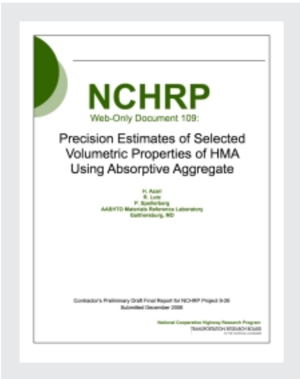

Precision Estimates of Selected Volumetric Properties of HMA Using Absorptive Aggregate

DETAILS

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State Department of Transportation Laboratories:

- Florida Department of Transportation, Gainesville, Florida
- Georgia Department of Transportation, Forest Park, Georgia
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- Indiana Department of Transportation, Indianapolis, Indiana
- Iowa Department of Transportation, Ames, Iowa
- Kansas Department of Transportation, Topeka, Kansas
- Kentucky Transportation Cabinet, Frankfort, Kentucky
- Maryland State Highway Administration, Easton, Maryland
- Maryland State Highway Administration, Greenbelt, Maryland
- Maryland State Highway Administration, Hancock, Maryland
- Maryland State Highway Administration, Lutherville, Maryland
- Minnesota Highway Department, Maplewood, Minnesota
- Montana Department of Highways, Helena, Montana
- Nebraska Department of Roads, Lincoln, Nebraska
- Nevada Department of Transportation, Carson City, Nevada
- New York Department of Transportation, Albany, New York
- Oklahoma Department of Transportation, Oklahoma City, Oklahoma
- South Carolina Department of Transportation, Columbia, South Carolina
- Utah Department of Transportation, Salt Lake City, Utah
- Virginia Department of Highways and Transportation, Richmond, Virginia
- Washington Department of Transportation, Tumwater, Washington
- Wyoming State Highway Department, Cheyenne, Wyoming

Other Participating Laboratories:

- Asphalt Institute, Lexington, Kentucky
- Federal Highway Administration, Lakewood, Colorado
- Payne & Dolan Inc., Waukesha, Wisconsin
- Rieth-Riley Construction Company, Indianapolis, Indiana

ABSTRACT

This report presents the results of a study to prepare precision estimates for AASHTO and ASTM standards used to determine selected volumetric properties of HMA using absorptive aggregates. An interlaboratory program was conducted with 27 participating laboratories to develop precision statements applicable to (1) AASHTO T312 used to prepare and determine the density of HMA specimens using a Superpave gyratory compactor, (2) AASHTO T166 used to determine bulk specific gravity of compacted asphalt mixtures, (3) ASTM D2041 used to determine maximum specific gravity of bituminous paving mixtures, and (4) ASTM D6752 used to determine bulk specific gravity and density of compacted bituminous mixtures using an automatic vacuum sealing method. It was found that precision estimates for D2041 and T312 using absorptive aggregates were significantly different from those using non-absorptive aggregates. The precision estimates for T166 and D6752 of mixtures with absorptive aggregates were found comparable to those of mixtures with non-absorptive aggregates. Draft precision estimates regarding mixtures with absorptive aggregates are proposed and included in this report. In addition to the ILS study, an experiment was conducted in the AMRL laboratory to determine the effects of aging time on mixtures containing absorptive aggregates and to provide guidance on appropriate laboratory aging time. The result of aging time study indicated that absorptive aggregates continued to absorb asphalt for a prolonged period of 32 hours; however, the laboratory aging time should be limited to a shortened period of 4 hours in which the changes in volumetric properties has not yet become significant.

EXECUTIVE 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. In Phase 1 of the NCHRP 9-26 project [1] precision estimates were developed for T312 using mixtures with non-absorptive aggregates. In this process, the precision estimates for the related standard test methods of 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 was updated. Furthermore, a first cut precision statement was prepared for ASTM D6752, Standard Method of Test for Bulk Specific Gravity and Density of Compacted Mixtures Using Automatic Vacuum Sealing Method. The objective of Phase 4 of the NCHRP 9-26 project was to develop precision estimates for the same standard test methods using mixtures with absorptive aggregates.

Another objective of Phase 4 was to examine the effect of aging time on selected volumetric properties of mixtures with absorptive aggregates and to recommend an appropriate laboratory aging time for these type mixtures. Currently, 2 hours of laboratory aging time is recommended by the Standard Practice R30 [2] for the volumetric design of mixtures with non-absorptive aggregates. However, mixtures with absorptive aggregates may require further conditioning in order to better represent conditions during production.

The experimental plan for the Phase 4 study selected two absorptive aggregate sources with differing levels of absorption. The binder for the study was a PG 64-22 meeting the requirements of AASHTO MP1, Standard Specification for Performance Graded Asphalt Binder. The plan required determining the test precision and aging time applicable to the coarse and fine mixes with absorptive aggregates. A maximum aggregate size of 19.0-mm was used for the coarse mix and 9.5-mm for the fine mix. This resulted in four mixtures from the 2 aggregate sources each having two gradations.

Twenty-seven laboratories volunteered to participate in the interlaboratory program to determine precision estimates of the selected volumetric properties applicable to AASHTO 312. The test samples for the interlaboratory study 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 four mixtures 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 were statistically analyzed and precision estimates prepared. The study resulted in

proposed precision statements for mixtures with absorptive aggregates in a format for consideration by standards committees for D2041, T166 (Method A), D6752, and T312.

To evaluate the effect of aging time, the four mixtures described prior were aged for a wide range of 2, 4, 8, 16, and 32 hours. The changes in number of volumetric properties as a result of change in aging time were examined. The volumetric properties included bulk specific gravity (T 166 and D6752), maximum specific gravity (D2041), percent air voids (T312), and percent absorbed asphalt. Based on the significance of the volumetric changes from statistical and practical standpoints an aging time of 4 hours was recommended for asphalt mixtures with absorptive aggregate.

CHAPTER 1- INTRODUCTION AND RESEARCH APPROACH

1.1 Introduction

Under the National Cooperative Highway Research Program (NCHRP) Project 09-26, the AASHTO Materials Reference Laboratory (AMRL) is conducting a multi-phase research project to determine or update estimates of precision in AASHTO test methods for asphalt binder and hot-mix asphalt (HMA). The objective of Phase 1 of Project 09-26 was to determine precision estimates for selected volumetric properties of HMA using non-absorptive aggregates [1]. This report details the results of Phase 4 of NCHRP 09-26, an extension of the Phase 1 activities. This phase expands upon the Phase 1 objective to produce estimates of precision for the same test methods using absorptive aggregates, having 4-5% water absorption, as opposed to non-absorptive aggregates. Also included in this study is the result of the experiment to examine the effects of oven aging on laboratory-prepared specimens containing absorptive aggregates.

1.1.1 Problem Statements

The Phase 4 project involves two studies, an Interlaboratory study and an aging time study.

1.1.1.1 Interlaboratory Study (ILS) Problem Statement

Superpave hot-mix asphalt mix designs rely on the volumetric properties of HMA in order to satisfy conditions set in AASHTO Specification M323, Superpave Volumetric Mix Design. The ILS in Phase 1 of NCHRP [1] provided estimates of precision for various test methods associated with HMA mix design when non-absorptive aggregates are used. However, the applicability of the precision estimates for mixtures prepared with absorptive aggregates is not known. In this respect, the ILS in Phase 1 will be repeated in Phase 4 using mixture with absorptive aggregates.

1.1.1.2 Aging Time Study Problem Statement

AASHTO Standard Practice R35, Superpave Volumetric Design for Hot-Mix Asphalt (HMA), references AASHTO Standard Practice R30, Mixture Conditioning of Hot-Mix Asphalt (HMA), for conditioning (aging) specimens in order to "simulate the plant-mixing and construction effects on the mixture" [2]. According to R30, this mixture conditioning is "designed to allow for binder absorption during the mixture design" [2]. Standard Practice R30 currently specifies 2 hours for conditioning laboratory prepared specimens. This time has been determined to be sufficient for non-absorptive aggregates but Note 1 of R30 states that absorptive aggregates may require further conditioning in order to better represent conditions during production. As a result, R30 does not provide specific guidance when absorptive aggregates are encountered. The "Aging Time Study" was a designed experiment whose results will attempt to give that guidance.

1.1.2 Research Objectives

The two objectives of Phase 4 of NCHRP Project 09-26 are:

I. Determine estimates of precision for the following standard test methods when HMA designs containing absorptive aggregates are used:

1.	AASHTO T312	Preparing and Determining the Density of Hot Mix Asphalt (HMA) Specimens by Means of the Gyratory Compactor
2.	AASHTO T166	Bulk Specific Gravity of Compacted Hot Mix Asphalt Using Saturated Surface –Dry Specimens
3.	ASTM D2041	Theoretical Maximum Specific Gravity and Density of Bituminous Paving Mixtures
4.	ASTM D6752	Bulk Specific Gravity and Density of Compacted Bituminous Mixtures Using Automatic Vacuum Sealing Method

II. Determine the effects of aging time on selective volumetric properties of mixtures containing absorptive aggregates and to provide guidance on appropriate laboratory aging time.

1.2 Scope of Study

The scope of the project involved the following major activities:

I. Design four hot mix asphalt mixtures using two absorptive aggregate sources and two nominal maximum aggregate sizes (Chapter 2).

   i. Source 1, 9.5-mm design
   ii. Source 1, 19-mm design
   iii. Source 2, 9.5-mm design
   iv. Source 2, 19-mm design

II. Produce approximately 600 specimens to send to participating laboratories for the ILS (Chapter 3).

III. Analyze results and develop precision estimates for the selected volumetric properties (Chapter 4).

IV. Study the effects of aging time on hot mix asphalt mixtures containing absorptive aggregates (Chapter 5).

V. Make conclusions and recommendations based on the analysis of results in Chapter 4 and Chapter 5 (Chapter 6).

CHAPTER 2- MATERIALS SELECTION AND MIX DESIGN

This section highlights the results of the relevant work performed prior to the Interlaboratory Study and Aging Time Study. Included is information regarding the selection of materials used in the project and the four asphalt mix designs needed to complete the project tasks.

2.1 Materials Selection

Materials used in the study were selected with the assistance of various outside agencies. The following sections list the pertinent material sources and the supplier.

2.1.1 Source 1 Aggregate

The first aggregate source chosen was a crushed caliche aggregate obtained from a stockpile near Ragland, New Mexico. The State of New Mexico has successfully used the stone in several highway projects.

Typical Test Properties of the Coarse Aggregate measured in 2003 and recorded by the State of New Mexico are given below.

- Bulk Specific Gravity = 2.349
- Percent Absorption = 5.0%

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

(a) Coarse Aggregate

- Water Absorption by AASHTO T85-91, Standard Method of Test for Specific Gravity and Absorption of Coarse Aggregate = 5.0%
- Bulk Specific Gravity by AASHTO T85-91 = 2.325

(b) Fine Aggregate

- Water Absorption by AASHTO T84-00, Standard Method of Test for Specific Gravity and Absorption of Fine Aggregate = 5.6%
- Bulk Specific Gravity by AASHTO T84-00 = 2.350

2.1.2 Source 2 Aggregate

The second aggregate source chosen was a crushed basalt aggregate obtained from a stockpile near Glenoma, Washington. The State of Washington has used this stone in several highway projects.

Typical Test Properties of the Coarse Aggregate measured in 2003 and recorded by the State of Washington are given below.

- Bulk Specific Gravity = 2.446
- Percent Absorption = 3% - 4%

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

(a) Coarse Aggregate

- Water Absorption by AASHTO T85-91, Standard Method of Test for Specific Gravity and Absorption of Coarse Aggregate = 4.3%
- Bulk Specific Gravity by AASHTO T85-91 = 2.428

(b) Fine Aggregate

- Water Absorption by AASHTO T84-00, Standard Method of Test for Specific Gravity and Absorption of Fine Aggregate = 5.1%
- Bulk Specific Gravity by AASHTO T84-00 = 2.350

2.1.3 Asphalt Binder

The binder used in all mix designs was a PG 64-22 grade 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.

2.2 Mix Designs

Four HMA Mix designs were created during the study. Information for each of the four designs can be found in Appendix A. Mix designs were created using the following AASHTO Standards:

- AASHTO R35, Standard Practice for Superpave Volumetric Mix Design for Hot-Mix Asphalt (HMA)
- AASHTO M323, Standard Specification for Superpave Volumetric Mix Design

CHAPTER 3- INTERLABORATORY STUDY EXPERIMENTAL PLAN

3.1 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 [3]. The absolute minimum number of laboratories required for the development of a precision statement is specified as 6 in E691 with a preferred minimum of 30.

The study involved two mix designs using two different sources of absorptive aggregates, resulting in four mixtures. The two mix designs included a fine gradation with 9.5-mm nominal maximum aggregate size and a coarse gradation with 19.0-mm nominal maximum aggregate size. The four mixtures are referred to as: S1-9.5-mm (Source 1, 9.5-mm design), S1-19-mm (Source 1, 19-mm design), S2-9.5-mm (Source 2, 9.5-mm design), and S2-19-mm (Source 2, 19-mm design). For each mix design, each participant was asked to determine the theoretical maximum specific gravity of three approximately 2000 g loose mix test specimens by D2041. 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 compacted specimens according to T166, determine the bulk specific gravity of the same compacted specimens as described in D6752 using the automatic vacuum sealing method, and calculate the relative density at N_{ini} and N_{des} as described in T312.

3.2 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 who have all the equipment and accessories needed for the completion of the tests included in the study.
5. Preference will be given to laboratories which had been participating 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 [4, 57].

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 Research 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 22 State DOTs, 1 research laboratories, 2 private sector laboratories, a FHWA laboratory, and AMRL. 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, 4].

3.3 Test Samples and Test Protocols

3.3.1 Sample Preparation

Samples were prepared by AMRL staff in their Proficiency Sample Facility located at the National Institute of Standards and Technology (NIST) using procedures developed for the AMRL HMA Proficiency Sample Program [5].

The laboratory mix formulas provided in Appendix A were used to prepare S1-9.5-mm, S1-19-mm, S2-9.5-mm, and S2-19-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 eight mix operations shown in Table 3-1.

Table 3-1- ILS sample production schedule

Production Date	Source	Size	Test	No. of Specimens
Date1	1	9.5	G_{mm} (D2041)	100
Date2	2	19	G_{mm} (D2041)	85
Date3	2	9.5	G_{mb} (T166 & D6752)	110
Date4	2	9.5	G_{mm} (D2041)	85
Date5	1	19	G_{mb} (T166 & D6752)	110
Date6	1	19	G_{mm} (D2041)	85
Date7	2	19	G_{mb} (T166 & D6752)	110
Date8	1	9.5	G_{mb} (T166 & D6752)	110
			Total	795

The samples were boxed and marked as shown in Figure 3-1. Each laboratory received one set of sample from each of the four mixtures for G_{mm} measurements and one set of sample from each of the four mixtures for gyratory compaction. Each set contained three replicate samples chosen at random from the samples selected for distribution.

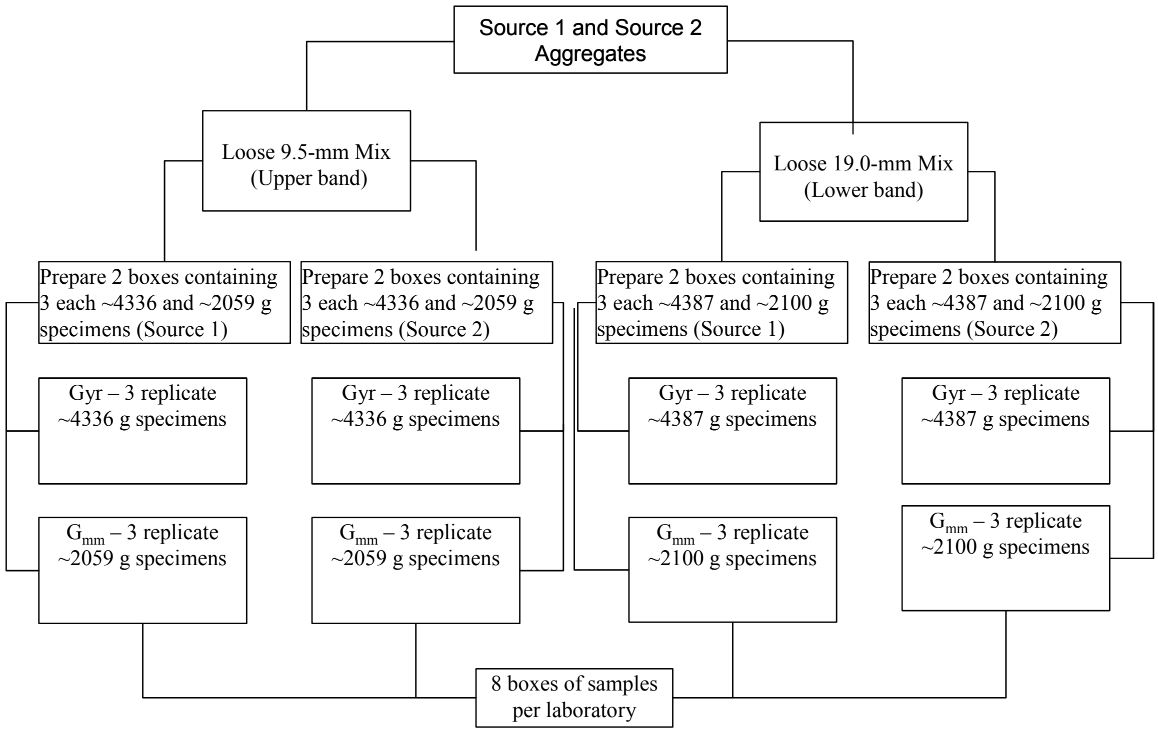

Figure 3-1- Box samples for participating laboratories

3.3.2 Test Protocols and Instruction and Data Forms for Participants

The test properties determined and the protocols followed in the study are shown in Table 3-2. The instruction and data forms shown in Appendix B 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.

Table 3-2- Test properties determined in the ILS study

Test Properties	**Test Protocols**
Maximum Specific Gravity (G_{mm})	ASTM D2041-00
Bulk Specific Gravity (G_{mb})	ASTM D6752
	AASHTO T166-00
Relative Density	AASHTO T312, N_{ini}
	AASHTO T312, N_{des}

CHAPTER 4- INTERLABORATORY TEST RESULTS AND ANALYSIS

4.1 Test Data

The data obtained in the ILS study are shown in Table 4-1 for the Source 1 mixtures and in Table 4-2 for the Source 2 mixtures. Empty cells indicate that the laboratory did not submit data. Shaded cells indicate data that were eliminated from analysis as described in Section 4.2.

4.2 Method of Analysis

Test data from this study were displayed graphically using box plots. The box plot is a graphical data analysis technique for determining if differences exist between various levels of a 1-factor model. The box plot is in fact 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.

Prior to the analysis, any partial sets of data were eliminated by following the procedures described in E691 in determining repeatability (S_r) and reproducibility (S_R) estimates of precision [3]. Data exceeding critical *h* and *k* values were eliminated as described in Sections 4.3, 4.4, and 4.5. Once identified for elimination, the same data were eliminated from any smaller subsets analyzed.

Tests for statistical significance on the ILS data were performed using *t*-test and *F*-test. All *t*-tests assumed a one-tailed t distribution for 1% level of significance. The t-test was to determine if bias in volumetric properties was statistically significant. All F-tests were also performed at 1% level of significance. The *F*-test was to determine if S_r and S_R precision estimates of the volumetric properties were significantly different.

4.3 Theoretical Maximum Specific Gravity, G_{mm}

4.3.1 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 mixture and the amount of 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 conducting weighing 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.

For mixtures containing absorptive aggregates the supplemental procedure provided in Section 11 of D2041 is to be followed. This is to prevent error in the G_{mm} measurement due to absorption of water into mixture. If the pores of the aggregates are not thoroughly sealed by the bituminous film, they may become saturated with water during the vacuum procedure. To correct this, the sample need to be surface dried that is the sample is spread on a flat tray with a nonabsorptive surface and placed in front of a fan to remove surface moisture. The agglomerations of mixture are broken by hand and the mixture is stirred occasionally for about 2 hours. The tray and sample is weighed at 15-min intervals. When the loss in mass is less than 0.05% for an interval, the sample is considered surface dried. Possible sources of error are loss of mixture particles during the dry back process and variation in mixture dryness.

Study participants were asked to determine the G_{mm} of three replicate 9.5-mm mixtures and three replicate 19.0-mm mixtures of the two aggregate sources 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. The participants were also asked to follow the dry back procedure that is explained in Section 11 of D2042-00 for mixtures containing porous aggregates.

4.3.2 Precision Estimates

Columns 2 and 3 of Table 4-1 and Table 4-2 provide G_{mm} values of the four mixtures. Twenty-five laboratories submitted full sets of G_{mm} data for S1-9.5-mm, S1-19-mm, and S2-9.5-mm mixtures. Twenty-two laboratories submitted full sets of G_{mm} data for S2-19-mm mixture. The data are displayed on box plots in Figure 4-1. Few sets of data were eliminated from the G_{mm} analysis based on h- and k-statistics (Figure 4-2). The eliminated data are shown shaded in Table 4-1 and Table 4-2. All remaining data were re-analyzed according to E691 method to determine the S_r and S_R precision estimates shown in Table 4-3. For each gradation, 9.5-mm and 19-mm, the precision estimates of Source 1 and Source 2 mixtures were then combined as presented in Table 4-4.

4.3.3 Tests for Significance

Tests for statistical significance were performed using the *t*-test and *F*-test on the precision estimates in Table 4-4. The result of the *t*-test on G_{mm} is shown in Table 4-5.

The comparison of computed and critical t values for 1% level of significance indicates that there is no significant difference in the average G_{mm} values for the 9.5-mm mixture and 19.0-mm mixtures (compare 2.292 and 2.291 in Table 4-4).

The results of *F*-test on precision estimates for G_{mm} are shown in Table 4-7. The comparison of computed and critical F values for 1% level of significance indicates that there is no significant difference in either S_r or S_R estimates of G_{mm} using 9.5-mm and 19.0-mm mixtures (compare 0.004 with 0.005 for S_r estimate and 0.009 with 0.011 for S_R estimate in Table 4-4). Therefore, the precision estimates for G_{mm} of 9.5-mm and 19.0-mm mixtures with absorptive aggregates were combined as provided in Table 4-9.

4.3.4 Comparison of Precision Estimates Using Mixtures with Absorptive Aggregates vs. Mixtures with Non-Absorptive Aggregates

The precisions estimates of G_{mm} measured from mixtures with absorptive aggregates were compared with the precision estimates for G_{mm} of mixtures with non-absorptive aggregates determined in Phase 1 of this project [1]. Table 4-9 provides the precision estimates from the two phases and Table 4-10 provides the results of an F test on the precision estimates. As shown in the tables, the S_r and S_R estimates of G_{mm} obtained in this study are significantly different from the estimates obtained in Phase 1 of the project (compare 0.005 vs. 0.002 for S_r estimate and 0.01 vs. 0.004 for S_R estimate in Table 4-9).

4.3.5 Precision Statements

Appendix C provides the proposed precision statement for D2041-00 for mixtures with absorptive aggregates. The precision statement is prepared by combining the precision estimates of 9.5-mm and 19-mm mixtures.

4.4 Bulk Specific Gravity, G_{mb}

4.4.1 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 [6]. Volumetric properties such as air voids, voids in mineral admixtures, 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 5000 g according to T312 and determine the G_{mb} of three replicates in each set of S1-9.5-mm, S1-19.0-mm, S2-9.5-mm, and S2-19.0-mm specimens according to D6752. 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 D6752, 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}.

4.4.2 Precision Estimates

4.4.2.1 T166 Test Data

Twenty-five laboratories submitted full sets of T166 data for S1-9.5-mm, S1-19-mm, and S2-9.5-mm mixtures. Twenty-two laboratories submitted full sets of data for the S2-19-mm mixture (See Table 4-1 and Table 4-2, Columns 4 & 5). The data are displayed in box plot form in Figure 4-3 . All data were analyzed according to E691 method [3]. The data from laboratory 23 were eliminated from the S1-19-mm mixture analysis based on h- and k-statistics (See Figure 4-4). All remaining data were re-analyzed according to E691 method to determine the S_r and S_R precision estimates shown in Table 4-3. For each gradation, 9.5-mm and 19-mm, the precision estimates of Source 1 and Source 2 mixtures were then combined as presented in Table 4-4.

4.4.2.2 D6752 Test Data

Twenty-five laboratories submitted full sets of D6752 data for S1-9.5-mm, S1-19-mm, and S2-9.5-mm mixtures. Twenty-three laboratories submitted full sets of data for S2-19-mm mixture (See Table 4-1 and Table 4-2, Columns 6 & 7). The data are displayed in box plot form in Figure 4-5. All data were analyzed using E691 method. Five sets of data from four laboratories were eliminated from D6752 G_{mb} analysis based on *h*- and *k*-statistics (See Figure 4-6). All remaining data were re-analyzed according to E691 method to determine the S_r and S_R precision estimates shown in Table 4-3. For each gradation, 9.5-mm and 19-mm, the precision estimates of Source 1 and Source 2 mixtures were then combined as presented in Table 4-4.

4.4.3 Tests for Significance

The *t*-test was performed to examine the bias in the measured G_{mb} properties of 9.5-mm and 19-mm mixtures measured according to T166 and D6752 test methods. The results of the *t*-test shown in Table 4-5 indicate a significant difference in the average G_{mb} values of 9.5-mm and 19.0-mm mixtures measured according to either T166 or D 6752. In both cases, the average specific gravity determined from the 9.5-mm mixtures was significantly lower than the average specific gravity determined from the 19-mm mixtures as shown in Table 4-4 (compare 2.153 vs. 2.182 using T166 and 2.133 vs. 2.149 using D6752). The bias in the G_{mb} values from the two gradations is apparent when viewing the box plots in Figure 4-3 and Figure 4-5.

A *t*-test was performed to determine if the average G_{mb} values obtained using D6752 differed significantly from average G_{mb} values obtained by T166. The results of *t*-test shown in Table 4-6 indicate significant differences in the T166 and D6752 G_{mb} values at 1% level of significance. In both cases, the average specific gravity determined from tests performed using D6752 was significantly lower than the average specific gravity determined from tests performed using T166 (compare 2.133 vs. 2.153 for 9.5-mm and 2.149 vs.2.182 for 19.0-mm in Table 4-4). The bias in test results from the two test methods is apparent when comparing the box plots in Figure 4-3 and Figure 4-5. This trend was also observed for T166 and D6752 G_{mb} values of mixtures with non-absorptive aggregates in Phase 1 of the project [1].

An F test was performed to examine the difference in the variances of G_{mb} values. The results of the F test indicate that there is a good agreement between precision estimates of 9.5-mm and 19.0-mm mixtures using either T166 or D 6753 data (compare S_r of 0.011 with 0.010 for T166 and S_r of 0.012 with 0.013 for D6752 in Table 4-4). In addition, there is a good agreement between precision estimates of T166 and D6753 using either 9.5-mm or 19-mm mixtures (compare S_R of 0.018 with 0.020 for 9.5-mm and S_R of 0.016 with 0.023 for 19-mm in Table 4-4).

4.4.4 Comparison of Precision Estimates Using Mixtures with Absorptive Aggregates vs. Mixtures with Non-Absorptive Aggregates

The precision estimates of G_{mb} were compared for mixtures with absorptive aggregates and mixtures with non-absorptive aggregates (Phase 1). Table 4-9 provides the precision estimates of the G_{mb} values and Table 4-10 provides the results of an F test on the precision estimates. As observed from the tables, S_r and S_R estimates of G_{mb} measured according to either T166 or D6752 are comparable for absorptive and non-absorptive mixtures (compare S_r of 0.010 with 0.013 using T166 and S_R of 0.023 with 0.020 using D6752 in Table 4-9).

4.4.5 Precision Statements

4.4.5.1 T 166

As indicated in 4.4.3, there is no significant difference between the repeatability and reproducibility estimates of G_{mb} measurements from the 9.5-mm and 19.0-mm mixtures (Compare S_r of 0.011 with 0.010 and S_R of 0.018 with 0.016 in Table 4-4). Therefore, it appears that it is appropriate to combine the estimates from 9.5-mm and 19-mm mixtures. Appendix D includes a proposed precision statement for T166 based on G_{mb} precision estimates of 9.5-mm and 19-mm mixtures with absorptive aggregates. Since precision estimates of mixtures with absorptive aggregates and mixtures with non-absorptive aggregates were found comparable for T166, the combining of the precision estimates from this study and the precision estimates from Phase 1 of the project is recommended.

4.4.5.2 D6752

As indicated in 4.4.3, the precision estimates for D6257 were not significantly different for 9.5-mm and 19-mm mixtures (compare S_r of 0.012 with 0.013 and S_R of 0.020 with 0.023). Therefore, it is appropriate to combine the repeatability and reproducibility estimates of G_{mb} measurements from 9.5-mm and 19-mm mixtures. Appendix E includes a draft precision statement for D6752 based on G_{mb} precision estimates of 9.5-mm and 19-mm mixtures with absorptive aggregates. Since precision estimates for D6752 using mixtures with absorptive aggregates and mixtures with non-absorptive aggregates were found to be comparable, the combining of the precision estimates from this study and the precision estimates from Phase 1 of the project is recommended.

4.5 Relative Density at N_i and N_d

4.5.1 Introduction

T312 describes a method for preparing 150-mm diameter cylindrical specimens of HMA using the Superpave gyratory compactor. The resulting specimens 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 loose mix replicates from each of S1-9.5-mm, S1-19-mm, S2-9.5-mm, and S2-19.0-mm mixtures. They were asked to prepare gyratory specimens from each of the as-received samples and determine the relative density at N_i (8 gyrations) and N_d (100 gyrations).

4.5.2 Precision Estimates

The relative density values were reported for the four mixtures at N_i and N_d computed using T166 G_{mb} and D6752 G_{mb} values. The data are provided in Table 4-1 and Table 4-2, Columns 8 through 15. The data are displayed in box plots in Figure 4-7, Figure 4-9, Figure 4-11, and Figure 4-13. All data were analyzed using ASTM E691 method. Based on *h*- and *k*-statistics of relative densities at N_i and N_d, few number of relative density data were eliminated from the analysis of each mixture (Figure 4-8, Figure 4-10, Figure 4-12, and Figure 4-14). All remaining data were re-analyzed using E691 method to determine the S_r and S_R precision estimates shown in Table 4-3. For each gradation, 9.5-mm and 19-mm, the precision estimates of Source 1 and Source 2 mixtures were then combined as presented in Table 4-4.

4.5.3 Tests for Significance

A *t*-test was performed to examine the bias in the computed relative densities at N_{in} and N_d of 9.5-mm and 19-mm mixtures using T166 G_{mb} and D6752 G_{mb} values. The results of the *t*-test shown in Table 4-5 indicate a significant difference in the average relative densities of 9.5-mm and 19.0-mm mixtures computed using either T166 G_{mb} or D 6752 G_{mb}. At both N_{in} and N_d, the relative densities determined from 9.5-mm mixtures were significantly lower than the relative densities determined from 19-mm mixtures (compare relative densities of 94.0% vs. 95.2% at N_d using T166 G_{mb} in Table 4-4). The bias in relative density values from the two gradations is apparent when viewing the box plots in Figure 4-7, Figure 4-9, Figure 4-11, and Figure 4-13.

A *t*-test was performed to determine if the average relative densities computed using D6752 G_{mb} values differed significantly from the average relative densities using T166 G_{mb} values. The *t*-test results shown in Table 4-6 indicate significant differences between relative densities computed using T166 G_{mb} and D6752 G_{mb} values for either 9.5-mm or 19.0-mm mixtures. In both cases, relative densities determined using T166 G_{mb} were significantly greater than relative densities determined using D6752 G_{mb} (compare relative densities at N_d of 95.2% using T166 G_{mb} vs. 93.9% using D6752 G_{mb} values for the 19-mm mixture in Table 4-4). The bias in relative densities from the two methods is apparent when comparing the box plots in Figure 4-7 and Figure 4-11 and the box plots in Figure 4-9 and Figure 4-13.

An F test was conducted to investigate the differences between precision estimates for relative densities of 9.5-mm and 19-mm mixtures and relative densities computed using T166 G_{mb} and D6752 G_{mb} data. It is indicated from the computed F values in Table 4-7 that at either N_{in} or N_d, the precision estimates for relative densities of 9.5-mm and 19-mm mixtures were not significantly different. Similarly, the comparison of computed and critical F values in Table 4-8 indicates that at either N_{in} or N_d, the precision estimates for relative densities computed using T166 G_{mb} and D6752 G_{mb} data were not significantly different.

4.5.4 Comparison of Precision Estimates Using Mixtures with Absorptive Aggregates vs. Mixtures with Non-Absorptive Aggregates

The precisions estimates for relative densities measured from mixtures with absorptive aggregates were compared with the precision estimates for relative densities of mixtures with non-absorptive aggregates obtained in Phase 1 of the project [1]. Table 4-9 provides the precision estimates from the two phases and Table 4-10 provides the result of an F test on the precision estimates. As observed from the tables, the S_r and S_R estimates of relative density for mixtures with absorptive aggregates are significantly greater than the precision estimates for mixtures with non-absorptive aggregates. Since relative density is calculated from G_{mm} and G_{mb} values, the larger precision estimates for relative density of mixtures with absorptive aggregates is caused by the larger precision estimates for G_{mm} and G_{mb} of mixtures with absorptive aggregates as compared with G_{mm} and G_{mb} of mixtures with non-absorptive aggregates (see Table 4-9).

4.5.5 Precision Statement

As indicated above, there is a significant difference between repeatability and reproducibility estimates for relative density measurements of mixtures with absorptive aggregates and mixtures with non-absorptive aggregates. Therefore, a separate precision statement for T312 associated with relative density of mixtures with absorptive aggregates is suggested. Appendix F includes the proposed precision statement for relative density of mixtures with absorptive aggregates. The proposed estimates were prepared by combining the estimates for relative densities at N_{in} and N_d of 9.5-mm and 19-mm mixtures, computed using T166 G_{mb} and D6742 G_{mb} data.

Table 4-1- Volumetric properties of three replicates of Source 1 mixtures in the ILS study

	Gmm, D2041	Gmm, D2041	Gmb, T166	Gmb, T166	Gmb, D6752	Gmb, D6752	T166 Rel. Density Nin, T312	T166 Rel. Density Nin, T312	T166 Rel. Density Nd, T312	T166 Rel. Density Nd, T312	D6752 Rel. Density Nin, T312	D6752 Rel. Density Nin, T312	D6752 Rel. Density Nd, T312	D6752 Rel. Density Nd, T312
Lab No	S1- 9.5mm	S1- 19mm	S1- 9.5mm	S1- 19mm	S1- 9.5mm	S1- 19mm	S1- 9.5mm	S1- 19mm	S1- 9.5mm	S1- 19mm	S1- 9.5mm	S1- 19mm	S1- 9.5mm	S1 - 19mm
1	2.276	2.258	2.156	2.191	2.137	2.171			94.7	97.0			93.9	96.1
	2.277	2.276	2.168	2.177	2.156	2.158			95.2	95.7			94.7	94.8
	2.276	2.273	2.176	2.175	2.162	2.154			95.6	95.7			95.0	94.7
2	2.282		2.164		2.154		84.5		94.8		84.2		94.4	
	2.278		2.173		2.164		84.5		95.4		84.2		95.0	
	2.283		2.189		2.181		84.6		95.9		84.3		95.5	
3	2.287	2.263	2.179	2.184	2.166	2.162	83.7	85.2	95.3	96.5	83.2	84.3	94.7	95.5
	2.295	2.276	2.185	2.226	2.171	2.208	83.6	86.5	95.2	97.8	83.1	85.8	94.6	97.0
	2.291	2.267	2.181	2.188	2.169	2.170	83.6	85.6	95.2	96.5	83.1	84.9	94.7	95.7
4	2.286	2.289	2.153	2.168	2.136	2.068	83.4	84.4	94.2	94.7	82.7	80.5	93.4	90.3
	2.298	2.287	2.152	2.177	2.133	2.147	82.9	84.9	93.6	95.2	82.1	83.7	92.8	93.9
	2.283	2.282	2.168	2.178	2.153	2.151	84.1	84.9	94.9	95.4	83.5	83.8	94.3	94.2
5		2.250		2.161		2.134		85.3		96.0		84.2		94.8
		2.250		2.188		2.149		86.5		97.2		85.0		95.5
		2.264		2.178		2.154		85.7		96.2		84.7		95.1
6	2.286	2.274	2.164	2.192	2.142	2.162	83.4	85.4	94.7	96.4	82.5	84.2	93.7	95.1
	2.286	2.276	2.166	2.179	2.142	2.153	83.2	84.8	94.7	95.7	82.3	83.8	93.7	94.6
	2.292	2.283	2.173	2.188	2.155	2.157	83.3	84.7	94.8	95.8	82.6	83.6	94.0	94.5
7	2.269	2.276	2.189	2.187	2.166	2.160	84.4	86.1	96.5	96.1	83.6	85.0	95.4	94.9
	2.283	2.275	2.183	2.197	2.166	2.166	83.7	85.0	95.6	96.6	83.0	83.8	94.9	95.2
	2.281	2.281	2.184	2.184	2.164	2.153	83.8	84.4	95.7	95.7	83.0	83.2	94.8	94.4
8	2.284	2.287	2.160	2.166	2.140	2.133	83.6	83.0	94.5	94.7	82.8	81.7	93.7	93.3
	2.283	2.290	2.154	2.175	2.134	2.142	83.3	84.5	94.3	95.0	82.6	83.2	93.5	93.5
	2.289	2.290	2.160	2.201	2.138	2.173	83.5	84.9	94.3	96.1	82.7	83.8	93.4	94.9
9	2.282	2.284	2.183	2.178	2.148	2.158	84.5	85.1	95.6	95.4	83.2	84.3	94.1	94.5
	2.287	2.288	2.152	2.193	2.128	2.171	82.9	85.4	94.1	95.8	82.0	84.5	93.0	94.9
	2.285	2.289	2.154	2.176	2.135	2.157	83.6	84.8	94.3	95.1	82.9	84.1	93.4	94.2
10	2.274	2.273	2.177	2.197	2.164	2.172	84.3	85.5	95.7	96.6	83.8	84.6	95.1	95.5
	2.279	2.269	2.178	2.181	2.165	2.162	84.0	86.0	95.6	96.1	83.5	85.3	95.0	95.3
	2.284	2.270	2.166	2.194	2.152	2.166	83.5	85.6	94.8	96.7	82.9	84.5	94.2	95.4
11	2.287	2.280	2.170	2.200	2.144	2.177	83.6	85.4	94.9	96.5	82.6	84.5	93.7	95.5
	2.283	2.282	2.187	2.169	2.161	2.064	84.2	84.2	95.8	95.0	83.2	80.1	94.7	90.4
	2.282	2.268	2.149	2.174	2.126	2.154	83.2	85.1	94.2	95.8	82.3	84.3	93.1	95.0
12	2.286	2.282	2.173	2.198	2.165	2.183	83.4	85.5	95.1	96.3	83.1	84.9	94.7	95.6
	2.296	2.294	2.165	2.184	2.154	2.166	82.9	84.4	94.3	95.2	82.5	83.7	93.8	94.4
	2.288	2.292	2.190	2.174	2.178	2.157	84.0	83.9	95.7	94.9	83.5	83.3	95.2	94.1
13	2.274	2.276	2.165	2.181	2.162	2.157	83.6	84.6	95.2	95.8	83.5	83.7	95.1	94.8
	2.281	2.265	2.177	2.194	2.161	2.167	83.9	85.3	95.4	96.8	83.3	84.3	94.7	95.7
	2.271	2.264	2.172	2.194	2.161	2.170	84.2	85.2	95.6	96.9	83.8	84.3	95.2	95.8
14	2.288	2.284	2.160	2.191	2.148	2.176	83.1	84.8	94.4	95.9	82.6	84.2	93.9	95.2
	2.279	2.275	2.159	2.187	2.143	2.160	82.8	83.9	94.7	96.1	82.2	82.9	94.0	94.9
	2.278	2.280	2.176	2.179	2.200	2.160	83.2	84.2	95.5	95.5	84.1	83.4	96.6	94.7
15	2.292	2.289	2.200	2.220	2.184	2.196	84.0	85.5	96.0	97.0	83.3	84.6	95.3	95.9
	2.290	2.286	2.184	2.199	2.167	2.176	83.6	84.3	95.4	96.2	83.0	83.4	94.6	95.2
	2.289	2.287	2.193	2.205	2.176	2.185	84.0	84.8	95.8	96.4	83.4	84.0	95.1	95.5
16	2.326	2.302	2.124	2.178	2.082	2.095	80.4	84.0	91.3	94.6	78.8	80.8	89.5	91.0
	2.326	2.311	2.139	2.146	2.101	2.099	81.1	82.5	91.9	92.8	79.7	80.7	90.3	90.8
	2.322	2.317	2.135	2.164	2.099	2.121	81.4	83.1	91.9	93.4	80.1	81.4	90.4	91.5
17	2.286	2.278	2.143	2.178	2.114	2.135	83.3	85.4	93.7	95.6	82.1	83.7	92.5	93.7
	2.282	2.283	2.150	2.176	2.113	2.127	83.6	85.0	94.2	95.3	82.2	83.1	92.6	93.2
	2.285	2.283	2.168	2.163	2.131	2.112	84.3	84.4	94.9	94.7	82.8	82.4	93.2	92.5
18	2.273	2.265	2.165	2.192	2.152	2.172	83.9	85.5	95.2	96.8	83.3	84.7	94.7	95.9
	2.277	2.268	2.165	2.187	2.153	2.167	83.5	85.2	95.1	96.4	83.1	84.4	94.6	95.5
	2.277	2.270	2.171	2.169	2.156	2.141	83.8	84.6	95.3	95.5	83.2	83.5	94.7	94.3
19	2.304	2.277	2.171	2.201	2.152	2.177	83.5	85.5	94.2	96.6	82.7	84.6	93.4	95.6
	2.306	2.268	2.168	2.198	2.143	2.166	82.7	85.9	94.0	96.9	81.8	84.6	92.9	95.5
	2.294	2.301	2.174	2.185	2.155	2.151	83.5	83.8	94.8	95.0	82.7	82.5	93.9	93.5
20		2.262		2.177		2.148		86.2		96.2		85.1		95.0
		2.266		2.170		2.146		85.7		95.7		84.8		94.7
		2.257		2.165		2.139		85.8		95.9		84.7		94.7
21	2.277	2.266	2.172	2.173	2.158	2.150	85.0	85.5	95.4	95.9	84.5	84.6	94.8	94.9
	2.273	2.267	2.173	2.178	2.164	2.157	84.9	85.8	95.6	96.1	84.6	84.9	95.2	95.1
	2.277	2.268	2.146	2.171	2.137	2.152	83.6	85.6	94.2	95.7	83.2	84.8	93.8	94.9
22	2.272	2.272	2.160	2.183	2.136	2.156	84.3	86.1	95.1	96.1	83.4	85.0	94.0	94.9
	2.275	2.258	2.165	2.176	2.147	2.138	84.3	85.7	95.2	96.4	83.5	84.2	94.4	94.7
	2.271	2.262	2.157	2.170	2.135	2.137	84.1	85.6	95.0	95.9	83.2	84.3	94.0	94.5
23	2.286	2.284	2.147	2.133	2.127	2.102	83.8	83.1	93.9	93.4	83.0	81.9	93.0	92.0
	2.276	2.268	2.148	2.142	2.127	2.105	83.7	84.0	94.4	94.4	82.8	82.6	93.4	92.8
	2.287	2.265	2.153	2.150	2.130	2.117	83.5	84.8	94.1	94.9	82.6	83.5	93.1	93.5
24	2.269	2.254	2.165	2.184	2.150	2.159	84.2	86.4	95.4	96.9	83.7	85.4	94.7	95.8
	2.281	2.271	2.157	2.167	2.145	2.142	83.2	84.8	94.5	95.4	82.8	83.9	94.0	94.3
	2.273	2.269	2.172	2.186	2.161	2.158	84.2	85.2	95.5	96.3	83.8	84.1	95.1	95.1
25	2.283	2.279	2.140	2.170	2.123	2.148	82.7	84.5	93.7	95.2	82.1	83.7	93.0	94.2
	2.281	2.266	2.154	2.181	2.137	2.158	83.3	85.6	94.4	96.2	82.7	84.7	93.7	95.2
	2.279	2.288	2.151	2.161	2.135	2.137	83.3	84.0	94.4	94.4	82.7	83.0	93.7	93.4
26	2.271		2.138		2.111		83.6		94.1		82.5		93.0	
	2.260		2.156		2.136		84.6		95.4		83.8		94.5	
	2.270		2.153		2.130		84.5		94.8		83.6		93.8	
27	2.285	2.280	2.140	2.151	2.126	2.128	82.8	84.0	93.6	94.3	82.3	83.1	93.0	93.3
	2.290	2.283	2.145	2.144	2.131	2.121	82.8	83.4	93.7	93.9	82.2	82.6	93.0	92.9
	2.290	2.292	2.151	2.152	2.135	2.135	83.1	83.4	93.9	93.9	82.4	82.7	93.2	93.1

Table 4-2- Volumetric properties of three replicates of Source 2 mixtures in the ILS Study

	Gmm, D2041	Gmm, D2041	Gmb, T166	Gmb, T166	Gmb, D6752	Gmb, D6752	T166 Rel. Density Nin, T312	T166 Rel. Density Nin, T312	T166 Rel. Density Nd, T312	T166 Rel. Density Nd, T312	D6752 Rel. Density Nin, T312	D6752 Rel. Density Nin, T312	D6752 Rel. Density Nd, T312	D6752 Rel. Density Nd, T312
Lab No	S2- 9.5mm	S2- 19mm	S2- 9.5mm	S2- 19mm	S2- 9.5mm	S2- 19mm	S2- 9.5mm	S2- 19mm	S2- 9.5mm	S2- 19mm	S2- 9.5mm	S2- 19mm	S2- 9.5mm	S2- 19mm
1	2.293	2.312	2.141	2.179	2.123	2.146	82.9	83.9	93.4	94.2	82.2	82.6	92.6	92.8
	2.291	2.308	2.090	2.180	2.110	2.145	81.1	84.3	91.2	94.5	81.8	82.9	92.1	92.9
	2.298	2.304	2.127	2.193	2.100	2.156	82.3	85.2	92.6	95.2	81.2	83.8	91.4	93.6
2														
3	2.296	2.313	2.146	2.198	2.130	2.169	82.5	84.5	93.5	95.0	81.9	83.4	92.8	93.8
	2.302	2.305	2.169	2.208	2.137	2.181	83.4	84.7	94.2	95.8	82.2	83.7	92.8	94.6
	2.306	2.305	2.150	2.205	2.132	2.183	82.1	84.9	93.2	95.7	81.4	84.0	92.5	94.7
4	2.302	2.307	2.136	2.166	2.106	2.113	82.5	84.3	92.8	93.9	81.4	82.2	91.5	91.6
	2.299	2.305	2.131	2.164	2.095	2.125	82.5	84.3	92.7	93.9	81.2	82.8	91.1	92.2
	2.304	2.305	2.163	2.179	2.139	2.143	83.7	84.7	93.9	94.5	82.8	83.3	92.8	93.0
5	2.282	2.268	2.132	2.173	2.124	2.149	82.8	85.7	93.4	95.8	82.5	84.8	93.1	94.7
	2.297	2.286	2.132	2.191	2.115	2.153	82.5	85.5	92.8	95.8	81.9	84.1	92.1	94.2
	2.298	2.289	2.136	2.198	2.113	2.170	82.6	85.8	92.9	96.0	81.8	84.7	91.9	94.8
6	2.301	2.309	2.157	2.202	2.137	2.151	82.8	84.8	93.7	95.3	82.0	82.8	92.9	93.1
	2.298	2.305	2.157	2.206	2.127	2.157	82.8	85.2	93.8	95.7	81.6	83.3	92.6	93.6
	2.297	2.304	2.147	2.192	2.118	2.133	82.5	84.5	93.5	95.1	81.4	82.2	92.2	92.6
7	2.308	2.305	2.171	2.200	2.160	2.171		84.9	94.1	95.4		83.8	93.6	94.2
	2.304	2.307	2.175	2.181	2.157	2.120		84.0	94.4	94.5		81.6	93.6	91.9
	2.307	2.307	2.156	2.203	2.130	2.171		84.6	93.4	95.5		83.3	92.3	94.1
8	2.315	2.310	2.128	2.181	2.097	2.141	81.7	84.1	91.9	94.4	80.5	82.5	90.6	92.7
	2.310	2.308	2.145	2.187	2.115	2.144	82.4	84.4	92.9	94.7	81.3	82.7	91.6	92.9
	2.312	2.320	2.172	2.164	2.142	2.122	83.4	83.1	93.9	93.3	82.3	81.5	92.6	91.5
9	2.303	2.314	2.140	2.166	2.118	2.131	82.3	83.7	92.9	93.6	81.5	82.3	92.0	92.1
	2.302	2.309	2.131	2.180	2.110	2.142	82.1	98.6	92.6	94.4	81.3	96.8	91.7	92.8
	2.304	2.310	2.121	2.167	2.097	2.132	81.7	83.8	92.1	93.8	80.8	82.5	91.0	92.3
10	2.294	2.297	2.170	2.203	2.157	2.168	83.3	85.1	94.6	95.9	82.8	83.7	94.0	94.4
	2.295	2.295	2.159	2.195	2.140	2.169	82.9	85.2	94.1	95.6	82.2	84.3	93.2	94.5
	2.294	2.301	2.176	2.197	2.161	2.172	83.7	84.6	94.8	95.5	83.1	83.7	94.2	94.4
11	2.308		2.160		2.170		82.8		93.6		83.2		94.0	
	2.306		2.166		2.144		83.0		93.9		82.1		93.0	
	2.307		2.142		2.053		82.2		92.8		78.8		89.0	
12	2.308	2.315	2.168	2.185	2.153	2.157	82.6	83.5	93.9	94.4	82.0	82.4	93.3	93.2
	2.311	2.319	2.149	2.196	2.130	2.160	81.9	84.1	93.0	94.7	81.1	82.8	92.1	93.1
	2.308	2.323	2.154	2.168	2.130	2.133	82.2	83.1	93.3	93.3	81.3	81.7	92.3	91.8
13	2.306	2.300	2.164	2.198	2.146	2.164		83.6	93.8	95.5		82.3	93.1	94.1
	2.306	2.308	2.136	2.199	2.117	2.189		84.7	92.6	95.3		84.3	91.8	94.8
	2.303	2.318	2.144	2.202	2.126	2.178		84.3	93.1	95.0		83.4	92.3	94.0
14	2.310		2.147		2.137		81.6		92.9		81.2		92.5	
	2.312		2.133		2.085		81.1		92.3		79.2		90.2	
	2.313		2.129		2.113		80.6		92.0		80.0		91.4	
15	2.308	2.313	2.173	2.212	2.151	2.182	82.8	84.8	94.2	95.6	81.9	83.6	93.2	94.3
	2.306	2.320	2.186	2.189	2.168	2.157	83.0	83.9	94.8	94.4	82.3	82.7	94.0	93.0
	2.306	2.318	2.164	2.205	2.142	2.172	82.3	84.2	93.8	95.1	81.5	83.0	92.9	93.7
16	2.312		2.149		2.130	2.123	84.8		92.9		84.0		92.1	
	2.309		2.126		2.094	2.140	81.4		92.1		80.2		90.7	
	2.308		2.118		2.088	2.083	81.3		91.8		80.2		90.5	
17	2.292	2.315	2.136	2.183	2.100	2.125	83.1	84.5	93.2	94.3	81.7	82.2	91.6	91.8
	2.288	2.316	2.144	2.181	2.120	2.128	83.2	84.7	93.7	94.2	82.3	82.6	92.6	91.9
	2.294	2.314	2.140	2.162	2.103	2.097	83.3	84.1	93.3	93.4	81.8	81.6	91.7	90.6
18	2.307	2.310	2.135	2.198	2.110	2.167	81.9	84.6	92.5	95.1	81.0	83.4	91.4	93.8
	2.311	2.307	2.135	2.178	2.109	2.150	81.8	84.1	92.4	94.4	80.8	83.0	91.3	93.2
	2.310	2.311	2.141	2.190	2.115	2.152	81.8	84.6	92.7	94.7	80.8	83.1	91.5	93.1
19	2.284	2.278	2.171	2.184	2.148	2.150	84.6	85.4	95.0	95.9	83.7	84.1	94.0	94.4
	2.286	2.275	2.137	2.173	2.110	2.126	82.4	85.5	93.5	95.5	81.4	83.6	92.3	93.5
	2.288	2.281	2.142	2.202	2.115	2.167	82.6	85.7	93.6	96.5	81.6	84.4	92.4	95.0
20														
21	2.300	2.299	2.135	2.179	2.111	2.167	82.6	85.0	92.8	94.8	81.6	84.5	91.8	94.2
	2.308	2.322	2.126	2.171	2.103	2.145	82.2	83.5	92.1	93.5	81.3	82.5	91.1	92.4
	2.297	2.321	2.124	2.166	2.103	2.128	82.4	84.1	92.5	93.3	81.6	82.6	91.6	91.7
22	2.301	2.303	2.099	2.184	2.079	2.146	81.7		91.2	94.8	80.9		90.4	93.2
	2.305	2.303	2.111	2.173	2.077	2.128	82.0		91.6	94.4	80.6		90.1	92.4
	2.290	2.294	2.129	2.173	2.105	2.132	82.8		93.0	94.7	81.9		91.9	92.9
23	2.301	2.304	2.109	2.163	2.073	2.119	81.6		91.6	93.9	80.2		90.1	91.9
	2.301	2.309	2.111	2.162	2.080	2.118	82.0		91.7	93.6	80.8		90.4	91.7
	2.299	2.311	2.116	2.149	2.083	2.106	82.0		92.0	93.0	80.7		90.6	91.1
24	2.300	2.312	2.131	2.181	2.102	2.147	82.0	84.1	92.6	94.3	80.8	82.8	91.4	92.8
	2.296	2.308	2.144	2.173	2.122	2.138	82.8	83.8	93.4	94.1	81.9	82.5	92.4	92.6
	2.302	2.309	2.132	2.186	2.102	2.156	81.9	84.1	92.6	94.7	80.8	83.0	91.3	93.4
25	2.292	2.308	2.140	2.165	2.109	2.114	82.7	83.6	93.3	93.8	81.5	81.6	92.0	91.6
	2.289	2.301	2.130	2.183	2.103	2.148	82.4	84.6	93.1	94.9	81.3	83.2	91.9	93.3
	2.291	2.310	2.136	2.183	2.107	2.149	82.5	84.0	93.2	94.5	81.4	82.7	91.9	93.0
26	2.291	2.292	2.128	2.186	2.103	2.147	82.8	85.5	92.9	95.4	81.8	84.0	91.8	93.7
	2.282	2.294	2.148	2.191	2.127	2.159	83.9	85.6	94.1	95.5	83.1	84.4	93.2	94.1
	2.275	2.297	2.129	2.192	2.101	2.158	83.3	85.6	93.6	95.4	82.2	84.3	92.3	93.9
27	2.306	2.315	2.134	2.161	2.119	2.131	82.1	83.4	92.5	93.3	81.5	82.2	91.9	92.0
	2.302	2.309	2.139	2.140	2.120	2.110	82.5	83.2	92.9	92.7	81.8	82.0	92.1	91.4
	2.307	2.313	2.132	2.170	2.116	2.134	82.1	83.8	92.4	93.8	81.5	82.4	91.7	92.3

Table 4-3- Precision estimates of volumetric properties of the four mixtures

Property	Source-Design	No. of Labs	Average	S_x	Repeatability 1s (S_r)	Repeatability D2s	Reproducibility 1s (S_r)	Reproducibility D2s
Gmm, D2041	S1- 9.5mm	24	2.282	0.008	0.004	0.012	0.009	0.024
Gmm, D2041	S2- 9.5mm	24	2.301	0.008	0.003	0.009	0.009	0.024
Gmm, D2041	S1- 19mm	23	2.275	0.010	0.006	0.017	0.011	0.032
Gmm, D2041	S2- 19mm	20	2.307	0.009	0.005	0.013	0.010	0.027
Gmb, T166	S1- 9.5mm	25	2.164	0.013	0.009	0.026	0.016	0.045
Gmb, T166	S2-9.5mm	25	2.141	0.016	0.012	0.034	0.020	0.056
Gmb, T166	S1-19mm	24	2.181	0.012	0.011	0.031	0.016	0.045
Gmb, T166	S2-19mm	22	2.183	0.013	0.010	0.027	0.016	0.046
Gmb, D6752	S1-9.5mm	23	2.148	0.015	0.009	0.026	0.018	0.050
Gmb, D6752	S2-9.5mm	24	2.118	0.018	0.014	0.039	0.023	0.064
Gmb, D6752	S1-19mm	23	2.152	0.020	0.011	0.032	0.023	0.064
Gmb, D6752	S2-19mm	23	2.145	0.019	0.015	0.041	0.023	0.066
T166 Rel. Density N_{in}, T312	S1-9.5mm	23	83.7	0.44	0.42	1.17	0.60	1.68
T166 Rel. Density N_{in}, T312	S2-9.5mm	21	82.5	0.46	0.52	1.45	0.68	1.92
T166 Rel. Density N_{in}, T312	S1-19mm	24	84.9	0.72	0.62	1.74	0.94	2.63
T166 Rel. Density N_{in}, T312	S2-19mm	19	84.5	0.62	0.42	1.18	0.75	2.09
T166 Rel. Density N_d, T312	S1-9.5mm	24	94.9	0.54	0.49	1.36	0.72	2.02
T166 Rel. Density N_d, T312	S2-9.5mm	25	93.1	0.71	0.56	1.57	0.90	2.52
T166 Rel. Density N_d, T312	S1-19mm	25	95.7	0.82	0.61	1.71	1.01	2.83
T166 Rel. Density N_d, T312	S2-19mm	22	94.7	0.78	0.47	1.33	0.90	2.53
D6752 Rel. Density N_{in}, T312	S1-9.5mm	23	83.0	0.51	0.46	1.29	0.68	1.91
D6752 Rel. Density N_{in}, T312	S2-9.5mm	21	81.6	0.60	0.58	1.63	0.82	2.30
D6752 Rel. Density N_{in}, T312	S1-19mm	22	83.9	0.74	0.76	2.14	1.05	2.94
D6752 Rel. Density N_{in}, T312	S2-19mm	19	83.1	0.70	0.61	1.70	0.92	2.57
D6752 Rel. Density N_d, T312	S1-9.5mm	23	94.1	0.71	0.48	1.34	0.85	2.37
D6752 Rel. Density N_d, T312	S2-9.5mm	24	92.0	0.60	0.66	1.85	1.04	2.92
D6752 Rel. Density N_d, T312	S1-19mm	22	94.7	0.82	0.61	1.72	1.02	2.85
D6752 Rel. Density N_d, T312	S2-19mm	22	93.1	0.93	0.66	1.85	1.13	3.18

Table 4-4- Combined statistics of volumetric properties of Source 1 and Source 2 mixtures

Property	Design	Average	S_x	Repeatability 1s (S_r)	Repeatability D2s	Reproducibility 1s (S_R)	Reproducibility D2s
Gmm, D2041	9.5 mm	2.292	0.008	0.004	0.011	0.009	0.024
	19 mm	2.290	0.009	0.005	0.015	0.011	0.030
Gmb, T166	9.5 mm	2.153	0.015	0.011	0.030	0.018	0.051
	19 mm	2.182	0.013	0.010	0.029	0.016	0.046
Gmb, D6752	9.5 mm	2.132	0.017	0.012	0.033	0.021	0.058
	19 mm	2.149	0.019	0.013	0.037	0.023	0.065
T166 Rel. Density N_{in}, T312	9.5 mm	83.1	0.45	0.47	1.32	0.64	1.80
	19 mm	84.7	0.67	0.53	1.49	0.85	2.38
T166 Rel. Density N_d, T312	9.5 mm	94.0	0.63	0.52	1.47	0.82	2.28
	19 mm	95.2	0.80	0.55	1.53	0.96	2.69
D6752 Rel. Density $N_{in,}$ T312	9.5 mm	82.3	0.56	0.52	1.47	0.76	2.12
	19 mm	83.6	0.72	0.69	1.93	0.98	2.76
D6752 Rel. Density $N_{d,}$ T312	9.5 mm	93.1	0.65	0.58	1.61	0.95	2.66
	19 mm	93.9	0.88	0.64	1.78	1.08	3.02

Table 4-5- Results of t-test on bias of the volumetric properties of 9.5-mm and 19-mm mixtures

Compare 9.5 mm & 19.0 mm	Degrees of freedom	Critical t	Computed t	Decision
G_{mm}	89	2.369	0.49	Accept
G_{mb}- T166	94	2.367	10.28	Reject
G_{mb}- D6752	91	2.368	4.21	Reject
Rel. Density@ N_{in-} T166	85	2.371	13.02	Reject
Rel. Density@ N_{in-} D6752	94	2.367	8.37	Reject
Rel. Density@ N_d- T166	83	2.372	8.84	Reject
Rel. Density@ N_{d-} D6752	89	2.369	5.09	Reject

Note: The critical t values are for 99% level of confidence for one tailed test.

Table 4-6- Results of t-test on bias of the volumetric properties measured according to T166 and D6752

Compare T166 & D6752	Degrees of freedom	Critical t	Computed t	Decision
G_{mb}- 9.5 mm	95	2.366	6.23	Reject
G_{mb}- 19 mm	90	2.368	9.78	Reject
Rel. Density@ N_{in-} 9.5 mm	86	2.370	7.64	Reject
Rel. Density@ N_{in-} 19 mm	82	2.373	7.82	Reject
Rel. Density@ N_d- 9.5mm	94	2.367	6.87	Reject
Rel. Density@ N_{d-} 19 mm	89	2.369	7.41	Reject

Note: The critical t values are for 99% level of confidence for one tailed test.

Table 4-7- Results of F test for comparison of precision estimates for volumetric properties of 9.5-mm and 19-mm mixtures

Compare 9.5 mm & 19.0 mm	Degrees of freedom	Critical F	S_r Computed F	S_r Decision	S_R Computed F	S_R Decision
G_{mm}	42 & 47	2.073	1.908	Accept	1.499	Accept
G_{mb}- T166	49 & 45	2.015	1.096	Accept	1.258	Accept
G_{mb}- D6752	45 & 46	2.046	1.234	Accept	1.272	Accept
Rel. Density@ N_{in}- T166	42 & 43	2.089	1.267	Accept	1.737	Accept
Rel. Density@ N_{in}- D6752	46 & 48	2.021	1.088	Accept	1.386	Accept
Rel. Density@ N_d- T166	40 & 43	2.104	1.733	Accept	1.700	Accept
Rel. Density@ N_d- D6752	43 & 46	2.067	1.220	Accept	1.287	Accept

Note: The critical F values stand for 99% level of confidence.

Table 4-8- Results of F test for comparison of the precision estimates of T166 and D6752

Compare T166 & D6752	Degrees of freedom	Critical F	S_r Computed F	S_r Decision	S_R Computed F	S_R Decision
G_{mb}- 9.5 mm	46 & 49	2.013	1.206	Accept	1.277	Accept
G_{mb}- 19 mm	45 & 45	2.053	1.633	Accept	2.044	Accept
Rel. Density@ N_{in}- 9.5 mm	43 & 43	2.081	1.234	Accept	1.377	Accept
Rel. Density@ N_{in}- 19 mm	40 & 42	2.106	1.687	Accept	1.347	Accept
Rel. Density@ N_d- 9.5mm	46 & 48	2.021	1.207	Accept	1.357	Accept
Rel. Density@ N_d- 19 mm	43 & 46	2.067	1.354	Accept	1.260	Accept

Note: The critical F values stand for 99% level of confidence.

Table 4-9- Precision estimates of selective volumetric properties using mixtures with absorptive aggregates and mixtures with non-absorptive aggregates; "Finer" mixtures represent 9.5-mm in Phase 4 and 12.0-mm in Phase 1 studies and "coarser" mixtures represent 19-mm mixtures

Property	Mixtures	Phase 4 (Absorptive)				Phase 1 (Non-Absorptive)			
		Repeatability		Reproducibility		Repeatability		Reproducibility	
		1s (S_r)	D2s	1s (S_R)	D2s	1s (S_r)	D2s	1s (S_R)	D2s
G_{mm} (D2041)	Finer	0.004	0.011	0.009	0.024	0.002	0.006	0.004	0.011
	Coarser	0.005	0.015	0.011	0.03				
	Combined	0.005	0.013	0.010	0.027				
G_{mb} (T166)	Finer	0.011	0.030	0.018	0.051	0.008	0.023	0.015	0.042
	Coarser	0.010	0.029	0.016	0.046	0.013	0.037	0.015	0.042
	Combined	0.011	0.030	0.017	0.048				
G_{mb} (D6752)	Finer	0.012	0.033	0.020	0.057	0.011	0.030	0.020	0.056
	Coarser	0.013	0.037	0.023	0.065	0.015	0.043	0.020	0.056
	Combined	0.012	0.035	0.022	0.061				
Rel. Densities @ N_{in} & N_d (T312)	Finer	0.520	1.457	0.787	2.205	0.300	0.900	0.600	1.700
	Coarser	0.596	1.669	0.965	2.702	0.500	1.400	0.600	1.700
	Combined	0.558	1.563	0.876	2.453				

Table 4-10- Results of F test for comparing precision estimates of volumetric properties of mixtures with absorptive aggregates (Phase 4) and mixtures with non-absorptive aggregates (Phase 1)

Compare Phase 4 & Phase 1	Degrees of freedom	Critical F	S_r		S_R	
			Computed F	Decision	Computed F	Decision
G_{mm}	47 & 24	2.450	5.652	Reject	7.466	Reject
G_{mb}- T166 (9.5-mm & 12.5-mm)	49 & 24	2.443	1.837	Accept	1.477	Accept
G_{mb}- T166 (19-mm)	45 & 23	2.505	1.576	Accept	1.348	Accept
G_{mb}- D6752 (9.5-mm & 12.5-mm)	46 & 19	2.736	1.172	Accept	1.176	Accept
G_{mb}- D6752 (19-mm)	45 & 19	2.730	1.285	Accept	1.225	Accept
Rel. Density (9.5-mm & 12.5-mm)	183 & 49	1.780	3.065	Reject	1.772	Accept
Rel. Density (19-mm)	174 & 48	1.810	1.461	Accept	2.619	Reject

Note: The critical F values stand for 99% level of confidence.

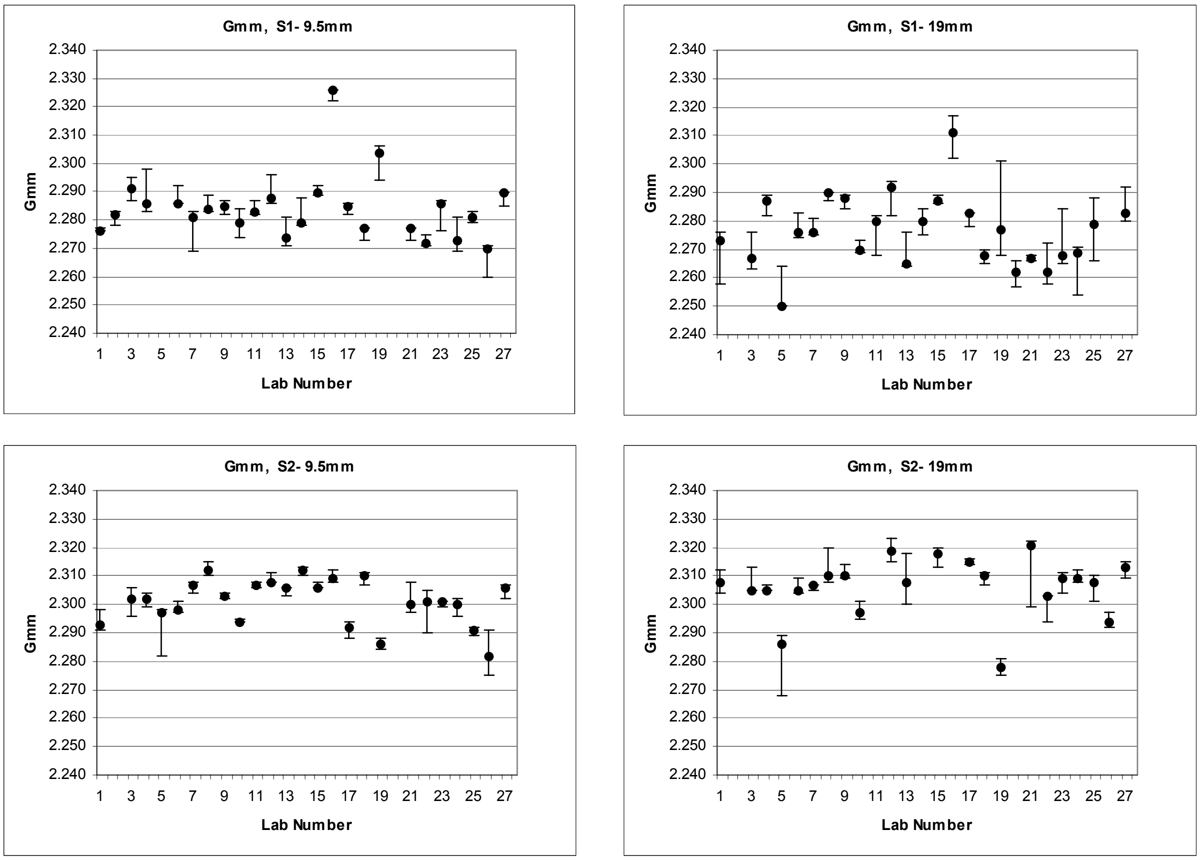

Figure 4-1- Box plots of maximum specific gravity (G_{mm}) -D2041

Figure 4-2- h and k consistency statistics of maximum specific gravity (G_{mm})-D2041

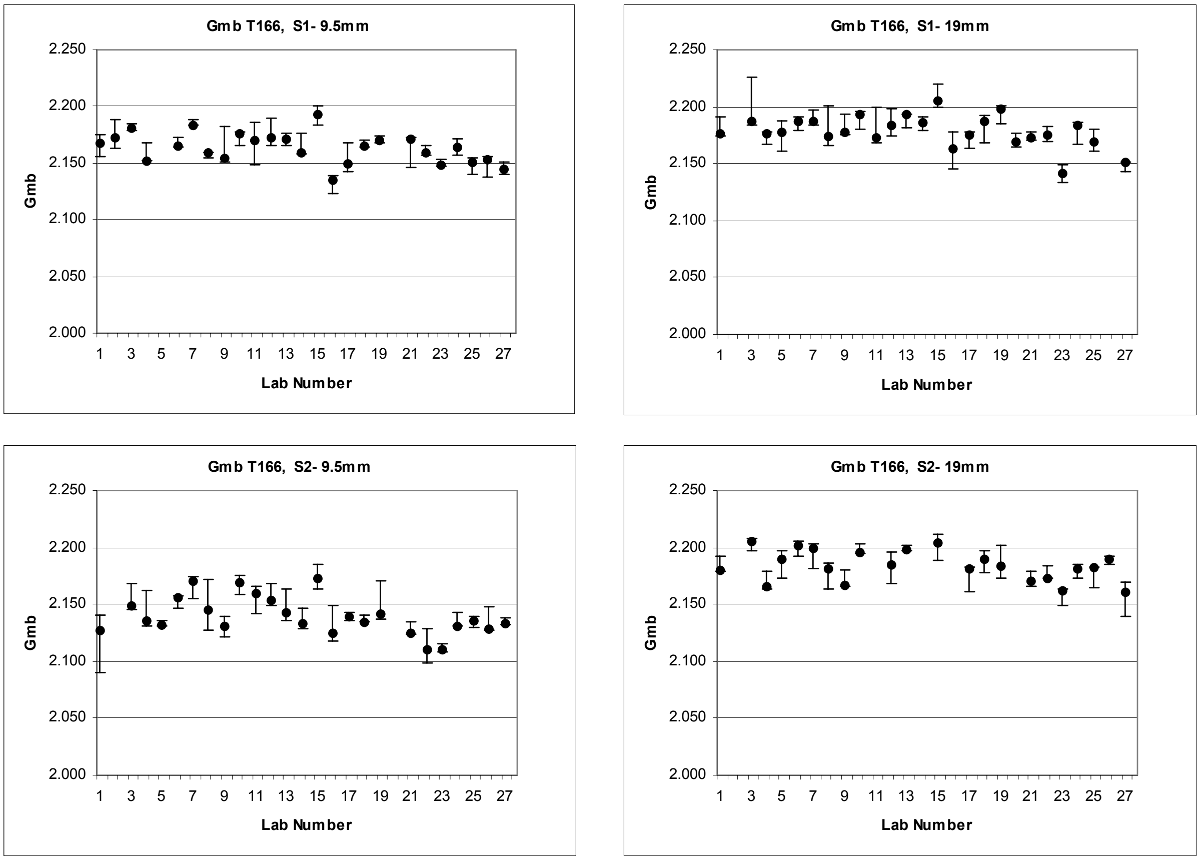

Figure 4-3- Box plots of bulk specific gravity (G_{mb})-T166

Figure 4-4- h and k consistency statistics of bulk specific gravity (G_{mb})-T166

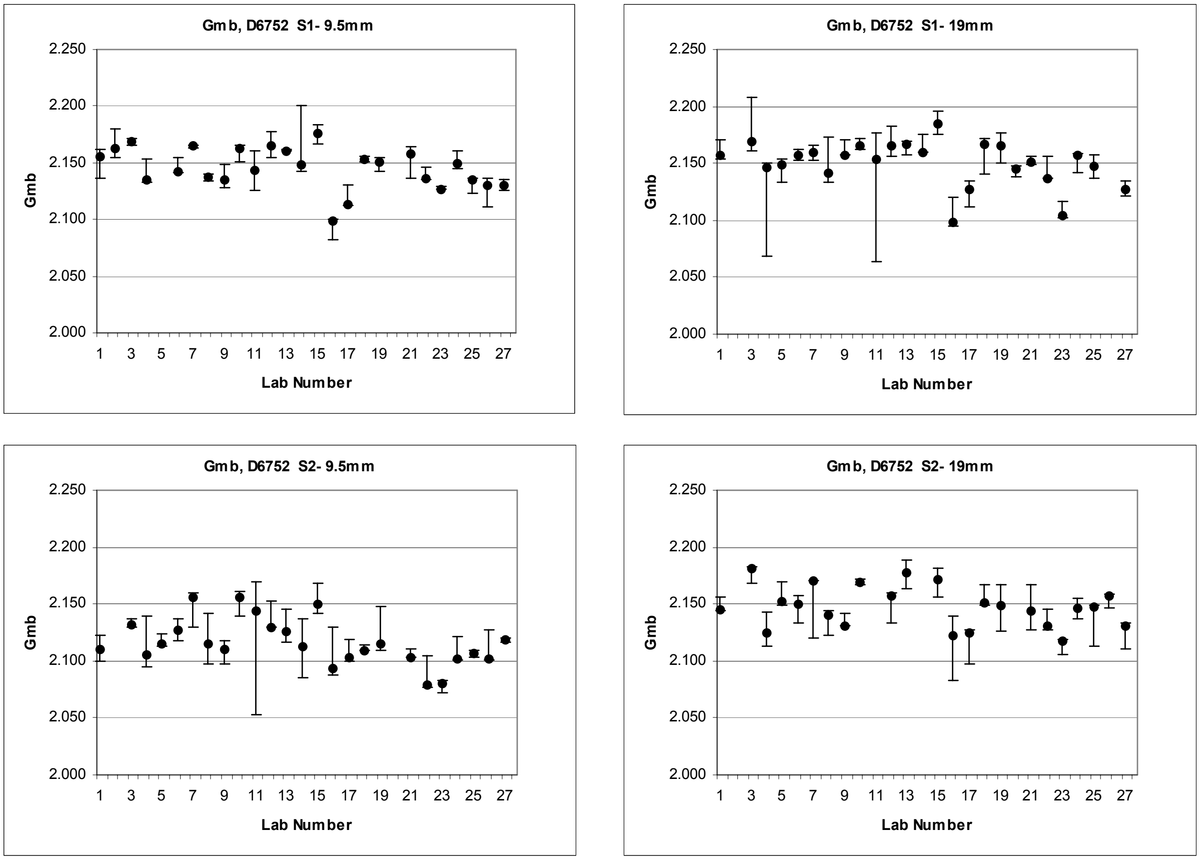

Figure 4-5- Box plots of bulk specific gravity (G_{mb})-D6752

Figure 4-6- h and k consistency statistics of bulk specific gravity (G_{mb})-D6752

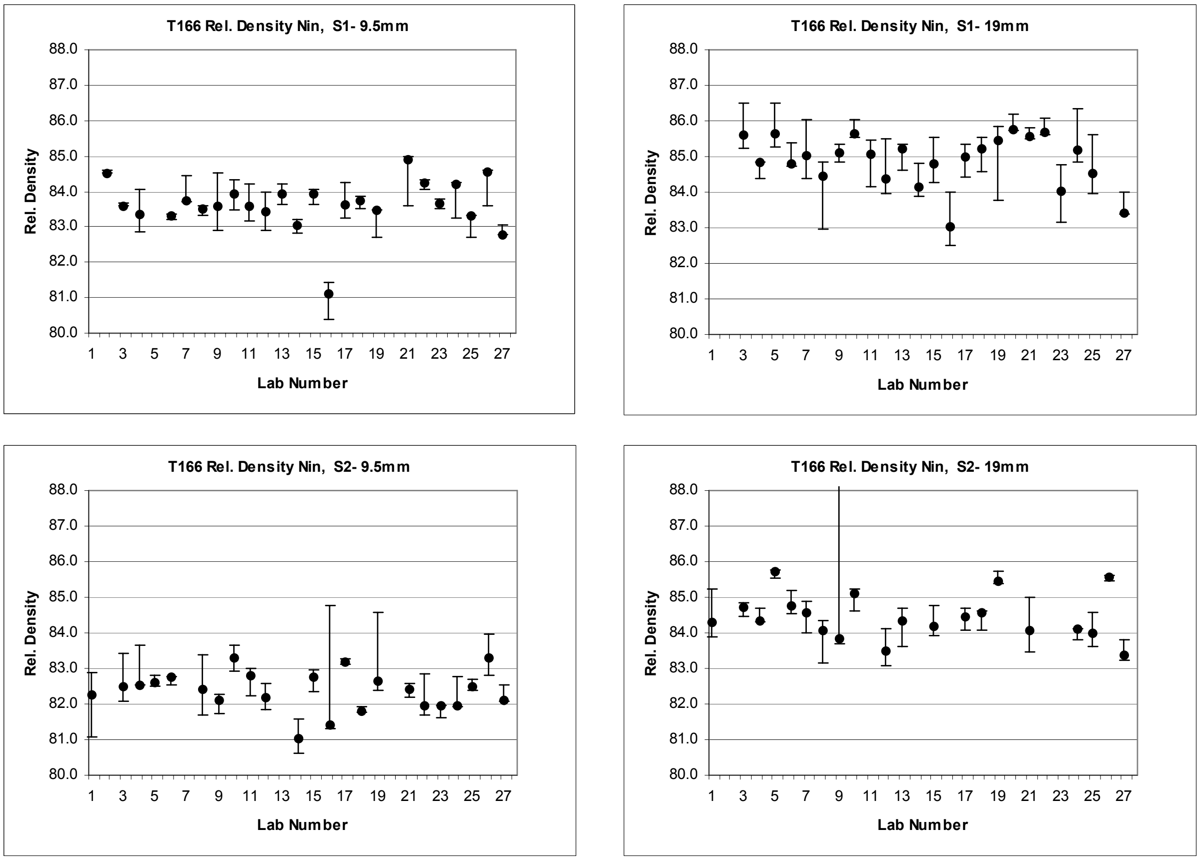

Figure 4-7- Box plots of relative density at N_{in} computed using T 166 G_{mb} values-T312

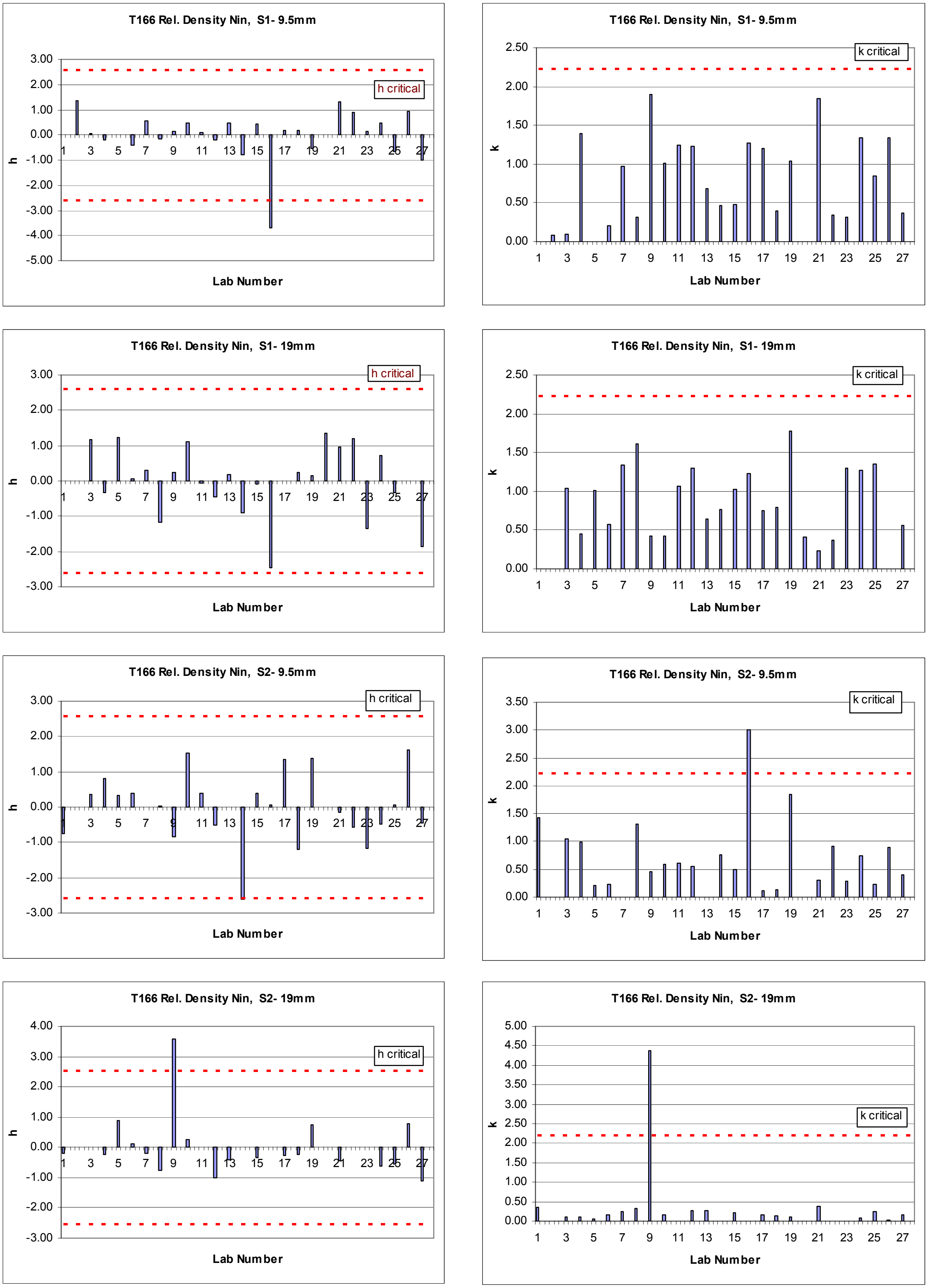

Figure 4-8- h and k consistency statistics of relative density at N_{in} using T166 G_{mb}-T312

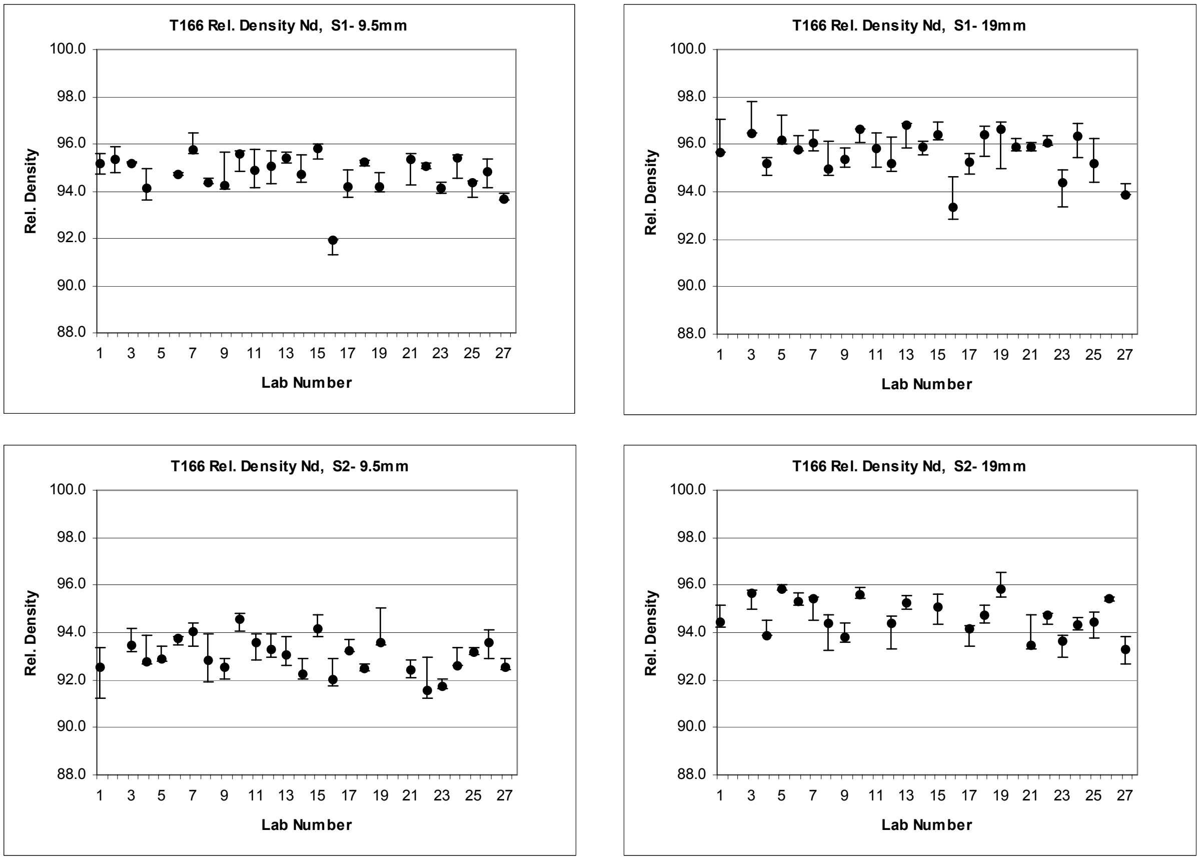

Figure 4-9 - Box plots of relative density at N_d computed using T 166 G_{mb} values-T312

Figure 4-10- h and k consistency statistics of relative density at N_d using T166 G_{mb}-T312

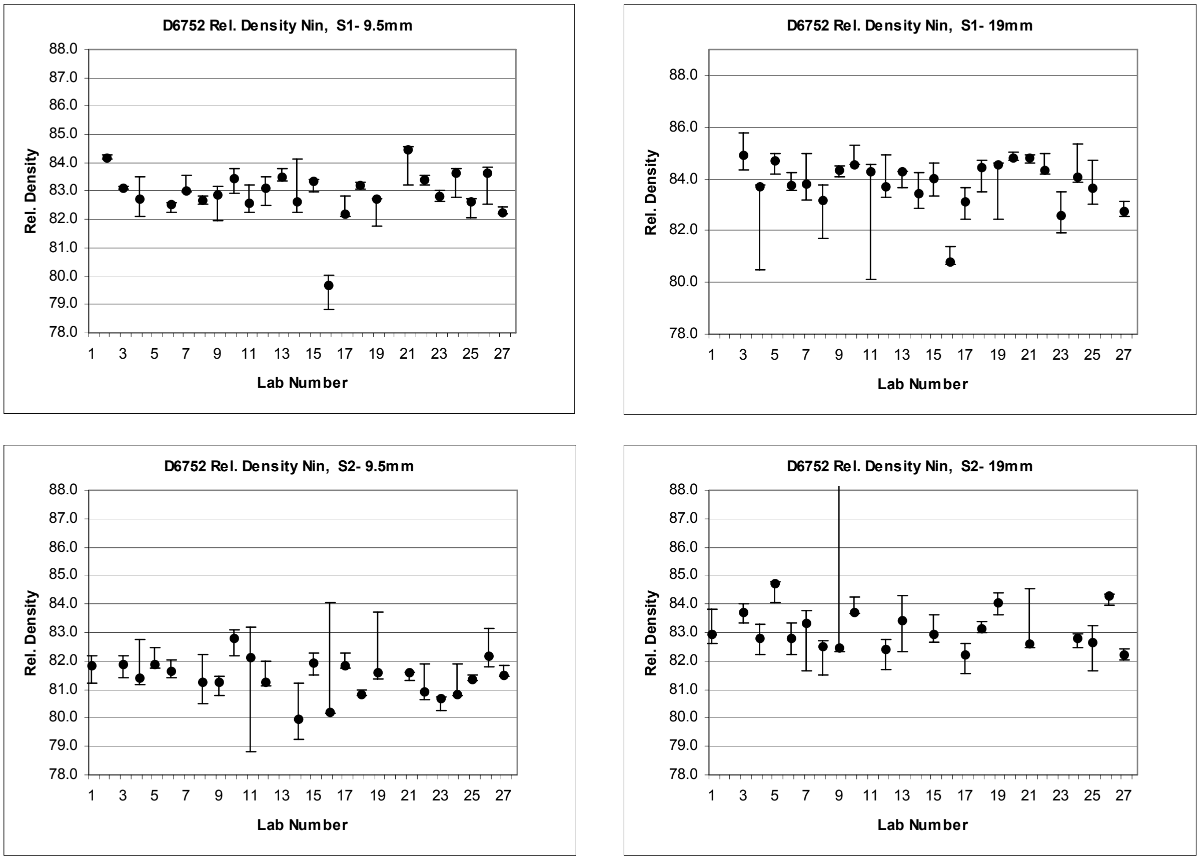

Figure 4-11- Box plots of relative density at N_{in} computed using D6752 G_{mb} values-T312

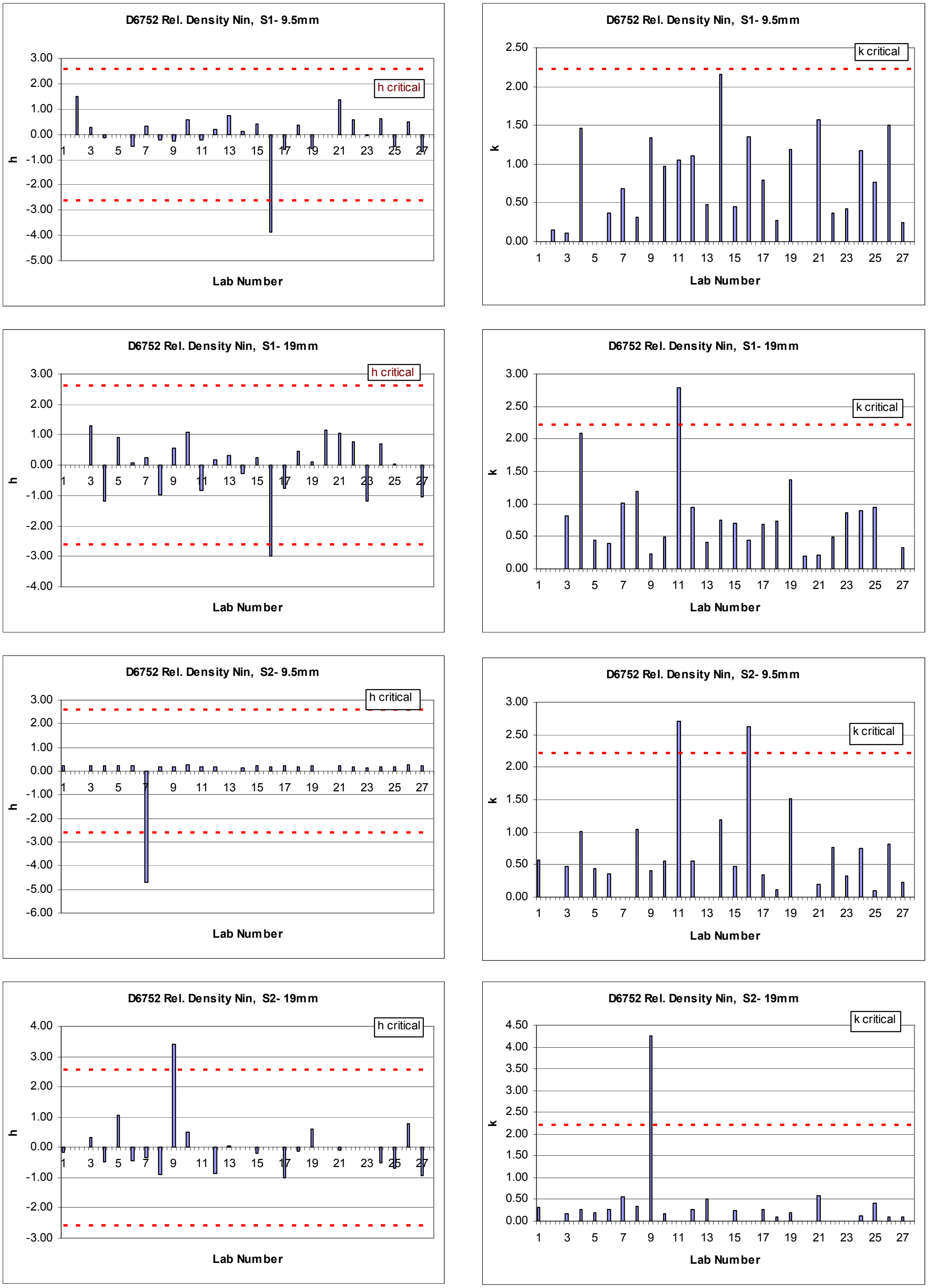

Figure 4-12- h and k consistency statistics of relative density at N_{in} using D6752 G_{mb}-T312

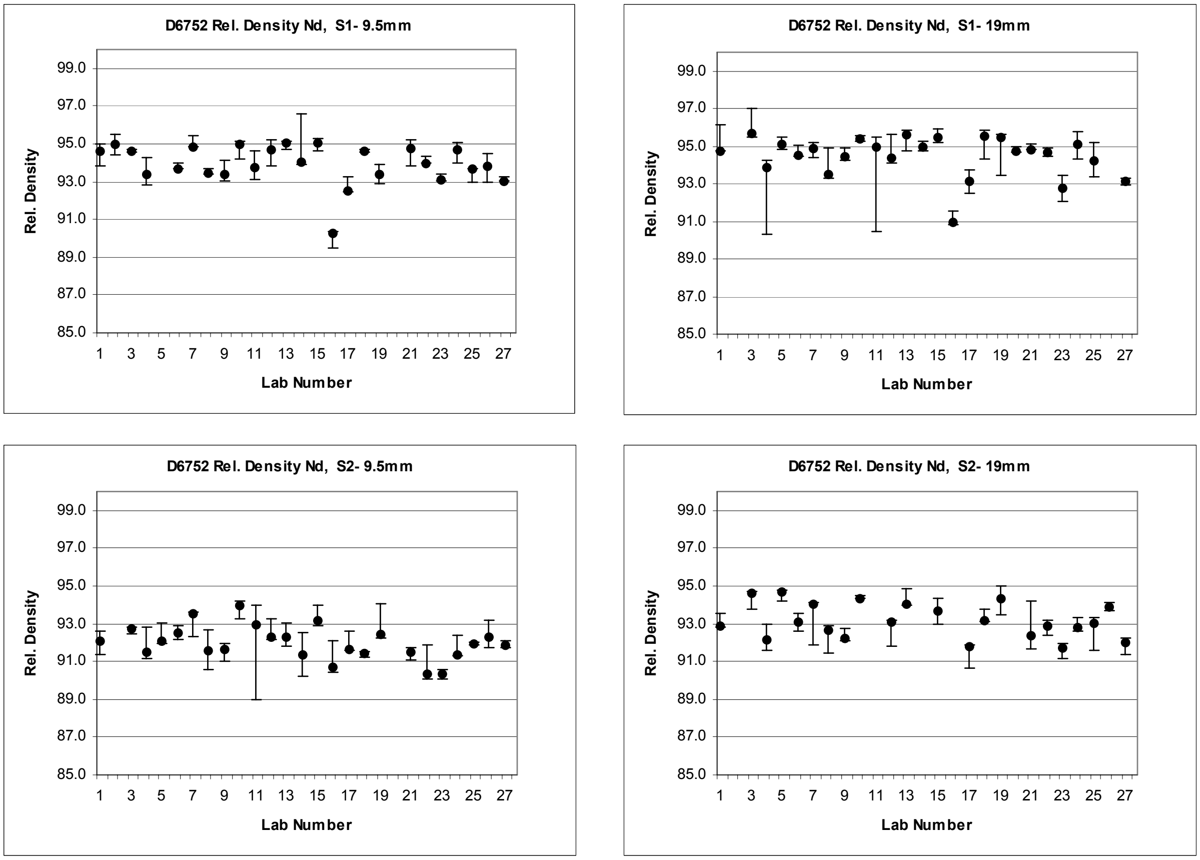

Figure 4-13- Box plots of relative density at N_d computed using D6752 G_{mb} values-T312

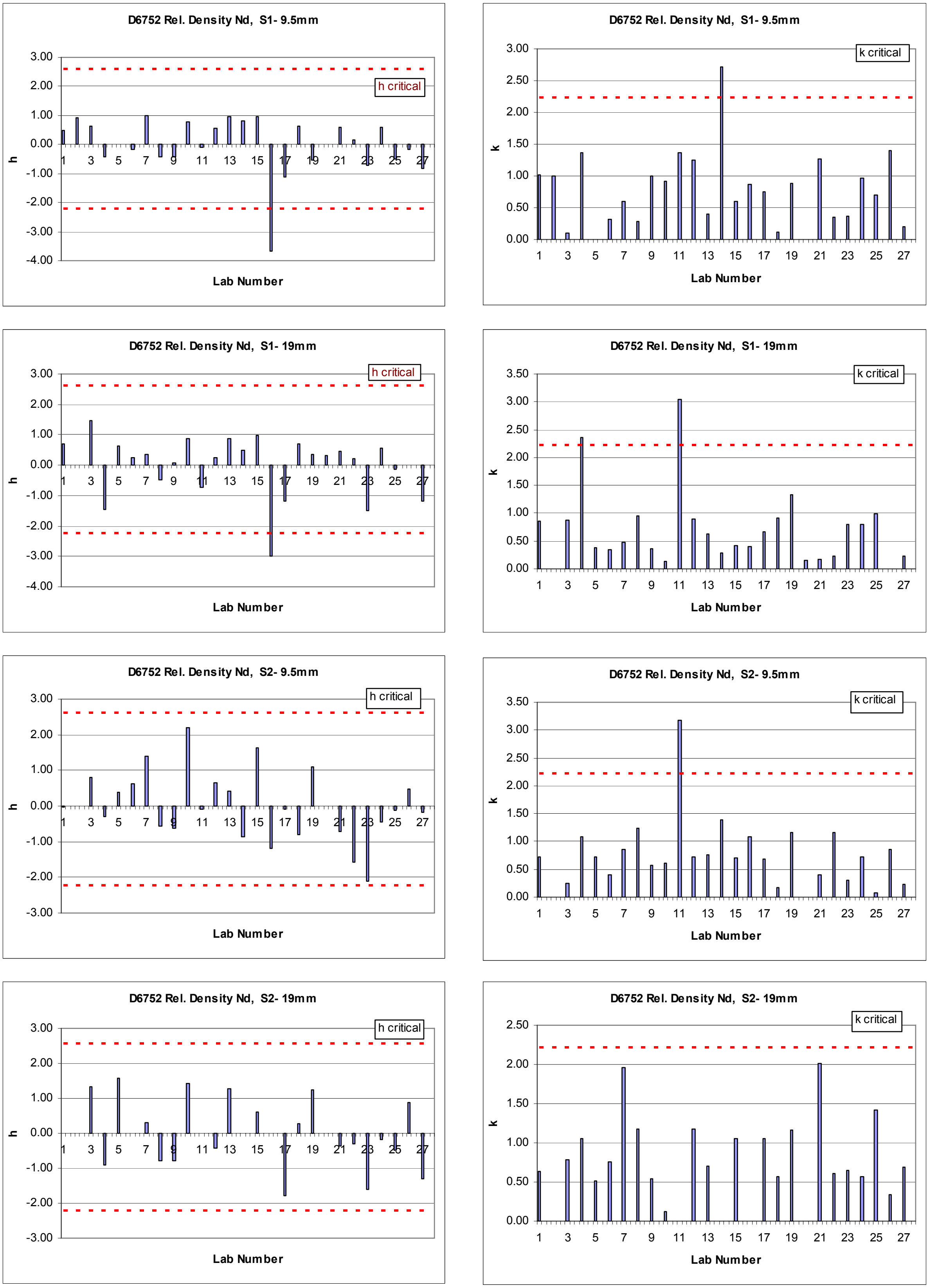

Figure 4-14- h and k consistency statistics of relative density at N_d using D6752 G_{mb}-T312

CHAPTER 5- AGING TIME STUDY

5.1 Experimental Design

The aging time experiment looked at asphalt mixtures with absorptive aggregates from two sources with two designs at five different aging/curing times. The goal of this experiment was to investigate how the volumetric properties of asphalt mixtures with absorptive aggregates change with increase in aging time and to propose an appropriate aging time based on the observed changes in volumetric properties.

Five input variables were addressed in the experiment.

1. Sample Source (Source 1 and Source 2 with differing levels of absorption)
2. Sample Design (9.5-mm and 19-mm nominal maximum aggregate size)
3. Aging Time (2 hr, 4 hr, 8 hr, 16 hr, and 32 hr)
4. Replicate (2 replicates)
5. Test (G_{mm} and G_{mb})

To evaluate all five variables, 80 specimens were required for the aging time experiment.

5.2 Sample Preparation and Testing

Sample batches 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 [5]. The 80 samples were then tested in the AMRL Bituminous Laboratory at NIST.

5.3 Analysis of Selective Volumetric Properties

To determine the appropriate aging time for the mixtures with absorptive aggregates, the changes in selective volumetric properties of the asphalt mixtures with the increase in aging time were evaluated. The selected volumetric properties included the maximum specific gravity (G_{mm}), bulk specific gravity (G_{mb}), percent air voids (V_a), and the percent absorbed asphalt. The asphalt film thickness as function of the effective binder content was also looked at. A summary of the measured properties are provided in Table 5-1. Between any pair of aging time, the trend of the change in volumetric properties was examined graphically and the significance of the change in number of properties was evaluated using a one-way analysis of variance.

5.3.1 Theoretical Maximum Specific Gravity (ASTM D2041)

The evaluation of G_{mm} is important in the aging study since the change in G_{mm} represents the change in the amount of asphalt absorbed into aggregates. Figure 5-1 shows the changes in specific gravity of the asphalt mixtures with the increase in aging time. As indicated in the figure, G_{mm} of the mixtures increase with the increase in aging time. Although, the G_{mm} values are expected to reach an asymptotic value at some point during the aging period, they continued to increase. This indicates that absorption of asphalt continued over the 32 hours of aging.

The ANOVA analysis was conducted to determine if the change in G_{mm} at various aging periods was significant. The result of the analysis is provided in Table 5-2. If a computed F value exceeds the critical value of 5.19, then the change in property between each pair of aging time is significant. Although G_{mm} of all four mixtures shows a positive trend with the increase in aging time (Figure 5-1), the significance of the change was different for different mixtures. As shown in Table 5-2, the change in G_{mm} of S1-9.5-mm mixture is not significant even after 32 hours of aging and the change in G_{mm} of S1-19-mm mixture is significant after 16 hours of aging. On the other hand, G_{mm} of S2-9.5 mm mixture changes significantly after 4 hours of aging and G_{mm} of S2-19- mm mixture changes significantly after 8 hours of aging. This points out that asphalt absorption in Source 1 aggregates occurs in slower pace than asphalt absorption in Source 2 aggregates.

5.3.2 Bulk Specific Gravity AASHTO T166

A change in G_{mb} represents the change in compactibility of the mixtures, which is a function of the quantity of asphalt absorption and the degree of asphalt hardening. As a result of extended aging time, not only more asphalt gets absorbed into aggregates but the non-absorbed portion of the asphalt binder hardens. Both absorption and hardening of asphalt binder would result in a decrease in compactibility and G_{mb} of the mixtures. This was clearly observed when mixtures were compacted after 32 hours of aging as all mixtures demonstrated very high resistance to compaction. The change in G_{mb} with the change in aging time is shown in Figure 5-2. As observed, other than S1-9.5 mm, the mixtures show the logical trend of decrease in G_{mb} with increase in aging time. The undefined trend of G_{mb} for the S1-9.5-mm mixture could be the result of sampling variation.

The significance of the change in G_{mb} for the four mixtures after each aging period was assessed using ANOVA. The ANOVA F values for G_{mb} property are provided in Table 5-3. Although a logical trend for G_{mb} of three out of four mixtures was shown in Figure 5-2, the significance of the trend was not indicted from the ANOVA results. As observed in Table 5-3, the computed F values for Source 1 mixtures do not exceed the critical F value of 5.19 even after 32 hours of aging and computed F values for Source 2 mixtures do not exceed the critical F value even after 16 hours of aging. The insensitivity of G_{mb} to aging time might be due to high shear force in gyratory compactor, which compact the specimens to similar heights regardless of their composition.

5.3.3 Percent Air Voids of Hot Mix Asphalt

The evaluation of the change in percent air void of the compacted asphalt mixtures, which represents the percent change in relative density at N-design provides a practical insight to the effect of aging time. Figure 5-3 shows the trend of the change in air voids with the change in aging time. As shown in the figure, the air voids increase with the increase in aging time. This trend is expected since the increase in aging time would allow more absorption and additional hardening of asphalt binder, which both phenomena reduce compactibility and consequently increase of percent air voids in the compacted mixtures.

The ANOVA F for the air void values are provided in Table 5-4. Although, the trend of the change in air voids with the change in aging time is positive for all four mixtures (Figure 5-3); ANOVA results indicates that the significance of the trends is not the same for the mixtures. The F values for air voids of Source 1 mixtures do not indicate a significant change even after 16 hours of aging. On the other hand, Source 2 mixtures show significant change in air voids at earlier time. The increase in air voids of S2-9.5-mm mixture is significant after 8 hours of aging and the increase in air voids of S2-19-mm mixture is significant after 4 hours of aging. Therefore, it can be concluded that laboratory aging time should not exceed 4 hours to prevent a significant change in the original mixture design.

The change in air voids as a function of aging time can also be evaluated from a practical point of view. Typically, a change in air voids more than ±0.5 % from the design is considered significant. Therefore, a difference of 1% between the air voids of a mixture at two different aging times would be significant. Figure 5-4 shows the difference in percent air voids of the four mixtures at each aging time with respect to the air voids at 2-hours aging time. In agreement with the statistical analysis, Figure 5-4 indicates that for S2-19-mm mixture a significant change of 1% in air voids occurs after 4 hours of aging while for Source 1 mixtures, the change in air void is not practically significant until after 16 hours of aging.

5.3.4 Percent Absorbed Binder Content

The evaluation of the change in the amount of absorbed asphalt might provide a better understanding of the effect of aging time. The percent absorbed asphalt was computed from maximum specific gravity of mixture, effective and bulk specific gravity of aggregates, specific gravity of asphalt, and the optimum binder content [7]. The computed percent absorbed asphalt values are provided in Table 5-1 and shown in Figure 5-5. It is observed from the figure that Source 1 mixtures are more absorptive from Source 2 mixtures. In addition, the percent absorption of asphalt increases with aging time for all four mixtures. To evaluate if the increase in absorption is significant from the practical stand point, the amount of change in absorbed asphalt at each aging time with respect to 2-hours aging time was computed as shown in Figure 5-6. In practice a change in binder content of ± 0.2 % from the design is significant. Therefore, a total change of 0.4% in the absorbed asphalt due to change in aging time would be significant. As indicated from Figure 5-6, the change in absorbed asphalt of S2-19-mm mixture is more than 0.4% after 4 hours of aging. This specifies that aging of this mixture for more than 4 hours would significantly change the original mixture design. Therefore, similar to the finding from the change in air voids, the change in absorbed binder indicates that laboratory aging time should not exceed 4 hours to prevent a significant change in the original mixture design.

5.3.5 Component Diagram of Asphalt Mixtures

In previous sections the differences between the volumetrics of the four mixtures and the changes in volumetrics after each aging time was presented in the percentage form. However, looking at the absolute volumes might provide additional insight to understanding the mixtures behavior. For this purpose, the component diagram approach was used to describe the changes in the volumes of the mixture elements [8]. A component diagram represents a compacted sample of a unit volume (cm^3) that displays the effective volume of aggregates (aggregate and water permeable pores), volume of absorbed binder, volume of effective binder, and volume of air in the form of discrete elements. Figure 5-7 through Figure 5-10 include the component diagrams of the four mixtures at various aging times. The following observations are made from the comparison of the figures:

1. The effective volume of aggregates is larger in the 19-mm mixtures (~0.83 cm^3 than in the 9.5-mm mixtures (~0.77 cm^3).
2. The volume of absorbed asphalt was greater for Source 1 mixtures (~0.06 cm^3) than for Source 2 mixtures (~0.04 cm^3).
3. The volume of effective asphalt was comparable for all mixtures (~0.1 cm^3), except for the S1-19-mm mixture that had the lowest volume of effective asphalt (~0.084 cm^3).
4. The volume of air was higher for the 9.5-mm mixtures (~0.08 cm^3) than for the 19-mm mixtures (~0.045 cm^3). This was caused by the lower effective volume of the aggregates in 9.5-mm mixtures.
5. Other than S1-9.5-mm mixture (Figure 5-7), which did not show a clear trend in volumetric changes, all mixtures clearly followed the logical trends in the volumetric changes (Figure 5-8 through Figure 5-10). As observed from the figures, effective volume of the aggregates decreased, volume of absorbed asphalt increased, volume of effective asphalt decreased, and volume of air increased with aging time.

As indicated from the findings above, the Source 2 mixtures which were most sensitive to aging have the least volume of absorbed asphalt and the most volume of effective asphalt. Since this is contrary to the consensus belief that mixtures with more absorptive aggregates are more susceptible to aging, a further investigation was done to compare the asphalt film thickness in the four mixtures.

5.3.6 Asphalt Binder Film Thickness

The asphalt film thickness should be a better indicator of the susceptibility of the mixture to aging than the volume of the absorbed and effective asphalt. This is because a mixture with high volume of effective binder might end up with a small asphalt film thickness if the specific surface area of the aggregates is large. A mixture with smaller asphalt film thickness is hypothesized to be more susceptible to aging than a mixture with larger asphalt film thickness. To test this hypothesis, the asphalt film thickness for the four mixtures was compared. The film thicknesses were computed using effective volume of asphalt binder and specific surface area of aggregates where specific surface area was calculated using the percent passing each sieve size and the specific surface area factors corresponding to those sieve sizes [7].

Figure 5-11 shows the computed film thicknesses of the four mixtures at various aging times. It is indicated from the figure that asphalt film thickness of all mixtures decreased with aging. While the asphalt film thicknesses were comparable for all mixtures (~10 micron), the S2-19-mm mixture had the largest asphalt film thickness of 14 micron. Since the S2-19-mm mixture was highly sensitive to aging, the hypothesis that mixtures with small asphalt film thickness are more susceptible to aging was not supported here. The reason might be due to the sampling variation of the limited number of specimens tested in this study.

Table 5-1- Average of the measured volumetric properties in the aging time study

Source	Design	Aging Time (h)	G_{mm}	G_{mb}	Air voids (%)	Absorbed Asphalt (%)	Asphalt Film Thickness (micron)
1	9.5	2	2.271	2.085	7.8	2.8	10.7
1	9.5	4	2.286	2.113	7.2	3.1	10.0
1	9.5	8	2.299	2.111	8.3	3.4	9.4
1	9.5	16	2.296	2.129	7.3	3.3	9.5
1	9.5	32	2.317	2.066	10.8	3.7	8.6
1	19	2	2.281	2.177	4.7	3.0	10.5
1	19	4	2.285	2.184	3.8	3.1	10.3
1	19	8	2.298	2.152	6.2	3.3	9.6
1	19	16	2.310	2.163	6.5	3.5	8.9
1	19	32	2.333	2.118	9.3	4.0	7.8
2	9.5	2	2.290	2.111	7.8	2.7	11.0
2	9.5	4	2.304	2.104	8.5	3.0	10.3
2	9.5	8	2.320	2.092	10.0	3.3	9.6
2	9.5	16	2.332	2.093	10.1	3.5	9.1
2	9.5	32	2.357	2.044	13.1	4.0	7.9
2	19	2	2.302	2.192	4.9	1.2	14.2
2	19	4	2.324	2.184	5.8	1.7	12.8
2	19	8	2.326	2.178	6.3	1.7	12.7
2	19	16	2.340	2.177	7.1	2.0	11.8
2	19	32	2.358	2.132	9.5	2.3	10.7

Table 5-2- Summary of results of ANOVA on G_{mm} values in the aging time study

Compare		Source 1- 9.5 mm		Source 1- 19 mm		Source 2- 9.5 mm		Source 2- 19 mm	
Hours	Hours	Computed F	Decision	Computed F	Decision	Computed F	Decision	Computed F	Decision
2	4	0.390	ACCEPT	0.054	ACCEPT	17.233	REJECT	4.286	ACCEPT
2	8	1.809	ACCEPT	0.764	ACCEPT	73.726	REJECT	5.101	ACCEPT
2	16	1.501	ACCEPT	2.3	ACCEPT	144.507	REJECT	13.127	REJECT
2	32	4.883	ACCEPT	7.146	REJECT	373.249	REJECT	27.773	REJECT
4	8	0.519	ACCEPT	0.413	ACCEPT	19.681	REJECT	0.035	ACCEPT
4	16	0.361	ACCEPT	1.652	ACCEPT	61.953	REJECT	2.411	ACCEPT
4	32	2.513	ACCEPT	5.962	REJECT	230.115	REJECT	10.238	REJECT
8	16	0.014	ACCEPT	0.413	ACCEPT	11.797	REJECT	1.862	ACCEPT
8	32	0.748	ACCEPT	3.237	ACCEPT	115.202	REJECT	9.069	REJECT
16	32	0.970	ACCEPT	1.338	ACCEPT	53.269	REJECT	2.712	ACCEPT

Note: The critical F value for 5% level of significance is 5.19.

Table 5-3- Summary of results of ANOVA on G_{mb} values in the aging time study

Compare		Source 1- 9.5 mm		Source 1- 19 mm		Source 2- 9.5 mm		Source 2- 19 mm	
Hours	Hours	Computed F	Decision	Computed F	Decision	Computed F	Decision	Computed F	Decision
2	4	1.419	ACCEPT	0.057	ACCEPT	0.142	ACCEPT	0.166	ACCEPT
2	8	1.271	ACCEPT	0.85	ACCEPT	1.047	ACCEPT	0.538	ACCEPT
2	16	3.505	ACCEPT	0.286	ACCEPT	0.94	ACCEPT	0.664	ACCEPT
2	32	0.62	ACCEPT	4.733	ACCEPT	12.824	REJECT	10.451	REJECT
4	8	0.004	ACCEPT	1.349	ACCEPT	0.418	ACCEPT	0.106	ACCEPT
4	16	0.463	ACCEPT	0.6	ACCEPT	0.351	ACCEPT	0.166	ACCEPT
4	32	3.915	ACCEPT	5.833	REJECT	10.266	REJECT	7.982	REJECT
8	16	0.554	ACCEPT	0.15	ACCEPT	0.003	ACCEPT	0.007	ACCEPT
8	32	3.666	ACCEPT	1.572	ACCEPT	6.543	REJECT	6.247	REJECT
16	32	7.072	REJECT	2.692	ACCEPT	6.821	REJECT	5.846	REJECT

Note: The critical F value for 5% level of significance is 5.19.

Table 5-4- Summary of results of ANOVA on air void values in the aging time study

Compare		Source 1- 9.5 mm		Source 1- 19 mm		Source 2- 9.5 mm		Source 2- 19 mm	
Hours	Hours	Computed F	Decision	Computed F	Decision	Computed F	Decision	Computed F	Decision
2	4	0.608	ACCEPT	1.069	ACCEPT	2.545	ACCEPT	12.161	REJECT
2	8	0.551	ACCEPT	2.893	ACCEPT	26.639	REJECT	25.849	REJECT
2	16	0.451	ACCEPT	4.128	ACCEPT	30.225	REJECT	61.559	REJECT
2	32	17.193	REJECT	26.942	REJECT	156.502	REJECT	270.353	REJECT
4	8	2.316	ACCEPT	7.479	REJECT	12.716	REJECT	2.55	ACCEPT
4	16	0.012	ACCEPT	9.398	REJECT	15.228	REJECT	18.998	REJECT
4	32	24.267	REJECT	38.473	REJECT	119.13	REJECT	167.835	REJECT
8	16	1.999	ACCEPT	0.109	ACCEPT	0.113	ACCEPT	7.627	REJECT
8	32	11.589	REJECT	12.177	REJECT	54.004	REJECT	129.008	REJECT
16	32	23.216	REJECT	9.978	REJECT	49.172	REJECT	73.899	REJECT

Note: The critical F value for 5% level of significance is 5.19.

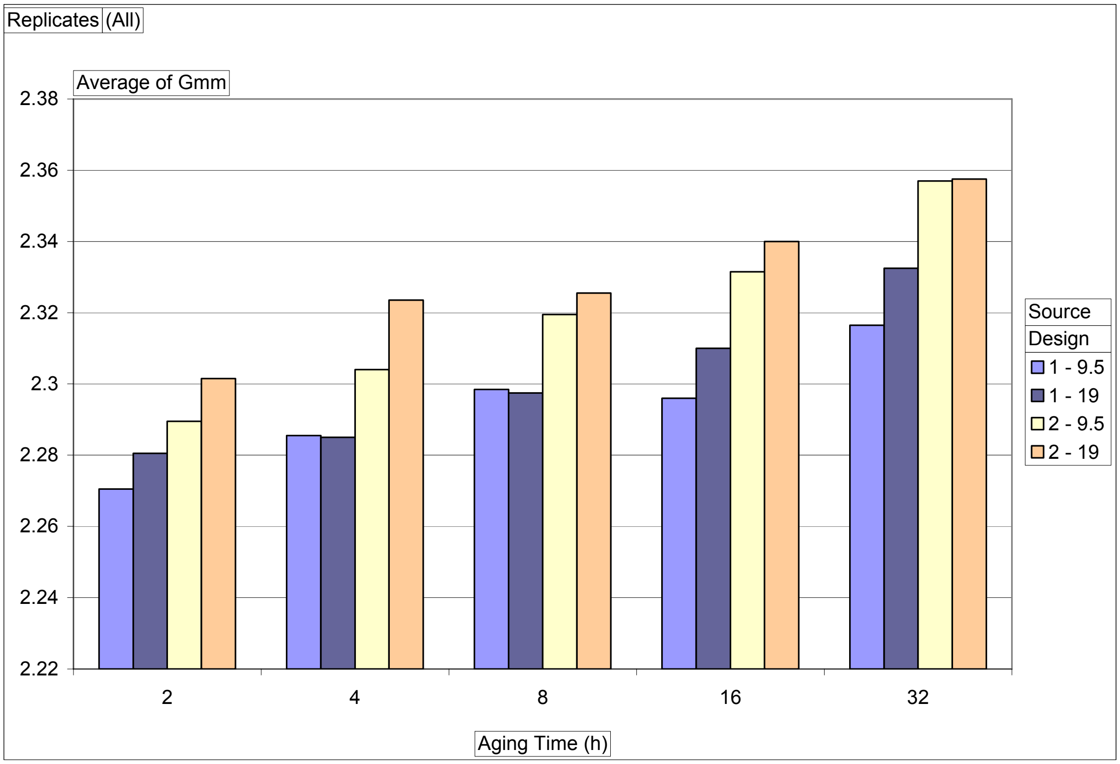

Figure 5-1- Average G_{mm} values of the four mixtures at various aging times

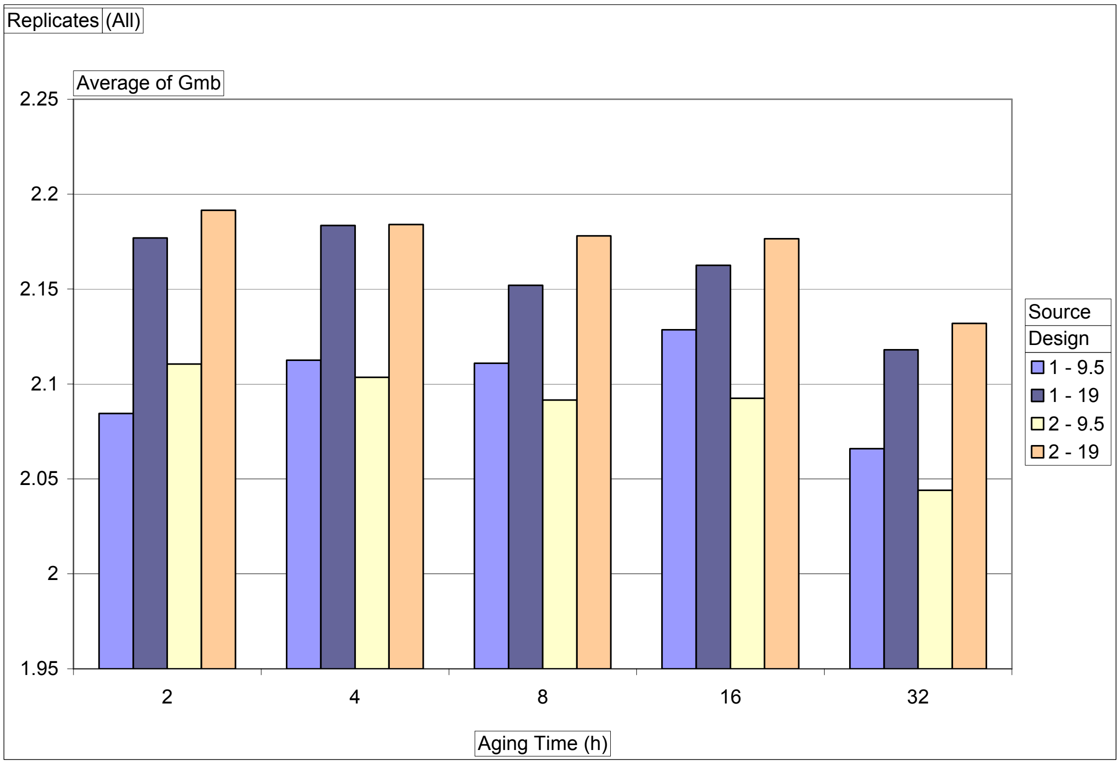

Figure 5-2- Average G_{mb} values of the four mixtures at various aging times

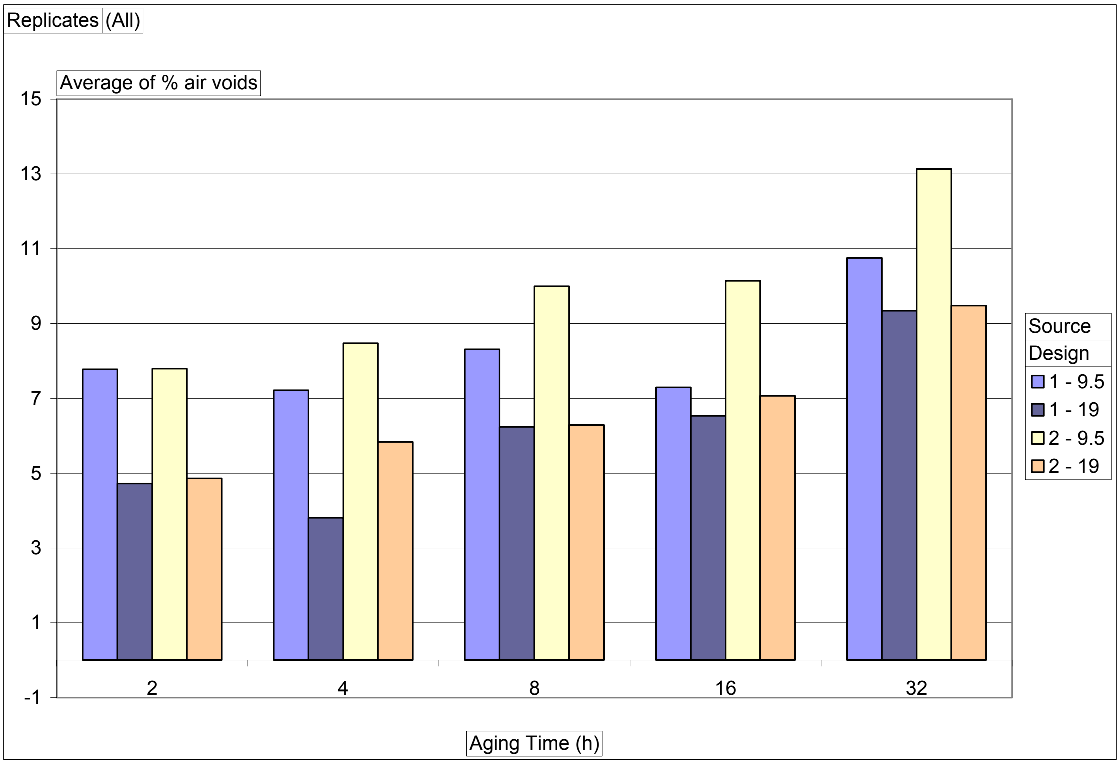

Figure 5-3- Average % air void values of the four mixtures at various aging times

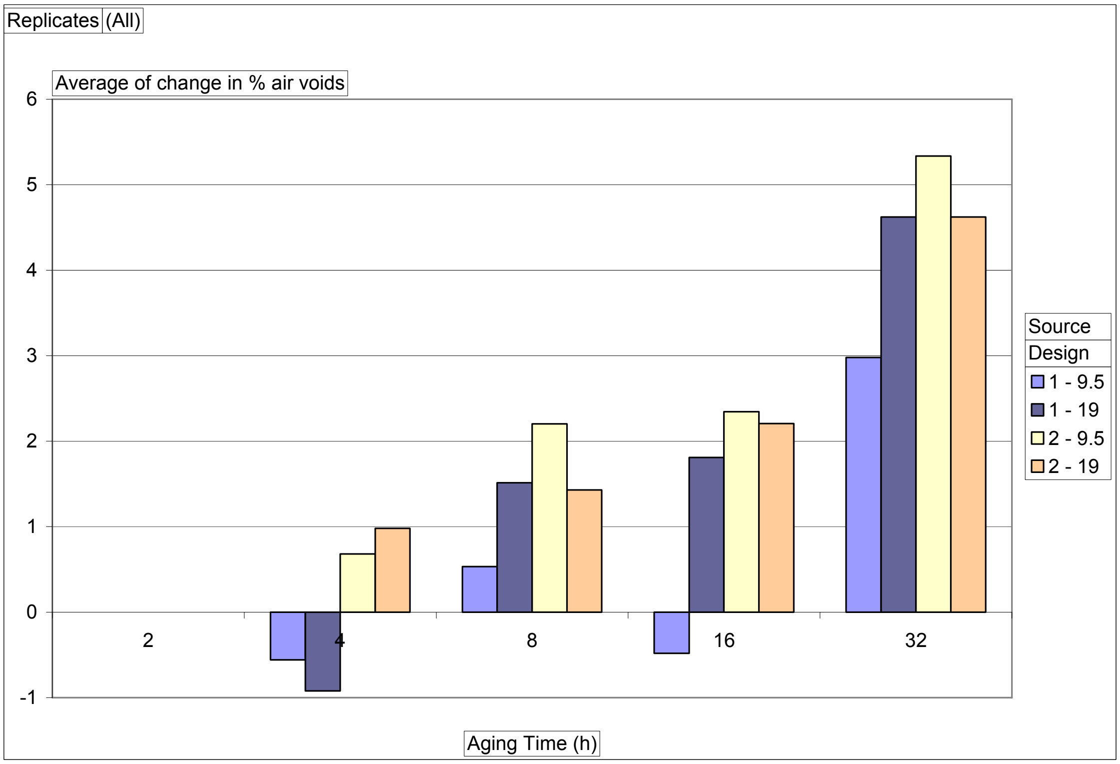

Figure 5-4- Average difference in % air voids of 2-hour and consequent aging times

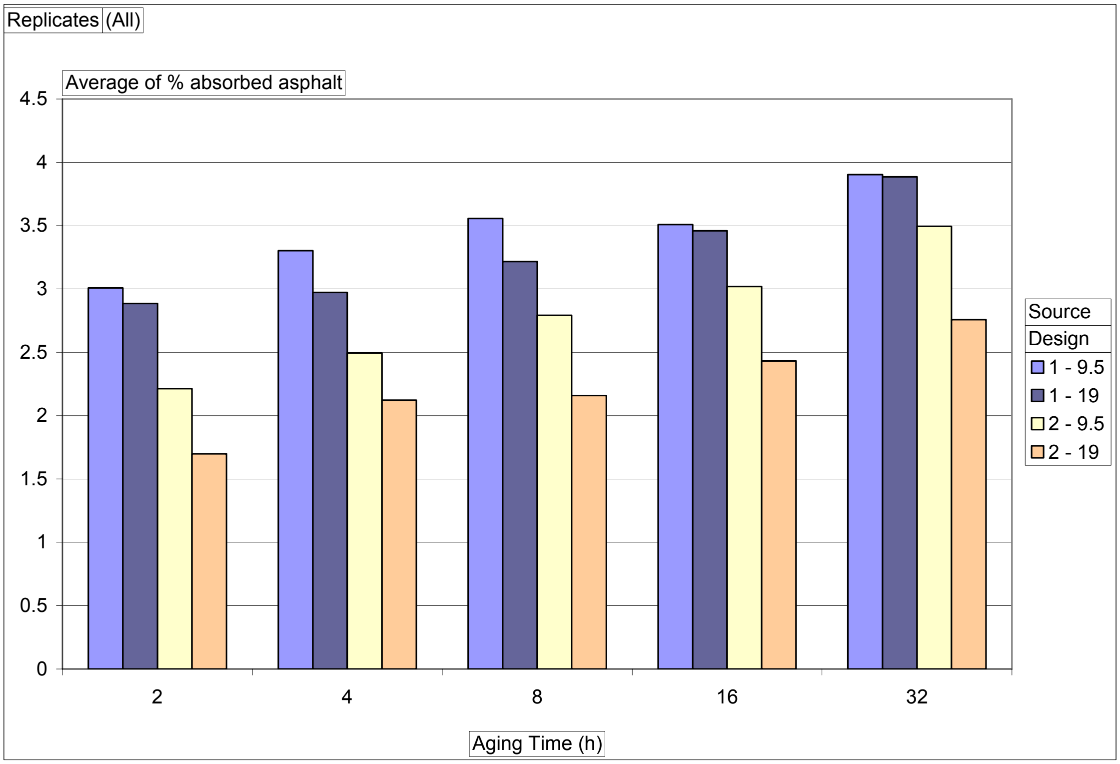

Figure 5-5- Average of % absorbed asphalt of the four mixtures at various aging times

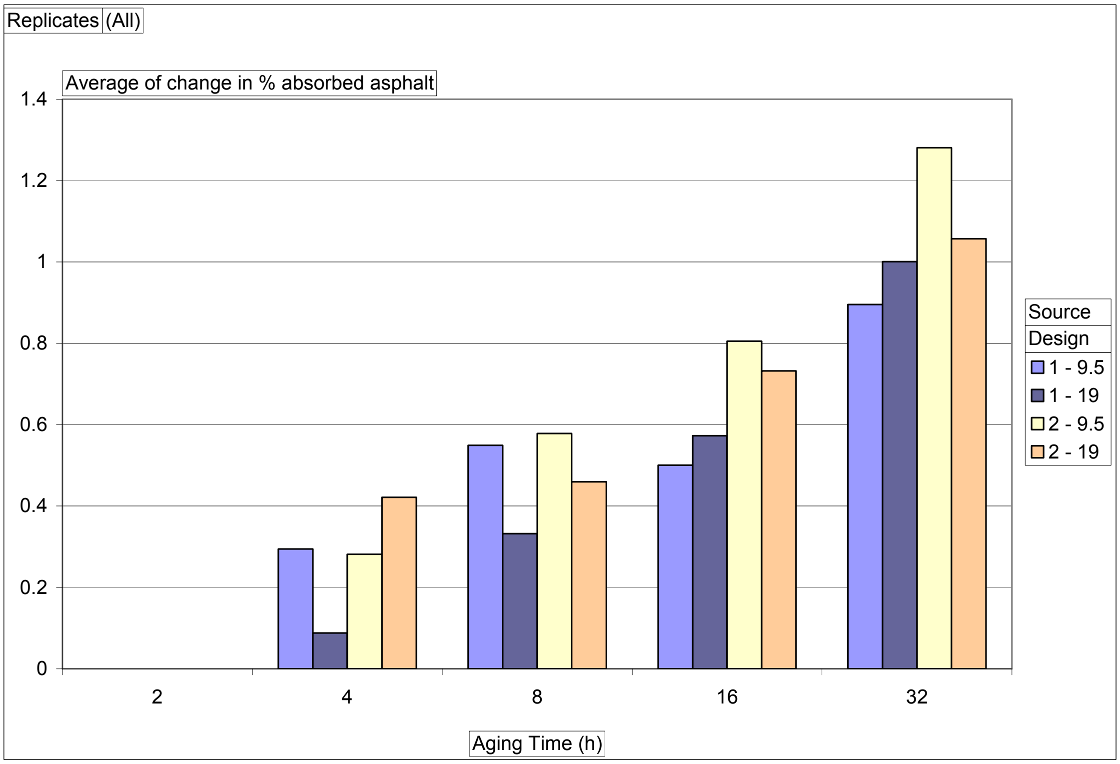

Figure 5-6- Average difference in % absorbed asphalt of 2-hour and consequent aging times

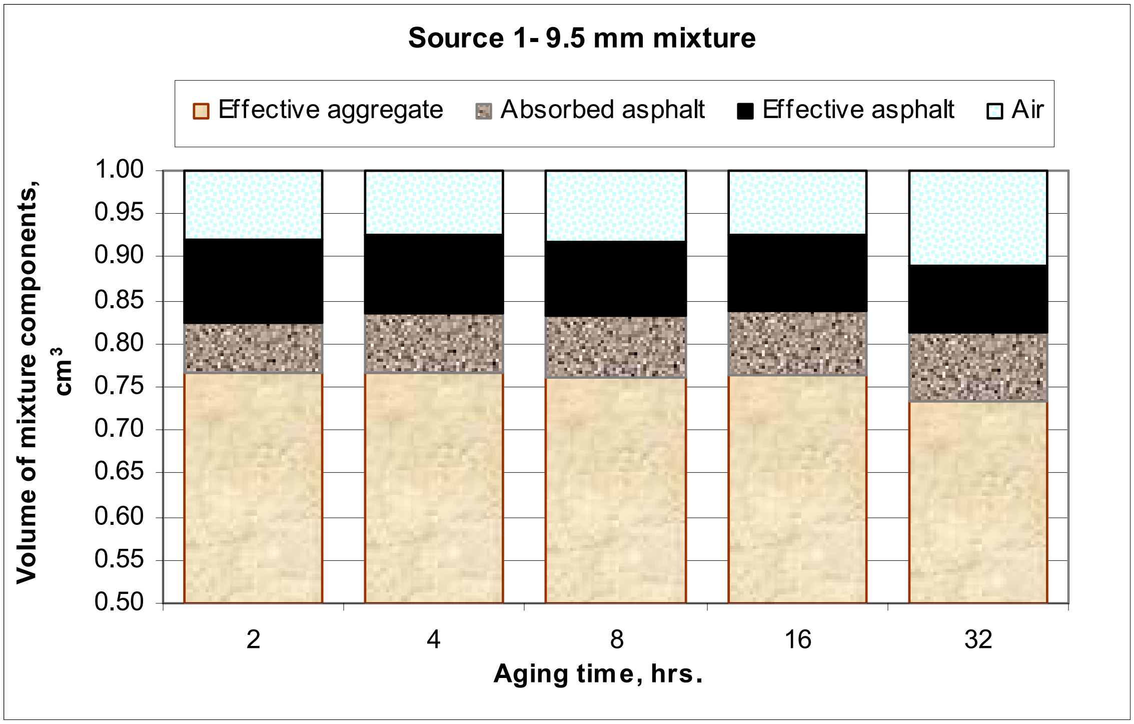

Figure 5-7- The volumetric component diagram of the S1-9.5-mm mixture at various aging times

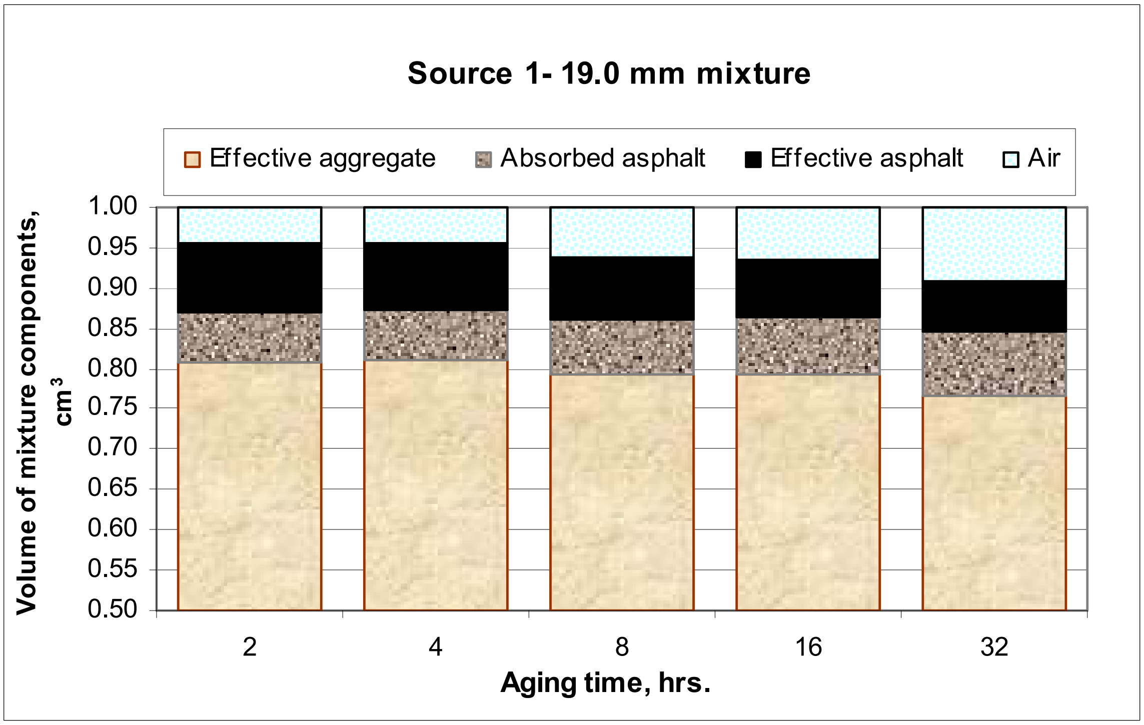

Figure 5-8- The volumetric component diagram of the S1-19-mm mixture at various aging times

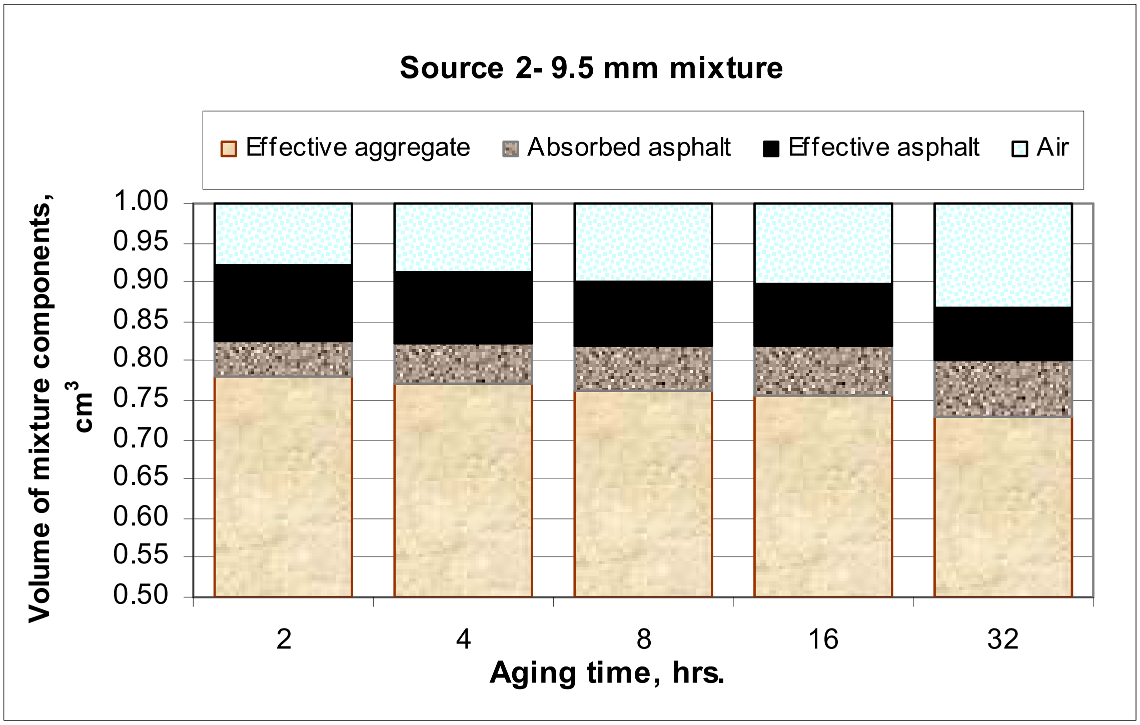

Figure 5-9- The volumetric component diagram of the S2-9.5-mm mixture at various aging times

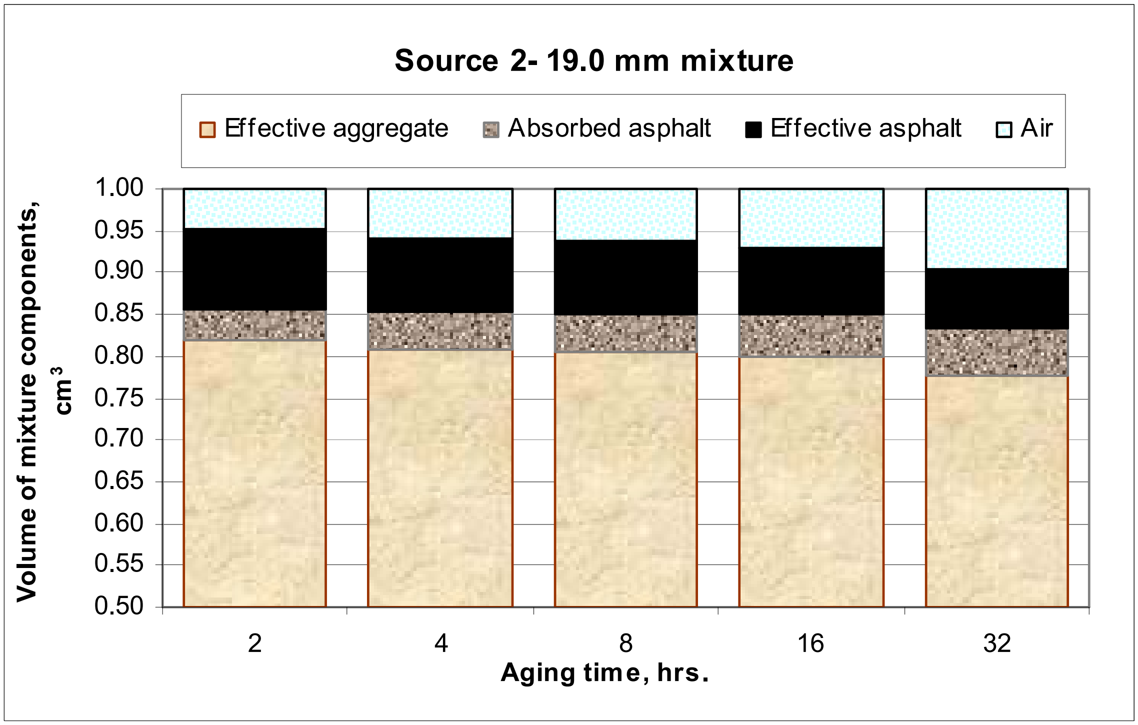

Figure 5-10- The volumetric component diagram of the S2-19-mm mixture at various aging times

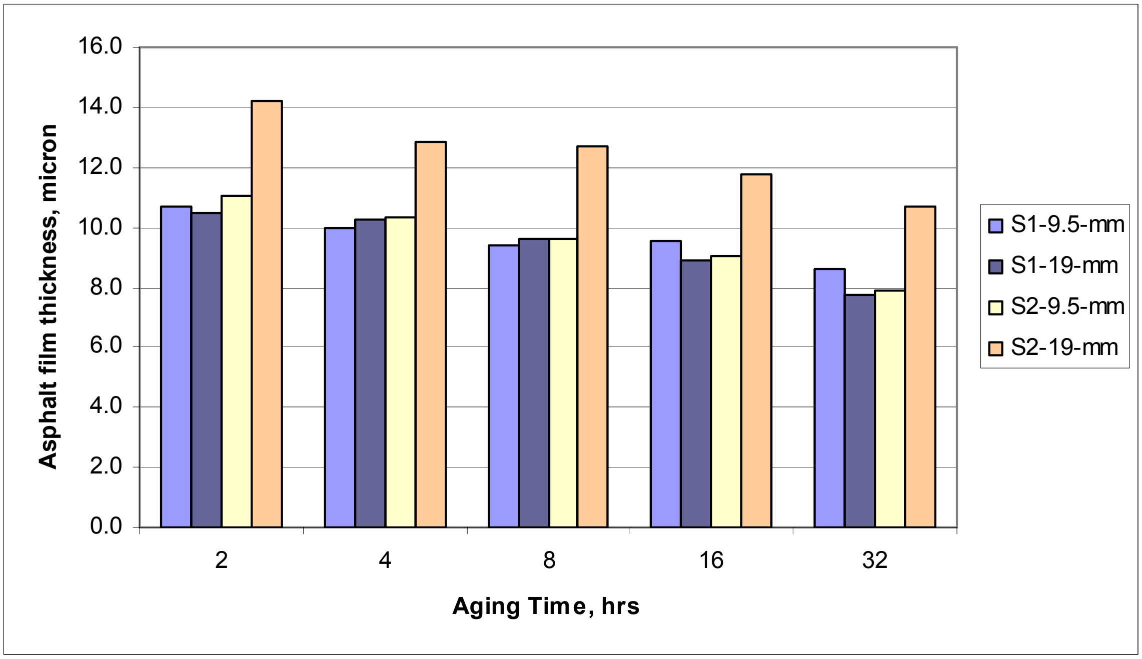

Figure 5-11- Asphalt film thickness in the four mixtures at various aging times

CHAPTER 6- CONCLUSIONS AND RECOMMENDATIONS

6.1 General

This study was conducted to prepare precision estimates for AASHTO and ASTM standards used to determine selected volumetric properties of HMA using absorptive aggregates. An interlaboratory study was planned to develop a precision statement applicable to (1) AASHTO T312, which is used to prepare and determine the density of HMA specimens using a Superpave Gyratory compactor, (2) AASHTO T166, which is used to determine bulk specific gravity of compacted asphalt mixtures, (3) ASTM D2041, which is used to determine maximum specific gravity of bituminous paving mixtures, and (4) ASTM D6752, which is used to determine bulk specific gravity and density of compacted bituminous mixtures using an automatic vacuum sealing method. The study was also aimed at evaluating the effect of aging time on the selective volumetric properties of HMA with absorptive aggregates. An appropriate laboratory aging time was intended to be proposed based on the observed effects of aging time. The study conclusions and recommendations are as follows.

6.2 Conclusions and Recommendations Related to Interlaboratory Study

6.2.1 ASTM D2041

6.2.1.1 Conclusions

The S_r and S_R estimates for D2041 of 9.5-mm and 19-mm mixtures were not significantly different. Therefore, the precision estimates from the two mixtures were combined and presented in a precision statement in Appendix C. The comparison of the precision estimates for D2041 computed using mixtures with absorptive aggregates versus mixtures with non-absorptive aggregates indicated that the precision estimates are significantly different.

6.2.1.2 Recommendations

Based on the significant difference in S_r and S_R precision estimates for D2041 of mixtures with absorptive aggregates versus mixtures with non-absorptive aggregates, the precision statement in Appendix C, which provides separate estimates for mixtures with absorptive aggregates and mixtures with non-absorptive aggregates, is recommended.

6.2.2 AASHTO T166

6.2.2.1 Conclusions

The S_r and S_R estimates for T166 of 9.5-mm and 19-mm mixtures with absorptive aggregates were not significantly different. Therefore, the precision estimates from the two mixtures were combined. The comparison of the precision estimates for T166 computed using mixtures with absorptive aggregates versus mixtures with non-absorptive aggregates indicated that the precision estimates are comparable and can be considered for combining.

6.2.2.2 Recommendations

Based on no significant difference in S_r and S_R precision estimates for T166 using mixtures with absorptive aggregates and mixtures with non-absorptive aggregates, the precision statement in Appendix D, which includes combined precision estimates of mixtures with absorptive aggregates and mixtures with non-absorptive aggregates, is recommended.

6.2.3 ASTM D6752 Results

6.2.3.1 Conclusions

The S_r and S_R estimates for D6752 of 9.5-mm and 19-mm mixtures with absorptive aggregates were not statistically significant. Therefore, the precision estimates from the two mixtures were combined.

The bulk specific gravity (G_{mb}) values obtained using D6752 were significantly lower than those obtained using T166. However, the S_r and S_R estimates obtained using T166 and D6752 were not significantly different for either 9.5-mm or 19-mm mixtures.

The comparison of the precision estimates for D6752 computed in this study with precision estimates for D6752 computed in Phase 1 [1] using mixtures with non-absorptive aggregates indicated that the precision estimates are comparable and can be considered for combining.

6.2.3.2 Recommendations

Based on no significant difference in S_r and S_R precision estimates for D6752 using mixtures with absorptive aggregates and mixtures with non-absorptive aggregates, the precision statement in Appendix E, which includes combined precision estimates of mixtures with absorptive aggregates and mixtures with non-absorptive aggregates, is recommended. The combined precision estimates can be included in AASHTO TP69, Bulk Specific Gravity and Density of Compacted Asphalt Mixtures Using Automatic Vacuum Sealing Method.

6.2.4 AASHTO T312

6.2.4.1 Conclusions

At either N_{in} or N_d, the relative density computed using D6752 G_{mb} was significantly lower than the relative density obtained using T166 G_{mb}. However, the corresponding S_r and S_R estimates for relative density using T166 G_{mb} and D6752 G_{mb} for either 9.5-mm or 19-mm mixtures with absorptive aggregates were not significantly different. Therefore, the precision estimates were combined. The comparison of the precision estimates for T 312 computed in this study with precision estimates computed in Phase 1 [1] using mixtures with non-absorptive aggregates indicated that the precision estimates are significantly different and should be presented separately.

6.2.4.2 Recommendations

Based on the significant difference in S_r and S_R precision estimates for T312 using mixtures with absorptive aggregates and mixtures with non-absorptive aggregates, the precision statement in Appendix F, which includes separate precision estimates for mixtures with absorptive aggregates and mixtures with non-absorptive aggregates, is recommended.

6.3 Conclusions and Recommendations Related to Aging Time Study

6.3.1.1 Conclusions

The effect of aging time on selective volumetric properties of four different mixtures with absorptive aggregates was investigated. Although data from this study indicated that absorption of asphalt into aggregates continued over the 32 hours of aging, the selection of proper aging time should not be based on the prolonged absorption period. The statistical analysis of G_{mm} and air void data indicated that volumetric properties of mixtures with absorptive aggregates could change significantly after 4 hours of aging. In addition, the change in air voids and asphalt absorption was shown to be practically significant after 4 hours of aging. Therefore, aging of a mixture with absorptive aggregates for a period longer than 4 hours might result in volumetrics that are significantly different from those at the original mixture design. Therefore, the laboratory aging of mixtures with absorptive aggregates should be limited to 4 hours to avoid deviation from the mixture design.

6.3.1.2 Recommendations

Based on the findings of this study a revision to AASHTO R 30 standard practice is recommended. A revision should specify a laboratory aging time that does not exceed 4 hours for volumetric testing of mixtures with absorptive aggregates. It is important to note that the suggested aging time is specific to laboratory procedure and does not apply to silo time since the laboratory aging does not necessarily represent the aging that takes place in the field. However, if mix is intended to be stored for a long time, the increased amount of absorption needs to be considered.

The aging of mixtures with absorptive aggregates has two phases. One phase is the absorption of asphalt into the pores of aggregates that affect the volumetrics of the mixture, and the other phase is the stiffening of the binder, which change the shear strength and stiffness of the material. This study aimed at determining the laboratory aging time that meets the volumetric requirements of the mixture design. It is of interest to determine the appropriate aging time for the performance testing of mixtures with absorptive aggregates. For example, the difference between 2 to 4 hours of aging might be significant for performance testing as shown by dynamic modulus or flow numbers of mixtures with absorptive aggregates. Therefore, it is recommended to examine the effect of aging time on performance testing of asphalt mixtures with absorptive aggregates. The simple performance tests (SPT) of dynamic modulus and flow number can be conducted and the change in performance with the change in aging time can be evaluated. The appropriate aging time for performance purpose can then be suggested based on the observed changes in dynamic modulus or flow number of the mixtures.

As mentioned above the aging process in laboratory includes both absorption and stiffening of the binder. However, the aging in silo is assumed to include mainly absorption and not the stiffening of the binder. The reason is that the mixtures in laboratory are spread thin in flat pans. The air that is in contact with the mixture oxidizes the binder and in result changes the mixture stiffness. In silo, the asphalt mixture is placed in a large pile. The exposure to the air does not necessarily result in age hardening of the binder and in result the mixture. Therefore, laboratory aging is assumed to cause more severe changes to the mixture than the aging in silo. For example, 32 hours of laboratory aging would destroy the binder and prevent the mixture to compact properly; however, 32 hours in silo might not cause any difficulty in the field compaction. In order to enable adjustment of the laboratory aging based on the expected field time aging, it is recommended that the correlation between the aging in a flat pan and aging in a silo to be investigated. This study could involve comparison of the performance of plant-aged and laboratory-aged specimens.

In the current study two sources of aggregates with 4% to 5% levels of absorption were used. It is recommended to conduct a similar study using aggregates with 3% to 4% absorption. The recommended study would increase the validity of the current study since the whole range of absorptive aggregates would be covered. In addition, it provides further insight to the current findings of the aging time study.

REFERENCES

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APPENDIX A

MIX DESIGN INFORMATION

Source 1 Design Information

Table 1 below gives the laboratory mix formulas used in the two Source 1 designs.

Table 1-Source 1 Design Formulas

	9.5 mm			19 mm		
Material	**Rice (g)**	**Bulk (g)**	**Percent (%)**	**Rice (g)**	**Bulk (g)**	**Percent (%)**
19 mm	0	0	--	0	0	--
12.5 mm	0	0	--	489.1	1021.8	23.3
9.5 mm	161.5	340.2	7.8	195.6	408.7	9.3
4.75 mm	541.5	1140.3	26.3	352.1	735.6	16.8
2.36 mm	598.5	1260.5	29.1	410.9	858.4	19.6
Sand	560.5	1180.3	27.2	469.6	980.9	22.4
Mineral Filler	38	80	1.8	39.1	81.7	1.9
Binder	159.2	335.2	7.7	143.9	300.6	6.9
Total	2059.2	4336.5	100	2100.3	4387.7	100

The designs above resulted in the properties shown in Table 2 below.

Table 2- Source 1 Properties

Property	9.5 mm Mix (%)	19 mm Mix (%)
Design Asphalt Content	7.73	6.85
Effective Asphalt Content	4.87	4.45
Binder Absorption	2.86	2.40

Source 2 Design Information

Table 3 below gives the laboratory mix formulas used in the two Source 2 designs.

Table 3 - Source 2 Design Formulas

	9.5 mm			19 mm		
Material	Rice (g)	Bulk (g)	Percent (%)	Rice (g)	Bulk (g)	Percent (%)
19 mm	0	0	--	236.9	414.6	9.4
12.5 mm	0	0	--	379.1	663.4	15.0
9.5 mm	204.6	409.1	9.3	450.2	787.8	17.8
4.75 mm	675.1	1350.2	30.7	402.8	704.8	16.0
2.36 mm	511.7	1023.3	23.3	355.4	621.9	14.1
Sand	654.8	1309.6	29.8	544.9	953.6	21.6
Mineral Filler	--	--	--	--	--	--
Binder	150.9	301.9	6.9	155.3	271.7	6.1
Total	2197.1	4394.1	100	2524.6	4417.8	100

The designs above resulted in the properties shown in Table 4 below.

Table 4 - Source 2 Properties

Property	9.5 mm Mix (%)	19 mm Mix (%)
Design Asphalt Content	6.87	6.15
Effective Asphalt Content	5.32	4.57
Binder Absorption	1.55	1.58

APPENDIX B

INSTRUCTIONS TO LABORATORIES FOR TESTING SAMPLES

NCHRP 9-26, Phase 4
Precision Statement for AASHTO T312
Source 2, 9.5-mm mix

April 3, 2006

To: Participants in NCHRP 9-26, Phase 4

From: Robert Lutz, Co-Principal Investigator

Subject: **Round 3 of NCHRP 9-26, Phase 4 (Source 2, 9.5-mm mix)**

We are sending to each of the 27 participating laboratories two boxes containing 9.5 mm loose mix for NCHRP 9-26. One box is labeled "**NCHRP 9-26: 9.5 mm – Source 2 Gyratory Samples**" and contains three, 9.5 mm loose mix samples weighing approximately 5000 g each. The other box is labeled "**NCHRP 9-26: 9.5 mm – Source 2 Rice Samples"** and contains three, 9.5 mm loose mix samples weighing approximately 2000 g each.

We would appreciate your cooperation in completing the tests as soon as possible in the order listed below, and according to the protocols indicated. The date by which all data should be reported is **April 21, 2006**. The data sheets and instructions are attached. You may email your data to rlutz@amrl.net. Alternatively, you may fax your data sheets to 301.975.8208 or mail them to:

AASHTO Materials Reference Laboratory
National Institute of Standards and Technology
100 Bureau Drive, Stop 8619
Gaithersburg, MD 20899-8619

All testing does not have to be performed by one individual. However, to permit comparisons of different methods and an estimate of single-operator precision, a single operator should make all three maximum specific gravity determinations; a single, but perhaps different, operator should compact all three gyratory specimens, and, a single, but perhaps different, operator should make all six bulk specific gravity determinations (all twelve, if additionally performing bulk specific gravity by the Corelok).
.
Attached are instructions for conducting the tests and data sheets for reporting the test results. Testing should be conducted in the following sequence:

Bulk Sp. Gr.	**Maximum Sp. Gr.**
1. Compact Specimens – T312-04	1. D2041-03
2. Corelok – D6752-03	
3. T166-05	

We appreciate your willingness to participate in this activity. If you have any questions, or you do not receive a full set of samples in good condition by April 7, 2006, please contact Robert Lutz at 301.975.3062.

Maximum Specific Gravity of Bituminous Paving Mixtures

ASTM D2041-03a

The three small boxes of mix labeled "NCHRP 9-26: 9.5 mm – Source 2 Rice Samples" are to be used for the maximum specific gravity test. The sample numbers are indicated on the outside of each sample box. The samples shall be heated in an oven at a temperature of 105 ± 5°C until soft enough to be separated.

- Each sample should be tested only once.
- Use either the **weighing-in-water (bowl)** or **weighing-in-air (bowl or flask)** method, and the mechanical agitation device described in the method.
- ***IMPORTANT: Follow the instructions described in Section 11 of the test method for drying back the mix, avoiding the steps that specify breaking large pieces of aggregate Specifically, decant the water from the container through a 75-µm (No. 200 sieve). Spread the sample on a flat, nonabsorptive tray, breaking up any agglomerations by hand, and dry the sample in front of a fan. Periodically, stir the sample by rolling it over upon itself. Weigh the tray and sample at 15-minute intervals. When mass loss is less than 0.05% for this interval, the sample is considered to be surface dry. This dry-back mass is substituted for *A* in the denominator in the equation for determining the maximum specific gravity.**

Weighing-in-Water Determination (Bowl)

	Sample #	Sample #	Sample #
Mass of Oven Dried Mix Sample in Air (0.1 g)			
Mass of Mix Sample in Air after the Dry-Back (0.1 g)			
Mass of Bowl in Water @ 25°C (0.1 g)			
Mass of Bowl+Mix in Water @ 25°C (0.1 g)			
Temperature of Water Bath (0.1°C)			
Maximum Specific Gravity, G_{mm} (0.001)			

Weighing-in-Air Determination (Flask or Bowl)

	Sample #	Sample #	Sample #
Mass of Oven Dried Mix Sample in Air (0.1 g)			
Mass of Mix Sample in Air after the Dry-Back (0.1 g)			
Mass of Bowl/Flask+Water @ 25°C (0.1 g)			
Mass of Bowl/Flask+Mix+Water @ 25°C (0.1 g)			
Temperature of Bowl/Flask and Contents (0.1°C)			
Maximum Specific Gravity, G_{mm} (0.001)			

Please submit all data by April 21, 2006

Name of Laboratory: ______________________ Phone No: ______________________

Tested by: ______________________ Date Tested: ______________________

Density of HMA Specimens by Means of the Superpave Gyratory Compactor

AASHTO T312-04

Verify the calibration of the gyratory compactor prior to compacting the specimens. The three large boxes of mix labeled "NCHRP 9-26: 9.5 mm –Source 2 Gyratory Samples" are to be compacted using the gyratory compactor. The sample numbers are indicated on the outside of each sample box. 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 gyratory compactor manufacturer, model, and angle verification method. **Please attach a copy of the printout for the specimen height after each gyration.**

Please submit all data by April 21, 2006.

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

Gyratory Manufacturer: ________________________ Model: ____________________

Angle Verification Method: ____

Name of Laboratory: ______________________________

Tested by: ______________________________

Phone No: ______________________________

Date Tested: ______________________________

Bulk Specific Gravity of Compacted Bituminous Mixtures Using Corelok

ASTM D6752-03

If applicable, perform the Corelok on the compacted specimens before performing T166.
Determine the bulk specific gravity of the compacted specimens at **25 ±0.5°C** using the Corelok before determining the bulk specific gravity by T166. Test each specimen twice and record the temperature, masses and the bulk specific gravity of each specimen below. The sample numbers are indicated on the outside of each sample box.

	Sample #		Sample #		Sample #	
	1st Test	2nd Test	1st Test	2nd Test	1st Test	2nd Test
(A) Sample mass in air (0.1 g)						
(B) Mass of dry, sealed specimen (0.1 g)						
(C) Mass of specimen after removal from sealed bag (0.1 g)						
(E) Mass of sealed specimen in water (0.1 g)						
(F_T) Apparent specific gravity of plastic sealing material						
Temperature of Water Bath (0.1°C)						
Bulk Specific Gravity, G_{mb} (0.001)						

Please submit all data by April 21, 2006.

Name of Laboratory: ________________________________

Tested by: ________________________________

Phone No: ________________________________

Date Tested: ________________________________

Bulk Specific Gravity of Compacted Bituminous Mixtures Using SSD Specimens

AASHTO T166-05

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. Perform this test on each specimen twice. After the first test, dry the specimen back to constant mass in front of a fan at room temperature. Constant mass is defined as the mass at which further drying under the fan does not alter the mass by more than 0.05%. Record the temperature, masses and the bulk specific gravity of the extruded specimen below. The sample numbers are indicated on the outside of each sample box.

	Sample #		Sample #		Sample #	
	1st Test	2nd Test	1st Test	2nd Test	1st Test	2nd Test
(A) Sample mass in air (0.1 g)						
(C) Sample mass in water (0.1 g)						
(B) SSD mass in air (0.1 g)						
Temperature of Water Bath (0.1°C)						
Bulk Specific Gravity, G_{mb} (0.001)						

Please submit all data by April 21, 2006.

Name of Laboratory: ______________________________

Tested by: ______________________________

Phone No: ______________________________

Date Tested: ______________________________

NCHRP 9-26, Phase 4
Precision Statement for AASHTO T312
***Source 2, 19--mm** mix*

April 26, 2006

To: Participants in NCHRP 9-26, Phase 4

From: Robert Lutz, Co-Principal Investigator

Subject: **Round 4 (LAST round) of NCHRP 9-26, Phase 4 (Source 2, 19-mm mix)**

We are sending to each of the 27 participating laboratories two boxes containing 19-mm loose mix for NCHRP 9-26. One box is labeled "**NCHRP 9-26: 19-mm – Source 2 Gyratory Samples**" and contains three, 19-mm loose mix samples weighing approximately 5000 g each. The other box is labeled "**NCHRP 9-26: 19-mm – Source 2 Rice Samples"** and contains three, 19-mm loose mix samples weighing approximately 2000 g each.

We would appreciate your cooperation in completing the tests as soon as possible in the order listed below, and according to the protocols indicated. The date by which all data should be reported is **May 19, 2006**. The data sheets and instructions are attached. You may email your data to rlutz@amrl.net . Alternatively, you may fax your data sheets to 301.975.8208 or mail them to:

AASHTO Materials Reference Laboratory
National Institute of Standards and Technology
100 Bureau Drive, Stop 8619
Gaithersburg, MD 20899-8619

All testing does not have to be performed by one individual. However, to permit comparisons of different methods and an estimate of single-operator precision, a single operator should make all three maximum specific gravity determinations; a single, but perhaps different, operator should compact all three gyratory specimens, and, a single, but perhaps different, operator should make all six bulk specific gravity determinations (all twelve, if additionally performing bulk specific gravity by the Corelok).
.

Attached are instructions for conducting the tests and data sheets for reporting the test results. Testing should be conducted in the following sequence:

Bulk Sp. Gr.	**Maximum Sp. Gr.**
1. Compact Specimens – T312-04	1. D2041-03
2. Corelok – D6752-03	
3. T166-05	

We appreciate your willingness to participate in this activity. If you have any questions, or you do not receive a full set of samples in good condition by May 5, 2006, please contact Robert Lutz at 301.975.3062.

Density of HMA Specimens by Means of the Superpave Gyratory Compactor

AASHTO T312-04

Verify the calibration of the gyratory compactor prior to compacting the specimens. The three large boxes of mix labeled "NCHRP 9-26: 19-mm – Source 2 Gyratory Samples" are to be compacted using the gyratory compactor. The sample numbers are indicated on the outside of each sample box. 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 gyratory compactor manufacturer, model, and angle verification method. **Please attach a copy of the printout for the specimen height after each gyration.**

Please submit all data by May 19, 2006.

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

Gyratory Manufacturer: __________________________ Model: ____________________

Angle Verification Method: ____

Name of Laboratory: _______________________________

Tested by: _______________________________

Phone No: _______________________________

Date Tested: _______________________________

Bulk Specific Gravity of Compacted Bituminous Mixtures Using Corelok

ASTM D6752-03

If applicable, perform the Corelok on the compacted specimens before performing T166.
Determine the bulk specific gravity of the compacted specimens at **25 ± 0.5°C** using the Corelok before determining the bulk specific gravity by T166. Test each specimen twice and record the temperature, masses and the bulk specific gravity of each specimen below. The sample numbers are indicated on the outside of each sample box.

	Sample #		Sample #		Sample #	
	1st Test	2nd Test	1st Test	2nd Test	1st Test	2nd Test
(A) Sample mass in air (0.1 g)						
(B) Mass of dry, sealed specimen (0.1 g)						
(C) Mass of specimen after removal from sealed bag (0.1 g)						
(E) Mass of sealed specimen in water (0.1 g)						
(F_T) Apparent specific gravity of plastic sealing material						
Temperature of Water Bath (0.1°C)						
Bulk Specific Gravity, G_{mb} (0.001)						

Please submit all data by May 19, 2006.

Name of Laboratory: ______________________________

Tested by: ______________________________

Phone No: ______________________________

Date Tested: ______________________________

Bulk Specific Gravity of Compacted Bituminous Mixtures Using SSD Specimens

AASHTO T166-05

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. Perform this test on each specimen twice. After the first test, dry the specimen back to constant mass in front of a fan at room temperature. Constant mass is defined as the mass at which further drying under the fan does not alter the mass by more than 0.05%. Record the temperature, masses and the bulk specific gravity of the extruded specimen below. The sample numbers are indicated on the outside of each sample box.

	Sample #		Sample #		Sample #	
	1st Test	2nd Test	1st Test	2nd Test	1st Test	2nd Test
(A) Sample mass in air (0.1 g)						
(C) Sample mass in water (0.1 g)						
(B) SSD mass in air (0.1 g)						
Temperature of Water Bath (0.1°C)						
Bulk Specific Gravity, G_{mb} (0.001)						

Please submit all data by May 19, 2006.

Name of Laboratory: ______________________________

Tested by: ______________________________

Phone No: ______________________________

Date Tested: ______________________________

APPENDIX C

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 deviations (1s limits) for mixtures containing aggregates with an absorption of less than 1.5 percent [A] and mixtures containing aggregates with an absorption of 4 percent to 5 percent [B] are shown in Table C-1. 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 d2s single operator limits shown in Table C-1.

1.1.2 Multilaboratory Precision - The multilaboratory standard deviations (1s limits) for mixtures containing aggregates with an absorption of less than 1.5 percent [A] and mixtures containing aggregates with an absorption of 4 percent to 5 percent [B] are shown in Table C-1. 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 d2s multilaboratory limits shown in Table C-1.

Table 0-1- Precision Estimates

	1s limit	d2s limit
Single Operator Precision:		
Aggregate with less than 1.5% absorption [A]	0.002	0.006
Aggregates with 4% to 5% absorption [B]	0.005	0.0013
Multilaboratory Precision:		
Aggregate with less than 1.5% absorption [A]	0.004	0.011
Aggregates with 4% to 5% absorption [B]	0.010	0.027

[A] *Based on an interlaboratory study described in NCHRP 9-26 Phase 1 Research Report involving twenty-six laboratories, two materials (a 12.5-mm mixture and a 19.0-mm mixture), three replicates, tested without use of Section 11.*

[B] *Based on an interlaboratory study described in NCHRP 9-26 Phase 4 Research Report involving twenty-six laboratories, four materials from two aggregate sources (absorptions between 4% to 5%) and two gradations (9.5-mm and 19.0-mm), three replicates, tested with use of Section 11.*

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 D

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 deviation has been found to be 0.012[A] (1s limit) for Method A, for mixtures containing aggregate with an absorption less than 1.5% and mixtures containing aggregate with an absorption between 4% to 5%. 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 0.033 (d2s limit).

1.1.2 Multilaboratory Precision - The multilaboratory standard deviation has been found to be 0.016 (1s limit) for Method A, for mixtures containing aggregate with an absorption less than 1.5% and mixtures containing aggregates with an absorption between 4% to 5% percent. 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 0.044 (d2s limit).

[A]*Based on two interlaboratory studies described in NCHRP 9-26 Phase 1 and Phase 4 Research Reports involving twenty-six laboratories. Phase 1 of the study included two mixtures (12.5-mm and 19.0-mm) containing aggregate with an absorption less than 1.5%. Phase 4 of the study included 4 mixtures from two aggregate sources (absorptions between 4% to 5%) and two gradations (9.5-mm and 19.0-mm). Specimens were compacted in three replicates according to AASHTO T312 150-mm diameter specimens with 4-7 percent air voids.*

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: ASTM PROVISIONAL TEST METHOD FOR BULK SPECIFIC GRAVITY AND DENSITY OF COMPACTED BITUMINOUS MIXTURES USING AUTOMATIC VACUUM SEALING METHOD, D6752

1. Precision and Bias

1.1 Precision

1.1.1 Single Operator Precision - The single operator standard deviation has been found to be 0.013[A] (1s limit) for Method A, for mixtures containing aggregate with an absorption less than 1.5% and mixtures containing aggregate with an absorption between 4% to 5%. 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 0.036 (d2s limit).

1.1.2 Multilaboratory Precision - The multilaboratory standard deviation has been found to be 0.021[A] (1s limit) for Method A, for mixtures containing aggregate with an absorption less than 1.5% and mixtures containing aggregates with an absorption between 4% to 5% percent. 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 0.059 (d2s limit).

[A]Based on two interlaboratory studies described in NCHRP 9-26 Phase 1 and Phase 4 Research Reports involving twenty-six laboratories. Phase 1 of the study included two mixtures (12.5-mm and 19-mm) containing aggregate with an absorption less than 1.5%. Phase 4 of the study included 4 mixtures from two aggregate sources (absorptions between 4% to 5%) and two gradations (9.5-mm and 19.0-mm). Specimens were compacted in three replicates according to AASHTO T312 150-mm diameter specimens with 4-7 percent air voids.

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

1.2 Precision

1.1.1 **Single Operator Precision** - The single operator standard deviations (1s limits) for mixtures containing aggregates with an absorption of less than 1.5 percent [A] and mixtures containing aggregates with an absorption of 4 percent to 5 percent [B] are shown in Table F-1. 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 d2s single operator limits shown in Table F-1.

1.1.2 **Multilaboratory Precision** - The multilaboratory standard deviations (1s limits) for mixtures containing aggregates with an absorption of less than 1.5 percent [A] and mixtures containing aggregates with an absorption of 4 percent to 5 percent [B] are shown in Table F-1. 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 d2s multilaboratory limits shown in Table F-1.

Table F-1- Precision Estimates

	1s limit	d2s limit
Single Operator Precision:		
Aggregate with less than 1.5% absorption [A]		
12.5-mm nominal maximum aggregates	0.3	0.9
19.0-mm nominal maximum aggregates	0.5	1.4
Aggregates with 4% to 5% absorption [B]	0.6	1.6
Multilaboratory Precision:		
Aggregate with less than 1.5% absorption [A]		
12.5-mm nominal maximum aggregates	0.6	1.7
19.0-mm nominal maximum aggregates	0.6	1.7
Aggregates with 4% to 5% absorption [B]	0.9	2.5

[A]Based on an interlaboratory study described in NCHRP 9-26 Phase 1 Research Report involving twenty-six laboratories, two mixtures (12.5-mm and 19-mm) containing aggregate with an absorption less than 1.5%. Specimens were compacted in three 150-mm diameter replicates with 4-5 percent air voids.

[A]Based on an interlaboratory study described in NCHRP 9-26 Phase 4 Research Report involving twenty-six laboratories, four mixtures from two aggregate sources (absorptions between 4% to 5%) and two gradations (9.5-mm and 19.0-mm). Specimens were compacted in three 150-mm diameter replicates with 4-7 percent air voids.

1.2 Bias

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