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Precision Estimates of Selected Volumetric Properties of HMA Using Non-Absorptive Aggregate

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- New York State Department of Transportation, Albany, New York
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SUMMARY

The Superpave system for the laboratory design of Hot Mixed Asphalt (HMA) requires the use of Superpave Gyratory Compactors for the compaction of specimens. The procedure used for this purpose is included in AASHTO T312, Standard Method of Test for Preparing and Determining the Density of Hot-Mix Asphalt (HMA) Specimens by Means of the Superpave Gyratory Compactor. This study was undertaken with the objective of developing a precision statement applicable to AASHTO T312 and updating the published precision statements for AASHTO T166, Standard Method of Test for Bulk Specific Gravity of Compacted Asphalt Mixtures Using Saturated Surface-Dry Specimens, and ASTM D2041-01, Standard Test Method for Theoretical Maximum Specific Gravity and Density of Bituminous Mixtures.

Another objective was to prepare first-cut precision estimates for ASTM provisional standards ASTM PS131, Standard Method of Test for Bulk Specific Gravity and Density of Compacted Mixtures Using Automatic Vacuum Sealing Method, and ASTM PS132, Standard Method of Test for Maximum Specific Gravity and Density of Bituminous Paving Mixtures Using Automatic Vacuum Sealing Method.

The experimental plan for the study selected an aggregate source that produced relatively uniform non-absorptive limestone aggregate. The binder for the study was a PG 64-22 meeting the requirements of AASHTO MP1, Standard Specification for Performance Graded Asphalt Binder. Twenty-seven laboratories (including AMRL) volunteered to participate in an interlaboratory program within the scope of the study. The plan required determining the test precision applicable to the coarse and fine mixes. A maximum aggregate size of 19.0-mm was used for the coarse mix and 12.5-mm for the fine mix.

The test samples were prepared by AMRL staff at the AMRL facility located at the National Institute of Standards and Technology (NIST). This involved determination of the design binder content for the two mixes in accordance with the requirements of AASHTO MP2, Standard Specification for Superpave Volumetric Mix Design and AASHTO PP28, Standard Practice for Superpave Volumetric Design for Hot-Mix Asphalt (HMA). With the design binder contents known, loose mix samples were then prepared according to PP28, and distributed to the participating laboratories for interlaboratory testing. The laboratories were provided with instructions to test the samples and test data collection forms. The test results from this study and existing data from the AMRL Proficiency Sample Program were statistically analyzed and precision estimates prepared.

The study resulted in proposed precision statements in a format for consideration by standards committees for D2041, T166 (Method A), and T312. It is recommended that AASHTO consider adopting D2041 as a replacement for T209, Standard Method of Test for Theoretical Maximum Specific Gravity and Density of Bituminous Paving Materials. The G_{mb} values obtained using T166 from specimens compacted with the Pine AFGC125X compactor were greater than those obtained from specimens compacted with the Troxler 4140 compactor. The relative density values determined using T312 from specimens compacted with the Pine AFGC125X compactor with the Troxler 4140 compactor. Although the range in air voids in specimens tested in this study were

limited, it may be appropriate to include the precision estimates proposed for T312 which are applicable to Superpave specimens in T269.

Precision estimates for PS131 and PS132 were prepared based on the results from this study but they are not recommended for adoption into standards until interlaboratory studies involving laboratories having more experience in performing the tests are conducted.

CHAPTER 1

INTRODUCTION AND RESEARCH APPROACH

1.1 INTRODUCTION

At the conclusion of research on asphalt in 1993, the Strategic Highway Research Program (SHRP), delivered as a final product, "The Superpave System TM." The system proposed methods of design of dense graded Hot Mixed Asphalts (HMA) for new pavements and overlays $(1)^1$. The system takes into account the traffic loading, environmental conditions, materials characteristics, and the level of performance expected of the HMA in the real world of service conditions. The design system established criteria for selecting, proportioning and combining the materials, densification or compaction requirements, and the volumetric properties of the mix.

As these new Superpave design procedures became available, the American Association of State Highway and Transportation Officials (AASHTO) Subcommittee on Materials (SOM) transformed them into national standards and published them as 'Provisional Standards'' (2). This step was taken to accomplish three objectives. First, maintain uniformity in the use of design procedures on a national basis. Second, make the standards available in a single volume, i.e., provide one-stop shopping. Third, provide a means for getting input from the practicing engineers and researchers for the improvement of the standards on a real-time basis.

The Provisional Standards continue to meet these objectives. Because of their application at the national level, the Superpave protocols have received extensive feedback from users and researchers. Since their first publication in 1993, most of these protocols have been revised and published on a yearly basis with the latest edition dated May 2002.

1.1.1 Problem Statement

Significant changes and improvements have occurred and continue to occur in the Superpave design procedures since the system was first introduced. One serious deficiency, which is widely acknowledged, relates to the absence of precision statements in most of the standards.

The Superpave mix design method relies on the volumetric properties of HMA at specified compaction levels. The desired compaction levels in the laboratory mix design are achieved by the use of gyratory compactors. The design computations and the volumetric properties for laboratory compacted specimens require determination of bulk and maximum specific gravities. There is concern about the validity of the precision statements in standards used to determine these properties. AASHTO T312-01, Standard Test Method for Preparing and Determining the Density of Hot-Mix Asphalt (HMA) Specimens by Means of the Superpave Gyratory Compactor (*3*) does not have a precision statement. ASTM D2041-00, Standard Test Method for Theoretical Maximum Specific Gravity and Density of Bituminous Mixtures (*4*) has

¹ Numbers in parentheses refer to the bibliography.

a precision statement but it may be obsolete because of many changes made to the standard. The precision statement for AASHTO T166-00, Standard Method of Test for Bulk Specific Gravity of Compacted Asphalt Mixtures Using Saturated Surface-Dry Specimens (*3*) may be outdated.

1.1.2 Research Objectives

This phase of Project 9-26 had the following objectives:

- (a) Develop a precision statement applicable to T312. (AASHTO Provisional Standard TP4-00 was the predecessor to T312. TP4-00 was used to test the 12.5-mm samples and T312 was used to test the 19.0-mm samples.)
- (b) Update precision statements currently published in T166 and D2041.
- (c) Develop precision estimates for ASTM PS131-01, Bulk Specific Gravity and Density of Compacted Bituminous Mixtures Using Automatic Vacuum Sealing Method (4); and PS132-01, Maximum Specific Gravity and Density of Bituminous Paving Mixtures Using Automatic Vacuum Sealing Method (4).
- (d) Compare the precision estimates obtained from this study to estimates obtained from data collected in the Proficiency Sample Program operated by the AASHTO Materials Reference Laboratory (5).
- (e) If possible, evaluate differences among models of Superpave gyratory compactors (SGCs).

1.2 SCOPE OF STUDY

This work was limited to the development of precision statements for the standards which provide information on the density or percent compaction of HMA made with non-absorptive aggregates. The following conditions limited the scope of the study:

- (a) Use materials that conform to the Superpave mix specifications.
- (b) Use only one source of relatively uniform non-absorptive aggregate. Use a 19.0-mm coarse gradation and a 12.5-mm fine gradation with Superpave upper and lower gradation bands as specified in MP2-00, Standard Specification for Superpave Volumetric Mix Design (2).
- (c) Use a single performance grade neat binder PG 64-22 from a single source.

Specific tasks included in the study were as follows.

Task 1 –Selection of Laboratories

Task 2 –Selection of Materials and Mix Designs

- Task 3 Preparation of Loose Mix Samples
 - Task 4 Preparation of Instructions for Participants
 - Task 5 Analysis of Data from Participants
 - Task 6 Preparation of Revisions to the Standards
 - Task 7 Preparation of a Final Report

CHAPTER 2

EXPERIMENTAL PLAN

2.1 OVERALL PLAN

The development of precision statements as outlined in Section 1.1.2 required participation of a number of laboratories in an interlaboratory study. The approach used for the development of such a program was based on ASTM E691-99, Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method (6). The absolute minimum number of laboratories required for the development of a precision statement is specified as six in E691 with a preferred minimum of 30.

The study involved two mix designs, one having a 12.5-mm fine gradation and the other a 19.0-mm coarse gradation. For each mix design, each participant was asked to determine the theoretical maximum specific gravity of three approximately 2300 g loose mix test specimens by D2041, and the maximum specific gravity of the same three loose mix test specimens according to PS132 using the automatic vacuum sealing method. In addition, for each mix design, each participant was asked to compact three approximately 5000 g loose mix specimens according to T312, determine the bulk specific gravity of the same compacted specimens as described in PS131 using the automatic vacuum sealing method, and calculate the relative density at N_{ini} and N_{des} as described in T312.

2.2 SELECTION OF LABORATORIES

The criteria developed for the selection of laboratories considered to be good candidates for this study were:

- 1. Its participation must be voluntary with no cost to the study.
- 2. The selection will be made from a mix of the State DOT and private sector laboratories.
- 3. The participants will agree to comply and strictly adhere to the requirements of the standards in question and the supplementary instructions and data sheets provided by the AMRL.
- 4. Preference will be given to laboratories that have all the equipment and accessories needed for the completion of the tests included in the study.
- 5. Preference will be given to laboratories that participated in the AMRL HMA Proficiency Sample Program which included TP4 or T312 for the past four years.
- 6. Preference will be given to laboratories accredited by the AASHTO Accreditation Program (5,7).

The most difficult criterion for the selection of laboratories was that they have the capability of performing all the tests included in the study. Three of the tests, D2041, PS131, and PS132, required new equipment not available in many laboratories. Accordingly, it was

expected that it might be necessary to accept laboratories that were not able to perform the full suite of tests.

All 50 central State DOT laboratories were invited to participate. Many expressed an interest, but were not equipped to perform all the tests. All laboratories that were properly equipped and expressed an interest were selected. It was more difficult to obtain non-DOT laboratories to participate. The NCHRP project panel assisted by identifying potential participants and a call for laboratories was included in the newsletter of National Asphalt Pavement Association.

The 27 laboratories participating included 20 State DOTs, 1 research laboratory, 4 private sector laboratories, AMRL, and a FHWA laboratory. These laboratories had the following characteristics relative to their recognition by the AASHTO Accreditation Program.

- 24 were accredited for HMA.
- 23 were accredited for T166 and T312
- 24 were accredited for D2041 and AASHTO T209, Standard Method of Test for Theoretical Maximum Specific Gravity and Density of Bituminous Paving Mixtures (3).

2.3 SELECTION OF MATERIALS

2.3.1 Aggregates

The crushed limestone aggregate selected for the study came from a relatively uniform geologic formation of limestone in the Lafarge Stone Quarry located in Frederick, Maryland. The aggregate is being used in the on-going NCHRP Study 9-19, Superpave Support and Models Management. Additionally, the State of Maryland has used the stone extensively in several of its highway projects and keeps a year-to-year record of the uniformity of material coming from the quarry that is measured in terms of tested properties. According to the records, the quarry has been in operation since 1859 and it has supplied about 150 million tons of stone since beginning operation.

Typical test properties of the coarse aggregate measured in the 2001-2002 timeframe and as recorded by the Maryland State Highway Administration are given below. The test methods used to determine the properties were not provided.

Bulk Specific Gravity = 2.71

Percent Absorption = 0.3 percent

Los Angeles Abrasion (percent loss) = 19 percent

Loose Unit Weight = $87.8 \text{ pcf} (1407 \text{ kg/m}^3)$

Rodded Unit Weight = $95.7 \text{ pcf} (1533 \text{ kg/m}^3)$

Testing performed on the aggregate by AMRL yielded the following results:

(a) Coarse Aggregate

Water Absorption by AASHTO T85, Standard Method of Test for Specific Gravity and Absorption of Coarse Aggregate (3) = 0.5 - 1.0 percent

Bulk Specific Gravity by AASHTO T85 = 2.67

Effective Specific Gravity by AASHTO PP28, Standard Practice for Superpave Volumetric Design for Hot-Mix Asphalt (HMA) (2) = 2.71

(b) Fine Aggregate

Water Absorption by AASHTO T84, Standard Method of Test for Specific Gravity and Absorption of Fine Aggregates (3) = 1.0 percent

Bulk Specific Gravity by AASHTO T84 = 2.64

2.3.2 Asphalt Binder

The binder used in both mixtures was a PG 64-22 grade asphalt binder obtained from the Chevron Refinery in Perth Amboy, New Jersey. This binder is one of the most commonly used grades in the United States and it has been used successfully on numerous research projects.

2.4 TEST SAMPLES AND TEST PROTOCOLS

2.4.1 Sample Preparation

Samples were prepared by AMRL staff in the Proficiency Sample Facility located at the National Institute of Standards and Technology (NIST) using procedures developed for the AMRL HMA Proficiency Sample Program (7).

The laboratory mix formulas shown in Table 1 were used to prepare the 12.5-mm and 19.0-mm maximum specific gravity (G_{mm}) test mixtures and the Superpave gyratory (Gyr) test mixtures. Each loose mix test sample was individually prepared during one of four mix operations: 12.5-mm G_{mm} , 12.5-mm Gyr, 19.0-mm G_{mm} , and 19.0-mm Gyr. The 12.5-mm and 19.0-mm mixtures resulted in the properties shown in Table 2. Ten extra mixtures were prepared during each mix operation. For each mix operation, the masses of the loose mixtures were determined and those mixtures resulting in the smallest variation in mass were selected for distribution.

The samples were boxed and marked as shown in Figure 1. Each laboratory received one set of 12.5-mm G_{mm} samples; one set of 12.5-mm Gyr samples; one set of 19.0-mm G_{mm} samples; and one set of 19.0-mm Gyr samples. Each set contained three replicate samples chosen at random from the samples selected for distribution.

2.4.2 Test Protocols and Instruction and Data Forms for Participants

The test properties determined and the protocols followed in the study are shown in Table 3. The instruction and data forms shown in Appendix A were used by participants to test the samples and report test results. Additionally, the method of weighing (in air or in water) was reported for D2041, and the SGC manufacturer and model number were reported for T312.

2.4.3 Sample Distribution

Samples were distributed to participating laboratories using the U.S. Postal Service and Federal Express. The 12.5-mm samples were shipped to participants and tested in June 2001 and the 19.0-mm samples were shipped and tested in December 2001.

CHAPTER 3

LABORATORY TEST RESULTS AND ANALYSIS

3.1 TEST DATA

The data obtained in this study are shown in Table 4 for the 12-5mm mixture and in Table 5 for the 19.0-mm mixture. Empty cells or portions of cells indicate that the laboratory did not submit data. Shaded cells indicate data that were eliminated from analysis as described in Section 3.2.

3.2 METHODS OF ANALYSIS

Different methods of analysis were used on the interlaboratory test data collected in the study and on the data from the AMRL Proficiency Sample Program.

3.2.1 Data from This Study

Test data from this study were displayed graphically using box plots generated by Dataplot (8) which was developed at NIST and is a free, public-domain, multi-platform software system for scientific visualization, statistical analysis, and non-linear modeling (See Figures 2, 7, and 12). The box plot is a graphical data analysis technique for determining if differences exist between the various levels of a 1-factor model (9). The box plot is a graphical alternative to a 1-factor ANOVA. It is also a useful technique for summarizing and comparing data from two or more samples. A box plot is structured in the following manner. The bottom x is the data minimum and the top x is the data maximum. The bottom of the box is the estimated 25 percent point and the top of the box is estimated 75 percent point. The middle x in the box is the data median.

In addition to eliminating any partial sets of data, data were eliminated from analysis by following the procedures described in E691 in determining repeatability (S_r) and reproducibility (S_R) estimates of precision. Data exceeding critical *h* and *k* values were eliminated as described in Sections 3.3, 3.4, and 3.5. Once identified for elimination, the same data were eliminated from any smaller subsets analyzed.

3.2.2 AMRL Proficiency Sample Data

AMRL operates proficiency sample programs for a number of construction materials. (5,7) AMRL gyratory proficiency sample pairs 7/8, 9/10, and 11/12 were the most recent gyratory proficiency samples available at the time of this study. Sample pair 7/8 was tested in 1999; sample pair 9/10 was tested in 2000, and sample pair 11/12 was tested in 2001. The test samples comprising all three sample pairs were mixed and compacted by 200 to 300 individual participants using materials and a job mix formula supplied by AMRL. (In the NCHRP study

the participants were provided with premixed samples ready for compaction.) All AMRL proficiency samples had a 12.5-mm maximum aggregate size. For AMRL proficiency sample pair 7/8, a single butter batch was split into two samples, and each sample was tested to determine the maximum specific gravity for sample pair 7/8. Two separate butter batches were used to determine the maximum specific gravity for AMRL proficiency sample pairs 9/10 and 11/12. Proficiency sample participants were asked to determine the bulk specific gravity and relative density in a manner similar to this study. The test results from sample pairs 7/8 and 9/10 permitted valid estimates of within laboratory variability. However, the bulk density data from sample pair 11/12 indicated that the samples were too dissimilar to permit valid estimates of within laboratory precision.

A bivariate normal tolerance region containing approximately 85% of the data with 95% confidence as described in a paper by Hall (10) was applied to the data from the AMRL proficiency sample pairs described above. Only data falling within the bivariate tolerance region for each pair were analyzed. Appendix B describes this approach and the rationale for screening AMRL proficiency sample data in this manner.

Once screened, the proficiency sample data were analyzed in the manner described in Appendix C to determine S_r and S_R precision estimates.

3.2.3 Tests for Statistical Significance

Tests for statistical significance on both data collected in this study and the AMRL proficiency sample data were performed using the "*T*-test" and "*F*-test" functions in Microsoft Excel. All *T*-tests assumed two samples with unequal variance and a one-tailed T distribution. For data in this study, *F*-tests, to determine if S_r estimates of precision were statistically different, were performed on the variances calculated from the three replicate determinations. For AMRL proficiency sample data, *F*-tests, to determine if S_r estimates of precision were statistically different, were performed on the variances calculated for the paired test results, taking into account any actual differences in the two samples comprising the sample pair.

3.3 THEORETICAL MAXIMUM SPECIFIC GRAVITY, G_{mm}

3.3.1 Introduction

The theoretical maximum specific gravity (G_{mm}) is a fundamental property of bituminous paving mixtures whose value is influenced by the composition of the mixture in terms of types and amounts of aggregate and bituminous materials. The G_{mm} is used in the calculation of air voids in compacted bituminous paving mixtures and the amount of asphalt binder bitumen absorbed by the aggregate. The G_{mm} also provides a target value for the compaction of paving mixtures.

The conventional method for determining the G_{mm} involves weighing a sample of loose paving mixture, placing it in a tared vacuum vessel (a bowl or flask), and adding sufficient water

at 25°C to cover it. Partial vacuum is applied to reduce the residual pressure in the vacuum vessel to 4 kPa or less, held for 15 ± 2 minutes, and then gradually released. The volume of the sample of paving mixture is obtained either by immersing the vacuum vessel in a water bath and weighing (weight in water), or by filling the vacuum vessel level full of water and weighing (weight in air). The G_{mm} is calculated from these mass and volume measurements. Possible sources of test variation when weighing in air include entrapping air bubbles under the lid and inadequate drying of the outside of the vacuum vessel. When making weighings in water, 'floaters' in the bowl could be lost when the bowl is immersed causing errors in the test result. Errors for either method could also result from variations in temperature and pressure.

Study participants were asked to determine the G_{mm} of three replicate 12.5-mm mixtures and three replicate 19.0-mm mixtures according to D2041-00. This test procedure differs significantly from D2041-95 and T209-99 by requiring continuous agitation, a larger test specimen, a constant partial vacuum (3.7 ± 0.3 kPa), a reduction in the number of allowable containers, and a procedure for placing the lid on the pycnometer.

After determining G_{mm} by D2041, the participants were asked to oven dry and retest each specimen and determine G_{mm} using PS132. This method for determining the G_{mm} involves placing a weighed oven-dry sample of loose paving mixture in a specially designed bag. The bag containing the sample is then placed inside another bag and placed inside a vacuum chamber. The sample is evacuated for approximately 1 minute to 4 kPa and automatically sealed. The bags containing the sample are removed and placed underwater in a large water tank. While completely submerged, the bag is cut open to allow water to enter the bag, and the submerged sample is weighed. The submerged weight is corrected for the influence of the bags to determine the sample volume. The dry mass and the volume are used to calculate G_{mm} .

3.3.2 Precision Estimates

3.3.2.1 D2041 Test Data

Twenty-six laboratories submitted full sets of G_{mm} data based on D2041 for the 12.5-mm and 19.0-mm mixtures (See Tables 4 and 5 –Column 3). The data are displayed on box plots in Figures 2a and 2c. The data from laboratory 11 were eliminated from the 12.5-mm mixture analysis, and the data from laboratories 5 and 22 were eliminated from the 19.0-mm mixture analysis based on *h*- and *k*-statistics (See Figures 3 and 4). All remaining data were re-analyzed with E691 software to determine the S_r and S_R precision estimates shown in Table 6. The 12.5mm and 19.0-mm mixture data remaining after the removal of outliers were separated into four data sets by method of weighing, and each of the four data sets was analyzed to determine the S_r and S_R precision estimates shown in Table 6.

3.3.2.2 PS132 Test Data

Twenty-one laboratories submitted full sets of G_{mm} data using the vacuum sealing method for the 12.5-mm mixture, and twenty-four laboratories submitted full sets of G_{mm} data

for the 19.0-mm mixtures (See Tables 4 and 5, Column 4). The data are displayed in Figures 2b and 2d. The data from laboratories 10 and 19 were eliminated from the 12.5-mm mixture analysis and the data from laboratory 22 were eliminated from the 19.0-mm mixture analysis based on *h*- and *k*-statistics (See Figures 5 and 6). All remaining data were re-analyzed using E691 software to determine the S_r and S_R precision estimates shown in Table 6.

3.3.2.3 AMRL Proficiency Sample Test Data

For AMRL gyratory proficiency sample pairs 9/10 and 11/12, data were analyzed as described in Appendices B and C to determine the S_r and S_R precision estimates shown in Table 6.

3.3.3 Tests for Significance

A comparison of D2041 G_{mm} test results from laboratories using the "weight in water" method and the "weight in air" method indicates that there is very little bias in test results from the two methods and that the S_r estimates (0.002) shown in Table 6 are the same. However, the results of the *F*-tests shown in Table 7, Column 4, Rows 3 and 4, indicate a statistically significant difference in the S_R estimates for the "weight in air" method (0.005 and 0.004) and the S_R estimates for the "weight in water" method (0.002 and 0.003). Consideration was given to making separate S_R estimates for the "weight in water" and "weight in air" methods in the proposed precision statement for D2041-00 noting a possible difference in multilaboratory precision when using the two methods. Unfortunately, AMRL gyratory proficiency sample data were not available to evaluate these test conditions separately; therefore, separate precision estimates are not presented (See Section 3.3.4.1). AMRL hopes to have proficiency sample data which will support a separate analysis of the two methods of test in the near future. If necessary, the precision statement can be revised.

The results of the *T*-test comparing the 12.5-mm, D2041 data to the 12.5-mm PS132 data indicate a statistically significant difference in the average G_{mm} values shown in Table 6 (2.550 vs. 2.542 respectively) at a 99 percent confidence level (See Table 7). Although not significant at a 99 percent confidence level, similar comparison of the 19.0-mm data did indicate a statistically significant difference in the average values at a 95 percent confidence level (2.562 vs. 2.557 respectively). This bias is somewhat evident in the box plots in Figure 2 and suggests that G_{mm} values obtained using PS132 may be lower than those obtained using D2041.

The results of F-tests shown in Table 7 indicate that the S_r and S_R precision estimates of G_{mm} data obtained using PS132 are statistically greater than the S_r and S_R precision estimates of G_{mm} data obtained using D2041. The greater variation in PS132 test data is clearly evident in Figure 2.

3.3.4 Precision Statements

3.3.4.1 D2041

Table 6 shows there is good agreement in the variability between the 12.5-mm and 19.0mm mixtures for the D2041 G_{mm} data obtained from this study. Appendix D shows a proposed revised precision statement for D2041-00 which includes precision estimates, for both size mixtures, of $S_r = 0.002$ and $S_R = 0.004$. Although tighter than the precision estimates currently found in D2041-00 as shown in Table 6, the proposed estimates seem to be suitable given the improvement made to D2041 and the fact that, through an oversight, the 2300 g, 19.0-mm specimens distributed to laboratories did not meet the 2500 g minimum sample mass requirements specified in D2041.

The precision estimates shown in Table 6 which resulted from the analysis of the 12.5mm, AMRL gyratory proficiency samples, involving over 200 laboratories, are a little higher, but compare favorably to the D2041 precision estimates from this study. Analysis of sample pair 9/10 yielded an S_r estimate of 0.003 and an S_R estimate of 0.006, while sample pair 11/12 resulted in an S_r estimate of 0.003 and an S_R estimate of 0.005. Laboratories testing the AMRL proficiency samples determined G_{mm} according to D2041-95 and T209-99 which were earlier versions of standards used in this study.

Based on the results of this study and the analysis of AMRL proficiency sample data, the precision estimates currently published in D2041 appear to be high and in need of revision. The precision estimates published in T209 look more appropriate than those published in D2041, but may need updating.

3.3.4.2 PS132

The G_{mm} precision estimates shown in Table 6 which resulted from the analysis of the 12.5-mm and 19.0-mm PS132 test results are greater than those obtained from the analysis of both D2041 data reported for this study and AMRL proficiency data. The increase in variability of test results obtained using PS132 may be due to the inexperience of the participants performing the test. Sixteen of the participants reported that they performed the test for the first time on the 12.5-mm mixtures, and reported very little additional experience when performing the test on the 19.0-mm mixture. Only two of the participants reported performing the test 25 times or more. The increased variation in PS132 G_{mm} values may indicate the test procedure needs adjustment. Some possible problems are the bags touching the sides of the bath during weighing, an incomplete evacuation of air during the vacuum sealing process, and compaction of the specimen during the vacuum sealing process.

3.4 BULK SPECIFIC GRAVITY, G_{mb}

3.4.1 Introduction

The proper measurement of bulk specific gravity (G_{mb}) of compacted HMA mixes is a major concern of the HMA industry. The G_{mb} of compacted asphalt mixtures is required for making volumetric calculations used during mixture design, field control, and construction acceptance (12). Volumetric properties such as air voids, voids in mineral aggregate, voids filled with asphalt, and percent maximum density at a certain number of gyrations are based on G_{mb} .

Study participants were asked to compact the as-received loose mixtures weighing approximately 4900 g according to T312 and determine the G_{mb} of three replicate 12.5-mm specimens and three replicate 19.0-mm specimens according to PS131. This method for determining G_{mb} involves placing a weighed sample in a specially designed bag. The bag containing the sample is then placed inside a vacuum chamber. The air in the bag containing the specimen is evacuated and the bag is automatically sealed. The mass of the sealed bag containing the specimen is determined. The sealed bag containing the specimen is immersed in water at 25°C and weighed. The G_{mb} is calculated from the resulting mass determinations, the immersed weight, and the apparent specific gravity of the plastic bag.

After determining G_{mb} by PS131, participants were asked to retest each specimen and determine G_{mb} according to T166, Method A. Method A of T166 involves determining the mass of an air-dried specimen (in this case a 150-mm diameter specimen compacted using a Superpave gyratory compactor), immersing the specimen in a water bath at 25°C, recording the weight after 3 to 5 minutes, removing the specimen, blotting it quickly with a damp cloth towel, and determining the saturated surface dry (SSD) mass in air. These mass and volume measurements are used to calculate G_{mb} . AMRL observes substantial variation in the techniques used to obtain the SSD condition of the specimen during laboratory assessments. Some reasons for this variation may be the difference in dampness of the towel used to blot the surface of the specimen, differences in temperature of the immersion bath, and differences in interpretations in achieving a SSD condition as quickly as possible.

3.4.2 Precision Estimates

3.4.2.1 T166 Test Data

Twenty-six laboratories submitted full sets of T166 data for the 12.5-mm and 19.0-mm mixtures (See Tables 4 and 5, Column 6). The data are displayed in Figures 7a and 7c. All data were analyzed using the E691 software. The data from laboratory 16 were eliminated from the 12.5-mm mixture analysis and the data from laboratories 4 and 22 were eliminated from the 19.0-mm mixture analysis based on *h*- and *k*-statistics (See Figures 8 and 9). All remaining data were re-analyzed with the E691 software to determine the S_r and S_R precision estimates shown in Table 8. The 12.5-mm and 19.0-mm mixture data sets was analyzed with the E691 software to determine the S_r and S_R precision estimates shown in Compaction. Each of the four data sets was analyzed with the E691 software to determine the S_r and S_R precision estimates shown in Table 8.

3.4.2.2 PS131 Test Data

Twenty-two laboratories submitted full sets of PS131 G_{mb} data for the 12.5-mm mixture and 19.0-mm mixtures (See Tables 4 and 5, Column 5). The data are displayed in box plot form in Figures 7b and 7d. All data were analyzed using E691 software. Data from laboratories 4 and 16 were eliminated from the 12.5-mm mixture analysis, and data from laboratories 22 and 25 were eliminated from the 19.0-mm mixture analysis based on *h*- and *k*-statistics (See Figures 10 and 11). All remaining data were re-analyzed with the E691 software to determine the S_r and S_R precision estimates shown in Table 8. The 12.5-mm and 19.0-mm mixture data remaining after the removal of outliers were separated into four data sets based on the mixture type, and compactor manufacturer and model used in compaction. Each of the four data sets was analyzed with the E691 software to determine the S_r and S_R precision estimates shown in Table 8.

3.4.2.3 AMRL Proficiency Sample Data

For comparison purposes, S_r and S_R precision estimates were determined for G_{mb} data from AMRL gyratory proficiency sample pairs 7/8, 9/10, and 11/12. Data were analyzed as described in Appendices B and C to determine the S_r and S_R precision estimates shown in Table 8. In addition, the results for sample pair 9/10 were separated into two data sets, one including data from laboratories that used a Pine compactor, and the other including data from laboratories that used a Troxler compactor. AMRL data includes the compactor manufacturer but not the compactor model number. The S_r and S_R precision estimates shown in Table 8 were determined for each data set.

3.4.3 Tests for Significance

As expected, the results of the *T*-test shown in Table 9 indicate a significant difference in the average G_{mb} for the 12.5-mm mixture and the average G_{mb} for the 19.0-mm mixture.

T-tests were performed to determine if the average G_{mb} values obtained using PS131 differed significantly from average G_{mb} values obtained by T166. The *T*-test results shown in Table 9 indicate statistically significant differences in the PS131 and T166 average G_{mb} values for both the 12.5-mm (2.362 vs. 2.386) and 19.0-mm (2.364 vs. 2.398) test data. In both cases, the average specific gravity determined from tests performed using PS131 was significantly lower than the average specific gravity determined from tests performed using T166. The bias in the test results from the two methods is apparent when viewing the box plots in Figure 7.

There is good agreement in the reproducibility estimates between the 12.5-mm and 19.0-mm mixtures (0.015 vs. 0.014) for the T166 data. However, the *F*-test result shown in Table 9 indicates a statistically significant difference between the repeatability for the 12.5-mm and 19.0-mm mixtures (0.008 vs. 0.013). The boxes in Figure 7a are noticeably smaller than those in Figure 7c.

T-tests were performed to determine if the average G_{mb} values obtained from specimens compacted using the Pine AFGC125X compactor differed significantly from average G_{mb} values obtained from specimens compacted using the Troxler 4140 compactor. The *T*-test results shown in Table 9 indicate statistically significant differences in the average density of specimens

compacted using the Pine compactor and the average density of specimens compacted using the Troxler compactor for the 12.5-mm and 19.0-mm T166 data, the 12.5-mm and 19.0-mm PS131 data, and AMRL proficiency sample pair 9/10 data. Analysis of data in this study and AMRL proficiency sample data indicates that the average density of specimens compacted with the Troxler compactor is lower than the average density of specimens compacted with a Pine compactor.

The results of *F*-tests shown in Table 9, comparing the 12.5-mm and 19.0-mm T166 G_{mb} data to the PS131 G_{mb} data, indicate that the S_r precision of G_{mb} data obtained on the 12.5-mm mixture using PS131 was statistically greater than the S_r precision of G_{mb} data obtained using T166 (0.011 vs. 0.008). In addition, the *F*-tests indicated that the S_R precision of G_{mb} data obtained on the 19.0-mm mixture using PS131 was statistically greater than the S_R precision of G_{mb} data obtained of the 19.0-mm mixture using PS131 was statistically greater than the S_R precision of G_{mb} data obtained on the 19.0-mm mixture using PS131 was statistically greater than the S_R precision of G_{mb} data obtained using T166 as shown in Table 8 (0.021 vs. 0.014).

The *F*-test results shown in Table 9, comparing data obtained from specimens compacted using the Pine compactor to data obtained from specimens compacted using the Troxler compactor, indicate that there is a significant difference in the S_r estimates for the 12.5-mm T166 data, the 12.5-mm PS131 data, and the AMRL proficiency sample pair 9/10 data. While the differences may be statistically significant, they are inconsistent and relatively small, and, therefore, may be insignificant from a practical standpoint.

3.4.4 Precision Statements

3.4.4.1 T 166

As indicated above, there is a significant difference between the repeatability for the 12.5-mm and 19.0-mm mixtures (0.008 vs. 0.013). Noting this difference, it appears appropriate to propose separate repeatability precision estimates for 12.5-mm and 19.0-mm mixtures. However, it is unclear from the data obtained whether the increase in single operator variability for 19.0-mm mixtures reflects problems with T166 or actual variation in the density of the specimens tested; that is, problems with the compaction process described in T312. Further research is needed to resolve this issue.

The current precision statement in T166 does not include a S_R estimate and, based on the results from this study, the S_r estimate appears to be high. The precision estimates published in ASTM D2726, Standard Test Method for Bulk Specific Gravity and Density of Non-Absorptive Compacted Bituminous Mixtures (4) agree more closely with the results obtained from this study.

The S_r and S_R estimates from the analysis of AMRL proficiency sample data shown in Table 8 are a little greater than the estimates resulting from the analysis of data obtained in this study. Differences of the magnitude observed are expected given the greater number of laboratories included in the AMRL Proficiency Sample Program.

Appendix E includes a proposed precision statement for T166 based on the findings from this study. It should be noted that the range in air voids covered by the 12.5-mm and 19.0-mm mixtures included in this study is limited. Work should continue to evaluate G_{mb} precision estimates for specimens covering a wider range of air voids.

3.4.4.2 PS131

The G_{mb} precision estimates shown in Table 8 resulting from the analysis of the 12.5-mm and 19.0-mm PS131 test results are greater than those obtained from the analysis of both T166 data reported for this study and AMRL proficiency sample data. The increase in variation in G_{mb} values obtained using the automatic vacuum sealing method may be due to the inexperience of the participants performing the test. Twelve of the participants reported that they performed the test for the first time on the 12.5-mm mixtures, and nine still reported having very little experience performing the test when it was performed on the 19.0-mm mixture. Seven of the participants reported performing the test 25 times or more. The increased variation in G_{mb} values may also be caused by the bags touching the sides of the bath during weighing, an incomplete evacuation of air during the vacuum sealing process, and pinholes developing in the bags after the vacuum sealing process.

3.5 RELATIVE DENSITY AT N_{ini} AND N_{des}

3.5.1 Introduction

T312 describes a method for preparing 150-mm diameter cylindrical specimens of HMA using the Superpave gyratory compactor. The resulting specimens are intended to simulate the density, aggregate orientation, and structural characteristics in an actual roadway when proper construction procedures are followed in the placement of a paving mix. T312 also describes procedures for calculating the relative density of a cylindrical specimen at any point in the compaction process from specimen height measurements, the G_{mb} of the specimen, and the G_{mm} of the mixture. Relative density values can be used for field control of a HMA production process.

Participants in this study were provided with three replicate 12.5-mm and three replicate 19.0-mm loose mix samples. They were asked to prepare gyratory specimens from each of the as-received samples and determine the relative density at N_{ini} (8 gyrations) and N_{des} (100 gyrations).

3.5.2 Precision Estimates

3.5.2.1 T312 Test Data

Twenty-six laboratories submitted full sets of relative density data at N_{ini} and N_{des} for both the 12.5-mm and 19.0-mm mixtures (See Tables 4 and 5, Columns 7 and 8). The data are

displayed in box plot form in Figure 12. All data were analyzed using the E691 software. Based on *h*- and *k*-statistics shown in Figures 13, 14, 15, and 16, the N_{ini} and N_{des} relative density data from laboratory 16 were eliminated from the 12.5-mm mixture analysis, the N_{ini} and N_{des} relative density data from laboratory 22 were eliminated from the 19.0-mm mixture analysis, and the N_{des} relative density data from laboratory 4 were eliminated from the 19.0-mm mixture analysis. All remaining data were re-analyzed with the E691 software to determine the S_r and S_R precision estimates shown in Table 10.

The 12.5-mm and 19.0-mm mixture, N_{ini} and N_{des} relative density data remaining after the removal of outliers were separated into four data sets based on the mixture type, and compactor manufacturer and model number used in the compaction. Each of the four data sets was analyzed with the E691 software to determine the S_r and S_R precision estimates shown in Table 10.

3.5.2.2 AMRL Proficiency Sample Test Data

For comparison purposes, S_r and S_R precision estimates were determined for relative density at N_{des} (100 gyrations) data from AMRL gyratory proficiency sample pairs 9/10 and 11/12. Data were analyzed as described in Appendices B and C to determine the S_r and S_R precision estimates shown in Table 10. In addition, the results for AMRL sample pair 9/10 were separated into two data sets, one including data from laboratories that used a Pine compactor, and the other including data from laboratories that used a Troxler compactor. The AMRL data identified the compactor manufacturer but not the compactor model number. The S_r and S_R precision estimates shown in Table 10 were determined for each data set.

3.5.2.3 T269

Air voids calculated and reported in AASHTO T269, Standard Test Method for Percent Air Voids in Compacted Dense and Open Bituminous Paving Mixtures (*3*) and ASTM D3203, Standard Test Method for Percent Air Voids in Compacted Dense and Open Bituminous Paving Mixtures (*4*) are equivalent to 100 minus the relative density at N_{des} . As a result, the precision estimates for relative density at N_{des} data from this study may be applied directly to T269 and D3203. The precision statement in ASTM D3203-94 states, "The precision of this test method depends on the precision of the test methods for bulk specific gravity and the theoretical maximum specific gravity. It is computed by a procedure described in ASTM Practice D4460, Standard Practice for Calculating Precision Estimates calculated from the precision estimates proposed for T166 and D2041 using D4460. The resulting precision estimates are in close agreement with those proposed for T312.

The precision estimates currently published in T269-97, shown in Table 10, were calculated from the precision estimates in T166 and T209. The estimates shown appear to be significantly higher than those proposed for T312. Although the range in air voids of the specimens tested in this study is limited, if D2041 is adopted as a replacement for T209, it may be appropriate to replace the precision estimates in T269 with those proposed for T312.

3.5.3 Tests for Significance

There is fairly good agreement in the N_{ini} and N_{des} reproducibility estimates shown on Table 10 for the T312, 12.5-mm and 19.0-mm relative density data (N_{ini} : 0.48 vs. 0.60, N_{des} : 0.59 vs. 0.57). However, the results of the *F*-tests shown in Table 11 indicate a statistically significant difference between the repeatability of the N_{ini} and N_{des} data for the 12.5-mm and 19.0-mm mixtures (N_{ini} : 0.32 vs. 0.49, N_{des} : 0.30 vs. 0.49). This is consistent with the findings relative to the G_{mb} data, since the relative density values are greatly influenced by the G_{mb} values.

T-tests were performed to determine if the average relative density of specimens compacted using the Pine AFGC125X compactor differed significantly from the average relative density of specimens compacted using the Troxler 4140 compactor. For the 12.5-mm data and AMRL proficiency sample pair 9/10 data, the *T*-test results shown in Table 11 indicated statistically significant differences in the relative density at N_{des}. In both cases the average relative density at N_{des} of specimens compacted with the Troxler compactor was lower than the average relative density at N_{des} of specimens compacted with a Pine compactor. The differences observed are consistent with those noted for the G_{mb} data for both compaction devices.

The *F*-test results shown in Table 11, comparing relative density data obtained from specimens compacted using the Pine AFGC125X compactor to relative density data obtained from specimens compacted using the Troxler 4140 compactor, indicate that there is a significant difference in the S_r estimates for the 12.5-mm data at N_{ini} and N_{des} . For both the 12.5-mm and 19.0-mm mixtures the S_r estimates for specimens compacted using the Troxler compactor are smaller than S_r estimates for specimens compacted using the Pine compactor, however, this trend was not supported by the AMRL proficiency sample pair 9/10 data. While the differences observed may be statistically significant, because of this inconsistency and the relative magnitude of the differences, they were judged to be insignificant from a practical standpoint.

3.5.4 Precision Statement for T312

As noted in Section 3.5.3, there is a significant difference between the repeatability of the N_{ini} and N_{des} data for the 12.5-mm and 19.0-mm mixtures (N_{ini} : 0.32 vs. 0.49, N_{des} : 0.30 vs. 0.49). Noting this difference, it appears to be appropriate, as it was for T166, to propose separate repeatability precision estimates for 12.5-mm and 19.0-mm mixtures. Here too, however, it is unclear from the data obtained whether the increase in single operator variability for 19.0-mm mixtures reflects actual variation in the relative density of the specimens tested, or a problem with the compaction process described in T312.

The S_r and S_R estimates from the analysis of AMRL proficiency sample relative density data at N_{des} are a little greater than the estimates resulting from the analysis of data in this study. These differences are a result of the increased variability of AMRL proficiency sample G_{mb} data noted earlier. Appendix F includes a proposed precision statement for T312 based on the findings from this study.

CHAPTER 4

CONCLUSIONS AND RECOMMENDATIONS

4.1 GENERAL

This study was conducted to prepare precision estimates for AASHTO and ASTM standards used to determine selected volumetric properties of HMA using non-absorptive aggregate. Specific study objectives were to (1) develop a precision statement applicable to AASHTO T312 used to prepare and determine the density of HMA specimens using a Superpave Gyratory compactor, (2) update precision statements currently published in AASHTO T166 used to determine bulk specific gravity of compacted asphalt mixtures and ASTM D2041 used to determine maximum specific gravity of bituminous paving mixtures, and (3) prepare first-cut precision estimates for ASTM Provisional Standards PS131 and PS132 which are used to determine bulk specific gravity and density of compacted bituminous mixtures using an automatic vacuum sealing method. The study conclusions and recommendations are as follows.

4.2 CONCLUSIONS AND RECOMMENDATIONS RELATED TO SPECIFIC STANDARDS

4.2.1 ASTM D2041, Standard Test Method for Theoretical Maximum Specific Gravity and Density of Bituminous Materials

Conclusions

1. The precision statement currently published in D2041-00 is in need of revision.

2. While the D2041 reproducibility estimates were statistically greater for the "weight in air" method than the "weight in water" method, there is not enough data available to provide separate precision estimates.

Recommendations

1. The precision statement in Appendix D should be adopted for D2041.

2. AMRL should collect data from participants in its Proficiency Sample Program to determine if separate S_r precision estimates are warranted for "weight in air" and "weight in water" methods.

4.2.2 AASHTO T166, Standard Test Method for Bulk Specific Gravity of Compacted Asphalt Mixtures Using Saturated Surface-Dry Specimens

Conclusions

1. The precision statement currently published in T166-00 is in need of revision.

2. The S_r precision estimates for the 19.0 mm mixture were statistically larger than that obtained for the 12.5 mm mixture.

3. The T166 bulk specific gravity (G_{mb}) values obtained from specimens compacted with the Pine AFGC125X compactor were greater than those obtained from specimens compacted with the Troxler 4140 compactor.

Recommendations

1. The precision statement proposed in Appendix E should be adopted for T166 (Method A). Separate S_r precision estimates are proposed for the 19.0 mm mixture and 12.5 mixtures. There was no apparent difference in the T166 S_R precision estimate for the 12.5 mm mixture and the 19.0 mm mixture as shown in the proposed precision statement.

2. Further research is needed to determine if the difference in the repeatability (S_r) of G_{mb} test results from 12.5-mm and 19.0-mm mixtures reflects potential problems with T166 or actual variation in the density of specimens tested.

4.2.3 ASTM D2041 Results Compared to ASTM PS132 Results

Conclusions

1. The variation in test results obtained using PS132 was statistically greater than that obtained using D2041.

2. The maximum specific gravity (G_{mm}) values obtained using PS132 were lower than those obtained using D2041.

Recommendation

1. A precision statement for PS132 should not be adopted until an interlaboratory study involving laboratories having more experience performing the test method is conducted.

2. Changes to PS132 to increase its precision should be investigated.

4.2.4 AASHTO T166 Results Compared to ASTM PS131 Results

Conclusions

1. The bulk specific gravity (G_{mb}) values obtained using PS131 were significantly lower than those obtained using T166.

2. The S_r estimates obtained using PS131 were statistically greater than those obtained using T166.

3. For the 19.0 mm mixture, the S_R estimate obtained using PS131 was statistically greater than the S_R estimate obtained using T166.

Recommendations

1. A precision statement for PS131 should not be adopted until an interlaboratory study involving laboratories having more experience performing the test method is conducted.

2. Changes to PS131 to increase its precision should be investigated.

4.2.5 AASHTO T312, Standard Method of Test for Preparing and Determining the Density of Hot-Mix Asphalt (HMA) Specimens by Means of the Superpave Gyratory Compactor

Conclusions

1. T312 needs a precision statement.

2. The T312 S_r estimate for the 19.0 mm mixture was statistically larger than that obtained for the 12.5 mm mixture.

3. The relative density values obtained from specimens compacted with the Pine AFGC125X compactor were greater than those obtained from specimens compacted with the Troxler 4140 compactor.

Recommendations

1. The precision statement proposed in Appendix F for T312 should be adopted. It includes separate S_r estimates for the 12.5-mm and 19.0-mm mixtures.

4.2.6 AASHTO T269, Standard Test Method for Percent Air Voids in Compacted Dense and Open Bituminous Paving Mixtures

Conclusions

1. The precision statement in T269 could be revised to include precision estimates applicable to Superpave specimens.

Recommendations

1. If AASHTO adopts D2041 as a replacement for T209, the precision estimates and conditions given in Appendix F for T312 could be added to the precision statement in T269 as precision estimates and conditions applying to Superpave specimens.

4.3 GENERAL CONCLUSIONS AND RECOMMENDATIONS

1. The bivariant analysis described by Hall (10) can be used effectively to screen AMRL proficiency sample data.

2. AMRL should attempt to develop precision estimates from existing asphalt binder and HMA proficiency sample data using the bivariate approach to screen the data.

3. Further research is needed to develop precision estimates for HMA mixtures with a wider range of air voids and having greater aggregate absorption.



Figure 1 -Box Samples for Participating Laboratories



Figure 2 – Box Plots for Maximum Specific Gravity (G_{mm}) Data



Figure 3 - h Consistency Statistics - Maximum Specific Gravity (G_{mm}) - D2041

Figure 5 - h Consistency Statistics - Maximum Specific Gravity (G_{mm}) -PS132

Figure 7 – Box Plots for Bulk Specific Gravity (Gmb) Data

Figure 8 - h Consistency Statistics - Bulk Specific Gravity (G_{mb}) - T166

Figure 10 - h Consistency Statistics - Bulk Specific Gravity (G_{mb}) -PS131

Figure 12 – Box Plots for Relative Density Data

Figure 13 - h Consistency Statistics - Relative Density (N_{ini}) -T312

Figure 15 - h Consistency Statistics - Relative Density (N_{des}) –T312

Material	12.5-mm, $G_{mm}(g)$	12.5-mm, Gyr (g)	19.0-mm, G _{mm} (g)	19.0-mm, Gyr (g)
19.0-mm aggregate			447	1010
12.5-mm aggregate	274	822	438	990
9.5-mm aggregate	268	805	204	460
4.75-mm aggregate	303	910	241	545
2.36-mm aggregate	273	820	239	540
Sand	414	1243	512	1155
Mineral Filler	10	30	9	20
Binder	69	207	86	195
Total:	1611	4837	2176	4915

Table 1 – Laboratory Mix Designs

Table 2 –HMA Design –Binder Content and Absorption

, 1	19.0-min mix, (percent)
4.28	3.97
3.59	3.37
0.7	0.6
	4.28 3.59 0.7

¹Determined Using PP28 (2)

	L.	e e
Property	12.5-mm Mixture	19.0-mm Mixture
Maximum Specific Gravity (G)	ASTM D2041-00	ASTM D2041-00
Property12.5-mm MixtureMaximum Specific Gravity (G_{mm})ASTM D2041-00Bulk Specific Gravity (G_{mb})ASTM PS132-01Bulk Specific Gravity (G_{mb})ASTM PS131-01Relative DensityAASHTO T166-00AASHTO T312, N _{ini} ASHTO T312, N _{ini}	ASTM PS132-01	
Maximum Specific Gravity (G _{mm}) Bulk Specific Gravity (G _{mb})	ASTM PS131-01	ASTM PS131-01
Buik Speenie Gruing (Gino)	AASHTO T166-00	AASHTO T166-00
Relative Density	AASHTO T312, N _{ini}	AASHTO T312, N _{ini}
	AASHTO T312, N _{des}	AASHTO T312, N _{des}

Table 3 - Test Properties Determined in Study

		Tuste	1210 11111 111	muit e Butu					
		Maximum Specific Gravi	ity	Bulk Specific C	aravity		Re	lative Density -	T 312
Lab Number	Weight in	G - D2041	G _PS132	G PS131	G . T166	N.	Ν.	Comp Manuf	Comp Model
Lab Number	weight in	2 549	2 534	2 371	2 201	92.2	02.8	Comp. Manur.	Comp. Woder
1	air	2.549	2.534	2.371	2.391	92.1	93.0	Pine	AGEC125X
	an	2.550	2.540	2.376	2.330	83.3	94.3		
		2.500	2.000	2.070	2,202	00.0	02.0		
2	water	2.540			2.392	03.5	93.9	Pine	AEGC125X
E	water	2.548			2.399	83.2	94.2	T IIIC	AIGOILOA
		2.551			2.402	83.7	94.2		
0		2.550	2.549	2.338	2.365	82.7	92.7	Dine	AFOOTOFY
3	water	2.547	2.547	2.327	2.358	82.5	92.6	Pine	AFGC125X
		2.551	2.541	2.341	2.349	81.9	92.1		
		2.551	2.540	2.373	2.404	83.4	94.2		
4	air	2.552	2.546	2.294	2.394	83.0	93.8	Pine	AFGC125Xa
		2.549	2.545	2.365	2.400	83.7	94.2		
		2.546	2.545	2.367	2.364	82.6	92.9		
5	water	2.554	2.571	2.359	2.368	82.5	92.7	Troxler	4140
		2.552	2.555	2.354	2.356	82.4	92.3		
		2.548	2.559	2.354	2.385	82.8	93.6		
6	water	2.549	2.534	2.360	2.384	83.0	93.5	Pine	AFGC125X
		2.546	2.561	2.375	2,400	83.5	94.3		
		2 545	2 540	2 390	2 402	84.3	94.4		
7	air	2 545	2 545	2 373	2.398	83.7	94.2	Pine	AFG1A
	c	2 550	2 548	2 407	2 411	84 1	94.5		-
		2.666	2.579	2.325	2.000	02.1	02.6		
8	-1-	2.546	2.573	2.335	2.362	00.1	93.0	Trovier	4140
5	air	2.545	2.531	2.335	2.390	83.5	93.9	TOXICI	4140
		2.553	2.575	2.351	2.390	83.4	93.6		
0		2.552			2.375	82.8	93.1	Treader	4140
9	water	2.551			2.381	83.5	93.3	Troxier	4140
		2.549			2.378	83.0	93.2		
10		2.550	2.517	2.379	2.401	83.5	94.1		
10	water	2.550	2.461	2.373	2.391	83.4	93.8	Pine	AFGC125X
		2.550	2.546	2.377	2.399	83.2	94.1		
		2.558			2.392	83.2	93.5		
11	water	2.564			2.397	83.0	93.5	Pine	AFGC125X
		2.566			2.398	83.2	93.4		
		2.550		2.380	2.394	83.1	93.9		
12	water	2.548	2.555	2.371	2.385	82.9	93.6	Pine	AFGC125X
		2.548	2,563	2.369	2.387	82.8	93.7		
		2.547	2.564	2.367	2,383	83.0	93.6		
13	water	2.549	2.552	2.381	2.397	83.4	94.0	Pine	AFGC125X
		2.547	2.542	2.407	2.422	84.6	95.1		
		2 551	2 553	2 346	2 371	83.2	92.9		
14	wator	2.549	2,556	2 341	2.071	83.0	02.0	Troxler	4140
	water	2.349	2.550	2.341	2.570	00.0	00.0		
		2.549	2.557	2.335	2.366	83.2	92.8		
15	-1-	2.560	2.504	2.373	2.409	83.6	94.1	Broyold	BGC-1
15	dii	2.556	2.512	2.380	2.411	02.9	94.3	Diovola	Dao I
		2.350	2.525	2.335	2.330	00.0	33.7		
16		2.553	2.525	2.360	2.368	82.7	92.7	Dina	AFOCIDEX
10	water	2.556	2.532	2.292	2.303	80.7	90.2	FILLE	AFGC125A
		2.552	2.513	2.277	2.289	80.5	89.6		
		2.550	2.553	2.352	2.388	84.0	93.6		
17	water	2.550	2.553	2.346	2.376	83.6	93.2	Iroxler	4140
		2.550	2.552	2.326	2.369	83.3	92.9		
		2.554	2.528	2.369	2.388	83.6	93.5		
18	air	2.551	2.551	2.374	2.396	84.2	93.9	Pine	AFGC125X
		2.552	2.482	2.375	2.397	84.4	93.9		
		2.548	2.628	2.360	2.384	84.0	93.6		
19	air	2.553	2.639	2.375	2.404	83.8	94.2	Troxler	4140
		2.554	2.634	2.377	2.395	83.7	93.8		
		2.553	2.518	2.333	2.361	82.5	92.5		
20	air	2.552	2.534	2.351	2.378	83.3	93.2	Troxler	4140
		2 551	2 538	2 352	2,369	83.1	92.9		
		2.546	2 543	2 354	2.000	83.6	02.0		
21	air	2.540	2.545	2.354	2 387	83.8	93.4	Troxler	4140B
	can	2 546	2 538	2 347	2 374	83.5	93.2	monior	
		2.546	2.000	2.047	2.074	00.0	00.2		
22		2.545			2.372	83.4	93.1	Troylor	4140
22	air	2.546			2.358	83.1	92.6	Troxier	4140
		2.547			2.366	83.1	93.0		
		2.553	2.559	2.343	2.384	83.7	93.3		
23	water	2.554	2.543	2.329	2.368	83.4	92.7	Troxier	4140
	ļ	2.551	2.555	2.331	2.365	83.1	92.6		
		2.540	2.545	2.363	2.382	83.2	93.8	_	
24	air	2.540	2.554	2.369	2.395	83.8	94.3	Brovold	BCG-1
		2.541	2.549	2.370	2.389	83.2	94.0		
		2.556	2.573	2.370	2.396	83.2	93.7		
25	water	2.557	2.529	2.341	2.398	82.9	93.8	Pine	AFGC125X
		2.558	2.559	2.384	2.409	83.5	94.2		
		2.554	2.519	2.380	2.390	83.1	93.6		
26	air	2,554	2,509	2,367	2,382	83.1	93.3	Brovold (Pine)	AFGB1A
	an	2,554	2.539	2.372	2,384	82 7	93.3	. ,	

Table 4 - 12.5-mm Mixture Data

Notes:

Shaded cells indicate data eliminated from analysis as described in Section 3.2.
 Empty cells or portions of cells indicate that the laboratory did not submit data. One laboratory did not submit any data.

	Maximum Specific Gravity Bulk Specific Gravity			Relative Density - T 31					
Lab Number	Weight in	G _{mm} - D2041	Gmm - PS132	G _{mb} - PS131	Gmb - T166	Nini	Ndee	Comp. Manuf.	Comp. Model
		2.562	2.554	- 110	2.376	82.4	92.7		
1	air	2.559	2.545		2.394	82.2	93.6	Pine	AGFC125X
		2.559	2.545	2.377	2.402	83.4	93.9		
		2.563			2.405	83.1	93.8		
2	water	2.564			2.407	83.2	93.9	Pine	AFGC125X
		2.564			2.407	83.5	93.9		
		2.557	2.533	2.371	2.397	83.5	93.7		
3	air	2.557	2.549	2.337	2.390	83.3	93.5	Pine	AFGC125X
		2.557	2.543	2.372	2.398	83.5	93.8		
4		2.568	2.561	2.385	2.431	83.9	94.7	Dine	
4	air	2.560	2.559	2.402	2.449	85.1	95.7	Pine	AFGC125X
		2.560	2.561	2.364	2.41/	83.8	94.4		
5	water	2.032	2.580	2.327	2.375	03.0	93.8	Trovler	4140
5	water	2.540	2.564	2.343	2.387	83.1 83.5	93.9	TTOXIET	4140
		2.555	2.504	2.342	2,300	83.0	04.0		
6	water	2.567	2.500	2.362	2 421	83.1	94.3	Pine	AFGC125X
	water	2 564	2 544	2.369	2 408	83.3	93.9		
		2.565	2.554	2.396	2.416	83.7	94.2		<u> </u>
7	air	2.560	2.549	2.371	2.385	82.7	93.2	Pine	AFGC125X
		2.562	2.555	2.383	2.403	83.3	93.8		
		2,565	2.558	2.367	2.410	83.2	93.9		
8	air	2.567	2.567	2.351	2.405	83.2	93.7	Troxler	4140
		2.567	2.586	2.337	2.381	82.9	92.8	2.8	
		2.560			2.383	83.5	93.1		1
9	water	2.562			2.373	82.9	92.6	Troxler	4140
		2.564			2.421	84.3	94.4		
		2.563	2.564	2.398	2.411	83.4	94.1		1
10	water	2.563	2.552	2.346	2.381	81.5	92.9	Pine	AFGC125X
		2.565	2.545	2.369	2.402	83.1	93.6		
		2.563			2.378	83.0	92.8		
11	water	2.560			2.408	84.2	94.1	Pine	AFGC125X
		2.562			2.416	84.5	94.3		
		2.565	2.565	2.369	2.395	83.3	93.4		
12	water	2.562	2.561	2.375	2.399	83.6	93.6	Pine	AFGC125X
		2.565	2.567	2.376	2.399	82.8	93.5		
		2.562	2.549	2.391	2.414	84.1	94.2		
13	water	2.563	2.549	2.401	2.401	83.5	93.7	Pine	AFGC125X
		2.564	2.552	2.382	2.405	83.6	93.8		
		2.564	2.559	2.352	2.392	83.6	93.3		
14	water	2.566	2.568	2.353	2.385	83.0	93.0	Troxler	4140
		2.564	2.564	2.359	2.398	83.7	93.5		
		2.564	2.565	2.347	2.392	82.4	93.3		
15	air	2.564	2.557	2.391	2.421	83.3	94.4	Brovold	BGC-1
		2.563	2.565	2.386	2.408	82.6	94.0		
16		2.562	2.534	2.357	2.389	83.1	93.2	Dino	AECOLOEX
10	water	2.565	2.525	2.328	2.361	82.1	92.0	Fille	AFGC125X
		2.563	2.539	2.342	2.376	82.9	92.7		
17	water	2.567	2.302	2.309	2.391	04.1	93.1	Trovler	4140
.,	walei	2.000	2.555	2.307	2.395	04.1	93.0	TTOXICT	4140
		2.304	2.557	2.370	2.393	00.0	93.3		
18	air	2.004	2.000	2.3/4	2.407	03.9 83 6	93.9 93.9	Pine	AFGC125X
	dii	2.502	2.560	2.303	2.390	94 A	93.0		
		2.566	2.500	2.300	2.429	837	93.6		<u> </u>
19	air	2.564	2.548	2,325	2,391	83.4	93.3	Troxler	4140
		2.562	2.551	2.328	2.389	82.9	93.3		
	Ì	2.563	2.548	2.310	2.371	82.9	92.5	l	1
20	air	2.564	2.541	2.364	2.393	83.5	93.3	Troxler	4140
		2.564	2.544	2.361	2.389	83.4	93.2		
		2.562	2.547	2.342	2.381	83.4	92.9		
21	air	2.560	2.549	2.356	2.391	83.9	93.4	Troxler	4140B
		2.561	2.547	2.376	2.408	84.2	94.0		
		2.546	2.464	2.278	2.392	83.9	93.9		
22	air	2.561	2.391	2.296	2.367	82.8	92.4	Troxler	4140
		2.561	2.460	2.331	2.434	85.2	95.0		
		2.565	2.576	2.377	2.404	84.2	93.7		
23	water	2.562	2.569	2.356	2.388	83.4	93.2	Troxler	4140
		2.563	2.569	2.383	2.411	84.0	94.1		<u> </u>
		2.549	2.637	2.374	2.392	83.7	93.7		
24	air	2.554	2.636	2.395	2.415	83.8	94.7	Brovold	BCG-1
	I	2.553	2.631	2.382	2.407	83.6	94.3		ļ
65		2.563	2.534	2.369	2.398	83.2	93.6	Divis	AFOOLOGY
25	water	2.561	2.541	2.368	2.417	83.4	94.4	Pine	AFGC125X
	ļ	2.561	2.501	2.258	2.409	83.4	94.1		<u> </u>
26			2.574	2.352	2.410	83.3	93.7	Broyold (Pipe)	AEGR1 A
20	air		2.5/0	2.343	2.385	82.0	92.7	Diovola (Fille)	AIGDIA
		0.507	2.0/4	2.351	∠.396	o2.9	93.1		<u> </u>
27	air	2.567	2.534			1			
21	aii	2.564	2.542						

Table 5 - 19.0-mm Mixture Data

Notes: 1. Shaded cells indicate data eliminated from analysis as described in Section 3.2. 2. Empty cells or portions of cells indicate that the laboratory did not submit data.

	No. of	No. of		Repeatability		Repeatability		Reprodu	ucibility
Data Source	Labs	Replicates	Average	1s (S _r)	d2s	1s (S _R)	d2s		
D2041, 12.5-mm Mix ^A	25	3	2.550	0.002	0.005	0.004	0.011		
D2041, 12.5-mm Mix, Wt in Air ^B	12	3	2.550	0.002	0.006	0.005	0.013		
D2041, 12.5-mm Mix, Wt in Water ^B	13	3	2.551	0.002	0.005	0.003	0.008		
D2041, 19.0-mm Mix ^A	24	3	2.562	0.002	0.006	0.003	0.009		
D2041, 19.0-mm Mix, Wt in Air ^B	12	3	2.562	0.002	0.006	0.004	0.012		
D2041, 19.0-mm Mix, Wt in Water ^B	12	3	2.563	0.002	0.005	0.002	0.005		
PS132, 12.5-mm Mix ^A	19	3	2.542	0.014	0.039	0.018	0.051		
PS132, 19.0-mm Mix ^A	23	3	2.557	0.009	0.025	0.022	0.063		
T209-99 AASHTO Book (<i>3</i>)	5	3		0.004	0.011	0.006	0.019		
D2041-00 ASTM Book (<i>4</i>)	~300	2		0.008	0.023	0.016	0.044		
AMRL PSP Samples 9 & 10, 12.5-mm Mix ^C	231	2	2.554	0.003	0.008	0.006	0.016		
AMRL PSP Samples 11 & 12, 12.5-mm Mix ^C	279	2	2.552	0.003	0.007	0.005	0.015		

Table 6 - Precision Estimates - Maximum Specific Gravity (G_{mm})

^A Outliers were eliminated and the remaining data were analyzed in the manner described in ASTM Practice E 691.

^B *Outliers excluded from the combined data set were eliminated. The remaining data were analyzed in the manner described in ASTM Practice E 691.*

^C Based on analysis of data falling within a bivariate normal tolerance region containing approximately 85% of the data with 95% confidence. (See Appendix B).

$Table \ 7 \ -Significance \ Levels \ (P-Values) \ for \ the \\Observed \ Differences \ in \ the \ Maximum \ Specific \ Gravity \ (G_{mm}) \ Data$

	T test	F test for S _r	F test for S_{R}
Data Compared	P-Value for the test of no difference in means	P-Value for the test of no difference in repeatability	P-Value for the test of no difference in reproducibility
D2041, 12.5-mm Mix D2041, 19.0-mm Mix		60.9%	19.8%
D2041, 12.5-mm Mix, Wt in Air D2041, 12.5-mm Mix, Wt in Water	16.2%	41.1%	0.6%
D2041, 19.0-mm Mix, Wt in Air D2041, 19.0-mm Mix, Wt in Water	2.2%	87.2%	0.0%
D2041, 12.5-mm Mix PS132, 12.5-mm Mix	0.1%	0.0%	0.0%
D2041, 19.0-mm Mix PS132, 19.0-mm Mix	3.0%	0.0%	0.0%

Note: Shaded cells indicate significant differences at a 1% level.

	No. of	No. of		Repeatability		Reproducibilit	
Data Source	Labs	Replicates	Average	1s (S _r)	D2s	1s (S _R)	d2s
T166, 12.5-mm Mix ^A	25	3	2.386	0.008	0.022	0.015	0.043
T166, 12.5-mm Mix, Pine AFGC125X ^B	11	3	2.392	0.008	0.024	0.014	0.040
T166, 12.5-mm Mix, Troxler 4140 ^B	10	3	2.376	0.007	0.021	0.012	0.033
T166, 19.0-mm Mix ^A	24	3	2.398	0.013	0.035	0.014	0.040
T166, 19.0-mm Mix, Pine AFGC125X ^B	12	3	2.401	0.012	0.035	0.015	0.043
T166, 19.0-mm Mix, Troxler 4140 ^B	9	3	2.392	0.013	0.036	0.013	0.036
PS131, 12.5-mm Mix ^A	20	3	2.362	0.011	0.030	0.019	0.054
PS131, 12.5-mm Mix, Pine AFGC125X ^B	8	3	2.368	0.012	0.034	0.018	0.050
PS131, 12.5-mm Mix, Troxler 4140 ^B	8	3	2.348	0.009	0.025	0.014	0.040
PS131, 19.0-mm Mix ^A	20	3	2.364	0.015	0.043	0.021	0.060
PS131, 19.0-mm Mix, Pine AFGC125X ^B	9	3	2.373	0.015	0.043	0.019	0.054
PS131, 19.0-mm Mix, Troxler 4140 ^B	8	3	2.352	0.015	0.042	0.019	0.053
T166 AASHTO Book, 4" dia. specimen				0.020	0.057		
D2726 ASTM Book, 4" dia. specimen	16	4		0.012	0.035	0.027	0.076
AMRL PSP Samples 7 & 8, 12.5-mm Mix ^{C,D}	231	2	2.487	0.008	0.022	0.020	0.058
AMRL PSP Samples 9 & 10, 12.5-mm Mix ^C	239	2	2.424	0.013	0.037	0.025	0.070
AMRL PSP Samples 9 & 10, Pine ^C	89	2	2.429	0.012	0.033	0.022	0.062
AMRL PSP Samples 9 & 10, Troxler ^C	120	2	2.422	0.014	0.040	0.027	0.076
AMRL PSP Samples 11 & 12, 12.5-mm Mix ^C	289	2	2.438	0.022 ^E	0.062 ^E	0.028	0.078

Table 8 - Precision Estimates - Bulk Specific Gravity (G_{mb})

^A Outliers were eliminated and the remaining data were analyzed in the manner described in ASTM Practice E 691.

^B Outliers excluded from the combined data set were eliminated. The remaining data were analyzed in the manner described in ASTM Practice E 691.

^{*C*} Based on analysis of data falling within a bivariate normal tolerance region containing approximately 85% of the data with 95% confidence.

^D Specimens compacted to N_{max}

 E A test to identify significant difference between the variance in the test results for samples 11 and 12, the F test, indicated with 99% probability that the sample variances were different. Therefore, the repeatability estimates are not valid.

$Table \ 9 \ -Significance \ Levels \ (P-Values) \ for \ the \\Observed \ Differences \ in \ the \ Bulk \ Specific \ Gravity \ (G_{mb}) \ Data$

	T test	F test for S _r	F test for S_R
Data Compared	P-Value for the test of no difference in means	P-Value for the test of no difference in repeatability	P-Value for the test of no difference in reproducibility
T166, 12.5-mm Mix T166, 19.0-mm Mix		0.2%	57.4%
T166, 12.5-mm Mix, Pine AFGC125X T166, 12.5-mm Mix, Troxler 4140	0.0%	0.3%	28.0%
T166, 19.0-mm Mix, Pine AFGC125X T166, 19.0-mm Mix, Troxler 4140	0.7%	16.0%	28.4%
PS131, 12.5-mm Mix, Pine AFGC125X PS131, 12.5-mm Mix, Troxler 4140	0.0%	0.2%	28.4%
PS131, 19.0-mm Mix, Pine AFGC125X PS131, 19.0-mm Mix, Troxler 4140	0.0%	36.1%	92.3%
AMRL PSP Samples 9 & 10, Pine AMRL PSP Samples 9 & 10, Troxler	0.2%	0.1%	9.8%
T166, 12.5-mm Mix PS131, 12.5-mm Mix	0.0%	0.7%	7.6%
T166, 19.0-mm Mix PS131, 19.0-mm Mix	0.0%	1.4%	0.1%

Note: Shaded cells indicate significant differences at a 1% level.

	No. of	No. of		Repeatability		Reprod	ucibility
Data Source	Labs	Replicates	Average (%)	1s (S _r)	d2s	1s (S _R)	d2s
T312, N _{ini} , 12.5-mm Mix ^A	25	3	83.3	0.32	0.91	0.48	1.36
T312, N _{ini} , 12.5-mm Mix, Pine AFGC125X ^B	11	3	83.3	0.37	1.05	0.52	1.49
T312, N _{ini} , 12.5-mm Mix, Troxler 4140 ^B	10	3	83.3	0.26	0.73	0.44	1.24
T312, N _{ini} , 19.0-mm Mix ^A	25	3	83.4	0.49	1.38	0.60	1.69
T312, N _{ini} , 19.0-mm Mix, Pine AFGC125X ^B	12	3	83.4	0.54	1.54	0.66	1.86
T312, N _{ini} , 19.0-mm Mix, Troxler 4140 ⁸	9	3	83.5	0.41	1.15	0.43	1.21
AMRL PSP Samples 9 & 10, N _{ini} , 12.5-mm Mix ^C	241	2	84.4	0.52	1.48	0.94	2.65
AMRL PSP Samples 11 & 12, N _{ini} , 12.5-mm Mix ^C	286	2	84.8	0.78 ^D	2.22 ^D	1.06	3.00
T312, N _{des} , 12.5-mm Mix ^A	25	3	93.6	0.30	0.85	0.59	1.67
T312, N _{des} , 12.5-mm Mix, Pine AFGC125X ^B	11	3	93.8	0.34	0.96	0.55	1.57
T312, N _{des} , 12.5-mm Mix, Troxler 4140 ^B	10	3	93.2	0.28	0.78	0.46	1.30
T312, N _{des} , 19.0-mm Mix ^A	24	3	93.6	0.49	1.40	0.57	1.60
T312, N _{des} , 19.0-mm Mix, Pine AFGC125X ^B	12	3	93.7	0.50	1.41	0.59	1.68
T312, N _{des} , 19.0-mm Mix, Troxler 4140 ^B	9	3	93.4	0.48	1.36	0.48	1.36
AMRL PSP Samples 9 & 10, N _{des} , 12.5-mm Mix ^C	238	2	94.8	0.53	1.51	0.98	2.77
AMRL PSP Samples 9 & 10, N _{des} , 12.5-mm Mix, Pine ^C	92	2	95.1	0.55	1.56	0.88	2.49
AMRL PSP Samples 9 & 10, N _{des} , 12.5-mm Mix, Troxler ^C	123	2	94.7	0.62	1.75	1.03	2.92
AMRL PSP Samples 11 & 12, N _{des} , 12.5-mm Mix ^C	284	2	95.6	0.85 ^D	2.40 ^D	1.11	3.13
T269 AASHTO Book, 4" dia. specimen ^E				0.51	1.44	1.09	3.08
D3203 ASTM Book, 4" dia. Specimen							
T312, Ndes, 12.5-mm Mix, D 4460 Calculation ^F				0.29	0.82	0.58	1.64
T312, Ndes, 19.0-mm Mix, D 4460 Calculation ^F				0.46	1.30	0.53	1.50

Table 10 - Precision Estimates - T 312 Relative Density

^A Outliers were eliminated and the remaining data were analyzed in the manner described in ASTM Practice E 691.

^B *Outliers excluded from the combined data set were eliminated. The remaining data were analyzed in the manner described in ASTM Practice E 691.*

^C Based on analysis of data falling within a bivariate normal tolerance region containing approximately 85% of the data with 95% confidence.

^D A test to identify significant difference between the variance in the test results for samples 11 and 12, the F test, indicated with 99% probability that the sample variances were different. Therefore, the repeatability estimates are not valid.

^E Precision estimates were computed according to ASTM Practice D4460 using published estimates of precision from the maximum and bulk specific gravity tests.

^F Precision estimates were computed according to ASTM Practice D4460 using estimates of precision for D2041 and T166 from data obtained in this study.

Data Compared	T test P-Value for the test of no difference in means	F test for S _r P-Value for the test of no difference in repeatability	F test for S _R P-Value for the test of no difference in reproducibility
T312, N _{ini} , 12.5-mm Mix T312, N _{ini} , 19.0-mm Mix	12.6%	0.7%	5.9%
T312, N _{ini} , 12.5-mm Mix, Pine AFGC125X T312, N _{ini} , 12.5-mm Mix, Troxler 4140	45.8%	0.1%	31.6%
T312, N _{ini} , 19.0-mm Mix, Pine AFGC125X T312, N _{ini} , 19.0-mm Mix, Troxler 4140	25.4%	2.2%	2.8%
T312, N _{des} , 12.5-mm Mix T312, N _{des} , 19.0-mm Mix	29.1%	0.5%	76.6%
T312, N _{des} , 12.5-mm Mix, Pine AFGC125X T312, N _{des} , 12.5-mm Mix, Troxler 4140	0.0%	0.1%	22.1%
T312, N _{des} , 19.0-mm Mix, Pine AFGC125X T312, N _{des} , 19.0-mm Mix, Troxler 4140	2.0%	43.8%	22.0%
AMRL PSP Samples 9 & 10, $N_{\rm des},$ 12.5-mm Mix, Pine AMRL PSP Samples 9 & 10, $N_{\rm des},$ 12.5-mm Mix, Troxler	0.0%	15.9%	50.2%

Table 11 –Significance Levels (P-Values) for the Observed Differences in the Relative Density Data

Note: Shaded cells indicate significant differences at a 1% level.

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APPENDIX A

INSTRUCTIONS TO LABORATORIES FOR TESTING SAMPLES

NCHRP 9-26 Precision Statement for AASHTO T312 12.5-mm Mix

Maximum Specific Gravity Of Bituminous Paving Mixtures

ASTM D2041-00, See Protocol Provided

The three small boxes of mix labeled 'NCHRP 9-26: Maximum Specific Gravity" are to be used for the maximum specific gravity test. The samples shall be heated in an oven at a temperature of $105 \pm 5^{\circ}$ C until soft enough to be separated. Use either the bowl or flask method, and the mechanical agitation device described in the method. Record the temperature, masses and the results below.

Weighing-in-Water Determination (Bowl)

	1	2	3
(a) Mass of Oven Dried Mix Sample in Air (0.1 g)			
(b) Mass of Bowl in Water @ 25°C (0.1 g)			
(c) Mass of Bowl+Mix in Water @ 25°C (0.1 g)			
(d) Temperature of Water Bath (0.1°C)			
a/a-(c-b) Maximum Specific Gravity, G _{mm} (0.001)			

Weighing-in-Air Determination (Flask or Bowl)

	1	2	3
(a) Mass of Oven Dried Mix Sample in Air (0.1 g)			
(b) Mass of Bowl/Flask+Water @ 25°C (0.1 g)			
(c) Mass of Bowl/Flask+Mix+Water @ 25°C (0.1 g)			
(d) Temperature of Bowl/Flask and Contents (0.1°C)			
a/(a+b-c) Maximum Specific Gravity, G _{mm} (0.001)			

Please submit all data by July 13, 2001

Name of Laboratory: _____ Tested by:

Tested by:	
Phone No:	
Date Tested:	

Maximum Specific Gravity Of Bituminous Paving Mixtures Using Corelok

See Protocol Provided

The three small boxes of mix labeled 'Maximum Specific Gravity' are to be used for the maximum specific gravity test. The samples shall be tested for D2041 and then dried back to constant mass. The samples shall be dried to constant mass in an oven at a temperature of $105 \pm 5^{\circ}$ C. Record the temperature, masses and the results below.

	1	2	3
(b) Mass of Oven Dried Mix Sample in Air (0.1 g)			
(a) Combined Mass of Two Plastic Bags (0.1 g)			
(c) Mass of Mix & Bags in Water @ 25°C (0.1 g)			
(d) Temperature of Water Bath $(0.1^{\circ}C)$			
(V _c) Apparent specific gravity of plastic sealing material			
b/a+b-c-(a/V _c) Maximum Specific Gravity, G _{mm} (0.001)			

Please submit all data by July 13, 2001

Name of Laboratory:	
Tested by:	
Phone No:	
Date Tested:	

Bulk Specific Gravity of Compacted Bituminous Mixtures Using Corelok

See Protocol Provided

If applicable, perform the Corelok on the compacted specimens before performing T166. Determine the bulk specific gravity of the compacted specimens at 25°C using the Corelok before determining the bulk specific gravity by T166. Record the temperature, masses and the bulk specific gravity of the extruded specimen below.

	1	2	3
(a) Sample mass in air (0.1 g)			
(b) Mass of dry, sealed specimen (0.1 g)			
(c) Mass of sealed specimen in water (0.1 g)			
(F _T) Apparent specific gravity of plastic sealing material			
(d) Temperature of Water Bath (0.1°C)			
a/b-c-(b-a/F _T) Bulk Specific Gravity, G _{mb} (0.001)			

Please submit all data by July 13, 2001

Name of Laboratory:	
Tested by:	
Phone No:	
Date Tested:	

Bulk Specific Gravity of Compacted Bituminous Mixtures Using SSD Specimens

AASHTO T166-00, See AASHTO Standard Specifications for Transportation Materials and Methods of Sampling and Testing (20th Edition)

After performing the bulk specific gravity with the Corelok, remove the compacted specimens from the sealed bag and determine the bulk specific gravity of the compacted specimens at 25° C using T166. Record the temperature, masses and the bulk specific gravity of the extruded specimen below.

	1	2	3
(a) Sample mass in air (0.1 g)			
(b) Sample mass in water (0.1 g)			
(c) SSD mass in air (0.1 g)			
(d) Temperature of Water Bath (0.1°C)			
a/(c-b) Bulk Specific Gravity, G _{mb} (0.001)			

Please submit all data by July 13, 2001

Name of Laboratory:	
Tested by:	
Phone No:	
Date Tested:	

Density of HMA Specimens by Means of the Superpave Gyratory Compactor

AASHTO TP4-00, See AASHTO Provisional Standards (2000 Edition)

Verify the calibration of the gyratory compactor prior to compacting the specimens. The three large boxes of mix labeled 'NCHRP 9-26: Gyratory" are to be compacted using the gyratory compactor. The mix shall be brought to the compaction temperature $(141-146^{\circ}C)$ by careful, uniform heating in an oven immediately prior to molding. Compact the specimens using 100 gyrations. Report the height of the specimens at N_{ini} (8 gyrations) and N_{des} (100 gyrations) to the nearest 0.1 mm. Please attach a copy of the printout for the specimen height after each gyration. Calculate and report the percent of maximum specific gravity at N_{ini} and N_{des}.

	1	2	3
Height @ N _{ini} (0.1 mm)			
Height @ N _{des} (0.1 mm)			
Percent G _{mm} @ N _{ini} (0.1 percent)			
Percent G _{mm} @ N _{des} (0.1 percent)			

Please submit all data by July 13, 2001

Please attach a printout of the specimen height after each gyration.

Gyratory Manufacturer:_____Model:_____

Name of Laboratory:	
Tested by:	
Phone No:	

Date Tested:

Maximum Specific Gravity Of Bituminous Paving Mixtures

ASTM D2041-00, See Protocol Provided

The three small boxes of mix labeled 'NCHRP 9-26 (19-mm): Maximum Specific Gravity" are to be used for the maximum specific gravity test. The samples shall be heated in an oven at a temperature of $105 \pm 5^{\circ}$ C until soft enough to be separated. Use either the bowl or flask method, and the mechanical agitation device described in the method. Record the temperature, masses and the results below.

		1	2	3
	Specimen No.			
(a) Mass of Oven Dried M	lix Sample in Air (0.1 g)			
(b) Mass of Bowl in Water @ 25°C (0.1 g)				
(c) Mass of Bowl+Mix in Water @ 25°C (0.1 g)				
(d) Temperature of Water Bath (0.1°C)				
a/a-(c-b) Maximum Spec	cific Gravity, G _{mm} (0.001)			

Weighing-in-Water Determination (Bowl)

Weighing-in-Air Determination (Flask or Bowl)

		1	2	3
	Specimen No.			
(a) Mass of Oven Dried Mix Sample in Air (0.1 g)				
(b) Mass of Bowl/Flask+Water @ 25°C (0.1 g)				
(c) Mass of Bowl/Flask+M				
(d) Temperature of Bowl/				
a/(a+b-c) Maximum Spe	cific Gravity, G _{mm} (0.001)			

Please submit all data by December 14, 2001

Name of Laboratory:

 Tested by:

 Phone No:

Date Tested:_____

Maximum Specific Gravity Of Bituminous Paving Mixtures Using Corelok

See Protocol Provided

The three small boxes of mix labeled 'NCHRP 9-26 (19-mm): Maximum Specific Gravity" are to be used for the maximum specific gravity test. The samples shall be tested for D2041 and then dried back to constant mass. The samples shall be dried to constant mass in an oven at a temperature of $105 \pm 5^{\circ}$ C. Record the temperature, masses and the results below.

		1	2	3
	Specimen No.			
(A) Combined Mass of Ty	wo Plastic Bags (0.1 g)			
(B) Mass of Oven Dried M	Aix Sample in Air (0.1 g)			
(C) Mass of Mix & Bags	in Water @ 25°C (0.1 g)			
(D) (A+B)-C				
(E) A/V _c				
V _c of the Plastic Bags				
(F) D-E				
(G) B/F Maximum Specific Gravity, G _{mm} (0.001)				
Temperature of Water Bat	th (0.1°C)			

Please submit all data by December 14, 2001

Name of Laboratory:	
Tested by:	
Phone No:	
Date Tested:	

Bulk Specific Gravity of Compacted Bituminous Mixtures Using Corelok

See Protocol Provided

If applicable, perform the Corelok on the compacted specimens before performing T166. Determine the bulk specific gravity of the compacted specimens at 25°C using the Corelok before determining the bulk specific gravity by T166. Record the temperature, masses and the bulk specific gravity of the extruded specimen below.

		1	2	3
	Specimen No.			
(a) Sample mass in air (0	0.1 g)			
(b) Mass of dry, sealed sp	pecimen (0.1 g)			
(c) Mass of sealed specin	nen in water (0.1 g)			
(F _T) Apparent specific g material	gravity of plastic sealing			
(d) Temperature of Wate	r Bath (0.1°C)			
a/[b-c-(b-a/F _T)] Bulk (0.001)	Specific Gravity , G _{mb}			

Please submit all data by December 14, 2001

Name of Laboratory:	
Tested by:	
Phone No:	
Date Tested:	

Bulk Specific Gravity of Compacted Bituminous Mixtures Using SSD Specimens

AASHTO T166-00, See AASHTO Standard Specifications for Transportation Materials and Methods of Sampling and Testing (21st Edition)

After performing the bulk specific gravity with the Corelok, remove the compacted specimens from the sealed bag and determine the bulk specific gravity of the compacted specimens at 25° C using T166. Record the temperature, masses and the bulk specific gravity of the extruded specimen below.

		1	2	3
	Specimen No.			
(a) Sample mass in air (0.1 g)				
(b) Sample mass in water (0.1 g)				
(c) SSD mass in air (0.1 g)				
(d) Temperature of Water Bath (0.1°C)				
a/(c-b) Bulk Specific Gravity, G _{mb} (0.001)				

Please submit all data by December 14, 2001

Name of Laboratory:

Lacoratory.	
Tested by:	
Phone No:	
Date Tested:	

Density of HMA Specimens by Means of the Superpave Gyratory Compactor

AASHTO T312-01, See AASHTO Standard Specifications for Transportation Materials and Methods of Sampling and Testing (21st Edition)

Verify the calibration of the gyratory compactor prior to compacting the specimens. The three large boxes of mix labeled 'NCHRP 9-26 (19-mm): Gyratory" are to be compacted using the gyratory compactor. The mix shall be brought to the compaction temperature (141-146°C) by careful, uniform heating in an oven immediately prior to molding. Compact the specimens using 100 gyrations. Report the height of the specimens at N_{ini} (8 gyrations) and N_{des} (100 gyrations) to the nearest 0.1 mm. Use the corresponding D2041 G_{mm} value and T166 G_{mb} value from columns 1, 2 and 3 on Pages 1 and 4 when calculating the Percent G_{mm} @ N_{ini} and N_{des} for columns 1, 2 and 3. Please attach a copy of the printout for the specimen height after each gyration. Calculate and report the percent of maximum specific gravity at N_{ini} and N_{des} .

		1	2	3
	Specimen No.			
Height @ N _{ini} (0.1 mm)				
Height @ N _{des} (0.1 mm)				
Percent G _{mm} @ N _{ini} (0.1 percent)				
Percent G _{mm} @ N _{des} (0.1 J	percent)			

Please submit all data by December 14, 2001

Please attach a printout of the specimen height after each gyration.

Gyratory Manufacturer:	Model:
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APPENDIX B

BIVARIANT TOLERANCE REGION – A TOOL FOR SCREENING AMRL PROFICIENCY SAMPLE DATA

In this study, AMRL proficiency sample data were analyzed to judge the suitability of the precision estimates determined and to validate the findings regarding the comparison of test procedures and compaction equipment.

The AMRL proficiency sample testing rounds involve paired test samples distributed to and tested by a large number of laboratories. The primary purpose of the program is to provide participants with a tool for comparing their test results to those of other laboratories. However, the reasons for participation vary, and in some cases the data obtained may not be the result of testing performed in exact accordance with the standard methods specified. Some laboratories participate to verify non-standard versions of test procedures. Some laboratories use the program to evaluate the competency of their technicians. Some technicians participate in order to become certified or to maintain certification. Some tests are performed by certified/qualified technicians, others are not. Some laboratories perform tests using test equipment that meets specification requirements, others may not. Many of the participating laboratories are assessed by AMRL and also accredited by AASHTO, however, many are not.

In order to use the data for comparison purposes it is necessary to screen the data so that only the core data remains. Since all AMRL proficiency sample rounds result in paired test results from each laboratory, the method chosen to eliminate spurious data prior to analysis involved defining a bivariate tolerance region as described Hall (10) and eliminating the data beyond the region defined. This technique allows the user to define a region to encompass a desired percentage of the data with a selected degree of confidence. For this study a region that encompassed approximately 85 percent of the data with 95 percent confidence was chosen. The figure below shows the bivariate tolerance region obtained when the technique was applied to the bulk specific gravity data from AMRL gyratory proficiency sample pair 9/10. The data outside the ellipse were eliminated from the analysis.

APPENDIX C

STATISTICAL METHODS FOR ANALYZING AMRL PROFICIENCY SAMPLE DATA

Since the two samples comprising a pair of AMRL proficiency samples are not identical in many cases, S_r (repeatability) estimates are obtained in the manner described by Youden (11) by applying the following equation to the paired data:

$$S_{r} = \sqrt{\frac{\sum((x_{i} - y_{i}) - (\bar{x} - \bar{y}))^{2}}{2(n-1)}}$$
(1)

where:

 S_r = repeatability estimate X_i = laboratory test result from the odd number sample of a pair Y_i = laboratory test result from the even number sample of a pair \overline{X} = average of all X_i \overline{Y} = average of all Y_i

This equation removes any actual differences in the samples and allows the paired test results to be treated as replicates. In some cases, when "F"exceeds "F critical"at an alpha level of 0.05, the differences between the samples comprising the pairs are judged to be too great. In these instances the S_r estimates are considered invalid and unsuitable as precision estimates.

 S_R precision estimates for AMRL proficiency sample data are obtained using the following equation:

$$S_{R} = \sqrt{\frac{n\sum_{i}x_{i}^{2} - (\sum_{i}x_{i})^{2}}{n(n-1)} + \frac{n\sum_{i}y_{i}^{2} - (\sum_{i}y_{i})^{2}}{n(n-1)}}{2}$$
(2)

where:

 S_R = reproducibility estimate

APPENDIX D

PRECISION STATEMENT FOR: ASTM STANDARD TEST METHOD FOR THEORETICAL MAXIMUM SPECIFIC GRAVITY AND DENSITY OF BITUMINOUS PAVING MIXTURES, D 2041-00

1. Precision and Bias

1.1 Precision

- **1.1.1** Single Operator Precision The single operator standard deviation has been found to be 0.002^A (1s limit) for mixtures containing aggregate with an absorption of less than 1.5 percent, having a nominal maximum aggregate size of 12.5-mm or 19.0-mm, and tested without use of Section 11. Therefore, the results of two properly conducted tests on the same material, by the same operator, using the same equipment, should be considered suspect if they differ by more than 0.006 (d2s limit).
- **1.1.2 Multilaboratory Precision**^B The multilaboratory standard deviation has been found to be 0.004^A (1s limit) for mixtures containing aggregate with an absorption of less than 1.5 percent, having a nominal maximum aggregate size of 12.5-mm or 19.0-mm, and tested without use of Section 11. Therefore, the results of two properly conducted tests on the same material, by different operators, using different equipment should be considered suspect if they differ by more than 0.011 (d2s limit).

^ABased on an interlaboratory study described in NCHRP Research Report 9-26 involving twenty-seven laboratories, two materials (a 12.5-mm mixture and a 19.0-mm mixture), and three replicates. The approximately 2300 g, 19.0-mm mixture specimens tested did not meet the minimum sample size requirement in D2041.

^BLimited research has indicated that the multilaboratory precision estimate for the "weight in water" method of test may be less than the multilaboratory precision estimate for the "weight in air" method of test. Further investigation is underway to determine if separate multilaboratory precision estimates are warranted.

1.2 Bias

No information can be presented on the bias of the procedure because no material having an accepted reference value is available.

APPENDIX E

PRECISION STATEMENT FOR: AASHTO STANDARD TEST METHOD FOR BULK SPECIFIC GRAVITY OF COMPACTED ASPHALT MIXTURES USING SATURATED SURFACE-DRY SPECIMENS, T 166-00

1. Precision

1.1 Precision

- **1.1.1** Single Operator Precision The single operator standard deviations (1s limits) for Method A, for mixtures containing aggregate with an absorption of less than 1.5 percent, are shown in Table F-1^A. The results of two properly conducted tests on the same material, by the same operator, using the same equipment, should be considered suspect if they differ by more than the d2s single operator limits shown in Table F-1^A.
- **1.1.2 Multilaboratory Precision** The multilaboratory standard deviations (1s limits) for Method A, for mixtures containing aggregate with an absorption of less than 1.5 percent, are shown in Table F-1^A. The results of two properly conducted tests on the same material, by different operators, using different equipment should be considered suspect if they differ by more than the d2s multilaboratory limits shown in Table F- 1^{A} .

	1s limit	d2s limit		
Single Operator Precision:				
12.5-mm nominal max. agg.	0.008	0.023		
19.0-mm nominal max. agg.	0.013	0.037		
Multilaboratory Precision:				
12.5-mm nominal max. agg.	0.015	0.042		
19.0-mm nominal max. agg.	0.015	0.042		

Table F-1 - Precision Estimates

^ABased on an interlaboratory study described in NCHRP Research Report 9-26 involving 150-mm diameter specimens with 4-5 percent air voids, twenty-six laboratories, two materials (a 12.5-mm mixture and a 19.0-mm mixture), and three replicates.

1.2 Bias

No information can be presented on the bias of the procedure because no material having an accepted reference value is available.

APPENDIX F

PRECISION STATEMENT FOR: AASHTO STANDARD TEST METHOD FOR PREPARING AND DETERMINING THE DENSITY OF HOT-MIX ASPHALT (HMA) SPECIMENS BY MEANS OF THE SUPERPAVE GYRATORY COMPACTOR, T 312-01

1. Precision and Bias

1.1 Precision

- **1.1.1** Single Operator Precision The single operator standard deviations (1s limits) for densities at N_{ini} and N_{des}, for mixtures containing aggregate with an absorption of less than 1.5 percent, are shown in Table H-1^A. The results of two properly conducted tests on the same material, by the same operator, using the same equipment, should be considered suspect if they differ by more than the d2s single operator limits shown in Table H-1^A.
- **1.1.2 Multilaboratory Precision** The multilaboratory standard deviations (1s limits) for relative densities at N_{ini} and N_{des} , for mixtures containing aggregate with an absorption of less than 1.5 percent, are shown in Table H-1^A. The results of two properly conducted tests on the same material, by different operators, using different equipment should be considered suspect if they differ by more than the d2s multilaboratory limits shown in Table H-1^A.

	1s limit	d2s limit	
	Relative Density (%)	Relative Density (%)	
Single Operator Precision:			
12.5-mm nominal max. agg.	0.3	0.9	
19.0-mm nominal max. agg.	0.5	1.4	
Multilaboratory Precision:			
12.5-mm nominal max. agg.	0.6	1.7	
19.0-mm nominal max. agg.	0.6	1.7	

Table H-1 - Precision Estimates

^ABased on an interlaboratory study described in NCHRP Research Report 9-26 involving 150-mm diameter specimens with 4-5 percent air voids, twenty-six laboratories, two materials (a 12.5-mm mixture and a 19.0-mm mixture), and three replicates.

1.2 Bias

No information can be presented on the bias of the procedure because no material having an accepted reference value is available.