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Precision Estimates for AASHTO Test Method T308 and the Test Methods for Performance-Graded Asphalt Binder in AASHTO Specification M320

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TRANSPORTATION RESEARCH BOARD OF THE NATIONAL ACADEMIES

Submitted by:

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TABLE OF CONTR	ENTS	iv
LIST OF FIGURES		vi
LIST OF TABLES.		vi
ACKNOWLEDGM	ENTS	vii
	duction and Research Approach	
	on	
1.1.1		
	Research Objectives	
	Study	
1.3 Proficienc	y Samples Used in Study	3
CHAPTER 2: Analy	vsis Technique	4
2.1 Technique	e Overview	4
2.2 Steps of A	nalysis	
2.2.1	Remove Unpaired and Null Data (Step 1)	5
2.2.2	Determine Invalid Data (Step 2)	7
2.2.3	Determine Outliers (Step 3)	8
2.2.4	Analysis of Core Data (Step 4)	9
2.3 Check for	Normality	10
CHAPTER 3. Posul	ts of Analysis and Estimates of Precision	11
	of the Data	
2	Determining the Asphalt Binder Content of Hot-Mix	11
5.2.1	Asphalt (HMA) by the Ignition Method	11
3.2.2	Flash and Fire Points by Cleveland Open Cup	
3.2.2	Specific Gravity of Semi-Solid Bituminous Materials	
		12
3.2.4	Effect of Heat and Air on a Moving Film of Asphalt	10
2.2.5	(Rolling Thin-Film Oven Test)	13
3.2.5	Determining the Flexural Creep Stiffness of Asphalt Binder	1 /
2.2.(Using the Bending Beam Rheometer (BBR)	14
3.2.6	Determining the Fracture Properties of Asphalt Binder in	1.5
	Direct Tension (DT).	15
3.2.7	Determining the Rheological Properties of Asphalt Binder	
	Using a Dynamic Shear Rheometer (DSR)	16
3.2.8	Viscosity Determination of Asphalt Binder Using a	
	Rotational Viscometer	17
CHAPTER 4: Conc	lusions and Recommendations	18
4.2 Conclus	ions and Recommendations Related to Specific Standards	
4.2.1	AASHTO T308-04, Determining the Asphalt Binder Content of	
	Hot-Mix Asphalt (HMA) by the Ignition Method	

	4.2.2	AASHTO T48-04, Flash and Fire Points by Cleveland Open Cup	18
	4.2.3	AASHTO T228-04, Specific Gravity of Semi-Solid Bituminous	
		Materials	19
	4.2.4	AASHTO T240-03, Effect of Heat and Air on a Moving Film of	
		Asphalt (Rolling Thin-Film Oven Test)	
	4.2.5	AASHTO T313-04, Determining the Flexural Creep Stiffness	
		of Asphalt Binder Using the Bending Beam Rheometer (BBR)	
	4.2.6	AASHTO T314-04, Determining the Fracture Properties of	
		Asphalt Binder in Direct Tension (DT)	
	4.2.7	AASHTO T315-04, Determining the Rheological Properties of	
		Asphalt Binder Using a Dynamic Shear Rheometer (DSR)	21
	4.2.8	AASHTO T316-04, Viscosity Determination of Asphalt Binder	
		Using a Rotational Viscometer	21
4.3	General	Conclusions and Recommendations.	
4.4		n Statement for AASHTO T308	
4.5		n Statement for AASHTO T48	
4.6		n Statement for AASHTO T228	
4.7	Precisio	n Statement for AASHTO T240	26
4.8	Precisio	n Statement for AASHTO T313	28
4.9		n Statement for AASHTO T314	
4.10		n Statement for AASHTO T315	
4.11	Precisio	n Statement for AASHTO T316	31
BIBLIOGR	APHY		33
APPENDIX	A De	escription of Hoaglin et al. Outlier Method	A-1
APPENDIX		xample of Analysis Technique	
APPENDIX		Immary Table for DSR Phase Angle Testing on Original Binder	
APPENDIX		ormal Summary Tables	
APPENDIX	E GI	raph and Analysis Results for AASHTO T308	E-1
APPENDIX	F Gi	raph and Analysis Results for AASHTO T48	F-1
APPENDIX		raph and Analysis Results for AASHTO T228	
APPENDIX	CH G	raph and Analysis Results for AASHTO T240	H-1
APPENDIX	CI Gi	raph and Analysis Results for AASHTO T313, Slope	I-1
APPENDIX	(J Gi	raph and Analysis Results for AASHTO T313, Stiffness	J-1
APPENDIX		raph and Analysis Results for AASHTO T314, Stress	
APPENDIX	L Gi	raph and Analysis Results for AASHTO T314, Strain	L-1
APPENDIX		raph and Analysis Results for AASHTO T315, Original	
APPENDIX	N Gi	raph and Analysis Results for AASHTO T315, RTFO	N-1
APPENDIX	CO GI	raph and Analysis Results for AASHTO T315, PAV	O- 1
APPENDIX	CP GI	raph and Analysis Results for AASHTO T316	P- 1

LIST OF FIGURES

Figure 1	Visual Representation of Analysis Technique	6
Figure 2	Graphical Representation of Using Inner 75% of Data to Determine	
	Invalid Data	7
Figure 3	Graphical Representation of Using Inner 75% of Data to Determine Outlie	ers8
Figure 4	Repeatability Graph for T240	12
Figure 5	Reproducibility Graph for T240	12
Figure 6	Determination of Invalid Data	B-2
Figure 7	Determination of Outliers	B-4

LIST OF TABLES

Table 1	Proficiency Samples Used in Analysis of T308	
Table 2	Proficiency Samples Used in Analysis of T240, T313, T314, T315, and T316	3
Table 3	Proficiency Samples Used in Analysis of T48 and T228	3
Table 4	Summary Table for T308, Percent Asphalt (%)	. 10
Table 5	Summary Table for T48, Flash Point (°C)	. 11
Table 6	Summary Table for T228, Specific Gravity	. 11
Table 7	Summary Table for T240, Change in Mass (%)	. 12
Table 8	Summary Table for T313, Slope (m-value)	. 13
Table 9	Summary Table for T313, Creep Stiffness (MPa)	. 13
Table 10	Summary Table for T314, Stress (MPa)	. 14
Table 11	Summary Table for T314, Percent Strain (%)	. 14
Table 12	Summary Table for T315, Original G*/sinð, (kPa)	
Table 13	Summary Table for T315, RTFO G*/sinð, (kPa)	. 15
Table 14	Summary Table for T315, PAV G*·sinδ, (kPa)	. 16
Table 15	Summary Table for T316, Viscosity (Pa·s)	. 16
Table 16	Table of Statistics and Limits	. B- 1
Table 17	Example Data	. B- 1
Table 18	Table of Statistics and Limits	.B-3
Table 19	Example Data	.B-3
Table 20	Summary Table for T315, Phase Angle for Original Binder	.C-1
Table 21	T308 Ignition Oven	.E-1
Table 22	T48 Cleveland Flash	.E-1
Table 23	T228 Specific Gravity	.E-1
Table 24	T240 RTFO Loss	.E-1
Table 25	T313 BBR Slope	.E-1
Table 26	T313 BBR Stiffness	.E-1
Table 27	T314 DT Stress	.E-1
Table 28	T314 DT Strain	.E-1
Table 29	T315 DSR Original	
Table 30	T315 DSR RTFO	
Table 31	T315 DSR PAV	.E-1
Table 32	T316 Rotational Viscosity	.E-1

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CHAPTER 1: INTRODUCTION AND RESEARCH APPROACH

1.1 INTRODUCTION

Under National Cooperative Highway Research Programs (NCHRP) Project 9-26, the AASHTO Materials Reference Laboratory (AMRL) is conducting a multi-phase research project to improve estimates of precision in AASHTO test methods for asphalt binder and hot-mix asphalt (HMA). The report from Phase 1 of Project 9-26 includes precision estimates of selected volumetric properties of HMA using non-absorptive aggregates [1]. The report from Phase 2 discusses the results of an investigation into the cause of variations in HMA bulk specific gravity test results using non-absorptive aggregates [2].

This report includes the results of Phase 3 of NCHRP 9-26 where data from the AMRL Proficiency Sample Program (PSP) are used to create or update precision estimates for a variety of test methods. This includes those specified in AASHTO Standard Specification M320, "Performance-Graded Asphalt Binder", and AASHTO Standard Test Method T308, "Determining the Asphalt Binder Content of Hot-Mix Asphalt (HMA) by the Ignition Method" [3,4].

Laboratories participating in the AMRL Proficiency Program receive annual or biannual shipments of paired proficiency samples which are tested according to specified AASHTO test methods [5,6]. The results of the testing are returned to AMRL for analysis, summarization, and reporting back to the laboratories. AMRL has an extensive database of test results for the broad range of construction materials included in its proficiency sample program. Data used in this study are for HMA ignition samples (T308), and for test methods for performance-graded asphalt binder and viscosity graded asphalt (M320). The proficiency samples included in these programs cover a range of test values and grades of materials.

This report includes a robust technique developed by AMRL for analyzing proficiency sample data. This technique is a four step methodology for shaving off extraneous results and analyzing the core data of a paired data set. The results of the analysis of the "core data" can then be used to obtain reliable single-operator and multilaboratory estimates of precision.

In this study, over 91 paired data sets comprised of over 28,000 test results were analyzed using the analysis technique developed. The analysis resulted in precision statements for eight separate test methods. In order to account for changes in test precision resulting from recent improvements in the test methods, only the most recent proficiency samples were used.

1.1.1 PROBLEM STATEMENT

AASHTO Standard Test Methods applicable to highway materials require periodic studies to determine estimates of precision. Some precision estimates become outdated as a result of improvements in the methods while other estimates need to be verified to see if they are still accurate. Others need to be expanded to take into account a wider range of materials while some newer test methods may not have precision estimates of any kind. This study addresses specific tests having these deficiencies.

1.1.2 RESEARCH OBJECTIVES

The objective of Phase 3 of NCHRP Project 9-26, herein referred to as the Phase 3 study, is to develop or update single-operator and multilaboratory precision estimates for the following test methods:

1. AASHTO T308	Determining the Asphalt Binder Content of Hot-Mix Asphalt (HMA) by the Ignition Method
2. AASHTO T48	Flash and Fire Points by Cleveland Open Cup
3. AASHTO T228	Specific Gravity of Semi-Solid Bituminous Materials
4. AASHTO T240	Effect of Heat and Air on a Moving Film of Asphalt (Rolling Thin-Film Oven Test)
5. AASHTO T313	Determining the Flexural Creep Stiffness of Asphalt Binder Using the Bending Beam Rheometer (BBR)
6. AASHTO T314	Determining the Fracture Properties of Asphalt Binder in Direct Tension (DT)
7. AASHTO T315	Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)
8. AASHTO T316	Viscosity Determination of Asphalt Binder Using a Rotational Viscometer

1.2 SCOPE OF STUDY

This work is limited to an evaluation of data collected from laboratories participating in the Performance Graded Asphalt Binder, Viscosity Graded Asphalt Cement, and Hot-Mix Asphalt Ignition Oven portions of the AMRL Proficiency Sample Program. There are 91 data sets analyzed and included in this report.

1.3 PROFICIENCY SAMPLES USED IN STUDY

Included in the study are the most recent AMRL proficiency samples that include the test methods covered in the Research Objectives (Section 1.1.2). These samples include multiple grades of material when it was possible to do so. The following tables describe the pertinent information for the samples used.

	ample ignation	Performance Grade	Viscosity Grade	Date of Final Report	Modified Binder
IGN	3 & 4	PG 64-22	AC 20	March 2002	No
IGN	5&6	PG 64-22	AC 20	March 2003	No
IGN	7&8	PG 52-34	AC 10	March 2004	No
	1 4 D	a b c	1 77		A TT A A A

 Table 1 – Proficiency Samples Used in Analysis of T308

The hot-mix asphalt ignition oven (IGN) samples listed in Table 1 are used in the analysis of T308.

Sample	Performance	•		Modified
Designation	Grade	Grade	Final Report	Binder
PGB 181 & 182	PG 64-16	AC 10	January 2001	No
PGB 183 & 184	PG 70-22		June 2001	No
PGB 185 & 186	PG 64-22	AC 20	January 2002	No
PGB 187 & 188	PG 76-22		May 2002	Yes
PGB 189 & 190	PG 64-22	AC 30	December 2002	No
PGB 191 & 192	PG 52-34	AC 10	May 2003	No
PGB 193 & 194	PG 64-22	AC 20	December 2003	No
PGB 195 & 196	PG 70-22		May 2004	No

Table 2 – Proficiency Samples Used in the Analysis ofT240, T313, T314, T315, and T316

The performance graded asphalt binder (PGB) samples listed in Table 2 are used in the analysis of T240, T313, T314, T315, and T316. (The PG 76-22 is an SBS modified binder.)

Sample	Performance	•	Date of	Modified
Designation	Grade	Grade	Final Report	Binder
BAC 181 & 182	PG 64-16	AC 10	January 2001	No
BAC 183 & 184	PG 70-22		June 2001	No
BAC 185 & 186	PG 64-22	AC 20	January 2002	No
BAC 187 & 188	PG 64-22	AC 30	May 2002	No
BAC 189 & 190	PG 64-22	AC 30	December 2002	No
BAC 191 & 192	PG 52-34	AC 10	May 2003	No
BAC 193 & 194	PG 64-22	AC 20	December 2003	No
PGB 195 & 196	PG 70-22		May 2004	No

Table 3 – Proficiency Samples Used in the Analysis of T48 and T228

The viscosity graded asphalt cement (BAC) samples and one PGB sample listed in Table 3 are used in the analysis of T48 and T228.

CHAPTER 2: ANALYSIS TECHNIQUE

2.1 TECHNIQUE OVERVIEW

The analysis method used to determine precision estimates for this study is designed to determine robust estimates of precision representative, as much as possible, of testing performed in accordance with the test standards. The desire is to obtain estimates that will compare favorably to those that might be obtained from a strictly controlled inter-laboratory study. A literature survey was conducted to investigate methods applicable to the AMRL PSP data. Where applicable, sources used for the development of the analysis technique will be referenced in the following sections. The method is designed to extract the core of the data from the data sets and then to analyze that core to determine repeatability and reproducibility precision estimates. It is these data that stand the best chance of representing testing performed in conformance with each of the test methods.

The AMRL Proficiency Sample Program is based on the testing of two samples of the same material having nearly identical, but not necessarily exactly identical, test properties. This type of program is described by Arni, Crandall and Blaine, and Youden [7,8,9]. One test is performed on each of the samples. This type of program provides two independent test results from each laboratory and allows for the evaluation of both within-laboratory and between-laboratory performance and for determining corresponding estimates of precision. The within-laboratory data are obtained under repeatability conditions by specifying the test method and by having testing in each laboratory performed by a single operator using the same equipment in a short period of time. The between-laboratory data are obtained under reproducibility conditions with different operators in different laboratories using different equipment.

The number of participants in the AMRL program is sufficiently large enough to ensure a statistically sound basis for determination of estimates of precision for standard test methods among laboratories using various types of equipment [10]. For most of the standards under consideration of this study, the number of participants is on the order of several hundred. Even for those tests for which the populations are smaller, the number of participants is sufficiently large (in the range of thirty to fifty) for a sound inter-laboratory study [10].

Due to the relatively large number of participants in the PSP it is expected that the original data obtained during a round of testing contains a significant number of test results submitted from laboratories whose testing procedures may not be in conformance to the test standards or whose equipment may not meet the requirements specified in the test methods. The analysis technique is an attempt to identify and eliminate those test results prior to determining the precision estimates.

The analysis method used in this study employs procedures to identify invalid data and outlying data by extrapolating to cutoff points in the extremes of a data set based on the spread of the most reliable data near the center (or median) of the data set. Precision estimates are then determined from the "core" of reliable data that remains after invalid data and outlying data are removed.

As shown in Figure 1, the analysis technique employs a four step process. First, null responses and unpaired data (i.e. where laboratories did not submit results for both samples, x and y) are removed (Section 2.2.1). Second, invalid data are removed (Section 2.2.2). Third, outliers are removed (Section 2.2.3). Forth and finally, traditional standard deviation-type analyses are performed on the remaining core data to obtain estimates of repeatability and reproducibility precision (Section 2.2.4).

The first three steps are applied to the between-laboratory results for each of the two samples and also to the within-laboratory results. The criteria in the first three steps used for the elimination process help to assure that the results for each of the test samples contain data representative of testing performed in conformance with the test method.

The within-laboratory, or repeatability, data to which the criteria are applied are numerically equal to the difference between the two results submitted, one for each of the two test samples, by each laboratory. The difference between the two results is adjusted for any difference between the median values for each of the two samples according to the following equation [9]:

Repeatability data point:

$$r_i = (x_i - y_i) - (x_{med} - y_{med}) \quad \text{for } i = 1 \text{ to } n \quad (\text{Equation } 1)$$

Where:

n = number of laboratories

 x_i = result from laboratory 'i' on sample 'x',

 y_i = result from laboratory 'i' on sample 'y',

 x_{med} = median of test results from all laboratories on sample x,

 y_{med} = median of test results from all laboratories on sample y.

2.2 STEPS OF ANALYSIS

2.2.1 REMOVE UNPAIRED AND NULL DATA (STEP 1)

The analysis technique will not work for null and unpaired data. As a result, all null and unpaired data from the x and y data sets are removed prior to being analyzed. Unpaired data result from participating laboratories that submit results for only one of the two samples. Null responses occur from laboratories that receive the PSP samples but do not submit any testing results.

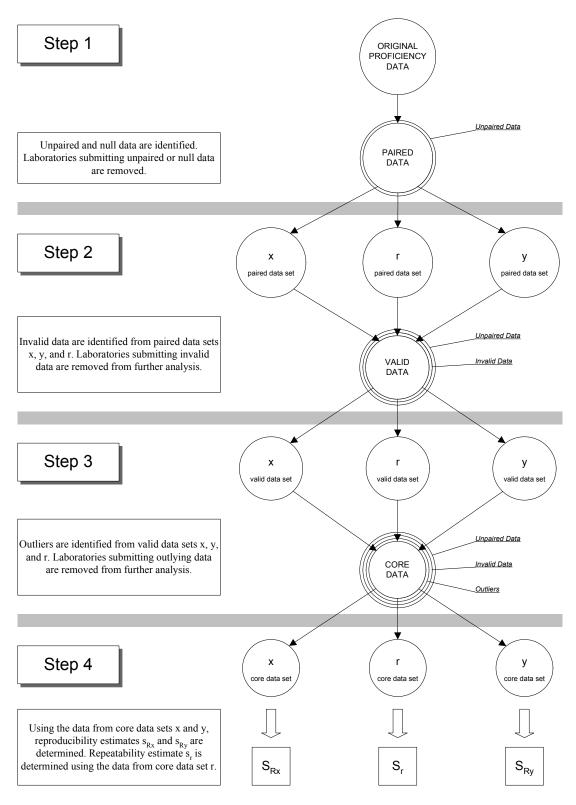


Figure 1 – Visual Representation of Analysis Technique

2.2.2 DETERMINE INVALID DATA (STEP 2)

Invalid data are defined as data falling above and below the values I_U and I_L , respectively; using Equations 2 and 3 based on Hoaglin et al [11,12]. See Appendix A for a more detailed description.

$$I_U = RI_{75U} + (1.555(RI_{75})) = \text{upper limit for invalid data}$$
(Equation 2)
$$I_L = RI_{75L} + (1.555(RI_{75})) = \text{lower limit for invalid data}$$
(Equation 3)

Where:

 $RI_{75} = RI_{75U} - RI_{75L}$ = the range of the inner 75% of data $RI_{75U} = 87.5^{\text{th}}$ percentile point of data (upper extent of the range of the inner 75 percent of all paired data) $RI_{75L} = 12.5^{\text{th}}$ percentile point of data (lower extent of the range of the inner 75 percent of all paired data)

Data determined to be invalid (i.e. falling beyond I_U and I_L) are beyond the equivalent of 4.725 standard deviations from the median value [11,12]. Even though this robust technique is applicable to Gaussian and non-Gaussian data [13], for normally distributed data, the probability is approximately 0.0000024 that data lying beyond I_U and I_L should be included in the population of results [11]. Any laboratory submitting invalid data is eliminated from further analysis. Figure 2 below gives a graphical representation of the location of the upper and lower limits for invalid data.

Appendix B gives a step-by-step example of how the equations are used to identify invalid data.

Using the Inner 75% to Identify Invalid Data

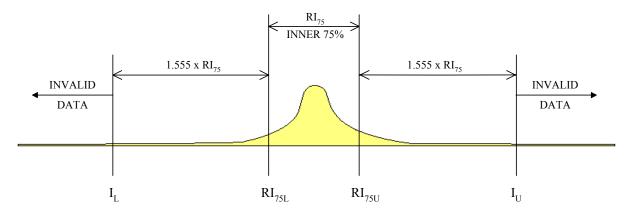


Figure 2 – Graphical Representation of Using the Inner 75% of Data to Determine Invalid Data

2.2.3 DETERMINE OUTLIERS (STEP 3)

Outliers are defined as data falling above and below the values O_U and O_L , respectively; using Equations 4 and 5 based on Hoaglin et al [11,12]. See Appendix A for a more detailed description.

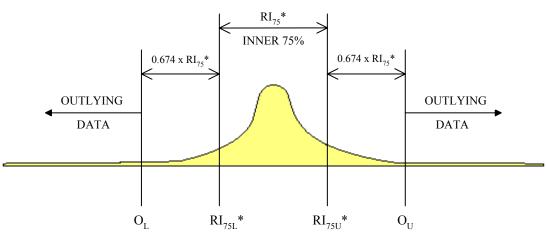
$$O_U = RI_{75U}^* + (0.674(RI_{75}^*)) =$$
 upper limit for outlying data (Equation 4)
 $O_L = RI_{75L} - (0.674(RI_{75}^*)) =$ lower limit for outlying data (Equation 5)

Where:

- $RI_{75}^{*} = RI_{75U}^{*} RI_{75L}^{*}$ = the range of the inner 75% of data without invalid data
- RI_{75U}^{*} = revised 87.5th percentile point of valid data (i.e. upper extent of the inner 75 percent of data remaining after the removal of invalid data)
- RI_{75L}^{*} = revised 12.5th percentile point of valid data (i.e. lower extent of the inner 75 percent of data remaining after the removal of invalid data)

Using the method described above, outliers fall beyond the equivalent of 2.7 standard deviations from the median value [11,12]. Similar to the method for determining invalid data, this technique is also applicable to Gaussian and non-Gaussian types of distributions [13]. However, the probability is approximately 0.007 [11] that data lying beyond the designated limits, O_U and O_L , should be included in the population of results for normally distributed data. Any laboratory submitting outlying results is eliminated from further analysis. Figure 3 below gives a graphical representation of the location of the upper and lower limits for outliers.

Appendix B gives a step-by-step example of how the equations are used to identify outlying data.



Using the Revised Inner 75% to Identify Outlying Data

Figure 3 – Graphical Representation of Using the Inner 75% of Data to Identify Outliers

2.2.4 ANALYSIS OF CORE DATA (STEP 4)

Once laboratories submitting either invalid or outlying data are eliminated, traditional standard deviation-type analyses are performed on the remaining data to determine repeatability and reproducibility precision estimates.

Since the two samples comprising a pair of AMRL proficiency samples are not identical in many cases, s_r (repeatability) estimates are obtained in the manner described by Youden [9] by applying the following equation to the paired data:

$$s_r = \sqrt{\frac{\sum \left[(x_i - y_i) - (\overline{x} - \overline{y}) \right]^2}{2(n-1)}}$$
 (Equation 6)

Where:

 s_r = repeatability estimate

- x_i = laboratory test result from the odd number sample of a pair
- y_i = laboratory test result from the even number sample of a pair

 \overline{x} = average of all x_i

 \overline{y} = average of all y_i

n = number of laboratories

This equation removes any actual differences in the samples and allows the paired test results to be treated as replicates.

Reproducibility estimates, s_{Rx} and s_{Ry} , are obtained independently for each of the two samples by applying the following equations for determining the sample standard deviations [3].

$$s_{Rx} = \sqrt{\left(\frac{\sum(x_i - \overline{x})^2}{n - 1}\right)}$$
 (Equation 7)
$$s_{Ry} = \sqrt{\left(\frac{\sum(y_i - \overline{y})^2}{n - 1}\right)}$$
 (Equation 8)

Where:

 s_{Rx} = reproducibility estimate for odd number sample pair

- s_{Ry} = reproducibility estimate for even number sample pair
- x_i = laboratory test result from the odd number sample of a pair
- y_i = laboratory test result from the even number sample of a pair
- \overline{x} = average of all x_i
- \overline{y} = average of all y_i
- n = number of laboratories

2.3 Check for Normality

According to ASTM E 177, the multiplier for determining the difference two-sigma (d2s) limits assumes an underlying normal distribution. To ensure the assumption of normality is a correct assumption, a comparison was made of the average 95% limits, for the differences between two results, by count to the pooled d2s limits for each of the 12 data groupings. The summary tables comparing the average 95% limits by count and the pooled d2s limits can be found in Appendix D. The Coefficient of Correlation from normal probability plotting can also be found in Appendix D.

CHAPTER 3: RESULTS OF ANALYSIS AND ESTIMATES OF PRECISION

3.1 TEST DATA

The individual results for each of the 91 proficiency data sets used to create precision estimates can be found in Appendices G to R. This chapter includes summaries of the data and the resulting precision estimates.

3.2 ANALYSIS OF THE DATA

The following tables and, in some cases, graphs display the results of the analyses. Precision estimates are based, where appropriate, on either the coefficients of variation (CV%) or the pooled standard deviation (1s) values. In one instance, an equation is used to express precision.

3.2.1 Determining the Asphalt Binder Content of Hot-Mix Asphalt (HMA) by the Ignition Method, AASHTO T308

Results from analyzing the data for Asphalt Binder Content of HMA by the Ignition Method are found in Appendix G.

					Average	Average Results		Repeatability			Reproducibility		cibility		
Sample	Sample	No. of	PG	AC	odd	even		odd	even	od	d	eve	en		
Туре	Numbers	Labs	Grade	Grade	samples	samples		samples	samples	samples		samples		samp	oles
							1s	CV%	CV%	1s	CV%	1s	CV%		
IGN	3 & 4	353	PG 64-22	AC 20	4.049	4.256	0.064	1.57	1.49	0.107	2.63	0.108	2.55		
IGN	5&6	399	PG 64-22	AC 20	4.802	5.098	0.072	1.49	1.41	0.119	2.47	0.116	2.28		
IGN	7&8	461	PG 52-34	AC 10	4.480	4.745	0.072	1.60	1.51	0.124	2.77	0.121	2.55		

Table 4 – Summary Table for T308, Percent Asphalt (%)

A review of the data shown in Table 4 indicates that the form of the precision estimates should be based on the sample standard deviation. The pooled repeatability sample standard deviation for the three pairs of samples analyzed is 0.069 percent. The corresponding pooled reproducibility sample standard deviation is 0.117 percent. The pooled estimates are derived using the following equation from Ku [14]:

$$s_{p} = \sqrt{\frac{(n_{1} - 1)s_{1}^{2} + (n_{2} - 1)s_{2}^{2} + \dots + (n_{k} - 1)s_{k}^{2}}{n_{1} + n_{2} + \dots + n_{k} - k}}$$
(Equation 9)

Where:

 s_p = pooled standard deviation

 $s_k = k^{\text{th}}$ standard deviation

 n_k = number of laboratories analyzed resulting in kth standard deviation

3.2.2 Flash and Fire Points by Cleveland Open Cup, T48

Results from analyzing the data for Flash Point by Cleveland Open Cup are found in Appendix H. There are no modified binders used in the analysis. Additionally, fire point data are not collected in the AMRL PSP.

					Average Results]	Repeatabil	ity	Reproducibility		Reproducibility	
Sample	Sample	No. of	PG	AC	odd	even		odd	even	od	d	eve	en
Туре	Numbers	Labs	Grade	Grade	samples	samples		samples	samples	sam	ples	samj	ples
							1s	CV%	CV%	1s	CV%	1s	CV%
BAC	181 & 182	107	PG 64-16	AC 10	271.7	272.3	2.4	0.9	0.9	9.1	3.3	9.6	3.5
BAC	183 & 184	98	PG 70-22		353.1	353.5	2.5	0.7	0.7	9.2	2.6	8.5	2.4
BAC	185 & 186	113	PG 64-22	AC 20	323.0	323.7	3.5	1.1	1.1	12.3	3.8	12.7	3.9
BAC	187 & 188	116	PG 64-22	AC 30	274.0	273.8	2.5	0.9	0.9	12.2	4.5	11.9	4.3
BAC	189 & 190	134	PG 64-22	AC 30	318.1	317.8	2.8	0.9	0.9	9.0	2.8	8.9	2.8
BAC	191 & 192	121	PG 52-34	AC 10	271.9	268.5	4.4	1.6	1.6	12.3	4.5	12.1	4.5
BAC	193 & 194	118	PG 64-22	AC 20	330.8	331.4	2.4	0.7	0.7	7.1	2.2	7.3	2.2
PGB	195 & 196	148	PG 70-22		350.8	350.8	1.7	0.5	0.5	7.7	2.2	7.4	2.1

Table 5 – Summary Table for T48, Flash Point (°C)

A review of the data shown in Table 5 indicates that the form of the precision estimates should be based on the sample standard deviation. The pooled repeatability sample standard deviation for the eight pairs of samples analyzed is 3°C. The corresponding pooled reproducibility sample standard deviation is 10°C. The pooled estimates are derived using Equation 9.

3.2.3 Specific Gravity of Semi-Solid Bituminous Materials, T228

Results from analyzing the data for Specific Gravity can be found in Appendix I. There are no modified binders used in the analysis.

					Average	e Results]	Repeatabil	ity	Reprodu	cibility	Reprodu	cibility
Sample	Sample	No. of	PG	AC	odd	even		odd	even	odd		eve	en
Туре	Numbers	Labs	Grade	Grade	samples	samples		samples sam		samples		samp	oles
							1s	CV%	CV%	1s	CV%	1s	CV%
BAC	181 & 182	104	PG 64-16	AC 10	1.0159	1.0157	0.0006	0.060	0.060	0.0012	0.119	0.0012	0.119
BAC	183 & 184	101	PG 70-22		1.0425	1.0428	0.0010	0.100	0.100	0.0016	0.156	0.0017	0.161
BAC	185 & 186	104	PG 64-22	AC 20	1.0330	1.0329	0.0008	0.076	0.076	0.0013	0.129	0.0013	0.124
BAC	187 & 188	112	PG 64-22	AC 30	1.0345	1.0344	0.0006	0.062	0.062	0.0011	0.110	0.0013	0.126
BAC	189 & 190	112	PG 64-22	AC 30	1.0308	1.0308	0.0006	0.061	0.061	0.0010	0.095	0.0008	0.077
BAC	191 & 192	121	PG 52-34	AC 10	1.0273	1.0274	0.0007	0.071	0.071	0.0013	0.127	0.0012	0.118
BAC	193 & 194	110	PG 64-22	AC 20	1.0058	1.0058	0.0006	0.062	0.062	0.0010	0.098	0.0008	0.079
PGB	195 & 196	137	PG 70-22		1.0404	1.0404	0.0007	0.067	0.067	0.0014	0.134	0.0013	0.125

Table 6 – Summary Table for T228, Specific Gravity

A review of the data shown in Table 6 indicates that the form of the precision estimates should be based on the sample standard deviation. The pooled repeatability sample standard deviation for the eight pairs of samples analyzed is 0.0008. The corresponding pooled reproducibility sample standard deviation is 0.0013. The pooled estimates are derived using Equation 9.

3.2.4 Effect of Heat and Air on a Moving Film of Asphalt (Rolling Thin-Film Oven Test), T240

Results from analyzing the data for change in mass using the RTFO can be found in Appendix J. One pair of modified binders, sample numbers 187 and 188, is used in the analysis.

					Average	e Results]	Repeatabil	ity	Reproducibility		Reprodu	cibility		
Sample	Sample	No. of	PG	AC	odd	even		odd	even	odd		odd		eve	en
Туре	Numbers	Labs	Grade	Grade	samples	samples		samples	samples	samples		samp	oles		
							1s	CV%	CV%	1s	CV%	1s	CV%		
PGB	181 & 182	170	PG 64-16	AC 10	-0.2740	-0.2646	0.0160	5.8	6.0	0.0570	20.8	0.0568	21.4		
PGB	183 & 184	172	PG 70-22		-0.0515	-0.0505	0.0087	16.9	17.2	0.0211	40.9	0.0203	40.3		
PGB	185 & 186	166	PG 64-22	AC 20	-0.2658	-0.2630	0.0149	5.6	5.7	0.0433	16.3	0.0424	16.1		
PGB	187 & 188	174	PG 76-22		-0.3435	-0.3363	0.0212	6.2	6.3	0.0722	21.0	0.0676	20.1		
PGB	189 & 190	171	PG 64-22	AC 30	-0.0358	-0.0369	0.0076	21.3	20.7	0.0219	61.1	0.0218	59.0		
PGB	191 & 192	191	PG 52-34	AC 10	-0.5133	-0.5107	0.0233	4.5	4.6	0.0827	16.1	0.0811	15.9		
PGB	193 & 194	176	PG 64-22	AC 20	-0.0321	-0.0336	0.0063	19.7	18.9	0.0219	68.1	0.0225	66.9		
PGB	195 & 196	191	PG 70-22		-0.0515	-0.0503	0.0074	14.3	14.6	0.0190	36.8	0.0184	36.6		

Table 7 – Summary Table for T240, Change in Mass (%)

A review of the data shown in Table 7 indicates that the standard deviation can be expressed as a function of the mass change (x) by using the equations described below.

The repeatability standard deviation, s_r , for the eight pairs of samples analyzed is determined to be best described using the following equation:

$$s_r = 0.0061 + 0.0363(x)$$
 (Equation 10)

The reproducibility standard deviation, s_R , for the eight pairs of samples analyzed is determined to be best described using the following equation:

$$s_R = 0.0153 + 0.1365(x)$$
 (Equation 11)

Where:

- s_r = reproducibility standard deviation
- s_R = reproducibility standard deviation
- x = mass loss in percent (a loss of mass is expressed as a negative number)

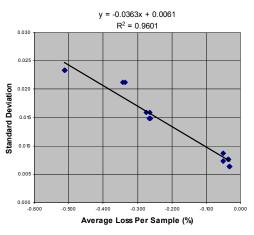


Figure 4 – Repeatability Graph for T240

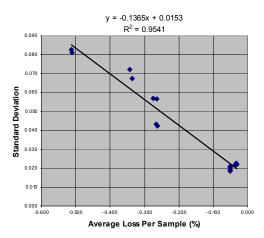


Figure 5 – Reproducibility Graph for T240

3.2.5 Determining the Flexural Creep Stiffness of Asphalt Binder Using the Bending Beam Rheometer (BBR), T313

3.2.5.1 SLOPE

Results from analyzing the data for BBR Slope can be found in Appendix K. One pair of modified binders, sample numbers 187 and 188, is used in the analysis.

					Average	e Results		Repeatability	r	Reprodu	cibility	Reproduc	cibility
Sample	Sample	No. of	PG	AC	odd	even		odd	even	ode	đ	eve	n
Туре	Numbers	Labs	Grade	Grade	samples	samples		samples	samples	samp	les	samp	les
							1s	CV%	CV%	1s	CV%	1s	CV%
PGB	181 & 182	174	PG 64-16	AC 10	0.3686	0.3697	0.0041	1.10	1.10	0.0085	2.31	0.0090	2.44
PGB	183 & 184	178	PG 70-22		0.3341	0.3339	0.0039	1.16	1.16	0.0067	2.01	0.0077	2.29
PGB	185 & 186	182	PG 64-22	AC 20	0.3303	0.3297	0.0037	1.12	1.13	0.0084	2.55	0.0090	2.72
PGB	187 & 188	189	PG 76-22		0.3735	0.3730	0.0039	1.04	1.04	0.0098	2.61	0.0089	2.39
PGB	189 & 190	185	PG 64-22	AC 30	0.3135	0.3135	0.0032	1.02	1.02	0.0076	2.41	0.0081	2.58
PGB	191 & 192	189	PG 52-34	AC 10	0.3106	0.3101	0.0031	0.99	0.99	0.0083	2.66	0.0086	2.76
PGB	193 & 194	196	PG 64-22	AC 20	0.3090	0.3086	0.0025	0.80	0.80	0.0058	1.88	0.0060	1.94
PGB	195 & 196	196	PG 70-22		0.3202	0.3197	0.0029	0.92	0.92	0.0083	2.58	0.0083	2.59

Table 8 – Summary Table for T313, Slope (m-value)

A review of the data shown in Table 8 indicates that the form of the precision estimates should be based on the coefficient of variation (CV%). The average repeatability coefficient of variation for the eight pairs of samples analyzed is 1.0 percent. The corresponding average reproducibility coefficient of variation is 2.4 percent. In each case, the average coefficient of variation is determined by calculating the "simple arithmetic average" as described in Section 8.4.2 of ASTM C802-96 [15].

3.2.5.2 STIFFNESS

Results from analyzing the data for BBR Stiffness can be found in Appendix L. One pair of modified binders, sample numbers 187 and 188, is used in the analysis.

					Average	e Results		Repeatabili	ty	Reprodu	cibility	Reprodu	cibility
Sample	Sample	No. of	PG	AC	odd	even		odd	even	od	d	eve	en
Туре	Numbers	Labs	Grade	Grade	samples	samples		samples	samples	samp	oles	samp	oles
							1s	CV%	CV%	1s	CV%	1s	CV%
PGB	181 & 182	179	PG 64-16	AC 10	182.8	179.2	6.1	3.32	3.39	14.7	8.03	14.5	8.09
PGB	183 & 184	188	PG 70-22		179.0	179.6	3.9	2.19	2.18	11.7	6.54	12.0	6.68
PGB	185 & 186	181	PG 64-22	AC 20	197.3	196.2	5.6	2.83	2.85	11.4	5.78	12.1	6.16
PGB	187 & 188	184	PG 76-22		125.4	125.5	3.2	2.59	2.59	7.8	6.22	8.4	6.73
PGB	189 & 190	192	PG 64-22	AC 30	216.7	216.1	5.8	2.68	2.69	12.8	5.89	13.2	6.09
PGB	191 & 192	181	PG 52-34	AC 10	225.9	225.4	4.9	2.18	2.18	14.0	6.21	14.5	6.45
PGB	193 & 194	193	PG 64-22	AC 20	158.7	158.9	3.6	2.28	2.28	8.8	5.55	8.2	5.19
PGB	195 & 196	187	PG 70-22		235.7	236.8	5.0	2.13	2.12	12.6	5.37	13.2	5.56

Table 9 – Summary Table for T313, Creep Stiffness (MPa)

A review of the data shown in Table 9 indicates that the form of the precision estimates should be based on the coefficient of variation (CV%). The average repeatability coefficient of variation for the eight pairs of samples analyzed is 2.5 percent. The corresponding average reproducibility coefficient of variation is 6.3 percent. In each case, the average coefficient of variation is determined by calculating the "simple arithmetic average" as described in Section 8.4.2 of ASTM C802-96 [15].

3.2.6 Determining the Fracture Properties of Asphalt Binder in Direct Tension (DT), T314

3.2.6.1 STRESS

Results from analyzing the data for Direct Tension Stress can be found in Appendix M. One pair of modified binders, sample numbers 187 and 188, is used in the analysis.

					Average	e Results]	Repeatabil	ity	Reprodu	cibility	Reprodu	cibility
Sample	Sample	No. of	PG	AC	odd	even		odd	even	od	d	eve	en
Туре	Numbers	Labs	Grade	Grade	samples	samples		samples	samples	samj	oles	samj	oles
							1s	CV%	CV%	1s	CV%	1s	CV%
PGB	181 & 182	37	PG 64-16	AC 10	2.785	2.816	0.265	9.5	9.4	0.532	19.1	0.572	20.3
PGB	183 & 184	44	PG 70-22		3.953	3.970	0.287	7.3	7.2	0.797	20.2	0.781	19.7
PGB	185 & 186	51	PG 64-22	AC 20	3.707	3.783	0.269	7.3	7.1	0.592	16.0	0.630	16.7
PGB	187 & 188	53	PG 76-22		3.353	3.402	0.205	6.1	6.0	0.601	17.9	0.577	16.9
PGB	189 & 190	59	PG 64-22	AC 30	3.775	3.860	0.306	8.1	7.9	0.878	23.3	0.939	24.3
PGB	191 & 192	53	PG 52-34	AC 10	4.172	4.108	0.261	6.3	6.3	0.648	15.5	0.681	16.6
PGB	193 & 194	60	PG 64-22	AC 20	4.034	3.967	0.255	6.3	6.4	0.735	18.2	0.787	19.9
PGB	195 & 196	54	PG 70-22		4.218	4.191	0.343	8.1	8.2	0.645	15.3	0.714	17.0

Table 10 – Summary Table for T314, Stress (MPa)

Following a review of the data in Table 10, coefficient of variation (CV%) was chosen as the estimate of precision. The average repeatability coefficient of variation for the eight pairs of samples analyzed was determined to be 7.4 percent. The corresponding average reproducibility coefficient of variation was determined to be 18.6 percent. In each case, the average coefficient of variation was determined by calculating the "simple arithmetic average" as described in Section 8.4.2 of ASTM C802-96 [15].

3.2.6.2 STRAIN

Results from analyzing the data for Direct Tension Strain can be found in Appendix N. One pair of modified binders, sample numbers 187 and 188, is used in the analysis.

					Average	Results		Repeatabil	ity	Reprodu	cibility	Reprodu	cibility
Sample	Sample	No. of	PG	AC	odd	even		odd	even	od	d	eve	en
Туре	Numbers	Labs	Grade	Grade	samples	samples		samples	samples	samj	oles	samj	oles
					_	-	1s	CV%	CV%	1s	CV%	1s	CV%
PGB	181 & 182	34	PG 64-16	AC 10	0.91	0.94	0.10	10.9	10.5	0.26	28.9	0.25	26.4
PGB	183 & 184	44	PG 70-22	-	1.83	1.85	0.24	13.3	13.2	0.65	35.5	0.72	38.7
PGB	185 & 186	53	PG 64-22	AC 20	1.38	1.45	0.17	12.2	11.6	0.34	25.0	0.37	25.2
PGB	187 & 188	54	PG 76-22	-	2.96	3.00	0.29	9.7	9.6	1.29	43.6	1.23	40.9
PGB	189 & 190	56	PG 64-22	AC 30	1.33	1.39	0.15	11.6	11.2	0.42	31.7	0.46	33.2
PGB	191 & 192	49	PG 52-34	AC 10	1.39	1.35	0.11	8.1	8.3	0.27	19.7	0.28	20.9
PGB	193 & 194	61	PG 64-22	AC 20	2.69	2.66	0.36	13.2	13.4	1.02	37.9	1.00	37.5
PGB	195 & 196	56	PG 70-22	1	1.36	1.35	0.17	12.8	12.9	0.39	28.8	0.41	29.9

Table 11 – Summary Table for T314, Percent Strain

A review of the data shown in Table 11 indicated that the form of the precision estimates should be based on the coefficient of variation (CV%). The average repeatability coefficient of variation for the eight pairs of samples analyzed was determined to be 11.4 percent. The corresponding average reproducibility coefficient of variation was determined to be 31.5 percent. In each case, the average coefficient of variation was determined by calculating the "simple arithmetic average" as described in Section 8.4.2 of ASTM C802-96 [15].

3.2.7 Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR), T315

3.2.7.1 Original Binder: G*/ sinð

Results from analyzing the data for DSR testing on original binder can be found in Appendix O. One pair of modified binders, sample numbers 187 and 188, was used in the analysis.

					Average	e Results		Repeatabili	ty	Reprodu	cibility	Reprodu	cibility
Sample	Sample	No. of	PG	AC	odd	even		odd	even	od	d	eve	en
Туре	Numbers	Labs	Grade	Grade	samples	samples		samples	samples	samp	oles	samp	oles
							1s	CV%	CV%	1s	CV%	1s	CV%
PGB	181 & 182	185	PG 64-16	AC 10	1.109	1.067	0.028	2.55	2.65	0.061	5.54	0.061	5.69
PGB	183 & 184	192	PG 70-22		1.345	1.348	0.032	2.40	2.39	0.083	6.14	0.078	5.80
PGB	185 & 186	189	PG 64-22	AC 20	1.287	1.280	0.026	2.00	2.01	0.070	5.46	0.073	5.71
PGB	187 & 188	189	PG 76-22		1.397	1.409	0.034	2.44	2.42	0.073	5.21	0.073	5.19
PGB	189 & 190	205	PG 64-22	AC 30	1.576	1.572	0.034	2.17	2.17	0.092	5.82	0.086	5.46
PGB	191 & 192	208	PG 52-34	AC 10	2.338	2.342	0.059	2.50	2.50	0.182	7.79	0.176	7.53
PGB	193 & 194	208	PG 64-22	AC 20	1.376	1.370	0.026	1.89	1.90	0.080	5.80	0.078	5.66
PGB	195 & 196	208	PG 70-22		1.444	1.448	0.030	2.08	2.08	0.095	6.61	0.095	6.53

Table 12 – Summary Table for T315, Original G*/ sinð

A review of the data shown in Table 12 indicated that the form of the precision estimates should be based on the coefficient of variation (CV%). The average repeatability coefficient of variation for the eight pairs of samples analyzed was determined to be 2.3 percent. The corresponding average reproducibility coefficient of variation was determined to be 6.0 percent. In each case, the average coefficient of variation was determined by calculating the "simple arithmetic average" as described in Section 8.4.2 of ASTM C802-96 [15].

3.2.7.2 RTFO Residue: G*/ sinð

Results from analyzing the data for DSR testing on RTFO residue can be found in Appendix P. One pair of modified binders, sample numbers 187 and 188, was used in the analysis.

					Average	e Results		Repeatabili	ty	Reprodu	cibility	Reprodu	cibility
Sample	Sample	No. of	PG	AC	odd	even		odd	even	od	d	eve	en
Туре	Numbers	Labs	Grade	Grade	samples	samples		samples	samples	samp	oles	samp	oles
							1s	CV%	CV%	1s	CV%	1s	CV%
PGB	181 & 182	186	PG 64-16	AC 10	2.374	2.274	0.079	3.34	3.49	0.182	7.67	0.174	7.63
PGB	183 & 184	184	PG 70-22		2.626	2.645	0.102	3.88	3.85	0.196	7.45	0.201	7.60
PGB	185 & 186	188	PG 64-22	AC 20	3.062	3.063	0.087	2.83	2.83	0.227	7.40	0.220	7.18
PGB	187 & 188	195	PG 76-22		2.972	2.975	0.110	3.70	3.69	0.264	8.88	0.236	7.94
PGB	189 & 190	198	PG 64-22	AC 30	3.641	3.646	0.107	2.93	2.93	0.268	7.36	0.254	6.96
PGB	191 & 192	199	PG 52-34	AC 10	7.683	7.733	0.230	2.99	2.97	0.780	10.15	0.732	9.46
PGB	193 & 194	204	PG 64-22	AC 20	3.185	3.191	0.097	3.04	3.03	0.241	7.57	0.243	7.62
PGB	195 & 196	205	PG 70-22		2.539	2.557	0.069	2.72	2.70	0.176	6.94	0.195	7.62

Table 13 – Summary Table for T315, RTFO G*/ $sin\delta$

A review of the data shown in Table 13 indicated that the form of the precision estimates should be based on the coefficient of variation (CV%). The average repeatability coefficient of variation for the eight pairs of samples analyzed was determined to be 3.2 percent. The corresponding average reproducibility coefficient of variation was determined to be 7.8 percent. In each case, the average coefficient of variation was determined by calculating the "simple arithmetic average" as described in Section 8.4.2 of ASTM C802-96 [15].

3.2.7.3 PAV Residue: G* sinð

Results from analyzing the data for the DSR testing on PAV residue can be found in Appendix Q. One pair of modified binders, sample numbers 187 and 188, was used in the analysis.

					Average	e Results	I	Repeatabili	ty	Reprodu	icibility	Reprodu	cibility
Sample	Sample	No. of	PG	AC	odd	even		odd	even	od	ld	eve	en
Туре	Numbers	Labs	Grade	Grade	samples	samples		samples	samples	sam	ples	sam	oles
							1s	CV%	CV%	1s	CV%	1s	CV%
PGB	181 & 182	181	PG 64-16	AC 10	4557	4489	249	5.5	5.6	695	15.3	656	14.6
PGB	183 & 184	178	PG 70-22		2310	2334	117	5.1	5.0	293	12.7	313	13.4
PGB	185 & 186	178	PG 64-22	AC 20	3830	3818	223	5.8	5.8	526	13.7	486	12.7
PGB	187 & 188	185	PG 76-22		1100	1102	61	5.6	5.6	167	15.1	157	14.3
PGB	189 & 190	182	PG 64-22	AC 30	4335	4340	143	3.3	3.3	597	13.8	603	13.9
PGB	191 & 192	185	PG 52-34	AC 10	3640	3673	171	4.7	4.7	660	18.1	660	18.0
PGB	193 & 194	188	PG 64-22	AC 20	2922	2937	137	4.7	4.7	364	12.5	359	12.2
PGB	195 & 196	199	PG 70-22		3163	3171	137	4.3	4.3	432	13.7	424	13.4

Table 14 – Summary Table for T315, PAV G* sinð

A review of the data shown in Table 14 indicated that the form of the precision estimates should be based on the coefficient of variation (CV%). The average repeatability coefficient of variation for the eight pairs of samples analyzed was determined to be 4.9 percent. The corresponding average reproducibility coefficient of variation was determined to be 14.2 percent. In each case, the average coefficient of variation was determined by calculating the "simple arithmetic average" as described in Section 8.4.2 of ASTM C802-96 [15].

3.2.8 Viscosity Determination of Asphalt Binder Using a Rotational Viscometer, T316

Results from analyzing the data for Viscosity Determination can be found in Appendix R. One pair of modified binders, sample numbers 187 and 188, was used in the analysis.

					Average	e Results]	Repeatabil	ity	Reprodu	cibility	Reprodu	cibility
Sample	Sample	No. of	PG	AC	odd	even		odd	even	od	d	eve	en
Туре	Numbers	Labs	Grade	Grade	samples	samples		samples	samples	samp	oles	samp	oles
							1s	CV%	CV%	1s	CV%	1s	CV%
PGB	181 & 182	142	PG 64-16	AC 10	0.277	0.272	0.004	1.27	1.30	0.015	5.40	0.015	5.52
PGB	183 & 184	176	PG 70-22		0.715	0.719	0.008	1.12	1.11	0.028	3.96	0.029	4.08
PGB	185 & 186	172	PG 64-22	AC 20	0.414	0.414	0.005	1.27	1.27	0.017	4.02	0.015	3.71
PGB	187 & 188	180	PG 76-22		1.621	1.638	0.020	1.25	1.23	0.070	4.34	0.069	4.19
PGB	189 & 190	179	PG 64-22	AC 30	0.439	0.439	0.005	1.18	1.18	0.016	3.59	0.016	3.55
PGB	191 & 192	192	PG 52-34	AC 10	0.290	0.291	0.005	1.69	1.68	0.012	4.06	0.013	4.31
PGB	193 & 194	202	PG 64-22	AC 20	0.445	0.445	0.005	1.22	1.22	0.020	4.54	0.020	4.48
PGB	195 & 196	195	PG 70-22		0.685	0.688	0.006	0.84	0.84	0.031	4.47	0.031	4.44

Table 15 – Summary Table for T316, Viscosity (Pa·s)

A review of the data shown in Table 15 indicated that the form of the precision estimates should be based on the coefficient of variation (CV%). The average repeatability coefficient of variation for the eight pairs of samples analyzed was determined to be 1.2 percent. The corresponding average reproducibility coefficient of variation was determined to be 4.3 percent. In each case, the average coefficient of variation was determined by calculating the "simple arithmetic average" as described in Section 8.4.2 of ASTM C802-96 [15].

CHAPTER 4: CONCLUSIONS AND RECOMMENDATIONS

4.1 GENERAL

This study was conducted to prepare precision estimates for AASHTO standards found in AASHTO Standard Specification M320, "Performance-Graded Asphalt Binder", and for AASHTO Standard Test Method T308, "Determining the Asphalt Binder Content of Hot-Mix Asphalt (HMA) by the Ignition Method". The study conclusions and recommendations are as follows:

4.2 CONCLUSIONS AND RECOMMENDATIONS RELATED TO SPECIFIC STANDARDS

4.2.1 AASHTO T308-04, Determining the Asphalt Binder Content of Hot-Mix Asphalt (HMA) by the Ignition Method

Commentary:

The current precision estimates for T308 are based on the results of four aggregate types, four replicates, and twelve participating laboratories using Method A only. This is a small number of laboratories compared to the number of labs in the AMRL proficiency sample program. The small number of laboratories may not capture all of the variability inherent in the test method. The precision and bias statement in Section 4.4 is based on testing by over 350 laboratories on three different paired aggregate samples and applies to both Method A and Method B.

Conclusion:

The precision statement derived from analyzing the AMRL PSP data comes from much larger data sets than the current estimates. These estimates reflect variability that is reflective of what is occurring in the laboratory setting.

Recommendation:

It is recommended that the precision and bias statement in Section 4.4 be adopted for T308.

4.2.2 AASHTO T48-04, Flash and Fire Points by Cleveland Open Cup

Commentary:

The precision estimates for flash point currently published in T48-04 are based on testing over ten years ago by eleven laboratories. Though seven oils were used in the study, only one asphalt (AC 10) was used. The precision and bias statement in Section 4.5 is based on testing by over 98 laboratories on eight different paired binder samples and four binder grades. Over time, the results of AMRL proficiency sample testing indicate that the degree of precision given in the current precision statement cannot be obtained. Two possible sources of variation are the

difficulty in achieving the required rate of temperature rise and improper application of barometric correction.

Conclusion:

The precision estimates for flash point should be revised.

Recommendation:

It is recommended that the precision and bias statement for "flash point" in Section 4.5 be adopted for T48.

4.2.3 AASHTO T228-04, Specific Gravity of Semi-Solid Bituminous Materials

Commentary:

The study showed that single-operator precision is slightly better than currently indicated in the test method and that the multilaboratory precision is significantly better than currently indicated. Information regarding testing of soft pitch tar or testing of asphalt at 15.6 °C was not available from AMRL data, therefore the precision estimates for those tests were not considered for revision by this study.

Conclusions:

The precision estimates currently published in T228-04 for specific gravity of asphalt determined at 25 °C should be revised.

Recommendations:

- (1) It is recommended that the precision and bias statement in Section 4.6 for determining the specific gravity of asphalt at 25 °C replace the precision and bias statement stated in section 14 of ASTM D70.
- (2) It is recommended that the precision estimates for "pooled values" for testing soft tar pitch and asphalt at 25 °C given in the current precision and bias statement of T228-04 be disregarded.

4.2.4 AASHTO T240-03, Effect of Heat and Air on a Moving Film of Asphalt (Rolling Thin-Film Oven Test)

Commentary:

The precision statement currently published in T240-03 does not include a precision estimate for the loss of mass determination. Test method T240-03 does not contain a statement regarding bias.

A review of the data showed that the standard deviation changed for different values of mass loss. Coefficient of variation also was not appropriate since standard deviation was not proportional to mass loss. However, the review indicated that the standard deviation can be expressed as a function of the mass change (x) by using an equation.

Conclusions:

The high coefficient of determination for the derived equation indicates it is the most informative form of precision for this method. Since this approach has not been commonly used in precision statements, a table with stratified estimates was included to assist the user.

Recommendations:

- (1) It is recommended that the precision and bias statement in Section 4.7 be adopted for T240.
- (2) The materials included in this study did not gain mass during testing. It is recommended that a study be conducted to develop precision estimates for materials that gain mass.

4.2.5 AASHTO T313-04, Determining the Flexural Creep Stiffness of Asphalt Binder Using the Bending Beam Rheometer (BBR)

Commentary:

The AMRL proficiency data analyzed in this study is more up to date than the AMRL data used for the current estimate of precision and reflect recent changes to the test method. The results of the study show that testing precision is better than indicated by the precision estimates currently provided in the test method.

Conclusion:

The precision and bias statement currently published in T313-04 should be revised.

Recommendation:

It is recommended that the revised precision and bias statement in Section 4.8 be adopted for T313.

4.2.6 AASHTO T314-04, Determining the Fracture Properties of Asphalt Binder in Direct Tension (DT)

Commentary:

A precision and bias statement is not provided in T314-04.

Conclusion:

A precision and bias statement is needed.

Recommendation:

It is recommended that the precision and bias statement in Section 4.9 be adopted for T314.

4.2.7 AASHTO T315-04, Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)

Commentary:

The AMRL proficiency data analyzed in this study is more up to date than the AMRL data used for the current estimate of precision and reflect recent changes to the test method. The results of the study show that testing precision is better than indicated by the precision estimates currently provided in the test method.

During the analysis of DSR data on "original" binder, the study observed that the "phase angle" and the testing variation for the determination of the "phase angle", δ , appeared to be different for the modified binder analyzed in this study when compared to the unmodified binders (See Appendix C). The difference between modified binders and unmodified binders was not apparent for determinations of G*, G*/sin δ , or G*·sin δ . It should be noted that only one modified binder was included in the study and that precision estimates for the determination of "phase angle" are not included in the proposed precision and bias statement contained within this report.

Conclusion:

The precision and bias statement currently published in T315-04 should be revised.

Recommendations:

- (1) It is recommended that the precision and bias statement in Section 4.10 be adopted for T315.
- (2) It is recommended that an additional study be conducted using modified binders if precision estimates are desired for the phase angle.

4.2.8 AASHTO T316-04, Viscosity Determination of Asphalt Binder Using a Rotational Viscometer

Commentary:

The AMRL proficiency data analyzed in this study is more up to date than the AMRL data used for the current estimate of precision and reflect recent changes to the test method. The results of the study show that testing precision is better than indicated by the precision estimates currently provided in the test method.

Conclusion:

The precision and bias statement currently published in T316-04 should be revised.

Recommendation:

It is recommended that the precision and bias statement in Section 4.11 be adopted for T316.

4.3 GENERAL CONCLUSIONS AND RECOMMENDATIONS

The analysis technique described in this study can be used effectively to analyze paired proficiency sample test data sets to obtain robust single operator and multilaboratory precision estimates for a variety of test methods.

A comparison of the 95% difference by count and the calculated d2s limits in Appendix D shows there is no real difference between the two numbers. Even when the values are not the same, there does not appear to be a large enough deviation that would require reporting the various d2s or d2s% limits reported in this report in a manner other than described in ASTM E 177 [16].

4.4 PRECISION STATEMENT FOR AASHTO T308

Determining the Asphalt Binder Content of Hot-Mix Asphalt (HMA) by the Ignition Method

X. Precision and Bias

X.1 Precision

Criteria for judging the acceptability of ignition burn results for asphalt content obtained by Method A or Method B are given in Table X.

- X.1.1 Single-Operator Precision (Repeatability) The figures in Column 2 of Table X are the standard deviations that have been found to be appropriate for the conditions of test described in Column 1. Two results obtained in the same laboratory, by the same operator using the same equipment, in the shortest practical period of time, should not be considered suspect unless the difference in the two results exceeds the values given in Table X, Column 3.
- X.1.2 Multilaboratory Precision (Reproducibility) The figures in Column 2 of Table X are the standard deviations that have been found to be appropriate for the conditions of test described in Column 1. Two results submitted by two different operators testing the same material in different laboratories shall not be considered suspect unless the difference in the two results exceeds the values given in Table X, Column 3.

	Standard Deviation	Acceptable Range of Two Test Results
Condition	$(1s)^{a}$	$(d2s)^a$
Condition	(18)	(028)
Single Operator Precision:		
Asphalt Content (%) Multilaboratory Precision:	0.069	0.196
Asphalt Content (%)	0.117	0.330

Table X – Precision Estimates

^a These values represent the 1s and d2s limits described in ASTM Practice C670.

Note – The precision estimates given in Table X are based on the analysis of test results from three pairs of AMRL proficiency samples. The data analyzed consisted of results from 353 to 461 laboratories for each of the three pairs of samples. The analysis included two binder grades: PG 52-34 and PG 64-22. Average results for asphalt content ranged from 4.049% to 5.098%. The details of this analysis are in NCHRP Final Report, NCHRP Project No. 9-26, Phase 3.

X.2 Bias – Any biases inherent to the ignition oven process used for test methods A and B, when testing for asphalt content and aggregate gradation, are accounted for by the determination and application of appropriate correction factors.

4.5 PRECISION STATEMENT FOR AASHTO T48

Flash and Fire Points by Cleveland Cup

X. Precision and Bias

- **X.1 Precision** Criteria for judging the acceptability of test results for flash point of asphalt binder obtained by this method are given in Table X. Criteria for judging the acceptability of fire point test results can be found in ASTM D92.
- X.1.1 Single-Operator Precision (Repeatability) The figures in Column 2 of Table X are the standard deviations that have been found to be appropriate for the conditions of test described in Column 1. Two results obtained in the same laboratory, by the same operator using the same equipment, in the shortest practical period of time, should not be considered suspect unless the difference in the two results exceeds the values given in Table X, Column 3.
- X.1.2 Multilaboratory Precision (Reproducibility) The figures in Column 2 of Table X are the standard deviations that have been found to be appropriate for the conditions of test described in Column 1. Two results submitted by two different operators testing the same material in different laboratories shall not be considered suspect unless the difference in the two results exceeds the values given in Table X, Column 3.

	Standard Deviation	Acceptable Range of Two Results
Condition	$(1s)^{a}$	$(d2s)^a$
Single Operator Precision:	× 7	, <i>, , , , , , , , , , , , , , , , , , </i>
Flash Point (°C)	3	8
Multilaboratory Precision:		
Flash Point (°C)	10	28

Table X – Precision Estimates

^a These values represent the 1s and d2s limits described in ASTM Practice C670.

Note 1 – The precision estimates for Flash Point given in Table X are based on the analysis of test results from eight pairs of AMRL proficiency samples. The data analyzed consisted of results from 98 to 148 laboratories for each of the eight pairs of samples. The analysis included four binder grades: PG 52-34, PG 64-16, PG 64-22, and PG 70-22. Average flash points ranged from 268.5 °C to 353.5 °C. The details of the analysis are in NCHRP Final Report, NCHRP Project No. 9-26, Phase 3.

X.2 Bias – The procedure of this test method has no bias because flash point and fire point can only be defined in terms of this test method.

4.6 PRECISION STATEMENT FOR AASHTO T228

Specific Gravity of Semi-Solid Bituminous Materials

X. Precision and Bias

- **X.1 Precision** Criteria for judging the acceptability of the relative density results obtained by this method are given in Table X.
- X.1.1 Single-Operator Precision (Repeatability) The figures in Column 2 of Table X are the standard deviations that have been found to be appropriate for the conditions of test described in Column 1. Two results obtained in the same laboratory, by the same operator using the same equipment, in the shortest practical period of time, should not be considered suspect unless the difference in the two results exceeds the values given in Table X, Column 3.
- **X.1.2 Multilaboratory Precision** (Reproducibility) The figures in Column 4 of Table X are the standard deviations that have been found to be appropriate for the conditions of test described in Column 1. Two results submitted by two different operators testing the same material in different laboratories shall not be considered suspect unless the difference in the two results exceeds the values given in Table X, Column 5.

	Single	-Operator	Multil	aboratory
	Standard	Acceptable Range of Two	Standard	Acceptable Range of Two
Condition	Deviation (1s) ^a	$\frac{\text{Results}}{(d2s)^a}$	Deviation $(1s)^{a}$	$\frac{\text{Results}}{(\text{d2s})^{\text{a}}}$
Asphalt:				
Specific Gravity (15.6 °C)	0.0011	0.0032	0.0018	0.0051
Specific Gravity (25 °C)	0.0008^{b}	0.0021 ^b	0.0013 ^b	0.0035 ^b
Soft Tar Pitch:				
Specific Gravity (15.6 °C)	0.0013	0.0038	0.0029	0.0083
Specific Gravity (25 °C)	0.00083	0.0023	0.0017	0.0048

Table X – Precision Estimates

^a These values represent the 1s and d2s limits described in ASTM Practice C670.

^b The precision estimates denoted by the superscript "b" are based on the analysis of test results from eight pairs of AMRL proficiency samples. The data analyzed consisted of results from 104 to 121 laboratories for each of the eight pairs of samples. The analysis included four binder grades: PG 52-34, PG 64-16, PG 64-22, and PG 70-22. Average specific gravities in the analysis ranged from 1.0058 to 1.0428. The details of this analysis arc in NCHRP Final Report, NCHRP Project No. 9-26, Phase 3.

Note – Values in Table X not marked with a superscript "b" are precision estimates retained from ASTM D70-03 Section 14, Table 1. These values were not part of the scope of the AMRL research activities described with the superscript "b".

X.2 Bias – No information can be presented on the bias of the procedure because no material having an accepted reference value is available.

4.7 PRECISION STATEMENT FOR AASHTO T240

Effect of Heat and Air on a Moving Film of Asphalt (Rolling Thin-Film Oven Test)

- X. Precision and Bias
- X.1 ... copy Section X.1 as it appears in T240-03 and renumber the section as necessary.
- **X.2 Precision for Loss of Mass** Criteria for judging the acceptability of change in mass results obtained by this method are given in Tables 1 and 2. Table 1 should be consulted as the final qualifier for precision purposes. Table 2 has been added for the convenience of the user.
- X.2.1 Single-Operator Precision (Repeatability) The equation in Column 2 of Table 1 indicates that the standard deviation of the test results (1s) can be expressed as a function of the mass change (X) for the conditions of test described in Column 1. Two results obtained in the same laboratory, by the same operator using the same equipment, in the shortest practical period of time, should not be considered suspect unless the difference in the two results exceeds the value determined by multiplying the 1s estimate determined in Column 2 for the average value of the two results by a factor of 2.83. This is shown in Table 1, Column 3.
- **X.2.2 Multilaboratory Precision** (Reproducibility) The equation in Column 2 of Table 1 indicates that the standard deviation of the test results (1s) can be expressed as a function of the mass change (X) for the conditions of test described in Column 1. Two results submitted by two different operators testing the same material in different laboratories shall not be considered suspect unless the difference in the two results exceeds the value determined by multiplying the 1s estimate determined in Column 2 for the average value of the two results by a factor of 2.83. This is shown in Table 1, Column 3.

		Acceptable
	Standard	Range of Two
	Deviation ^{a,b}	Test Results ^{a,b,c}
Condition	(1s)	(d2s)
Single Operator Precision:		
Mass Loss (%)	1s = 0.0061 + 0.0363(X)	$d2s = (0.0061 + 0.0363(X_{avg})) \times (2.83)$
Multilaboratory Precision:		-
Mass Loss (%)	1s = 0.00153 + 0.1365(X)	$d2s = (0.00153 + 0.1365(X_{avg})) \times (2.83)$

Table 1 – Precision Estimates

^a These values represent the 1s and d2s limits described in ASTM Practice C670.

 b X and X_{avg} should be entered into equations as positive numbers.

 c The value X_{avg} represents the average value of two test results.

Note – The precision estimates given in Table 1 are based on the analysis of test results from eight pairs of AMRL proficiency samples. The data analyzed consisted of results from 166 to 191 laboratories for each of the eight pairs of samples. The analysis included five binder grades: PG 52-34, PG 64-16, PG 64-22, PG 70-22 and PG 76-22 (SBS modified). The samples used in the analysis had an average loss of mass ranging from -0.05% to -0.51%. The equations for precision estimates are reliable only in situations when the change in mass is negative. The details of this analysis are in NCHRP Final Report, NCHRP Project No. 9-26, Phase 3.

	Standard	Acceptable Range of Two
	Deviation ^a	Test Results ^a
Condition	(1s)	(d2s)
Single Operator Precision:		
Mass Loss (%)		
0.0 to 0.1%	0.0079	0.0224
0.1 to 0.2%	0.0115	0.0327
0.2 to 0.3%	0.0152	0.0429
0.3 to 0.4%	0.0188	0.0532
0.4 to 0.5%	0.0224	0.0635
Multilaboratory Precision:		
Mass Loss (%)		
0.0 to 0.1%	0.0084	0.0236
0.1 to 0.2%	0.0220	0.0623
0.2 to 0.3%	0.0357	0.1009
0.3 to 0.4%	0.0493	0.1395
0.4 to 0.5%	0.0630	0.1781

^a The values represented in this table are the 1s and d2s limits described as stratified values. Table 1 of this standard should be consulted as the final qualifier for precision purposes.

4.8 PRECISION STATEMENT FOR AASHTO T313

Determining the Flexural Creep Stiffness of Asphalt Binder Using the Bending Beam Rheometer (BBR)

X. Precision and Bias

- **X.1 Precision** Criteria for judging the acceptability of creep stiffness and slope results obtained by this method are given in Table X.
- X.1.1 Single-Operator Precision (Repeatability) The figures in Column 2 of Table X are the coefficients of variation that have been found to be appropriate for the conditions of test described in Column 1. Two results obtained in the same laboratory, by the same operator using the same equipment, in the shortest practical period of time, should not be considered suspect unless the difference in the two results, expressed as a percent of their mean, exceeds the values given in Table X, Column 3.
- X.1.2 Multilaboratory Precision (Reproducibility) The figures in Column 2 of Table X are the coefficients of variation that have been found to be appropriate for the conditions of test described in Column 1. Two results submitted by two different operators testing the same material in different laboratories shall not be considered suspect unless the difference in the two results, expressed as a percent of their mean, exceeds the values given in Table X, Column 3.

Condition	Coefficient of Variation (1s%) ^a	Acceptable Range of Two Test Results (d2s%) ^a
Single Operator Precision:		
Creep Stiffness (MPa) Slope (m-value)	2.5 1.0	7.2 2.9
Multilaboratory Precision:		
Creep Stiffness (MPa)	6.3	17.8
Slope (m-value)	2.4	6.8

Table X – Precision Estimates

^a These values represent the 1s% and d2s% limits described in ASTM Practice C670.

Note – The precision estimates given in Table X are based on the analysis of test results from eight pairs of AMRL proficiency samples. The data analyzed consisted of results from 174 to 196 laboratories for each of the eight pairs of samples. The analysis included five binder grades: PG 52-34, PG 64-16, PG 64-22, PG 70-22 and PG 76-22 (SBS modified). Average creep stiffness results ranged from 125.4 MPa to 236.8 MPa. Average slope results ranged from an m-value of 0.308 to 0.374. The details of this analysis are in NCHRP Final Report, NCHRP Project No. 9-26, Phase 3.

Note – As an example, two tests conducted on the same material yield creep stiffness results of 190.3 MPa and 200.7 MPa, respectively. The average of these two measurements is 195.5 MPa. The acceptable range of results is then 7.2 percent of 195.5 MPa or 14.1 MPa. As the difference between 190.3 MPa and 200.7 MPa is < 14.1 MPa the results are within the acceptable range.

X.2 Bias – No information can be presented on the bias of the procedure because no material having an accepted reference value is available.

4.9 PRECISION STATEMENT FOR AASHTO T314

Determining the Fracture Properties of Asphalt Binder in Direct Tension (DT)

X. Precision and Bias

- **X.1 Precision** Criteria for judging the acceptability of failure stress and strain results obtained by this method are given in Table X.
- X.1.1 Single-Operator Precision (Repeatability) The figures in Column 2 of Table X are the coefficients of variation that have been found to be appropriate for the conditions of test described in Column 1. Two results obtained in the same laboratory, by the same operator using the same equipment, in the shortest practical period of time, should not be considered suspect unless the difference in the two results, expressed as a percent of their mean, exceeds the values given in Table X, Column 3.
- X.1.2 Multilaboratory Precision (Reproducibility) The figures in Column 2 of Table X are the coefficients of variation that have been found to be appropriate for the conditions of test described in Column 1. Two results submitted by two different operators testing the same material in different laboratories shall not be considered suspect unless the difference in the two results, expressed as a percent of their mean, exceeds the values given in Table X, Column 3.

	Coefficient of Variation	Acceptable Range of Two Test Results
Condition	$(1s\%)^{a}$	$(d2s\%)^a$
Single Operator Precision:		
Stress (MPa)	7.4	20.8
Strain (%)	11.4	32.2
Multilaboratory Precision:		
Stress (MPa)	18.6	52.5
Strain (%)	31.5	89.1

Table X – Precision Estimates

^a These values represent the 1s%, and d2s% limits described in ASTM Practice C670.

Note – The precision estimates given in Table X are based on the analysis of test results from eight pairs of AMRL proficiency samples. The data analyzed consisted of results from 34 to 61 laboratories for each of the eight pairs of samples. The analysis included five binder grades: PG 52-34, PG 64-16, PG 64-22, PG 70-22 and PG 76-22 (SBS modified). Average stress results ranged from 2.79 MPa to 4.22 MPa. Average strain results ranged from 0.91% to 3.00%. The details of this analysis are in NCHRP Final Report, NCHRP Project No. 9-26, Phase 3.

Note – As an example, two tests conducted on the same material yield stress results of 2.95 MPa and 3.15 MPa, respectively. The average of these two measurements is 3.05 MPa. The acceptable range of results is then 20.8 percent of 3.05 or 0.63 MPa. As the difference between 2.95 MPa and 3.15 MPa is < 0.63 MPa, the results are within the acceptable range.

X.2 Bias – No information can be presented on the bias of the procedure because no material having an accepted reference value is available.

4.10 PRECISION STATEMENT FOR AASHTO T315

Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)

X. Precision and Bias

- **X.1 Precision** Criteria for judging the acceptability of dynamic shear results obtained by this method are given in Table X.
- X.1.1 Single-Operator Precision (Repeatability) The figures in Column 2 of Table X are the coefficients of variation that have been found to be appropriate for the conditions of test described in Column 1. Two results obtained in the same laboratory, by the same operator using the same equipment, in the shortest practical period of time, should not be considered suspect unless the difference in the two results, expressed as a percent of their mean, exceeds the values given in Table X, Column 3.
- X.1.2 Multilaboratory Precision (Reproducibility) The figures in Column 2 of Table X are the coefficients of variation that have been found to be appropriate for the conditions of test described in Column 1. Two results submitted by two different operators testing the same material in different laboratories shall not be considered suspect unless the difference in the two results, expressed as a percent of their mean, exceeds the values given in Table X, Column 3.

	Coefficient of Variation	Acceptable Range of Two Test Results
Condition	$(1s\%)^{a}$	$(d2s\%)^a$
Single Operator Precision:		
Original Binder: G*/sino (kPa)	2.3	6.4
RTFO Residue: G*/sino (kPa)	3.2	9.0
PAV Residue: G*·sino (kPa)	4.9	13.8
Multilaboratory Precision:		
Original Binder: G*/sino (kPa)	6.0	17.0
RTFO Residue: G*/sino (kPa)	7.8	22.2
PAV Residue: G*·sinδ (kPa)	14.2	40.2

Table X – Precision Estimates

^a These values represent the 1s% and d2s% limits described in ASTM Practice C670.

Note – The precision estimates given in Table X are based on the analysis of test results from eight pairs of AMRL proficiency samples. The data analyzed consisted of results from 185 to 208 laboratories for each of the eight pairs of samples. The analysis included five binder grades: PG 52-34, PG 64-16, PG 64-22, PG 70-22 and PG 76-22 (SBS modified). Average original binder results for G*/sinð ranged from 1.067 kPa to 2.342 kPa. Average RTFO residue results for G*/sinð ranged from 2.274 kPa to 7.733 kPa. Average PAV residue results for G*·sinð averaged from 1100 kPa to 4557 kPa. The details of this analysis are in NCHRP Final Report, NCHRP Project No. 9-26, Phase 3.

Note – As an example, two tests conducted on the same PAV residue yield results of 1200 kPa and 1300 kPa, respectively. The average of these two measurements is 1250 kPa. The acceptable range of results is then 13.8 percent of 1250 kPa or 173 kPa. As the difference between 1200 and 1300 is < 173 kPa, the results are within the acceptable range.

X.2 Bias – No information can be presented on the bias of the procedure because no material having an accepted reference value is available.

4.11 PRECISION STATEMENT FOR AASHTO T316

Viscosity Determination of Asphalt Binder Using a Rotational Viscometer

X. Precision and Bias

- **X.1 Precision** Criteria for judging the acceptability of viscosity results obtained by this method are given in Table X.
- X.1.1 Single-Operator Precision (Repeatability) The figures in Column 2 of Table X are the coefficients of variation that have been found to be appropriate for the conditions of test described in Column 1. Two results obtained in the same laboratory, by the same operator using the same equipment, in the shortest practical period of time, should not be considered suspect unless the difference in the two results, expressed as a percent of their mean, exceeds the values given in Table X, Column 3.
- X.1.2 Multilaboratory Precision (Reproducibility) The figures in Column 2 of Table X are the coefficients of variation that have been found to be appropriate for the conditions of test described in Column 1. Two results submitted by two different operators testing the same material in different laboratories shall not be considered suspect unless the difference in the two results, expressed as a percent of their mean, exceeds the values given in Table X, Column 3.

	Acceptable
Coefficient of	Range of Two
Variation	Test Results
$(1s\%)^{a}$	$(d2s\%)^a$
1.2	3.5
4.3	12.1
	Coefficient of Variation (1s%) ^a 1.2

Table X – Precision Estimates

^a These values represent the 1s% and d2s% limits described in ASTM Practice C670.

Note – The precision estimates given in Table X are based on the analysis of test results from eight pairs of AMRL proficiency samples. The data analyzed consisted of results from 142 to 202 laboratories for each of the eight pairs of samples. The analysis included five binder grades: PG 52-34, PG 64-16, PG 64-22, PG 70-22 and PG 76-22 (SBS modified). Unmodified average viscosity results ranged from 0.272 Pa·s to 0.719 Pa·s. The modified binder average viscosity ranged from 1.621 Pa·s to 1.638 Pa·s. The details of this analysis are in NCHRP Final Report, NCHRP Project No. 9-26, Phase 3.

Note – As an example, two tests conducted on the same material yield viscosity results of 0.500 Pa \cdot s and 0.510 Pa \cdot s, respectively. The average of these two measurements is 0.505 Pa \cdot s. The acceptable range of results is then 3.5 percent of 0.505 Pa \cdot s or 0.018 Pa \cdot s. As the difference between 0.500 Pa \cdot s and 0.510 Pa \cdot s is < 0.018 Pa \cdot s, the results are within the acceptable range.

X.2 Bias – No information can be presented on the bias of the procedure because no material having an accepted reference value is available.

REFERENCES:

- [1] Spellerberg, P.A., Savage, D.A., and Pielert, J.H., "Precision Estimates of Selected Volumetric Properties of HMA Using Non-Absorptive Aggregate," NCHRP Web Document 54, 2003.
- [2] Spellerberg, P.A. and Savage, D.A., "An Investigation of the Cause of Variation in HMA Bulk Specific Gravity Test Results Using Non-Absorptive Aggregates," NCHRP Web Document 66, 2004.
- [3] AASHTO, Designation M320, "Performance-Graded Asphalt Binder", *Standard Specifications for Transportation Materials and Methods of Sampling and Testing*, 24th Edition, AASHTO, Washington, DC, 2004, CD-ROM.
- [4] AASHTO, Designation T308, "Determining the Asphalt Binder Content of Hot-Mix Asphalt (HMA) by the Ignition Method" *Standard Specifications for Transportation Materials and Methods of Sampling and Testing*, 24th Edition, AASHTO, Washington, DC, 2004, CD-ROM.
- [5] Pielert, J.H. and Spellerberg, P.A., "AASHTO Materials Reference Laboratory Thirty Years of Service to the Transportation Community," TR News, Number 183, Transportation Research Board, Washington, DC, March-April 1996, pages 22-28.
- [6] AMRL Web Site: <u>http://www.amrl.net</u>
- [7] Arni, H.T., "Precision of Air-Permeability, Turbidimeter, and No. 325 Sieve Fineness Data," *Fineness of Cement*, ASTM STP 473, American Society for Testing and Materials, 1970, pp 20-44.
- [8] Crandall, J.R. and Blaine, R. L., "Statistical Evaluation of Interlaboratory Cement Tests," Proceedings, American Society of Testing and Materials, ASTEA, Vol. 59, 1959.
- [9] Youden, W.J., "Statistical Techniques for Collaborative Tests," Association of Official Analytical Chemists, Inc., 1967: pp. 17 19.
- [10] ASTM, Designation E691-99, "Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method," *Annual Book of ASTM Standards*, Volume 14.02, ASTM, West Conshohocken, PA, 2001, Section 9.1.1.
- [11] Hoaglin, D.C., Iglewicz, B., Tukey, J. W., "Performance of Some Resistant Rules for Outlier Labeling," Journal of the American Statistical Association, Vol. 81, No. 396 (Dec., 1986), pp. 991-999.
- [12] Tukey, John W., Exploratory Data Analysis, Addison-Wesley Publishing Co., Reading, Mass., 1977, Chapter 2: (a) pp. 33, 43 and 44.
- [13] Carling, K., "Resistant Outlier Rules and the Non-Gaussian Case," Computational Statistics and Data Analysis, Vol. 33, 2000, pp. 249-258.
- [14] Ku, Harry H., "Statistical Concepts in Metrology," NIST Special Publication 300, Volume 1, 1969: p 316-40.
- [15] ASTM, Designation C802-96(2002), "Standard Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods for Construction Materials," *Annual Book of ASTM Standards*, Volume 4.02, ASTM, West Conshohocken, PA, 2001, Section 8.4.3.
- [16] ASTM, Designation E177-04, "Standard Practice for Use of the Terms Precision and Bias in ASTM Test Methods," *Annual Book of ASTM Standards*, Volume 4.02, ASTM, West Conshohocken, PA, 2001, Section 27.3.

BIBLIOGRAPHY:

AMRL Web Site: <u>http://www.amrl.net</u>

- Arni, H. T., "Precision of Air-Permeability, Turbidimeter, and No. 325 Sieve Fineness Data", *Fineness of Cement*, ASTM STP 473, American Society for Testing and Materials, 1970, pp 20-44.
- AASHTO, Designation M320, "Performance-Graded Asphalt Binder", *Standard Specifications for Transportation Materials and Methods of Sampling and Testing*, 24th Edition, AASHTO, Washington, DC, 2004, CD-ROM.
- AASHTO, Designation T308, "Determining the Asphalt Binder Content of Hot-Mix Asphalt (HMA) by the Ignition Method" Standard Specifications for Transportation Materials and Methods of Sampling and Testing, 24th Edition, AASHTO, Washington, DC, 2004, CD-ROM.
- ASTM, Designation C670-03, "Standard Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials," *Annual Book of ASTM Standards*, Vol. 4.02, ASTM, West Conshohocken, PA, 2003.
- ASTM, Designation C802-96, "Standard Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods for Construction Materials," *Annual Book of ASTM Standards*, Vol. 4.02, ASTM, West Conshohocken, PA, 2003.
- ASTM, Designation E177-04, "Standard Practice for Use of the Terms Precision and Bias in ASTM Test Methods," *Annual Book of ASTM Standards*, Vol. 14.02, ASTM, West Conshohocken, PA, 2001.
- ASTM, Designation E178-02, "Standard Practice for Dealing with Outlying Observations," *Annual Book of ASTM Standards,* Vol. 14.02, ASTM, West Conshohocken, PA, 2001.
- ASTM, Designation E691-99, "Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method," *Annual Book of ASTM Standards*, Vol. 14.02, ASTM, West Conshohocken, PA, 2001, Section 17.1.
- ASTM, Designation E1301-95(2003), "Standard Guide for Proficiency Testing by Interlaboratory Comparisons," Annual Book of ASTM Standards, Vol. 14.02, ASTM, West Conshohocken, PA, 2001, Section.
- Carling, K., "Resistant outlier rules and the non-Gaussian case", Computational Statistics and Data Analysis, Vol. 33, 2000, pp. 249-258.
- Coleman, H. W. and Steele, W. G., Experimentation and Uncertainty Analysis for Engineers, John Wiley & Sons, Inc, NY, 1999.
- Crandall, J. R. and Blaine, R. L., "Statistical Evaluation of Interlaboratory Cement Tests", Proceedings, American Society of Testing and Materials, ASTEA, Vol. 59, 1959.
- Dolciani, M. P., Sorgenfrey, R. H., Brown, R. G., Kane, R. B., Algebra and Trigonometry Book 2, Houghton Mifflin Company, Boston, 1984, pp. 343-347, 367, 673-674.
- Hoaglin, D. C., Iglewicz, B., Tukey, J. W., "Performance of Some Resistant Rules for Outlier Labeling", Journal of the American Statistical Association, Vol. 81, No. 396 (Dec., 1986), pp. 991-999.
- Mandel, J. and Lashof, T. W., "Interpretation and Generalization of Youden's Two-Sample Diagram", Journal of Quality Technology, Vol. 6, No.1, January 1974.
- Hall, I. J., Sheldon, D. D., "Improved Bivariate Normal Tolerance Regions with Some Applications", Journal of Quality Technology, Vol. 11, No. 1, January 1979.
- Gonick, L. and Woollcott, S., The Cartoon Guide to Statistics, HarperCollins Publishers, Inc., NY, 1993, pp. 20, 21.
- James, G. and James, R., Mathematics Dictionary, Van Nostrand Reinhold Company, NY, 1968, pp. 67, 126-127, 208-209.
- Schwartz, A., Calculus and Analytical Geometry, Holt, Rinehart and Winston, Inc, NY, 1967, pp. 205, 525
- Pielert, J.H. and Spellerberg, P.A., "AASHTO Materials Reference Laboratory Thirty Years of Service to the Transportation Community," TR News, Number 183, Transportation Research Board, Washington, DC, March-April 1996, pages 22-28.
- Spellerberg, P.A. and Savage, D.A., "An Investigation of the Cause of Variation in HMA Bulk Specific Gravity Test Results Using Non-Absorptive Aggregates," NCHRP Web Document 66, 2004.
- Spellerberg, P.A., Savage, D.A., and Pielert, J.H., "Precision Estimates of Selected Volumetric Properties of HMA Using Non-Absorptive Aggregate," NCHRP Web Document 54, 2003.
- Tukey, John W., Exploratory Data Analysis, Addison-Wesley Publishing Co., Reading, MA, 1977, Chapter 2: (a) p. 44.
- Youden, W. J., "Statistical Techniques for Collaborative Tests", Association of Official Analytical Chemists, Inc., 1967: (a) pp.18 19.

APPENDIX A: Description of Hoaglin et al. Outlier Method

The method of identifying invalid data and outliers is a slightly modified version of a method for determining extreme data values described by Hoaglin et al.¹ This method uses the range of the two inner quartiles of a data set to determine the cut off values for outlying data and extreme outlying data:

$IF_U = F_U \pm k(F_U - F_L)$	(Equation 12)
$IF_L = F_L \pm k(F_U - F_L)$	(Equation 13)

Where:

 IF_U = Upper cutoff point for extreme value determination IF_L = Lower cutoff point for extreme value determination F_U = Upper quartile F_L = Lower quartile k = constant k where k = 1.5 for outlying data and k = 3 for extreme outlying data

The analysis technique in this study uses cut off limits at the same locations by using the range of the inner 75% of the data rather than use the inner quartiles (i.e. inner 50%). This way, the cut offs are based on a larger number of laboratories and the technique is more robust. Since the inner range of data is increased from 50% to 75%, the *k* values are decreased accordingly from k = 1.5 and k = 3 to k = 0.674 and k = 1.555, respectively.

¹ Hoaglin, D. C., Iglewicz, B., Tukey, J. W., "Performance of Some Resistant Rules for Outlier Labeling," Journal of the American Statistical Association, Vol. 81, No. 396 (Dec., 1986), pp. 991-999.

APPENDIX B: Example of Analysis Technique

Example for Determining Invalid Data

DATA SOURCE: AASHTO T314 Direct Tension Failure Strain (%)

SAMPLES: Performance Graded Binder Samples 195 and 196

Table of Statistics and Limits		Sample	(Y-X) -
		196, (Y)	(Ymed-Xmed)
Count = Number of Laboratories	60	60	60
Median	1.355	1.31	0.05
0.875 Percentile	1.85	1.91625	0.315
0.125 Percentile	1.00625	0.9525	-0.2375
Range of Inner 75% = (87.5th Percentile Value) - (12.5th Percentile Value)	0.84375	0.96375	0.5525
(1.555) x (Range of Inner 75%) =Dist Beyond Inner 75% for 4.725 Std Dev	1.312031	1.498631	0.8591375
Invalid Upper Limit = (87.5 th Percentile) + [(1.555) x (Range of Inner 75%)]	3.162031	3.414881	1.1741375
Invalid Lower Limit = (12.5 th Percentile) - [(1.555) x (Range of Inner 75%)]	-0.30578	-0.54613	-1.0966375

Table 16 – Table of Statistics and Limits

The data at the right is in descending order for Sample 195, (X). The laboratory numbers were assigned in ascending order to make them easier to locate in the column. The data for Sample 195 appears in the Column 2. Data for Sample 196 appears in the Column 3. The fourth column, labeled (X-Y) - (Ymed -Xmed), is the difference between the Sample 196 result and the Sample 195 result for each laboratory minus the difference between the median value for Sample 196 and the median value for Sample 195. The values in this fourth column provide an indication of the variation that can be expected between two test results determined by an individual laboratory. This column is ultimately used to estimate the repeatability.

Column 2 in the table at the right, containing data for sample X, and the Table of Statistics and Limits above can be used to demonstrate how Invalid Data was determined. The 87.5th percentile was determined using a function available in Microsoft EXCEL software. The value corresponding to the 87.5th percentile is 1.85, as shown in the table above. Similarly, the value corresponding to the 12.5th percentile was determined to be 1.00625. The range of the Inner 75% of the data extends from the 87.5th percentile down to the 12.5th percentile, providing a range of 1.85 - 1.00625 = 0.84375. The limits for determining Invalid Data are located at 1.555times the Range of the Inner 75% beyond the 87.5th percentile and below the 12.5th percentile. (For normally distributed data, these upper and lower limits are equivalent to 4.725 standard deviations from the center of the data. Since Invalid Data having extreme values can greatly affect the average value of the data, the median is used to estimate the center of the data rather than the average value.) In this case, (1.555) x (Range of the Inner 75%) = $1.555 \times 0.84375 = 1.312031$, as shown in the table above. The upper limit for determining Invalid Data is then equal to the value of the (87.5th percentile) + (1.312031) = (1.85) + (1.312031) = 3.162031. There are two data points for sample X having values greater than 3.162031. Those values for Sample 195 were reported for laboratories #1 and #2 and are shown as gray shaded in Column 2 of the table at the right. Laboratories #1 and #2 are then eliminated from any further analysis. The lower limit for determining Invalid Data is equal to the value of the (12.5 th percentile) - (1.312031) = (1.00625) - (1.312031) = -0.30578. For Sample 195, there are no results reported below -0.305781, so no other data is determined to be invalid for Sample 195.

Similarly using Column 3 in the table at the right and the table above, Invalid Data is determined for Sample 196. Any data above 3.414881 or below -0.54613 are considered to be invalid. Again the results for laboratories #1 and #2 are above the upper limit and are shown as gray shaded in the data at the right in Column 3.

		DATA	
LAB	Sample 195, (X)	Sample 196, (Y)	(Y-X)- (Ymed-Xmed)
1	4.89	5.28	0.39
2	3.82	3.82	0
3 4	2.57 2.3	2.41 2.32	-0.16
4 5	2.034	2.32	0.02 0.177
6	2.004	1.46	-0.54
7	1.97	2.24	0.27
8	1.85	1.91	0.06
9	1.85	1.78	-0.07
10	1.85	1.63	-0.22
11	1.84	1.81	-0.03
12 13	1.82	1.92	0.1
14	1.82 1.77	1.2 1.67	-0.62 -0.1
15	1.76	1.28	-0.48
16	1.67	1.59	-0.08
17	1.66	1.45	-0.21
18	1.63	2.06	0.43
19	1.62	1.91	0.29
20 21	1.62	1.19 1.26	-0.43 -0.29
21	1.55 1.54	1.20	-0.29
23	1.54	1.39	-0.15
24	1.53	1.48	-0.05
25	1.53	0.72	-0.81
26	1.44	1.29	-0.15
27	1.428	1.517	0.089
28	1.42	1.71	0.29
29 30	1.39 1.36	1.12 1.38	-0.27 0.02
31	1.35	0.93	-0.42
32	1.31	1.36	0.05
33	1.28	1.2	-0.08
34	1.24	1.23	-0.01
35	1.24	0.71	-0.53
36 37	1.23 1.22	1.29 1.26	0.06 0.04
38	1.22	1.48	0.27
39	1.19	1.26	0.07
40	1.18	1.33	0.15
41	1.18	1.21	0.03
42	1.18	1.04	-0.14
43 44	1.17 1.16	1.57 1.42	0.4 0.26
45	1.13	1.08	-0.05
46	1.13	1.04	-0.09
47	1.099	1.33	0.231
48	1.09	1.33	0.24
49	1.09	1.2	0.11
50	1.08	1.05	-0.03 0.17
51 52	1.07 1.05	1.24 0.91	-0.14
53	0.98	0.99	0.01
54	0.97	1.06	0.09
55	0.84	1.27	0.43
56	0.808	0.702	-0.106
57 50	0.69	0.77	0.08
58 59	0.63 0.6	0.58 1	-0.05 0.4
59 60	0.6	0.38	-0.12
	oblo 17	Exam	-0.12

Table 17 – Example Data

The same criteria is applied to Column 4 of the table at the right, marked (Y-X) - (Ymed - Xmed). Any values above 1.1741375 or below -1.0966375 would be considered as Invalid Data. In this case, there are no values that are considered invalid. However, the results from laboratories #1 and #2 are shown as gray shaded and are not included in further analysis because the results for those two laboratories were invalid for Samples 195 and 196. Any laboratory having any invalid results in any of the columns at the right is totally removed from any further analysis of this data for reproducibility or repeatability.

The diagram below identifies the data points for laboratories #1 and #2 that are eliminated from further analysis. Using a similar process, the data remaining after eliminating results for laboratories #1 and #2 are then analyzed for Outliers.

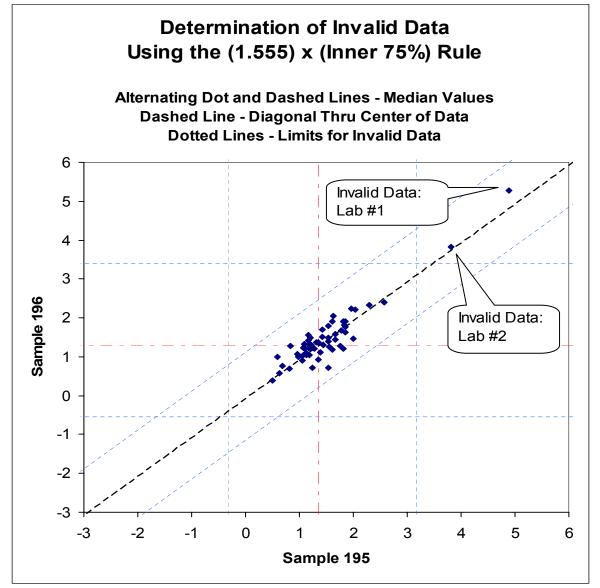


Figure 6 – Determination of Invalid Data

Example for Determining Outliers

TEST DATA: AASHTO T314 Direct Tension Failure Strain (%) SAMPLES: AMRL Performance Graded Binder Samples 195 and 196

Table of Statistics and Limits	Sample 195, (X)	Sample 196, (Y)	(Y-X)- (Ymed-Xmed)
Count = Number of Laboratories	58	58	58
Median	1.33	1.29	0.04
0.875 Percentile	1.84875	1.8975	0.30875
0.125 Percentile	0.98875	0.9375	-0.2475
Range of Inner 75% = (87.5th Percentile Value) - (12.5th Percentile Value)	0.86	0.96	0.55625
(0.674) x (Range of Inner 75%) = Dist Beyond 75% for 2.7 Std Dev	0.57964	0.64704	0.3749125
Outlier Upper Limit = (87.5 th Percentile) + [(0.674) x (Range of Inner 75%)]	2.42839	2.54454	0.6836625
Outlier Lower Limit = (12.5 th Percentile) - [(0.674) x (Range of Inner 75%)]	0.40911	0.29046	-0.6224125

Table 18 – Table of Statistics and Limits

Laboratories #1 and #2, whose results were determined to be Invalid Data, have been eliminated from the data at the right. The data remaining is arranged in descending order for Sample 195 and will be analyzed for Outliers in a manner similar to that previously applied to determine Invalid Data. Once again, the data for Sample 195 appears in Column 2. Data for Sample 196 appears in Column 3. The fourth column, marked (Y-X)-(Ymed-Xmed), is the difference between the Sample 196 result and the Sample 195 result for each laboratory minus the difference between the median value for Sample 196 and the median value for Sample 195. (New median values were calculated after laboratories # 1 and # 2 were removed.) The values in this fourth column provide an indication of the variation that can be expected between two test results determined by an individual laboratory. This column will ultimately be used to determine an estimate of repeatability.

Column 2 and the above Table of Statistics and Limits can be used to demonstrate how Outliers were determined. The 87.5th percentile, for the data remaining after the elimination of Invalid Data, was determined using a function available in Microsoft EXCEL software. The value corresponding to the 87.5th percentile is 1.84875, as shown in the table above. Similarly, the value corresponding to the 12.5th percentile was determined to be 0.98875. The range of the Inner 75% of the data extends from the 87.5th percentile down to the 12.5th percentile, providing a range of 1.84875 -0.98875 = 0.86. The limits for determining Outliers are located at 0.674 times the Range of the Inner 75% beyond the 87.5th percentile and below the 12.5th percentile. (For normally distributed data, these limits are equivalent to 2.7 standard deviations from the center of the data. Since Outliers having extreme values can greatly affect the average value of the data, the median is used to estimate the center of the data rather than the average value.) In this case, $(0.674) \times (Range of the Inner 75\%) = 0.674 \times 10^{-10}$ 0.86 = 0.57964, as shown in the table above. The upper limit for determining Invalid Data is then equal to the value of the (87.5th percentile) + (0.57964) = (1.84875) +(0.57964) = 2.42839. There is one point in Column 2 having a value greater than 2.42839. That value was reported by laboratory #3 and is shown as gray shaded at the top of Column 2. Laboratory #3 is then eliminated from any further analysis. The lower limit for determining Outliers is equal to the value of the (12.5th percentile) -(0.57964) = (0.98875) - (0.57964) = 0.40911. For Sample 195, there are no results reported below 0.40911, so no other point is determined to be an Outlier for Sample 195.

DATA					
LAB	Sample	Sample	(Y-X)-		
3	195, (X) 2.57	196, (Y) 2.41	(Ymed-Xmed) -0.12		
4	2.3	2.32	0.06		
5	2.034	2.211	0.217		
6	2	1.46	-0.5		
7	1.97	2.24	0.31		
8	1.85	1.91	0.1		
9	1.85	1.78	-0.03		
10	1.85	1.63	-0.18		
11	1.84	1.81	0.01		
12	1.82	1.92	0.14		
13	1.82	1.2	-0.58		
14	1.77	1.67	-0.06		
15	1.76	1.28	-0.44		
16	1.67	1.59	-0.04		
17	1.66	1.45	-0.17		
18	1.63	2.06	0.47		
19	1.62	1.91	0.33		
20	1.62	1.19	-0.39		
21	1.55	1.26 1.79	-0.25 0.29		
22 23	1.54 1.54	1.79	-0.11		
23 24	1.54	1.39	-0.01		
25	1.53	0.72	-0.77		
26	1.44	1.29	-0.11		
27	1.428	1.517	0.129		
28	1.42	1.71	0.33		
29	1.39	1.12	-0.23		
30	1.36	1.38	0.06		
31	1.35	0.93	-0.38		
32	1.31	1.36	0.09		
33	1.28	1.2	-0.04		
34	1.24	1.23	0.03		
35	1.24	0.71	-0.49		
36	1.23	1.29	0.1		
37	1.22	1.26	0.08		
38	1.21	1.48	0.31		
39	1.19	1.26 1.33	0.11		
40 41	1.18 1.18	1.33	0.19		
41	1.18	1.21	0.07 -0.1		
43	1.17	1.57	0.44		
44	1.16	1.42	0.3		
45	1.13	1.08	-0.01		
46	1.13	1.04	-0.05		
47	1.099	1.33	0.271		
48	1.09	1.33	0.28		
49	1.09	1.2	0.15		
50	1.08	1.05	0.01		
51	1.07	1.24	0.21		
52	1.05	0.91	-0.1		
53	0.98	0.99	0.05		
54	0.97	1.06	0.13		
55	0.84	1.27	0.47		
56	0.808	0.702	-0.066		
57	0.69	0.77	0.12		
58	0.63	0.58	-0.01		
59	0.6	1	0.44		
60	0.5	0.38	-0.08		

Table 19 – Example Data

Similarly using Table 17 and Column 3 of Table 18, Outliers are determined for Sample 196. Any point above 2.54454 or below 0.29046 would be considered to be an Outlier. There are no points that exceed the Outlier limits for Sample 196, however, laboratory #3 appears as gray shaded in Column 3 of Table 18 since laboratory #3 was previously eliminated based on results for Sample 195.

From Table 17, the upper and lower Outlier limits for the fourth column of Table 18, marked (Y-X)-(Ymed-Xmed), are 0.6836625 and -0.6224125, respectively. In the fourth column, the value for laboratory #25, -0.77, is beyond the lower Outlier limit. Therefore, -0.77 is considered to be an Outlier and laboratory #25 is eliminated from any further analysis. The results for laboratory #25 are shown as gray shaded in Table 18.

The diagram below identifies the points that were eliminated as Outliers. The core data points remaining after eliminating results from laboratories #1, #2, #3, and #25 (i.e. those points contained in the hexagon) were used in the final analysis to estimate repeatability and reproducibility.

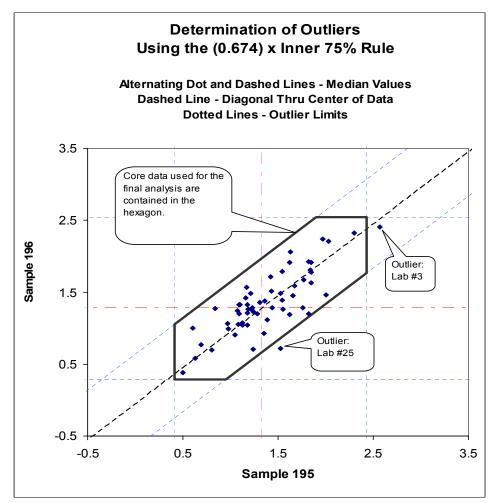


Figure 7 – Determination of Outliers

APPENDIX C: Summary Table for DSR Phase Angle Testing on Original Binder

						Average	e Results		Repeatabili	ty	Reprodu	cibility	Reprodu	cibility
	Sample	Sample	No. of	PG	AC	odd	even		odd	even	od	d	eve	en
	Туре	Numbers	Labs	Grade	Grade	samples	samples		samples	samples	samp	oles	samp	oles
								1s	CV%	CV%	1s	CV%	1s	CV%
	PGB	181 & 182	170	PG 64-16	AC 10	89.0	89.1	0.150	0.17	0.17	0.295	0.33	0.339	0.38
	PGB	183 & 184	173	PG 70-22		83.9	83.9	0.141	0.17	0.17	0.299	0.36	0.283	0.34
	PGB	185 & 186	171	PG 64-22	AC 20	87.7	87.7	0.142	0.16	0.16	0.279	0.32	0.284	0.32
$ \square $	PGB	187 & 188	184	PG 76-22		69.8	69.7	0.218	0.31	0.31	0.452	0.65	0.440	0.63
	PGB	189 & 190	186	PG 64-22	AC 30	87.2	87.2	0.114	0.13	0.13	0.248	0.28	0.235	0.27
	PGB	191 & 192	190	PG 52-34	AC 10	82.1	82.2	0.137	0.17	0.17	0.282	0.34	0.308	0.37
	PGB	193 & 194	194	PG 64-22	AC 20	86.3	86.3	0.115	0.13	0.13	0.247	0.29	0.244	0.28
	PGB	195 & 196	189	PG 70-22		85.7	85.7	0.120	0.14	0.14	0.240	0.28	0.235	0.27

Table 20 – Summary Table for T315, Phase Angle for Original Binder

Chapter 3 Sample 187 and sample 188 listed in the above table contains a modified binder.

APPENDIX D: Normal Summary Tables

Test T308	Ignition Oven			
Sample #	d2s (2.83xStDev)	5% diff (By Count)	R-Squared for Norm Prob Plot	Input Analyzer Best Fit
3	0.30	0.30	0.9921	Normal
4	0.31	0.31	0.9892	Beta
5	0.34	0.33	0.9934	Normal
6	0.33	0.33	0.9876	Normal
7	0.35	0.35	0.9913	Beta
8	0.34	0.34	0.9917	Normal
average =	0.33	0.33		

Sample #	d2s (2.83xStDev)	5% diff (By Count)	R-Squared for Norm Prob Plot	Input Analyze Best Fit
181	26	26	0.9798	Normal
182	27	26	0.9859	Weibull
183	26	27	0.9533	Normal
184	24	24	0.9664	Normal
185	35	34	0.9834	Triangular
186	36	35	0.9898	Normal
187	35	36	0.9222	Erlang
188	34	35	0.9347	Gamma
189	26	25	0.9903	Triangular
190	25	25	0.9944	Normal
191	35	34	0.9780	Weibull
192	34	34	0.9580	Triangular
193	20	20	0.9807	Normal
194	21	21	0.9755	Normal
195	22	22	0.9781	Normal
196	21	21	0.9751	Normal
average =	28	28		

ecific Ca

Test T313BBR Stiffness

average

181 182

Test T228	Specific Gravi	ty		
	d2s	5% diff	R-Squared for	
Sample #	(2.83xStDev)	(By Count)	Norm Prob Plot	Best Fit
181	0.0034	0.003	0.9258	Weibull
182	0.0034	0.003	0.9206	Weibull
183	0.0046	0.005	0.9507	Lognormal
184	0.0048	0.005	0.9353	Lognormal
185	0.0037	0.004	0.9243	Normal
186	0.0036	0.004	0.9210	Lognormal
187	0.0032	0.003	0.9158	Weibull
188	0.0037	0.004	0.9073	Triangular
189	0.0028	0.003	0.8818	Weibull
190	0.0037	0.004	0.8625	Weibull
191	0.0037	0.004	0.9159	Normal
192	0.0034	0.003	0.9362	Beta
193	0.0028	0.003	0.9116	Weibull
194	0.0022	0.002	0.8554	Weibull
195	0.0039	0.004	0.9604	Normal
196	0.0037	0.004	0.9535	Weibull
average =	0.0035	0.0036		

Table 21 – T308 Ignition Oven

Test T240 RTFO Loss d2s 5% diff R-Squared for Sample # (2.83xStDev) (By Count) Norm Prob Plot Input Analyzer Best Fit 0.16 0.16 0.06 0.16 0.16 0.06 0.9940 0.9885 0.9954 181 182 183 Normal Normal Normal 184 0.06 0.06 0.9831 Erlang 0.9783 185 0.12 0.12 Beta 0.9826 0.9965 0.9934 0.9840 Normal Normal 186 187 0.12 0.12 0.20 0.20 188 0.19 Normal Weibull 0.06 0.06 190 191 192 0.9840 0.9799 0.9964 0.9940 0.06 0.00 Weibull 0.23 Norma 0.23 0.23 Normal 193 194 0.06 0.06 0.9896 Normal Normal 195 0.05 0.05 0.9829 Erlang Normal 196 0.05 0.9831 average 0.12 0.12

Table 24- T240 RTFO Loss

Test T313	BBR Slope			
	d2s	5% diff	R-Squared for	Input Analyzer
Sample #	(2.83xStDev)	(By Count)	Norm Prob Plot	Best Fit
181	0.024	0.024	0.9907	Normal
182	0.026	0.025	0.9930	Normal
183	0.019	0.019	0.9941	Beta
184	0.022	0.021	0.9949	Erlang
185	0.024	0.023	0.9900	Normal
186	0.025	0.025	0.9930	Normal
187	0.028	0.027	0.9954	Weibull
188	0.025	0.025	0.9914	Weibull
189	0.021	0.021	0.9917	Gamma
190	0.023	0.023	0.9905	Normal
191	0.023	0.023	0.9946	Normal
192	0.024	0.024	0.9926	Weibull
193	0.016	0.016	0.9934	Weibull
194	0.017	0.016	0.9884	Beta
195	0.023	0.023	0.9921	Normal
196	0.023	0.023	0.9936	Beta
average =	0.023	0.022		
	Table 25	L T313	BBR Slo	no

Table 25- T313 BBR Slope

Test T314DT Strain					
d2s	5% diff	R-Squared for	Input Analyzer		

Test T314 DT Stress						
	d2s	5% diff	R-Squared for	Input Analyzer		
Sample #	(2.83xStDev)	(By Count)	Norm Prob Plot	Best Fit		
181	1.5	1.5	0.9596	Weibull		
182	1.6	1.6	0.9650	Normal		
183	2.3	2.2	0.9685	Beta		
184	2.2	2.1	0.9403	Triangular		
185	1.7	1.6	0.9779	Triangular		
186	1.8	1.8	0.9697	Normal		
187	1.7	1.6	0.9495	Beta		
188	1.6	1.6	0.9723	Triangular		
189	2.5	2.4	0.9778	Triangular		
190	2.7	2.6	0.9827	Triangular		
191	1.8	1.8	0.9820	Triangular		
192	1.9	1.9	0.9854	Beta		
193	2.1	2.1	0.9163	Triangular		
194	2.2	2.3	0.9194	Beta		
195	1.8	1.8	0.9734	Normal		
196	2.0	2.0	0.9737	Normal		
average =	2.0	1.9				
Τa	Table 27- T314 DT Stress					

Sample #	(2.83xStDev)	(By Count)	Norm Prob Plot	Best Fit
181	0.7	0.7	0.9703	Normal
182	0.7	0.7	0.9827	Gamma
183	1.8	1.8	0.9818	Weibull
184	2.0	1.9	0.9766	Beta
185	1.0	0.9	0.9689	Triangular
186	1.0	1.0	0.9815	Beta
187	3.7	3.5	0.9820	Weibull
188	3.5	3.3	0.9906	Triangular
189	1.2	1.1	0.9856	Normal
190	1.3	1.3	0.9773	Normal
191	0.8	0.7	0.9911	Triangular
192	0.8	0.8	0.9898	Beta
193	2.9	2.8	0.9875	Normal
194	2.8	2.7	0.9850	Weibull
195	1.1	1.1	0.9842	Erlang
196	1.1	1.1	0.9784	Beta
average =	1.7	1.6		
	Table 2	8- T314	DT Strai	n

Test 315 DSR on PAV 5% diff R-Squared for Input Analyzer d2s

Sample #	(2.83xStDev)	(By Count)	Norm Prob Plot	Best Fit
181	1966	1944	0.9901	Normal
182	1855	1886	0.9767	Normal
183	830	810	0.9960	Triangular
184	887	867	0.9963	Normal
185	1489	1468	0.9821	Normal
186	1375	1372	0.9914	Normal
187	471	465	0.9958	Normal
188	444	434	0.9859	Weibull
189	1689	1686	0.9893	Normal
190	1706	1703	0.9882	Normal
191	1866	1833	0.9969	Normal
192	1866	1834	0.9943	Normal
193	1029	1010	0.9952	Normal
194	1016	1005	0.9951	Normal
195	1222	1212	0.9918	Normal
196	1200	1191	0.9930	Normal
average =	1307	1295		

Table 31- T315 DSR PAV

0.2 Table 29- T315 DSR Original

Test 316 Brookfield Viscosity d2s 5% diff R-Squared for Input Analyzer

Sample #	(2.83xStDev)	(By Count)	Norm Prob Plot	Best Fit
181	0.042	0.040	0.9578	Beta
182	0.042	0.040	0.9563	Beta
183	0.080	0.080	0.9867	Normal
184	0.083	0.080	0.9855	Normal
185	0.047	0.050	0.9634	Lognormal
186	0.043	0.040	0.9596	Erlang
187	0.199	0.190	0.9968	Beta
188	0.194	0.190	0.9910	Normal
189	0.045	0.040	0.9655	Normal
190	0.044	0.040	0.9710	Normal
191	0.033	0.030	0.9623	Erlang
192	0.036	0.035	0.9633	Gamma
193	0.057	0.060	0.9781	Beta
194	0.056	0.058	0.9810	Gamma
195	0.087	0.085	0.9954	Weibull
196	0.086	0.083	0.9953	Weibull
average =	0.073	0.071		

Table 32- T316 Rotational Viscosity

Test 315	DSR on RTFC)	
	d2s	5% diff	R-Squared for
Sample #	(2.83xStDev)	(By Count)	Norm Prob Plc
181	0.52	0.51	0.9959
182	0.49	0.48	0.9935
100			0.0001

183	0.55	0.55	0.9891	Normal
184	0.57	0.56	0.9889	Normal
185	0.64	0.63	0.9914	Normal
186	0.62	0.62	0.9815	Normal
187	0.75	0.73	0.9937	Weibull
188	0.67	0.65	0.9938	Normal
189	0.76	0.76	0.9805	Normal
190	0.72	0.71	0.9909	Normal
191	2.21	2.18	0.9939	Normal
192	2.07	2.05	0.9872	Gamma
193	0.68	0.68	0.9890	Normal
194	0.69	0.69	0.9804	Gamma
195	0.50	0.49	0.9863	Gamma
196	0.55	0.54	0.9860	Gamma
average =	0.81	0.80		

Norma Normal

Table 30- T315 DSR RTFO

averag

	183	0.23	0.23	0.9939
_	184	0.22	0.22	0.9902
-	185	0.20	0.19	0.9926
	186	0.21	0.21	0 9914

0.17 0.17

d2s 5% diff R-Squared for Sample # (2.83xStDev) (By Count) Norm Prob Plot

Test 315 DSR on Original

183	0.23	0.23	0.9939	Normal
184	0.22	0.22	0.9902	Weibull
185	0.20	0.19	0.9926	Normal
186	0.21	0.21	0.9914	Beta
187	0.21	0.20	0.9949	Beta
188	0.21	0.20	0.9952	Beta
189	0.26	0.25	0.9957	Normal
190	0.24	0.24	0.9903	Normal
191	0.52	0.51	0.9909	Normal
192	0.50	0.49	0.9928	Normal
193	0.23	0.22	0.9878	Gamma
194	0.22	0.22	0.9834	Erlang
195	0.27	0.27	0.9913	Normal
196	0.27	0.27	0.9907	Normal

0.17 0.17

185 186 18 188

d2s 5% diff R-Squared for Sample # (2.83xStDev) (By Count) Norm Prob Plot Best Fit 181 182 183 0.9725 0.9896 0.9857 Normal Normal Normal 33 33 184 34 33 0.9951 Erlang 0.9899 Beta Normal Triangular Weibull 34 33 0.9907 0.9789 22 24 24 Normal 189 36 37 35 37 0.9896 0.9896 0.9916 0.9911 0.9888 190 Normal Norm 41 192 41 Weibull 0.9820 0.9933 193 194 25 23 25 23 Normal Beta 195 36 37 35 0.9850 0.9815 Normal 196 37 Beta

Table 26- T313 BBR Stiffness

R-Squared for Input Analyzer Best Fit

Weibull Weibull

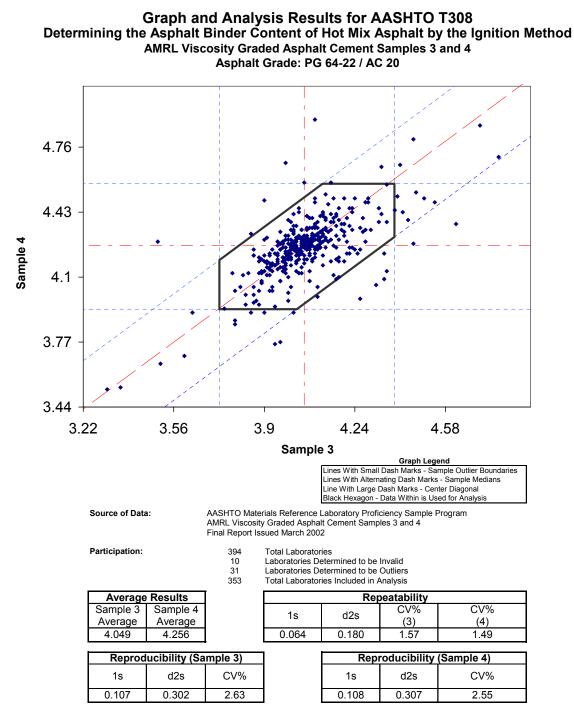
0.9947 0.9957

Table 23- T228 Specific Gravity

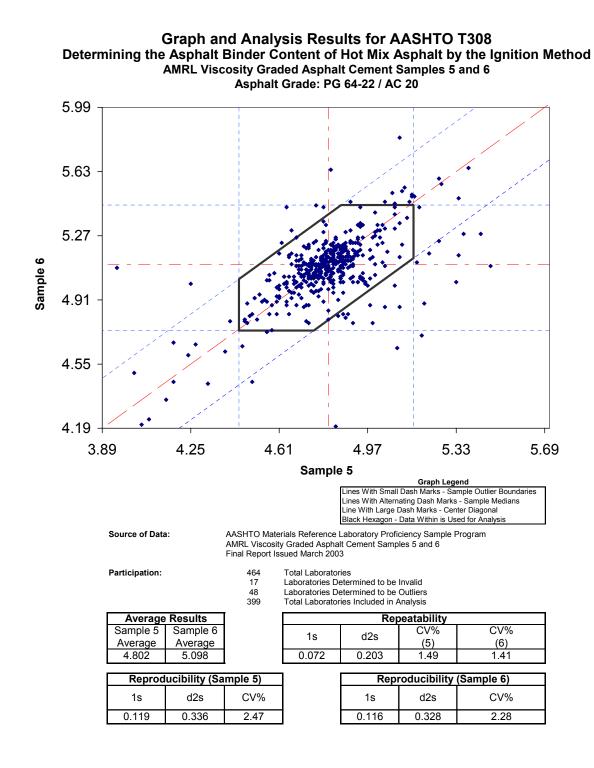
Input Analyzer

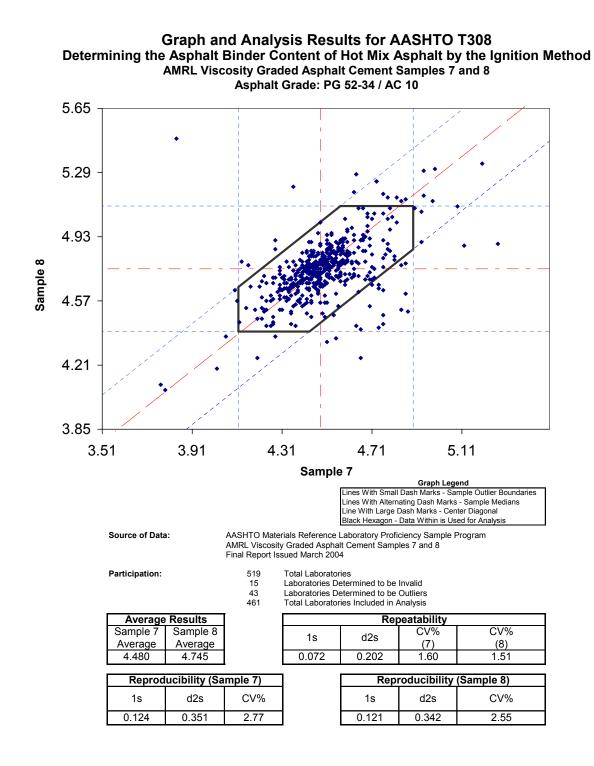
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APPENDIX E

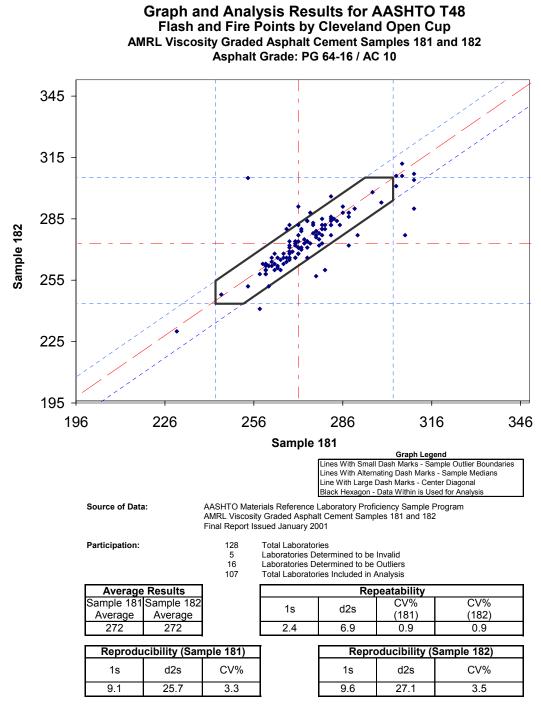


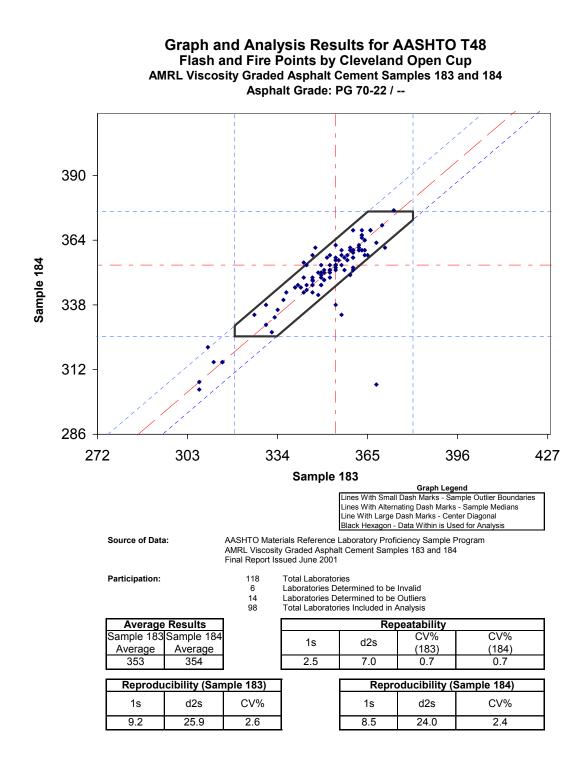
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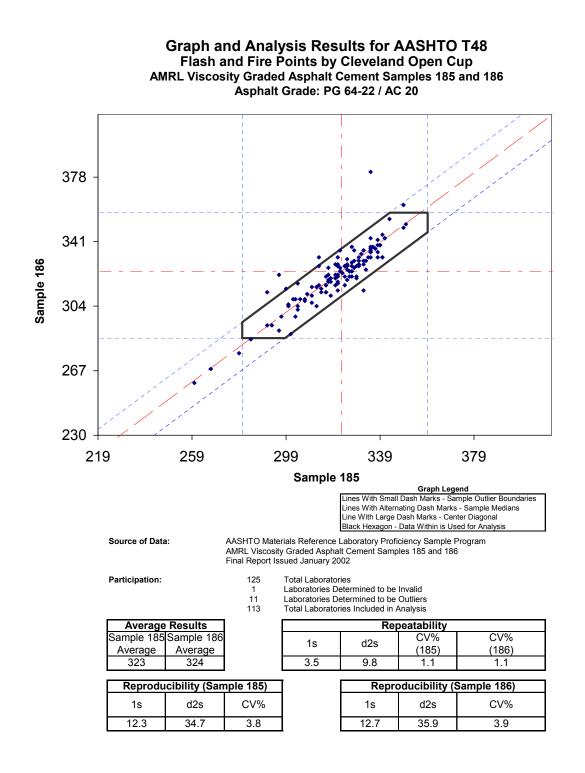


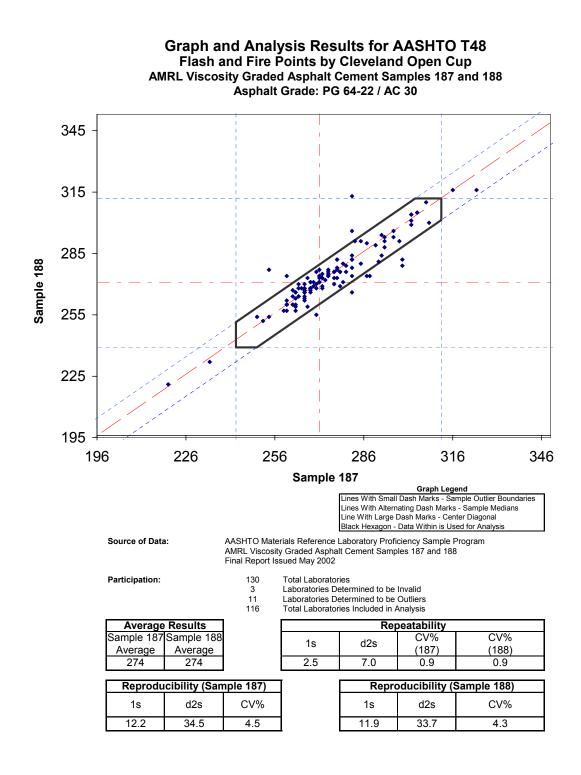


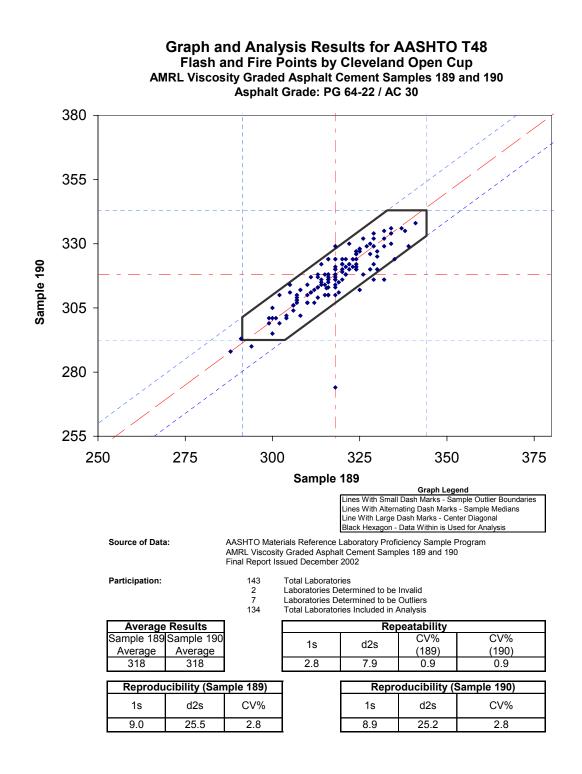
APPENDIX F

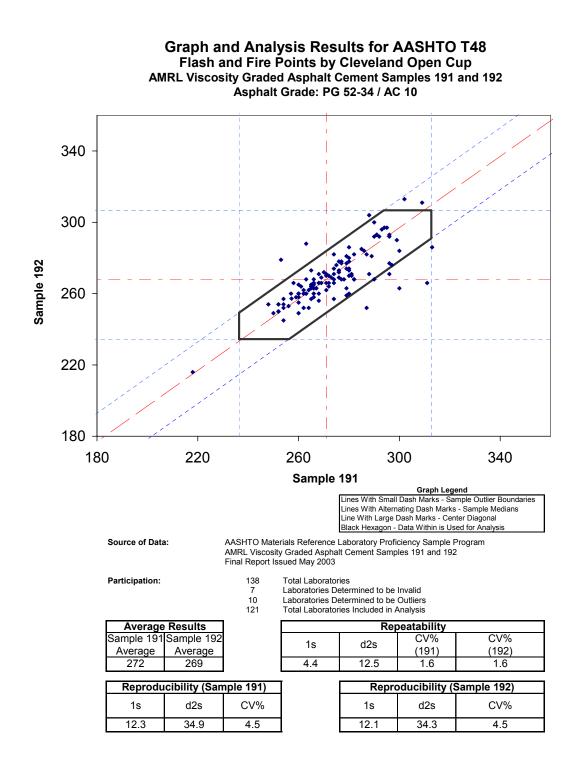


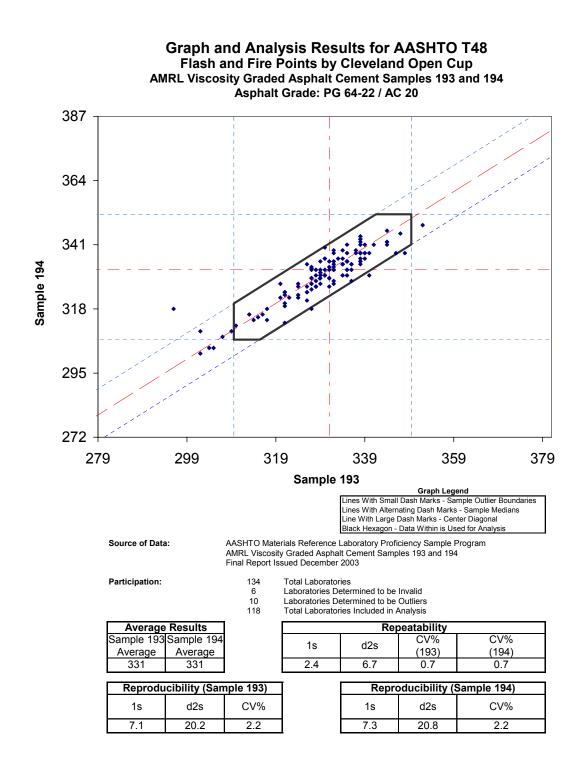


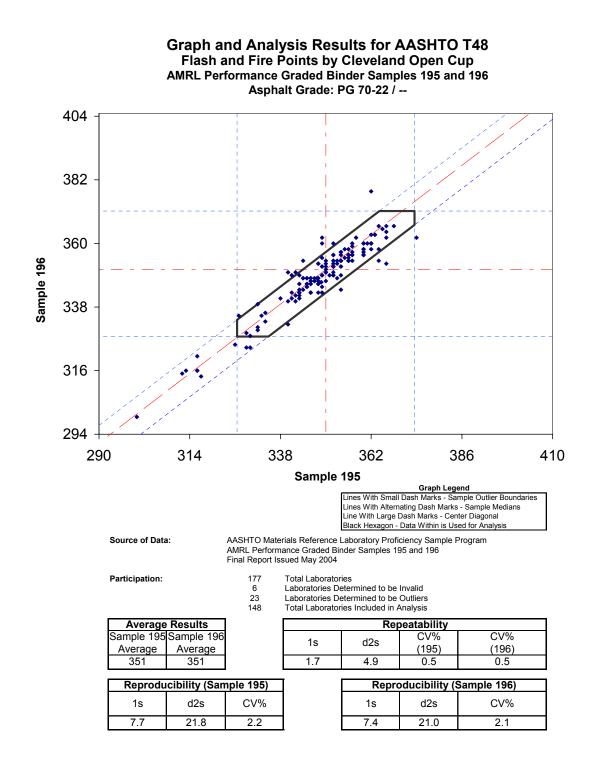




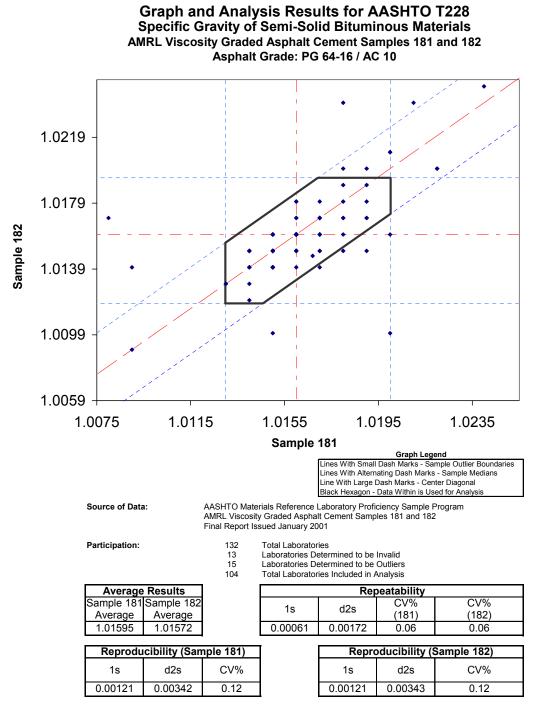


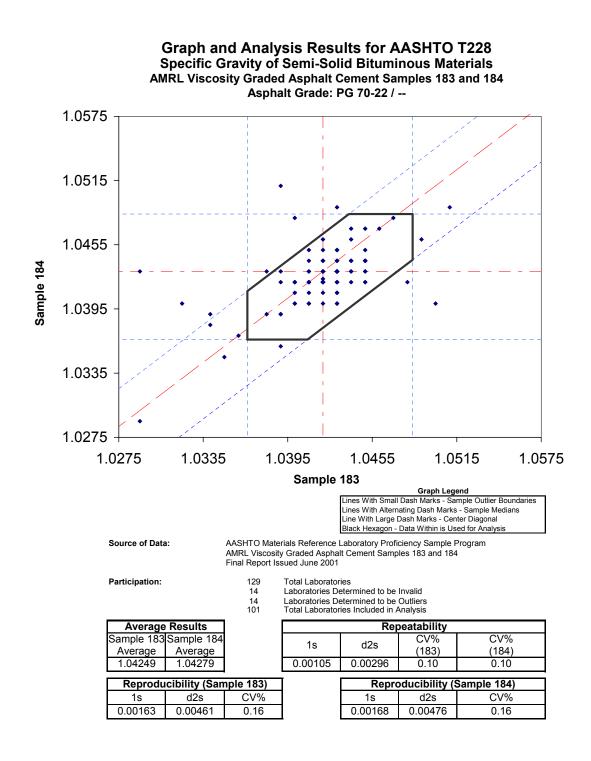


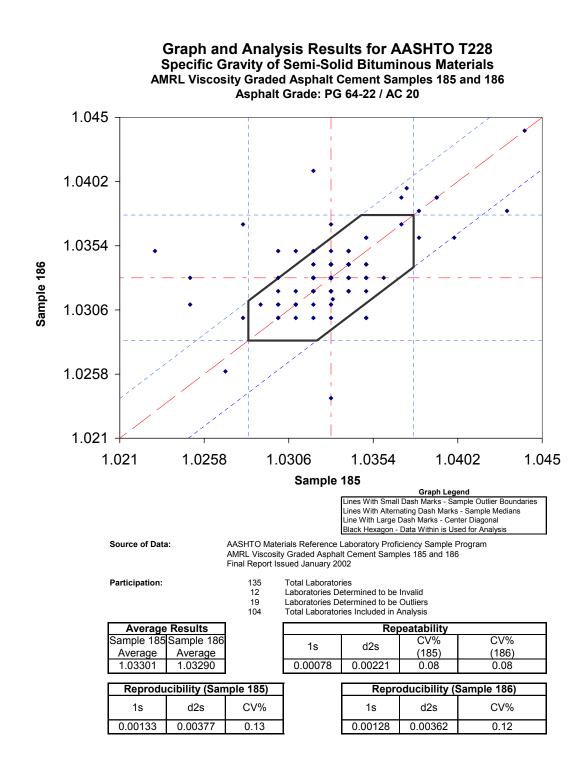


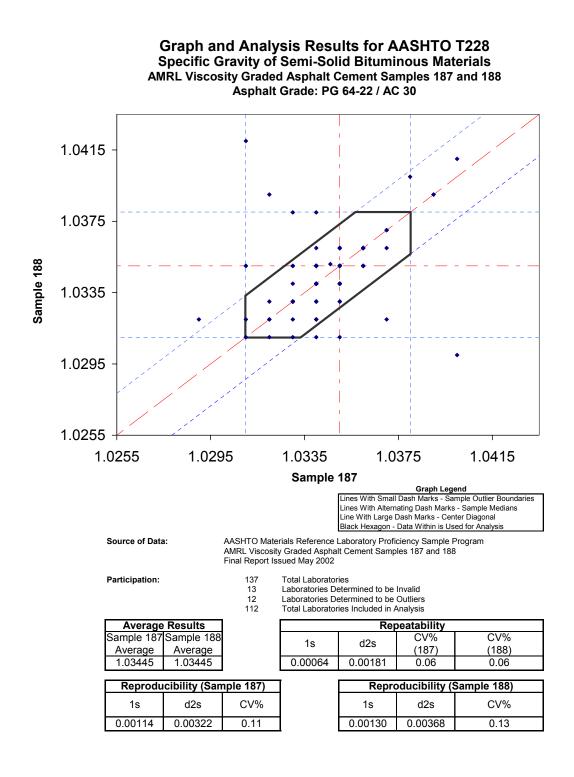


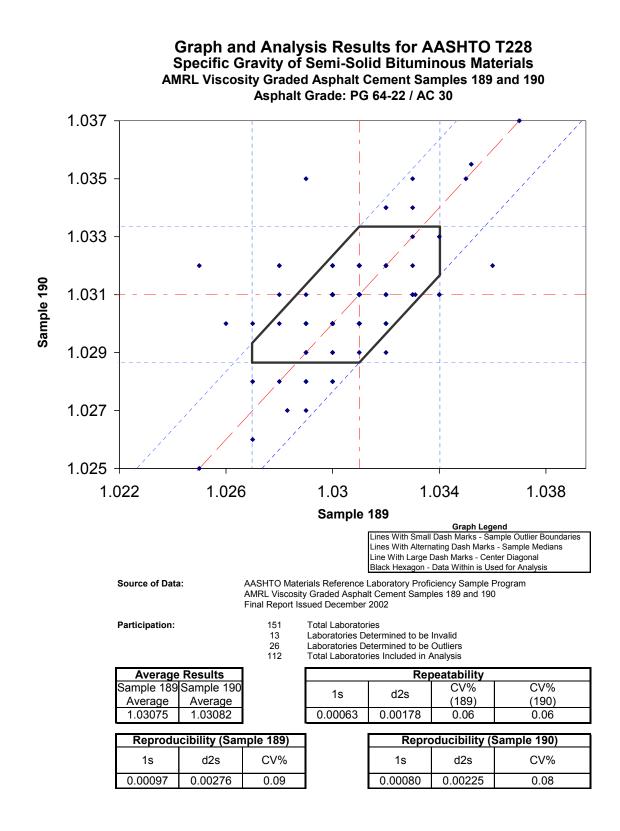
APPENDIX G

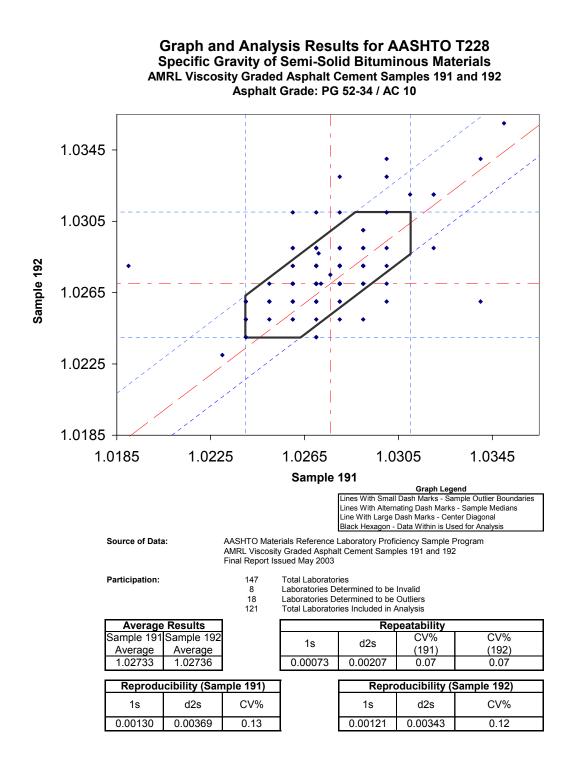


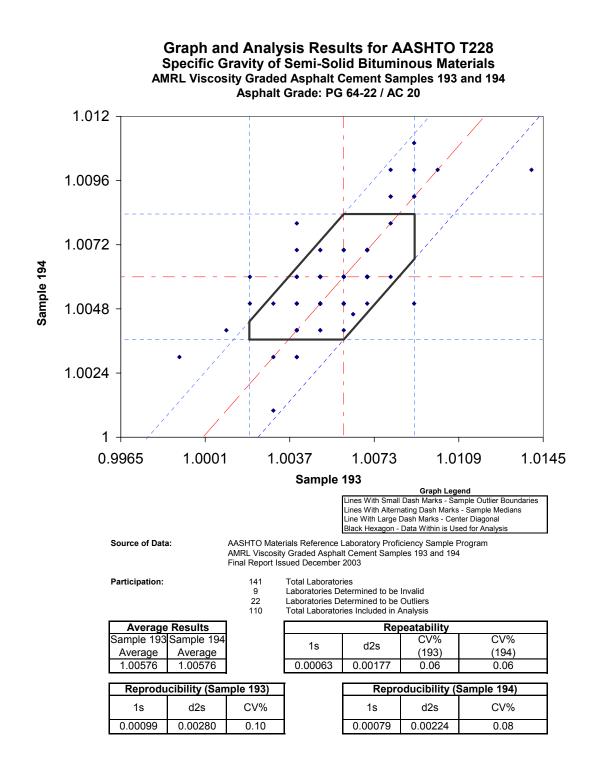


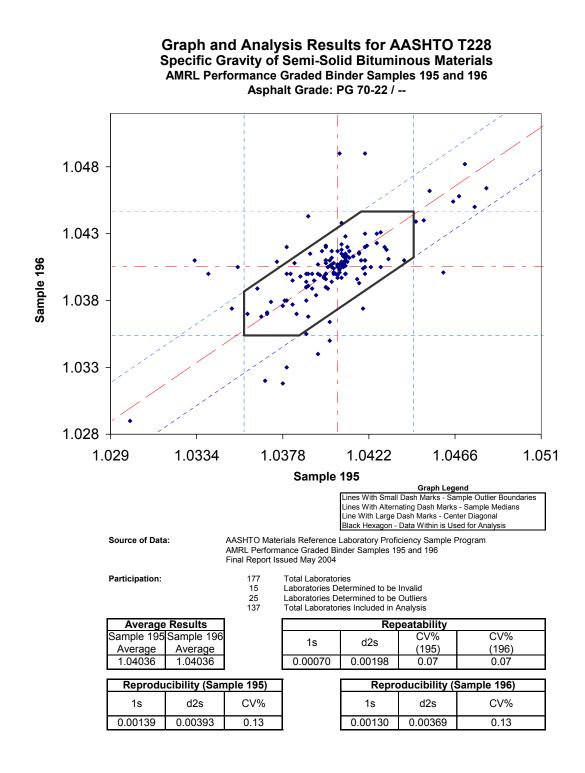




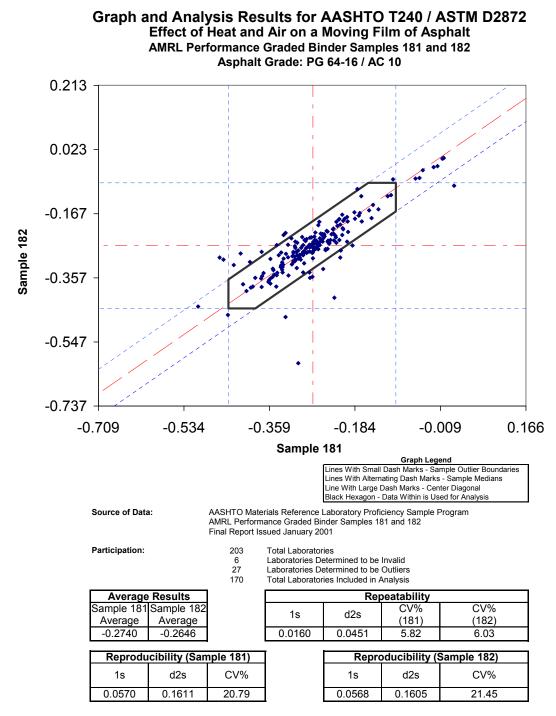


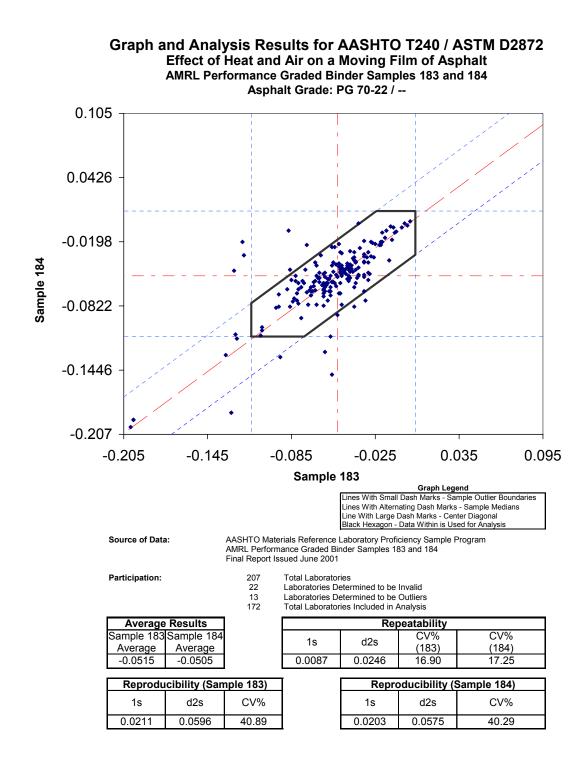


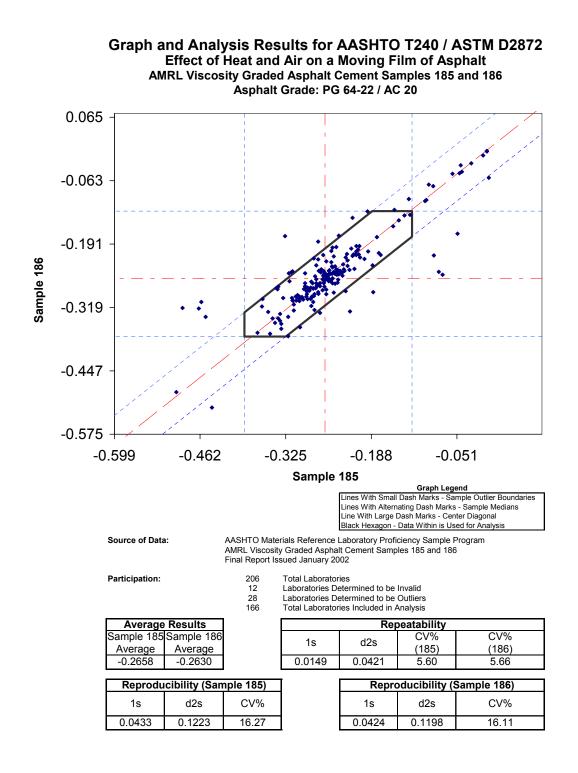


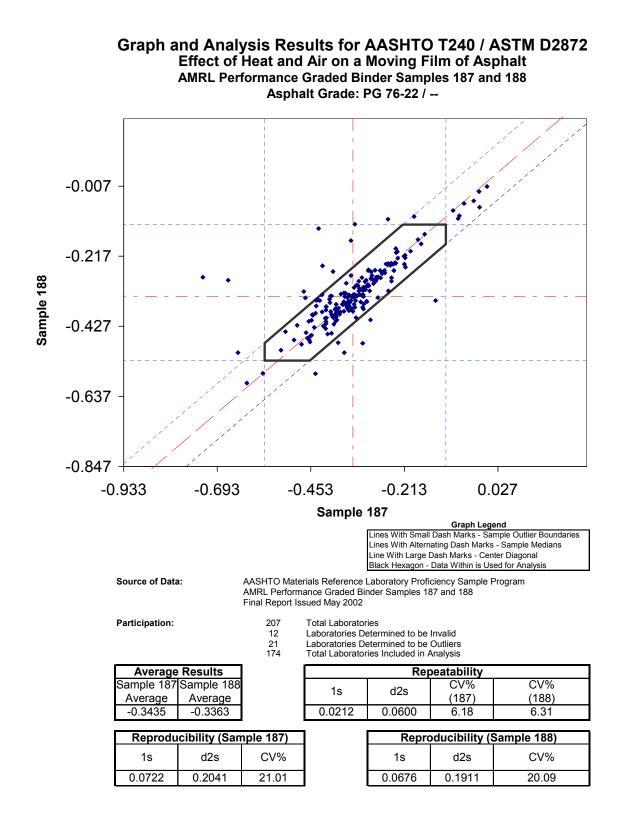


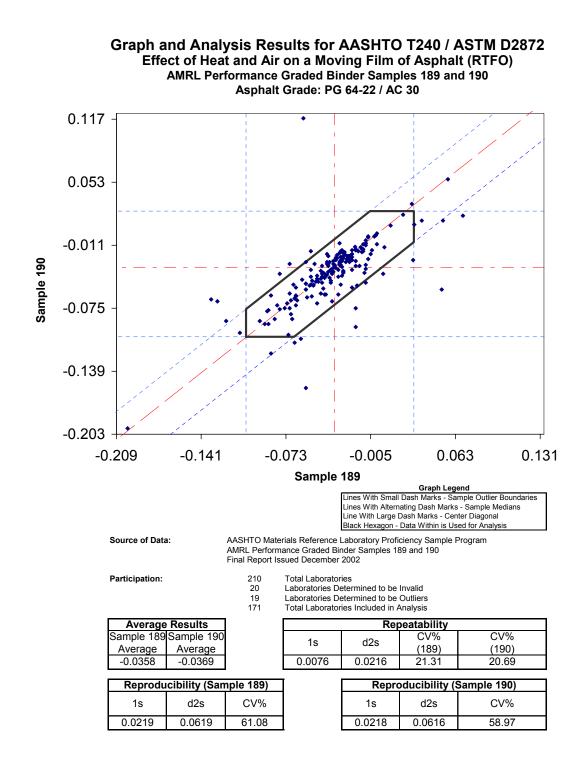
APPENDIX H

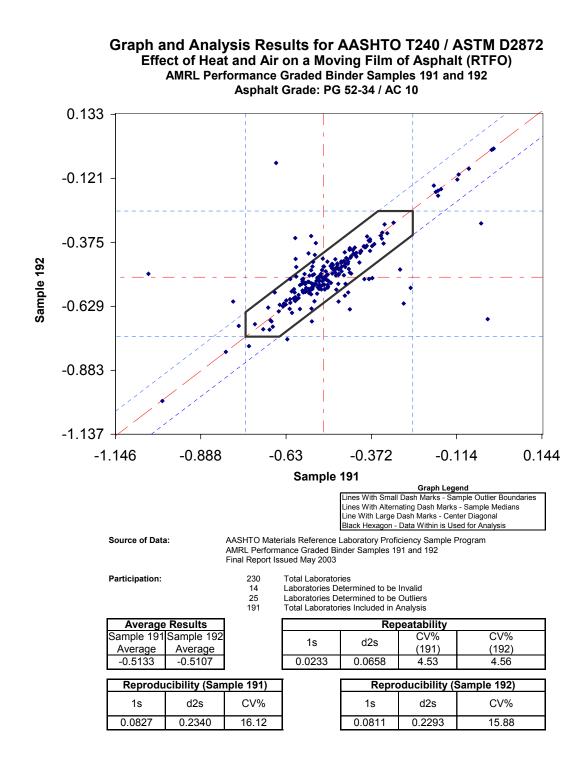


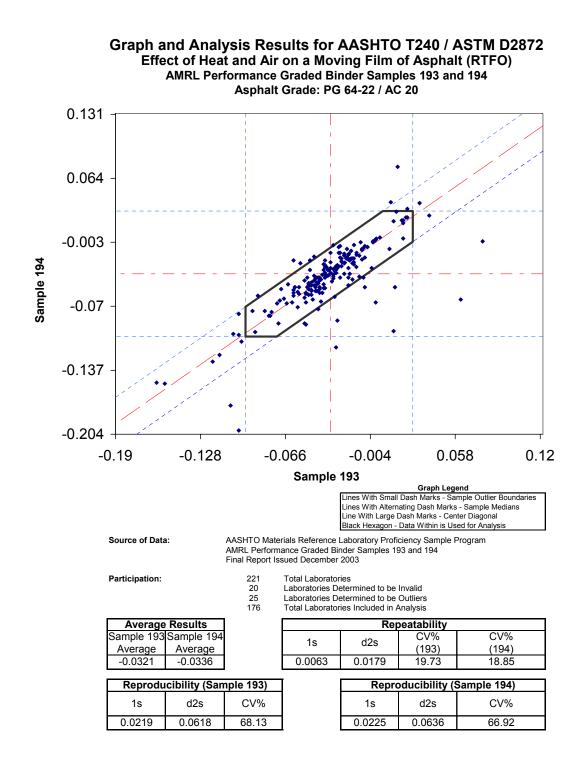


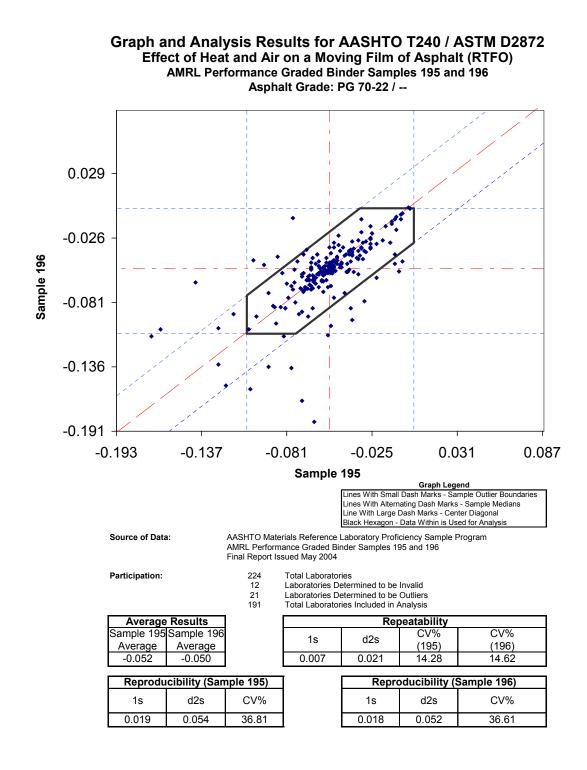




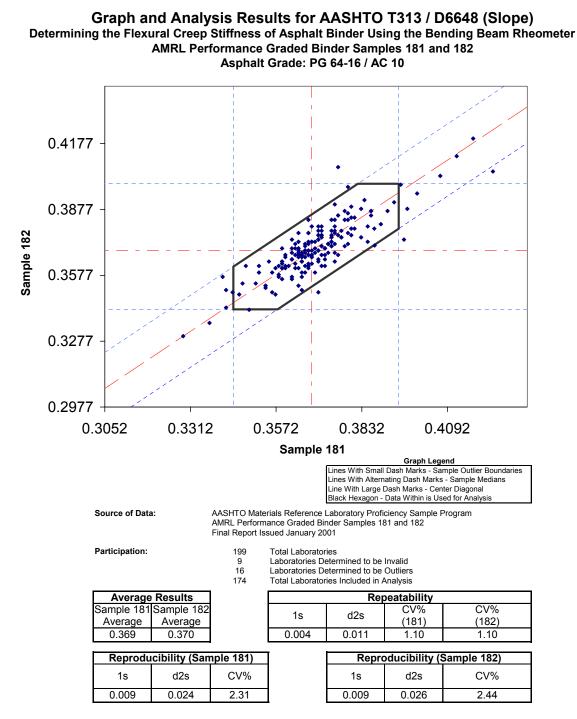


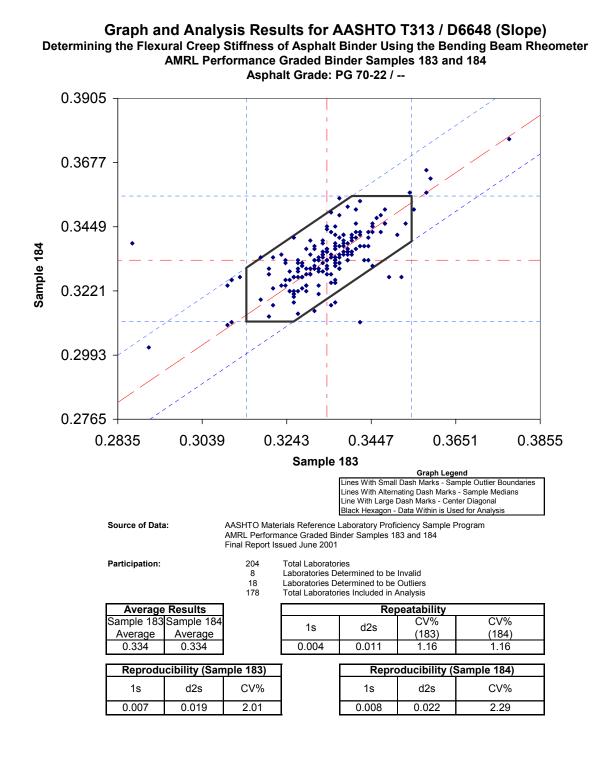


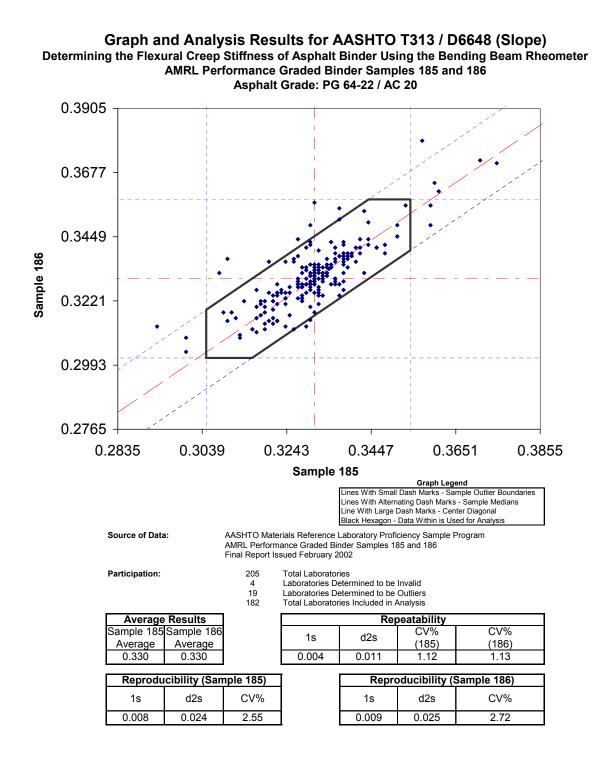


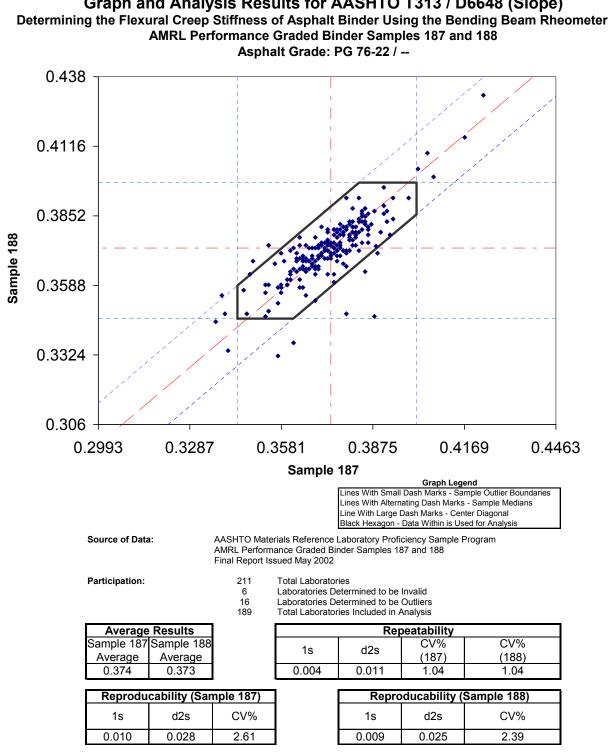


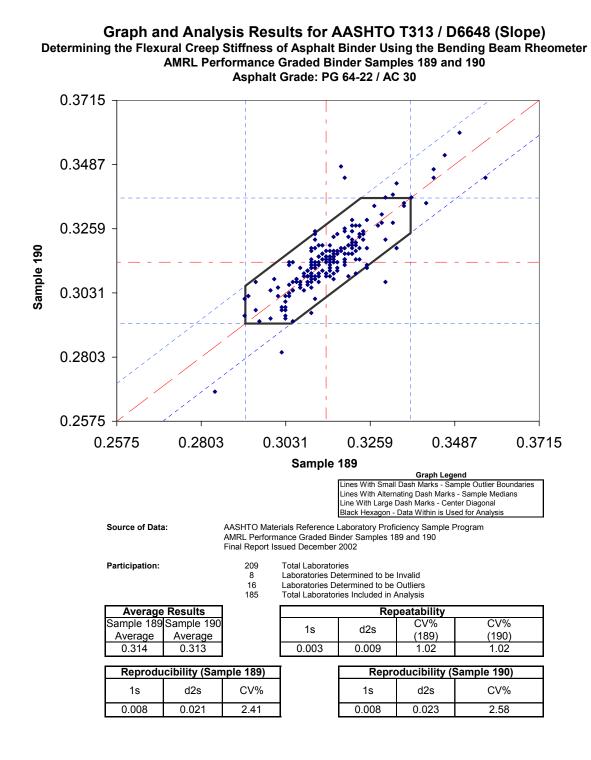
APPENDIX I

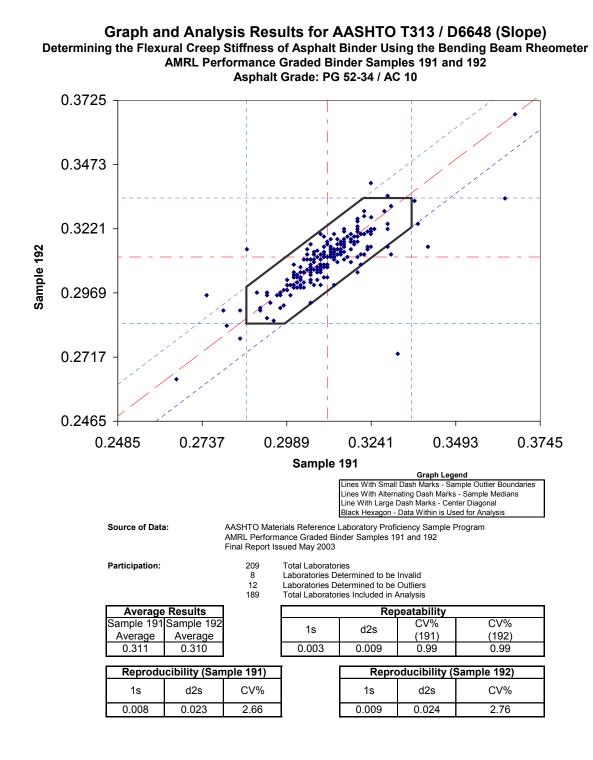


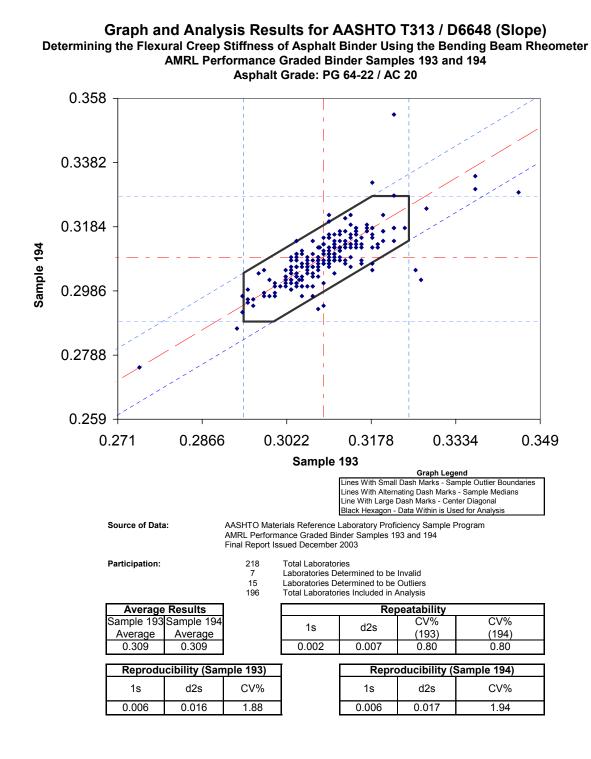


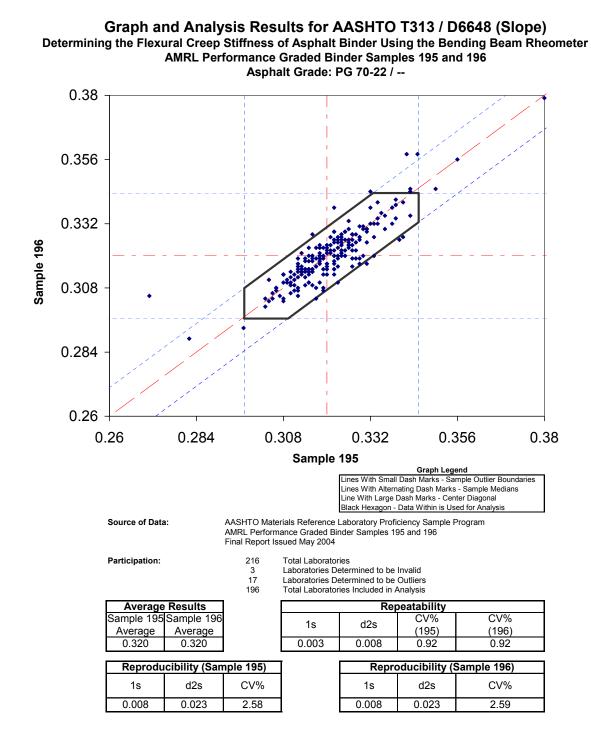




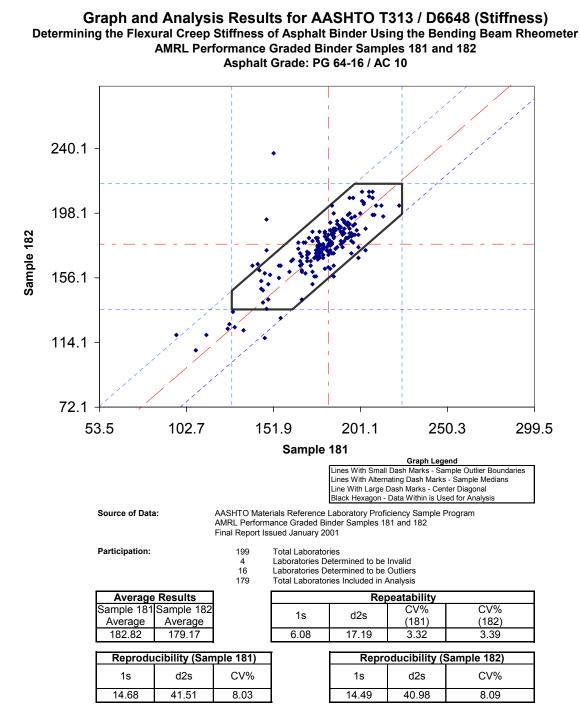


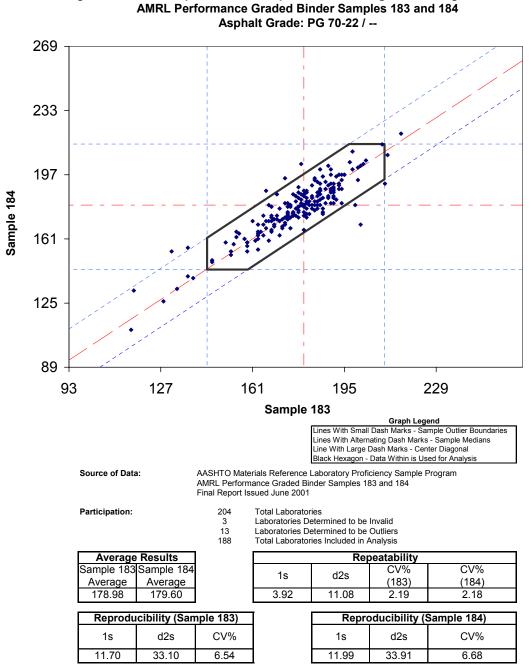


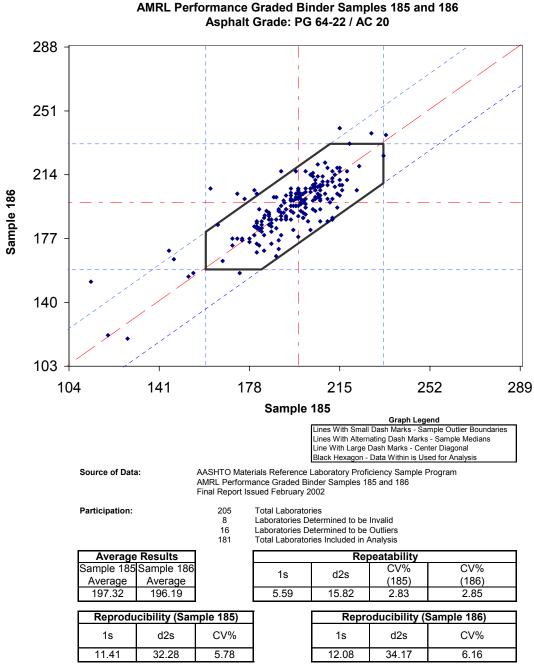




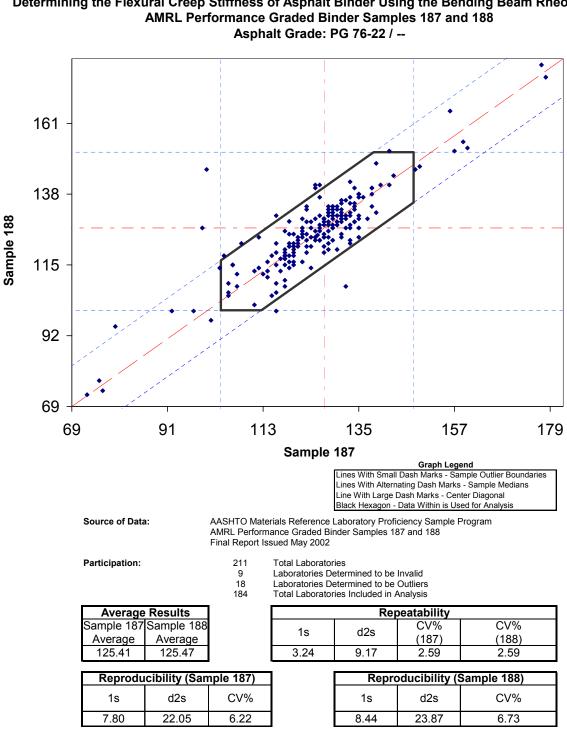
APPENDIX J



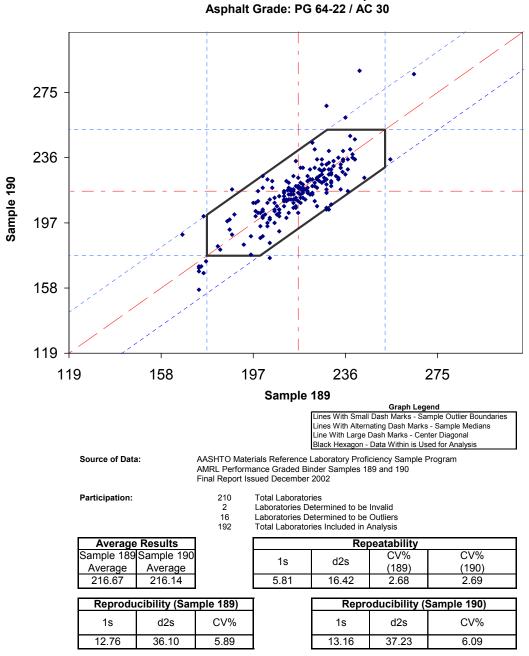




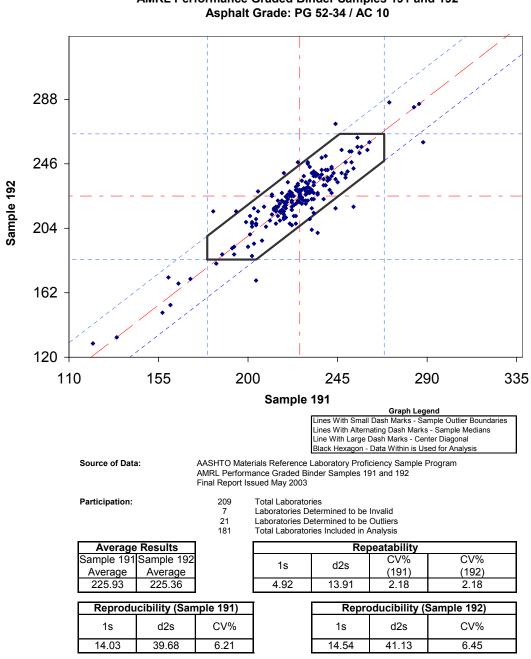
Graph and Analysis Results for AASHTO T313 / D6648 (Stiffness) Determining the Flexural Creep Stiffness of Asphalt Binder Using the Bending Beam Rheometer AMRL Performance Graded Binder Samples 185 and 186 Asphalt Grade: PG 64-22 / AC 20



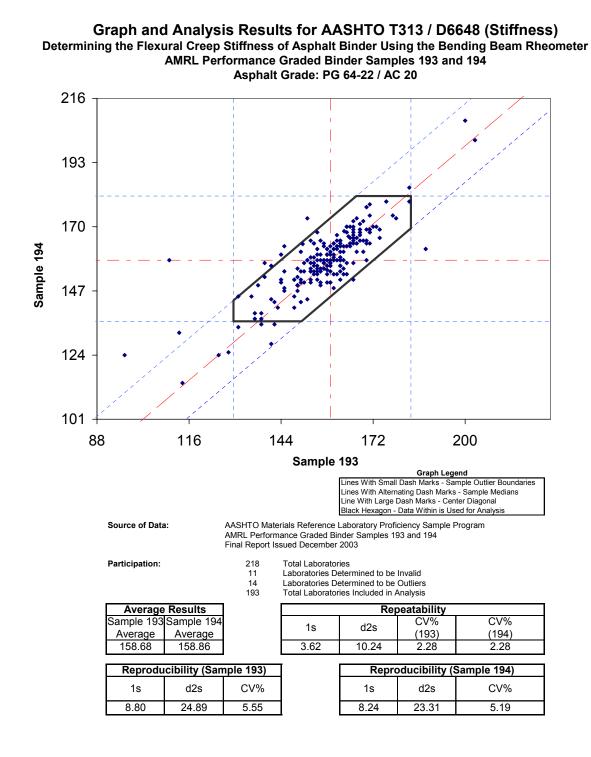
Graph and Analysis Results for AASHTO T313 / D6648 (Stiffness) Determining the Flexural Creep Stiffness of Asphalt Binder Using the Bending Beam Rheometer

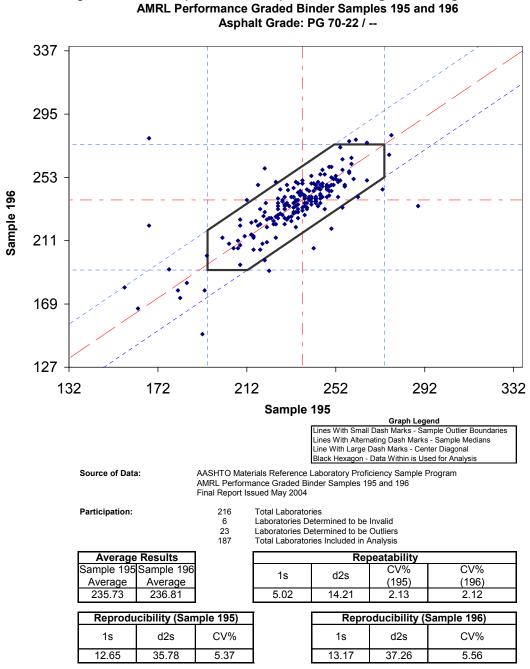


Graph and Analysis Results for AASHTO T313 / D6648 (Stiffness) Determining the Flexural Creep Stiffness of Asphalt Binder Using the Bending Beam Rheometer AMRL Performance Graded Binder Samples 189 and 190 Asphalt Grade: PG 64-22 / AC 30



Graph and Analysis Results for AASHTO T313 / D6648 (Stiffness) Determining the Flexural Creep Stiffness of Asphalt Binder Using the Bending Beam Rheometer AMRL Performance Graded Binder Samples 191 and 192 Asphalt Grade: PG 52-34 / AC 10

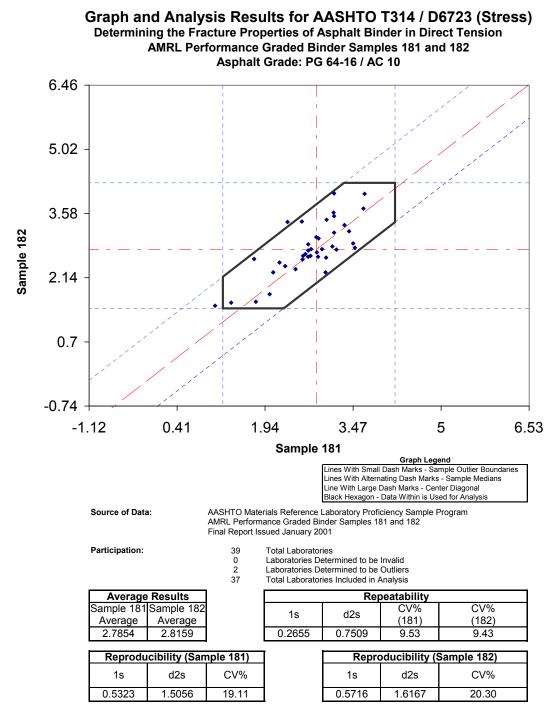


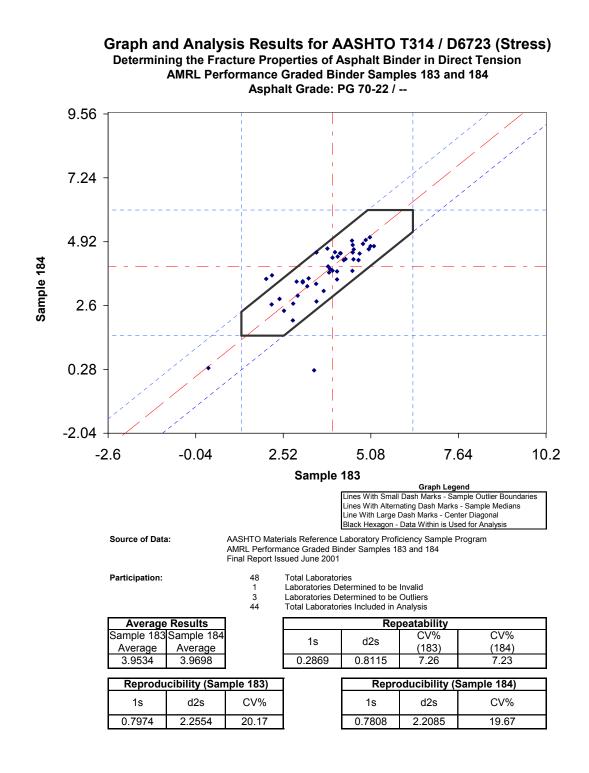


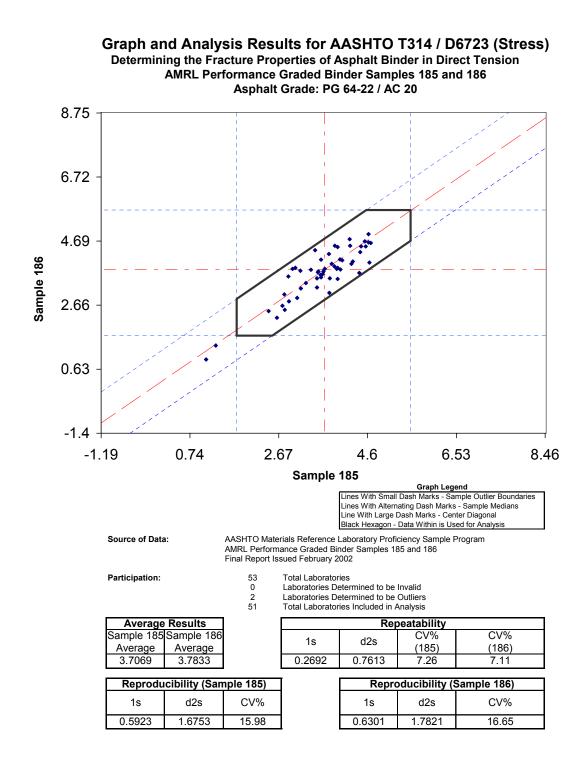
Graph and Analysis Results for AASHTO T313 / D6648 (Stiffness) Determining the Flexural Creep Stiffness of Asphalt Binder Using the Bending Beam Rheometer

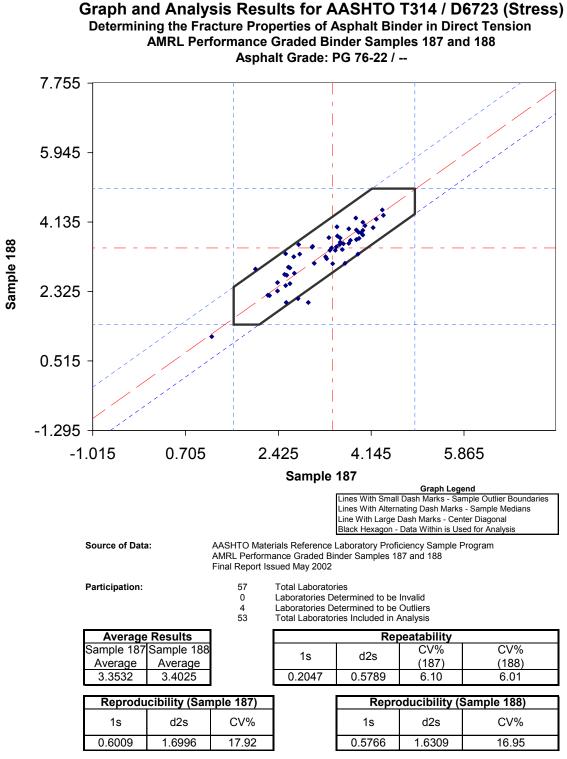
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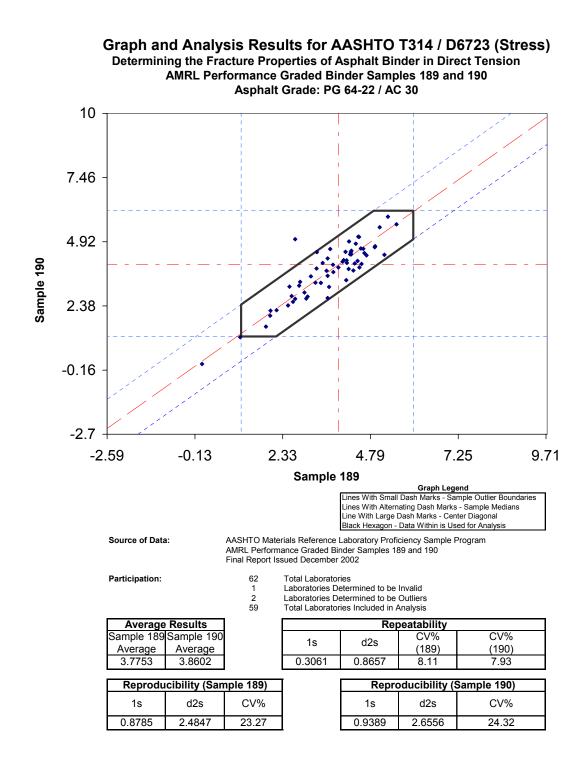
APPENDIX K

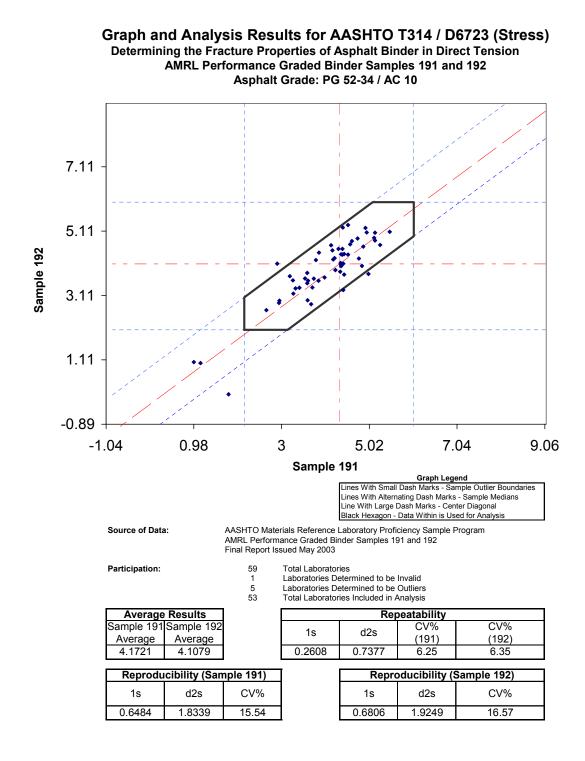


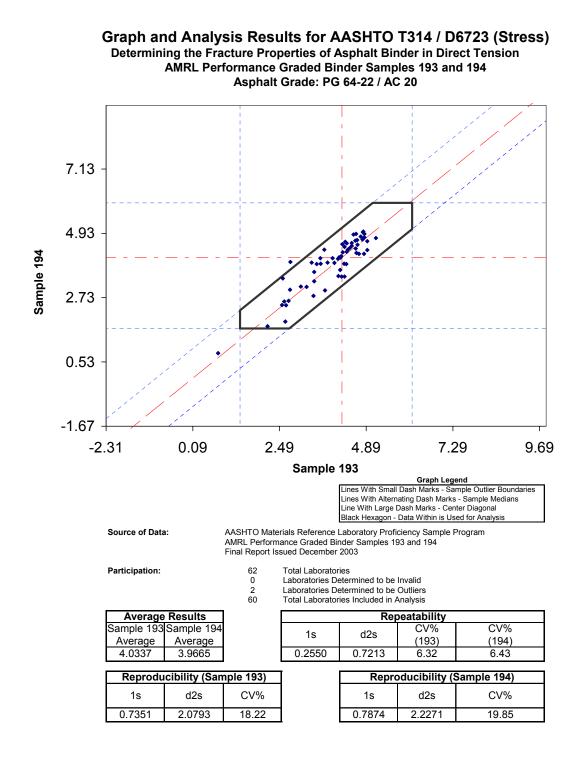


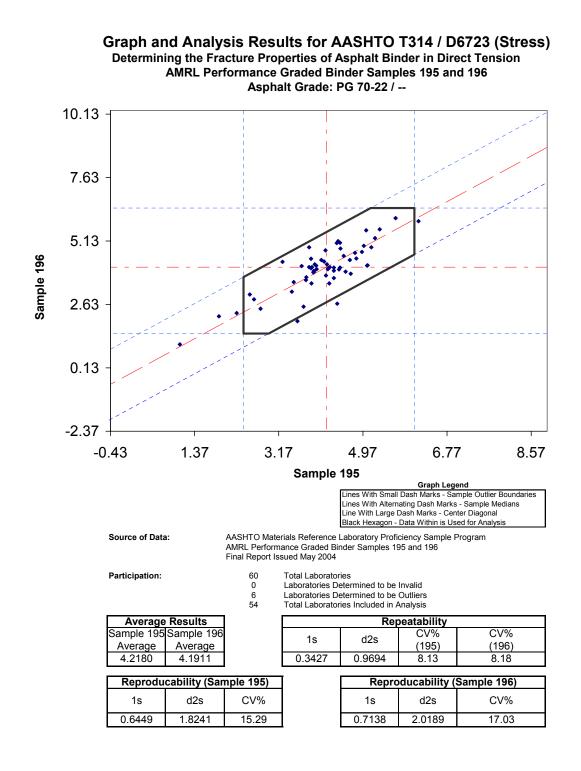




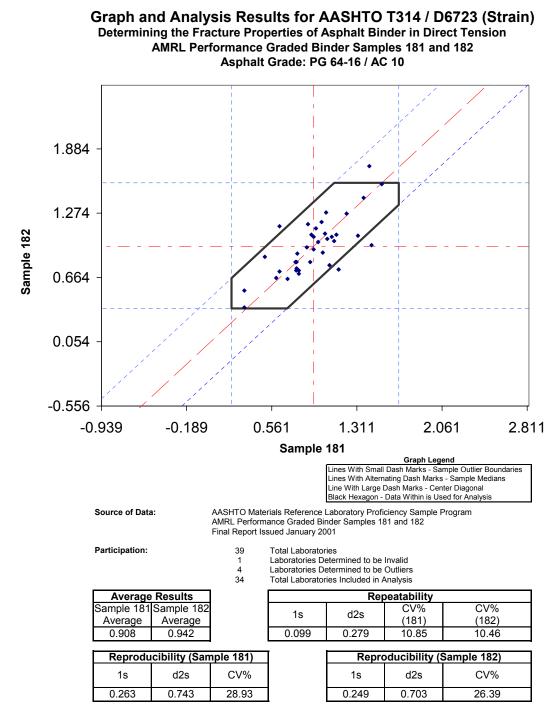


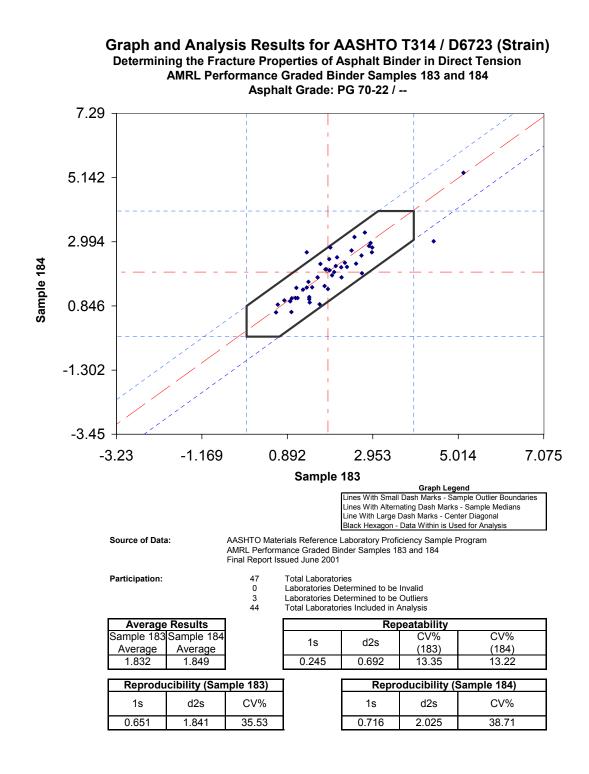


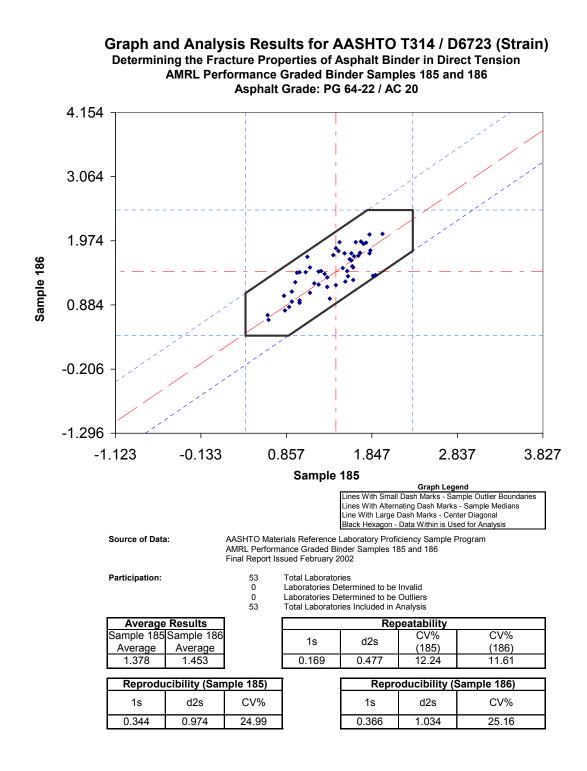


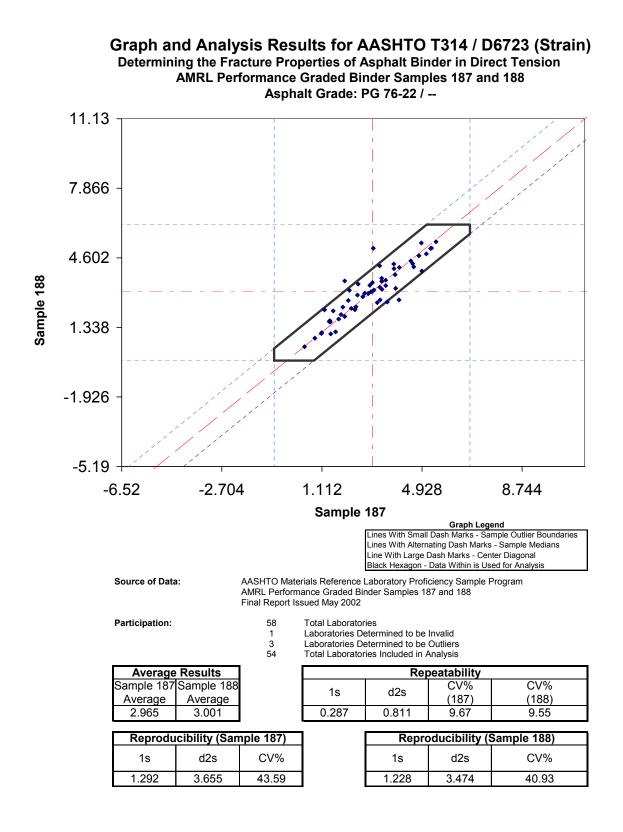


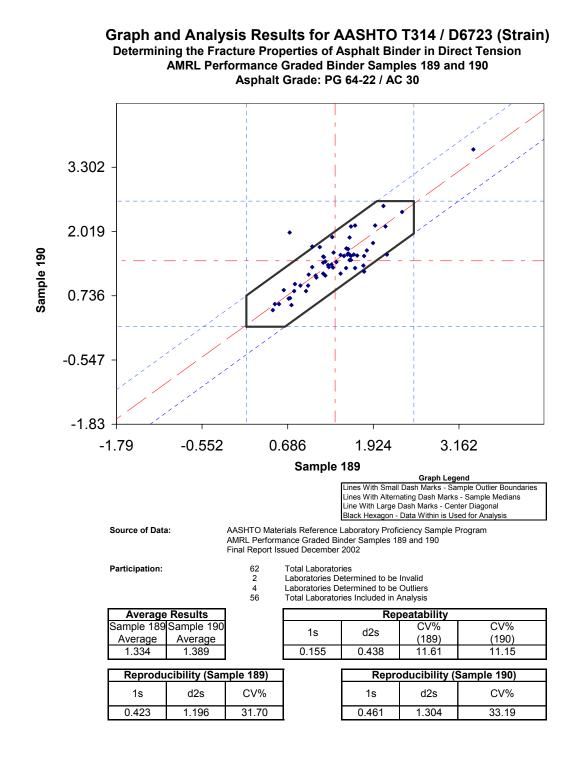
APPENDIX L

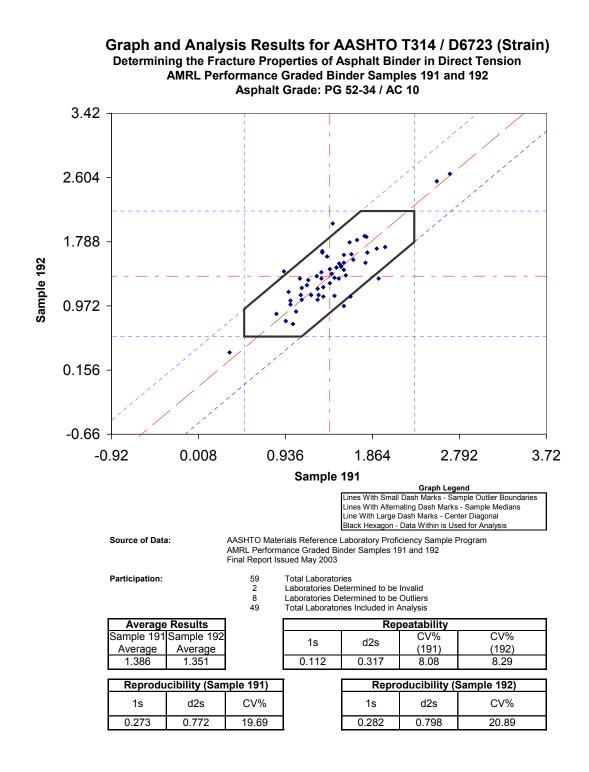


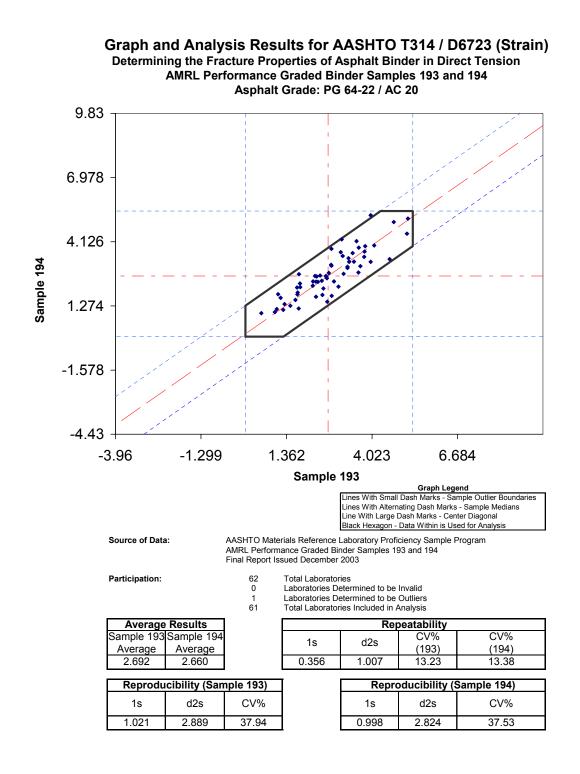


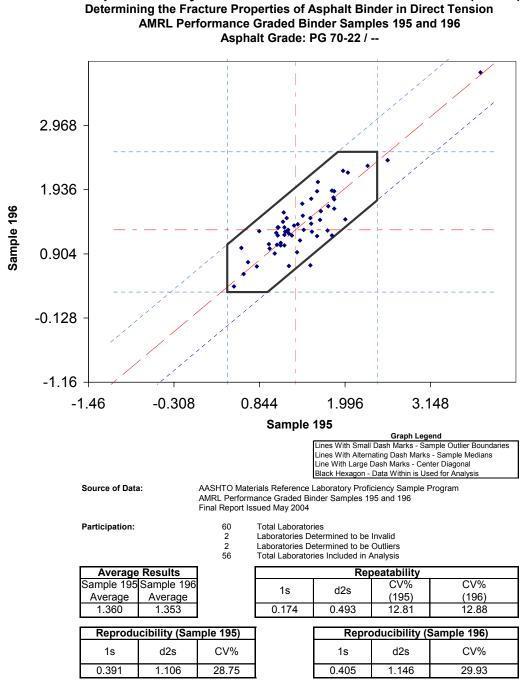






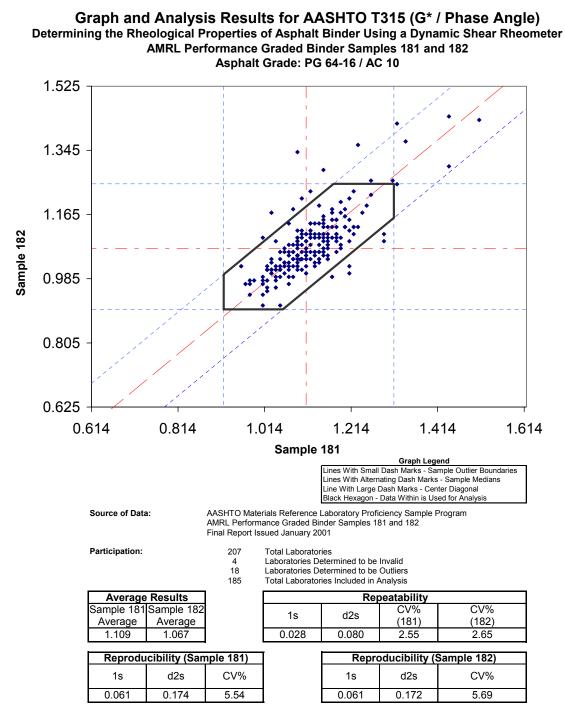


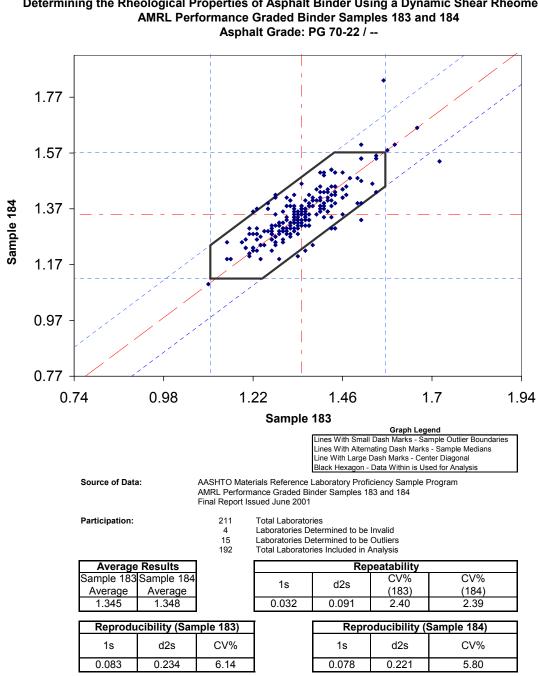




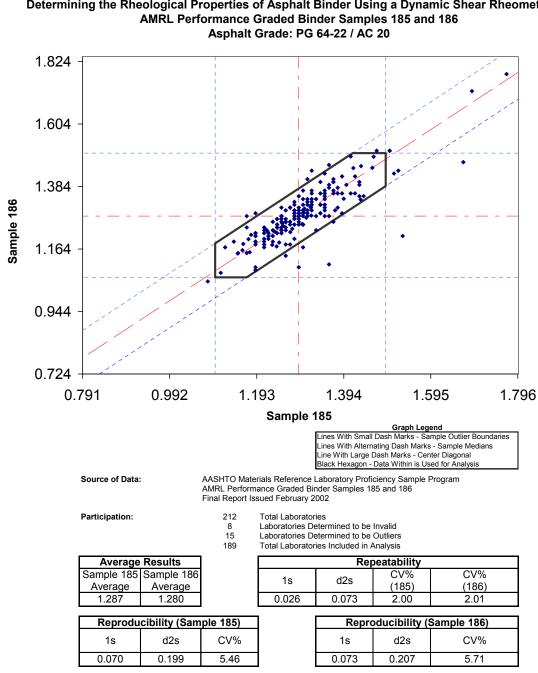
Graph and Analysis Results for AASHTO T314 / D6723 (Strain)

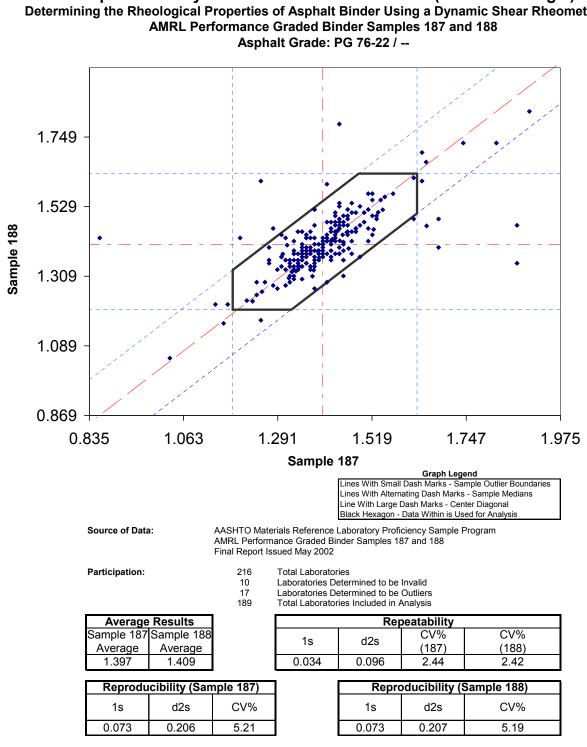
APPENDIX M

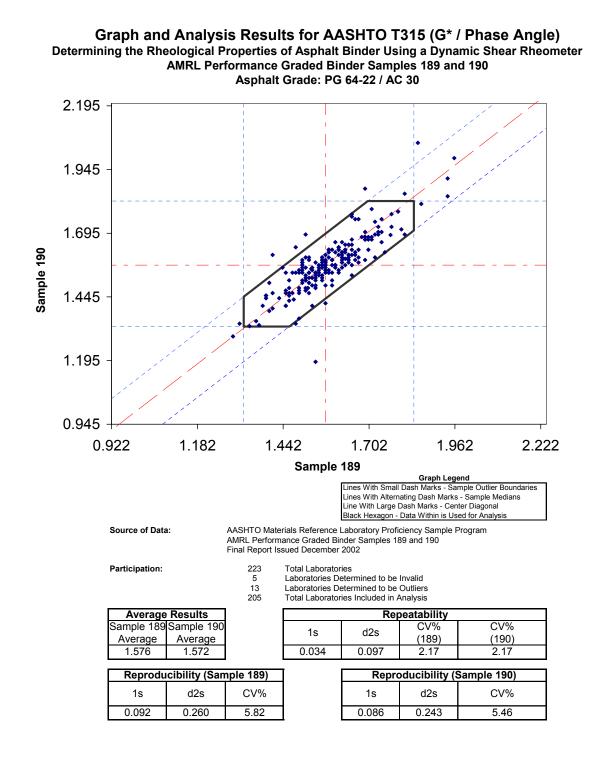


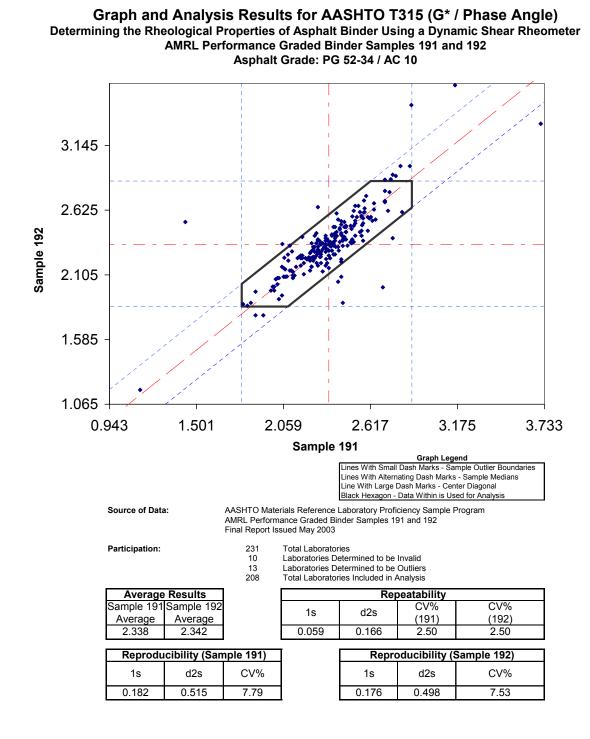


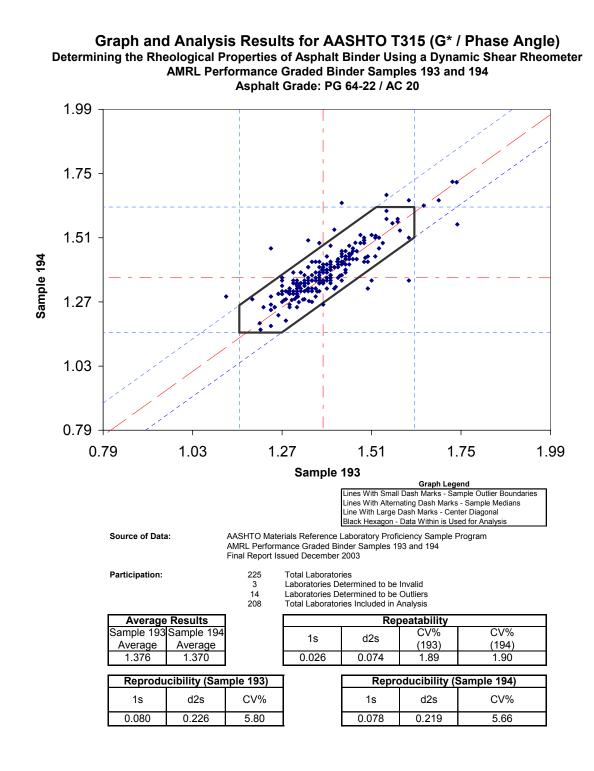
Graph and Analysis Results for AASHTO T315 (G* / Phase Angle) Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer

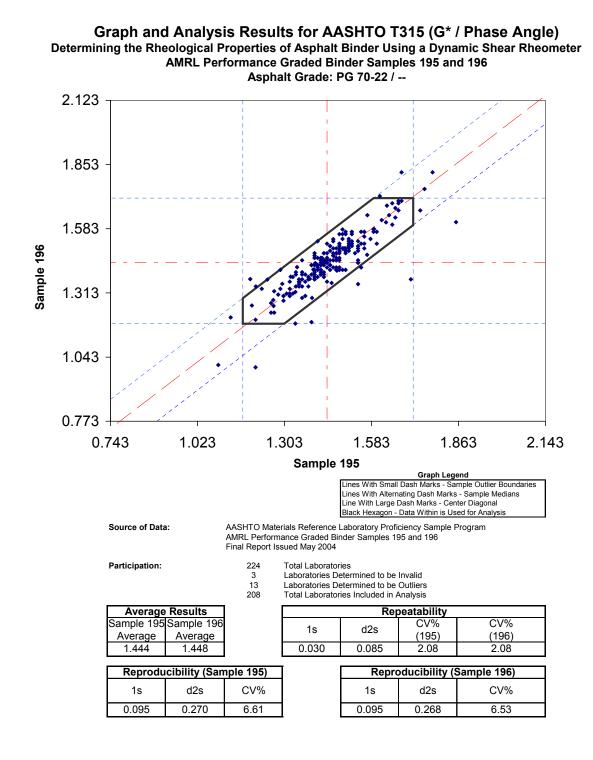




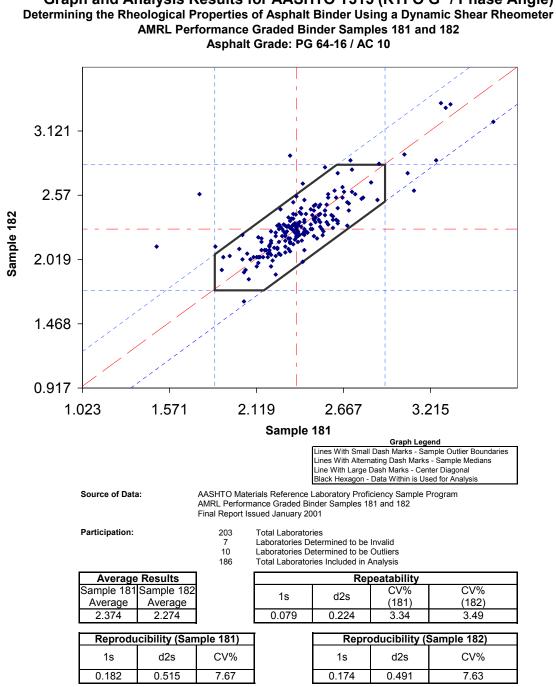




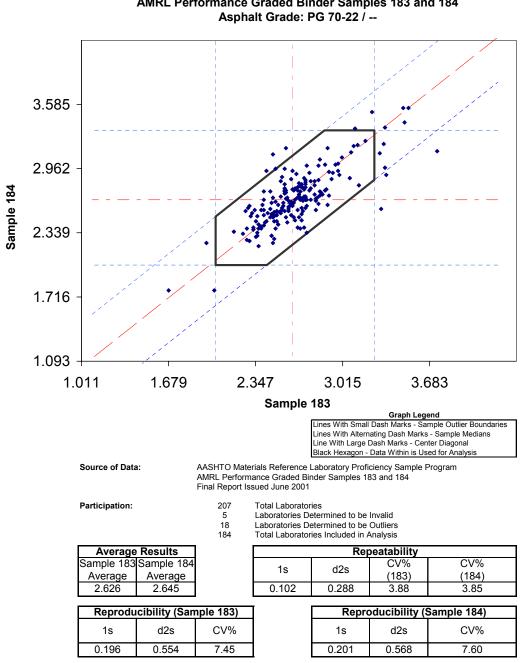




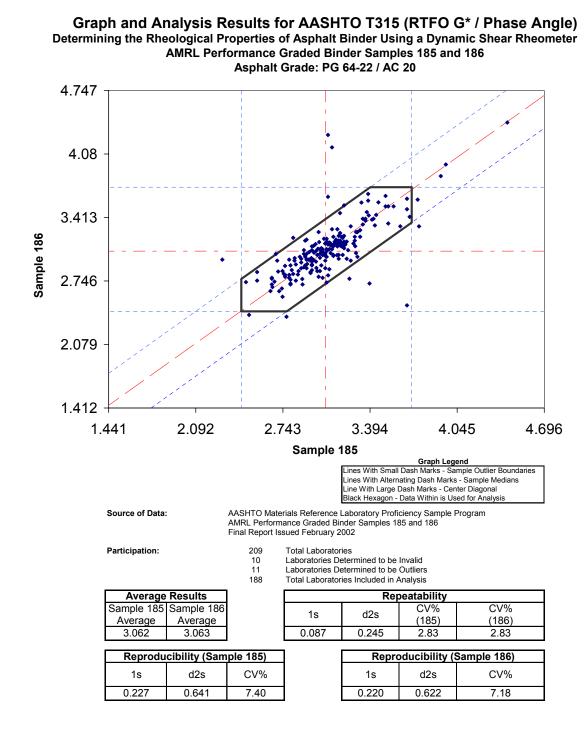
APPENDIX N

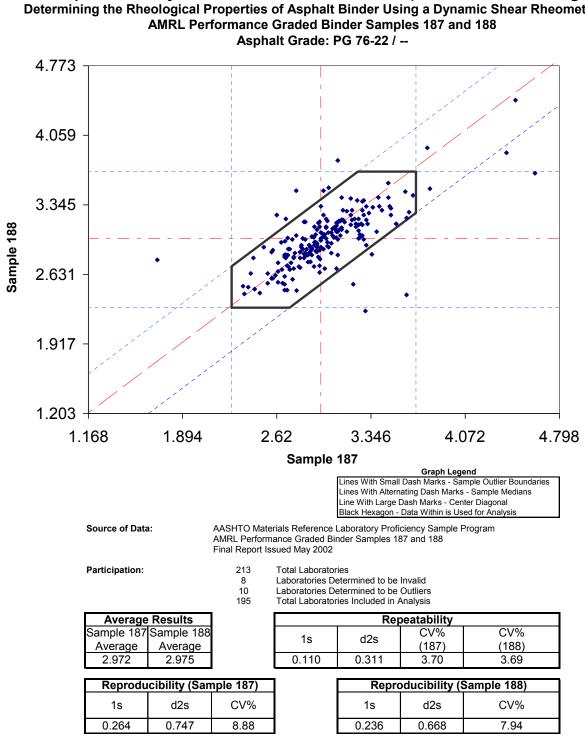


Graph and Analysis Results for AASHTO T315 (RTFO G* / Phase Angle)

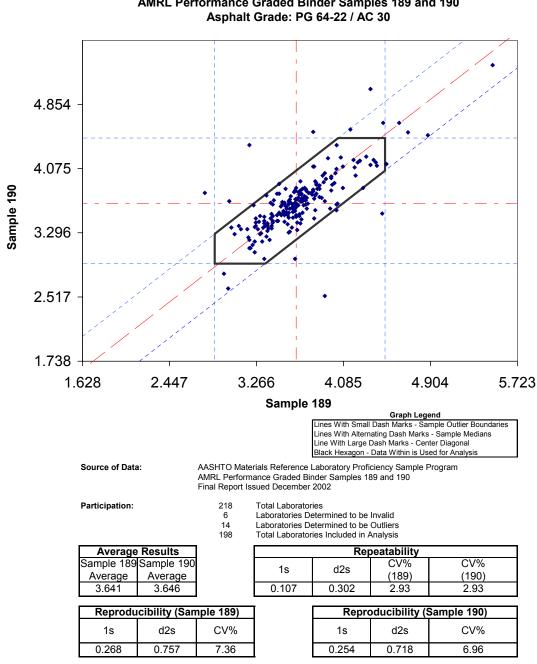


Graph and Analysis Results for AASHTO T315 (RTFO G* / Phase Angle) Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer AMRL Performance Graded Binder Samples 183 and 184 Asphalt Grade: PG 70-22 / --

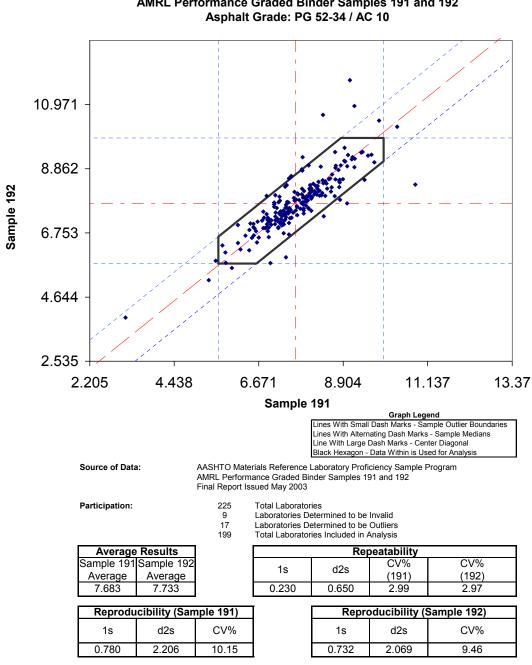




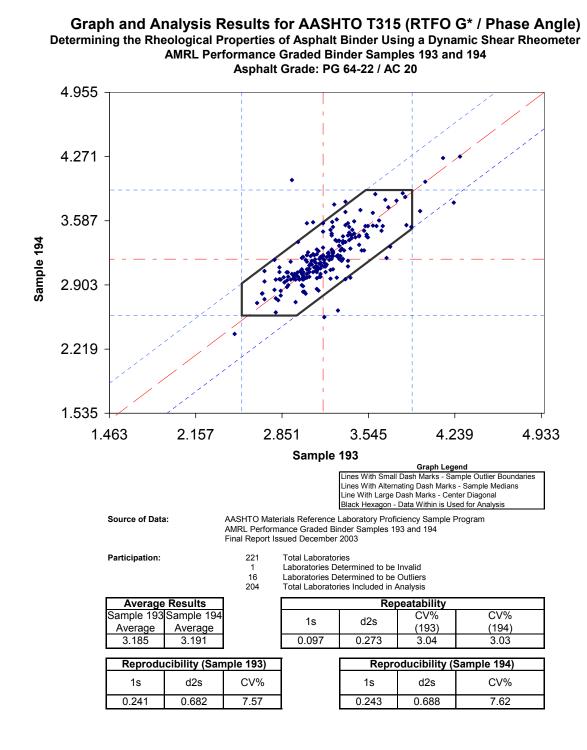
Graph and Analysis Results for AASHTO T315 (RTFO G* / Phase Angle) Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer

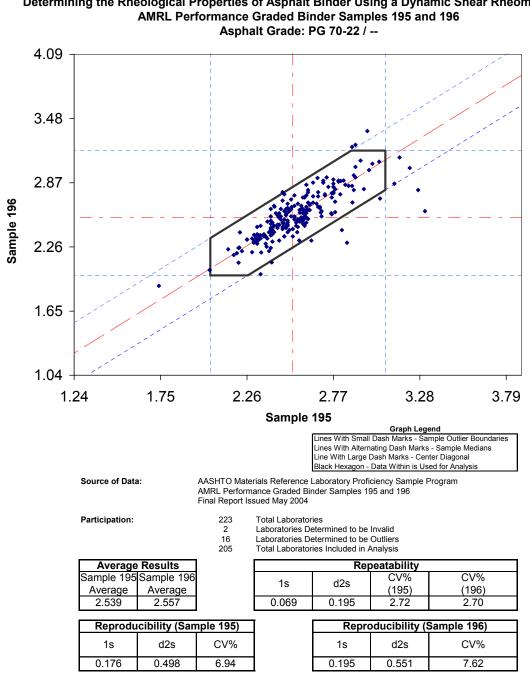


Graph and Analysis Results for AASHTO T315 (RTFO G* / Phase Angle) Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer AMRL Performance Graded Binder Samples 189 and 190 Asphalt Grade: PG 64-22 / AC 30



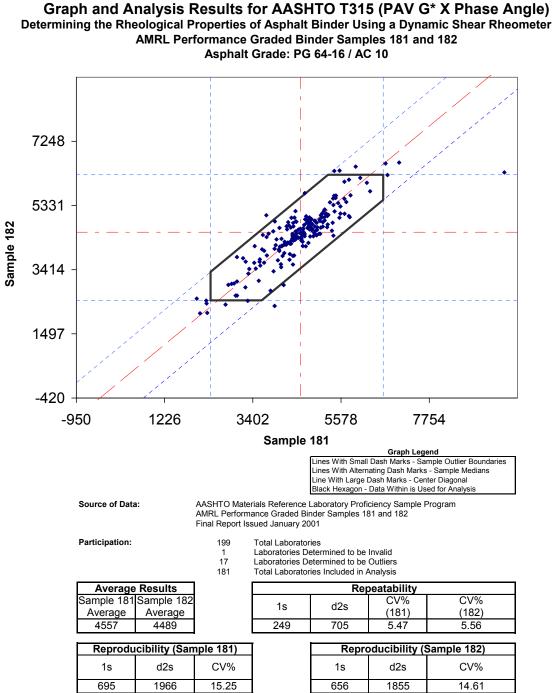
Graph and Analysis Results for AASHTO T315 (RTFO G* / Phase Angle) Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer AMRL Performance Graded Binder Samples 191 and 192 Asphalt Grade: PG 52-34 / AC 10

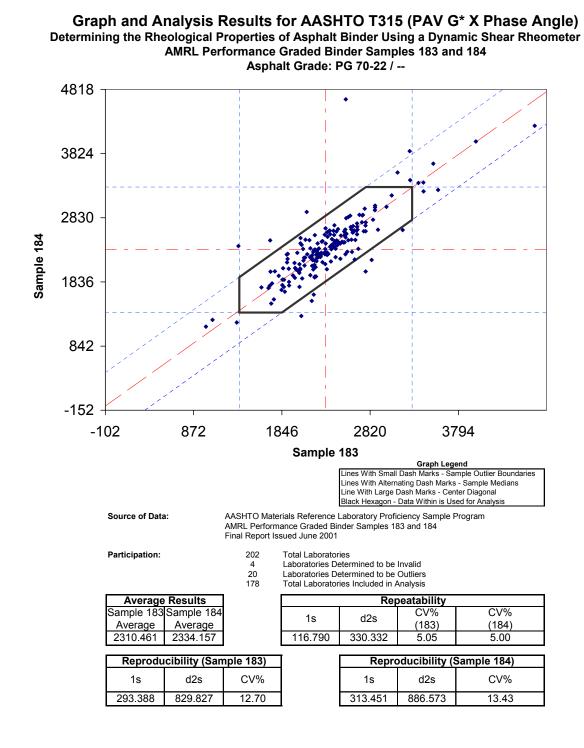


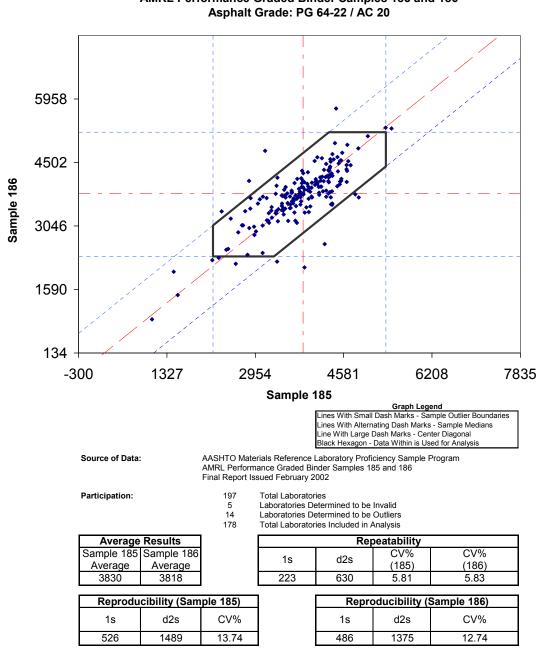


Graph and Analysis Results for AASHTO T315 (RTFO G* / Phase Angle) Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer

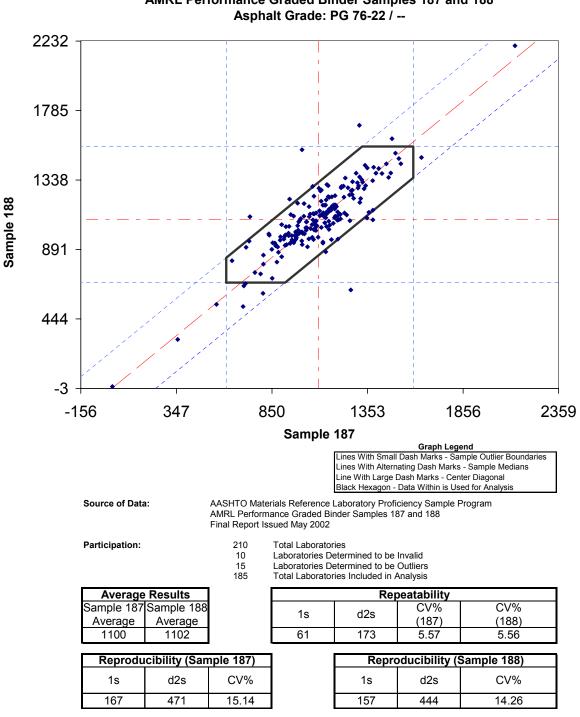
APPENDIX O



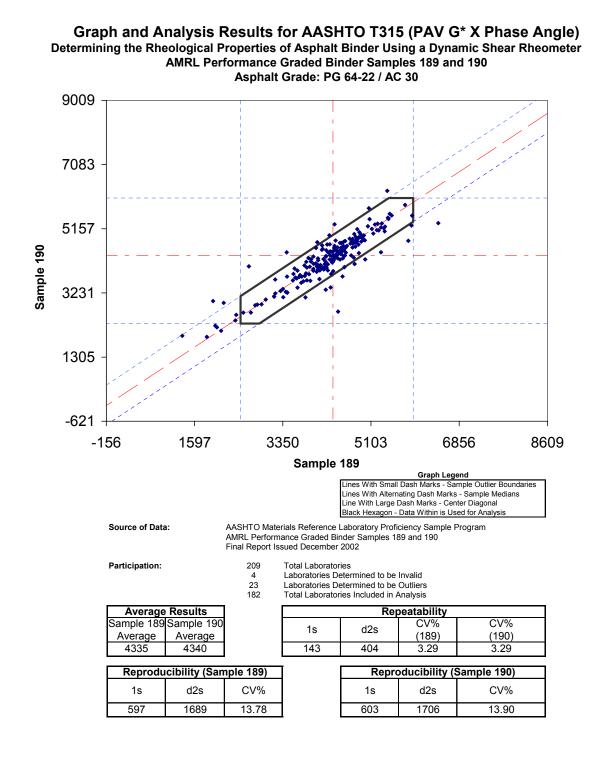


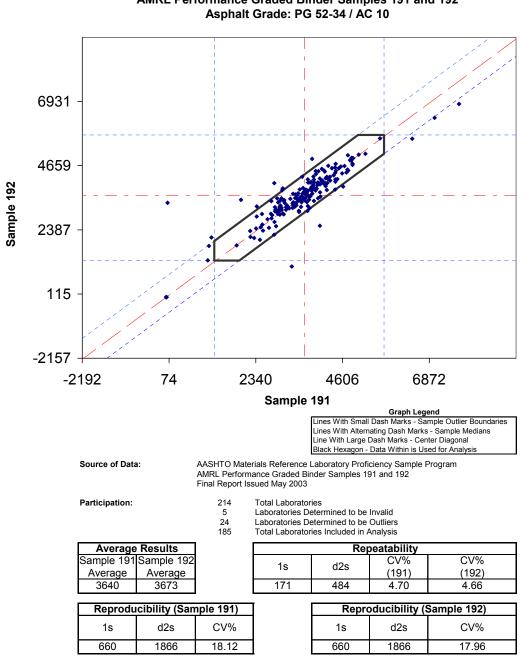


Graph and Analysis Results for AASHTO T315 (PAV G* X Phase Angle) Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer AMRL Performance Graded Binder Samples 185 and 186 Asphalt Grade: PG 64-22 / AC 20

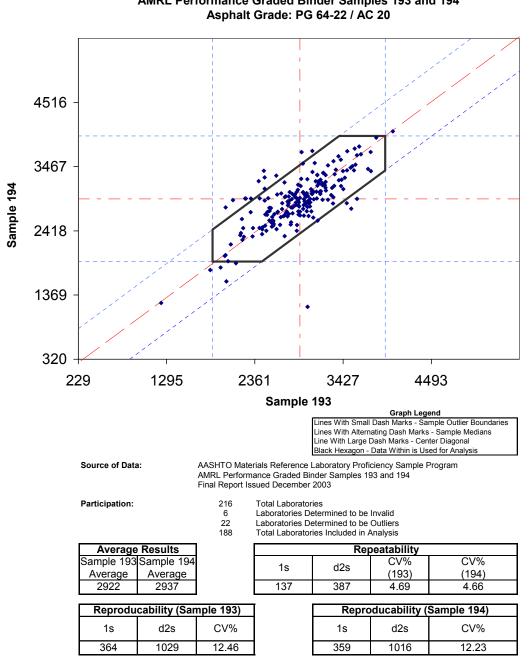


Graph and Analysis Results for AASHTO T315 (PAV G* X Phase Angle) Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer AMRL Performance Graded Binder Samples 187 and 188 Asphalt Grade: PG 76-22 / --

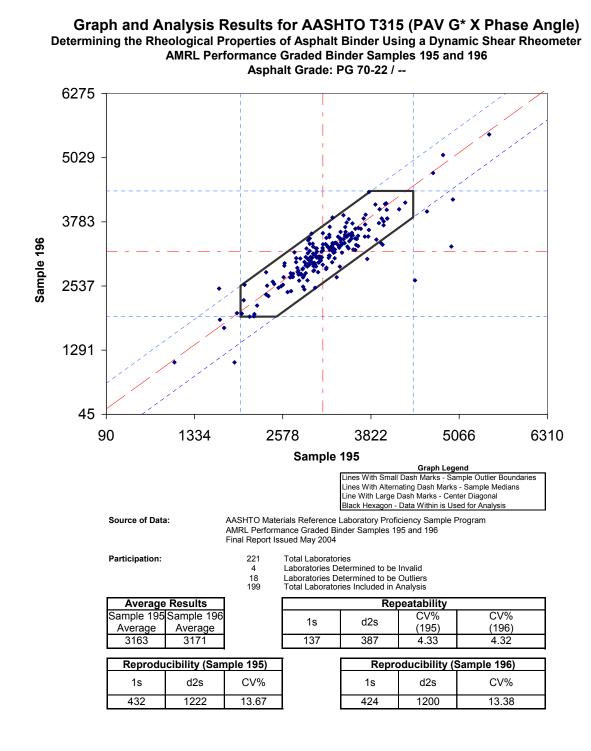




Graph and Analysis Results for AASHTO T315 (PAV G* X Phase Angle) Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer AMRL Performance Graded Binder Samples 191 and 192 Asphalt Grade: PG 52-34 / AC 10



Graph and Analysis Results for AASHTO T315 (PAV G* X Phase Angle) Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer AMRL Performance Graded Binder Samples 193 and 194 Asphalt Grade: PG 64-22 / AC 20



APPENDIX P

