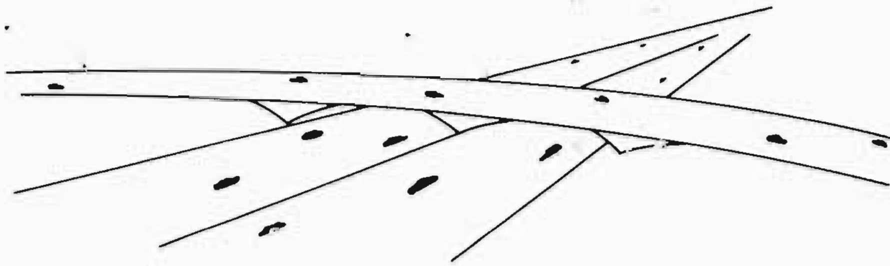


# DEPARTMENTAL RESEARCH

Report Number: **SS-17.1**

## DETERMINATION OF ASPHALT AND MOISTURE CONTENT BY THE NUCLEAR METHOD



TEXAS HIGHWAY DEPARTMENT

# DETERMINATION OF ASPHALT AND MOISTURE CONTENT BY THE NUCLEAR METHOD

By

James R. White, Jr.
Resident Engineer
District 14

SPECIAL STUDIES REPORT
NUMBER SS 17.1

August 1971

# PREFACE

The use of nuclear testing has been shown to be accurate and speedy in control of moisture and density in subgrade and base materials. This thesis was designed to apply nuclear principles to the control of asphalt content in hot mix asphaltic concrete and explore other practical applications in every day laboratory and field control operations.

The author wishes to express grateful appreciation to Dr. Clyde E. Lee of the Department of Civil Engineering who served as thesis supervisor and gave valuable guidance and help in every phase of this project. Thanks are also given to Dr. B. F. McCullough of the Civil Engineering Department for his assistance in preparing this report.

Special thanks are expressed for the life of Mr. Thomas K. Wood, District Engineer, who died after a long illness on March 19, 1971. Without his inspiration, faith and help, this project would not have materialized.

Acknowledgments and thanks are extended to Mr. J. M. Owens, District Engineer, District 14, Texas Highway Department for his patience and help, and likewise to Mr. James F. Todd, District Laboratory Engineer, who provided facilities, guidance, and counsel throughout this project.

The author also wishes to thank Mr.Gilbert L. Galletin for his advice and help in preparing mix designs and samples, and to Messrs. Thomas Roy Bullock, David T. Parsons, Gerald L. Turner, Jr., and Marvin L. Stark for their assistance.

Thanks are also due Sheila Huntress, Mrs. Maureen Tucker and all others who assisted with the manuscript.

The financial support for this project was furnished by the Texas Highway Department.

James R. White

August , 1971

Austin, Texas

# ABSTRACT

A nuclear asphalt content gauge was evaluated to determine the variables which affect precision of measurement, to investigate the practicality of using the instrument in field control of hot mix alphaltic concrete, and to explore other uses for the gauge in normal testing operations. In measuring asphalt content, particular attention was given to compensating for any moisture in the samples and to developing a common calibration curve in which asphalt content is expressed as a percent of absolute volume. The use of 1000 gram molded cylindrical hot mix samples for calibrating the gauge was explored. Highway base and subgrade materials were used in a moisture calibration study to determine the feasibility of using the nuclear gauge in other areas of control. It was concluded that the gauge is a practical, accurate, safe, and usable tool for measuring asphalt and moisture content in highway materials.

TABLE OF CONTENTS

## LIST OF FIGURES

LIST OF TABLES

# CHAPTER 1

# INTRODUCTION

In the determination of asphalt content by reflux extraction methods, problems such as the time required to perform the test and possible inaccurate test results due to absorptive aggregates have prevented consistently high level quality control. Nuclear methods for measuring asphalt content show promise of being accurate, speedy, clean and will allow performance of more tests for better quality control. In addition, these methods may be used as standard laboratory tests to determine the moisture content of soils.

This project was conceived in order to evaluate practical applications for nuclear instrumentation and to implement the findings directly into normal Texas Highway Department testing operations where applicable.

## CHAPTER 2

## DESCRIPTION AND OPERATING PRINCIPLES OF EQUIPMENT

The Troxler Model 2226 Asphalt Content Gauge was used in this study. As suggested by the instrument configuration shown in Figure 1, this instrument is intended for use in the laboratory (central or field), and is not designed to monitor "in place" asphalt content of compacted materials.

Figure 1. Nuclear Asphalt Gauge

Guage operation is based on the principle of neutron moderation; the same principle which has been used successfully in determining soil moisture (1) (2).* "Fast" neutrons emitted by the source are slowed or moderated primarily by the presence of hydrogen and counted by Helium-3 detector tubes after passing through a sample (2). Since hydrogen is present in asphalt (3), the number of fast neutrons which pass through the sample without modification is directly proportional (within certain limits) to the amount of asphalt in the sample assuming that no other neutron modifiers are present.

The gauge actually responds to total hydrogen density (2) and does not distinguish between hydrogen in asphalt or in water. For calibration purposes, weight and volume of the sample are kept constant thereby resulting in count rate being proportional to percent asphalt rather than to asphalt density expressed as weight per volume of sample. Carbon, phosphorous, and chlorine are also good moderators of fast neutrons although much less efficient than hydrogen (3).

A general expression for gauge response is thus:

---

*Numbers in parenthesis indicate entries in the Bibliography.

Count Rate = f(Asphalt, Water, Chemically bound hydrogen, and Chemical composition of aggregate).

Other variables which affect count rate (therefore calibration) are discussed later.

In the Troxler gauge, a standard stainless steel sample pan, approximately 9 by 7 by 4 inches, is filled with the mix and inserted into a drawer located between the source and detector tubes for testing. The radioactive source used is 300 millicuries of Americium Beryllium and the detection system utilizes Helium-3 detector tubes. The system uses the direct transmission technique which has proven superior to backscatter in other studies involving nuclear density gauges (1).

A multiple detector system which monitors standard radiation counts simultaneously with test counts is used to minimize the effects of electronic drift. The read-out is automatically compensated for changes in count rate which may have occurred due to system variables.

Figure 2 shows a schematic layout of the system. The photographs in Figure 3 show a sample in the open drawer and the gauge read-out display.

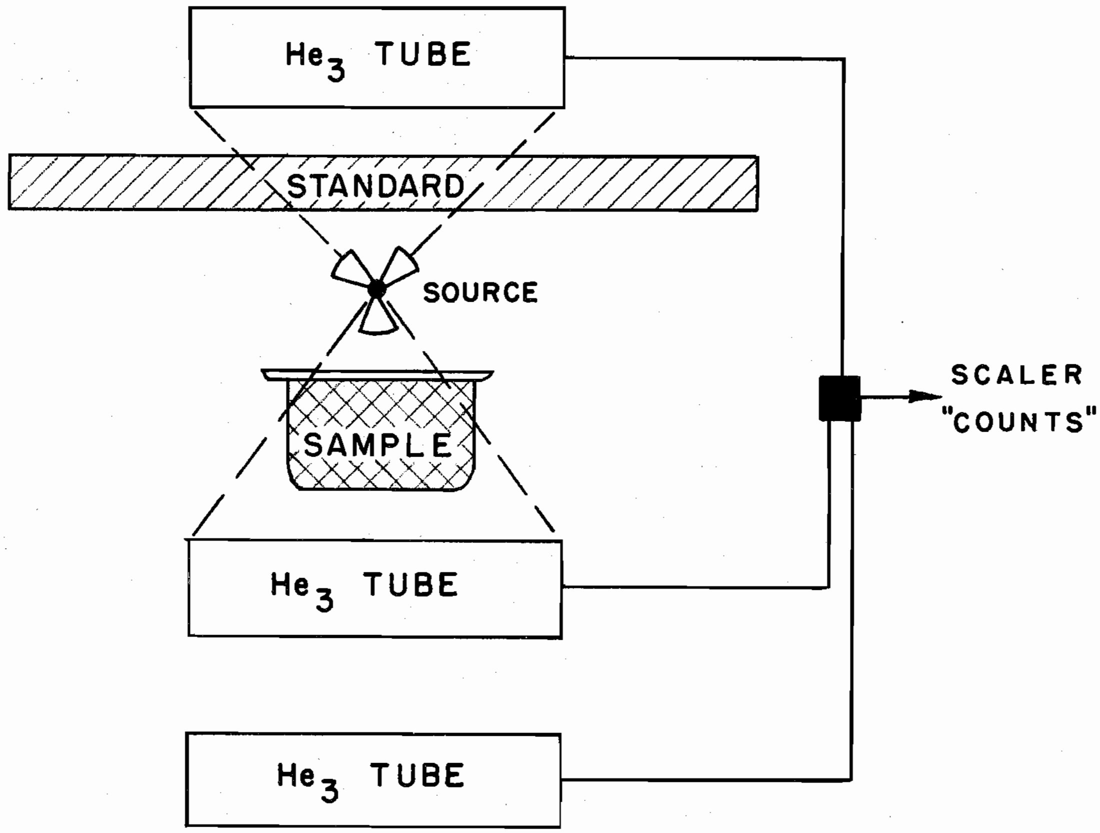



Figure 2. System Schematic

a) Sample Placement in Drawer


b) Gauge Readout

Figure 3. Photographs of Operation

The gauge is designed to operate on standard 60 Hz, 115 $\pm$ 10 volt AC. It weighs 125 pounds and is 16 inches cubic in size.

## CHAPTER 3

## LITERATURE REVIEW

A review of the literature indicates that the following variables affect determination of asphalt content by nuclear methods.

1. Materials in Mix (Combination)
2. Asphalt Type
3. Gauge Stability
4. Sample Preparation
5. Moisture in Aggregate
6. Gradation

Probably the earliest work on this subject was done in 1955 at the University of Wyoming by Lamb and Zoller (2). In their work, the direct transmission technique was used with a Ra - D - Be source and an enriched boron - flouride 10 detector tube. Asphalt mix samples were constructed approximately 2 x 11 x 14 inches. A relationship between count rate, percent asphalt, and unit weight of specimen was presented to demonstrate the feasibility of using nuclear methods.

In about 1962, Varma and Reid (3) did considerable experimentation with a Nuclear-Chicago down hole probe using samples composed of alum, sand and water, and also asphalt samples. Their work explored the chemistry of asphalts, the theory of neutron scattering and indicated that the type of asphalt would probably be a major factor in establishing calibration. Cylindrical samples 8 inches by 8 inches with an access hole in the middle were molded for study.

Howard and Covault (4) determined that it was not feasible to use the neutron backscatter technique for measuring the asphalt content of in-place bituminous concrete pavement. Apparently, the basic problem was that underlying materials seriously influenced the count rate. No mention was made of the type of device used.

In about 1963, Walters (5) of the Colorado Department of Highways perfected a procedure for determining asphalt content at the job site. In his method, a sample of the mix was picked up behind the paver, and placed in a one gallon container. The container was inserted in a small test chamber at a temperature of approximately 200$^{\circ}$ to 230$^{\circ}$ F and then tested by the backscatter technique using the Troxler surface moisture gauge. It was found that asphalt penetration value and aggregate gradation were the main variables which affected calibration. Four master calibration curves which related neutron count ratio to asphalt content for two penetration grades of asphalt and two standard aggregate gradations were developed. Using nuclear results obtained from these calibration curves, the difference between nuclear and plant calculated and nuclear and the reflux method was no greater than 0.3% asphalt, 95% of the time.

In a discussion following this article, Qureshi (6) confirmed Walters' findings using the same equipment; however, his samples were molded square and apparently only one material was investigated. Problems were encountered with the stability of the equipment, thus requiring the use of count ratio rather than counts. It was stated that points deviated a maximum of ± 1/4 percent asphalt from the derived calibration curve.

The first generation instrument (Model AC - 200) of the Troxler asphalt gauge was evaluated at the University of Southwestern Louisiana in about 1967. The same configuration of sample presently used (7 x 9 x 4 inch stainless steel pan filled to capacity) was prepared by carefully combining aggregates and asphalt. All molding was done at the same bulk density.

A statistical procedure for determining the counting time was presented. For the materials studied, approximately eight minutes counting time was required for a precision of ± 0.2% asphalt, 95% of the time. The effect of aggregate gradation on neutron transmission was recognized and improvements in gauge design were also recommended.

Grey of the Pennsylvania Department of Highways (8) began evaluation of a gauge developed by Nuclear Chicago Corporation in about 1967. The gauge consisted of an $Am^{241}$ Be source, four $He^3$ detectors, a preamplifier, and a sample cavity interposed between the source and detectors. The count ratio calibration procedure was used with the standard count being corrected for background count. Variables recognized in the laboratory calibration of this instrument were aggregate type and gradation, asphalt effect, and nonuniformity of sample preparation. Standard deviations in the range of .03% to .09% asphalt were obtained in the laboratory for various mixes. Values obtained in the field by nuclear methods at one plant had about the same variation as those from extraction tests.

Further field studies were carried on by Grey using the same gauge and reported in 1970 (9, 10). In this work, the aggregate effect was further verified along with possible errors due to entrapped moisture. No gradation effect was noticed when comparing one aggregate of two widely different gradations. A slight calibration shift was noted for different batches of asphalt from the same manufacturer. Comparison of nuclear and extraction test results indicated that one is about as reliable as the other.

As part of an overall study of nuclear test equipment, the Nebraska Department of Roads evaluated the Troxler AC - 200 Asphalt Content Gauge and published a progress report in January 1968 (11). Nuclear values were compared with extraction values obtained from a highly accurate method of extraction. A procedure for duplicate testing of split samples was used in order to have some measure of precision on the same sample. Field nuclear asphalt contents were generally found to be lower than extraction values which is just the opposite as found in most of the references previously cited. The presence of carbon in limestone dust was found to affect nuclear test results. Temperature of the mix had a significant effect on count rate. On the basis of some 559 tests on 18 projects, average standard deviations of $\pm$ 0.19% for nuclear and $\pm$ 0.06% for extraction were obtained. The plant asphalt contents ranged from 3.9% to 7.4% on these projects. The standard deviations included variations in material and also sampling errors. It was concluded that even though the variation in nuclear was larger than that for the extraction test, the nuclear test had the advantage of being much faster. Work done at a later date indicated that duplicate nuclear testing rather than counting for longer periods of time yielded more accurate nuclear test results.

In December 1970, Hughes (12) published what is probably the latest information on this subject. The Troxler Model 2226 (the same instrument used in this study) was evaluated in both the laboratory and in the field lab and compared with conventional reflux values. The aggregate and asphalt effects were examined, and it was concluded that precision attained in the laboratory was equivalent to 0.06% asphalt content with a 45 second count. A change in gradation did not require recalibration; however, the coarser mixes reduced the precision of measurement considerably.

The gauge had to be recalibrated each time a different aggregate was used and periodically during field use in order to compensate for changes in aggregate, asphalt and moisture. Sample preparation and uniformity were found to be very important. No temperature effect was noted. It was concluded that the nuclear method was superior due to speed, and the accuracy was at least as good as that of the reflux extractor.

## CHAPTER 4

## INSTRUMENT CHECK PROCEDURES

In addition to check procedures suggested by the manufacturer, other studies were made to determine the operating characteristics of the gauge.

### Environmental Factors

Past experience has shown that objects in the area of a nuclear gauge affect the count rate (1). For this reason, an analysis of variance experiment was designed to determine if placement of the Troxler Model 2226 gauge had any effect on readings.

With the gauge placed on a plywood stand 34 inches high, "1" position (approximately 2 1/2 minute counts) readings were obtained on a 6% asphalt content sample of hot mix at room temperature of approximately 74$^{\circ}$ F. The instrument was placed in a random sequence against a brick wall, a plaster wall, in the middle of a room with no object within three feet, and in the middle of a room with the gauge directly on a concrete floor. The readings are shown in Table 1 along with the Analysis of Variance (AOV) table.

Table 1

Environmental Study

| | Plaster Wall | Brick Wall | Middle Room | Concrete Floor |
|---|---|---|---|---|
| | 44678 | 44969 | 44393 | 45887 |
| | 44846 | 44862 | 44379 | 46026 |
| | 44965 | 45050 | 44555 | 46077 |
| | 44858 | 44994 | 44623 | 45822 |
| | 44815 | 44917 | 44365 | 45854 |
| Avg.Count= | 44832 | 44958 | 44463 | 45933 |
| Avg. Diff. in Indicated Asphalt Content* | +0.25% | +0.35% | 0 | +0.95% |

*With reference to middle of room

AOV Table

| Source of Variance | Degrees Freedom | Sums of Squares | Mean Square | F Level |
|---|---|---|---|---|
| Between Treatments | 3 | 5,901,547 | 1,967,182 | 186** |
| Within Treatments | 16 | 169,029 | 10,564 | |
| | 19 | 6,070,576 | | |

**Highly significant ($1 - \alpha = .99$)

From this analysis, it may be concluded that the variation in gauge readings resulting from placing the gauge at locations other than on a stand in the middle of a room is greater than would be expected due to chance alone in 99 of 100 cases.

Using the Duncan Multiple Range test (13), differences in means for each treatment were examined. Comparing each mean with all others, at a 95% confidence level all possible mean combinations were significantly different except those obtained against the plaster and brick walls. It was therefore concluded that all readings should be obtained on the stand with objects greater than three feet distant. All further laboratory studies reported herein were made with the gauge on the plywood stand in the middle of a room at approximately $74^{\circ}$ F.

Count Stability

An attempt was made to evaluate both short and long term guage stability by counting neutrons transmitted through a 6,000 gram molded sample. The mix was primarily limestone, contained 6% asphalt (Gulf States AC-10) and had a Texas Highway Department Type D gradation (14, 15). Table 13 (shown on p. 60 of this thesis) shows the gradation specifications of this mix.

Short Term. When the sample had cooled to approximately $70^{o}$ F after preparation, twenty "1-position" repeated counts were taken and the standard deviation (denoted sigma - $\sigma$) was computed to be $\pm$ 95 counts. In terms of asphalt content, this corresponds to about $\pm$ 0.06% as determined from a calibration curve (developed in later studies in this report) for the material under test and having a slope of 1604 counts per 1% asphalt. If four 1-position counts were averaged, the standard deviation (68% confidence level) for the mean would probably be: $s_x = \sigma/\sqrt{4} = .06/2 = \pm 0.03\%$ asphalt, or twice this value ($\pm$ 0.06%) for the 95% confidence level.

Long Term. Two studies were made to evaluate long term gauge stability. Figure 4 indicates the pattern of standard counts obtained over a 40 day period with no sample and the drawer closed. Each point is an average of four 1-position counts taken in succession during a 15 minute interval. The standard deviation for the mean values was calculated to be $\pm$ 36 counts (about $\pm$ 0.02% asphalt), or for each individual observation: $\sigma = \sqrt{4}\,(36) = 72$ counts. This corresponds to about $\pm$ 0.05% asphalt.

Figure 5 shows the results of counts over a 125 day period using the same sample as in the short term

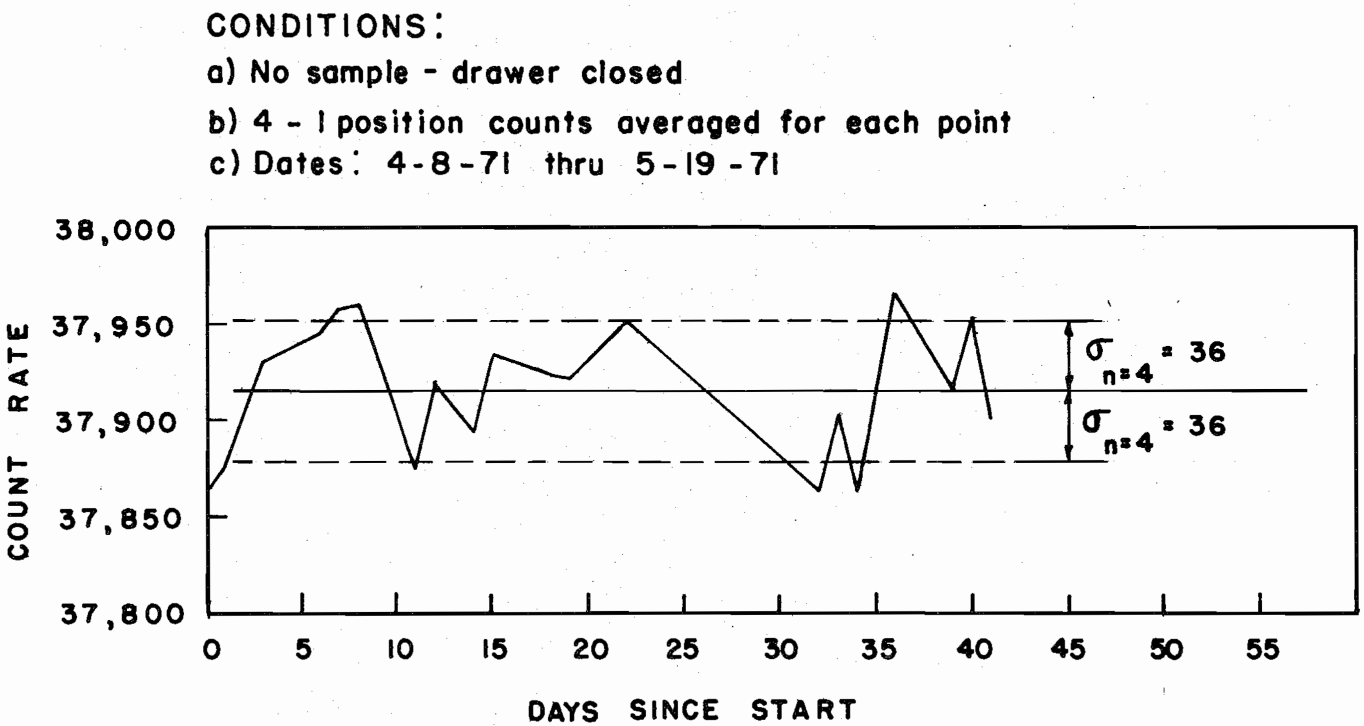
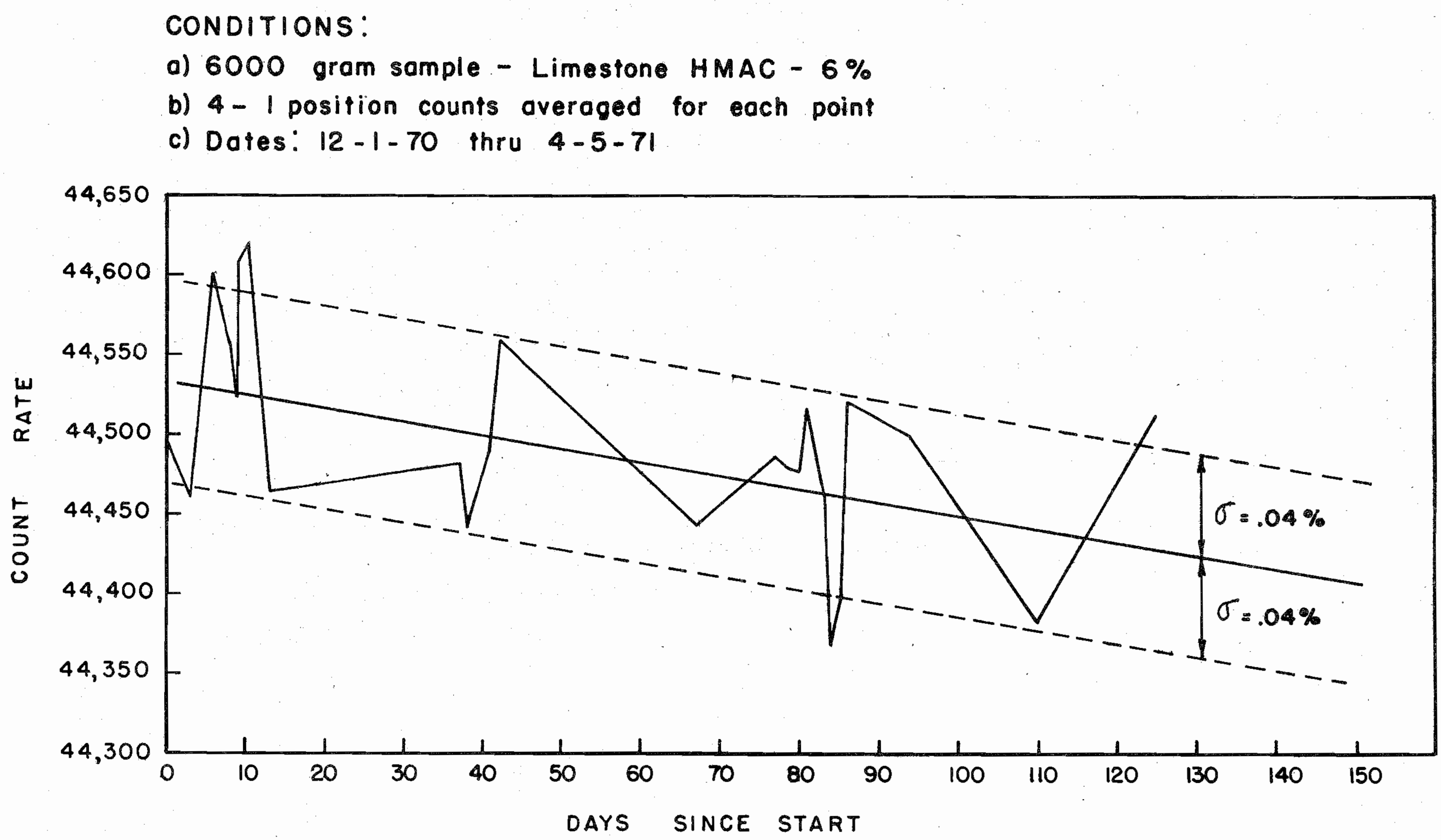



Figure 4: Long Term Stability, No Sample

Figure 5. Long Term Stability, 6000 gram HMAC Sample



analysis. Each point represents four 1-position counts averaged. It appears that age caused a loss of volatiles over this period; therefore a trend line was constructed and 68% of the mean values fell inside an equivalent ± 0.04% band around this line. This would indicate a standard deviation for the mean of 4 tests of about ± 0.02% asphalt.

Results of the short and long term tests taken together would indicate that a precision no better than ± 0.03%, 68% of the time, or ± 0.06%, 95% of the time could be expected when averaging four 1-position counts. As a practical matter, it should be expected that precision will be less due to less than ideal operating conditions and inherent material variables.

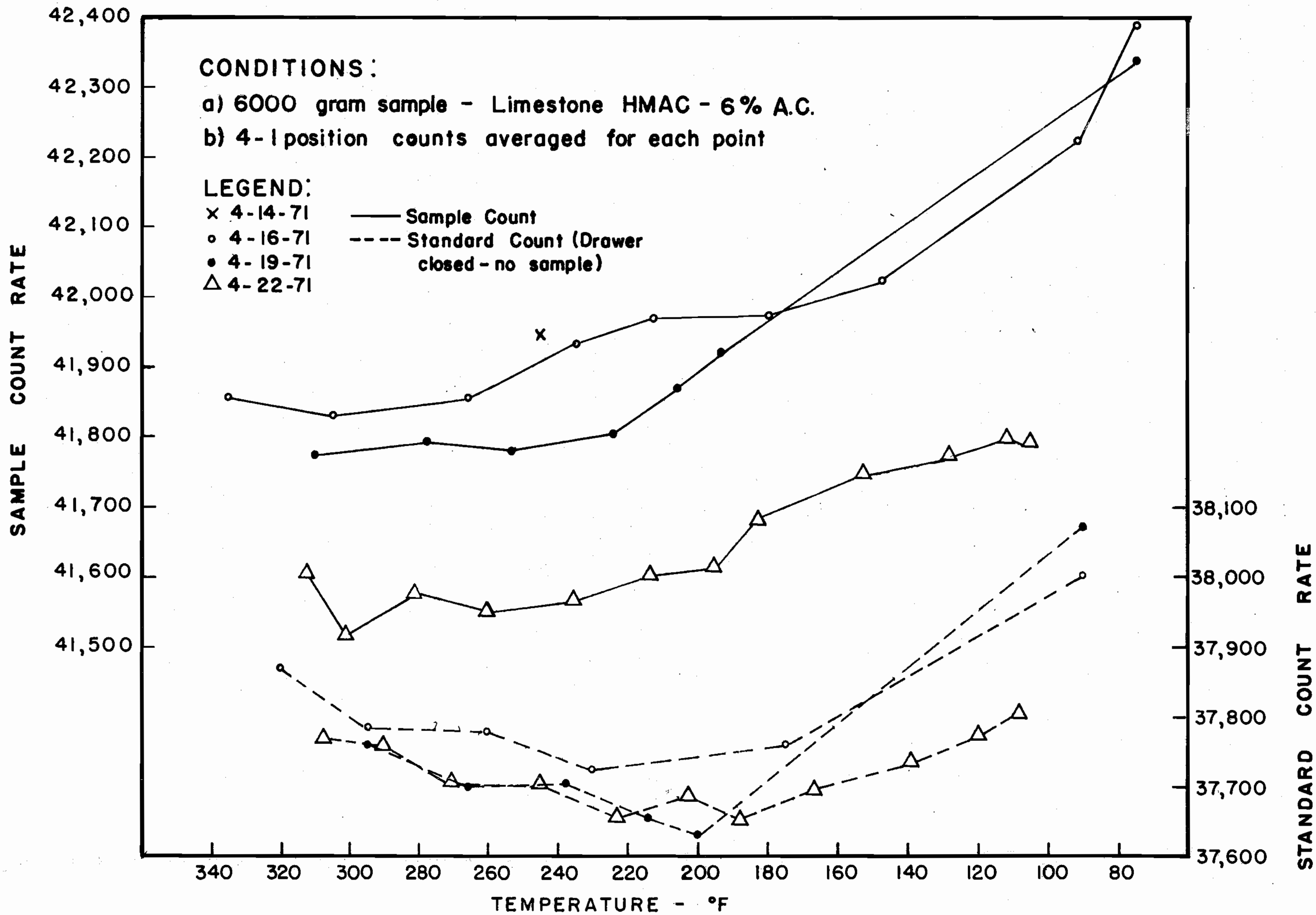
It is recognized that the self-standardizing feature of this gauge may allow equal precision for shorter counting periods (12). However, at the start of this investigation, information was not available to verify this point, and for the sake of uniformity, the decision was made to continue averaging four 1-position counts.

<u>Temperature Stability</u>

The effects of specimen temperature on gauge performance were investigated by heating the same pan of Hot Mix Asphaltic Concrete (HMAC) several times to $300^{\circ}$ F or higher without remolding, and then allowing it to cool while counts were being obtained. Each point in Figure 6 represents four 1-position counts averaged. Four standard counts with no sample in the drawer were obtained after each set of test counts. The results indicate that the test count is reasonably stable in the range $230^{\circ}$ F to $330^{\circ}$ F and then begins to increase at a rate of about 50 counts per $20^{\circ}$ F down to $100^{\circ}$ F. This effect was also noted when testing other samples at $250^{\circ}$ F then again at $70^{\circ}$ F. The standard count (drawer closed no sample) decreased until the temperature dropped to about $200\text{-}220^{\circ}$ F then began to increase as temperature dropped. The progressively lower sample count obtained each time the sample was reheated probably indicates a loss of volatiles from the asphalt.

In order to avoid a temperature effect, results of this study indicate that testing should be done between temperatures of $230^{\circ}$ F and $300^{\circ}$ F. Either a regression equation incorporating a temperature variable, or a

Figure 6. Temperature Stability Test



separate calibration for each $10^{\circ}$ F below $230^{\circ}$ should be developed to obtain satisfactory precision. The decision was made to perform all tests as close to $250^{\circ}$ F as possible and develop a separate calibration at $70^{\circ}$ F if needed.

Radiation Levels

The radiation level monitored at the front and top surface of the gauge was 1 to 2 millirems per hour or about the same dose as obtained from an average luminous dial wrist watch. At a distance of one meter, the radiation level drops to 0.1 mr/hr which is well below Texas Health Department and Atomic Energy Commission requirements. When compared to the presently used asphalt extraction procedure which utilizes liquids that are toxic and flammable, it appears that the nuclear method is perhaps potentially less hazardous if ordinary safety procedures are followed. The instrument operator should, however, be familiar with radiation safety practices, such as safety thru distance, time, shielding and personal radiation monitoring equipment (film badge, dosimeter, survey meter).

## CHAPTER 5

## FACTORS AFFECTING CALIBRATION FOR HOT MIX ASPHALTIC CONCRETE

In this chapter, the major variables discussed in the literature are investigated with particular emphasis on the effect of moisture in the mix.

### Sample Preparation

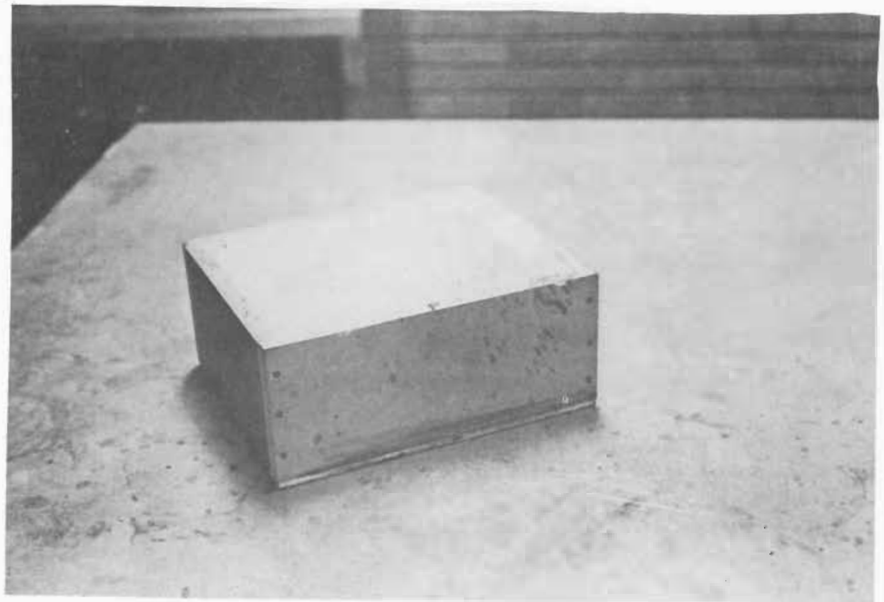
Initial attempts to calibrate the gauge indicated that uniformity of sample preparation was extremely important. Figure 12 (see p. 34) demonstrates this fact by the standard errors for each curve. Curve 1) had a standard error of ± 0.19% asphalt because the molding procedure was not as well controlled as that in curve 2) which had a standard error of ± 0.08% asphalt. In this particular case, the major errors were in uniformity of sample volume.

Rather than using convential sample splitting techniques after mixing, all specimens were combined from sieved materials according to particle size and gradation of materials available (14). Full pan samples were mixed in two equal batches since mixing facilities were not available to handle 6000 gram batches. Appendix A gives details on sample preparation and testing.

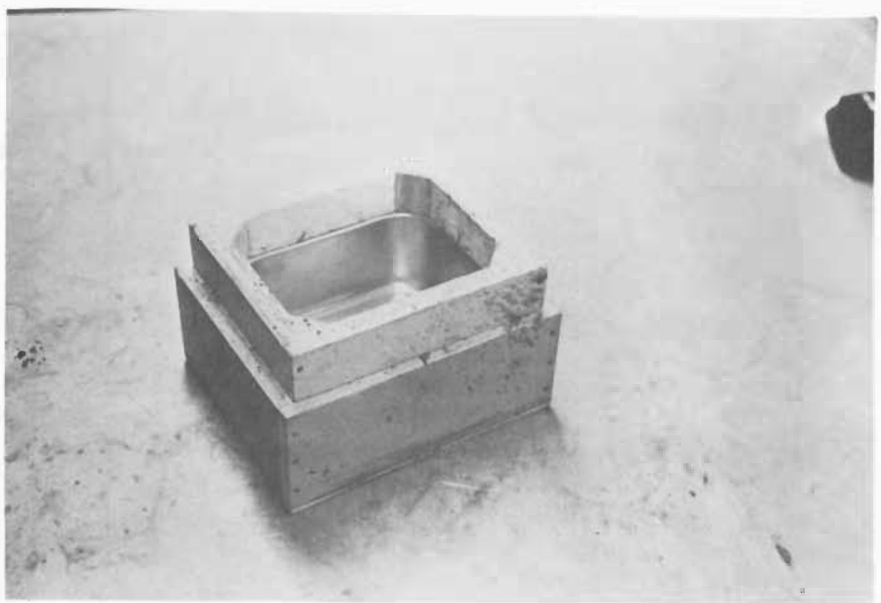
Figures 7 thru 9 show the equipment used to mold large samples. A mold was constructed to reinforce the sample pan in order to minimize expansion during pressing operations. A 1/2-inch thick steel plate was cut to fit into the top of the pan, then fitted to a standard mechanical press. Material placed in the pan was compressed in one operation regardless of the amount used. Sample height was controlled by using a gauge constructed from two pipes, one fitting inside the other, placed over the press stem as shown in Figure 9.

Figure 10 shows the volume calibration of the sample pan used throughout this project. Volumes were determined by adding known weights of water into the pan.

Since the nuclear gauge actually indicates hydrogen density (or asphalt density), it was reasoned that there might be some advantage to calibrating the gauge readings under conditions such that asphalt density would be numerically equal to percent asphalt. In this case, asphalt density is defined as the scale weight of asphalt in the mix which occupies a known pan volume. By making

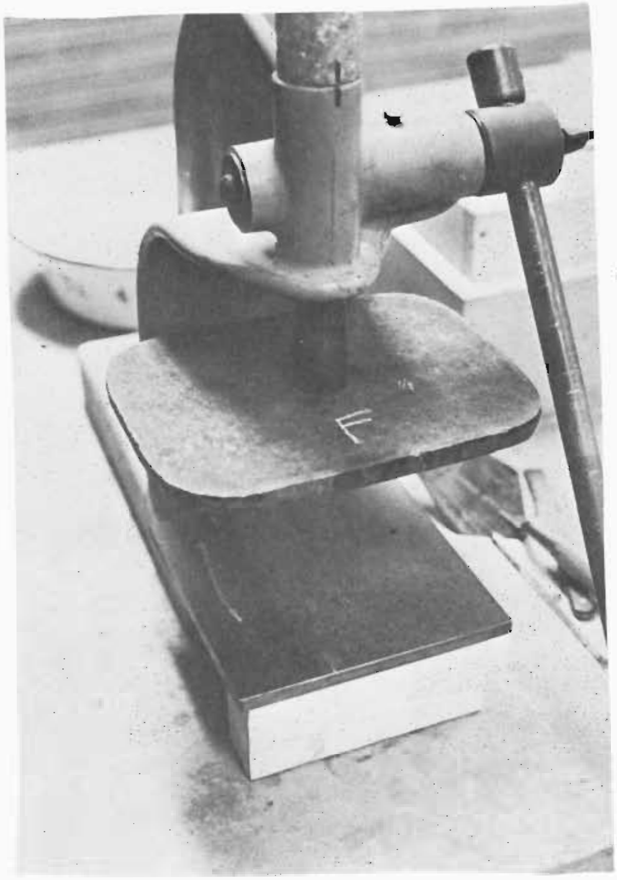


a) Mold

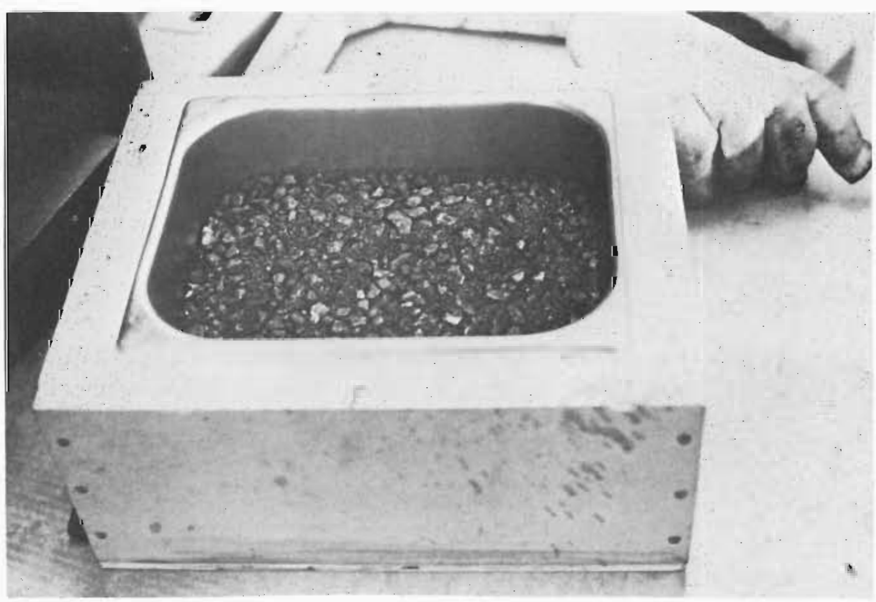


b) Mold with Sample Pan and
Overflow Ring

Figure 7. Sample Mold



a) Mechanical Press Fitted to Receive 1/2" Steel Press Plate



b) 3000 gram Sample After Molding

Figure 8. Molding Press and Molded Sample

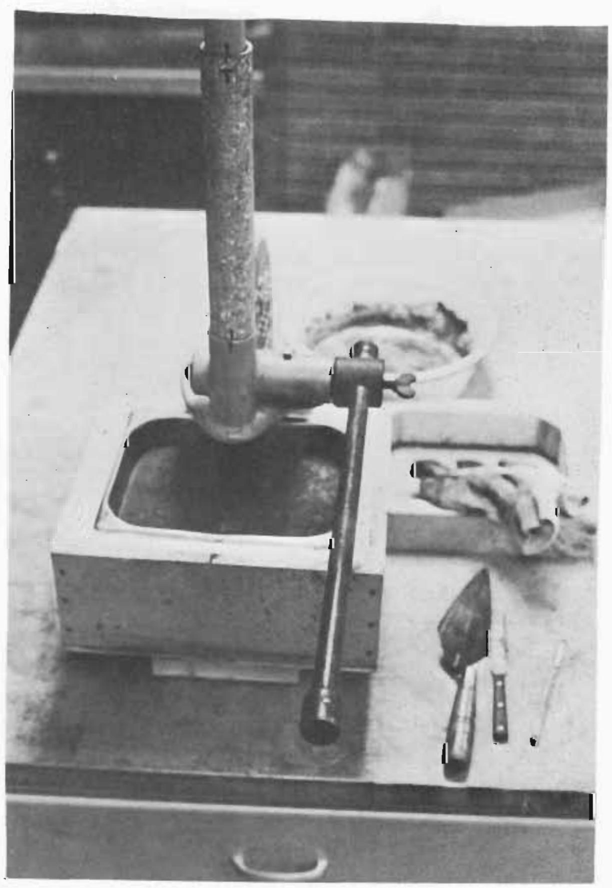
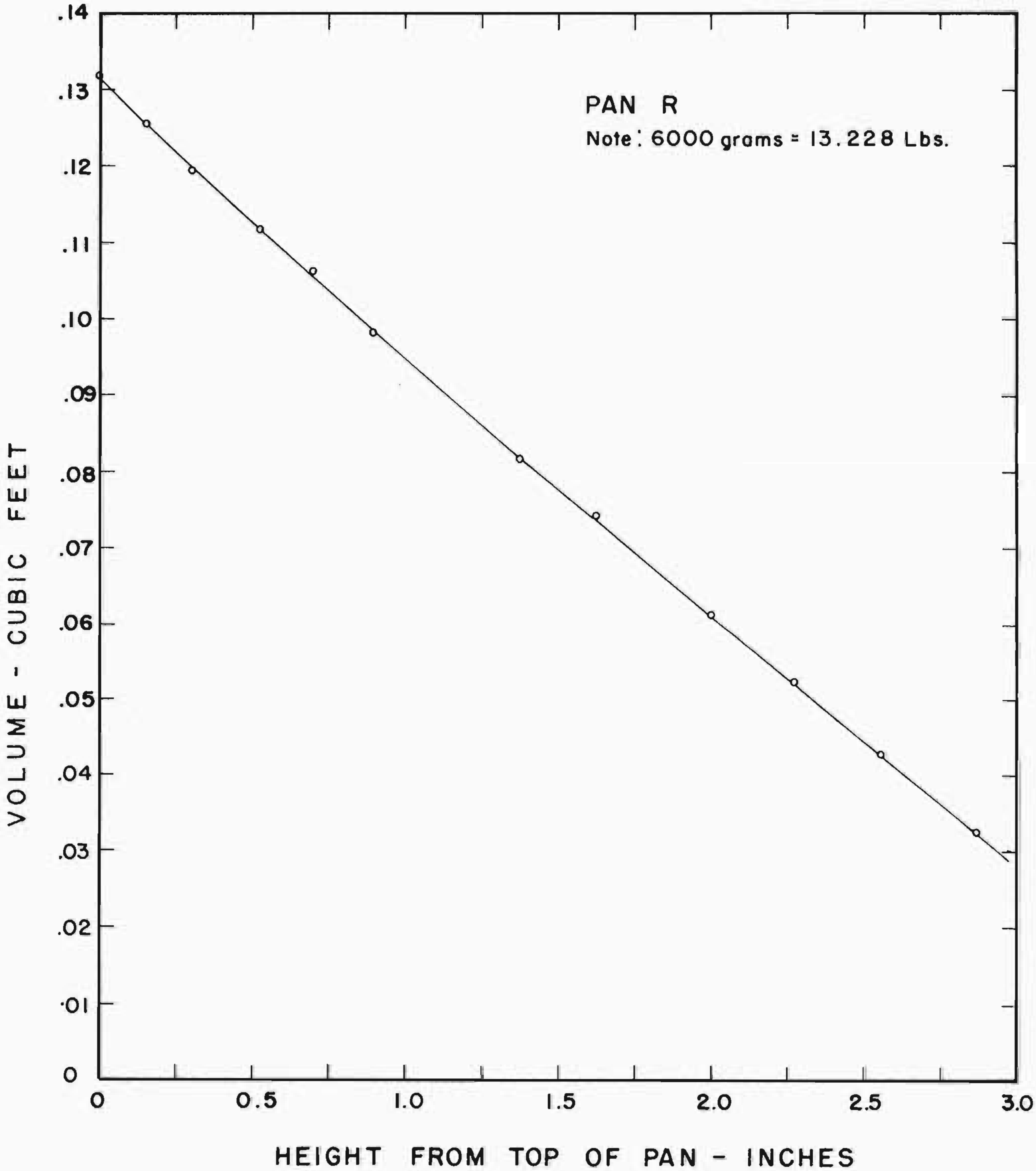


Figure 9. View showing Pressing Operation and Height Gauge Fitted Over Press Stem

Figure 10. Pan Calibration

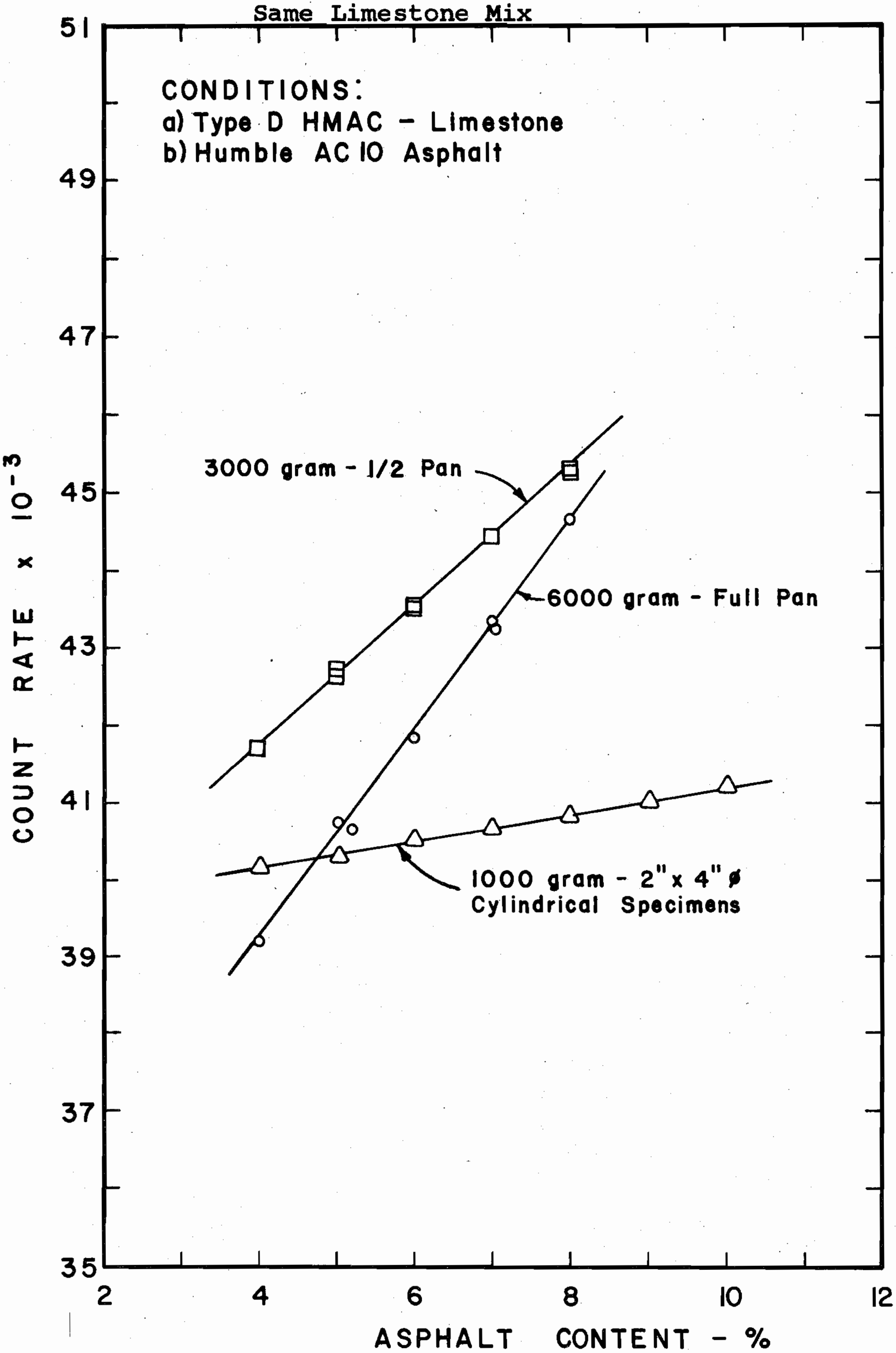


the molded density of the specimens 100 pounds per cubic foot, asphalt density would be equal to percent asphalt. For normal weight materials, this could be approximated by manipulation of the weight-volume relationship at some median asphalt percent, and then holding this relationship constant for all calibration samples. In some cases, the densities required to obtain this equivalency were greater than could be achieved. This calibration technique was dropped from consideration because it was impractical. It should be noted, however, that for normal unit weight materials, 13.2 pounds (6000 grams) of material with 6% asphalt compacted to the top of the pan (0.132 cu.ft.) will approximate 6 pounds per cubic foot asphalt density.

Sample Size Effect

In order to save calibration time, smaller specimens than that recommended by the manufacturer were tried. The gauge response to different size specimens is shown in Figure 11. The curve for 6000 grams represents a full pan, 3,000 grams represents one-half pan, and the 1000 gram curve was developed using standard Texas High Department two inch high by four inch diameter

Figure 11. Sample Size Effect on Same Limestone Mix



molded cylindrical specimens. The mix combination and asphalt were the same in each case. Asphalt content was determined by scale weight.

Apparently, the difference in slope between the 3000 and 6000 gram curves is due to gauge influence area, since samples for both curves were molded at about the same density. It can be seen that the sensitivity (slope factor) of the gauge with reference to precision is considerably reduced as the sample size is reduced. For a gauge standard deviation of ± 95 counts, the following table shows the relative effect of each sample size for the limestone mix used.

Table 2

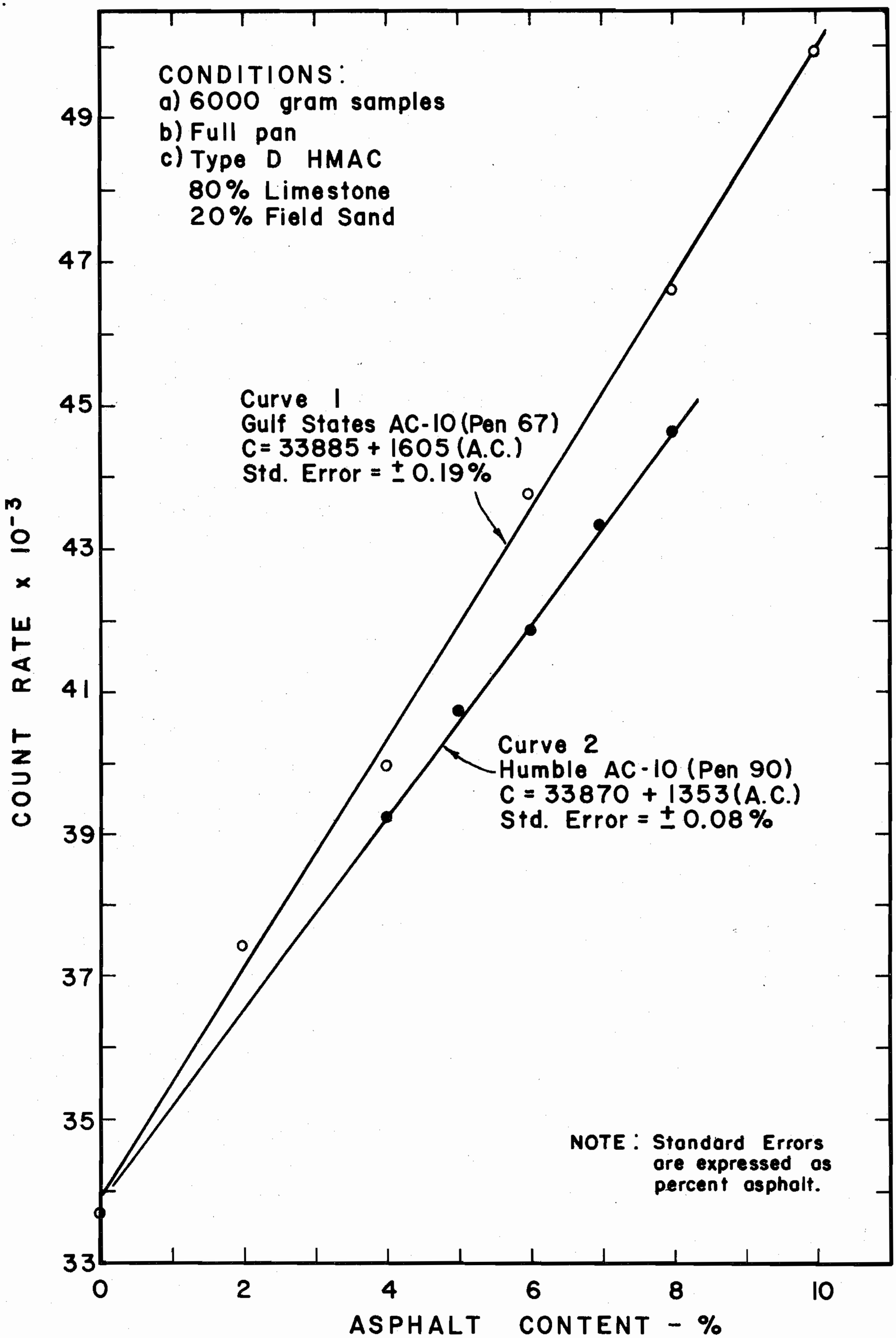
Relative Effect of Sample Size

| Sample Size (grams) | Slope (Counts per % Asphalt) | Relative Sensitivity (% Asphalt) |
|---|---|---|
| 6,000 | 1353 | ± .07 |
| 3,000 | 904 | ± .11 |
| 1,000 | 274 | ± .35 |

## Asphalt Effect

Figure 12 shows the effect caused by two different asphalts when the same type and combination of materials was used. This shift in calibration is well documented in the literature (12).

Figure 12. Sample Preparation and Asphalt Effects



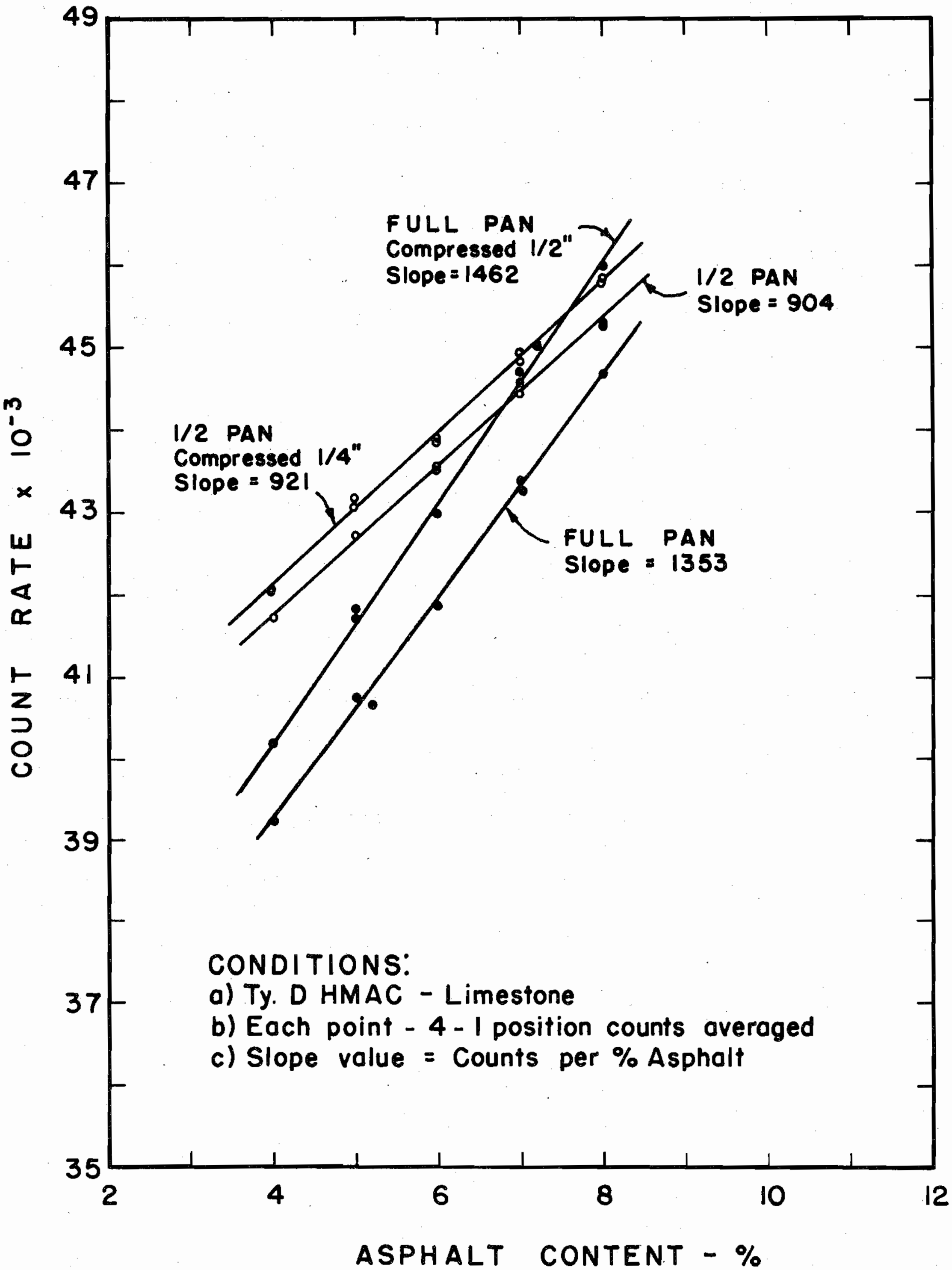
Density Effect

The effect of an error in volume (or an increase in sample density) is shown in Figure 13. The 3000 gram samples (1/2 pan) were compressed 1/4 inch and the 6000 gram samples 1/2 inch. An increase in density at a known asphalt content causes the count rate to increase, thus indicating an erroneously high asphalt content. The regression equations also show an increase in slope with higher density.

From this study, it is concluded that calibration should be done at the highest practical density in order to gain the most sensitive calibration slope and thus minimize inherent statistical variations in count rate. The largest weight which will completely fill the sample pan should be used, and this weight and volume must be held constant during calibration and in field control.

This conclusion was not carried forward in the remaining sections of this report, because it was desired to compare other mixes on an equal basis with those previously tested.

Figure 13. Density Effect



## Gradation Effect

The effect of aggregate gradation on calibration was investigated by molding four fine graded (56% sieve size 3/8 - 10) and four coarse graded (74% sieve size 3/8 - 10) samples of the same limestone mix previously used. All samples weighed 3000 grams and contained 6% asphalt by weight of mixture. The indicated asphalt contents in Table 3 were obtained by using the appropriate curve in Figure 11.

Table 3

Gradation Study

Asphalt Content

| Coarse Gradation | Fine Gradation |
|---|---|
| 5.94 | 6.05 |
| 6.00 | 5.98 |
| 6.00 | 6.01 |
| 5.96 | 5.89 |

An analysis of variance indicated that the "within treatment" variation was no different from the "between treatment" variation. At least for this material and sample size, aggregate gradation does not appear to be a significant variable.

The use of all coarse graded samples to develop a calibration curve has an effect on calibration precision as will be shown in a later study. Due to segregation problems, coarse graded samples tend to be less homogeneous than well graded samples.

Segregation in well graded samples can also cause problems when attempting to use a "no asphalt" sample as a 0% point on the calibration curve. Before asphalt was added, counts were obtained on all carefully prepared 3000 gram samples of the various mixes used in this study. All testing was done at the same volume and temperature (250° F) after the materials had been allowed to dry in a 200° F oven overnight. Table 4 shows the results of this investigation.

Table 4

Effect of Segregation on Calibration Precision

| Material | Maximum Count Difference (0% Asphalt) | Calibration Curve Slope Counts/ % Asph | Equivalent Spread in terms of Asphalt Content |
|---|---|---|---|
| Limestone | 350 | 904 | 0.4% |
| Siliceous Gravel | 300 | 911 | 0.3% |
| Lightweight | 530 | 592 | 0.9% |

In relative terms, the lightweight material was coarser than the other materials. Efforts were made to prevent segretation as the material went into the pan; however, visual observation indicated only limited success.

It is concluded that segregation rather than gradation has a significant effect on calibration, and 0% asphalt appears to be a poor calibration point. Use of this and one other point for approximating calibration should be limited to cases where it is desired to monitor asphalt contents in a relatively narrow range around the control asphalt content. More accurate calibration slopes may be obtained when the 0% point is used in conjunction with several other points at different asphalt contents. Priority should be given to using asphalt points only to establish an accurate calibration slope near the design asphalt content.

<u>Moisture Effect</u>

Considerable effort was made to determine the effect of moisture on calibration using a mix combination consisting of 80% absorptive limestone and 20% sand.

<u>Factorial Experiment</u>. The initial phase of this moisture investigation was to design a factorial experiment

in which asphalt and wet aggregate were to be mixed and tested at 250° F. A $2^3$ factorial experiment was used which included asphalt content, moisture content, and volume as the main effects. Held constant were the gradation (Type D), the asphalt, and the testing temperature (as close to 250° F as possible). Volume was involved because of its significant effect on calibration precision as previously demonstrated. Count rate was the dependent (Y) variable and levels of the following independent variables were used.

| Variable | Levels of Variable | |
|---|---|---|
| $x_1$ - Asphalt Content | (+)*<br>(-) | 7%<br>5% |
| $x_2$ - Moisture Content | (+)<br>(-) | Moisture<br>No Moisture |
| $x_3$ - Volume (Change in Density) | (+)<br>(-) | 1/2 Pan<br>1/2 Pan Compressed 1/4 inch |

*(+) = high level; (-) = low level

The following regression model was assumed:

$$y = b_o + b_1x_1' + b_2x_2' + b_{12}x_1'x_2' + b_3x_3' + b_{13}x_1'x_3' + b_{23}x_2'x_3' \quad (1)$$

Where: $y$ = Count Rate

$x_1'$ = Asphalt Content = $(x_1 - \bar{x}_1)$

$x_2'$ = Moisture Content = $(x_2 - \bar{x}_2)$

$x_3'$ = Volume = $(x_3 - \bar{x}_3)$

$\bar{x}_i$ = Mean of $x_i$

Asphalt content was computed as a percent of the combined weight of dry aggregate plus asphalt. Moisture content was expressed as a percent of dry aggregate weight. This calculation procedure was used, because it follows present practice in the Texas Highway Department.

Duplicate 3000 gram samples were prepared for each treatment combination in order to investigate repeatability.

The procedure was to first soak the graded aggregate, then heat it to the highest obtainable temperature. Usually, when the mix temperature reached about 225° F, the moisture loss was extremely rapid and almost uncontrollable. After a few trial mixes, it became apparent that moisture could not be controlled at a specified level.

Knowing the weights of dry aggregate and asphalt, moisture contents were computed on the basis that any weight loss was due to moisture evaporation, and the known weight differential was moisture left in the aggregate. All weighing was done hot in order to compensate for convection currents. Asphalt was added to the mixture at about 225° F, and mixing was done by hand until cooling prevented further mixing. The sample was then heated to 250° F after which it was again mixed until the aggregates were properly coated. Since mixing lowered the temperature, the sample was again placed in the oven, brought to 250° F, placed in the gauge sample pan, compressed to a known volume, and then tested. Four 1-position counts were averaged. After each operation, a reference weight was obtained to check moisture content.

The data was coded for the mean ($\bar{x}$) value of each variable in order to show the true underlying effects. A tabulation of the raw data is shown in Table 5. The volume variable was precoded for simplicity, and the other variables were coded during the computation process.

Table 5

Data - 3000 Gram Limestone Samples

Moisture $2^3$ Factorial Experiment

| Specimen No. | Y<br>Avg Count Rate | $x_1$<br>Asphalt Content, % | $x_2$<br>Asphalt Content, % | $x_3$<br>Volume |
|---|---|---|---|---|
| 1 | 43089 | 5.00 | 0.0 | -1 |
| 2 | 43201 | 5.00 | 0.0 | -1 |
| 3 | 44828 | 7.00 | 0.0 | -1 |
| 4 | 44961 | 7.00 | 0.0 | -1 |
| 5 | 43789 | 5.00 | 0.55 | -1 |
| 6 | 43457 | 5.00 | 0.27 | -1 |
| 7 | 45544 | 7.34 | 0.16 | -1 |
| 8 | 45135 | 7.03 | 0.13 | -1 |
| 9 | 42709 | 5.00 | 0.0 | +1 |
| 10 | 42703 | 5.00 | 0.0 | +1 |
| 11 | 44582 | 7.00 | 0.0 | +1 |
| 12 | 44445 | 7.00 | 0.0 | +1 |
| 13 | 43153 | 5.00 | 0.48 | +1 |
| 14 | 43638 | 5.39 | 0.32 | +1 |
| 15 | 45026 | 7.00 | 0.61 | +1 |
| 16 | 45254 | 7.00 | 0.94 | +1 |
| $\bar{x}$ | | 6.048 | 0.2163 | 0 |

Using a stepwise regression program (STEP 01), an Analysis of Variance table was derived using the coded data, and Table 6 ranks the importance of the main effects and interaction terms by F value.

Table 6

AOV Table - Moisture $2^3$ Factorial
Experiment-Data Corrected
for Means

| SV | df | SS | MS | F |
|---|---|---|---|---|
| Due to | | | | |
| Reg. | 6 | 14,193,464 | 2,365,577 | |
| A.C. | 1 | 12,773,783 | 12,773,783 | 1,031 |
| Vol. | 1 | 771,410 | 771,410 | 62 |
| M.C. | 1 | 609,800 | 609,800 | 49 |
| AC x MC | 1 | 27,269 | 27,269 | 2 |
| MC x Vol. | 1 | 6,231 | 6,231 | 0.5 |
| AC x Vol. | 1 | 4,971 | 4,971 | 0.4 |
| From | | | | |
| Reg | 9 | 111,516 | 12,391 | |

It can be seen that asphalt content, volume, and moisture content, in that order, are the most important variables. The significance of the interaction terms is very small considering how much they add to regression. For all practical purposes, only the first three terms need to be included in the regression equation. The following equation was derived and solved for asphalt content assuming a constant volume of 1/2 pan.

Counts = 44095 + 875(AC-6.048) + 923(MC-0.2163)-228(Vol.)
R = 0.9947
Std. Error = $\pm$ 0.13% Asphalt

Rearranging and Substituting:
Counts = 44095 - 5292 - 200 - 228 + 875(AC) + 923(MC)
Counts = 38375 + 875(AC) + 923(MC)

$$AC = \frac{\text{Counts} - 38375}{875} - \frac{923(MC)}{875}$$

$$AC = \frac{\text{Counts} - 38375}{875} - 1.055(MC) \qquad (2)$$

Conditions for use of equation:

a) Type D Mix - 80% TCS Limestone
20% CA Field Sand
b) 3000 gram samples, compacted to 2 inch height in sample pan
c) Asphalt Range - 4 to 8%
d) Asphalt - Humble AC 10
e) Moisture Range - 0 to 0.9%
f) Four - 1-position counts averaged

The coefficient for moisture indicates that the gauge is slightly more sensitive to moisture than it is to asphalt (at least in this case). A count rate change which yields a difference of 0.10% asphalt will yield an equivalent difference of 0.11% moisture. Therefore, a little more moisture than is actually present must be subtracted in the above equation.

Figure 14 shows how each of the variables affect count rate. It should be noted that the moisture slope is more than the asphalt slope, thus accounting for the above factor.

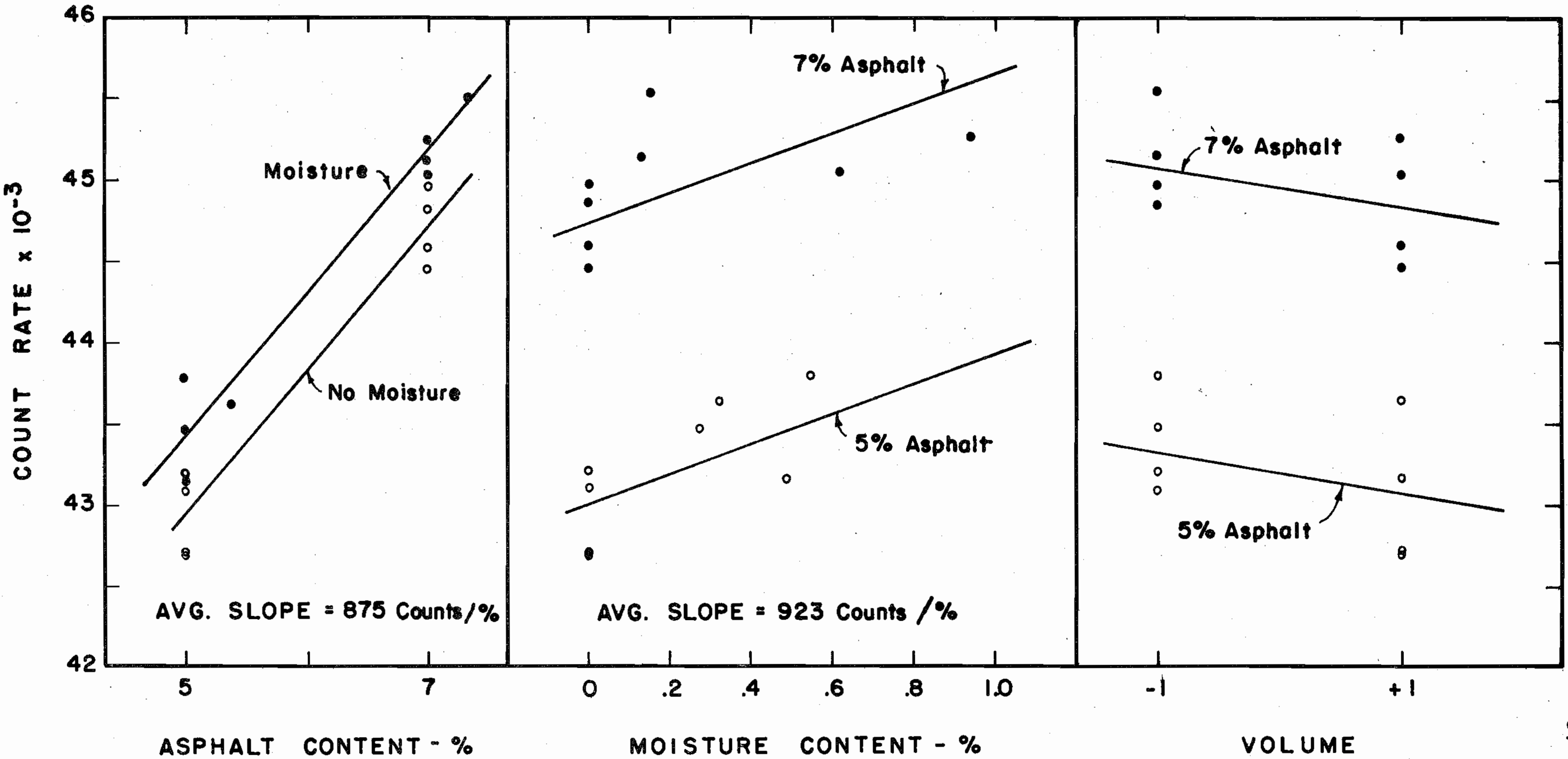
This regression equation (2) is usable in the field; however, its performance (note the standard error) will not be as good as that for full pan samples, because of the lower slope sensitivity. Also, the physical difficulty of trying to combine asphalt, water, and aggregate at high temperatures probably contributed to some error in the calculated moisture contents.

Modified Experiments. Using the information gained from the factorial experiment, a second study was developed for the purpose of establishing the moisture coefficient for 6000 gram samples.

In the previous experiment, scale weights indicated that all the moisture had evaporated from the duplicate 3000 gram samples before they could be mixed together and reheated for testing as 6000 gram full pan samples. Also, at the time of this study, facilities were not available to mix 6000 grams of material in one batch. This necessitated that asphalt

CONDITIONS:
a) 3000 gram samples
b) Type D HMAC - Limestone
c) Temp. ~250°F

Figure 14. Count Rate vs. Asphalt Content, Moisture Content, and Volume



and moisture contents be analyzed separately regarding their effect on calibration.

In this case, the approach was first to compare results obtained from the factorial experiment with that obtained from separate tests on additional 3000 gram asphalt samples and moisture samples. Then, a comparative analysis was made between the 3000 gram and 6000 gram experiments to see if the moisture coefficients were the same.

The primary difference in the two studies utilizing 3000 gram samples was that moisture specimens were tested under a heated condition in one case (factorial), whereas they were not in the other. Thus, the temperature effect (previously shown to be a variable) was not accounted for in one case. Since calibration curves for 1000 gram asphalt samples were found to be parallel at 70° and 250° F, it is assumed that the 3000 gram moisture samples would likewise yield parallel curves (same slope, hot or cold), only the intercept would change. Using this assumption, the ratio of the slope of the asphalt curve to the slope of the moisture curve should remain unchanged. The question then, was whether gauge response to different sample volumes changes the value of the asphalt - moisture slope ratio.

Separate 3000 gram Experiments. In each of these studies, volume was held constant at 1/2 pan of material, asphalt contents were calculated as percent of total weight, and moisture contents calculated as percent of oven dry weight.

Table 7

Data - 3000 Gram Limestone Samples

Asphalt and Moisture Separated

| Specimen No. | 1/2 Pan Count Rate | Asphalt Content, % | Moisture Content, % |
|---|---|---|---|
| 1 | 42709 | 5.0 | 0.0 |
| 2 | 42703 | 5.0 | 0.0 |
| 3 | 44582 | 7.0 | 0.0 |
| 4 | 44445 | 7.0 | 0.0 |
| 5 | 41702 | 4.0 | 0.0 |
| 6 | 41714 | 4.0 | 0.0 |
| 7 | 43523 | 6.0 | 0.0 |
| 8 | 43550 | 6.0 | 0.0 |
| 9 | 45334 | 8.0 | 0.0 |
| 10 | 45312 | 8.0 | 0.0 |
| 11 | 38461 | 0.0 | 0.0 |
| 12 | 38592 | 0.0 | 0.0 |
| 13 | 42672 | 0.0 | 4.95 |
| 14 | 42598 | 0.0 | 4.72 |
| 15 | 42468 | 0.0 | 4.50 |
| 16 | 42393 | 0.0 | 4.58 |
| 17 | 42296 | 0.0 | 4.51 |
| 18 | 42068 | 0.0 | 4.09 |
| 19 | 42220 | 0.0 | 4.27 |
| 20 | 42329 | 0.0 | 4.27 |
| $\bar{x}$ | | 3.0 | 1.795 |

Tables 5 and 7 show the raw data for each experiment. The data was coded for the means and the stepwise program was used to derive the following equation:

$$\text{Counts} = 42584 + 853(AC - 3.0) + 875(MC-1.795)$$
$$R = 0.9987$$
$$\text{Std. Error} = \pm 0.11\% \text{ Asphalt}$$

Rearranging and Substituting:

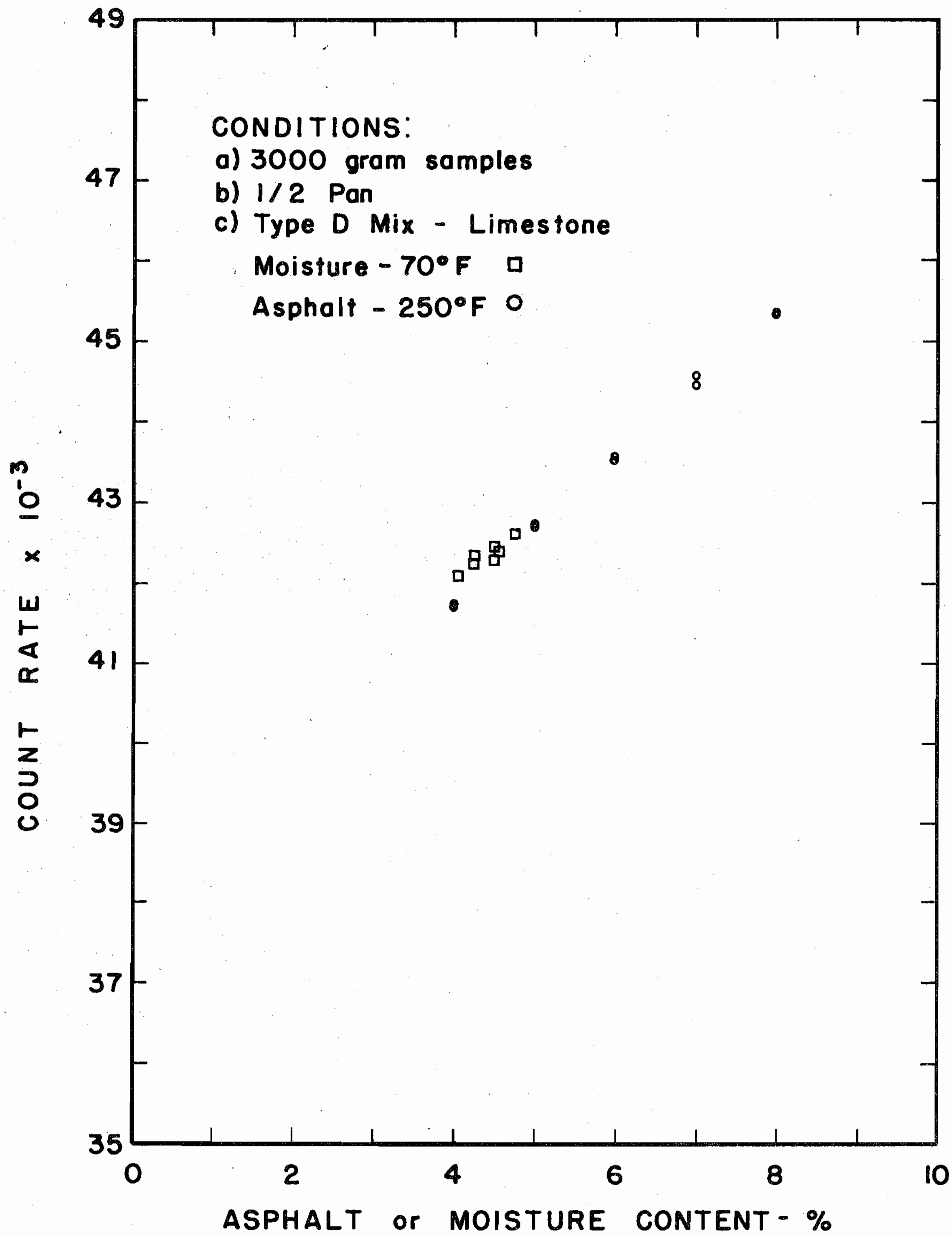
$$\text{Counts} = 42584 - 2559 - 1571 + 853(AC) + 875(MC)$$
$$\text{Counts} = 38454 + 853(AC) + 875(MC)$$
$$AC = \frac{\text{Counts} - 38454}{853} - \frac{875}{853}(MC)$$

$$\boxed{AC = \frac{\text{Counts} - 38454}{853} - 1.025(MC)} \qquad (3)$$

The moisture coefficient in equation (2) was 1.055 as compared to 1.025 in equation (3). A graphical representation of the data used to derive equation (3) is shown in Figure 15.

6000 Gram Experiment. The duplicate 3000 gram samples containing asphalt and no moisture were combined into 6000 gram full pan samples in order to study the asphalt effect alone. Using the same mix combination, additional 6000 gram moisture samples were mixed to

Figure 15. Calibration for Moisture and Asphalt Separated, 3000 Gram Samples



show the moisture effect. Moisture contents were calculated as percent of oven dry weight.

The data was combined, as shown in Table 8, coded for the means, and analyzed by the stepwise program to obtain the calibration equation.

Table 8

Data - 6000 Gram Limestone Samples

Asphalt and Moisture Separated

| Specimen No. | Full Pan Count Rate | Asphalt Content,% | Moisture Content,% |
|---|---|---|---|
| 1 | 39219 | 4.0 | 0.0 |
| 2 | 40777 | 5.0 | 0.0 |
| 3 | 41880 | 6.0 | 0.0 |
| 4 | 43381 | 7.0 | 0.0 |
| 5 | 44682 | 8.0 | 0.0 |
| 6 | 41302 | 0.0 | 4.95 |
| 7 | 40940 | 0.0 | 4.73 |
| 8 | 37535 | 0.0 | 2.52 |
| 9 | 39776 | 0.0 | 4.12 |
| 10 | 38585 | 0.0 | 3.16 |
| 11 | 37562 | 0.0 | 2.59 |
| 12 | 36639 | 0.0 | 1.94 |
| $\bar{x}$ | | 2.50 | 2.001 |

The following calibration equation was derived:

Counts = 40190 + 1378 (AC - 2.50) + 1516(MC-2.001)
R = 0.9992
Std. Error = $\pm$ 0.08% Asphalt

Rearranging and Substituting:

$$\text{Counts} = 40190 - 3450 - 3035 + 1378(AC) + 1516(MC)$$

$$\text{Counts} = 33705 + 1378(AC) + 1516(MC)$$

$$AC = \frac{\text{Counts} - 33705}{1378} - \frac{1516}{1378}(MC)$$

$$\boxed{AC = \frac{\text{Counts} - 33705}{1378} - 1.100(MC)} \qquad (4)$$

Conditions for use of equation:

a) Type D Mix - 80% TCS Limestone
20% CA Sand
b) 6000 gram samples, compacted to top of sample pan
c) Asphalt Range - 0 to 8% (% Aggr + Asph)
d) Asphalt - Humble AC 10
e) Moisture Range - 0 to 5% *(% Dry Wt. Aggr.)
f) Four - 1-position counts averaged

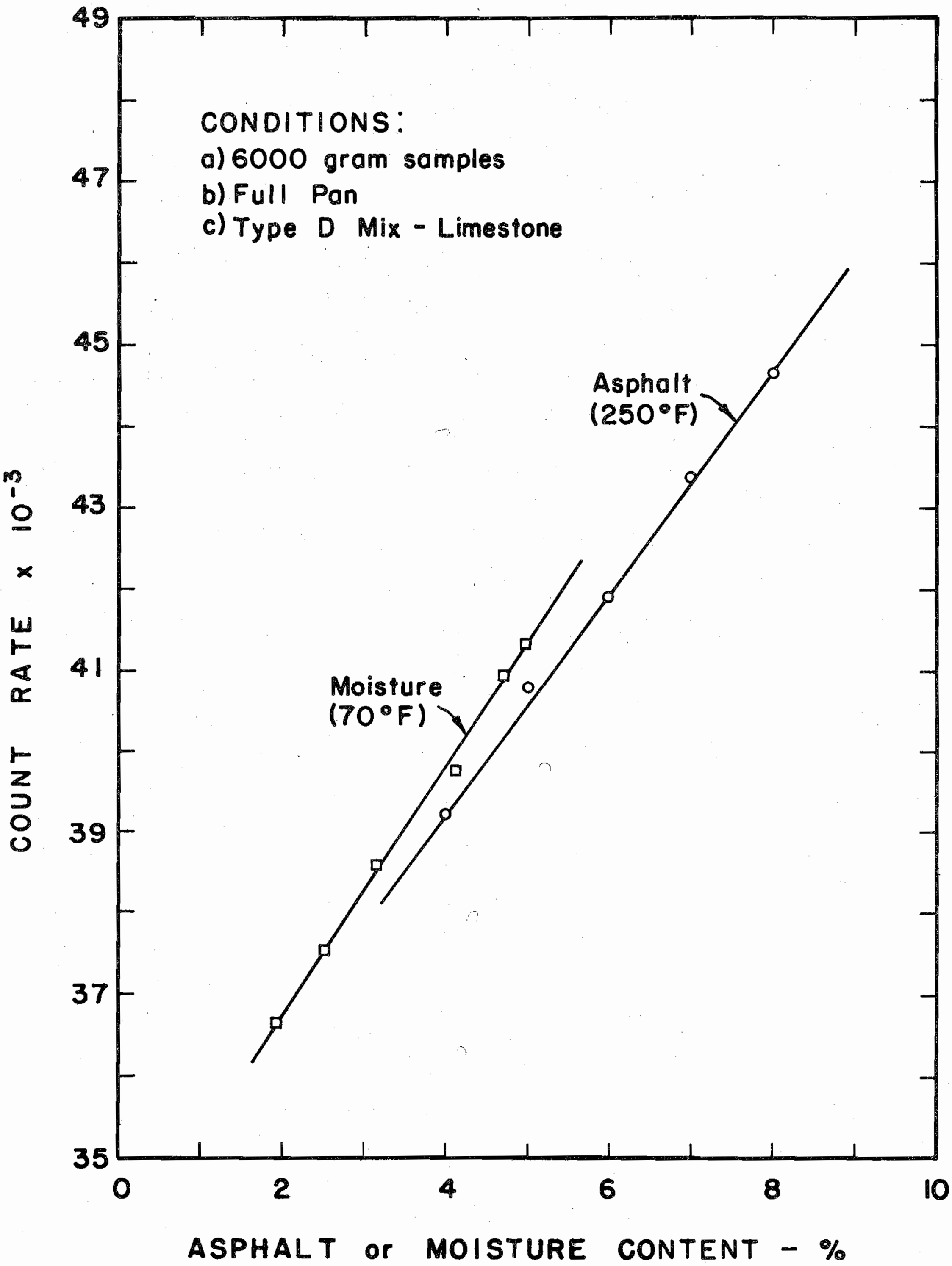
*See later discussion p. 55

The moisture coefficient of 1.100 in this equation was higher than for either of the 3000 gram experiments (1.055 and 1.025) indicating that gauge response may be slightly different for the two sample volumes.

The data in Table 8 is shown in Figure 16.

A sensitivity analysis was performed to determine what error would be introduced if the moisture

Figure 16. Calibration for Moisture and Asphalt Separated, 6000 Gram Samples



coefficient varied from 1.025 to 1.100. Table 9 shows the results of this analysis.

Table 9

Sensitivity Analysis

Error in Moisture Coefficient

| | Percent Moisture (MC) | | |
|---|---|---|---|
| | 0.1% | 0.5% | 1.0% |
| Coeff = 1.100 | 0.11 | 0.55 | 1.10 |
| Coeff = 1.025 | 0.10 | 0.51 | 1.03 |
| Error in AC | 0.01% | 0.04% | 0.07% |

This analysis shows that if the hot asphalt mix has a moisture content of 1.0%, there is the possibility of introducing a 0.07% error in determination of asphalt content if the wrong moisture coefficient is used in the predictive equation. Since normal hot bin samples for this mix were found to contain from 0.1% to 0.3% moisture, the possible error in asphalt content is reduced to 0.02%.

Therefore, it is concluded that the moisture coefficient of 1.10 in equation (4) is a practical value when moisture contents of less than 1%, expressed as percent of weight of dry aggregate, are present in the hot mix. This coefficient is valid only for the materials and conditions under which it was derived.

Sample Weight Effect

A $2^3$ factorial experiment was designed using 6000 gram full pan samples of the limestone mix to investigate the effect of sample weight. Duplicate samples which had been mixed for the previous 3000 gram factorial experiment were combined and used in this investigation. Some of these samples contained moisture initially, but upon reheating and mixing, all the moisture evaporated leaving a constant weight speciman less than 6,000 grams. Weight loss due to mixing and transfer was accounted for in all samples. The following variables were also included.

| Variable | Levels of Variable |
|---|---|
| $x_1$ - Asphalt Content | (+) 7%<br>(-) 5% |
| $x_2$ - Sample Weight (Varies) | (+) $>$ 5,990 grams<br>(-) $<$ 5,990 grams |
| $x_3$ - Volume | (+) Full Pan<br>(-) Full Pan Compressed 1/2 inch |

The data for this experiment is shown in Table 10.

Table 10

Data - 6,000 Gram Limestone Samples

Weight $2^3$ Factorial Experiment

| | $y$ | $x_1$ | $x_2$ | $x_3$ |
|---|---|---|---|---|
| Specimen No. | Avg. Count Rate | Asphalt Content | Sample Wt. Grams | Volume |
| 1 | 41,698 | 5.00 | 5994 | -1 |
| 2 | 44,719 | 7.00 | 5992 | -1 |
| 3 | 41,835 | 5.03 | 5966 | -1 |
| 4 | 45,058 | 7.20 | 5970 | -1 |
| 5 | 40,777 | 5.00 | 5997 | +1 |
| 6 | 43,381 | 7.00 | 5990 | +1 |
| 7 | 40,672 | 5.23 | 5967 | +1 |
| 8 | 43,262 | 7.05 | 5961 | +1 |
| $\bar{x}$ | | 6.064 | 5980 | 0 |

Each independent variable was coded for its mean, and the stepwise regression program was used to develop the AOV table in Table 11. The same conceptual regression model used in the previous $2^3$ factorial, equation (1), was assumed for this experiment.

Table 11

AOV Table - Weight $2^3$ Factorial Experiment

Data Corrected for Means

| SV | df | SS | MS | F |
|---|---|---|---|---|
| Due to Reg. | 6 | 19,966,254 | 3,327,709 | |
| A.C. | 1 | 16,321,194 | 16,321,194 | 2,061 |
| Vol. | 1 | 3,497,120 | 3,497,120 | 442 |
| Wt.xVol. | 1 | 93,122 | 93,122 | 12 |
| Wt. | 1 | 28,258 | 28,258 | 4 |
| A.C.xVol. | 1 | 21,320 | 21,320 | 3 |
| A.C.xWt. | 1 | 5,240 | 5,240 | 1 |
| From Reg | 1 | 7,918 | | |

As shown by the F values, asphalt content and volume were the two most important variables, with an interaction term for weight times volume being third, and the term for weight fourth. This indicates that sample weight may be allowed to vary as much as minus 40 grams, in this case, with no deteremental effect to calibration. Based on this finding, it is concluded that a ± 25 gram error from 6000 grams would be an allowable variation for ordinary control.

The actual regression model is of little use at this point, since weight and volume are held constant in practical usage. However, with the first three terms in regression, the model had a correlation coefficient of 0.9984 and a standard error of $\pm$ 0.09%. The regression model containing only asphalt content will be discussed under calibration studies.

## Material Effect - Calibration Studies

The effect of material on calibration is demonstrated by the following examples. Carefully graded samples, uniform temperature, controlled volume, and aggregates dried to constant weight at $200^{\circ}$ F were used in each case. Table 12 indicates each mix combination.

Table 12

Material Combinations

| Mix No. | Type | % Coarse Aggregate | % Limestone Screenings | % Field Sand | Asphalt |
|---|---|---|---|---|---|
| 1 | Limestone | 66 | 14 | 20 | GS AC-10 |
| 2 | Limestone | 66 | 14 | 20 | H AC-10 |
| 3 | Siliceous Gravel | 63 | 20 | 17 | H AC-10 |
| 4 | Lightweight | 65 | 25 | 10 | H AC-10 |

GS AC-10 - Gulf States AC-10 (Pen 67)
H AC-10 - Humble AC-10 (Pen 90)

Table 13 shows the aggregate gradations for each mix.

Table 13

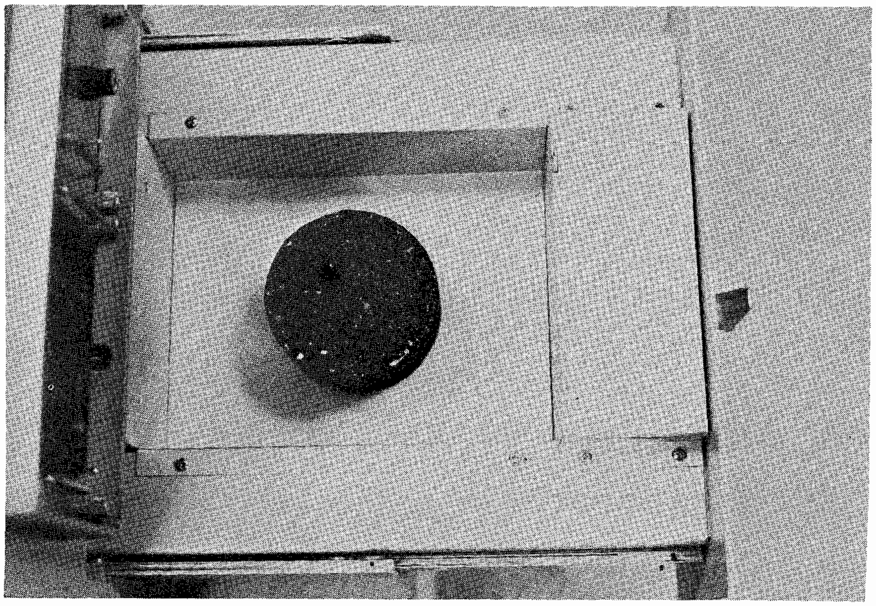
Mix Gradations

| Sieve Size | Mix No. & Type | | | | Type D Specs. |
|---|---|---|---|---|---|
| | 1-Ls. | 2-Ls. | 3-Sil.Gr. | 4-Lw. | |
| 1/2-3/8 | 94.4 | 95.2 | 94.0 | 89.0 | 95-100 |
| 3/8-4 | 36.1 | 35.4 | 32.1 | 44.9 | 20- 50 |
| 4-10 | 23.0 | 23.5 | 27.8 | 13.5 | 10- 30 |
| +10 | 64.7 | 63.7 | 65.9 | 69.4 | 50- 70 |
| 10-40 | 9.9 | 10.5 | 10.8 | 11.7 | 0- 30 |
| 40-80 | 11.0 | 8.2 | 7.5 | 7.3 | 4- 25 |
| 80-200 | 12.2 | 14.6 | 13.4 | 9.5 | 3- 25 |
| Pass 200 | 2.2 | 3.0 | 2.4 | 2.1 | 0- 6 |
| Avg. Dry Bulk Sp. Gr. | 2.576 | 2.576 | 2.631 | 1.751 | - |

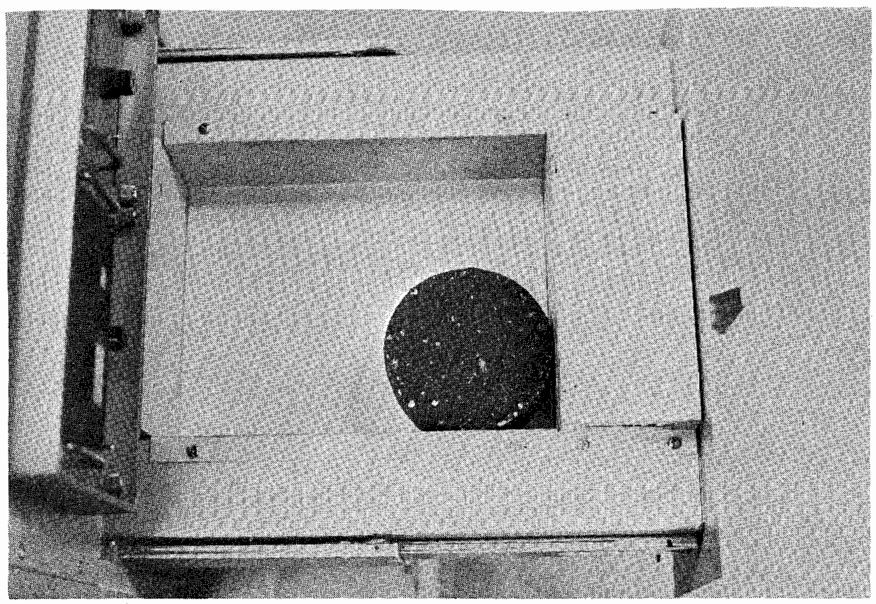
Standard Gyratory Press Samples. In the course of normal design and control operations for hot mix, the Texas Highway Department prepares cylindrical specimens which are two inches high by four inches in diameter and weigh between 900 and 1000 grams. These specimens are molded with a gyratory shear press using Test Method Tex - 206 - F (15). Using these specimens would fit very well into standard laboratory operations.

Initially, an analysis was made of gauge response to various positions of a sample in the drawer. Different placements, both horizontal and vertical, were tried. It was found that counts were highest in the bottom, front left corner, but a more sensitive calibration slope was obtained at the bottom, center of the drawer. However, efforts to place the sample at the exact center yielded more count variation than at other positions. Exact centering over the same spot was difficult to achieve without some kind of permanently fixed template. A small error in lateral placement caused an undesirable fluctuation in count rate. In an attempt to minimize this fluctuation, a circle was drawn on the bottom (center) of the drawer, and the samples were visually placed in this circle for testing. Figure 17 shows the different placement positions.

Three specimens at each of five asphalt contents were molded and three 1-position counts obtained for



a) Center Placement



b) Corner Placement

Figure 17. Gyratory Shear Press Sample Placement

each specimen and position. Texas Highway Department procedures for determining optimum asphalt content require that three samples be molded at each of five different asphalt contents, and taking three counts would minimize the counting error inherent in the gauge.

Figure 18 compares the effect on calibration of placing the specimen in the corner and center (bottom) of the drawer. In this case, the samples were tested at about 240° F.

The samples were very tender after the hot molding process; therefore, it was decided to allow them to cool before testing with the hope of obtaining more reliable results. Figures 19 and 20 show the relationship between hot (240° F) and cold (70° F) calibration for each of the two placement positions. As previously noted, the calibration slopes are higher for the center than for the corner position, and they are almost parallel for the two temperatures within each case. However, standard errors and correlation coefficients are better for the corner position than for the center position. These indices also show little difference between hot and cold calibration regarding precision.

Figure 18. Comparison of the Center and Corner Calibration Positions

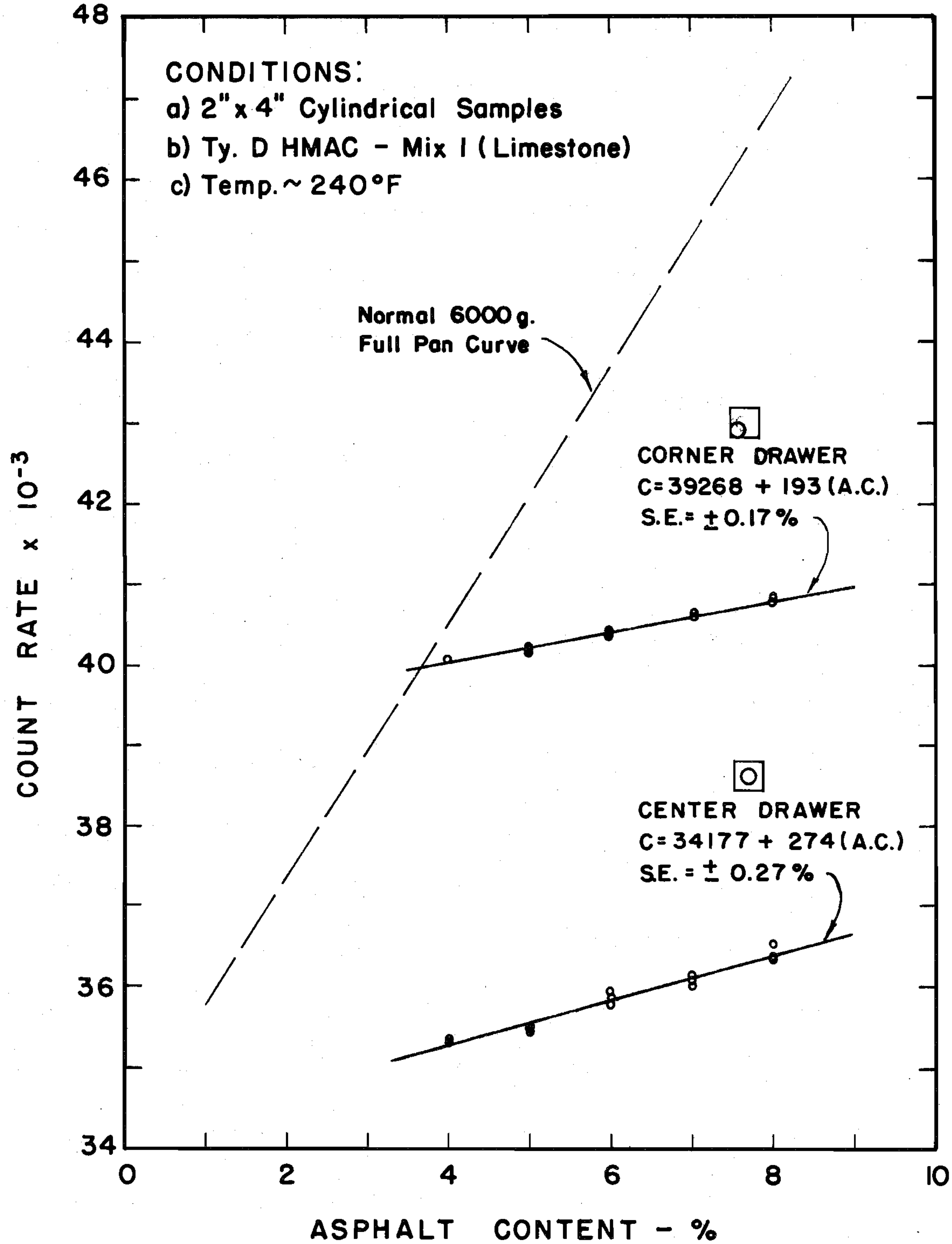


Figure 19. Calibration Using 2"x4" Round Specimens

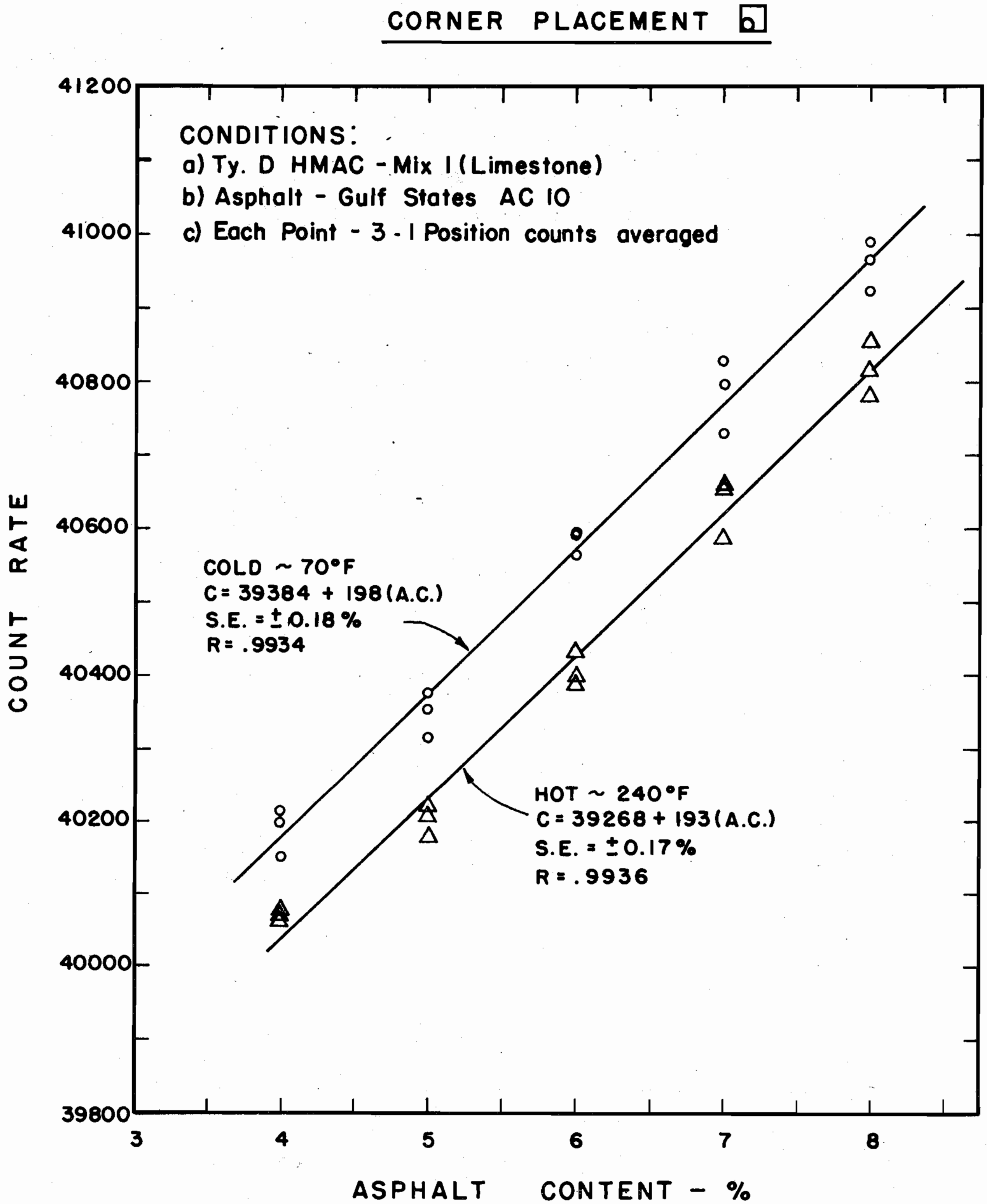
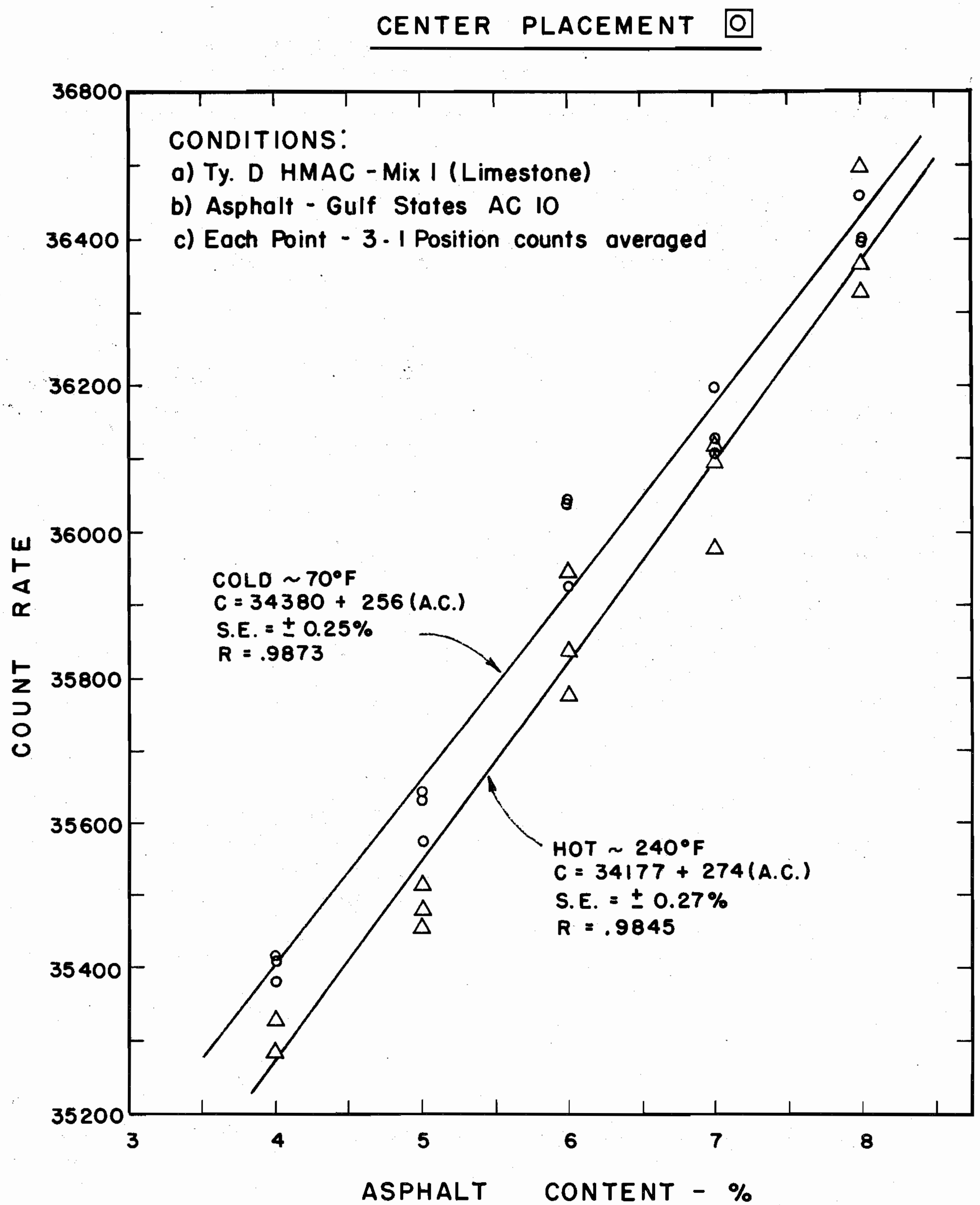


Figure 20. Calibration Using 2"x4" Round Specimens



Based on this data, it is concluded that more reliable tests results will be obtained if the corner position is used even though the center offers better sensitivity.

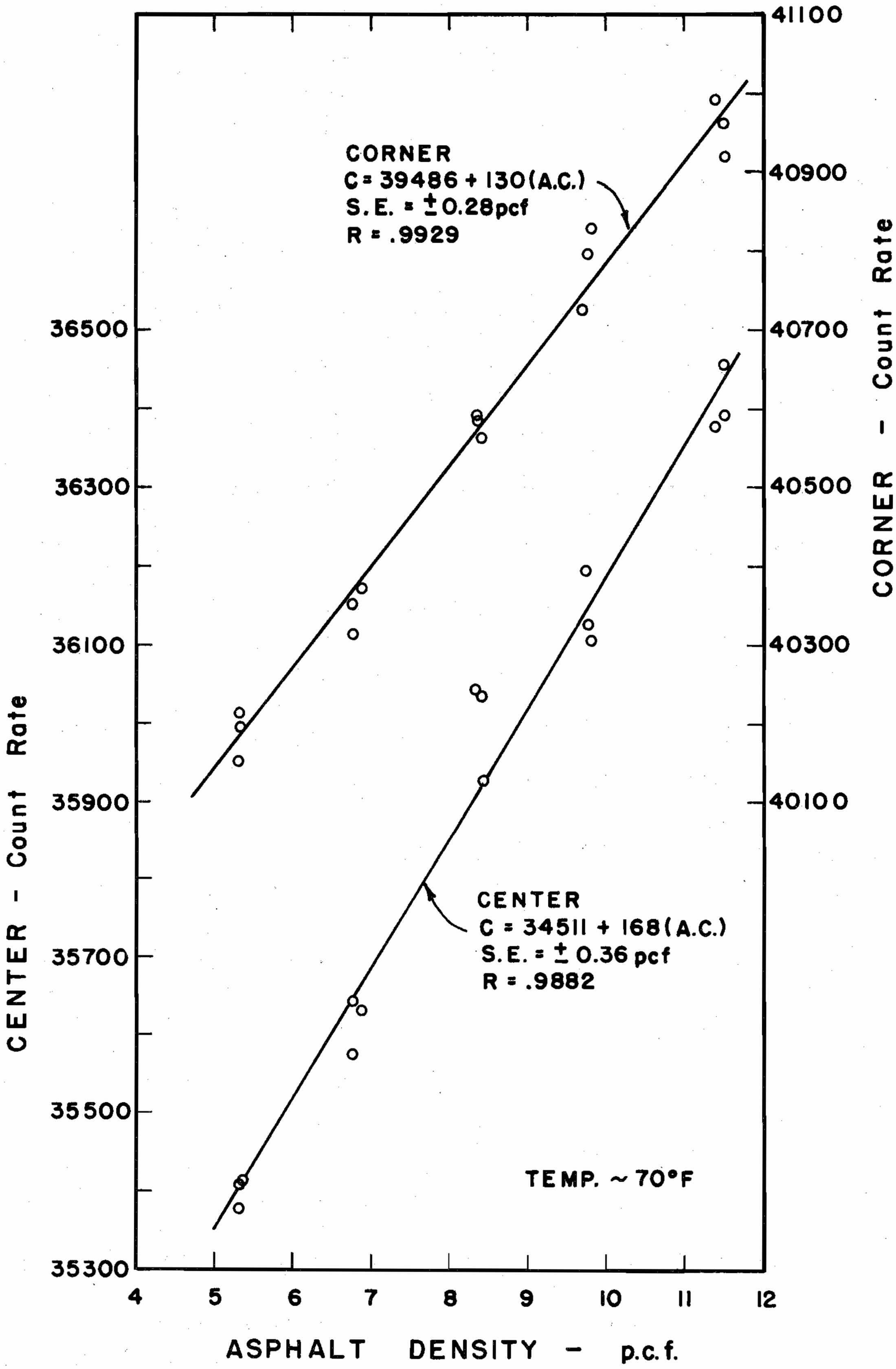
An investigation was made to determine whether calibration for asphalt density rather than for percent asphalt would improve precision.

The volume of each specimen was determined using Test Method Tex - 207 - F. Weight of the asphalt which had been added was then divided by the volume of the specimen to obtain asphalt density. A calibration plot for the center and corner positions at 70$^{o}$ F appears in Figure 21. Conversion of the standard errors from pounds per cubic foot to percent do not indicate any improved precision for this material and calibration method.

It was found that preparation, weighing, and molding must be done with extreme care since nonuniform samples or small weighing errors are critical for the relatively small changes in count rate that are obtained from these small samples.

Extreme care must also be taken to use the same molding procedure (manual vs. motorized) in the field control as was used to develop calibration.

Figure 21. Calibration Using Asphalt Density



Results of this investigation indicate that reliability and precision of measurement will be reduced if this type of sample is used to control asphalt content.

Calibration Curves- 1/2 Pan Samples. Two approaches were made to the problem of calibrating for specimens containing asphalt, but no moisture. The first was to use the conventional method of plotting count rate (y) versus asphalt content (x) expressed as a percent of total specimen weight. The second was to convert asphalt content to percent of absolute volume.

For this study, absolute volume is defined as the volume of asphalt in the specimen expressed as a percent of the total bulk volume of the mix. Dry bulk specific gravities of the various aggregate mixes were determined using the Bryant Method (17). These gravities, the asphalt specific gravity, and the unit weight of water at 77$^{\circ}$ F (the same temperature at which the asphalt specific gravity was determined) were then used to calculate bulk volume of the aggregate and asphalt. The asphalt bulk volume was then divided by the total bulk volume (asphalt plus aggregate) to obtain what is termed asphalt absolute volume. This method was developed by the Texas Transportation Institute (18) and later reported by Zeigler (19).

For the 1/2 pan samples, Table 14 shows the data used in each approach. Four 1-position counts were averaged for each calibration point.

Table 14

Data - 1/2 Pan Calibration Samples

I. Limestone (Mix 2) - 3000 Gram Samples

| Count Rate | Asphalt Content (% Total Wt.) | Asphalt Content (% Absolute Volume) |
|---|---|---|
| 42709 | 5.0 | 11.72 |
| 42703 | 5.0 | 11.72 |
| 44582 | 7.0 | 15.99 |
| 44445 | 7.0 | 15.99 |
| 41702 | 4.0 | 9.54 |
| 41714 | 4.0 | 9.54 |
| 43523 | 6.0 | 13.89 |
| 43550 | 6.0 | 13.89 |
| 45334 | 8.0 | 18.05 |
| 45312 | 8.0 | 18.05 |

II. Siliceous Gravel (Mix 3) - 3000 Gram Samples

| | | |
|---|---|---|
| 41161 | 3.0 | 7.40 |
| 41094 | 3.0 | 7.40 |
| 42042 | 4.0 | 9.72 |
| 42125 | 4.0 | 9.72 |
| 43083 | 5.0 | 11.97 |
| 43053 | 5.0 | 11.97 |
| 43927 | 6.0 | 14.16 |
| 43894 | 6.0 | 14.16 |
| 44762 | 7.0 | 16.29 |
| 44780 | 7.0 | 16.29 |

III. Lightweight (Mix 4) - 2,000 Gram Samples

| | | |
|---|---|---|
| 41588 | 6.0 | 9.89 |
| 41530 | 6.0 | 9.89 |
| 42157 | 7.0 | 11.46 |
| 42129 | 7.0 | 11.46 |
| 42691 | 8.0 | 13.01 |
| 42752 | 8.0 | 13.01 |
| 43432 | 9.0 | 14.53 |
| 43348 | 9.0 | 14.53 |
| 43917 | 10.0 | 16.04 |
| 43873 | 10.0 | 16.04 |

Figures 22 and 23 compare calibration curves for each material and method of calibration. Table 15 shows a summary of the calibration results. For comparison purposes, equation (3), which contains moisture as a variable, was converted to percent of absolute volume and also included.

Larger changes in count rate for a given change in asphalt content resulted when the percentages were expressed in terms of total weight of specimen; however, the material effect was reduced when percent of absolute volume was used. Standard errors for the absolute volume method were also larger than for percent of total weight.

There would be an advantage to using percent of absolute volume if the points in Figure 23 were sufficiently close to allow the use of one common

Table 15

Summary of Calibration Results - 1/2 Pan Samples

| Material | Calibration Method | Sample Density & Weight | Calibration Equation | Standard Error | Correlation Coefficient |
|---|---|---|---|---|---|
| Limestone | % T.W. | 107.3 PCF<br>3,000 g | $AC = \frac{Counts - 38135}{904}$ | $\pm 0.07\%$ | 0.9991 |
| Limestone | % A.V. | 107.3 PCF<br>3,000 g | $AC = \frac{Counts - 37683}{425}$ | $\pm 0.13\%$ | 0.9993 |
| Limestone | % A.V. | 107.3 PCF<br>3,000 g | $AC=\frac{Counts-38377}{377} - 1.026(MC)$ | $\pm 0.37\%$ | 0.9971 |
| Siliceous Gravel | % T.W. | 100.0 PCF<br>3,000 g | $AC = \frac{Counts - 38435}{911}$ | $\pm 0.07\%$ | 0.9989 |
| Siliceous Gravel | % A.V. | 100.0 PCF<br>3,000 g | $AC = \frac{Counts - 38107}{410}$ | $\pm 0.10\%$ | 0.9996 |
| Light-weight | % T.W. | 71.5 PCF<br>2,000 g | $AC = \frac{Counts - 38007}{592}$ | $\pm 0.08\%$ | 0.9987 |
| Light-weight | % A.V. | 71.5 PCF<br>2,000 g | $AC = \frac{Counts - 37741}{385}$ | $\pm 0.12$ | .9988 |

Figure 22. Calibration Curves for 1/2 Pan Samples Using Asphalt Content as Percent of Total Weight

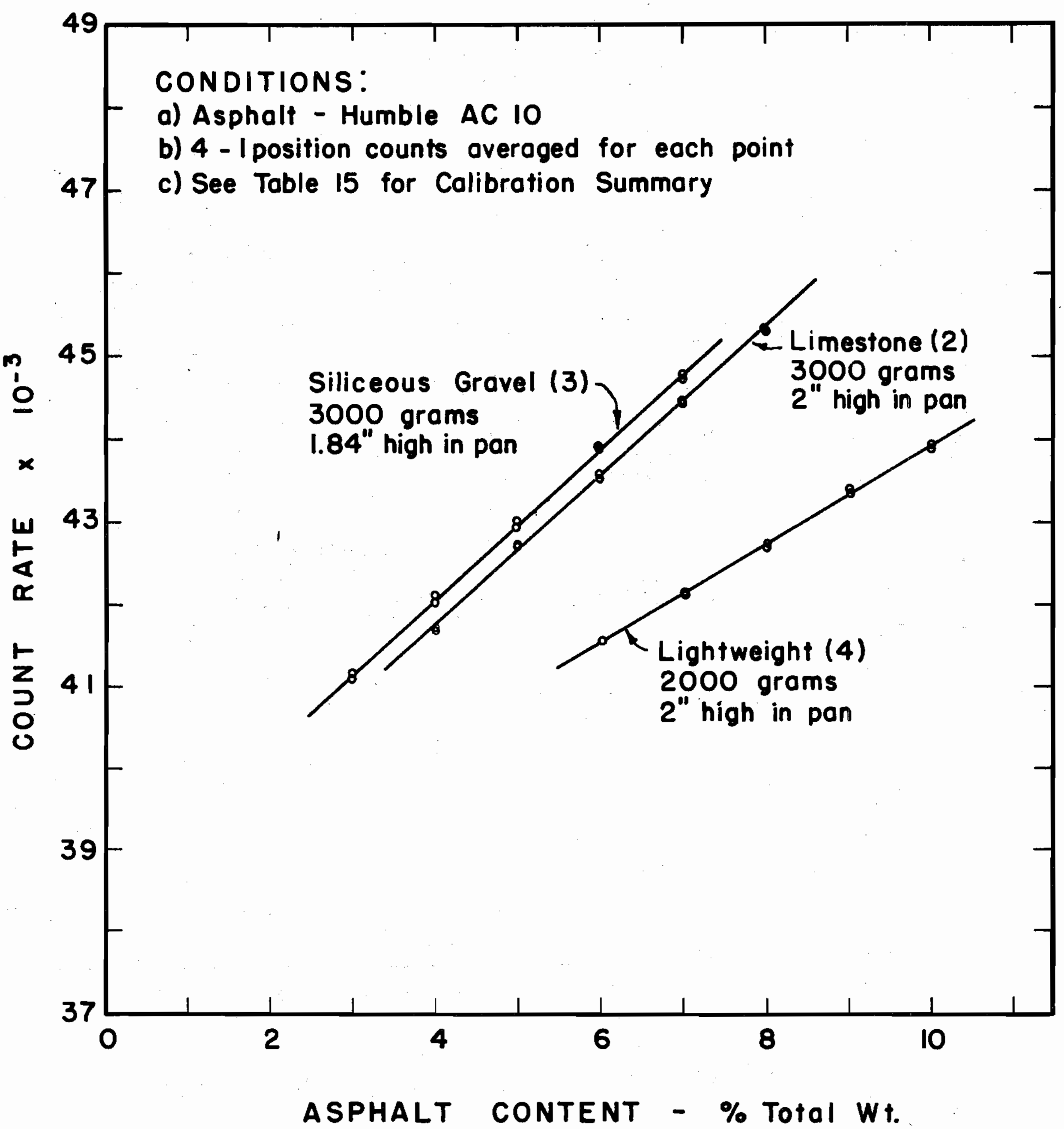
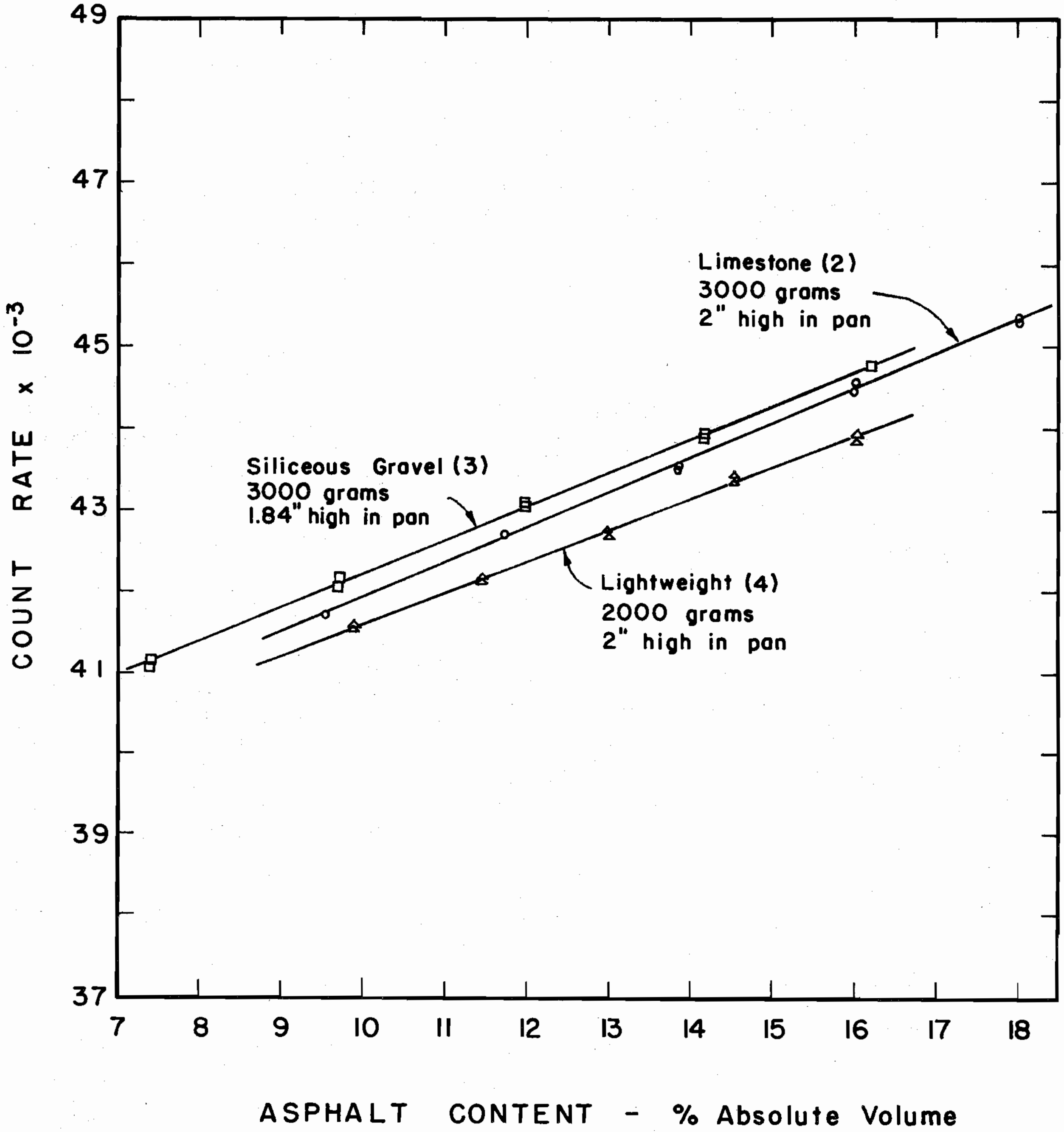


Figure 23. Calibration Curves for 1/2 Pan Samples Using Asphalt Content as Percent of Absolute Volume



calibration equation; however, this is probably not desirable since sensitivity of count rate is much less.

Calibration Curves - Full Pan Samples. The same two calibration alternatives were investigated using full pan samples. In this study, the duplicate samples previously used were combined and the data in Table 16 was obtained.

Table 16

Data - Full Pan Calibration Samples

I. Limestone (Mix 2) - 6000 gram samples

| Count Rate | Asphalt Content (% Total Wt.) | Asphalt Content (% Absolute Volume) |
|---|---|---|
| 39219 | 4.0 | 9.54 |
| 40777 | 5.0 | 11.72 |
| 41880 | 6.0 | 13.89 |
| 43381 | 7.0 | 15.99 |
| 44682 | 8.0 | 18.05 |

II. Siliceous Gravel (Mix 3) - 6000 grams samples

| Count Rate | Asphalt Content (% Total Wt.) | Asphalt Content (% Absolute Volume) |
|---|---|---|
| 37806 | 3.0 | 7.40 |
| 39168 | 4.0 | 9.72 |
| 40497 | 5.0 | 11.97 |
| 41989 | 6.0 | 14.16 |
| 43463 | 7.0 | 16.29 |

III. Lightweight (Mix 4) - 4000 gram samples

| | | |
|---|---|---|
| 39302 | 6.0 | 9.89 |
| 40437 | 7.0 | 11.46 |
| 41092 | 8.0 | 13.01 |
| 42251 | 9.0 | 14.53 |
| 42820 | 10.0 | 16.04 |

The data in Table 16 is shown in Figures 24 and 25, and Table 17 summarizes calibration results. Equation (4) which contains the moisture variable was converted to percent of absolute volume and shown for comparison purposes.

The individual material calibration equations in which asphalt content is expressed as a percent of total specimen weight was again the most sensitive. Segregation in the relatively coarse graded lightweight mix is believed to have affected precision as reflected by the standard error.

The significant finding in this study of full pan specimens is that expressing asphalt content as a percent of absolute volume practically eliminates the material effect as shown in Figure 25. Gauge response to 1/2 pan samples continues to show a material effect as indicated in Figure 23. For the combined full pan data, the use of

Table 17

Summary of Calibration Results - Full Pan Samples

| Material | Calibration Method | Sample Density & Weight | Calibration Equation | Standard Error | Correlation Coefficient |
|---|---|---|---|---|---|
| Limestone | % T.W. | 100.5 PCF<br>6,000 g | $AC = \frac{Counts - 33870}{1353}$ | $\pm$0.08% | 0.9990 |
| Limestone | % A.V. | 100.5 PCF<br>6,000 g | $AC = \frac{Counts - 33,199}{635} - 1.115(MC)$ | $\pm$0.17% | 0.9992 |
| Siliceous Gravel | % T.W. | 100.5PCF<br>6,000 g | $AC = \frac{Counts - 33,517}{1414}$ | $\pm$0.05% | 0.9997 |
| Light-weight | % T.W. | 67.0 PCF<br>4,000 g | $AC = \frac{Counts - 34,100}{885}$ | $\pm$0.20% | 0.9942 |
| Limestone, Siliceous Gr., & Light-weight Combined | % A.V. | - | $AC = \frac{Counts - 33108}{631}$ | $\pm$0.29% | 0.9959 |

Figure 24. Calibration Curves for Full Pan Samples Using Asphalt Content as Percent of Total Weight

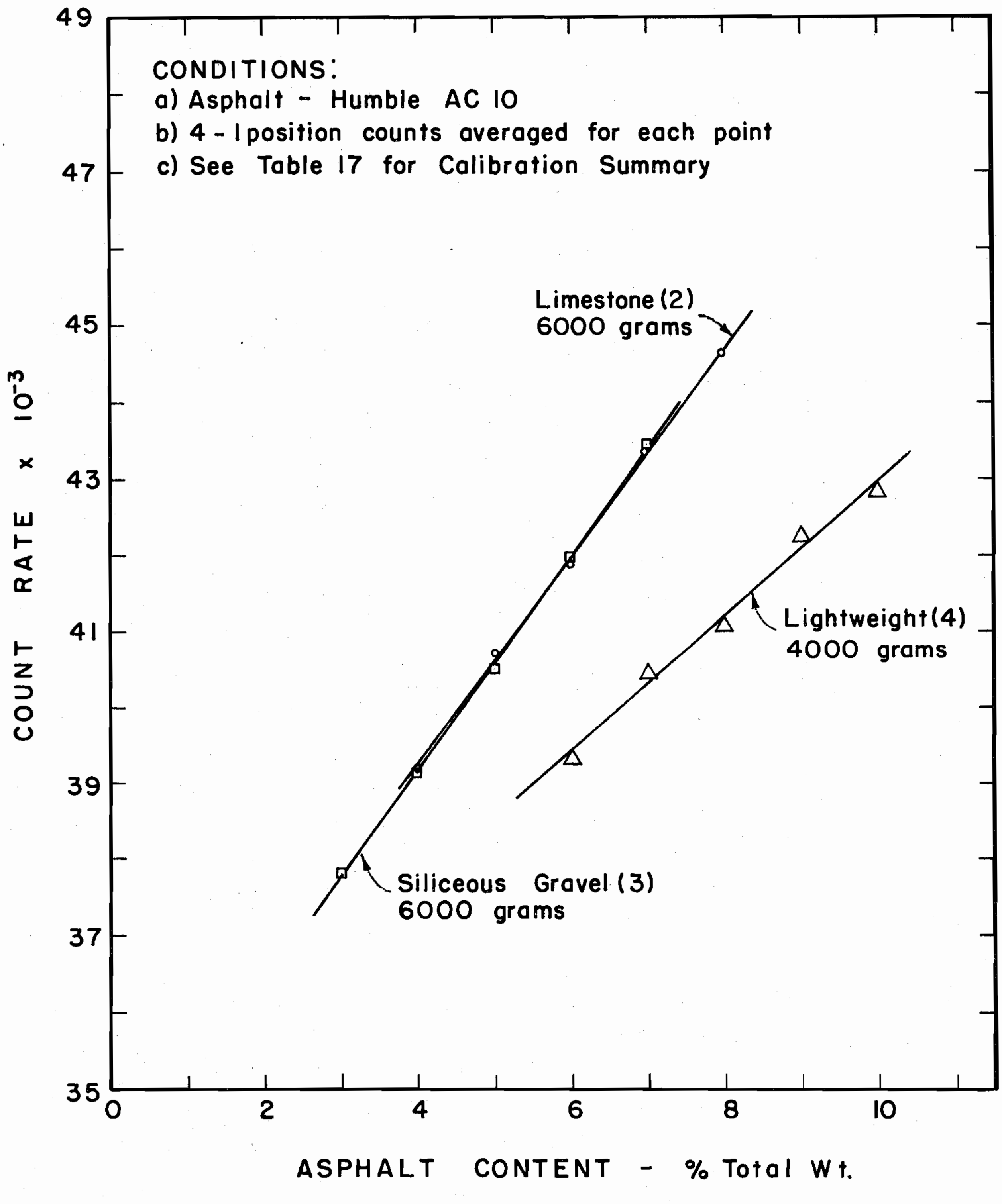
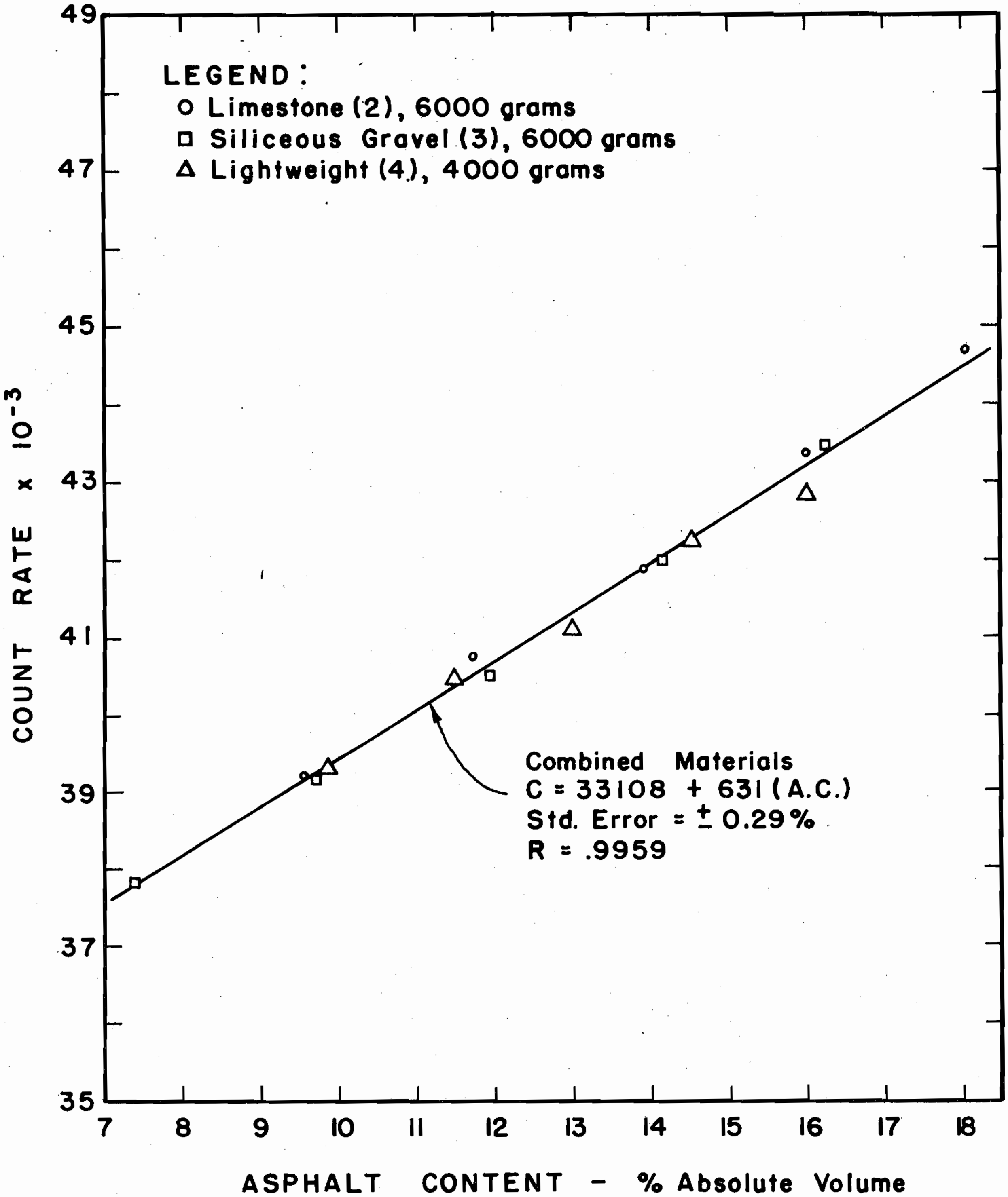


Figure 25. Calibration Curves for Full Pan Samples Using Asphalt Content as Percent of Absolute Volume



one common calibration curve yielded a standard error of about $\pm 0.3\%$, which is within acceptable tolerances. Individual material calibrations improved the standard error only slightly.

CHAPTER 6

# REPEATABILITY STUDIES

An analysis of variance was used to determine whether there was any difference between count rates obtained from molding five separate specimens (one each day), remolding the same specimen kept hot over that period of time, and one reheated to 250° F from room temperature then remolded each day. A siliceous gravel mix containing 5% asphalt and having a Type D gradation (Table 13) was used, and all samples weighed 3000 grams. Asphalt contents shown in Table 18 were obtained from a previously developed calibration curve for the subject material.

Table 18

Gauge Repeatability Study

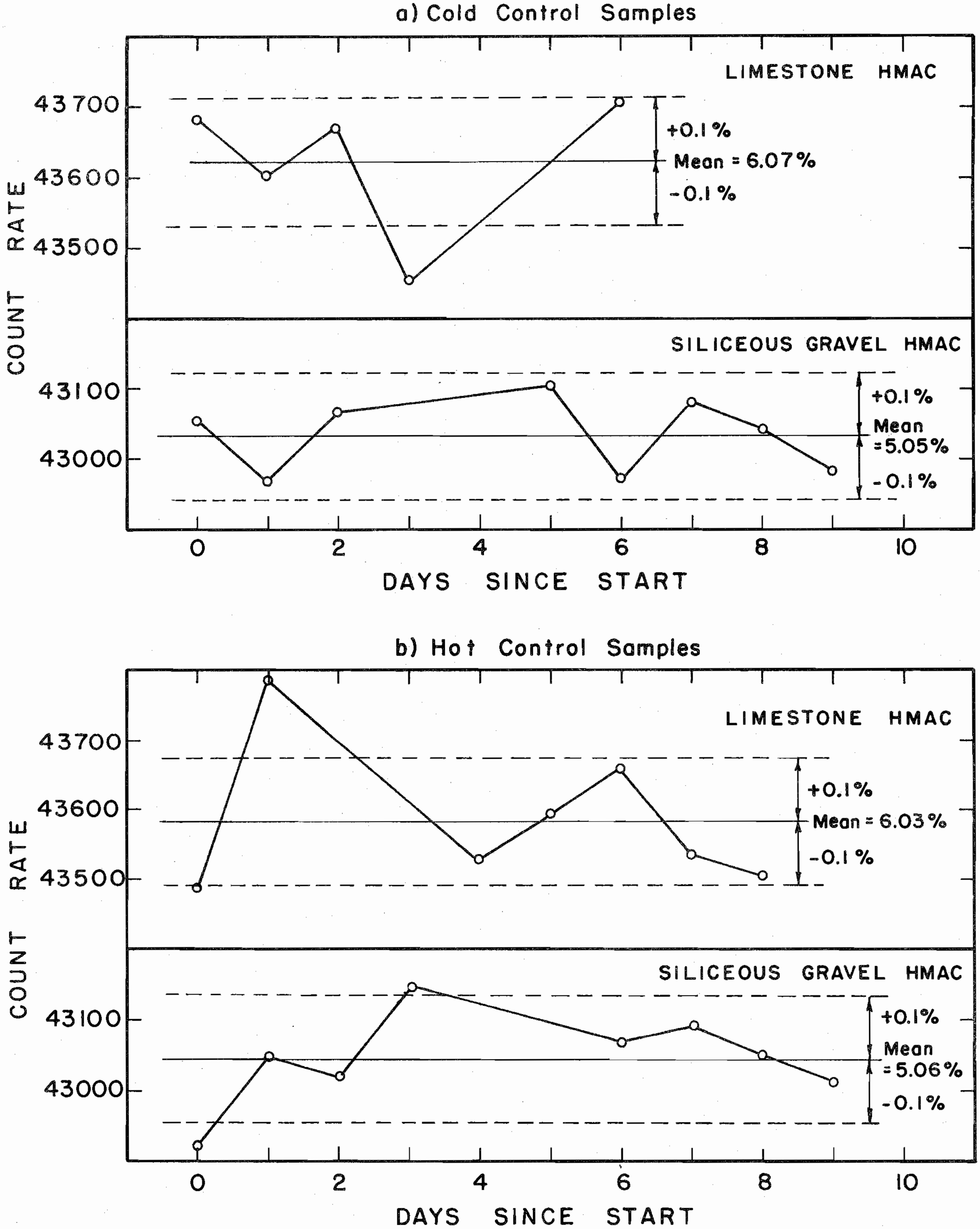
| | Samples 1 thru 5 | Hot Control Sample | Cold Control Sample |
|---|---|---|---|
| | 5.10 | 5.06 | 4.98 |
| | 4.92 | 5.03 | 5.09 |
| | 5.07 | 5.17 | 5.12 |
| | 5.06 | 5.09 | 4.98 |
| | 5.14 | 5.11 | 5.10 |
| $\sigma$ = | ±.08 | ±.05 | ±.07 |

The "within treatment" variation was found to be no different from the "between treatment" variation. The total spread in asphalt content for the five samples was 0.22% and for the two remolded control samples, 0.14%. Standard deviations for each series are about twice the level indicated in the stability studies (as expected) and about the level claimed by the manufacturer.

The above study was carried further to check repeatability and accuracy using the absorptive limestone and to check the feasibility of being able to delay testing for several days (as might be required for record testing purposes). Figure 26 compares 3000 gram samples of the siliceous gravel (5% A.C.) and limestone (6% A.C.) mixes over a period of several days. The same samples were remolded each day. One set (hot) was kept in the oven at 200° F before testing at 250° F, the other (cold) was heated from room temperature. Repeatability for the limestone appears to be a little more erratic than for the gravel. The reason for the relatively high mean values is probably due to the statistical derivation of the regression equations which were developed using five asphalt contents.

Further studies of repeatability are presented in the next chapter.

Figure 26. Repeatability Study-
3000 gram Samples




NOTE: All tests performed at 250°F.

## CHAPTER 7

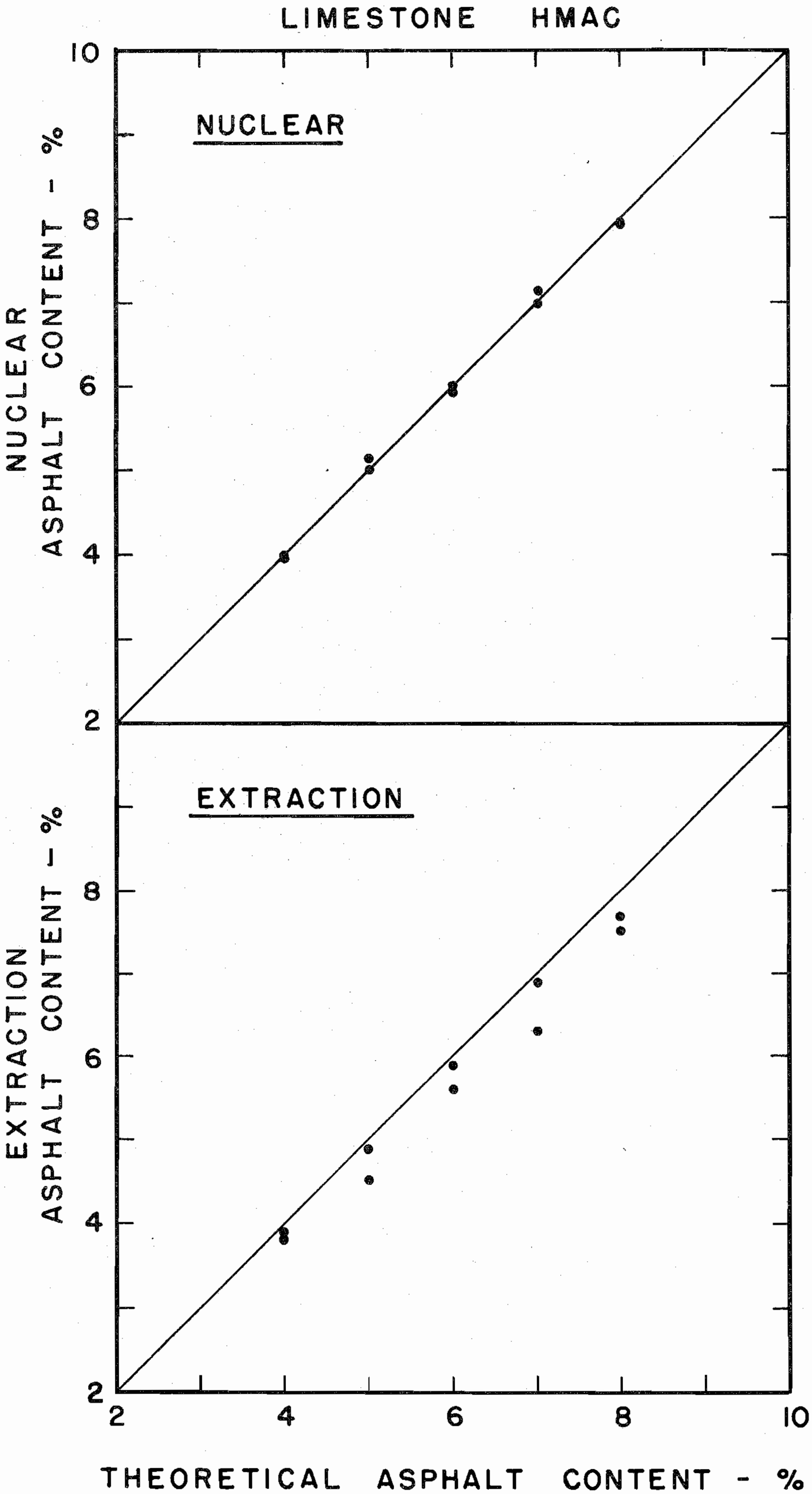
## CORRELATION STUDIES - NUCLEAR AND EXTRACTION TESTS

After nuclear tests were run, the laboratory prepared full pan (6000 gram and 4000 gram) samples were split and asphalt extraction tests (15) performed on each half in accordance with Test Method Tex-210-F. The extraction values were then correlated with theoretical asphalt contents as determined by scale weights (14). For comparison purposes, nuclear values for each of the duplicate 1/2 pan samples (Figure 22) were also correlated with the same theoretical values. Figures 27 thru 29 show the results of these correlations with respect to material type.

It comes as no surprise that the extraction tests are biased on the low side of theoretical asphalt content since this was previously recognized in the literature (12). The nuclear values should correlate well since they were the same as used for calibration. The date spread

Figure 27. Correlation Studies





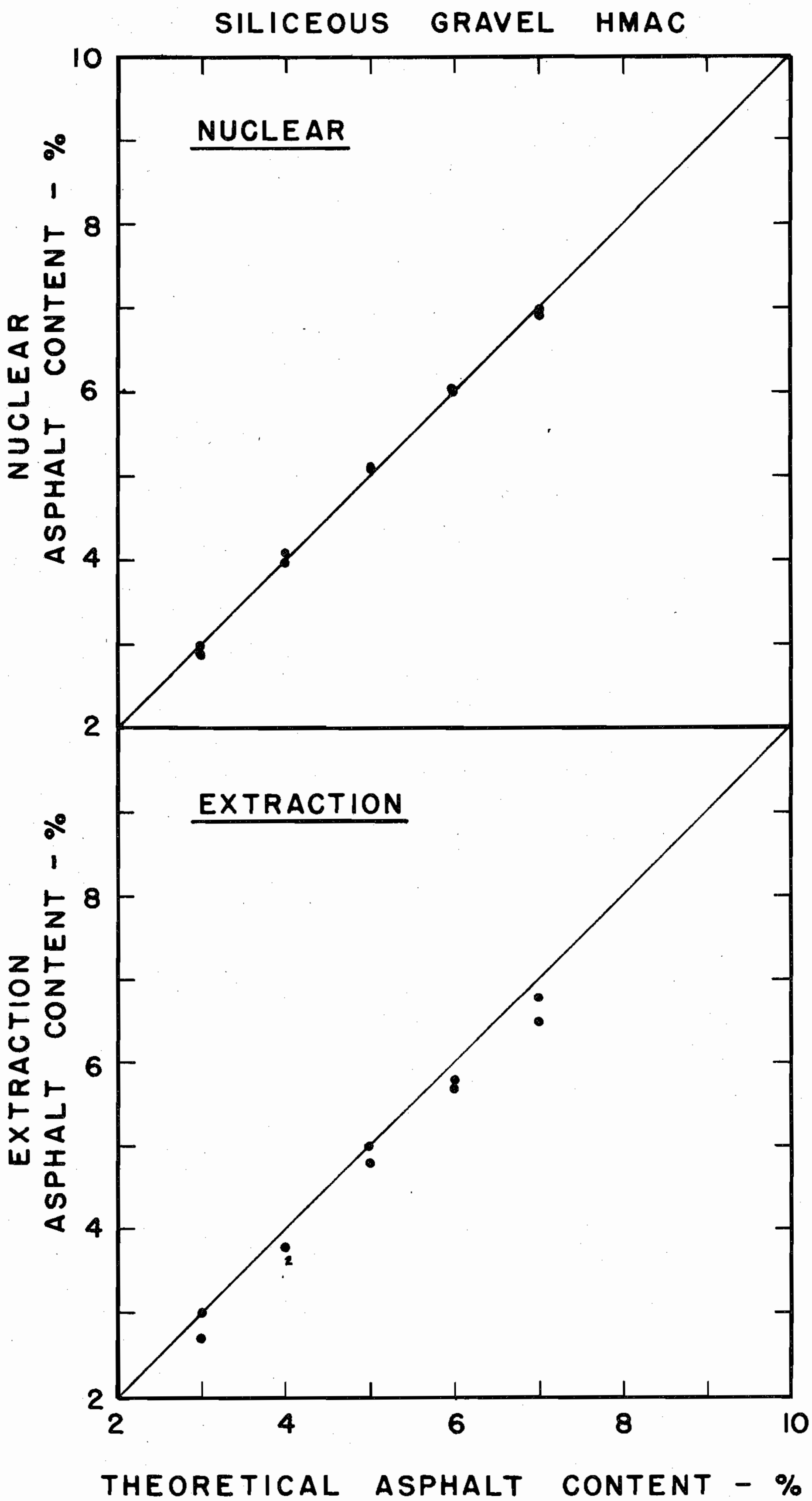
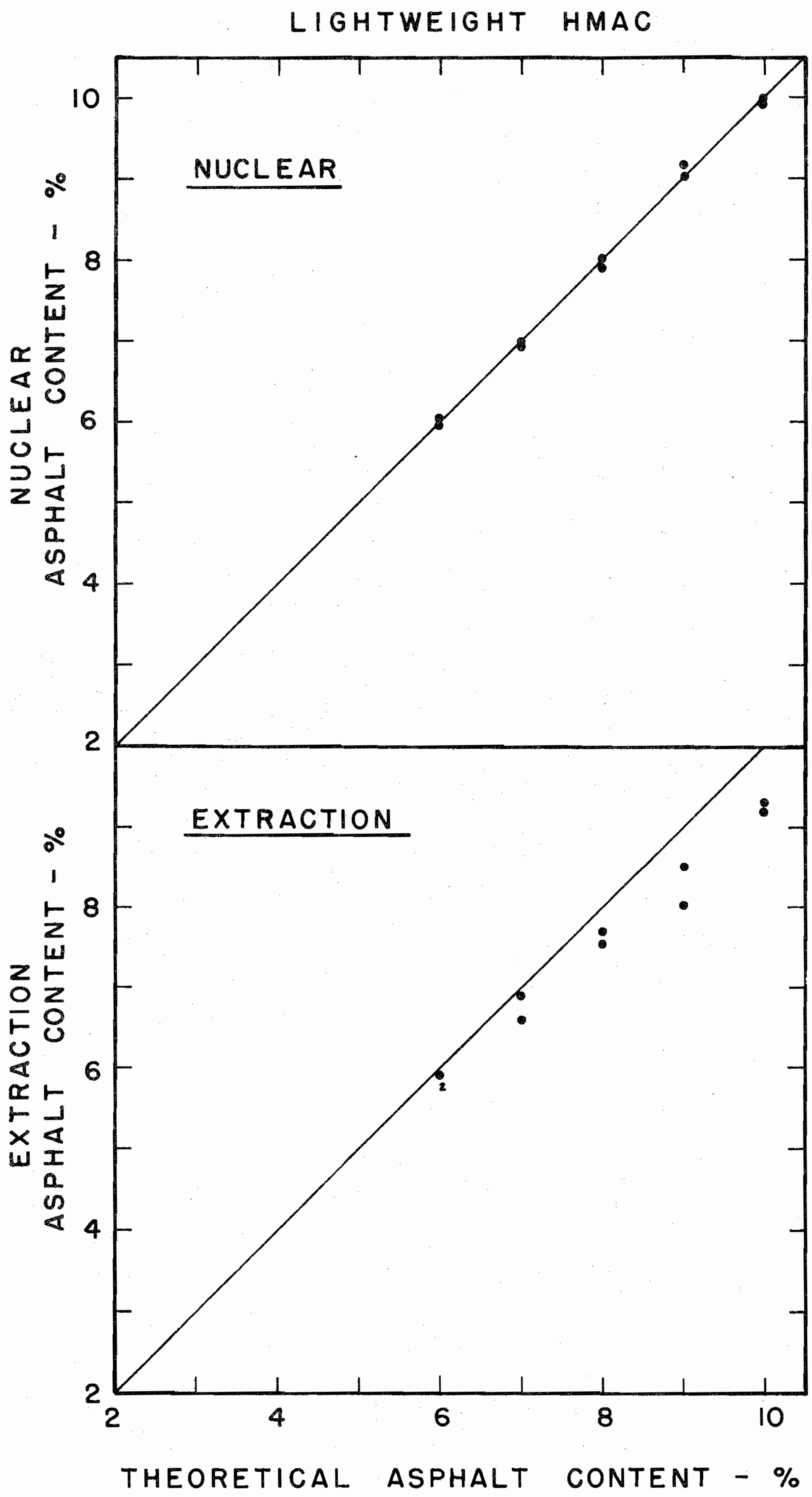

SILICEOUS GRAVEL HMAC
NUCLEAR
NUCLEAR ASPHALT CONTENT - %
EXTRACTION
EXTRACTION ASPHALT CONTENT - %
10
8
6
4
2
2
4
6
8
10
THEORETICAL ASPHALT CONTENT - %

Figure 29. Correlation Studies





in each figure gives some idea of the relative precision of each method.

Table 19 indicates the average difference between duplicate extraction and nuclear tests.

Table 19

Average Differences Between Duplicate Extraction and Nuclear Tests

| Material & Mix | Avg. Asphalt Content Difference Between Duplicate Samples | |
|---|---|---|
| | Extraction | Nuclear |
| Limestone - 2 | 0.32 | 0.07 |
| Siliceous Gravel - 3 | 0.18 | 0.05 |
| Lightweight - 4 | 0.22 | 0.09 |

It is also interesting to note the average difference in nuclear readings for samples containing asphalt (Table 19) and for samples containing no asphalt (Table 4). The repeatability for samples containing asphalt is much better than for samples containing no asphalt.

This study indicates that the nuclear method is clearly more repeatable than the extraction method within the range of asphalt contents tested.

# CHAPTER 8

## FIELD STUDIES WITH HOT MIX ASPHALTIC CONCRETE

A construction project was chosen on which to evaluate the nuclear gauge under field laboratory conditions. The project utilized the limestone mix (Mix 2 - Table 12) and was rigidly controlled by specifications concerning gradation, asphalt content, and temperature. Plant asphalt content was set by scale weight (percent of total batch weight) at 6.2%. Data was gathered over a period of two weeks during which time additional aggregate materials were continually being hauled to the mixing plant as was asphalt from various storage tanks operated by the producer. A variety of weather caused substantial fluctuation of stockpile moisture.

For reasons previously discussed, it was planned to use 6000 gram samples in the nuclear gauge and occasionally check 3000 gram samples.

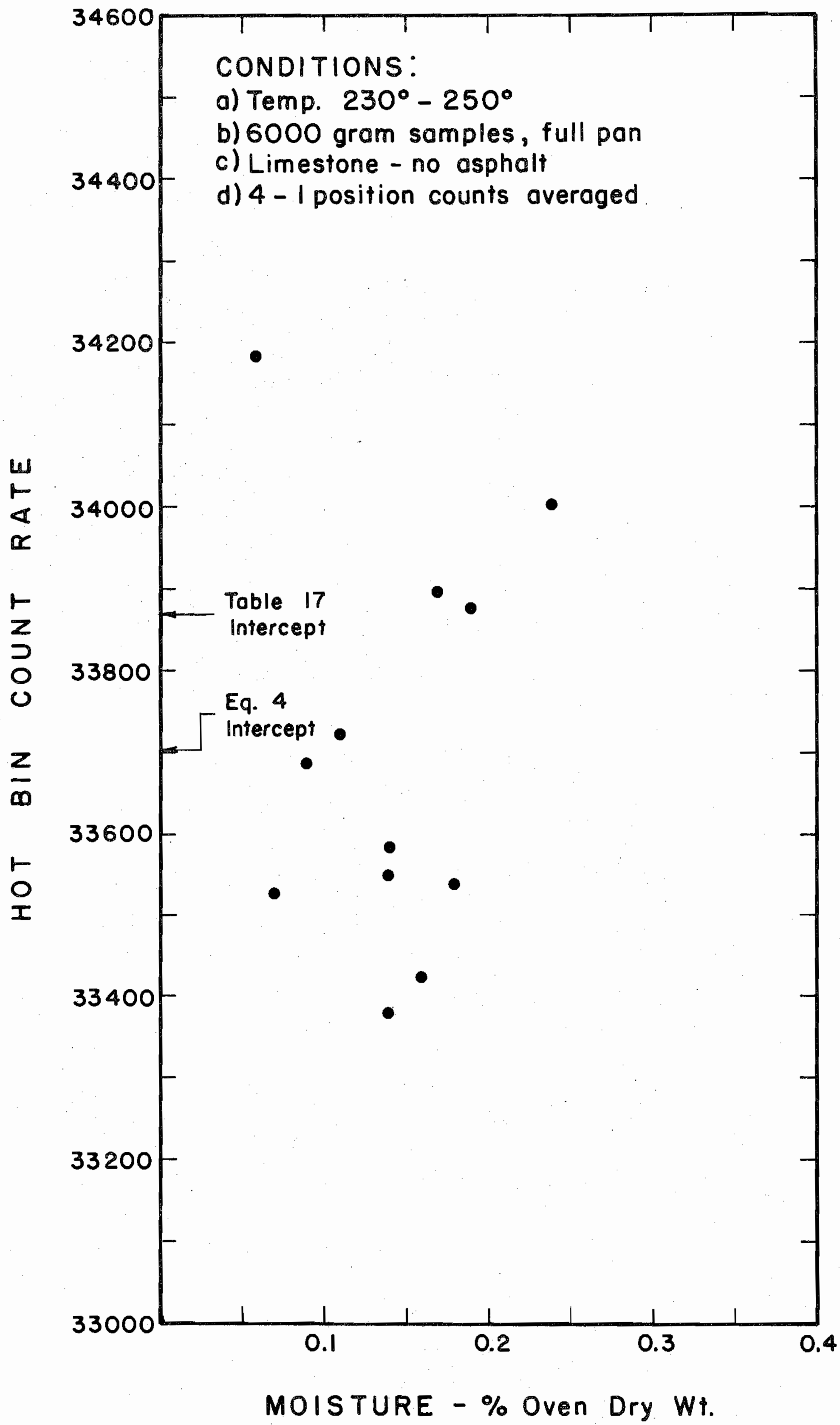
Aggregates from the hot bins were sampled to check moisture contents by oven drying and by correlation with nuclear count rate. The point of sampling for hot mix was in the haul truck just after the mix was dumped from the pug mill. As many extraction tests as possible were obtained to correlate with nuclear values. Standard counts were obtained daily to monitor operation of the gauge.

Certain stability problems were encountered in the field laboratory. It was observed that sharp surges in A. C. line voltage of ± 5 volts, as measured with an ordinary voltmeter, caused erratic counting. This usually occurred when the hot mix plant was turned on or off. Also, the combination of moisture in the air and dust caused significant increases in count rate. These problems, although frustrating at times, were not insurmountable, and the gauge was subsequently returned to the manufacturer for modification. Field operations after the gauge had been modified and used in the field for one week, indicated that these problems had been corrected; however, the calibration curves shifted. This presented no real problem, since an adjustment could be made to the y intercept for the difference, leaving the slope the same.

Hot Bin Samples

Attempts were made to adjust the calibration curve for moisture by testing hot bin samples (0% asphalt), then moving the y intercept up or down as indicated, keeping the slope constant. It was suspected that false count rates were being obtained in this procedure because of the possibility that any moisture present, however small, was being continuously driven off as a result of high temperature ($230^{o}$-$300^{o}$ F) and handling. About 15 minutes was required to sample, combine, mold, and test the hot, raw aggregate. In an attempt to correct this problem, the sample for nuclear testing was combined at the hot bins by approximate volume, then sealed in a container to prevent moisture loss. At the same time, additional samples from each hot bin were obtained and sealed in moisture tight containers. These samples were used to determine moisture content by oven drying. A plot of moisture content (percent of dry weight) versus hot bin count rate (based on 6,000 gram samples) is shown in Figure 30. This figure indicates that the projected y intercept (0% moisture) is somewhat lower than that predicted by any of the previously developed calibration equations (Table 17).

Figure 30. Moisture in Hot Bin Samples



Based on this information and previous experience with trying to control moisture in the laboratory, it is entirely possible that the nuclear hot bin samples contained very little, if any, moisture during testing. From a practical standpoint, it was extremely difficult to sample and test these materials without losing moisture in the process. The loss of a few grams of moisture represented a substantial percentage for the sample weight used.

It might be argued that just as much, if not all moisture is lost between the time materials are weighed from the hot bins and mixed with asphalt (where it is assumed that moisture is sealed in). This may be true in some plants; however, in this case, batching times averaged about 45 seconds whereas laboratory handling required about 15 minutes.

Based on the above discussion, it was concluded that nuclear moisture tests on hot bin samples would not be as accurate as the standard oven dry moisture tests, at least for this material and range of moisture contents.

Hot Mix Samples

After sampling the mix from the haul trucks, 6000 grams were compacted level with the top of the pan and four 1-position counts were obtained and averaged. Exactly the same molding procedure and testing temperature (250° F) as used in calibration was used in the field. It was not always possible to obtain a companion hot bin moisture test for each nuclear asphalt test. Extraction tests were run on the average of one a day.

On two occasions during field testing, calibration checks were made to verify the laboratory developed asphalt curve (Figure 16). One check indicated a calibration shift to the low and one to the high side of the established curve. In both cases, the check samples were oven dried prior to adding asphalt from the current day's haul. It was concluded that equation (4) represented an average condition and could justifiably be used in the field.

Only one moisture test was secured during the first two days of testing. Because of laboratory experience with trying to keep moisture in hot mix, it was not anticipated that there would be any after asphalt was mixed with the aggregate. After studying the hot bin samples and nuclear test results from the first two days, it was concluded that the gauge was detecting moisture in addition to asphalt. During this period, testing time was fixed at about one test every 10 to 30 minutes for a one man operation.

Table 20 shows all of the data gathered on this project. Figure 31 shows frequency distribution plots which indicate the variations experienced. The mean value for total asphalt and moisture content was about 0.16% higher than the 6.2% asphalt added at the plant by scale weights. When moisture was taken into account (Equation 4), the mean value for nuclear asphalt content was 6.21%. Extraction tests yielded a mean asphalt content of 6.1%.

The standard deviation ($\sigma$) for nuclear asphalt content ($\pm$0.11%) was about half that for asphalt extraction tests ($\pm$ 0.25%), thus indicating superior repeatability for the nuclear test, at least for this material.

Table 20

Field Data - Limestone HMAC Samples

| Sample Number | Date | 6000 g Count Rate | Total % (Asphalt + Moisture) | % Moisture | % Moisture x 1.100 | Net Asphalt Content,% | Asphalt Extraction Test,% |
|---|---|---|---|---|---|---|---|
| 1 | 5-13-71 | 42678 | 6.51 | - | - | - | 6.3 |
| 2 | 5-13 | 42416 | 6.32 | 0.17 | 0.19 | 6.13 | - |
| 3 | 5-13 | 42432 | 6.33 | - | - | - | - |
| 4 | 5-13 | 42420 | 6.32 | - | - | - | - |
| 5 | 5-13 | 42037 | 6.05 | - | - | - | 5.7 |
| 6 | 5-14 | 42673 | 6.51 | - | - | - | 6.0 |
| 7 | 5-14 | 42498 | 6.38 | - | - | - | - |
| 8 | 5-14 | 42538 | 6.41 | - | - | - | - |
| 9 | 5-14 | 42560 | 6.43 | - | - | - | - |
| 10 | 5-14 | 42026 | 6.04 | - | - | - | - |
| 11 | 5-14 | 42274 | 6.22 | - | - | - | - |
| 12 | 5-17 | 42511 | 6.39 | - | - | - | 6.2 |
| 13 | 5-17 | 42308 | 6.24 | 0.14 | 0.15 | 6.09 | - |
| 14 | 5-17 | 42414 | 6.32 | - | - | - | - |
| 15 | 5-18 | 42409 | 6.32 | 0.09 | 0.10 | 6.22 | 6.1 |
| 16 | 5-19 | 42524 | 6.40 | 0.07 | 0.08 | 6.32 | 6.0 |
| 17 | 5-19 | 42337 | 6.26 | 0.14 | 0.15 | 6.11 | - |
| 18 | 5-19 | 42267 | 6.21 | 0.11 | 0.12 | 6.09 | - |
| 19 | 5-20 | 42570 | 6.43 | - | - | - | 6.0 |
| 20 | 5-20 | 42595 | 6.45 | 0.12 | 0.13 | 6.32 | 5.7 |
| 21 | 5-25 | 42769 | 6.58 | - | - | - | 6.5 |
| 22 | 5-25 | 42427 | 6.33 | 0.10 | 0.11 | 6.22 | - |

(Table 20 cont'd)

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 23 | 5-25 | 42436 | 6.34 | 0.14 | 0.15 | 6.19 | - |
| 24 | 5-25 | 42724 | 6.54 | 0.24 | 0.26 | 6.28 | - |
| 25 | 5-26 | 42454 | 6.35 | - | - | - | 6.3 |
| 26 | 5-26 | 42868 | 6.65 | 0.18 | 0.20 | 6.45 | 6.3 |
| 27 | 5-26 | 42484 | 6.37 | 0.19 | 0.21 | 6.16 | - |
| 28 | 5-26 | 42515 | 6.39 | 0.16 | 0.18 | 6.21 | - |
| | $\bar{x}$ | | 6.36 | 0.14 | | 6.21 | 6.10 |
| | $\sigma$ | | $\pm$0.14 | | | $\pm$0.11 | $\pm$0.25 |

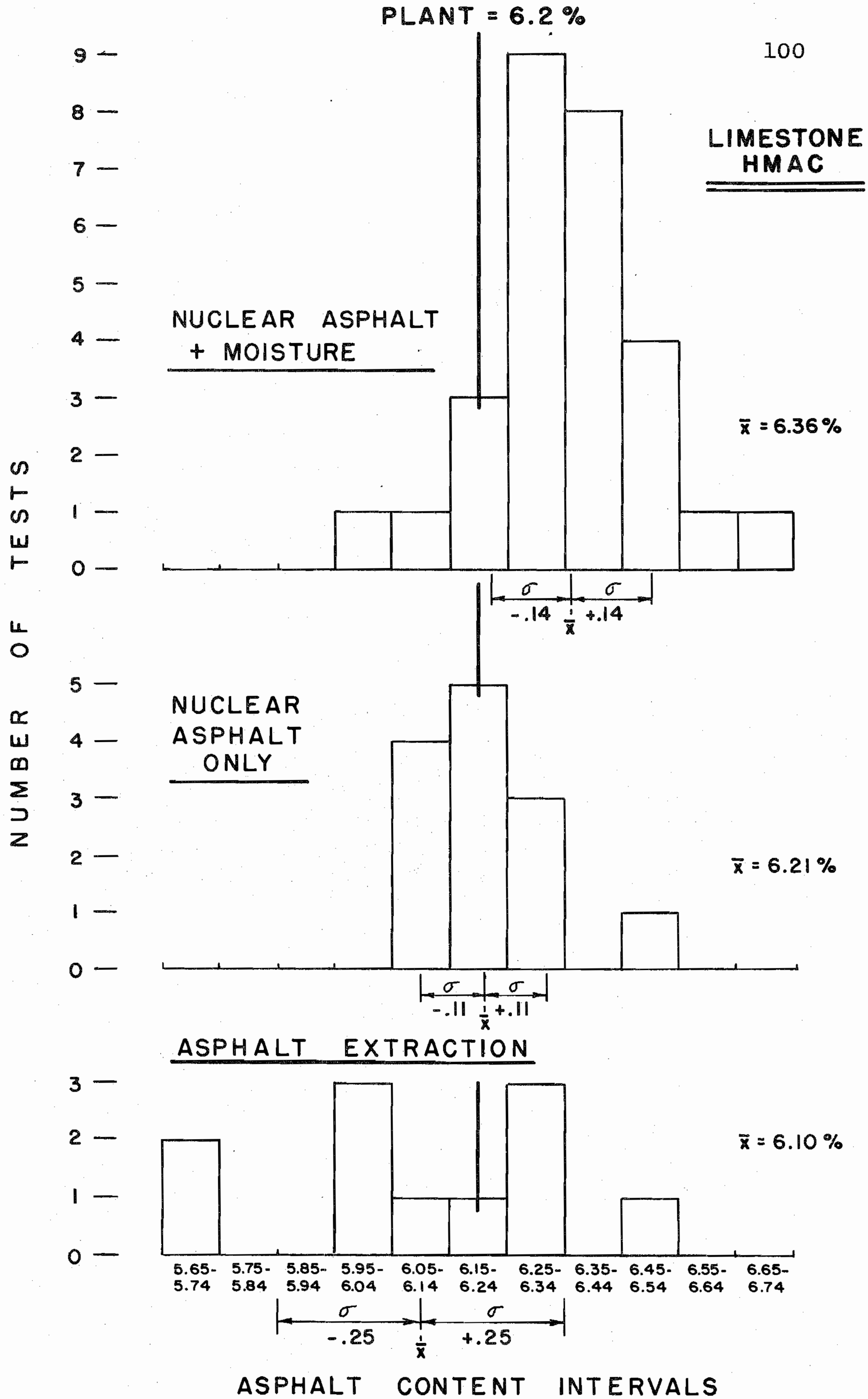



Figure 31. Frequency Distribution for Field Tests

CHAPTER 9

# LABORATORY STUDIES WITH MOISTURE AND SOIL

Since the nuclear gauge offers the advantage of testing speed, it was decided to evaluate the gauge for moisture in various soils.

Past experience has indicated the existence of a material effect when calibrating other gauges for moisture in soil (1)(16). Several subgrade and base materials were used in this study, and the oven dry moisture test was used as the calibration standard. Moisture contents were based on percent of dry weight (dried at $200^{\circ}$ F $\pm$ $10^{\circ}$) and each nuclear value was based on the average of four 1-position counts.

## Moisture Calibration - Subgrade Soils

Five types were used, one of which was a 50% - 50% mixture of two clays and another had 4% lime added. All materials were 100% finer than the 40 mesh sieve, and the soil constants are shown in Table 21.

Table 21

Subgrade Soil Constants

| Material | Liquid Limit | Plastic Limit | Plasticity Index | Volumetric Shrinkage |
|---|---|---|---|---|
| *Adobe | 26.4 | 12.0 | 14.4 | 23.3 |
| Black Clay | 75.6 | 24.3 | 51.3 | 57.9 |
| Yellow Clay | 81.0 | 21.2 | 59.8 | 56.8 |
| 50% Black-50% Yellow | - | - | - | - |
| Yellow Clay + 4% Lime | 62.8 | 36.2 | 26.6 | 37.2 |

*Calcareous, sandy clay, lean

The yellow and black clays and calcareous adobe material came from the same geologic formation (Taylor Series). The yellow clay with lime was similar, but came from another location.

It was not always possible to hold weight and volume relationships constant for calibration comparisons, since the unit weight and compaction characteristics of each material were different. A weight of 3,500 grams was chosen because this amount completely filled the pan for the clay materials and came within one-half inch of filling for the adobe material.

Each material was molded at several moisture contents. Known amounts of water were added to dry soil, allowed to cure (or equalize) for several days, and thoroughly mixed. The samples were then compacted into the pan and tested in the nuclear gauge. After testing, the complete sample was oven dried to obtain percent moisture.

Figures 32 and 33 show calibration relationships for these soils. For the clays, all the calibration slopes are about parallel, and the 50% mixture is about half way between the two parent materials. Two points are shown for the yellow clay and lime; however, there was not enough data for a separate calibration.

At least for these materials, individual calibration curves must be developed to obtain tolerable accuracy. It appears that calibration may be estimated if the percent combination of two materials and their calibration curves are known.

In each case, the correlation coefficient and standard error indicate excellent agreement with the oven dry test. With careful sample preparation and possibly duplicate points, precisions in the range of 0.1% to 0.4% moisture may be expected 68% of the time for materials similar to those tested.

Figure 32. Clay subgrade Calibration Curves

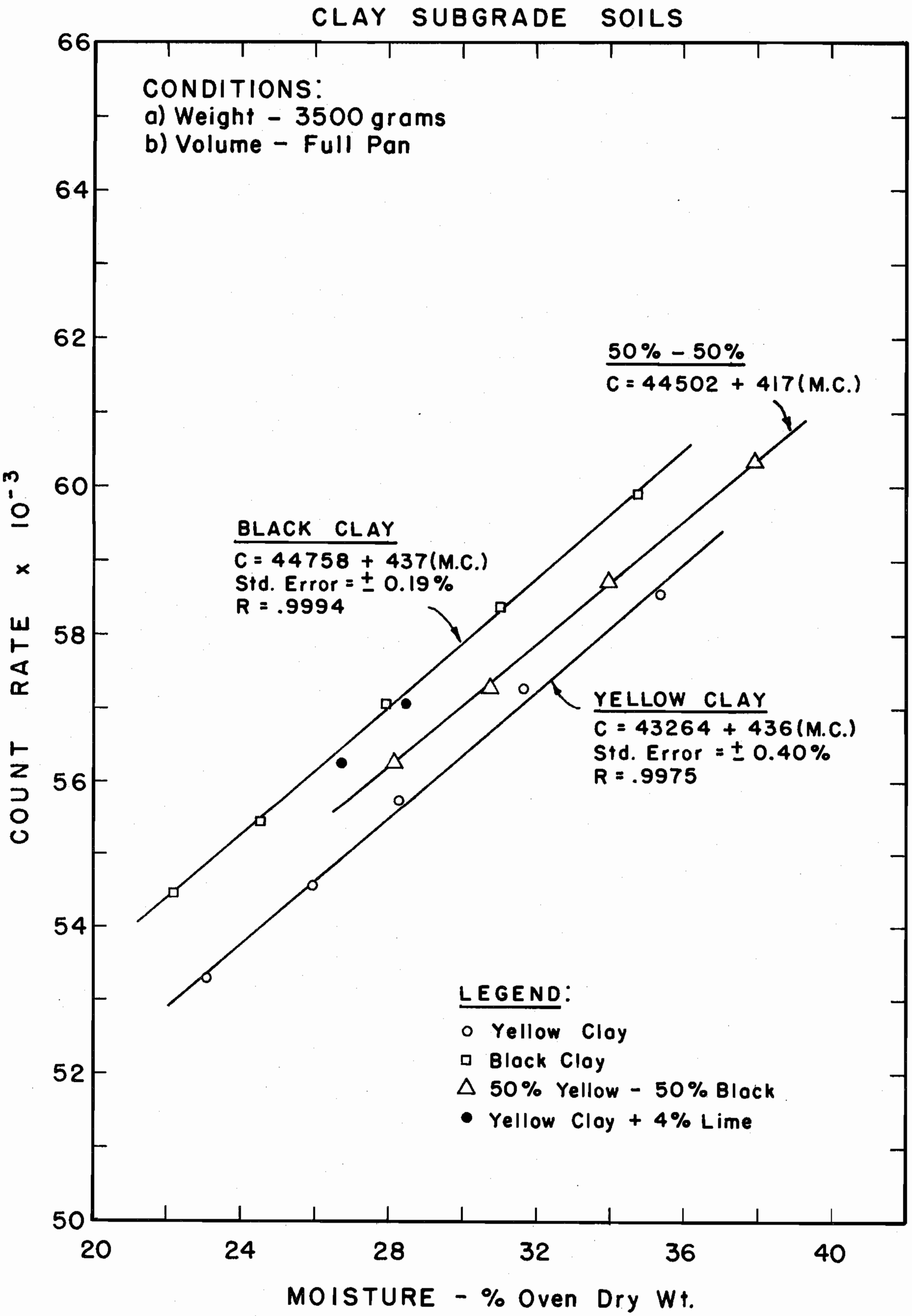
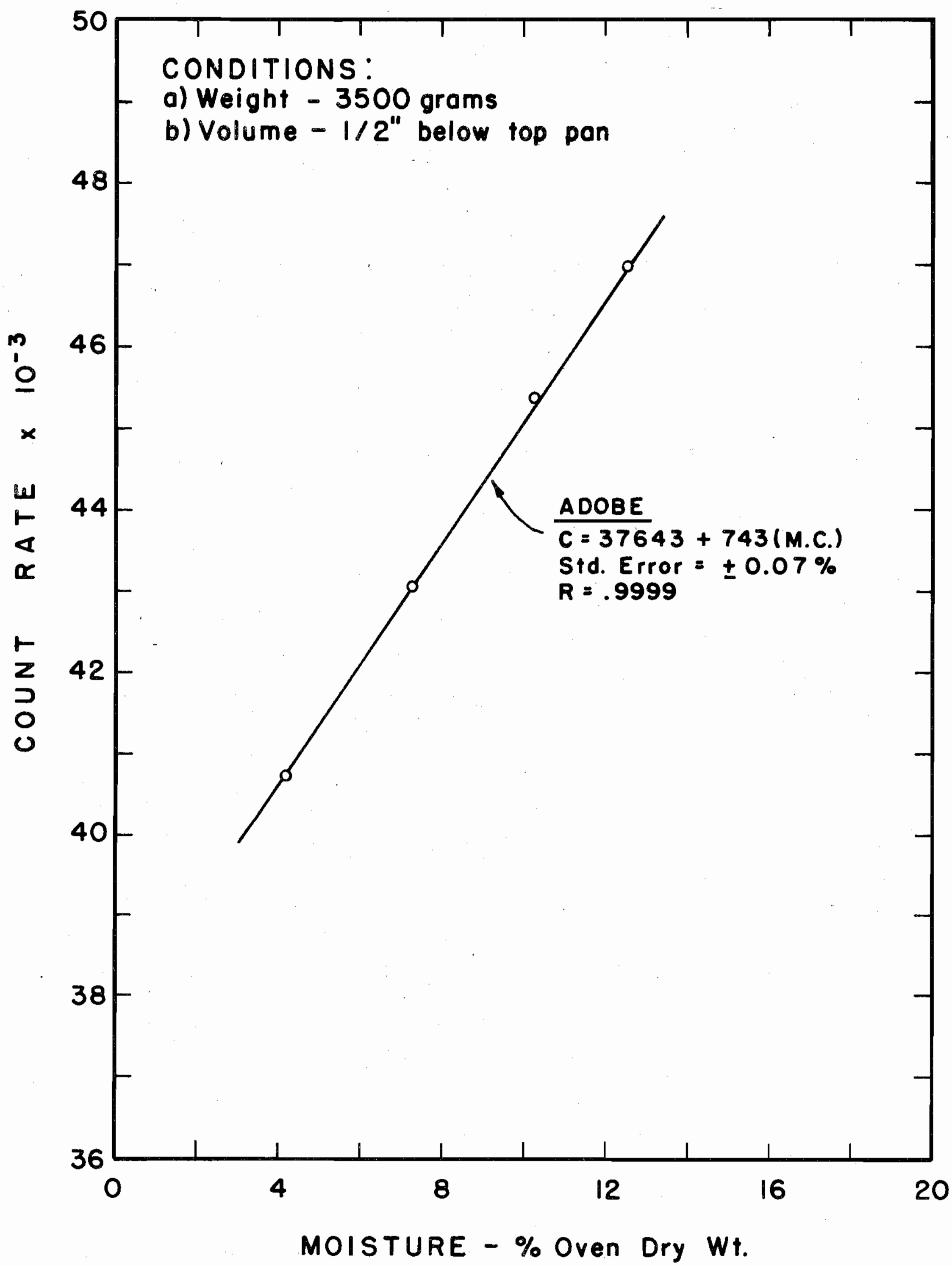


Figure 33. Adobe Subgrade Calibration Curve



The necessity of calibrating for individual soils makes use of this gauge impractical for job control except where only a few soil types are encountered.

## Moisture Calibration - Base Materials

This investigation was limited to non-stabilized materials processed by crushing or from pit run sources. Three limestones, a combination of limestone and sandstone and a siliceous gravel were used, each having a relatively coarse gradation. The raw limestone-sand mix previously described in the hot mix section of this report was also included.

Tables 22 and 23 give the soil constants and gradation data for each material.

Table 22

Base Material Soil Constants

| Material | Liquid Limit | Plastic Limit | Plasticity Index | Volumetric Shrinkage |
|---|---|---|---|---|
| Georgetown Limestone | 20.1 | 15.3 | 4.8 | 6.7 |
| Onion Creek Limestone | 19.0 | 15.7 | 3.3 | 4.5 |
| Magnesium Limestone | 24.0 | 19.9 | 4.1 | 5.1 |
| Cap Mtn. Ls-Ss | 21.9 | 17.4 | 4.5 | 9.2 |
| Taylor Gravel | 21.2 | 12.3 | 8.9 | 13.4 |

Table 23

Base Material Gradations

| Sieve Size | Georgetown Limestone | Onion Ck Limestone | Magnesium Limestone | Cap.Mtn. Limestone-Sandstone | Taylor Gravel |
|---|---|---|---|---|---|
| | Percent of Material Retained | | | | |
| 1 1/4" | 3 | 9 | 4 | 15 | 4 |
| 7/8" | 19 | 20 | 21 | 31 | 9 |
| 5/8" | 27 | 26 | 31 | 41 | 15 |
| 3/8" | 41 | 35 | 44 | 50 | 27 |
| No. 4 | 55 | 42 | 55 | 58 | 40 |
| No. 10 | 66 | 49 | 64 | 63 | 48 |
| No. 20 | 71 | 56 | 68 | 65 | 53 |
| No. 40 | 76 | 66 | 71 | 68 | 60 |
| Pass No. 40 | 24 | 34 | 29 | 32 | 40 |

The testing operation was coordinated with routine laboratory tests for determination of optimum moisture and maximum density in soils. Specimens prepared, compacted, and tested (15) in accordance with Test Method Tex - 113 - E were broken down, and 6000 grams recompacted to the top of the sample pan. Count rates were then obtained and correlated with percent moisture as determined by oven drying.

Figure 34 shows a plot of all base material calibration data. For purposes of clarity, calibration slopes were not shown. Table 24 summarizes the regression

Figure 34. Base Material Calibration Data

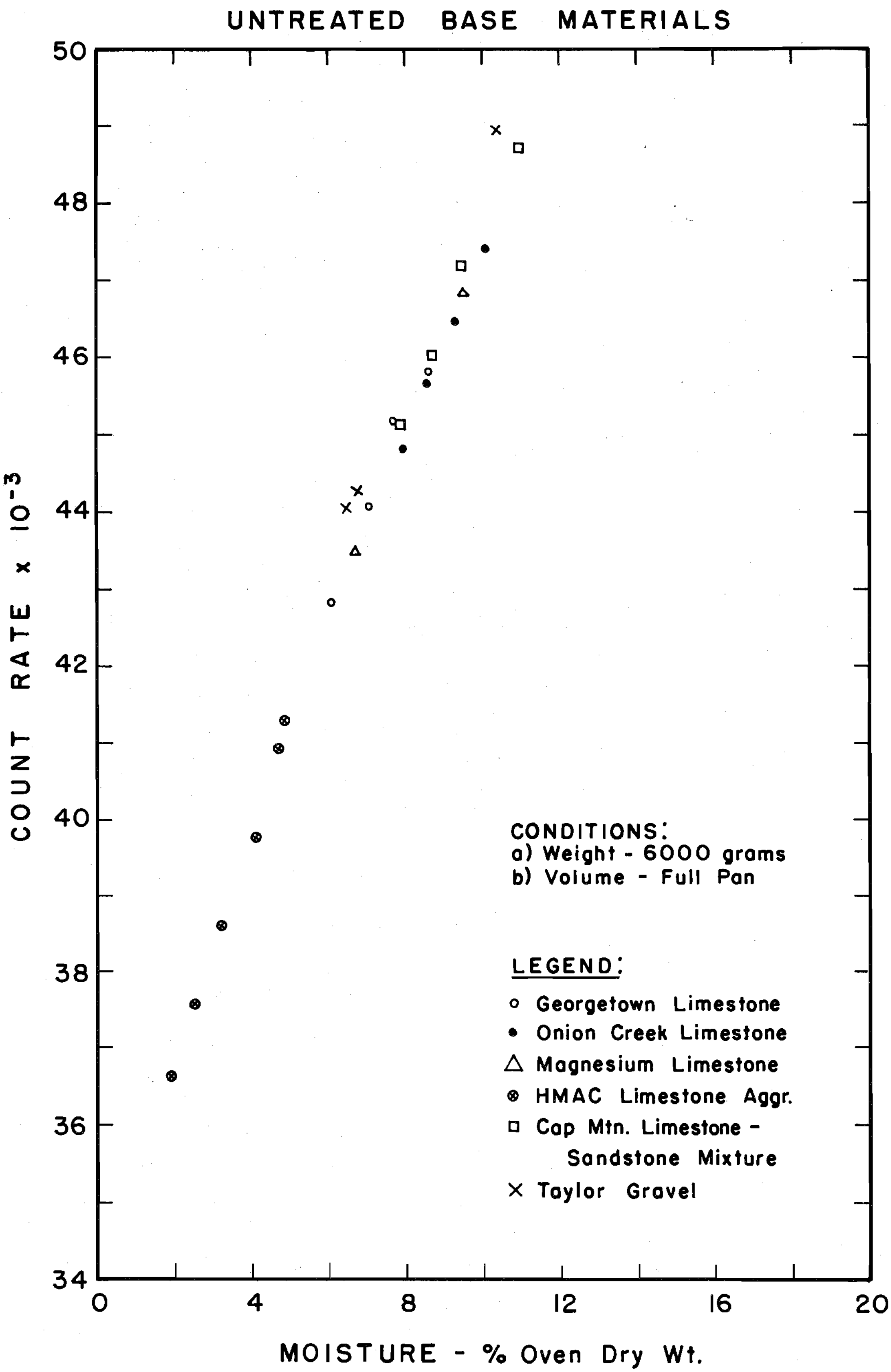


Table 24

Regression Data - Base Materials

| Material or Group of Materials | y Intercept, Counts | Slope, Counts per % Moisture | Std. Error, % Moisture | Corr. Coeff. |
|---|---|---|---|---|
| Georgetown Limestone | 35591 | 1204 | ± .21 | .9876 |
| Onion Creek Limestone | 35223 | 1212 | ± .08 | .9975 |
| Magnesium Limestone - Not enough data for separate analysis. | | | | |
| Cap Mountain Limestone-Sandstone Mixture | 35918 | 1166 | ± .12 | .9971 |
| Taylor Gravel | 35691 | 1272 | ± .06 | .9998 |
| Hot Mix Mat'ls (80% Limestone 20% Sand)* | 33648 | 1533 | ± .07 | .9986 |
| Group Combination - Georgetown, Onion, Mag. and Cap. Mtn. | 35734 | 1174 | ± .16 | .9939 |
| Group Combination - Georgetown, Onion, and Magnesium Limestones | 35954 | 1141 | ± .16 | .9932 |

*The hot mix limestone material was from the same source as the Georgetown limestone.

data for each material by type and combinations of like materials.

Depending on the degree of accuracy desired, either one of the individual calibrations, or a combination curve may be used. With the exception of the Georgetown limestone, standard errors for individual materials averaged about $\pm$ 0.08% moisture, while material groupings were twice that amount. The hot mix limestone combination was not included in either group because it obviously had a different slope and y intercept. This may be due to the much finer nature of this mix or the true gauge response could be quadratic rather than linear in this area. A general regression equation for the limestones tested is as follows:

$$MC = \frac{\text{Counts} - 35{,}734}{1174}$$

Std Error = $\pm$ 0.16% Moisture
Corr. Coeff. = 0.9939

Additional data, duplicate samples, or the use of moisture as a percent of absolute volume would probably strengthen this equation. In any event, it appears that a single calibration curve for limestones is practical, and a reasonable degree of precision (Std. Error $\sim$ 0.2%) may be expected; whereas, individual calibrations were needed for the subgrade materials. This data is even more significant when considering the coarse nature of the materials tested.

These findings are also significant because of the possible use of this gauge in controlling asphalt content in "black base." Many times, the gradation of this material is very coarse. Since gradation does not seem to affect calibration, there appears to be an additional practical use for the gauge.

# CHAPTER 10

# DISCUSSION

It is felt that this study indicates the feasibility of using the nuclear method for field controlling asphalt content in bituminous mixes and moisture content in base materials. When properly calibrated, the nuclear gauge provides better precision than extraction tests as evidenced by standard errors in the laboratory study and standard deviations in the field. Based on experience with granular materials in the moisture study, there appears to be no reason why this method could not be used to control asphalt content in "black base".

## Potential Laboratory Uses

There are other potential uses for the gauge which would easily fit into standard Texas Highway Department laboratory operations. In addition to completely replacing the asphalt extraction test, a few of these uses are:

1. Determining moisture contents in laboratory compaction tests (Test Method Tex - 113 - E).
2. Use on construction projects in conjunction with the volumeter to determine moisture contents in base materials and soils.
3. Concurrent control of asphalt content in stabilized bases and subgrades, asphalt content in HMAC, and moisture contents in bases and soils on several projects from a centralized field laboratory.
4. Determination of moisture contents in concrete aggregate.
5. Determination of asphalt content in rock asphalt.
6. Where the cohesiometer test is used as a means of control, the gauge could be calibrated to determine asphalt or moisture content in the 2 inch by 6 inch cylindrical specimens.

## Additional Research

There are also benefits which could be gained from using the gauge in additional research concerning:

1. Establishment of a correlation procedure which could be used to calibrate the small portable nuclear moisture devices presently used in construction.
2. Determination of cement or lime content in subgrades and bases.
3. Measure moisture content in concrete.
4. Use of three 2 inch by 4 inch cylindrical HMAC specimens tested simultaneously to determine if calibration sensitivity for this method can be improved.
5. Investigation of additional materials using asphalt contents calculated as percent of absolute volume to determine if one common calibration curve can be used for all asphalts and materials. The precision of this calibration method could probably be improved.
6. Use of percent of absolute volume in conjunction with moisture tests to determine if a common calibration curve is practical.
7. Prediction of gauge response to asphalt based on known response to moisture.

8. Use in soil identification.
9. Correlation with soil constants (liquid limit, plastic limit, plasticity index, shrinkage).

Methods Other Than Nuclear

The nuclear method of testing has the potential of improving testing efficiency over a wide range of laboratory operations; however, there are other methods of determining moisture and asphalt content which are reported to be just as fast and less expensive than the nuclear method. For instance, the Nebraska Department of Roads (11) has developed a pycnometer method for asphalt content determination which requires less than one half hour per test. It is reported to be very dependable and about equal to their extraction test accuracy (which is the best reported in the publications reviewed). Equipment costs for this method are about ten to fifteen dollars as compared to $6,500 for the nuclear gauge.

Also, the use of a microwave oven would shorten drying times to about ten minutes, thus improving testing efficiency tremendously, and the investment required would be between $500 and $2000. It has been reported that the microwave oven has no adverse effect on drying soil constants and extraction tests, although there may be more radiation danger than with the nuclear gauge (20).

To say the least, the dawn of nuclear methods and other new testing innovations have presented the highway engineer with a challenge to improve testing efficiency and thus the end product itself.

# CHAPTER 11

## CONCLUSIONS

Based on the laboratory and field investigations and the engineering studies which are described in preceding chapters of this report, the following conclusions are warranted.

1. Nuclear gauge operating procedure:

   a) A clear space of at least three feet should be maintained around the gauge during testing, and the gauge should be operated on the same table or stand on which it is calibrated.

   b) During testing, the temperature of the asphalt mixture should be in the range $265^{\circ}$ F $\pm$ $35^{\circ}$ F in order to minimize the effects of temperature on instrument performance. Either a regression equation incorporating a temperature variable, or a separate calibration curve for the desired sample temperature below this range should be developed to obtain satisfactory testing precision.

c) The effects of power line voltage fluctuations, dust, humidity, and ambient temperature in a field operating environment should be checked by taking gauge standard counts over a period of at least one half day, or until the user is satisfied that these effects are non-existent. During the field operations, lasting approximately two weeks, these factors were found to affect testing precision tremendously and precluded operating the gauge outside a dry, clean environment. Modifications to the instrument made by the manufacturer in June, 1971, adequately eliminated these problems when the instrument was operated in a typical air conditioned field laboratory located adjacent to an asphalt mixing plant.

2. Calibration considerations:

   a) Care should be exercised in preparing materials, and in controlling the <u>volume</u> as well as the weight of laboratory specimens in order to obtain the best possible calibration of the nuclear gauge. The largest weight of material which can be compacted level with the top of the sample pan produces a calibration curve with the greatest slope. The same weight and volume of material used in calibration must be used in subsequent measurements of asphalt or moisture content in the field.

   b) The highest precision feasible may be obtained by calibrating the gauge for individual aggregate-asphalt mix combinations. Standard errors obtained from 4000 and 6000 gram-full pan samples of HMAC ranged from $\pm$ 0.05% to $\pm$ 0.20% for nominal asphalt contents between 3% and 10%. Based on count stability tests previously discussed, a precision no better than ± 0.03%, 68% of the time, or ± 0.06%, 95% of the time can be expected when averaging four 1-position counts on a typical full pan sample of hot mix.

c) By expressing asphalt content as percent of absolute volume, it was found that a single calibration curve could be developed for full pan samples with a standard error of ± 0.30% asphalt for the mixes evaluated. In this study, absolute volume was based on the bulk volume of aggregate particles as determined by a technique which compensates for water absorption during the determination of specific gravity of the aggregate. Asphalt content was then computed as a percent of the total bulk volume (asphalt plus aggregate). The aggregates used included limestone, siliceous gravel, and lightweight, and one asphalt grade was used.

d) Regardless of the calibration method used, a change in asphalt source requires recalibration of the gauge.

e) The use of two known asphalt contents which are molded no more than $\pm$ 1% from the expected value to be measured, rather than one asphalt specimen molded at optimum and one at 0% asphalt, is recommended as a quick and reasonably accurate method for checking calibration in the field. For the sake of speed, calibration could be initially based on two known asphalt contents molded in the field, and these same two points duplicated on a day to day basis until enough data was obtained to refine the calibration curve slope. This data would also reflect the variation which could be expected due to changes in asphalt obtained from the same source and aggregate from the same producer. It is estimated, based on experience gained in this study, that this method of developing calibration will result in testing precisions comparable to that obtained from using five different known asphalt contents as done in Chapter 5.

3. Specimen size considerations:

   a) Standard two inch high by four inch diameter cylindrical specimens of asphalt mix molded in a gyratory shear press may be used for calibrating the instrument and for controlling asphalt content at the mixing plant. The precision of measurement is much less than that obtained when using full pan specimens, and the control process is slowed by the time required for molding the small specimens.

   b) Special means for controlling the position of the specimen in the gauge drawer must be provided.

4. Effect of moisture on asphalt content measurements:

   a) Field studies of asphalt content in a limestone HMAC mixture indicated that the precision of measurement was much better when compensation was made for moisture in the aggregate. Standard deviations of ± 0.11% asphalt were determined by nuclear measurements (using a laboratory developed curve for the limestone mix and ± 0.25% by extraction tests on the same mix in the field laboratory. The plant asphalt content during this time was controlled by scale weight at 6.2%. Hot bin moisture contents were less than 0.25% and averaged about 0.14% during the same period.

b) The nuclear gauge responds to moisture differently than to asphalt. For the materials studied, and a 6000 gram-full pan sample, regression studies of laboratory data showed that 0.1% asphalt was equivalent to about 0.11% moisture. Asphalt content was expressed as a percent of the total weight of the mixture, and moisture content as a percent of the dry weight of aggregate. This equivalency would be expected to change with any change in asphalt source.

5. Moisture measurement in soils and base materials:

   a) Laboratory studies of moisture in subgrade soils indicated that individual calibration curves should be developed for each type of soil in order to obtain reasonable precision.

   b) Laboratory studies of moisture in untreated base materials indicated that one common calibration curve could be used for coarse limestone base materials with a standard error of $\pm$ 0.16%. Moisture contents in this study ranged from 6% to 11%.

6. There are several laboratory and field operations in which the nuclear gauge may be used practically. Optional uses include:

   a) Determination of moisture in compaction test specimens.

b) Use in conjunction with the volumeter for field moisture tests.

c) Concurrent control from a centralized field laboratory of moisture in soils (base and/or subgrade materials) and control of asphalt in hot mix surface coarses or in "black base".

7. In terms of initial investment, the cost of the nuclear gauge is relatively high ($6,500); however, its various optional uses (both in standard testing and in research) and the increased precision and speed (10 to 30 minutes per test) with which tests can be obtained, make the investment attractive, especially when spread over a long period of time.

8. Precision, speed, and safety of the nuclear method were found to be superior to the asphalt extraction method presently used by the Texas Highway Department for field control of asphalt content at a mixing plant.

## APPENDIX A

### Suggested Calibration and Test Procedure

I. Calibration Procedure

A. Make the decision as to how asphalt contents will be expressed.

1. Percent of total weight
2. Percent of absolute volume

B. It is assumed that percent of total weight is chosen since this produces the most accurate results.

C. Procedure

1. A gradation of proposed materials is obtained and a mix combination is designed in accordance with instructions in Texas Highway Department Bulletin C-14.
2. The total sample weight used in calibration varies depending on the type of aggregate used. Generally, the largest weight which can be compacted level with the top of the pan is the most desirable. For normal aggregates this weight varies between 6,000 and 7,000 grams and for lightweight aggregates, about 4,000 to 5,000 grams. The weight chosen should be held constant throughout calibration and control testing within limits of ± 25 grams.
3. Depending on the mixing equipment available, the samples may be weighed and mixed in 1/2 batch or full batch quantities.

4. Choose from 2 to 5 asphalt contents within $\pm$ 2% of optimum asphalt content of the material used, and mix single or duplicate samples, depending on the degree of precision desired, at each asphalt content. An alternate method would be to choose 2 asphalt contents, one 1% above and one 1% below optimum, and mold several duplicate samples at each point.
5. In this report 1/2 batches were weighed and mixed at 5 asphalt contents.
6. Prior to adding the asphalt planned for use in construction, dry the combined mix samples in a 200° F oven to constant weight.
7. After drying, bring the samples to a temperature of about 260° F to 300° F, then add asphalt.
8. All weights should be checked hot to compensate for convection currents. Any discrepancy should be corrected by adding or removing a few grams of sand or other fines.
9. Add the asphalt when it is about 300° F.
10. Mix the sample to achieve uniform coating of the aggregate, then return the sample to the oven and bring to 260° F.
11. The gauge sample pan should be preheated and the press plate lightly oiled to prevent asphalt from sticking. A small rammer should also be preheated in case it is required to correct sample finishing.
12. The sample pan is placed in the molding block, the sample removed from the oven and placed in the pan in about 1/2 batch increments.
13. Lightly rod the sample with a trowel as it is placed in the pan and try to control segregation by eye.
14. The top of the sample should be troweled fairly level and smooth prior to placing in the press.

15. Place the molding block with the pan in the press and lower the press plate to a predetermined height, usually flush with the top of the pan.
16. Remove the sample pan from the molding block and correct any surface irregularities with the small rammer.
17. Warm up the nuclear asphalt gauge for at least 30 minutes prior to testing, and after this time obtain enough standard counts to insure stable operation.
18. Place the pan with sample in the gauge and obtain four 1-position counts. Other counting positions on the gauge may be used if desired.
19. During this operation, the temperature of the mix should be between 240°and 260° F.
20. Plot the count rate obtained against corresponding asphalt content as determined by scale weight, and develop the calibration curve using other points. Calculate the slope of the curve.
21. Maintain a clear space of at least three feet around the gauge to minimize environmental effects, and use the same sample pan for all calibration points and field control.
22. Develop a moisture calibration curve using the same general procedure, sample weight and volume with the following exceptions:

    a) Do not heat the mix for testing
    b) Add no asphalt, only moisture
    c) Do not heat sample pan
    d) Correlate count rate with moisture as determined by percent of oven dry weight. Calculate the slope of the curve.
    e) Divide the slope of the moisture curve by the slope of the asphalt curve to obtain the moisture coefficient in equation (4).

II. Field Control Testing

A. Obtain standard counts to insure stable instrument operation.

B. Determine moisture in hot bin aggregates by oven drying.

C. Obtain a sample of hot mix from a haul truck and using the same calibration conditions (weight, volume, temperature, and counting procedure) obtain a nuclear count rate for the sample.

D. Use the calibration curve or equation to determine asphalt content.

E. Reduce the asphalt content by the amount of moisture in the hot bin aggregates times the ratio of the slope of moisture to asphalt calibration curves (as determined in C-22-e above). This subtraction process yields the net asphalt content of the mix.

## BIBLIOGRAPHY


1. Bingham, J. M., White, J. R., "District Evaluation of Nuclear Type Moisture-Density Instruments," Research Report No. 37-2F, Texas Highway Department, February, 1966.

2. Lamb, D. R. and Zoller, J. H., "Determination of Asphalt Content of Bituminous Mixtures by Means of Radioactive Isotopes," HRB Proceedings, Vol. 35, pp. 322-326, 1956.

3. Varma, Man Mohan and Reid, G. W., "Determination of Asphalt Contents in a Paving Mixture by Thermal Neutrons," Highway Research Record No. 66, pp. 73-83, 1963.

4. Howard, P. K. and Covault, D. O. "Use of Nuclear Methods to Measure Mineral Filler Content and Asphaltic Content of Bituminous Concrete," Highway Research Record No. 66, pp. 84-85, 1963.

5. Walters, H. W., "Nuclear Asphalt Content Determination at the Job Site," Highway Research Record No. 117, pp. 54-66, 1966.

6. Qureshi, Tajamal Hussain, "Discussion - Nuclear Asphalt Content Determination," Highway Research Record No. 117, pp. 66-70, 1966.

7. Metcalfe, T. B., Averitt, W. K., and Larue, F. L., "Asphalt Content by Nuclear Devices," University of Southwestern Louisiana, Department of Chemical Engineering Research Bulletin, 1967.

8. Grey, R. L., "Determination of Asphalt Content in Hot Bituminous Mixes With a Portable Nuclear Asphalt Content Gage," Highway Research Record No. 248, pp. 77-81, 1968.

9. Grey, R. L., "Asphalt Content Studies by the Nuclear Method," Final Report for Phase II, Pennsylvania Department of Highways Research Project No. 64-15, May, 1970.

10. Grey, R. L., "Evaluation of Bituminous Compaction Procedures Using Nuclear Gauges," Final Report for Phase II, Pennsylvania Department of Highways Research Project No. 64-15, March, 1970.

11. Inghram, D., "Nuclear Test Equipment Investigation-Troxler Nuclear Asphalt Content Gage," Progress Report, Nebraska Department of Roads, Research Study 64-5, January, 1968. Correspondence Included.

12. Hughs, C. S., "Evaluation of a Nuclear Asphalt Content Gauge," Virginia Highway Research Council (Department of Highways and University of Virginia) VHRC 70-R26, December, 1970.

13. Hicks, C. R., "Fundamental Concepts in the Design of Experiments," Holt, Rinehart and Winston, Inc., 1964.

14. "Construction Bulletin C-14," Texas Highway Department, Construction Division, September, 1964.

15. "Manual of Testing Procedures, Volume 1," Texas Highway Department.

16. "Use of a Nuclear Moisture-Density Gauge in District 14, Texas Highway Department," unpublished data and studies compiled by James F. Todd, 1965 thru 1971.

17. Bryant, J. S., "Determination of the Moisture Absorption Characteristics of Lightweight Aggregate," M.S. Thesis, Texas A & M University, January, 1959.

18. Hargett, E. R., Gallaway, B. M., and Scott, W. W., Jr., "Synthetic Aggregates for Asphaltic Concrete Mixes," Research Report No. 110-1F, Texas Transportation Institute, 1969.

19. Zeigler, Carol D., "Design and Construction of Asphaltic Concrete Pavement Using Volumetric Design Specifications," Departmental Research Report Number SS 15.2, Texas Highway Department, January, 1971.

20. "Use of a Microwave Oven for Rapid Drying of Aggregate Samples," State Department of Highways, Division of Highways - State of Colorado, October, 1969.