FACTORS INFLUENCING THE APPLICATION OF NUCLEAR TECHNIQUES TO SOIL COMPACTION CONTROL

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ABSTRACT

The use of nuclear backscatter moisture-density gages for soil compaction control has gained a great deal of favor during the past several years. However, before this type of equipment can be applied to routine field control, several factors influencing the operation of the gage must be investigated.

Various techniques for expressing nuclear results were studied, and the results indicate that the use of count ratio at constant high voltage should be adopted. This technique along with the use of standard calibration blocks provide for reproducibility of results and account for aging to some extent. These factors are especially important from the standpoint of recalibrating the gages.

Results of tests performed on various materials and calibration blocks of different chemical composition indicate that material composition has a major effect on the development of calibration curves for the density gages. This was not found to be true for the moisture gages. Soil pH as an indication of soil type was investigated, and calibration curves based on this parameter were developed. The effect of grain size distribution resulted in a different calibration curve for coarse grained versus fine grained soils.

Along with typical calibration curves, guidelines for field application were developed. A statistical decision theory based on a "t" test was also developed to aid in making a decision involving the validity of using a given calibration curve.
INTRODUCTION

The rapid determination of soil density and moisture content is of the utmost importance in the control of highway construction. 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.

The potential uses and adaptation 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 nondestructiveness. 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. However, widespread usage of the nuclear gages for final field control has not been universally adopted primarily due to the limited knowledge concerning long term gage stability and reproducibility as well as the fact that the response of the common nuclear density backscatter device is dependent to some degree upon the substrate material type.

* See List of References.
PURPOSE

The purposes of the study reported herein were 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.
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 glueing 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 1 and 2. From Figure 1, 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 40,469 cpm in July 1964. This represents a decrease far outside the reliable error for the gage. In contrast, the variation of self standards in the moisture gage was within the reliable error (Figure 2).

Also, it is noted from Figure 1 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 3 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\textsuperscript{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 RaBe source, a high degree of reproducibility is also shown. A further discussion of the techniques of expressing results based on reproducibility and aging effects is provided in the section of this text pertaining to Procedural Factors.
FIG. 1 VARIATION OF DENSITY GAGE READINGS WITH PLATEAU VOLTAGE
LEGEND: RaBe SOURCE
-○- JULY 1962
-x-x- JUNE 1964
-Δ- JUNE 1966

FIG. 2 VARIATION OF MOISTURE GAGE READINGS WITH PLATEAU VOLTAGE
FIG. 3 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 $^{137}$Cs source illustrated in Figures 1 and 3 is 33 years in contrast to the 1620 year half-life of the RaBe source shown in Figures 2 and 3. Therefore a nuclear gage utilizing a $^{137}$Cs 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 $^{137}$Cs source. These data appear in the lower portion of Figure 3. The general equations of radioactive decay are:

\[
N(t) = N_0 e^{-\lambda t} \quad \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots (1)
\]
\[
A(t) = \lambda N(t) \quad \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots (2)
\]
\[
T = \frac{\ln 2}{\lambda} \quad \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots (3)
\]

where

- $N(t)$ = No. of undecayed atoms at time $(t)$
- $N_0$ = No. of undecayed atoms at time $(t = 0)$
- $A(t)$ = Activity at time $(t)$
- $A_0$ = Activity at time $(t = 0)$
- $t$ = Time from $t = 0$ (yrs)
- $\lambda$ = Proportionality constant $(\text{yrs}^{-1}) = 0.021$ for $^{137}$Cs
- $T$ = Half-life (yrs) = 33 yrs for $^{137}$Cs
Therefore the theoretical decay ratio becomes:

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

(4)

For the actual decay ratio:

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

(5)

Table 1

<table>
<thead>
<tr>
<th>t (yrs)</th>
<th>e^{\lambda t}</th>
<th>R_T</th>
<th>\frac{CPM(t=0)}{CPM(t=t)}</th>
<th>R_A</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 (January 1962)</td>
<td>e^{(0.021)0}</td>
<td>1.000</td>
<td>\frac{\43,215}{\43,215}</td>
<td>1.000</td>
</tr>
<tr>
<td>0.83 (October 1962)</td>
<td>e^{(0.021)(0.83)}</td>
<td>1.018</td>
<td>\frac{\43,215}{\42,531}</td>
<td>1.016</td>
</tr>
<tr>
<td>1.17 (February 1963)</td>
<td>e^{(0.021)(1.17)}</td>
<td>1.025</td>
<td>\frac{\43,215}{\42,181}</td>
<td>1.025</td>
</tr>
<tr>
<td>1.50 (June 1963)</td>
<td>e^{(0.021)(1.50)}</td>
<td>1.032</td>
<td>\frac{\43,215}{\41,880}</td>
<td>1.032</td>
</tr>
<tr>
<td>1.67 (August 1963)</td>
<td>e^{(0.021)(1.67)}</td>
<td>1.036</td>
<td>\frac{\43,215}{\41,543}</td>
<td>1.040</td>
</tr>
<tr>
<td>2.58 (July 1964)</td>
<td>e^{(0.021)(2.58)}</td>
<td>1.056</td>
<td>\frac{\43,215}{\40,469}</td>
<td>1.068</td>
</tr>
<tr>
<td>3.92 (November 1965)</td>
<td>e^{(0.021)(3.92)}</td>
<td>1.086</td>
<td>\frac{\43,215}{\39,696}</td>
<td>1.089</td>
</tr>
</tbody>
</table>

Figure 4 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 3...
can be attributed to decay of the source. For the RaBe 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 4. As pointed out previously, the decrease in self-standard counts for the moisture gage utilizing the RaBe source was almost negligible.

It was 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 will not eliminate the need for recalibration, but will merely serve to reduce the required frequency of recalibration."

Interpreting the data obtained from Figure 3 along with Mr. Pocock's discussion, it can be concluded that for a testing period of approximately 2.5 years the use of the count ratio eliminated the need to recalibrate the
FIG. 4 COMPARISON OF ACTUAL AND THEORETICAL DECAY RATIOS
nuclear gage containing the Cs\(^{137}\) source. It cannot, however, be concluded due to the limited test interval, that recalibration will never be required when the count ratio is utilized.

The previous paragraphs have been concerned with the effect of aging on nuclear results over extended periods of time. For short periods of time (several months) aging does not appear to affect the standard counts for either the moisture or density gages. Results of this are shown by Figure 5 which is a plot of standard counts versus time for a particular instrument system. The density gage indicated utilizes a Cs\(^{137}\) source which has previously been shown to be the most critical with respect to aging. However, results as shown by Figure 5 indicate that standard counts remain constant over relatively short periods of time.
FIG. 5 VARIABILITY OF STANDARD COUNTS
Procedural Factors

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 6 illustrates the above based on the data for Figure 1. 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 6. 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 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 \( R_A \).
FIG. 6 ILLUSTRATION OF DENSITY GAGE READINGS WITH PLATEAU VOLTAGE
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:

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

\[ t = t': \quad HV = B, \quad \text{Count Per Minute} = C_B'; \quad \text{Count Ratio} = R_B \]

however, from Figure 6

\[ R_A \neq R_B \]

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 recalibration 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 1, 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 have been recalibrated 1.8 yrs. 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:

\[
e^{-\lambda t} = \frac{C_U}{C_U''} \quad \ldots \quad \ldots \quad \ldots \quad \ldots (6)
\]

\[
e^{(0.021)t} = \frac{47,500}{43,215}
\]

\[
e^{(0.021)t} = 1.099
\]

\[
t = \frac{\ln 1.099}{(0.021)}
\]

\[
t = \frac{0.0943}{0.021}
\]

\[
t = 4.5\ \text{yrs.}
\]

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 1.

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 7 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 2) 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\textsuperscript{137} source, will require recalibration due to source decay (aging) provided the method used to express results is a count-ratio-varying high voltage.

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 years. 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 $R'_A - R_A \geq$ reliable error of ($R_A$). Consequently, since this difference in count ratios was negligible for a 2.5
Fig. 7 Comparison of count ratio and count per minute procedure for moisture calibration.
year period; the time required to cause a recalibration (count ratio-constant high voltage) for a nuclear gage using a Cs\textsuperscript{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\textsuperscript{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.
SUBSTRATE MATERIALS

Density Gage

Material Composition

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 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 for all material types is made. However, mass absorption coefficients are a function of both the nuclear particle energy and the 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 8.

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 9 illustrates calibration curves for heavy liquids, 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 Virginia Highway Research Council. 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
FIG. 8 RELATIONS BETWEEN MASS ABSORPTION COEFFICIENT AND THE ENERGY OF GAMMA RADIATION FOR ELEMENTS COMMONLY FOUND IN SOIL (FROM PARSONS & LEWIS)
a chemical analysis of the crushed limestone and crushed quartzite studied at Purdue, is shown in Table 2. It is seen that Blocks No. 1, 2, and 5 have an appreciable quantity of silicon dioxide ($\text{SiO}_2$) 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 is calcium oxide (CaO). It is quite obvious that for the $\text{SiO}_2$ 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 2
Chemical Analysis of Selected Materials

<table>
<thead>
<tr>
<th>Material</th>
<th>$\text{SiO}_2$ (%)</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 9 shows the effect of material type for nuclear density gage with a Cs$^{137}$ 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
FIG. 9  EFFECT OF MATERIAL TYPE ON NUCLEAR DENSITY READINGS
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 8, 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 experiment to determine material type.

It is recognized that the pH method perhaps 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 may tend to yield a basic

* For the gage using a Cs$^{137}$ source, the magnitude of the deviation ranged from 12 pci to 18 pci: while the range between calibration curves was 25 pci to 35 pci with the gage using the RaBe source.
pH while physically speaking, the acidic element would generally dominate the overall average mass absorption coefficient for the soil. Also, soluble salts in the soil mass of different chemical properties than that of the soil itself may result in the measurement of a pH value that is not truly indicative of 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 10 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 8 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 (i.e., 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 10 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 (i.e., material type), its application to field testing produced calibration curve parameters that reduce errors of estimate for the nuclear readings.
FIG. 10 DENSITY CALIBRATION CURVES DEVELOPED BY SOIL pH GROUPING FOR FIELD DATA
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. 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 11.

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 11. Field data tend to substantiate this concept as illustrated by Figure 12. The figure is representative of all basic (pH \( \geq 7.3 \)) 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. However, in studies made during July of 1966 this same trend was observed using a
**FIG. 11** EFFECT OF GRAIN SIZE DISTRIBUTION UPON NUCLEAR DENSITY READINGS FOR SELECTED MATERIALS
FIG. 12  COMPARISON OF FIELD CALIBRATION CURVES FOR GRANULAR AND FINE GRAINED MATERIALS (pH ≥ 7.3)
different nuclear system and it would appear that distinct parallel calibration curves exist for coarse grained versus fine grained materials.
SUMMARY OF RESULTS

Guidelines for Use of Nuclear Gages

This section presents guidelines for the use of nuclear moisture density gages for routine compaction control in Indiana. 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. Standard - 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. Many researchers have suggested the use of a leveling course to be placed between the gage and substrate to accomplish this seating. However, results during this study have not conclusively substantiated this concept and no recommendation concerning this factor can be made.
D. Standard Blocks - A standard reference block should be used to insure proper functioning of the density gage as well as to 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 2\(\frac{1}{4}\)" x 2\(\frac{1}{4}\)" 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 be adopted for field use. A typical moisture calibration curve is shown in Figure 13. If the gage is to be
FIG. 13  MOISTURE CALIBRATION CURVE
FOR INSTRUMENT NO. 1
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. 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.

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 percent, 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 16 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 A.

III. Density Gages

A. Calibration curves-Typical density calibration curves are presented in this section as Figures 14 and 15. These 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 suggested.

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.
FIG. 14  DENSITY CALIBRATION CURVES FOR FINE-GRAINED SOILS FOR INSTRUMENT NO. 1

pH $\approx 7.2$: $D = -308.94 \times \text{CR} + 197.86$

pH $\approx 7.3$: $D = -332.02 \times \text{CR} + 194.93$
FIG. 15  DENSITY CALIBRATION CURVE FOR
GRANULAR MATERIALS FOR INSTRUMENT
NO. 1
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 A.

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 judgment will have to be made by the engineer.

IV. Summary - The guidelines 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 are obtained, this concept can be further investigated.
It is highly desirous 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 A

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.

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 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.

$$t = \frac{(X_A - X_P) - \mu_o)}{S} \sqrt{N}$$

$$t = \text{test statistic}$$

$$X_A = \text{actual density or moisture determined by check test}$$

$$X_P = \text{predicted density or moisture determined from calibration curve}$$
\[
\overline{(X_a - X_p)} = \text{average difference of } N \text{ observations}
\]
\[
\mu_o = \text{expected value of } \overline{(X_a - X_p)} = 0
\]
\[
N = \text{number of check tests}
\]
\[
S = \text{standard deviation of } N \text{ observations}
\]
The variable \(\overline{(X_a - X_p)}\) 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:

\[
H: \quad \overline{(X_a - X_p)} = 0
\]

\[
A: \quad \overline{(X_a - X_p)} \neq 0 \quad \ldots \ldots \ldots \ldots \ldots \quad (8)
\]

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 judgment 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 and 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 16 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 the
FIG. 16 TYPE II ERROR ASSOCIATED WITH NUMBER OF CHECK TESTS FOR DIFFERENT NUCLEAR GAGES
soil in question should be .05. From Figure 16, 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 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 appendix.

A. Suggested Procedure:

1. Determine the required number (N) of check tests necessary based on acceptable probability of Type II error (β) (Figure 16). It is suggested that a β error of .05 be adopted.

2. Conduct the (N) check tests as previously described.

3. Determine the predicted density or moisture value by the appropriate calibration curves for the count ratio found from 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 \((X_A - X_F)\) 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 3 the test statistic \((t')\) based upon
   \(N\) observations for \(\alpha = .05\).

9. If \((-t' \leq t \leq t')\) accept the calibration curve field control.

10. If \((t > t')\) or \((t < -t')\), recalibration for the particular soil in question must be accomplished.
Table 3

Values of $t'$ for Various Number of Check Tests
at .05 Level of Significance ($t' = t; \text{ where } \nu = N-1 (\mathcal{F}, \nu)$

<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>
<td>5</td>
<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>
Table 4
Suggested Worksheet for Significance Test Computation

<table>
<thead>
<tr>
<th>No.</th>
<th>Xₐ (1)</th>
<th>CRₐ</th>
<th>Xₚ (2)</th>
<th>Xₐ - Xₚ (3)</th>
<th>(Xₐ - Xₚ)² (4)</th>
<th>∑(Xₐ-Xₚ) (5)</th>
<th>∑(Xₐ-Xₚ)² (6)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td></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>
<td></td>
<td></td>
</tr>
<tr>
<td>N</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Where:
- \( Xₐ \) = Check test (density) (moisture)
- \( CRₐ \) = Check test Count Ratio
- \( Xₚ \) = Predicted (Density)(Moisture)
- \( Xₐ - Xₚ \) = Difference of (Density)(Moisture)
- \( (Xₐ - Xₚ)² \) = Square of difference

A. Calculate average difference
\[
(Xₐ-Xₚ) = \frac{\sum(Xₐ-Xₚ)}{N} = (5) * \]

B. Compute Standard deviation \( S \)
\[
S^2 = \frac{\sum(Xₐ - Xₚ)^2 - \sum(Xₐ - Xₚ)^2}{N-1} \]
\[
S^2 = \frac{(6) - \frac{(5)^2}{N}}{N-1} \]
\[
S = \sqrt{S^2} \]

C. Compute test statistic \( t \)
\[
t = \frac{(Xₐ-Xₚ)}{\sqrt{N} \frac{S}{N}} \]

D. Determine \( t' \) from Table 4 based on \( N \) observations
\[
t' = \frac{\text{ }}{\text{ }} \]

E. Use calibration curve if
\[
-t' \leq t \leq + t' \text{ or } -D \leq C \leq + D \]

F. Do not use calibration curve otherwise

* Numbers in ( ) reference to column numbers.
SELECTED REFERENCES


