46.0</td>
</tr>
<tr>
<td>4 (Virginia)</td>
<td>-</td>
<td>55.8</td>
<td>44.2</td>
</tr>
<tr>
<td>5 (Virginia)</td>
<td>74.4</td>
<td>-</td>
<td>25.6</td>
</tr>
<tr>
<td>Crushed Quartzite</td>
<td>97.2</td>
<td>-</td>
<td>2.8</td>
</tr>
<tr>
<td>Crushed Limestone</td>
<td>12.1</td>
<td>47.1</td>
<td>40.8</td>
</tr>
</tbody>
</table>

Figure 16 shows the effect of material type for nuclear density gage with a Cs⁴⁷ source. For the nuclear gage with the RaBe source, identical patterns for the crushed materials and calibration blocks were noted with the exception that a larger deviation between the limestone and quartzite curves occurred. It is felt that a possible explanation for this event is directly related to the type of source used in each gage.

RaBe has the major portion of its energy spectra at two energy levels, 0.61 Mev and 0.35 Mev. Since a portion of the initial energy
is lost due to the physical events that occur in the system, and there also exists some radiation at energies of 0.18 Mev at the lower spectrum value, it is suggested that the possibility for radiation levels being found at or below the 0.3 Mev energy range is quite probable.

Since a Cs$^{137}$ radiation source has an energy level in which the initial level of the photon energy exists at 0.66 Mev, a subsequent smaller portion of photon energies may be expected to occur at the 0.3 Mev level. Therefore smaller deviations between chemically different soils may be expected to occur for a Cs$^{137}$ source due to its radiation energy spectrum existing at energy levels where mass absorption coefficients for most soil elements are almost identical.

As seen in Figure 15, for energy levels below 0.3 Mev, a rather large deviation in mass absorption coefficients occurs between calcium and silicon. Because of this, the concept of using soil pH as an indicator to correlate the mass absorption was used as a field expediant to determine material type.

It is recognized that the pH method can only be used in a general way to indicate material type due to the fact that it is possible for a large proportion of an element in a soil to produce a weak acid while a small proportion of another element may produce a strong base. The titrating effect of the weak acid and strong base solution would tend to yield a basic pH while physically speaking, the acidic element would generally dominate the overall average mass absorption coefficient for the soil. However, field tests were conducted using pH as an indicator due to its relative ease of use in the field in contrast to a more complicated procedure of obtaining a quantitative analysis of soil composition.
Figure 17 shows the results of nuclear density tests conducted in the field. All densities were obtained by the sand-cone method. It is seen that two distinct calibration curves were developed; one for basic soils and one for acidic soils. Assuming that a basic soil would generally correspond to those soils containing large quantities of calcium and iron, and acidic soils would correspond to elements shown in Figure 16 that are distinct from the calcium and iron for energy levels below 0.3 Mev, it would be expected that a basic soil will absorb more of the nuclear particles than an acidic soil (ie...fewer counts would be recorded and a basic soil calibration curve would then plot below an acidic soil calibration curve). The general relationship of the basic and acidic curves for data in Figure 17 tend to verify this concept.

The standard error of estimate for the basic soil curve was $\pm 0.010$ (count ratio) and $\pm 0.015$ (count ratio) for the acidic soil curve. The overall standard error of estimate for all data regardless of pH was $\pm 0.017$ (count ratio). Based on these results, it can be stated that although use of pH cannot be completely correlated to mass absorption coefficients (ie...material type), its application to field testing produced calibration curve parameters that reduce errors of estimate for the nuclear readings.

Moisture in Material Mass

The effect of material type (mass absorption coefficients) on density count readings has been previously discussed. As very few soils, either in a natural state or those applied to engineering applications, exist
FIG. 17 DENSITY CALIBRATION CURVES DEVELOPED BY SOIL pH GROUPING FOR FIELD DATA
in a dry state; the effect of moisture in the substrate material upon nuclear density readings was investigated.

It was concluded that no quantitative effects were noted for the data analyzed. In an earlier report, Burgers (2) noted the possibility of this effect existing (Figure 13). The results by Burgers were obtained on four different soils. However, in his work, soil type was generally associated with a given range of moisture contents, and effect of material type was neglected.

Since water possesses a discrete mass absorption coefficient, it is felt that moisture might have some effect on nuclear density readings. As of yet, a definite method of accurately determining the relative magnitude of this effect is unknown. A report from the Road Research Laboratory (7) states:

"It is estimated that the variation of calibration due to the hydrogen contained in water would not be more than 1-1.5 pounds per cubic foot in bulk density for a variation in moisture content of 10 per cent."

Grain Size Distribution

Reference has been made to the importance of mass absorption coefficients of various soil elements for nuclear determination. However, for a particular material type, the question arises, can similar mass absorption coefficients be defined at various compositions of the soil, or at different grain size distributions?

To investigate this effect, two materials were tested. They were subsequently crushed and hand picked to produce a finer (denser) gradation.
FIG. 18 LABORATORY CALIBRATION CURVE SHOWING EFFECT OF MOISTURE CONTENT UPON NUCLEAR DENSITY READINGS
For a given gradation, each material was blended to yield identical grain size distributions. Results of this portion of the study are shown in Figure 19.

Both instruments tested produced similar count reductions for both the quartzite and limestone materials as the open graded material was crushed finer. From the results shown, it is concluded that the nuclear gage did not "record" identical mass absorption coefficients for the same material at the grain size distribution indicated on Figure 19. Field data tend to substantiate this concept as illustrated by Figure 20. The figure is representative of all basic (pH>7.3) field materials plotted as granular versus fine grained.

However, it should be noted, that it is not felt that for every possible grain size distribution for a given material type, deviations between calibration curves can be expected to exist. Rather, it is felt that at a certain state of grain size distribution, this effect is negligible. The data shown are indicative only for aggregates and soil aggregate mixes and are limited in quantity, preventing a general conclusion to be expressed.

Moisture Gage

Material Type

The concepts involved in correlating moderation and absorption of fast neutrons to the amount of water in a substrate mass has been briefly stated in the section "Principles of Operation" of this report. Although hydrogen (water) is both a low absorber and a highly effective moderator, it is not the only element that produces these physical reactions.

The results of both the laboratory and field tests indicated that, for the soil materials investigated, material type had no apparent effect
FIG. 19 EFFECT OF GRAIN SIZE DISTRIBUTION UPON NUCLEAR DENSITY READINGS FOR SELECTED MATERIALS
FIG. 20  COMPARISON OF FIELD CALIBRATION CURVES FOR GRANULAR AND FINE GRAINED MATERIALS (pH > 7.3)
upon results. Figure 21 shows the moisture calibration curves developed from the laboratory and field investigations.

Burn (3) has found that the absorption effect of iron and potassium present in a Leda clay caused variations from the laboratory calibration curve obtained on inert soil substitutes. He states:

"The absorption effect of potassium on the neutron flux was investigated... (it) was found to cause a significant decrease in counting rate."

He further adds:

"The absorption effect of iron was... found to have a significant effect on the counting rate. At the measured proportions of iron in Leda clay, the decrease in neutron activity would account for 85 to 90 per cent of the observed discrepancy."

Conventional Methods of Determining Moisture Content

For the data obtained in Figure 21, it is evident that the field calibration curve plots above the laboratory curve. The largest deviation between curves occurs at low moisture values. It is felt that this effect can be attributed partially to the difference in obtaining field moisture contents in comparison to the relative ease and accuracy of laboratory procedures.

A further possible explanation for the discrepancy between curves may be found in the fact that at relatively low moisture values, the nuclear gage measures an average moisture over a depth of approximately 8 to 10 inches (See Figure 14). Standard moisture content determinations were performed
FIG. 21. A COMPARISON OF FIELD AND LABORATORY MOISTURE CALIBRATION CURVES
on material obtained from the sand cone density hole, which in general, did not exceed 6 inches in depth. Therefore, if a moisture gradient caused by drying of the surface existed, the gage would record a larger moisture reading than the corresponding standard moisture content determination.

Although conventional oven drying tests are standardized, the test procedure for soils containing a high clay content may lead to erroneous results. Partridge and Ridgen (8) have shown the effect of clay content on moisture measurements. This effect is illustrated in Figure 22.

A moisture gage will measure all neutron moderation encompassed in the substrate system. As the clay content is increased in a soil containing water, an increased resistance to the bound water being driven out of the system by heat is encountered. Although a nuclear moisture unit will measure all forms of hydrogen present, conventional oven drying tests under standardized conditions do not effectively drive out all the water in the heavy clay system. As a result, the nuclear method measures one quantity of water present while the conventional oven test measures a reduced quantity of water.
FIG. 22. EFFECT OF CLAY CONTENT ON MOISTURE MEASUREMENTS (PARTDRIGE AND RIGDEN)
SUMMARY

Instrument Stability

1. Throughout the test program the nuclear self standard counts per minute for the gage with the Cs\textsuperscript{137} source decreased according to the natural law of radioactive decay.

2. For the nuclear gage using a RaBe source, the decrease in self standard counts per minute was within the reliable error for the entire test period.

3. All instruments showed a high degree of timer accuracy (± 0.1 sec) for the entire test period.

4. For consistent count per minute readings, internal battery voltage should be close to 6 volts; however, use of a count ratio procedure will eliminate voltage effects.

5. Operating temperatures for nuclear testing are dependent upon instrument type. The operating temperature range is more restrictive for moisture readings than for density readings.

Procedural Factors

1. Heavy liquids cannot be used to determine a density calibration curve for soils due to their differing atomic structures. They can, however, serve as excellent standards for an overall periodic system check.

2. A standard system need not be homogeneous if it will be used purely as a periodic system check. However, control over gage placement and orientation must be exercised throughout the testing period.
3. Use of an air gap between the bottom of the nuclear gage and the surface on which it rested minimized variations in the standard readings.

4. The use of a count ratio procedure at constant high voltage eliminated aging (source decay) effects for the gage containing Cs$^{137}$ over a 2.5 year test period.

5. Aging effects for the nuclear gage with RaBe source were not evident for the test period due to its long half-life.

6. The use of a count ratio at variable high voltage for the purpose of maintaining a self standard reading within the reliable error limits did not eliminate the effect of aging or source decay.

7. Use of a count ratio at both constant and variable high voltage resulted in reduced data scatter over identical test results expressed as counts per minute.

8. Use of a leveling course under the nuclear gage to reduce air voids was found to be of questionable value.

**Substrate Material**

1. The depth of nuclear particle penetration is a function of source to detector distance and magnitude of the test variable (density or moisture).

2. Material type (chemical composition) affects nuclear density readings due to variable mass absorption coefficients of the elements involved.

3. The magnitude of variation between calibration curves for various material types was a function of the type of radioactive source.
4. Soil material pH served as a reasonable indicator of material type in reducing errors of estimate associated with densities determined from a single calibration curve.

5. No categorical effects of moisture content on density readings were noted for the soils tested.

6. For a given mass density, the fine (dense) graded materials recorded less nuclear density counts than the same materials at a more coarse (open) gradation.

7. Material type did not influence the moisture gage readings for the soils studied, although there is evidence that soil elements other than hydrogen will influence soil moisture readings.
SELECTED REFERENCES


RECOMMENDED PROCEDURES FOR USE OF NUCLEAR GAGES

This section presents guidelines for the use of nuclear moisture density gages for routine compaction control in Indiana. However, the concepts discussed can be adopted to any locality with certain modifications. The following procedures are based on experiences gained in this study; it is felt that by following the recommended format, reliable results can be obtained with these instruments. It should be noted that these recommendations are not considered to be the ultimate, but represent the best techniques developed up to the present time. Furthermore, the following general procedures apply to all nuclear gages, regardless of manufacturer.

I. General Concepts Involved in the Use of Nuclear Gages

A. Method of Reporting Results - The use of a count ratio procedure at constant voltage (as defined in the main text of this report) should be adopted for expressing all nuclear counts for the gages.

B. Air Gap - In order to achieve more consistent readings, the use of an air gap to obtain the instrument standard readings is recommended. The same air gap device can be used with all gages and it need not be elaborate in construction. A simple wooden platform approximately twelve inches in height has been found to be highly satisfactory.

C. Leveling Course - It is highly desirable to obtain a flat surface on the test area in order to insure proper seating of the gage. It is recommended that a leveling course be used on
the soil only if surface irregularities cannot be eliminated under the gage. This leveling course should be the same soil as that being tested and should be placed in the thinnest possible layer. The leveling course can best be obtained by sieving a representative quantity of soil from an area adjacent to the actual test site through a No. 10 or 20 mesh sieve onto the pre-leveled test area.

A recent development in this area of research has been the use of a constant air gap between the gage and the soil while determining a nuclear reading. This air gap minimizes the problem of seating the gage. The recommended air gap is usually 1/2 inch although this is not a set limit. It is recommended that further work along these lines be investigated and this technique possibly be adopted in the future in lieu of the leveling course technique.

D. Standard Blocks - A standard reference block should be used to insure proper functioning of the density gage as well as check reproducibility of results. The blocks can also be used to establish count ratio plateau curves for the gages. Three conditions should be met: (1) each gage should have its own individual block (2) the blocks should be made of a material which will not change density or chemical composition with time and (3) the gage should always be placed on this block in the same orientation. These conditions are necessary to insure that the gage is influenced by the same volume of material for all readings. As the block does not need to be homogeneous in order to
serve as a standard, it is suggested that concrete blocks of not less than 24" x 24" x 12" in size be made for each gage. These blocks should be stored in some central location where periodic check tests can be performed. These tests should be performed at least every three months. When the count ratio of the standard block at a given voltage is outside of the established reliable error, it will be necessary to recalibrate the gage. This is especially critical for gages utilizing sources which have a relatively short half-life. Any adjustments or changes made on the equipment by the manufacturer will also necessitate checking the gage to determine if recalibration is warranted.

II. Moisture Gages

A. Calibration Curves - It is recommended that the laboratory moisture calibration curves as shown by Figures 23 and 24 be adopted for field use. If the gage is to be utilized on granular materials at low moisture contents, a comparison of the expected depth of penetration and depth of the granular material should be made. In using the moisture gages the following procedure is recommended.

1. On each project where the gages are to be used several check tests should be performed to insure that the calibration curve is valid for that project before actually using the data for moisture control. These check tests may be made by following the steps indicated below:

   a) Test sites on typical soils should be prepared by providing a smooth level surface on which to place the nuclear gage.
FIG. 23. MOISTURE CALIBRATION CURVE
FOR INSTRUMENT NO. 1

$M = 45.30(CR) - 0.765$
Nuclear counts should be taken on the test area and an average count ratio determined.

b) A sand cone density test should then be performed on the exact area where the nuclear gage had been placed. The sand cone density obtained is used to determine the pounds of water per cubic foot of soil and to serve as density gage check tests which are discussed in a later section of this appendix.

c) The moisture content of the material taken from the density hole should be determined by standard laboratory oven-dry techniques. If this cannot be accomplished, field moisture determinations should be made using one of several techniques now in use.

d) After obtaining values for the dry density and moisture content in per cent, the moisture data should be converted to pounds of water per cubic foot. This value should then be plotted with its corresponding nuclear count ratio on the laboratory calibration curve.

2. The required number of check tests should be determined from Figure 29 for the particular Type II error ($\beta$) desired.

3. The suggested format to be used in either accepting or rejecting the moisture calibration curve based on the results of the check tests is shown in Appendix B.

III. Density Gages

A. Calibration Curves - Density calibration curves for Instrument No. 1 are presented in this Appendix as Figures 25, and 26. These
FIG. 25 DENSITY CALIBRATION CURVES FOR FINE-GRAINED SOILS FOR INSTRUMENT NO. 1
FIG. 26 DENSITY CALIBRATION CURVE FOR GRANULAR MATERIALS FOR INSTRUMENT NO. 1

\[ D = -253.4 \times (CR) + 186.88 \]
curves were obtained under field conditions and are recommended for use in field compaction control. The curves are based on two primary types of soils: (1) subgrade or embankment soils and (2) subbase materials. The first category is further subdivided on the basis of soil pH into a basic and acidic classification.

In order to utilize the density gage, the following procedure is recommended.

1. On a given project, several check tests should be performed on the typical soils involved. In making these tests, a procedure similar to that described for the moisture gages should be followed (as pertains to obtaining a flat surface, etc.) It is necessary to obtain an average count ratio and a sand cone density for each check test. Also, for the subgrade or embankment soils, it will be necessary to measure the pH of the soil. This measurement can be made by either using a portable, battery operated pH meter or by using soil color charts. Both of these tests are simple to perform and the equipment involved is relatively inexpensive.

2. The required number of tests necessary to ascertain the validity of a given calibration curve as well as a suggested format that allows a statistical decision to accept or reject the curve are presented in Appendix B.

3. If the decision to reject the calibration curve is made a new calibration curve should be developed. This is accomplished by performing further tests on the construction materials and
plotting this data as count ratio versus wet density as determined by the sand cone test. The number of tests required to establish a calibration curve will vary from material to material and the final judgement will have to be made by the engineer.

IV. Summary - The recommended procedure presented in the preceding paragraphs should make it possible to adopt the nuclear gages for the routine control of field compaction. It is felt that as field data are collected, a further insight into the method of obtaining calibration curves may be gained. For the density gages, it is now felt that a single calibration curve cannot be valid for all soils. On the other hand, it would seem that a calibration curve for each soil would be impractical from the standpoint of the difficulty involved in gathering this amount of data. The best alternative at the present time appears to be in the adoption of a family of calibration curves, based on soil pH. This family of calibration curves can be developed after further field data are gathered and the results analyzed.

It appears that a single calibration curve for the moisture gage can be adopted for the materials commonly found in highway construction. As more field data is obtained this concept can be further investigated. It is highly desirable that a detailed record be kept of all nuclear data obtained in the field. Specific importance should be placed on determining material composition and its effect on nuclear readings.
APPENDIX B

STATISTICAL DECISION PROCEDURE FOR
CALIBRATION CURVE ACCEPTANCE
STATISTICAL DECISION PROCEDURE FOR CALIBRATION CURVE ACCEPTANCE

This appendix deals with the development and suggested format to be used in conducting a statistical study of acceptance for a calibration curve to be employed with nuclear backscatter devices.

The exact procedure to be used in a study of this nature is truly a complex analysis and perhaps, beyond the level of present methods of statistical analysis. In order to provide a solution compatible with acceptable significance test methods and present knowledge of the distribution effects of the variables involved in nuclear backscatter devices, several simplifying assumptions have been made. The analysis is based upon a significance test between a given calibration curve (laboratory or field developed) and the "true" calibration curve that the tested material inherently possesses.

The calibration curves were developed using a regression analysis. In all curves the independent variable was considered to be the nuclear count ratio reading and the dependent variable as either density or moisture. A condition required by the least squares analysis is that the error in the independent variable (count ratio) is small (i.e. a fixed value) compared to the variability of the dependent variable (density or moisture). For both density and moisture calibration curves the assumption of this condition was made.

It is recognized that the validity of this assumption can be questioned. The error associated with a count ratio of a moisture gage may be as large as the variability of moisture measurement by standard oven drying techniques. The assumption might be more valid for the density calibration curve because the variability of sand cone density determinations may be as large as ± 4.9 pcf (2).
Although the distribution of nuclear count readings is Poisson, the distribution of a count ratio reading is unknown as it is a ratio of Poisson distributions. The situation is further complicated by the fact that as the number of one minute tests used to determine an average nuclear count is increased, the distribution of the count may approach normality. Consequently, the distribution of a count ratio may range between a ratio of Poisson distributed random variables to a ratio of normally distributed random variables.

Another aspect of the calibration curves that was investigated was the homogeneity of variance along the regression lines. This was done to check uniformity of variances over the entire range of data used to establish the regression lines. Cochran's test for homogeneity was used. Results for both density and moisture calibration curves did not reject the hypothesis of homogeneous variances for a level of significance ($\alpha$) of .05. Results are shown in Figures 27 and 28.

A significance test can be used to test the hypothesis that the mean of a normal distribution has a specified value. If the actual density or moisture obtained from a check test minus the predicted value obtained from the calibration curve is defined as the random variable and is normally distributed, then the optimum procedure for testing the hypothesis that the mean of this difference is equal to zero is based upon the test statistic

\[
    t = \left( \frac{(X_A - X_P)}{\sigma} \right) \sqrt{N} \quad \text{............................ (9)}
\]

where

- $t$ = test statistic
- $X_A$ = actual density or moisture determined by check test
- $X_P$ = predicted density or moisture determined from calibration curve
FIG. 27. STANDARD DEVIATION OF TEST VARIABLE (SAND CONE DENSITY MINUS PREDICTED DENSITY) FOR COUNT RATIO RANGES CONSIDERED IN HOMOGENEITY TEST.
Fig. 28. Standard deviation of test variable (oven dry moisture minus predicted moisture) for count ratio ranges considered in homogeneity test.
\[ (X_A - X_p) = \text{average difference of } N \text{ observations} \]

\[ \mu_0 = \text{expected value of } (X_A - X_p) = 0 \]

\[ N = \text{number of check tests} \]

\[ S = \text{standard deviation of } N \text{ observations} \]

The variable \( (X_A - X_p) \) is assumed to be independently normally distributed over the entire count ratio range.

The decision to either accept or reject the calibration curves can be denoted by:

\[
H: \quad (X_A - X_p) = 0 \\
A: \quad (X_A - X_p) \neq 0 \quad \ldots \ldots \ldots \ldots \ldots \ldots (10)\]

where \( H \) is the hypothesis that the predicted and "true" calibration curves are identical and \( A \) represents the alternative that they are not identical. Since it is possible for the "true" calibration curve to be either above or below the predicted calibration curve, a two sided \( t \) test is conducted. If the value taken on by the test statistic as a result of the check test falls in the rejection region, then the calibration curves cannot be adopted. Likewise, if the value is within the acceptance region, the calibration curves are accepted for field use.

The probability of rejecting the hypothesis when it is really true is the probability of the type one error (\( \alpha \)). In this case \( \alpha = .05 \).

The probability of accepting the hypothesis when it is really false is called the type two error (\( \beta \)). \( \beta \) is not known unless a specific alternative is given. This alternative is a judgement decision associated with accepting the calibration curve when in reality they should not be used.
I. Determinations of the Required Number of Check Tests

The decision of the acceptable $\beta$ error must be decided before the number of check tests required can be determined. The associated risk of the $\beta$ error is a function of the true difference between means, $|\mu - \mu_0|$, the standard deviation ($\sigma$), and the number of tests required (check tests). Therefore, by selecting a value that corresponds to the difference in means (actual value minus predicted) a $\beta$ error is then the probability of not detecting this difference when a sample size of (N) is used for a given $\alpha$ and $\sigma$. The difference in means selected for the density calibration curves of Instrument No. 1 was 4 pounds per cubic foot. That is, when the difference between calibration curves is greater than 4 pounds per cubic foot, the given calibration curve is unsatisfactory. If the above occurs, the probability of accepting the given calibration curve when it should be rejected is $\beta$. This difference for both moisture calibration curves was arbitrarily chosen to be 1.5 pounds per cubic foot of moisture. Figure 29 illustrates the probability of type II errors associated with the number of check tests used for the various nuclear gages. The standard deviation ($\sigma$) of the random variable is unknown. Therefore an estimate of it must be made to determine the required number of check tests for a given ($\beta$). The standard deviation ($\sigma$) was taken to be 3.0 pounds per cubic foot for the density gage and 1.0 per cubic foot for the moisture gage. The decision was based on an examination of the calibration data.

As an example, it is desired to determine the number of check tests required for the Instrument No. 1 density gage. It is decided that the $\beta$ error for determining a mean difference of 4 pounds per cubic foot between the given calibration curve and the true calibration curve for
FIG. 29. TYPE II ERROR ASSOCIATED WITH NUMBER OF CHECK TESTS FOR DIFFERENT NUCLEAR GAGES
the soil in question should be .05. From Figure 29, the required
number of check tests is found to be 10. If conditions at the job
site were such that only 6 check tests were conducted, the $\beta$ error
for the decision would be approximately .25.

That is, the probability of accepting the hypothesis that the
calibration curve is valid when it is really false is .25 if only these
6 tests were used.

II. Significance Test Procedure For Calibration Curve Acceptance.

It has been previously stated that the (t) test is used to decide
whether a given calibration curve will be accepted or rejected for use
with a nuclear gage. The discussion of the (t) test can be found in most
statistics books. Therefore, only a suggested format for arriving at
a decision based upon computation of the (t) statistic is given below.
Tables and a recommended worksheet are included at the end of this appen-
dix.

A. Suggested Procedure:

1. Determine the required number (N) of check tests necessary
based on acceptable probability of Type II error ($\beta$) (Figure 29).
It is suggested that a $\beta$ error of .10 be adopted.
2. Conduct these (N) check tests as described in Appendix A.
3. Determine the predicted density or moisture value by the
appropriate calibration curves shown in Figures 23 to 26 for
the count ratio found for the check test.
4. Calculate the difference in density or moisture determined
from the check test and the predicted value found in step 3 for
each check test ($X_A - X_p$).
5. Compute \( \overline{X_A} - \overline{X_p} \) based on \( N \) observations.

6. Compute the standard deviation of the \( N \) observations.

7. Compute the value of the test statistic \( t \).

8. Determine from Table 4 the test statistic \( t' \) based upon \( N \) observations for \( \alpha = .05 \).

9. If \( (-t' \leq t \leq t') \) accept the calibration curve as shown in Figures 23 to 26 and use them for field control.

10. If \( (t > t') \) or \( (t < -t') \), recalibration for the particular soil in question must be accomplished and the calibration curves shown in Figure 23 to 26 are invalid.
Table 4

Values of $t'$ for Various Number of Check Tests at .05 Level of Significance ($t' = t_{\nu}^{\alpha/2}$; where $\nu = N-1$)

<table>
<thead>
<tr>
<th>N</th>
<th>$t'$</th>
<th>N</th>
<th>$t'$</th>
<th>N</th>
<th>$t'$</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>12.71</td>
<td>9</td>
<td>2.306</td>
<td>16</td>
<td>2.131</td>
</tr>
<tr>
<td>3</td>
<td>4.303</td>
<td>10</td>
<td>2.262</td>
<td>17</td>
<td>2.120</td>
</tr>
<tr>
<td>4</td>
<td>3.182</td>
<td>11</td>
<td>2.228</td>
<td>18</td>
<td>2.110</td>
</tr>
<tr>
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<td>2.776</td>
<td>12</td>
<td>2.201</td>
<td>19</td>
<td>2.101</td>
</tr>
<tr>
<td>6</td>
<td>2.571</td>
<td>13</td>
<td>2.179</td>
<td>20</td>
<td>2.093</td>
</tr>
<tr>
<td>7</td>
<td>2.447</td>
<td>14</td>
<td>2.160</td>
<td>21</td>
<td>2.086</td>
</tr>
<tr>
<td>8</td>
<td>2.365</td>
<td>15</td>
<td>2.145</td>
<td>22</td>
<td>2.080</td>
</tr>
</tbody>
</table>
**Suggested Worksheet for Significance Test Computation**

**Instrument Number (Name):** ____________________________  **(Density)(Moisture) Calibration Curve Number:** ____________

**Project Number:** ____________________________  **Selected β Error:** ____________________________

**Required Number of check tests:** ____________________________

<table>
<thead>
<tr>
<th>Check test No.</th>
<th>Check test (density) (moisture) $X_A (1)$</th>
<th>Check test Count Ratio $C_{RA}$</th>
<th>Predicted (Density)(Moisture) $X_p (2)$</th>
<th>Difference of (Density)(Moisture) $X_A - X_p (3)$</th>
<th>Square of difference $(X_A - X_p)^2 (4)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$N$</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**A. Calculate average difference**

$$\bar{X}_A - X_p = \frac{\sum (X_A - X_p)^2}{N}$$

**B. Compute Standard deviation ($s$)**

$$s^2 = \frac{\sum (X_A - X_p)^2 - \frac{\sum (X_A - X_p)^2}{N}}{N-1}$$

$$s = \sqrt{s^2}$$

**C. Compute test statistic ($t$)**

$$t = \frac{\bar{X}_A - X_p}{s} \sqrt{N}$$

**D. Determine $t'$ from Table 4 based on $N$ observations**

$$t' =$$

**E. Use calibration curve if**

$$-t' \leq t \leq +t' \text{ or } \quad -D \leq C \leq +D$$

**F. Do not use calibration curve otherwise**

*Numbers in ( ) referenced to column numbers.*