

FINAL REPORT

REFINEMENT OF MOISTURE CALIBRATION CURVES FOR NUCLEAR GAGE

by

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(The opinions, findings, and conclusions expressed in this report are those of the authors and not necessarily those of the sponsoring agencies.)

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SUMMARY

Over the last three years the Virginia Highway Research Council has directed a research effort toward improving the method of determining the moisture content of soils with a nuclear gage. The first task in this research was the determination of the correct moisture calibration curve for the nuclear gages being used (Troxler Models 227 and 2401). Forty-three soils sampled from the three physiographic areas of Virginia and having optimum moisture contents ranging from 5% to 40% were tested. It was found that for the Model 227 gage two calibration curves produced better results than did one calibration curve. With one curve the standard error was 2.7 pcf (0.04 g/cc), while for two curves the errors were 1.4 and 1.7 pcf (0.02 and 0.03 g/cc), which are judged to be acceptable for compliance testing. From an investigation of the physical and chemical properties of the soils tested it was concluded that the separation of the two curves could be based on optimum moisture content, and that the separation value should be 21%. At this point in the study, the testing of the Model 227 gage was discontinued because the Virginia Department of Highways had replaced all of these gages with the Model 2401.

The Model 2401 gage requires only one calibration curve, which has a standard error of 1.7 pcf (0.03 g/cc). Using this gage and its single calibration curve, five moisture standards were developed. A zero moisture content standard was developed by using dry C-190 sand, and four standards were developed by mixing different portions of either $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$ or $\text{Na}_3\text{PO}_4 \cdot 12 \text{H}_2\text{O}$ with Ottawa silica C-190 sand. These standards had respective moisture contents of 9.3, 16.1, 21.0 and 25.8 pcf (0.15, .26, .34, and .41 g/cc). The curve of best fit for these standards had a standard error of only 0.57 pcf (0.01 g/cc). Also the curve was approximately parallel to the curve previously determined from the soils. At approximately 25 pcf (0.40 g/cc) moisture content, the curve from the standards gives a moisture content about 2 pcf (0.03 g/cc) higher than the curve from the soils; and at around 5 pcf (0.08 g/cc) moisture content, the former gives a moisture content about 1 pcf (0.02 g/cc) higher than does the latter.

The manufacturer's curve generally gives a lower moisture content than does either of the other two curves at the same count ratio.

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PREVIOUS RESEARCH

Initial Work

The Virginia Highway Research Council was among the pioneers in research on portable nuclear density and moisture gages. Council research was started in 1959, and by 1965 the gages were used in Virginia to test for compliance with specifications on the moisture content and density of soils.

In 1965 the Research Council sponsored a Correlation and Conference of Portable Nuclear Density and Moisture Systems in Charlottesville(1).^{*} After several years, use of the moisture standards prepared for this correlation conference were discontinued, since the calibration curves obtained with them were unsatisfactory for all soils. Temporarily this problem was solved by the use of a field calibration; that is, by obtaining a correlation point with conventional methods and drawing a curve parallel to the original calibration curve obtained from the moisture standards. Though satisfactory in many cases, field calibration is inefficient and subject to misuse in the hands of those who are not well experienced with nuclear gages.

The problems in developing reliable moisture standards are:

1. If physical standards containing water are used, then moisture stability is a problem; evaporation or movement of water cause changes in the properties of the standards.
2. If chemical standards containing hydrogen are used, the obtainment of realistic densities is a problem. In addition, it is not easy to obtain a variety in chemical composition while holding the hydrogen content constant.

^{*}Numbers in parentheses refer to entries in the list of references.

Anday-Hughes Study

In early 1969 M. C. Anday and C. S. Hughes III of the Research Council undertook a study to develop more satisfactory standards. (2) It was believed that most of the problems in Virginia were encountered in testing soils with moisture contents greater than 30%. Also, it was felt that the moisture calibration curves might have a shape similar to the ones shown in Figure 1 rather than just the linear shape characterizing the curve being used.

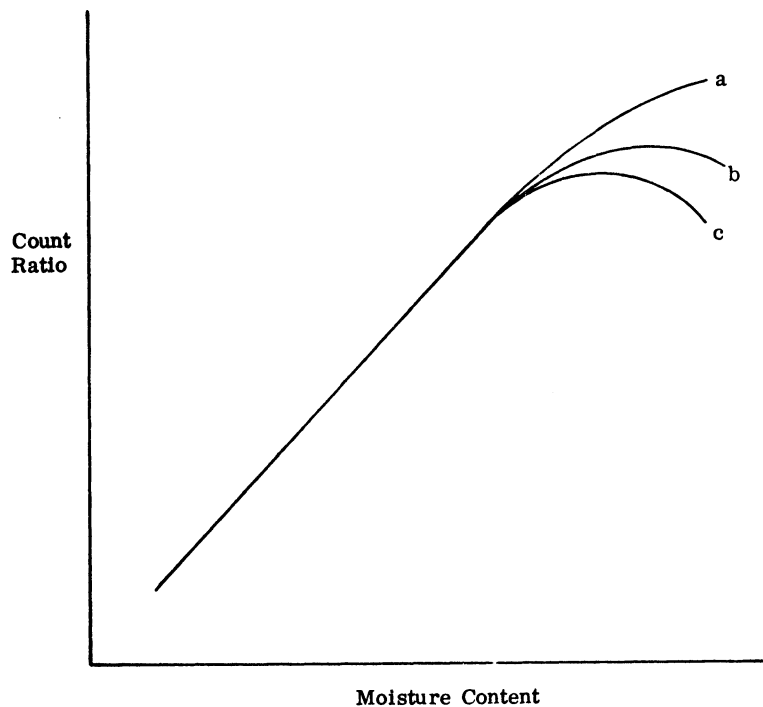


Figure 1. Possible shapes of moisture calibration curve.
(From reference 2.)

In the working plan for the Anday-Hughes study four objectives were stated as follows:

1. Determine the shape of the calibration curve for different soils.
(At the time of the study it was felt that the shape of the moisture calibration curve above 30% moisture must be determined (whether a, b, or c in Figure 1). Even though soils with moisture contents this high are rare in Virginia they are occasionally encountered. It was felt that for soils with moisture contents less than 30%, which are represented by the straight-line portion of the calibration curve, the curve need only be verified.)

2. Determine the accuracy of a single calibration curve.
3. Develop primary standards.

(Primary standards are ones that contain a known amount of hydrogen in the form of either water or some other material. The standards prepared for the conference in 1965 were primary, but problems that arose after the conference led to the discontinuance of their use.)

4. If objective number 3 is unsuccessful, then the development of secondary standards will be pursued.

(Secondary moisture standards are ones that are assigned moisture content values because the amount of hydrogen in each is not known.)

To achieve objectives 1 and 2, the researchers sampled ten soils from different geological formations to provide a wide spread in chemical and physical properties. The optimum moisture contents of the soils ranged from 5% to 40%.

Each soil was compacted in a 17" (43.2 cm) diameter by 7" (17.8 cm) high mold at six moisture contents and two predetermined densities. After nuclear measurements were obtained with the Troxler 227 gage, the moisture content and density of each molded sample were determined by conventional methods. The results of the tests are shown in Figure 2 with the nuclear count ratio plotted versus the actual moisture content in pcf (g/cc) as determined by the conventional method. The count ratio for each test is the moisture content reading divided by the moisture standard count for that nuclear gage that day. The linear curve of best fit for the data is shown on Figure 2 and has a standard error of 3.0 pcf (0.05 g/cc).

It was felt by the researchers that the standard error of 3.0 pcf (0.05 g/cc) was too high to allow the nuclear gage to be used for specification compliance tests. Since the variability in data was high, they felt it was necessary to determine the cause of the variability before embarking on objectives 3 and 4, the development of primary or secondary standards. In their interim report number 1 to the Federal Highway Administration, they requested that objectives 3 and 4 be changed to one as follows:

Objective 3. Determination of the quantitative effects of several specific chemicals on the moisture determination. More specifically, it is intended to select at least two soils with low percentages of any of the problem chemicals and also with little chemically bound hydrogen; to add varying amounts of iron, potassium, titanium and manganese; and to experimentally measure the effects of these chemicals on the moisture count rate. Also, other chemicals such as boron and chlorine may be tested⁽³⁾.

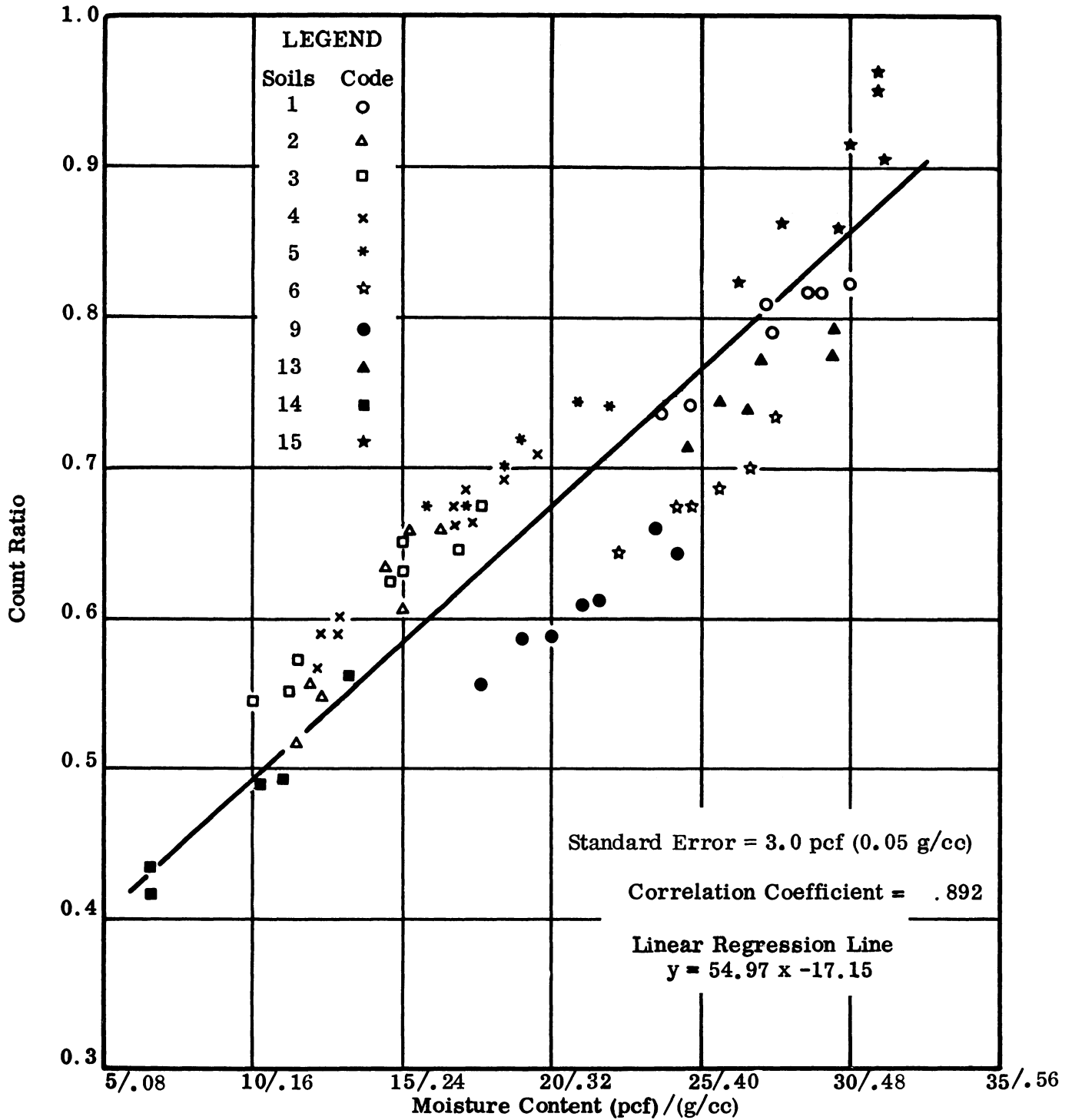


Figure 2. Single calibration curve for Model 227 gage.

In response to the above request, the Federal Highway Administration requested Anday and Hughes to terminate their project and submit a final report. However, while awaiting a reply from the FHWA, Anday and Hughes had begun an abbreviated investigation into the effects of some chemicals. The two chemicals tested were iron and titanium, which have large capture cross sections that the researchers thought might interfere with nuclear moisture readings. From adding each chemical at two dosages to a particular soil, no trends or effects were found.

The final report by Anday and Hughes summarized their findings in attempting to determine the cause of the large variability of the data. ⁽⁴⁾ Besides checking the effect of iron and titanium on the nuclear moisture readings, they investigated the loss on ignition values obtained at 1,000°C to see if any trends were evident and found none.

Another possible cause of the large variability, one that the Federal Highway Administration suggested for investigation, was the source of each soil. Although the soils were sampled from three physiographic areas, no trends were indicated by separating the soils on the basis of these three areas.

After no cause of the large variability was found, it became apparent to the researchers that the data points could possibly be divided into two groups that could be represented by two calibration curves. With two calibration curves instead of one, the variability would decrease and thus the accuracy would increase.

In order to establish the proper calibration curve, some property of the soils that separates them into two groups must be known. It was at this time that the researchers thought the soils could be grouped by their physical properties, and a correlation was sought for the following properties:

1. Atterberg limits
2. Maximum density
3. Optimum moisture content
4. Gradation
5. Specific gravity
6. Classification (HRB)

After an evaluation of all the physical properties of the soils tested, it was determined that the data could be grouped by the following properties at the separation points indicated:

1. Optimum Moisture Content — 18%
2. Percent Passing No. 200 Screen — 48%
3. Percent Sand — 30%

Figure 3 shows the two calibration curves for the two groups of data. Also shown are the standard error and the equation for each curve. With two calibration curves, the standard errors are 1.5 and 0.84 pcf (0.02 and 0.01 g/cc), as compared to 3.0 pcf (0.05 g/cc) for the single curve.

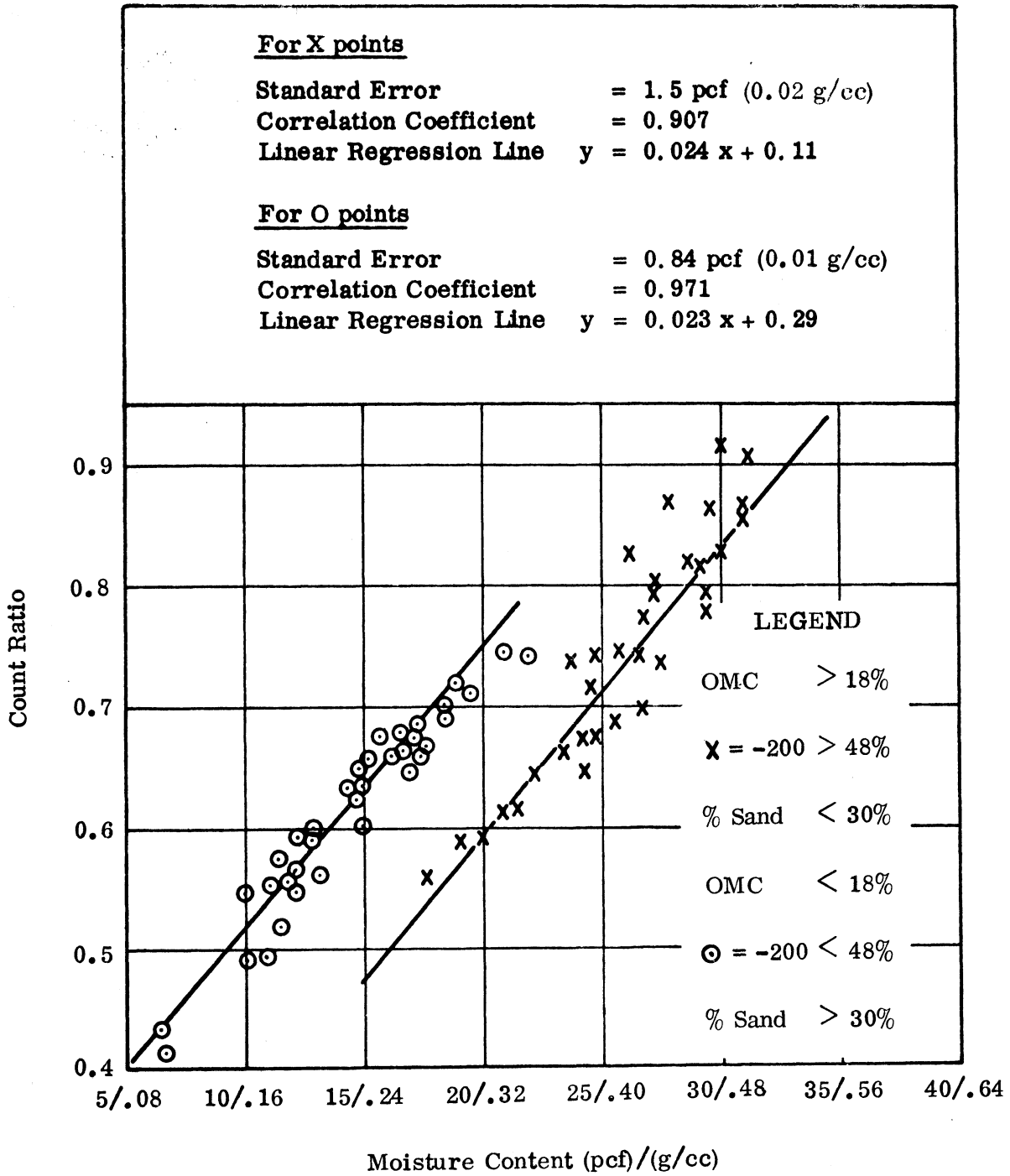


Figure 3. Separation of data on the basis of physical properties for Model 227 gage.

In addition to the final report as summarized above, the Federal Highway Administration suggested that a working plan for a new study be prepared to accomplish the new objective proposed earlier and original objectives 3 and 4. The working plan⁽⁵⁾ was prepared and submitted in September 1970 and outlined the research concluded with this present final report.

PRESENT STUDY

Objectives

The working plan for this study stated four objectives as follows:

1. Generalize the conclusion that soils can be divided into two or more distinct groups by the three physical properties: optimum moisture content, percent passing the No. 200 screen, and percent sand.
2. Determine if the above conclusion is applicable to a more modern gage than the one used in the Anday and Hughes study.
3. Develop primary standards.
4. If objective number 3 is not successful, then develop secondary standards.

In the study by Anday and Hughes one nuclear gage was used throughout the study. It was felt prior to this study that there might be differences between two models of gages in their number of calibration curves as well as the slope of the curves. It was for these reasons that objectives 1 and 2 of the present study were proposed.

Interim Report No. 1

In interim report No. 1⁽⁶⁾ of this study the procedures for pursuing objectives 1 and 2 and the results obtained were discussed. The following is a summary of the interim report.

The Troxler Model 227 gage used in the Anday and Hughes study was tested on some additional soils in the present study to fulfill objective number 1. From the analysis of the Anday-Hughes data and the additional data it was determined that the standard error was reduced to 2.7 pcf (0.04 g/cc) when a single calibration curve of best fit was used. An error of this magnitude was still felt to be too large for most specifications and field calibrations would have to be made for some soils in order to use the gage effectively. Therefore, the data were analyzed as in the previous study to determine if they could be divided into two groups that could be represented by two calibration curves. From an

analysis of all the physical properties of the soils, it was determined that the optimum moisture content results showed a trend. The other two properties, percent passing the No. 200 screen and percent sand, that had indicated a division in the previous study did not show an apparent division of the same soils as did the optimum moisture content.

However, at two other optimum moisture content division lines the percent passing the No. 200 screen in one case and the percent sand in the other case indicated a separation in the data. When the standard errors were determined for these separations, they were larger than those determined for the separation by the optimum moisture content only. Also, since this gage is no longer being manufactured or used in Virginia, the groupings other than by optimum moisture content did not appear to be pertinent and are not reported.

The correlation of the optimum moisture content with a certain calibration curve is practical for the Virginia Highway Department because at the time of the field density testing the optimum moisture content is known and available to the inspector.

In the Anday and Hughes study the division point between the two calibration curves was determined to be approximately 18% optimum moisture. After the testing of additional soils in this study the division point was determined to be 21%. Also, the variability of the data for each curve increased. The standard error of 0.84 pcf (0.01 g/cc) for the lower moisture curve in the previous study increased to 1.4 pcf (0.02 g/cc) after the additional testing. For the higher moisture soils, those above 21% optimum moisture content, there was a small increase in the standard error; from 1.5 pcf (0.02 g/cc) to 1.7 pcf (0.03 g/cc).

Figure 4 shows the two calibration curves determined for the Model 227 gage and the curve provided by the gage manufacturer (dashed line). The manufacturer's curve was the one provided to the Highway Department's inspectors. When problems arose in making moisture determinations with the Model 227 gage, adjustments to the curve were made for high moisture soils. In other words, the manufacturer's curve was shifted to the right, or toward higher moisture contents. In Figure 4 it can be seen that these adjustments substantiate the findings of this study. The manufacturer's curve and the high moisture calibration curve are approximately parallel to each other and about 3 standard deviations, or about 5.0 pcf (0.08 g/cc), apart. However, field adjustment is no longer a problem to the Highway Department in Virginia, since these gages are no longer used.

At this point in the study research with the Model 227 gage was discontinued, since the gage is obsolete.

The second objective of this study involved the use of a more modern nuclear gage, the Troxler Model 2401. During the additional testing with the Model 227 gage, tests were carried out with the Model 2401 gage. The data were analyzed to determine if any separation as indicated with the Model 227 gage was possible with the Model 2401. An examination of all soil properties showed no indication of a separation and, further, that the standard error of a single curve, as shown in Figure 5, is 1.7 pcf (0.03 g/cc), which is considered reasonable. This standard error for the single calibration curve

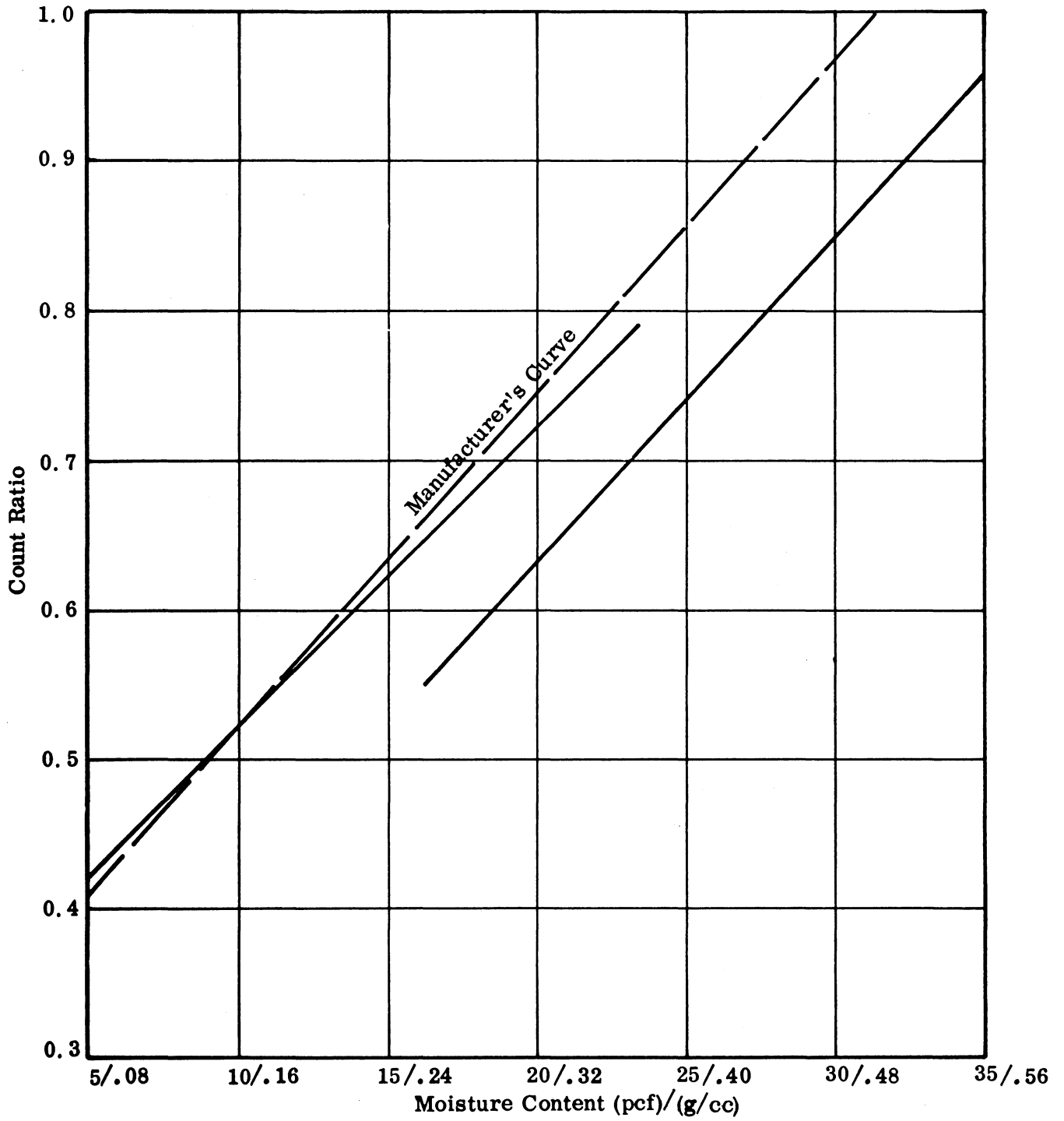


Figure 4. Calibration curves for Model 227 gage.

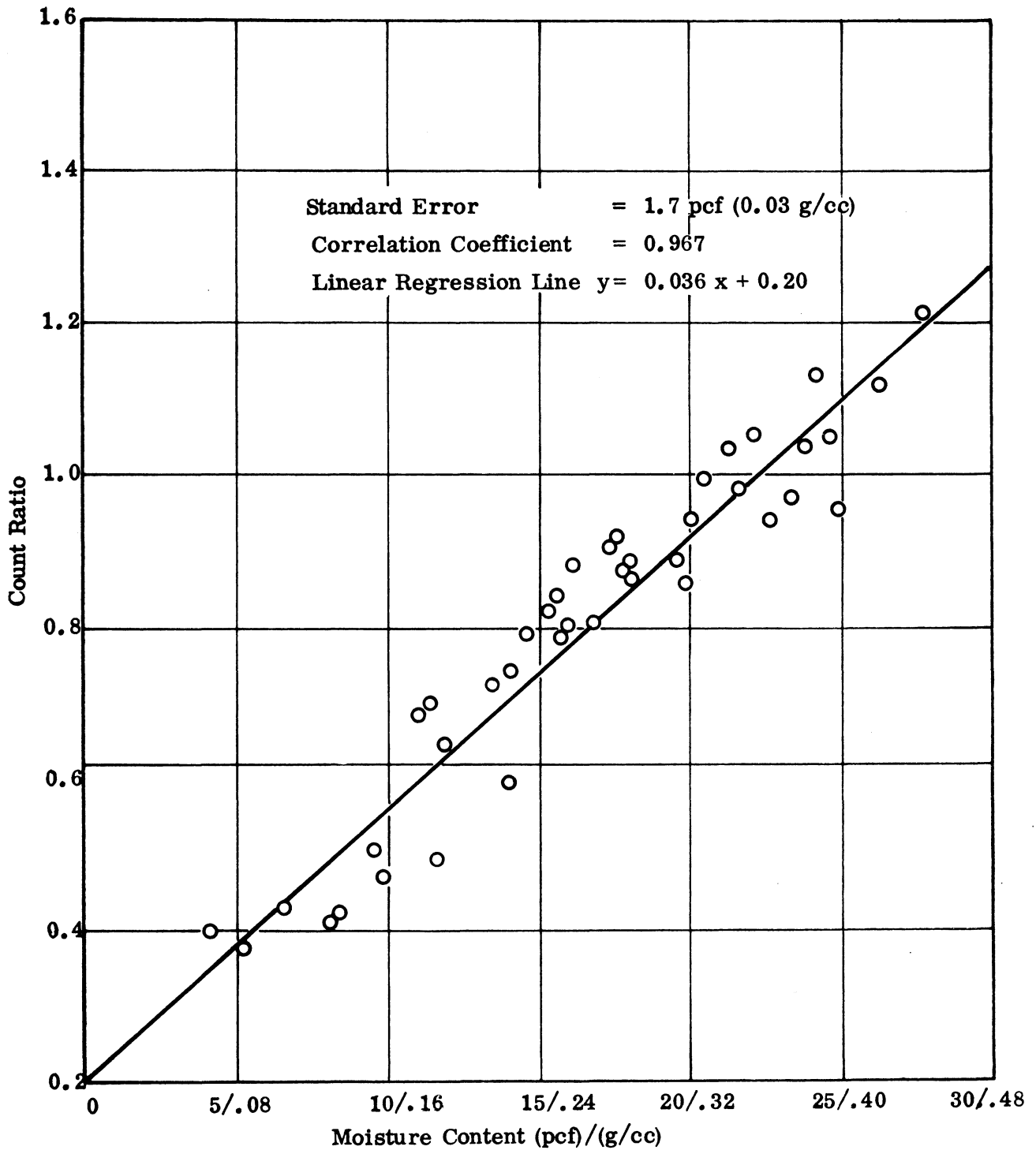


Figure 5. Single calibration curve for Model 2401 gage.

of the Model 2401 gage may be smaller than that for the single calibration curve of the Model 227 due to the following reasons:

1. The geometry of the gage,
2. the strength of the isotropic source,
3. the source type,
4. the detector system, and
5. other technological changes incorporated in the Model 2401.

Due to these changes, the slope of the calibration curve for the Model 2401 is also steeper than the slopes of the curves for the Model 227.

Figure 6 shows the soil calibration curve determined in this study and the manufacturer's calibration curve (dashed line) for the Model 2401 gage. It can be seen that the two cross each other around 6 pcf (0.10 g/cc) moisture content. At a count ratio of 0.2, or near zero moisture content, the two curves differ by 1 pcf (0.02 g/cc) moisture content. In other words, the manufacturer's curve will give a higher moisture content than the soils curve in this area. Above 6 pcf (0.10 g/cc) moisture content the two curves diverge, with the manufacturer's curve giving a lower moisture content than the soils curve for the same count ratio. At 25 pcf (0.40 g/cc) moisture content the two curves differ by approximately 4 pcf (0.06g/cc) moisture content.

Since the Model 2401 gage requires only one calibration curve, the development of standards for use with it was somewhat easier. With the one curve, moisture standards near the bottom and the top of the curve are needed as well as a check value for these standards near the middle of the curve.

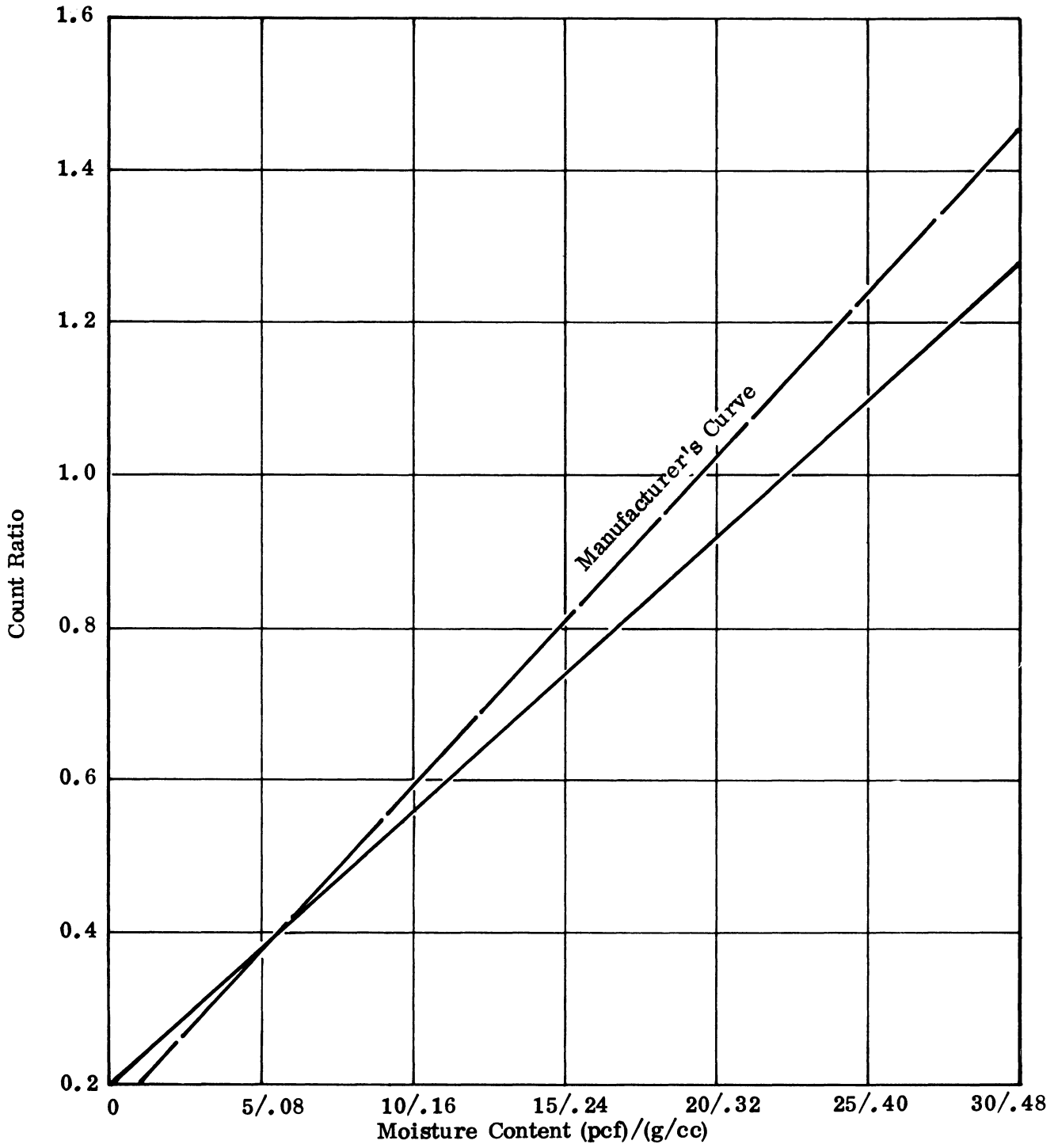


Figure 6. Calibration curves for Model 2401 gage.

Objective number 3 was to develop primary standards. As explained earlier, primary standards have a known amount of hydrogen, which is read by the nuclear gage. In the past, the development of primary standards has been attempted with little success. Several possible reasons for the lack of success are:

1. The moisture standards were set up on the basis of one calibration curve, when actually two or more curves may have been needed;
2. the material in the standards was not stable;
3. the standards were not uniform;
4. the precise amounts of moisture or hydrogen in the standards were not known; and
5. the densities of the standards were not comparable to the densities of soils in the field, which generally are above 80 pcf (1.28 g/cc).

Past Council attempts to develop moisture standards using dry sand, sand saturated with water, dry sand mixed with a chemical, and different chemicals met with only moderate success.⁽¹⁾ With the wet sand standard, problems were encountered with the evaporation of water as well as in determining the actual moisture content. With the standard made of dry sand mixed with a chemical other problems were encountered. The density of the standard was lower than that of most soils encountered in the field, and the standard was not a homogeneous mix of chemical and sand. The chemical standards provided the high moisture contents needed but problems were encountered in obtaining the desired density.

Sand Standards

After all these problems were considered, the development of standards for this study was started. First, dry sand was molded for use as the zero percent moisture standard. In order to obtain a uniform standard, it was decided to use Ottawa C-190 silica sand. The sand was placed in a lucite box approximately 4 cubic feet (0.11 m³) in volume (2 feet (0.61 m) by 2 feet (0.61 m) by 1 foot (0.31 m)). An immersion-type vibrator was then used to compact the sand to the maximum obtainable density. As the sand settled from compaction, more was added until the box was completely full and compacted. The sand was leveled with the top of the box, and a polyethylene sheet was fastened over the mold to keep out dust and prevent the absorption of moisture.

The mold was then weighed to determine its mass unit weight. Since the Ottawa sand contained zero percent moisture, the mass unit weight was also the dry unit weight, which for this mold was 109.5 pcf (1.75 g/cc).

Nuclear readings were then taken from the standard for two weeks to get an accurate count ratio. As explained earlier, the count ratio of the nuclear gage is the

actual moisture content reading off the mold divided by the standard count for that particular gage that day. The count ratio for the Model 2401 gage on the dry sand standard was determined to be 0.170 and varied only 0.002 over the period of testing. From the manufacturer's calibration curve this count ratio indicated an average moisture content of 0.3 pcf (0.005 g/cc).

After the count ratio for the dry sand standard was established, it was decided to see if a wet sand standard could be developed that was reasonably stable at the highest possible moisture content without adhesion of moisture to the bottom of the nuclear gage. A piezometer tube was attached to the side of the lucite box at the bottom seam. Water was added to the sand through the tube and allowed to come to equilibrium for a day. To determine the mass unit weight and moisture content of the standard, it was weighed after the second day. Nuclear tests were then run, after which more water was added to the sand and the above procedure repeated. When the water was allowed to come to equilibrium, it was evident that the capillary action of the water in the sand caused the sand to be damper at a higher height in the mold than indicated by the piezometer tube, which produced an incompatibility between the nuclear readings and the actual water content. The addition of water was continued until water just started to adhere to the bottom of the gage, which occurred when the height of water in the standard was approximately 11 inches (27.94 cm) from the bottom of the mold, or 1 inch (2.54 cm) from the surface of the standard. It was assumed that the moisture content in the mold was the maximum possible to obtain with a wet standard without free surface water exposed to the nuclear gage. The moisture content of the standard was determined to be 18.6 pcf (0.30 g/cc) or 17.1%.

Due to evaporation, water had to be added every day to keep the water level up to 11 inches (27.94 cm). It was decided then that a wet sand standard was not stable enough to be used to reproduce moisture calibration curves for nuclear gages and thus it was not included in any analysis.

Chemical Standards

As mentioned earlier, chemicals, either by themselves or mixed with sand, had been tried as standards. As also explained earlier, these efforts did not prove to be very successful. The Council's chemist, G. G. Clemená, was consulted and he suggested several stable chemicals that might be mixed with sand to form standards. Table 1 shows three of the chemicals. Each has a certain number of water molecules chemically held. Since the nuclear gage reads the amount of hydrogen in a material, the hydrogen in the water molecules is read. The second column of Table 1 gives the molecular weights of the chemicals, which include the weight of the chemically held water. If the weight of the water molecules in each chemical is divided by the total molecular weight of the chemical, then column 3 is derived. Another way of viewing the water fraction is that for every pound (453.59 g) of the chemical being tested, the nuclear gage reads the water fraction ratio shown in the table as moisture content. As an example, if one pound (453.59 g) of $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$ is tested with a nuclear gage, the moisture content reading would indicate 0.511 pound (231.79 g) of hydrogen, or moisture, present.

TABLE 1

SUGGESTED STABLE CHEMICALS FOR USE IN STANDARDS

<u>Chemical</u>	<u>Molecular Weight (g)</u>	<u>Water Fraction</u>
$\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$	246.48	0.511
$\text{Na}_3\text{PO}_4 \cdot 12 \text{H}_2\text{O}$	380.12	0.568
$\text{Na}_2\text{SO}_4 \cdot 10 \text{H}_2\text{O}$	322.19	0.559

One problem encountered in previous studies was the low density of the moisture standards. The solution to this problem was achieved as follows. If a chemical is used alone, a realistic field density of 80 pcf (1.28 g/cc) or more can not be obtained. However, sand could be molded near 110 pcf (1.76 g/cc) but without any moisture in it. In order to use the advantage of both the sand and the chemical, it was decided to mix them together. The sand would provide most of the density while the chemical would provide the hydrogen for the moisture content reading.

It was felt at this time in the development of standards, that standards near 10, 20, and 30 pcf (0.16, .32, and .48 g/cc) used with the dry sand standard at 0 pcf (0 g/cc) moisture content would provide a well defined moisture calibration curve.

The first standard was designed for 10 pcf (0.16 g/cc) moisture content and was made with $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$ and C-190 Ottawa silica sand. It had a mass density of 107.8 pcf (1.73 g/cc). In determining the amount of $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$ to use, the desired moisture content, 10 pcf (0.16 g/cc), was divided by the water fraction, 0.511, for this chemical. This quotient gives the amount of chemical necessary for one cubic foot (0.03 m³), so it was multiplied by the volume of the mold, 4.0 cubic feet (0.11 m³), to derive the total amount of chemical needed. The remaining volume of the mold was then determined to derive the amount of sand needed.

The total weight of the $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$ and C-190 sand to be placed in the mold was divided into 20 equal portions. Each portion was thoroughly mixed to assure as much uniform gradation as possible prior to placing the material in the mold. Also, the $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$ was passed through a No. 10 sieve before mixing with the sand to make the sizes of the grains of the two materials nearly the same.

After ten portions were placed in the mold, an immersion-type vibrator was used to compact the bottom half of the standard. The remaining ten portions were prepared and placed in the mold separately until the mold was nearly filled. The vibrator was then reinserted the full depth of the standard to obtain maximum density. While the standard was being compacted, most of the remaining portions were added until the mold was completely filled and compacted as densely as possible. The design density was not obtained, therefore, some mix was left over.

The mixture was leveled to the top of the box and a polyethylene sheet was placed over it and sealed prior to weighing. The weight of the standard was then determined and the mass density ascertained. Nuclear tests for density and moisture content were then run on the standard every day for several weeks. Since that time, tests have been performed about once a week to determine if the standard is remaining stable.

As noted above, the designed mass density of 107.8 pcf (1.73 g/cc) was not reached — the actual density was determined to be only 100.4 pcf (1.61 g/cc). Since the designed density was not achieved some of the mixture was not used, and thus the designed moisture content also was not reached. The deficiency in moisture content was in the same proportion as the deficiency in density, since the portions were assumed to be uniform. To correct the designed moisture content of 10.0 pcf (0.16 g/cc) to the actual moisture content of the standard, the ratio of 100.4 pcf (1.61 g/cc) divided by 107.8 pcf (1.73 g/cc) was multiplied by 10.0 pcf (0.16 g/cc) moisture content. The result gave an actual corrected moisture content of 9.3 pcf (0.149 g/cc) for the standard. The count ratio determined for the standard was 0.492, which indicates a moisture content of 7.7 pcf (0.12 g/cc) from the manufacturer's curve.

At this stage, it was felt that a better mixture and standard could be achieved by using a smaller grain sand. A sieve analysis of the $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$ indicated that its grain sizes approximated those of C-109 Ottawa silica sand (Figure 7). Therefore, a standard was designed with C-109 sand and $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$ to replace the previously fabricated 10 pcf (0.16 g/cc) moisture content standard. This standard was molded and prepared as was the former one. The mass density was designed for 100 pcf (1.60 g/cc), since the designed density of the first standard was not achieved. However, because this sand had a maximum dry density of 103.4 pcf (1.66 g/cc) as compared to 109.5 pcf (1.75 g/cc) for the C-190 sand, the maximum density attained was only 96.0 pcf (1.54 g/cc). The moisture content was corrected for the deficiency in density and was determined to be 9.6 pcf (0.154 g/cc).

This standard was tested for several weeks, and then discarded in favor of the first standard because it was speculated that the C-109 sand and the $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$ had segregated during compaction. It was felt that since the two components of the standard had approximately the same grain size but varied about 1.0 in their specific gravities, the heavier sand grains had migrated to the bottom during vibration. The count ratio determined for this standard was 0.484, for a moisture content value of 7.5 pcf (0.12 g/cc) from the manufacturer's curve.

At this point, it was concluded that C-190 sand was superior to C-109 sand in providing a uniform gradation within the standards. Also, it was felt that there was a possibility of obtaining a better standard by using a chemical other than that used in the first mold. Therefore, $\text{Na}_3\text{PO}_4 \cdot 12 \text{H}_2\text{O}$ with a water fraction of 0.568 was used with C-190 sand at a moisture content of 20 pcf (0.32 g/cc). A design density of 108.5 pcf (1.74 g/cc) was attempted. The density obtained was determined to be 87.5 pcf (1.40 g/cc), thus the moisture content was 16.1 pcf (0.26 g/cc).

Since the density for the $\text{Na}_3\text{PO}_4 \cdot 12 \text{H}_2\text{O}$ did not approach the design density value, it was decided to go back to the $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$. Since the last standard had

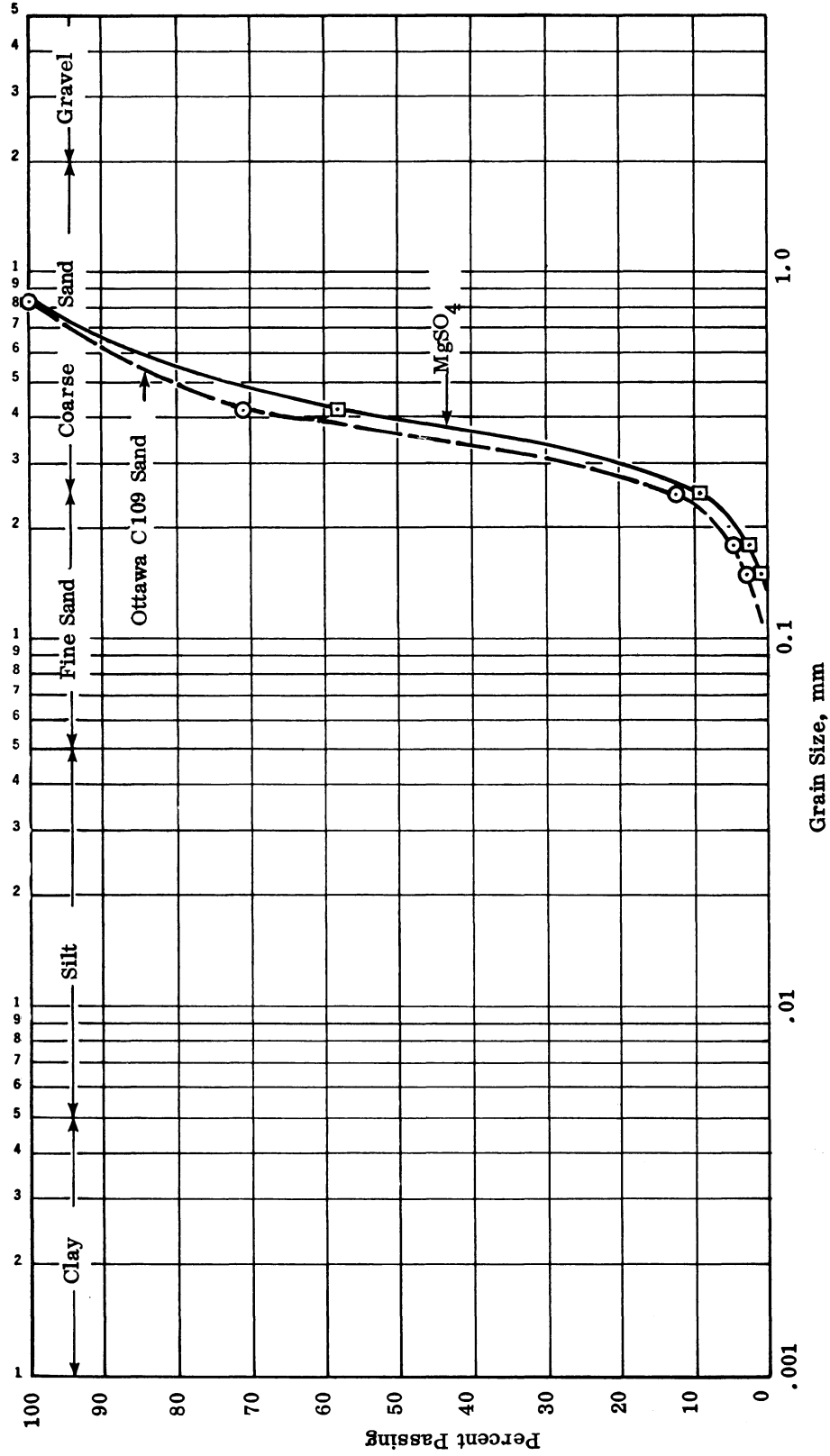


Figure 7. Gradation curves of C109 Ottawa silica sand and MgSO₄.

been designed for 20 pcf (0.32 g/cc) moisture content and corrected to 16.1 pcf (0.26 g/cc), it was decided to allow for the correction and design the next standard for 25 pcf (0.40 g/cc) moisture content and 100 pcf (1.60 g/cc) density. Again, however, the design values were not attained. The actual density was only 83.9 pcf (1.34 g/cc) and the corrected moisture content was 21.0 pcf (0.336 g/cc). The results of nuclear tests run on this standard indicated it to be stable and uniform, so it was retained.

Although four standards, including the dry sand, had now been developed, it was felt that a higher moisture standard was needed. $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$ was chosen to be mixed with C-190 sand for a standard designed for 100 pcf (1.60 g/cc) density and 35 pcf (0.56 g/cc) moisture content. The values attained were 73.8 pcf (1.18 g/cc) density and 25.8 pcf (0.41 g/cc) moisture content. Several weeks of nuclear testing indicated the standard to be **stable** and uniform and it was retained.

In attempting a more satisfactory standard, it was believed that since the density of the standard with $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$, dropped below 80 pcf (1.28 g/cc), a higher moisture content standard incorporating $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$ would have a density lower than 70 pcf. It was then decided to try $\text{Na}_3\text{PO}_4 \cdot 12 \text{H}_2\text{O}$ mixed with C-190 sand again. This mold was designed to have a density of 100 pcf (1.60 g/cc) and a moisture content of 28 pcf (0.45 g/cc), but the respective values attained were 75.5 pcf (1.21 g/cc) and 21.1 pcf (0.338 g/cc). Since the density was below 80 pcf, it was felt that it was not possible to make a standard with a high moisture content using either $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$ or $\text{Na}_3\text{PO}_4 \cdot 12 \text{H}_2\text{O}$.

During the molding and testing of the above moisture standards, two others were molded and tested but were discarded because of their lack of a uniform gradation. One was made from $\text{Na}_2\text{SO}_4 \cdot 10 \text{H}_2\text{O}$ and C-190 sand and the other from $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$ and C-190 sand.

Results

Table 2 is a summary of the data for all the standards molded and tested in this study except the wet sand standard.

At this time in the study six moisture standards were thought to be satisfactory for calibration purposes. Although two standards had moisture contents around 21 pcf (0.34 g/cc), they were made of different sand-chemical mixtures. In order to determine which of the two was the more satisfactory, nuclear readings were obtained for several months to get an average count ratio on the five chemical standards. The moisture calibration curve for this Model 2401 Troxler nuclear gage on these standards was determined twice using the linear regression equation. The first curve was determined using the data from five standards (i.e., excluding Mold #7). The standard error for this curve was 0.57 pcf (0.01 g/cc). The second analysis used the same data, except Mold #7 was substituted for Mold #5. The standard error for this curve was 1.25 pcf (0.02 g/cc), or an increase of 0.68 pcf (0.01 g/cc) over that of the first curve. Therefore, it was decided to discard Mold #7.

TABLE 2
SUMMARY OF DATA ON MOISTURE STANDARDS

Mold No.	Sand	Chemical	Design Values -- pcf (g/cc)		Actual Values -- pcf (g/cc)		Remarks
			Density	Moisture Content	Density	Moisture Content	
1	C 190	—	108.5 (1.74)	0	108.5 (1.74)	0 -	OK
2	C 190	MgSO ₄ · 7 H ₂ O	107.8 (1.73)	10.0 (0.16)	100.4 (1.61)	9.3 (0.149)	OK
3	C 109	MgSO ₄ · 7 H ₂ O	100.0 (1.60)	10.0 (0.16)	96.0 (1.54)	9.6 (0.154)	Nonuniform; discarded
4	C 190	Na ₃ PO ₄ · 12 H ₂ O	108.5 (1.74)	20.0 (0.32)	87.5 (1.40)	16.1 (0.26)	OK
5	C 190	MgSO ₄ · 7 H ₂ O	100.0 (1.60)	25.0 (0.40)	83.9 (1.34)	21.0 (0.336)	OK
6	C 190	MgSO ₄ · 7 H ₂ O	100.0 (1.60)	35.0 (0.56)	73.8 (1.18)	25.8 (0.41)	OK
7	C 190	Na ₃ PO ₄ · 12 H ₂ O	100.0 (1.60)	28.0 (0.45)	75.5 (1.21)	21.1 (0.338)	OK but discarded in favor of Mold No. 5
8	C 190	Na ₂ SO ₄ · 10 H ₂ O	98.2 (1.57)	10.0 (0.16)	97.8 (1.57)	10.0 (0.16)	Nonuniform; discarded
9	C 190	MgSO ₄ · 7 H ₂ O	95.0 (1.52)	10.0 (0.16)	96.3 (1.54)	10.1 (0.162)	Discarded; did not need.

Figure 8 shows the soils and manufacturer's curves with their standard errors imposed over the data from each of the five standards. The linear regression line for the five standards and its standard error are also shown. As can be seen the standards curve and the soils curve are roughly parallel. At around 25 pcf (0.40 g/cc) moisture content they vary about 2 pcf (0.03 g/cc). However, at the lower moisture contents, the two curves approach each other and the difference is only 1 pcf (0.02 g/cc) around 5 pcf (0.08 g/cc) moisture content.

The plots of the soils and manufacturer's curves show a larger difference at 25 pcf (0.40 g/cc) than that between the soils and standards curves. At 25 pcf (0.40 g/cc) the two curves vary approximately 4 pcf (0.06 g/cc). As the moisture content decreases, the two curves approach each other and cross at around 6 pcf (0.10 g/cc) moisture content. Below this moisture content, they diverge again to about 1 pcf (0.02 g/cc) difference near 0 moisture content.

From the figure it can be seen that the standards curve will give a higher moisture content than the other two curves for the same count ratio for soils with moisture contents greater than 2 pcf (0.03 g/cc). For soils with moisture contents greater than 6 pcf (0.10 g/cc), the manufacturer's curve will give the lowest moisture content of the three curves for the same count ratio.

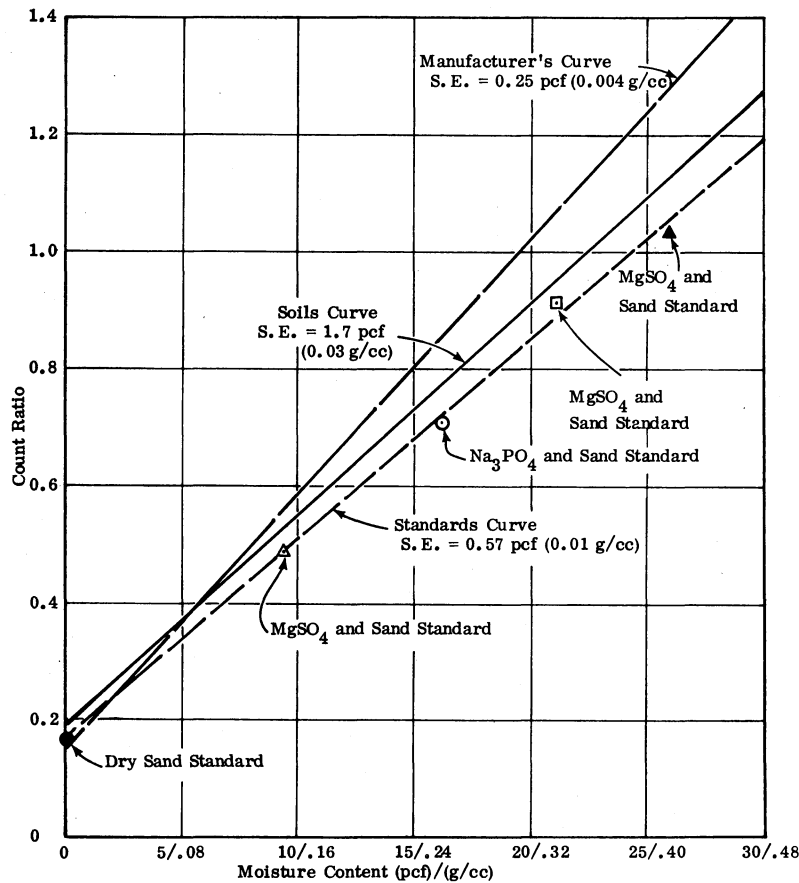


Figure 8. Moisture calibration curves for Model 2401 gage.

CONCLUSIONS

The following conclusions can be drawn from the work carried out in this study:

1. Two calibration curves with a linear relationship between the count ratio and the moisture content are necessary for accurate readings from the Model 227 gage.
2. The two calibration curves for the Model 227 gage are best divided by the optimum moisture content of the soil. The separation value for the optimum moisture contents seems to be around 21%.
3. The Model 2401 gage needs only one calibration curve and this produces a standard error of 1.7 pcf (0.03 g/cc).
4. Five moisture standards were developed at 0, 9.3, 16.1, 21.0, and 25.8 pcf (0, 0.15, .26, .34, and .41 g/cc) moisture contents. The calibration curve from these standards has a standard error of 0.57 pcf (0.01 g/cc).
5. The soils curve and standards curve are nearly parallel. The difference between the two varies from 1 to 2 pcf (0.02 to 0.03 g/cc).
6. The manufacturer's curve gives a lower moisture content than do the other two curves for the same count ratio, except for moisture contents below 6.0 pcf (0.10 g/cc). The difference between this curve and the soils calibration curve increases with moisture content. At 6 pcf (0.10 g/cc) moisture content there is no difference, while at 25 pcf (0.40 g/cc) moisture content the difference is approximately 4 pcf (0.06 g/cc).

RECOMMENDATION

The primary chemical moisture standards developed in this study are stable and give satisfactory results. The dry sand standard (zero pcf moisture content) and the $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$ and C-190 sand standards at approximately 9 and 21 pcf (0.14 and 0.34 g/cc) moisture contents will be reproduced in the Central Office Laboratories of the Virginia Department of Highways. If, as anticipated, these standards produce a curve similar to the one developed in this study, a recommendation as to their adoption for use will be made to the Virginia Department of Highways.

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