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16. Abstract

The current performance grading (PG) specification for asphalt binders was developed based on the Strategic Highway Research Program (SHRP) and is based primarily on the study of unmodified asphalt binders. Over the years, experience has proven that the PG grading system, while good for ensuring overall quality, fails in some cases to predict rutting and cracking performance, particularly as it applies to softer but highly modified binders. Therefore, it is necessary to improve current asphalt binder performance parameters and associated specification limits, especially for modified binders.

This report documents the laboratory evaluation of several new tests for rutting, fatigue, and adhesion property of asphalt binders, including the multiple stress creep and recovery (MSCR) test, linear amplitude sweep (LAS) test, double edged notched tension (DENT) test, elastic recovery test, pull-off test, pneumatic adhesion tensile testing instrument (PATTI), dynamic mechanical analyzer (DMA) test, and surface energy test. It was found that the MSCR test and associated specification works better than the current G*/sin δ-based PG specification, especially for those highly modified asphalt binders (such as PG64-34). MSCR round robin results among five laboratories clearly indicated that both $J_{nr0.1}$ and $J_{nr3.2}$ results are very repeatable and reproducible, but both J_{nrdiff} and $R_{0.1}$ have pretty high variability. Since J_{nrdiff} is one of the parameters for grading asphalt binder, caution should be exercised when applying the MSCR specification. The $R_{3.2}$ results are acceptable in terms of repeatability and reproducibility. Based on the laboratory test results, users should exercise some caution when grading the slightly modified asphalt binders (such as PG64-28) using the MSCR test and associated specification. This study further confirms the poor relationship between the parameter G*sinδ and the binder fatigue resistance. Neither the MSCR nor the elastic recovery test shows good correlation with the asphalt mix OT cracking test. Both the LAST and the DENT tests provide similar ranking to that of asphalt mix OT cracking test. The DSR-based LAS test is recommended for asphalt binder fracture test, since the DSR has been widely used in the last 20 years. Additionally, only the PATTI test is a promising test for evaluating adhesive properties of asphalt binders. All three other tests (the pull-off test, DMA, and surface energy test) were not successful in this study for evaluating asphalt binder adhesion property.

Obviously, these laboratory findings need further field validation. Additionally, one needs always to keep in mind that the binder alone does not determine field performance of asphalt mixes. Mix characteristics as well as the pavement structure itself, traffic, and the environment within which it is located have a significant role in determining pavement performance.

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by

Fujie Zhou, Ph.D., P.E.
Research Engineer
Texas A&M Transportation Institute

Hongsheng Li
Research Associate
Texas A&M Transportation Institute

Peiru Chen
Research Associate
Texas A&M Transportation Institute

and

Tom Scullion
Senior Research Engineer
Texas A&M Transportation Institute

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TEXAS A&M TRANSPORTATION INSTITUTE
College Station, Texas 77843-3135

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CHAPTER 1
INTRODUCTION

The current performance grading (PG) specification for asphalt binders was developed based on the Strategic Highway Research Program (SHRP) and is based primarily on the study of unmodified asphalt binders. Over the years, experience has proven that the PG grading system, while good for ensuring overall quality, fails in some cases to predict rutting and cracking performance, particularly as it applies to modified binders (such as those with polymers or other types of modifiers). Specifically, recent studies on mixes from out of state (Minnesota) found that the mixes from Minnesota's Cold Weather Road Research Facility (MnRoad) have substantially improved cold weather cracking properties than mixes currently used in Texas. while these mixes still pass Hamburg rutting requirements, in spite of the binders being much softer, according to typical PG measurement, than would be used on Texas roads, according to typical PG measurement. To eliminate this type of apparent discrepancy on softer but highly modified binders, a new Multiple Stress Creep and Recovery (MSCR) test-based specification was proposed and has been adopted by the American Association of State Highway and Transportation Officials (AASHTO). It is believed that using the MSCR test which directly measures the permanent deformation properties of asphalt binder and the new specification, softer but highly modified binders will be allowed. However, these claims have not been verified with tests on Texas mixtures. Additionally, another weakness of the PG grading system is that it has no test method or criteria to screen out the premature adhesive or moisture damage failures of asphalt binder-aggregates. Therefore, it is necessary to improve current asphalt binder performance parameters and associated specification limits, especially for modified binders. Specifically, the major objectives of research project 0-6674 are:

- Determine if the new AASHTO MSCR-based binder grading system is superior to the current TxDOT system.
- Identify/develop a simple test method or methods to characterize fracture and adhesive properties of modified and unmodified asphalt binders and associated tentative specification limits.
- Determine if asphalt binders not currently used in Texas would potentially improve overlay performance and conduct an associated cost-benefit analysis.
- Identify optimal asphalt binder/aggregate combinations for different environmental zones in Texas.
- Develop and initially populate a catalogue of all these measured (binder, binder/fine aggregate mastic, and asphalt mix) properties with relevant information that can be used to track the field performance of pavements constructed using these asphalt binders.

This report documents the laboratory work focused on part of the first two objectives.

ORGANIZATION OF THE REPORT

This report is composed of five chapters. Following this introduction (Chapter 1), Chapter 2 describes the laboratory evaluation of MSCR test repeatability and its correlation with asphalt mix rutting properties. Chapter 3 focuses on identifying a simple fracture test for asphalt binder fatigue properties. The asphalt binder adhesion tests are discussed in Chapter 4. Finally, Chapter 5 provides a summary of findings and conclusions of this project.

CHAPTER 2
LABORATORY EVALUATION OF MSCR TEST: REPEATABILITY AND CORRELATION WITH ASPHALT MIX RUTTING TEST

INTRODUCTION

The Superpave PG binder specification, AASHTO M320 (*1*) is now used binders in Texas and other States to buy and sell paving asphalt binders. The current high temperature PG specification for asphalt binders uses the stiffness-based G*/sinδ as the rutting parameter, and the basic assumption is that a stiffer binder is rut resistant. However, the inadequacy of the current specification parameter, G*/sin δ, to correctly grade the superior field performance of modified asphalt binders has been demonstrated by several researchers (*2*, *3, 4*). To address this issue, researchers have explored several different rheological properties to replace the existing PG high temperature specification parameter G*/sinδ. These properties include repeated load creep and recovery testing, Zero Shear Viscosity (ZSV), and low frequency dynamic viscosity, η'. The relationship to asphalt mix rutting, ease of testing, and repeatability of each procedure have also been evaluated (*5-10*). After years of development, debate, and discussion, AASHTO finally adopted the MSCR test to characterize asphalt binder rutting resistance. However, TxDOT still has two major concerns with the MSCR test: its repeatability and correlation with asphalt mix rutting performance. Additionally, one may wonder about the ranking difference between the MSCR specification and the G*/sinδ specification. This chapter will address these two concerns and related issues.

BACKGROUND MSCR TEST

The MSCR test is based on the repeated creep and recovery test which was recommended as a high temperature parameter under the National Cooperative Highway Research Program (NCHRP) project 9-10, "*Characterization of Polymer Modified Binder in Superpave Mix Design*" (*11*). In this test a Dynamic Shear Rheometer (DSR) is used to apply a constant stress to a sample of asphalt binder for 1 second. After the load is removed the sample is then allowed to relax for 9 seconds. The test, as run in the NCHRP 9-10 work, is repeated for 50 to 100 cycles. The creep test has been used for many years to evaluate the creep-recovery of a material. This test closely models the loading associated with traffic passing over a spot in the roadway. For this reason the 9-10 researchers felt it should be the experimental procedure for evaluating asphalt binders. In the NCHRP 9-10 project, the repeated creep and recovery test was performed at only one creep stress level of 300 Pa repeated for 100 creep and recovery cycles. To determine the stress dependence of an asphalt binder, D' Angelo and Dongre (*12, 13, 14*) extended the NCHRP 9-10 repeated creep and recovery test and modified it by using increasing stress levels and renamed it the MSCR test. The idea behind the approach is to get the non-recovered compliance (J_{nr}-) as a measure of the high temperature specification as it relates to the binder contributions in the roadway permanent deformation (Figure 1). The test introduces stress sensitivity as well as a recoverable strain response and nonrecoverable response; that is, an alternative to elastic recovery specifications (ASTM D5976, AASHTO T-51).

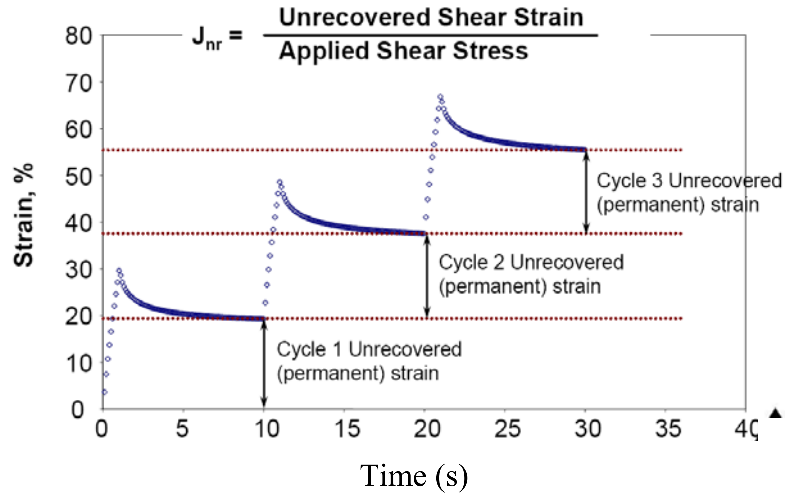


Figure 1. J_{nr} Definition.

Different versions of the MSCR test have been proposed. The latest one found in AASHTO TP 70 is summarized below:

> *Asphalt binder is first aged using T 240 (RTFO). A sample of the RTFO-aged asphalt is tested using T 315 (DSR). The 25-mm parallel plate geometry is used with a 1-mm gap setting. The sample is tested in creep at two stress levels followed by recovery at each stress level. The stress levels used are 0.1 kPa and 3.2 kPa. The creep portion of the test lasts for 1 s which is followed by a 9-s recovery. Ten creep and recovery cycles are tested at each stress level.*

Table 1 presents the comparison between the current PG specification and the new MSCR specification. When compared with current PG specification, one of the biggest changes in the MSCR specification is the way of adjusting traffic speed and volume. In the current PG grading system, traffic speed and volume are adjusted by “grade bumping” (or testing at higher temperatures than indicated by the climate); in the new MSCR grading system, traffic speed and volume are adjusted by changing the J_{nr} value but keeping the same test temperature as indicated by the climate. For example, a PG76-22 binder would be graded at 76°C under the current specification, but the same PG76-22 binder would be graded at 64°C when used in a scenario where the highest pavement temperature (i.e., determined using LTPPBind 3.1) is 64°C. To adjust traffic speed and volume, the new MSCR specification (Table 2) grades the binder as PG64-22S, PG64-22H, PG64-22V, or PG64-22E, depending on the measured J_{nr} value.

In terms of test method and rutting parameter, the MSCR system is better, at least in concept, than the current PG system because the MSCR system is established on repeated shearing load test and permanent deformation (strain), which better simulates what is happening in the field. However, whether or not it is better in reality than the current system needs to be verified in the laboratory and the field test sections, especially for Texas mixes. Additionally, the variability of MSCR has not been well defined so far. Bahia (*15*) recently reported that MSCR has very high variability. Therefore, the precision of MSCR test needs to be defined through a round robin test under this study.

In summary, the issues and concerns about the MSCR test are its repeatability and precision and the correlation with the asphalt mix rutting test, which are addressed in this chapter.

Table 1. Major Differences between G*/sinδ and MSCR-J_{nr} Grading Systems.

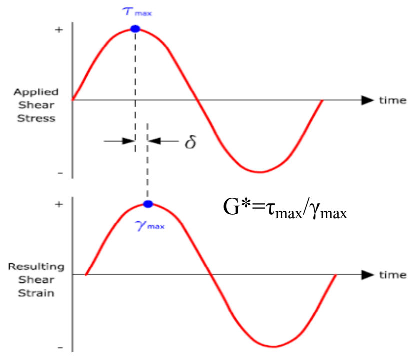
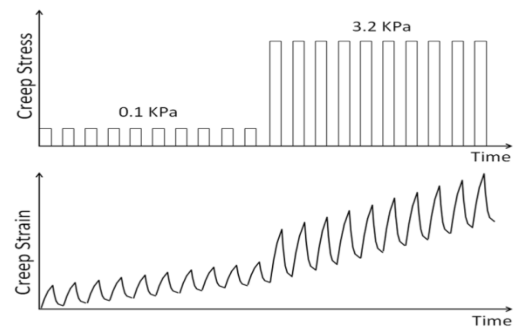
Item	Current PG Specification	MSCR-Based Specification
Test method	AASHTO T-315, stiffness test 	AASHTO TP 70, repeated shearing load test 
Rutting parameter	G*/sinδ, stiffness-based	J_{nr}, permanent strain-based
Adjust for traffic speed and volume	"grade bumping" or testing at higher temperatures than indicated by the climate	Testing at the same temperature as indicated by the climate, but adjusting required J_{nr} maximum value
Variability	Well defined and known	Not well defined (or unknown)

Table 2. Summary of New High Temperature PG Binder Specification for PG64-XX Grade.

Design Traffic Level (ESALs, Millions)	New PG Designation	Original binder (AASHTO T315@64°C)	RTFO Aged Binder MSCR (AASHTO TP70)@64°C	
			$J_{nr3.2}$(kPa^{-1})	%J_{nrdiff}
Standard (<10 m)	PG64-22S	G*/Sin δ >1.0 kPa	≤ 4.0	≤ 75 %
Heavy (10-30 m)	PG64-22H	G*/Sin δ >1.0 kPa	≤ 2.0	≤ 75 %
Very Heavy (>30 m)	PG64-22V	G*/Sin δ >1.0 kPa	≤ 1.0	≤ 75 %
Extreme (>30 m+ standing traffic)	PG64-22E	G*/Sin δ >1.0 kPa	≤ 0.5	≤ 75 %

REPEATABILITY AND PRECISION OF THE MSCR TEST

In order to evaluate the repeatability and reproducibility of the MSCR test, TTI conducted an inter-laboratory study (or Round Robin test) in six independent laboratories following *ASTM E691-11 Standard Practice for Conducting an Inter-laboratory Study to Determine the Precision of a Test Method* (*16*). A brief description of ASTM E691-11 is presented in Appendix A. The purpose of the inter-laboratory study was to develop information needed for a precision statement of the MSCR test. In each laboratory the same certified technician performed the test using the same equipment. Under this study, two TTI labs, the TxDOT central lab at Cedar Park, UT-A, UT-SA, and an FHWA lab participated in the round robin test, as listed in Table 3.

Table 3. Participating Labs for MSCR Inter-Laboratory Study.

Lab No.	Lab Name
1	TTI-Kinexus
2	TTI-DSRII
3	UT-Austin
4	TxDOT C11374406
5	FHWA
6	UT-San Antonio

MSCR Inter-Laboratory Test: Binders, Test Results, and Analysis

Selected Asphalt Binders

Three asphalt binders, PG58-28, PG64-22, and PG70-34, were used for the inter-laboratory Round Robin test (Table 4). PG64-22 and PG58-28 binders are often used in Texas. PG70-34 binder is a highly modified binder with a very good elastic-recovery property. It is believed that these three significantly different binders are good representatives of asphalt binders for checking the repeatability and reproducibility of the MSCR test.

TTI researchers aged all three asphalt binders through a rolling thin film oven (RTFO) test and then shipped them to each participating lab for the MSCR test following AASHTO TP 70. The test temperature used for all the three binders was 64°C, which is a reasonable test temperature for Texas hot weather.

Table 4. Asphalt Binders.

Binder PG	PG58-28	PG64-22	PG70-34
Binder Number	A	B	C

Test Results

The reported test results include J_{nr} @ 0.1 kPa shear stress ($J_{nr0.1}$), Recovery @ 0.1 kPa shear stress ($R_{0.1}$), Jnr @ 3.2 kPa shear stress ($J_{nr3.2}$), Recovery @ 3.2 kPa shear stress ($R_{3.2}$), and Stress Sensitivity (J_{nrDiff}). Detailed test results from each laboratory are presented in Table 5.

Table 5. Inter-Laboratory MSCR Test Results.

Lab	$J_{nr0.1}$ (kPa-1)			$J_{nr3.2}$ (kPa-1)			J_{nrdiff} (%)		
	Binder A	Binder B	Binder C	Binder A	Binder B	Binder C	Binder A	Binder B	Binder C
1	4.26	1.23	0.13	5.39	1.40	0.15	26.4	13.7	11.7
	4.20	1.23	0.13	5.13	1.46	0.14	22.1	18.7	6.8
	4.19	1.23	0.13	5.24	1.42	0.14	24.9	15.7	9.4
	4.12	1.15	0.13	5.11	1.33	0.13	23.9	15.4	1.0
	4.22	1.16	0.13	5.27	1.42	0.13	24.9	22.1	1.6
	4.00	1.16	0.13	5.10	1.36	0.14	27.6	16.7	2.7
	4.02	1.20	0.13	5.21	1.37	0.14	29.4	13.9	6.8
2	4.03	1.26	0.14	4.84	1.42	0.13	20.1	12.5	-1.6
	4.09	1.28	0.14	4.97	1.45	0.15	21.5	13.0	1.2
	4.11	1.30	0.15	4.95	1.45	0.15	20.2	11.3	-0.2
	4.11	1.25	0.14	5.01	1.41	0.14	21.8	12.4	-3.0
	4.12	1.25	0.14	4.98	1.41	0.14	20.7	12.1	2.2
	4.13	1.21	0.14	5.19	1.36	0.13	25.4	12.1	-5.6
	4.25	1.27	0.12	5.13	1.43	0.14	20.6	12.3	12.7
3	3.82	1.23	0.12	4.57	1.37	0.12	19.7	11.4	1.2
	3.76	1.16	0.11	4.54	1.29	0.11	20.7	11.0	0.8
	3.75	1.20	0.10	4.52	1.34	0.10	20.8	11.4	1.4
	3.87	1.19	0.12	4.59	1.32	0.12	18.6	10.6	1.6
	3.61	1.15	0.12	4.35	1.29	0.13	20.5	11.9	3.3
	3.79	1.17	0.12	4.60	1.33	0.12	21.3	13.3	2.4
	3.76	1.19	0.12	4.55	1.32	0.12	20.9	10.7	0.5
4	4.27	1.24	0.13	5.17	1.41	0.15	21.2	13.8	11.8
	4.07	1.36	0.14	4.94	1.55	0.14	21.3	13.9	5.7
	4.52	1.25	0.14	5.47	1.42	0.15	21.1	13.2	5.2
	4.25	1.26	0.15	5.17	1.43	0.16	21.6	14.0	6.7
	4.15	1.30	0.15	5.01	1.48	0.17	20.6	13.9	10.4
	4.08	1.29	0.14	4.99	1.47	0.15	22.3	13.3	6.7
	4.03	1.29	0.13	4.92	1.46	0.15	22.0	13.6	10.5
5	4.40	1.30	0.13	5.40	1.40	0.14	23.9	15.2	10.5
	4.40	1.30	0.14	5.50	1.50	0.14	22.9	13.9	-2.8
	4.20	1.30	0.14	5.20	1.40	0.15	23.3	14.3	8.3
	4.20	1.30	0.13	5.20	1.50	0.14	21.8	13.7	10.8
	4.20	1.30	0.13	5.10	1.40	0.14	21.7	13.9	3.7
	4.00	1.30	0.13	4.90	1.50	0.14	21.5	13.2	8.1
	4.10	1.30	0.15	5.00	1.50	0.14	22.8	15.9	-5.7
6	4.20	1.05	0.09	5.25	1.46	0.08	25.0	39.4	11.0
	3.95	1.25	0.12	5.29	1.54	0.11	33.8	23.6	14.2
	2.49	1.24	0.12	3.19	1.43	0.10	28.2	15.1	10.3
	2.31	0.99	0.12	2.92	1.41	0.11	26.5	42.1	9.3
	2.20	0.71	0.12	2.73	0.78	0.09	24.2	9.8	19.2
	2.44	0.71	0.12	3.06	0.79	0.10	25.6	10.3	13.9
	4.10	1.25	0.12	5.39	1.48	0.11	31.4	18.4	11.6

Table 5. Inter-Laboratory MSCR Test Results (Continued).

Laboratory	$R_{0.1}$ (%)			$R_{3.2}$ (%)		
	Binder A	**Binder B**	**Binder C**	**Binder A**	**Binder**	**Binder C**
1	6.5	11.4	89.5	1.1	4.4	88.6
	6.9	12.0	89.3	1.3	4.2	88.6
	6.7	12.2	89.5	1.3	4.0	88.9
	6.9	11.3	89.0	1.4	5.1	88.9
	6.7	14.5	88.8	1.3	5.1	88.9
	7.6	13.2	88.5	1.3	5.2	88.5
	10.7	13.1	87.9	1.2	5.2	87.6
2	7.1	12.0	89.4	1.4	4.9	89.6
	7.8	11.3	89.1	1.4	4.7	88.9
	7.2	11.5	89.3	1.4	4.9	89.1
	8.0	11.1	89.2	1.4	5.0	89.7
	7.8	12.2	89.2	1.3	5.0	89.2
	9.1	12.4	89.2	1.4	5.2	89.8
	6.5	11.4	90.8	1.3	4.9	89.6
3	7.9	12.0	90.7	1.5	5.2	90.8
	7.9	12.2	90.7	1.5	5.4	90.8
	8.0	12.3	91.1	1.6	5.3	91.2
	6.5	11.7	90.7	1.5	5.3	90.7
	8.2	12.9	90.1	1.7	5.5	90.0
	8.4	13.4	90.5	1.5	5.3	90.5
	8.4	11.9	90.4	1.5	5.4	90.5
4	5.8	10.6	89.6	1.3	4.9	88.9
	6.2	10.0	89.6	1.4	4.3	89.1
	5.5	10.4	89.2	1.2	4.8	88.9
	6.0	10.5	89.0	1.3	4.7	88.4
	5.5	10.3	89.2	1.3	4.6	88.2
	7.0	9.7	89.4	1.3	4.6	88.8
	6.5	9.8	89.6	1.4	4.6	89.2
5	7.0	11.5	89.9	1.3	4.6	89.0
	6.5	11.1	88.6	1.2	4.5	89.1
	7.0	11.4	89.3	1.3	4.7	88.7
	6.9	10.8	89.8	1.4	4.5	88.9
	6.9	11.4	89.3	1.3	4.6	89.1
	7.3	10.8	89.6	1.5	4.5	89.0
	7.3	11.6	88.1	1.4	4.5	89.0
6	4.6	15.4	90.0	-2.7	1.8	91.2
	8.7	10.4	89.0	-2.6	1.4	90.6
	8.0	9.2	90.1	-1.0	1.8	91.2
	8.8	18.0	89.2	-0.6	2.0	90.3
	9.8	13.1	88.7	-0.3	6.8	90.9
	8.0	12.9	89.8	-0.8	6.6	91.2
	7.0	10.9	89.5	-2.6	1.8	90.7

Data Analysis

Following *ASTM E691-11*, the round robin test results were analyzed for each asphalt binder to develop information needed for a precision statement on MSCR test. The main tasks of this phase were to: 1) determine whether the collected data were consistent enough to form the basis for a test method precision statement, 2) investigate and act on any data considered inconsistent, and 3) obtain the precision statistics on which the precision statement can be based.

Consistency verification of the test results is important because the presence of outliers may lead to invalidation of the analysis. A simple one-way analysis of variance can check data consistency. For ease of analyzing the data, the results are represented in the form of a table where each row contains data from one laboratory for three binders and the columns contains the data obtained from all laboratories for a certain parameter. The data are then divided into cell statistics, intermediate statistics, precision statistics, and consistency statistics, as described in the following paragraphs. Note that Lab6 data were seriously deviated from others. Thus, Lab 6 was removed from the analysis. The test results for Labs 1-5 were used to conduct the inter-laboratory study analysis.

All detailed data analyses are presented in Appendix B. As an example, Table 6 presents the J_{nr100} data. The values for Repeatability Standard Deviation (s_r) and Reproducibility Standard Deviation (s_R) are used to determine the 1s, 1s%, d2s, and d2s% values for repeatability (within-lab) and reproducibility (between-lab), respectively. A quality review of the data was initiated to ensure that there were no obvious errors that would require removal of some data points from the participating laboratories. Although there were some reporting errors, the errors were corrected before conducting the analysis.

Table 6. Initial Preparation of Jnr0.1 Test Results for Binder A.

Lab	Test Results, x							$\bar{x}$	s	d	h	k
	1	2	3	4	5	6	7					
1	4.263	4.201	4.191	4.124	4.218	3.998	4.023	4.14543	0.10119	0.08797	0.45	0.91
2	4.026	4.095	4.114	4.115	4.124	4.135	4.251	4.12276	0.06689	0.06531	0.33	0.60
3	3.816	3.764	3.746	3.870	3.607	3.789	3.760	3.76464	0.08136	-0.29281	-1.48	0.73
4	4.265	4.073	4.522	4.254	4.154	4.076	4.035	4.19699	0.16931	0.13953	0.71	1.51
5	4.400	4.400	4.200	4.200	4.200	4.000	4.100	4.21429	0.14639	0.12546	0.68	1.22
	$\bar{\bar{x}}$	$s_{\bar{x}}$	s_r	s_R								
	4.089	0.185	0.120	0.216								

Data Consistency Check

Tables of the "h" and "k" consistency statistics were created for each of the five parameters; Table 7 provides an example. The "h"value can also be seen in Figures 2 and 3. The data in the consistency tables can be used with graphs to identify whether inconsistent data should be eliminated when determining the precision estimates. In the above case, none of the "h" values exceeds the critical value, however the "h" values of binders A and C for Lab 3 are close to the critical value. Figure 1 shows that most of the "h" values for Labs1, 2, and 4 are positive, while they are negative for Lab3, which means all Lab3 results are lower than all the other labs (Figure

3). Due to the limitation of participating lab numbers, none of those data were removed from this analysis.

Table 7. Consistency Statistic "h" for $J_{nr0.1}$[A].

Laboratory	Binder A	Binder B	Binder C
1	0.31	-0.96	-0.13
2	0.18	0.32	0.62
3	-1.75	-1.15	-1.67
4	0.58	0.73	0.84
5	0.68	1.06	0.33

Note: A critical value=1.74.

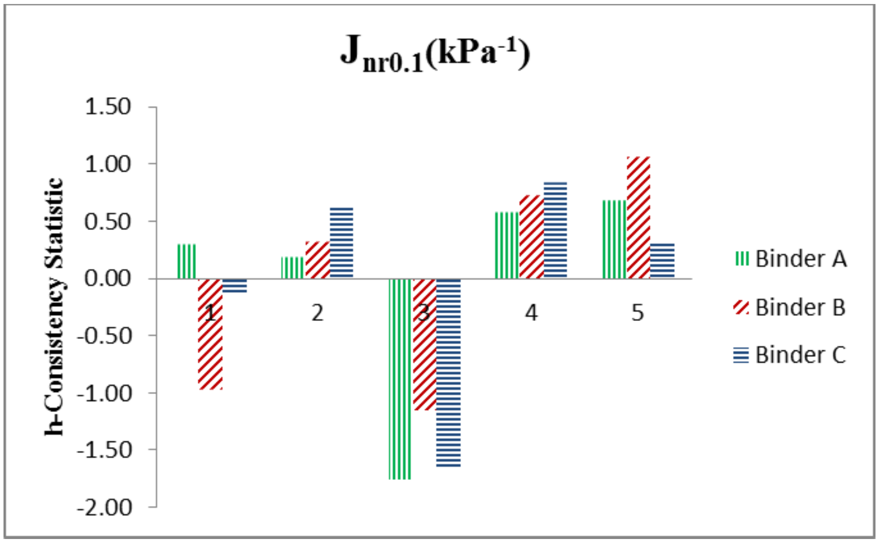


Figure 2. h Value for $J_{nr0.1}$ – Materials within Laboratories.

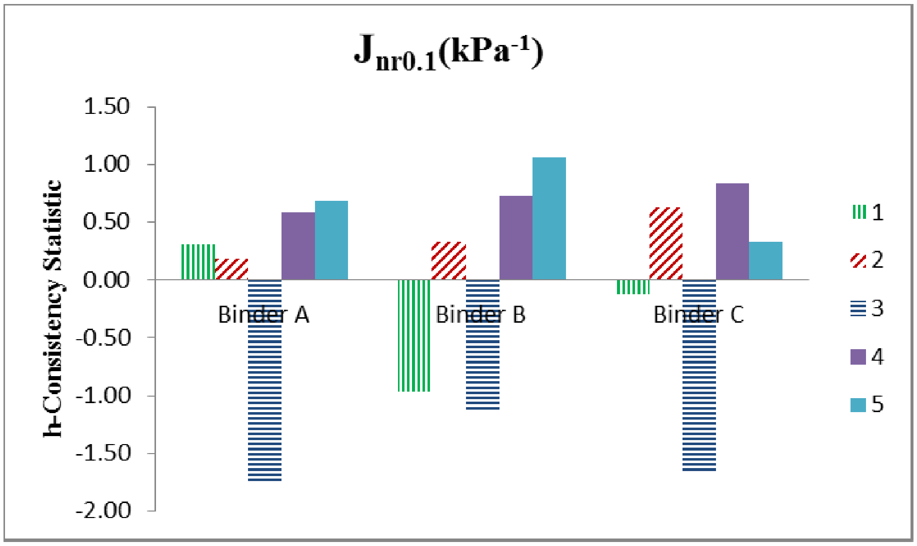


Figure 3. h Value for $J_{nr0.1}$ – Laboratories within Materials.

Repeatability and Reproducibility

After examining all the test results, the repeatability and reproducibility were calculated based on all the data collected from Lab1-Lab5. The calculated results for $J_{nr0.1}$, $J_{nr3.2}$, J_{nrdiff}, $R_{0.1}$, and $R_{3.2}$, are listed in Tables 8-12, respectively.

Table 8. Repeatability and Reproducibility Calculations for $J_{nr0.1}$.

ID	Binder	$\bar{\bar{x}}$	$s_{\bar{x}}$	s_r	s_R	r	R	Repeatability		Reproducibility	
								1s%	d2s%	1s%	d2s%
A	PG58-28	4.08900	0.18499	0.11952	0.21556	0.33467	0.60358	2.9%	8.2%	5.3%	14.8%
B	PG64-22	1.24500	0.05179	0.02968	0.05863	0.08311	0.16417	2.4%	6.7%	4.7%	13.2%
C	PG70-34	0.13200	0.01100	0.00700	0.01200	0.01984	0.03497	5.4%	15.0%	9.4%	26.4%

Table 9. Repeatability and Reproducibility Calculations for $J_{nr3.2}$.

ID	Binder	$\bar{\bar{x}}$	$s_{\bar{x}}$	s_r	s_R	r	R	Repeatability		Reproducibility	
								1s%	d2s%	1s%	d2s%
A	PG58-28	5.00500	0.27600	0.15100	0.31000	0.42395	0.86771	3.0%	8.5%	6.2%	17.3%
B	PG64-22	1.40900	0.05629	0.04219	0.06852	0.11813	0.19185	3.0%	8.4%	4.9%	13.6%
C	PG70-34	0.13800	0.01300	0.00600	0.01400	0.01773	0.04039	4.6%	12.9%	10.5%	29.3%

Table 10. Repeatability and Reproducibility Calculations for J_{nrdiff}.

ID	Binder	$\bar{\bar{x}}$	$s_{\bar{x}}$	s_r	s_R	r	R	Repeatability		Reproducibility	
								1s%	d2s%	1s%	d2s%
A	PG58-28	22.29000	2.01000	1.50200	2.44400	4.20592	6.84414	6.7%	18.9%	11.0%	30.7%
B	PG64-22	13.65400	1.98230	1.47248	2.40582	4.12293	6.73630	10.8%	30.2%	17.6%	49.3%
C	PG70-34	4.18800	3.00900	4.52000	5.15400	12.65624	14.43167	107.9%	302.2%	123.1%	344.6%

Table 11. Repeatability and Reproducibility Calculations for $R_{0.1}$.

ID	Binder	$\bar{\bar{x}}$	$s_{\bar{x}}$	s_r	s_R	r	R	Repeatability		Reproducibility	
								1s%	d2s%	1s%	d2s%
A	PG58-28	7.19800	0.71900	0.85400	1.06900	2.39215	2.99333	11.9%	33.2%	14.9%	41.6%
B	PG64-22	11.59200	0.94653	0.65002	1.12164	1.82005	3.14060	5.6%	15.7%	9.7%	27.1%
C	PG70-34	89.51000	0.65100	0.51100	0.80500	1.42968	2.25290	0.6%	1.6%	0.9%	2.5%

Table 12. Repeatability and Reproducibility Calculations for R_{3200}.

ID	Binder	$\bar{\bar{x}}$	$s_{\bar{x}}$	s_r	s_R	r	R	Repeatability		Reproducibility	
								1s%	d2s%	1s%	d2s%
A	PG58-28	1.35900	0.10600	0.06900	0.12400	0.19448	0.34624	5.1%	14.3%	9.1%	25.5%
B	PG64-22	4.84200	0.30681	0.25932	0.38959	0.72611	1.09084	5.4%	15.0%	8.0%	22.5%
C	PG70-34	89.27500	0.81300	0.35100	0.87600	0.98345	2.45159	0.4%	1.1%	1.0%	2.7%

Note that in the tables above:

- "$\bar{x}$" represents the average value from the participating labs for each binder sample.
- "s_r" represents the within-lab (single operator) standard deviation, or 1s. Dividing this value by the average value generates the Repeatability coefficient of variation, or 1s%.

Multiplying s_r by 2.8 generates the acceptable range of two test results under repeatability conditions, or d2s. Dividing this product by the average value results in the d2s%.

- "s_R" represents the between-lab standard deviation, or 1s. Dividing this value by the average value generates the Reproducibility coefficient of variation, or 1s%. Multiplying s_R by 2.8 generates the acceptable range of two test results under reproducibility conditions, or d2s. Dividing this product by the average value results in the d2s%.

Summary of the MSCR Inter-Laboratory Test

Based on the test results presented above, the following conclusions are offered:

- Both $J_{nr0.1}$ and $J_{nr3.2}$ results are very repeatable and reproducible.
- Both J_{nrdiff} and $R_{0.1}$ have pretty high variability. As shown in Table 2, J_{nrdiff} is one of the parameters for grading asphalt binder. Therefore, cautions should be exercised when applying the MSCR specification.
- The $R_{3.2}$ results are acceptable in terms of repeatability and reproducibility.

ASPHALT BINDER RANKING BASED ON J_{NR} AND G*/SIN δ CRITERIA

Nine asphalt binders ranging from the softest (PG58-34) to the hardest (PG76-22) were selected for this comparison. Both the MSCR and PG grading tests were performed at 64°C, which is the temperature pavements experience in Texas, based on AASHTO TP70. Figure 4 shows the test results. It is clear that both J_{nr} and G*/sin δ criteria provide the same overall ranking from the most rutting resistant to the least: PG76-XX, PG70-XX, PG64-XX, and PG58-XX. The advantage of the MSCR test is to consider the influence of recoverable deformation on rutting resistance. For example, PG64-34 has better rutting performance than PG64-28, followed by PG64-22. The difference among the binders with the same high temperature PG cannot be identified by current G*/sin δ based specification. Therefore, J_{nr} is overall better than G*/sin δ. The MSCR test results also clearly showed the significant benefit of using softer, highly modified binder to improve asphalt binder rutting resistance. However, it is necessary to evaluate how much the improvement in asphalt binder can be transferred into the rutting resistance of asphalt mixes, because it is the asphalt mix (asphalt binder and aggregates) rather than asphalt binder alone that is paved on the road. The following section investigates the rutting resistance of these softer, modified binders in the asphalt mixes and the correlation between J_{nr} and asphalt mix rutting test results.

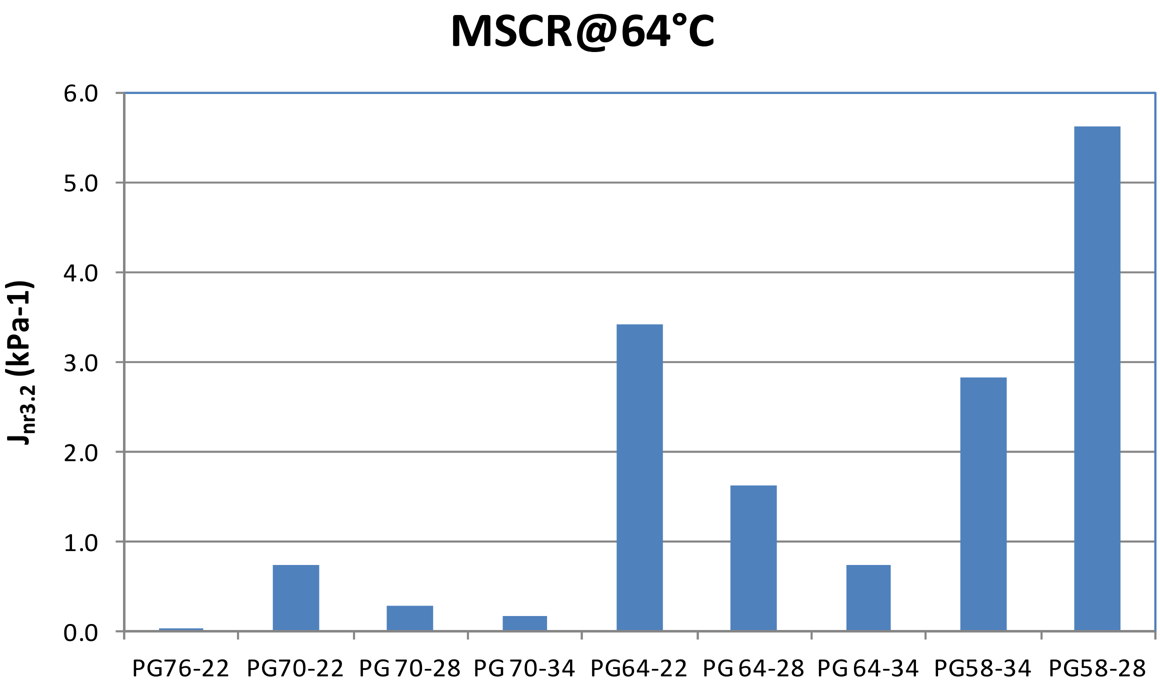


Figure 4. MSCR Test Results of Nine Asphalt Binders.

CORRELATION BETWEEN MSCR BINDER TEST AND ASPHALT MIX RUTTING TEST

The second concern discussed previously is the correlation between the MSCR binder test and the asphalt mix rutting test. Without a good correlation between the two types of tests, it is difficult for TxDOT to implement the MSCR test with confidence. Thus, the following sections will investigate whether or not there is a good correlation between the two types of tests.

Materials and Asphalt Mix Rutting Test

Five asphalt binders and three aggregates were selected for this study. The five binders are PG64-22, PG64-28, PG64-34, PG70-22, and PG76-22; the three aggregates include limestone, crushed gravels, and granite. A full factorial design with a total of 15 mixes was used for this study. For each aggregate type, the same asphalt binder content and gradation were used for all the mixes, and the only variable was asphalt binder type. Table 13 shows the optimum asphalt binder for each mix. The gradations of the mixes are shown in Figure 5.

Table 13. Optimum Asphalt Content of Each Mix.

Aggregates	PG64-22	PG64-28	PG64-34	PG70-22	PG76-22
Limestone (Type D)	4.8%				
Crushed Gravel (Type C)	4.6%				
Granite (Superpave D)	5.5%				

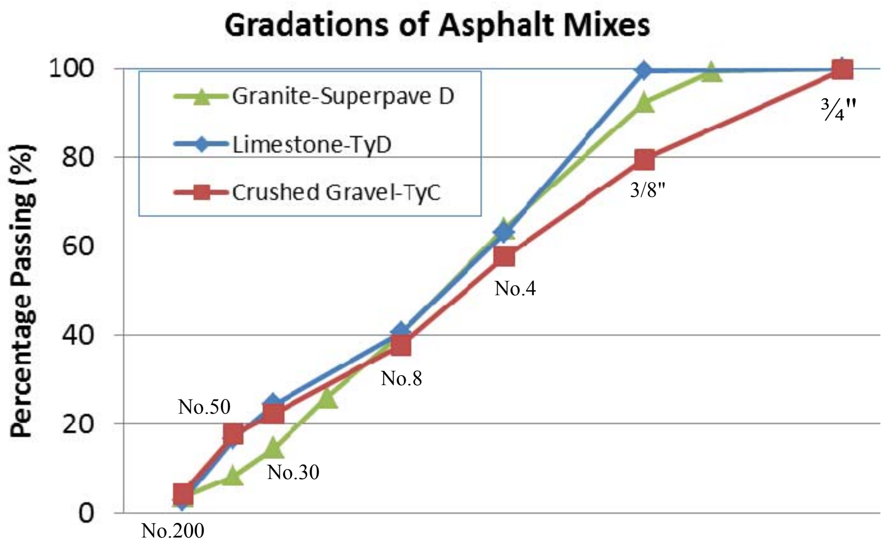


Figure 5. Aggregate Gradations of Mixes Used in This Study.

The Hamburg Wheel Tracking Test (HWTT) is the standard test for evaluating rutting resistance of asphalt mixes in Texas. So the HWTT was selected here to assess the validity of the MSCR binder test. The HWTT was conducted at a temperature of 50°C in accordance with TEX-242-F (*17*), *Test Procedure for Hamburg Wheel-Tracking Test (HWTT)*. A Superpave gyratory compactor was used to mold cylindrical specimens with a diameter of 150 mm and a height of 62 mm. A masonry saw was used to cut along the edge of the cylindrical specimens. The target air void of specimens was 7 percent ± 1 percent. To evaluate the rutting susceptibility and moisture resistance, specimens were submerged under water at a temperature of 50 °C during the test, and a linear variable differential transducer (LVDT) device measured deformations of specimens. The stop criterion was a rut depth of 12.5 mm or 20,000 passes.

Test Results and Analysis

Figures 6, 7, and 8 show the HWTT results of 15 mixes (5 binders and 3 types of aggregates). When examining the results, the following observations are made:

- First, the three PG64-XX binders, PG64-22, PG64-28, and PG64-34, have different rutting performance under the HWTT, regardless of aggregate types, although they have the same high temperature PG grade. The mixes with PG64-34 binder have superior rutting performance to those with either PG64-22 or PG64-28 binder. Therefore, the MSCR test and associated specification are better than the current G*/sin δ-based PG specification.
- The PG64-34 and PG70-22 binders that are graded as PG64-V showed similar performance in terms of rutting only when the stripping part of the HWTT curves are ignored. Additionally, the mixes with PG76-22 binder graded as PG64-E, regardless of aggregate types, had the least rut depth. Thus, the MSCR test and associated specification work as it should.

- There is not much difference in rutting performance between the mixes with PG64-22 and PG64-28 binders, which seems to prove that the current G*/sin δ-based PG specification works just fine.

In summary, it seems that the MSCR test and associated specification works better than the current G*/sin δ-based PG specification. However, some caution should be exercised when grading the slightly modified asphalt binders (such as PG64-28).

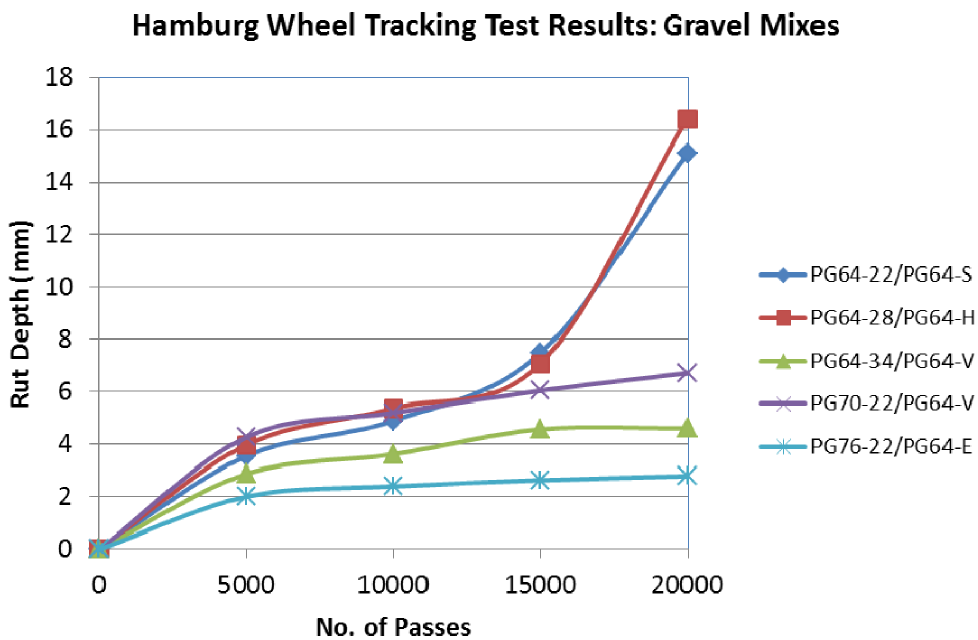


Figure 6. HWTT Results of Gravel Mixes with Five Binders.

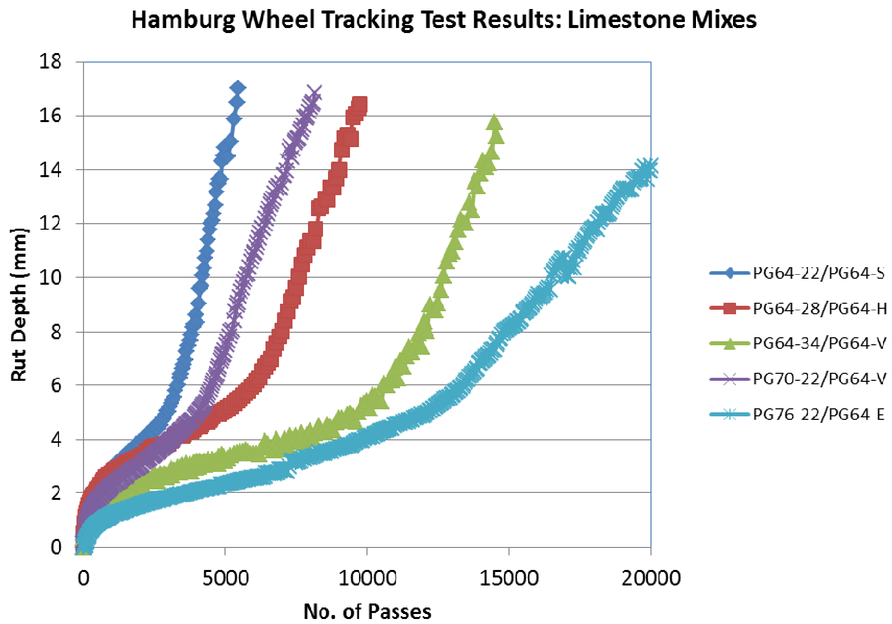


Figure 7. HWTT Results of Limestone Mixes with Five Binders.

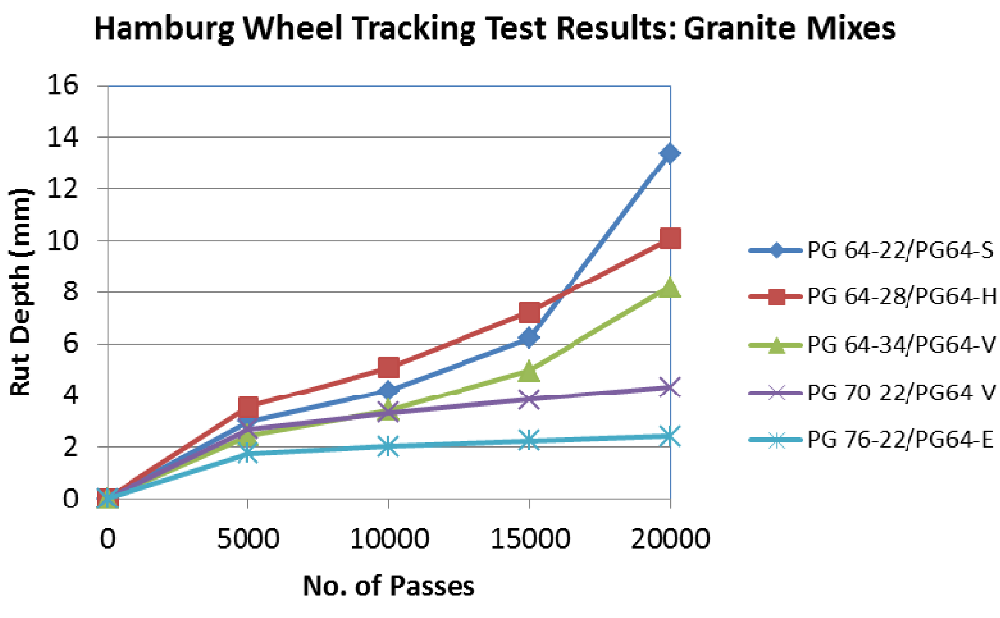


Figure 8. HWTT Results of Granite Mixes with Five Binders.

CONCLUSIONS AND RECOMMENDATION

This chapter evaluated the MSCR test with a focus on its repeatability and the validity of differentiating rutting performance of five types of asphalt binders. Based on the research results obtained previously, the following conclusions and recommendation are offered:

- MSCR Round Robin results among five laboratories clearly indicated that both $J_{nr0.1}$ and $J_{nr3.2}$ results are very repeatable and reproducible. But both J_{nrdiff} and $R_{0.1}$ have pretty high variability. Since J_{nrdiff} is one of the parameters for grading asphalt binder, caution should be exercised when applying the MSCR specification. The $R_{3.2}$ results are acceptable in terms of repeatability and reproducibility.
- Asphalt mix rutting test results showed that that the MSCR test and associated specification works better than the current G*/sin δ-based PG specification, especially for those highly modified asphalt binders (such as PG64-34). However, some caution should be exercised when grading the slightly modified asphalt binders (such as PG64-28).
- Based on the laboratory test results, the research team recommends that TxDOT implement the MSCR test and associated specification.

Certainly, the above conclusions and recommendation still need to be validated through field test sections constructed at different environments and traffic conditions.

CHAPTER 3
IDENTIFICATION OF SIMPLE FRACTURE TESTS FOR ASPHALT BINDERS

INTRODUCTION

The current asphalt binder PG specification was developed based on the SHRP asphalt program, and it was based primarily on the study of unmodified asphalt binders. It has long been known that the weakest part of the PG specification is the fatigue cracking parameter and associated criteria. The current PG binder specification employs parameter G*sinδ to quantify asphalt binder fatigue resistance. Many studies have questioned the parameter G*sinδ (*18*-*22*). Parameter G* sinδ is a binder stiffness parameter and is measured under relatively small strain conditions at a fixed frequency (10 rad/s) and only a few cycles of loading without damage, which is significantly different from the very complicated fatigue phenomenon that features much more cycles of loading and fatigue damage. To address this issue, many highway agencies adopted the elastic-recovery test to identify the existence of polymers in the modified binder and to enhance the current PG fatigue parameter, which is often called the PG Plus specification. Meanwhile, substantial research has been made to develop/identify a new asphalt binder fatigue test. Table 14 summarizes existing binder fatigue test methods and associated features for each method. Overall, there are four major developments in terms of asphalt binder fatigue test:

1. **Linear amplitude sweep test**: Bahia et al. initially proposed using DSR in a repeated constant strain/stress mode (or time sweep) to characterize the fatigue behavior of asphalt binders in 2001 (*18*). The proposed procedure is conceptually sound but not without its flaws. Anderson et al. found that the DSR-based time sweep test was not suitable for characterizing the fatigue behavior because of unstable flow and edge fracture effects in some cases (*23*). Most recently, Bahia and his associates developed a new, promising test named the linear amplitude sweep (LAS) test (*24, 25,26*). The LAS test results correlated fairly well with LTPP field fatigue cracking data (*25, 26*).
2. **Multiple stress creep recovery test**: The MSCR test was developed mainly for characterizing rutting resistance of asphalt binders (*13, 14*), but the MSCR recovery can be potentially used as one parameter to evaluate the delayed elastic response of asphalt binder. PG plus tests with a similar purpose such as elastic recovery could be replaced if MSCR recovery correlates well with fatigue resistance.
3. **Double edged notched tension (DENT) test**: Researchers at Queen's University initially proposed evaluation of the energy needed for fracturing ductile materials to get a measure of the fatigue cracking resistance of asphalt binders (*27*). It is a ductility tension test, but the sample is notched on two edges. Most recently, researchers at the Federal Highway Administration (FHWA) employed the DENT test to characterize the binders used in the FHWA accelerated loading facility (ALF) fatigue test lanes, and found very good rank-correlation between the DENT test results and the observed fatigue cracking under ALF loading (*28*).
4. **Dynamic mechanical analysis (DMA) mortar test**: Kim et al. used the DMA mortar test to characterize binder fatigue and healing potential of asphalt binders (*29*). Different from other tests in which the sample is asphalt binder only, the DMA mortar test uses asphalt mastic made of asphalt binder and sands or fine aggregates (*30*).

One of the good features of using the DMA mortar test for characterizing binder fatigue is to clearly define fatigue failure and avoid the unstable flow and edge fracture effect. The downside of the DMA mortar test is the influence of sand or fine aggregates on asphalt binder fatigue resistance. This is especially true when the test is to serve the purpose of a binder purchase specification. More discussion on the DMA fatigue cracking test is provided in Chapter 4. Therefore, this chapter evaluates all existing and the latest asphalt binder fatigue tests and compares them with asphalt mix fatigue cracking test data. Based on the comparisons, a simple and promising fatigue test for asphalt binders is recommended at the end of this chapter.

OBJECTIVES

The objectives of this study were to 1) evaluate the fatigue resistance of various binders (unmodified and modified), 2) verify the binder test results through two fatigue tests (Overlay Test and push-pull fatigue test) of asphalt mixes containing the same binders, and 3) identify a simple and promising asphalt binder fatigue test.

EXPERIMENTAL TEST PLAN

In order to achieve the above objectives, a series of laboratory tests including both asphalt binder and mix tests was planned. Figure 9 shows the overall experimental test plan.

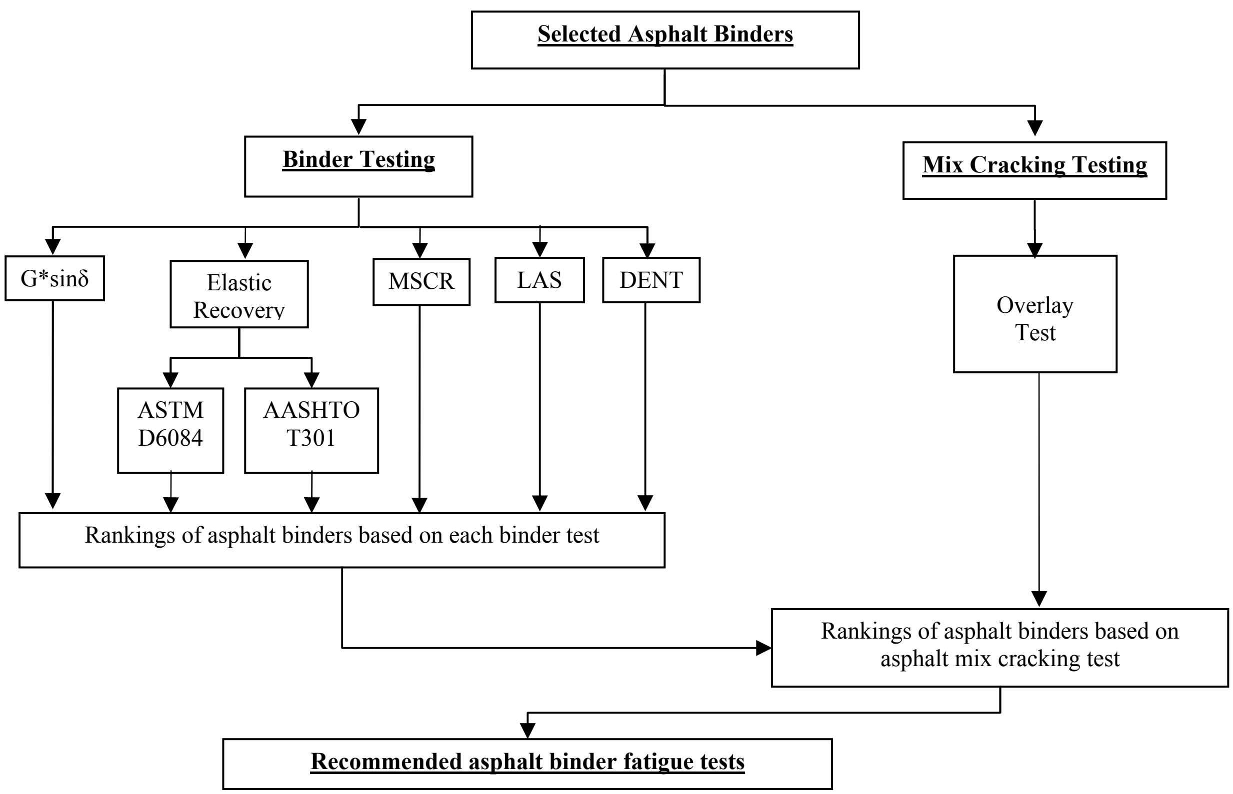
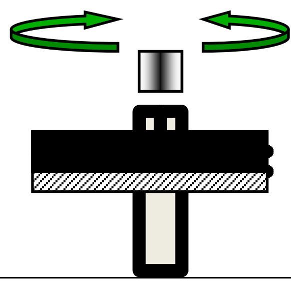
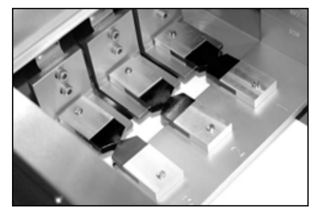


Figure 9. Experimental Plan.

Table 14. Existing Laboratory Tests for Characterizing Asphalt Binder Fatigue Resistance.

Item	**G* Test**	**MSCR Test**	**Time Sweep Test**	**Linear Amplitude Sweep Test**	**Elastic Recovery Test**	**Double Edge Notch Tension Test**	**DMA Mortar Test**
Test method	AASHTO T 315	AASHTO TP 70	NCHRP 9-10 (*18*)	Bahia et al. (*24, 25, 26*)	AASHTO T301 ASTM D6084	Ontario Ministry of Transportation Test Method LS-299 (*21*)	Kim et al. (*29*)
Parameter	G*sinδ	Recovery (%)	Fatigue life	Fatigue lives at different strain levels	Elastic recovery (%)	Critical tip opening displacement (CTOD)	Fatigue life
Test equipment	DSR				Ductility test machine	Ductility test machine with capability of measuring the force and displacement	Advanced DSR
Test specimen	Asphalt binder only and easy to prepare 				Asphalt binder only and easy to prepare 	Asphalt binder only and easy to prepare 	Asphalt binder + fine aggregates and much longer time to prepare 
Loading mode	Shear				Tension		Shear
Beyond LVE range	No	Yes	Yes	Yes	Yes	Yes	Yes
Correlation with field fatigue distress	Lots of concerns	TBD	TBD	Preliminarily validated with LTPP sections	Used for decades	Validated with FHWA-APT fatigue test sections	TBD

TBD-To be determined.

ASPHALT BINDERS, TESTS, RESULTS, AND ANALYSIS

Asphalt Binders

The same five asphalt binders used for the MSCR test evaluation in Chapter 2 were employed here. They are PG64-22, PG64-28, PG64-34, PG70-22, and PG76-22. Except for the PG64-22 binder, the other four binders are polymer modified binders.

Asphalt Binder Testing

A total of five tests were performed in this study. Detailed information is described below.

PG Grading Test

The six asphalt binders were tested following the American Association of State Highway and Transportation Officials T315 "Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)" and T313 "Determining the Flexural Creep Stiffness of Asphalt Binder Using the Bending Beam Rheometer (BBR)."

Elastic Recovery Test

The elastic recovery test is conducted using the ductility device following TxDOT's elastic recovery test, which is a modified version of American Society for Testing and Materials (ASTM) D6084 Method A "Standard Test Method for Elastic Recovery of Bituminous Materials by Ductilometer." The major difference between TxDOT's method and the ASTM D6084 is the test temperature. TxDOT's test method requires performing the elastic recovery test at 50°F (10°C) rather than 77°F (25°C) at the ASTM specification. Another difference is that TxDOT uses original asphalt binder for the elastic recovery test.

In the elastic recovery test, the binder specimens were pulled apart in a ductilometer and held after reaching a specified elongation. The specimens were then cut in the middle of the elongation and the percent recovery of each specimen was determined.

MSCR Test

The MSCR test is conducted using the DSR following AASHTO TP70 "Multiple Stress Creep Recovery (MSCR) Test of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)." Each RTFO-aged specimen was tested at the same temperature of 64°C. It is hypothesized that the higher the degree of elastic recovery under the MSCR test, the binder is more fatigue resistance. This hypothesis will be tested here by comparing the ranking of binder recovery to the ranking of the asphalt mix fatigue characteristics measured under the push-pull fatigue test.

LAS Test

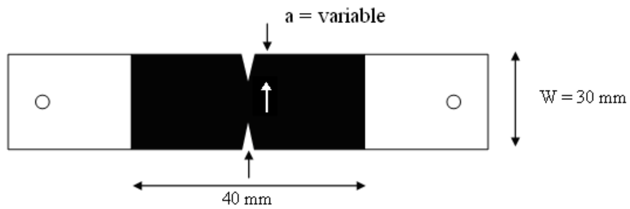
The LAS test, compared to the time sweep test, is an accelerated method test and it uses the DSR with the standard 8 mm parallel plate geometry. Either RTFO- or pressure aging vessel (PAV)-aged asphalt binder can be used. Basically the LAS test determines two parameters (*a* and *b*) of asphalt binder fatigue law ($N_f = a \times \gamma_{max}^{b}$). As noted by Bahia and his associates (*24-26*), the LAS test consists of two steps: a frequency sweep test and an amplitude sweep test. The frequency sweep test at very low strain amplitude of 0.1% is used to obtain undamaged material properties and accordingly the parameter *b* of the fatigue law. The amplitude sweep test with a series of cyclic loads at systematically linearly increasing strain amplitudes at a constant frequency of 10 Hz is used to determine the parameter *a* of the fatigue law through the viscoelastic continuum damage (VECD) mechanics analysis. For the linear amplitude sweep test, loading begins with 100 cycles of sinusoidal loading at 0.1%. Each successive loading step consists of 100 cycles at a rate of increase of 1% applied strain until reaching 30% applied strain (*25*). Note that the combination of the frequency and amplitude sweeps tests takes approximately 10 min plus conditioning time. Bahia and his associates reported that the LAS test results have a good correlation with the fatigue data of LTPP test sections (*25*).

Specifically for this study the PAV-aged specimens with two replicates were used for each binder, and the LAS test was conducted at 77°F (25°C). The test data were analyzed using an Excel© macro provided by Bahia's group, and the two parameters *a* and *b* were automatically calculated. The results presented later are the average value of the two replicates.

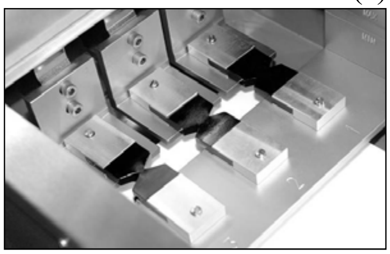
DENT Test

The DENT test and the calculated critical tip opening displacement (CTOD) were originally developed at Queen's University of Canada (*27*). Recently, FHWA made some minor changes relative to the Ontario Ministry of Transportation test method for more southern conditions and developed a draft method in AASHTO format (*28*). Basically, it is believed that the energy needed for fracturing ductile materials consists of two parts: an essential portion of work performed in the local region of the advancing crack creating two surfaces and non-essential work away from the local region of cracking/tearing associated with ductility, plasticity and yielding. To determine the essential work of fracture and the CTOD, the DENT test is performed using similar specimens with different ligament lengths (such as 5, 10, 15 mm). Figure 10 shows a schematic of the sample in the DENT test defining ligament length, the test samples in a force-ductility instrument, and typical test results. Gibson et al. reported that the parameter CTOD relates extremely well with the FHWA-ALF fatigue test results (*28*). Large CTOD test results indicate better fatigue resistance.

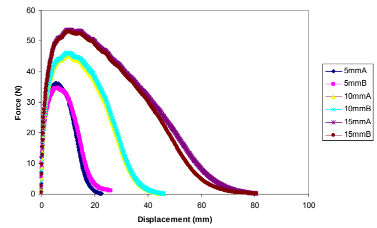
For this study, the DENT test was conducted at 77°F (25°C) for all six asphalt binders with the PAV-aged specimens and two replicates used for each asphalt binder. The results reported later are the average values of the two replicates.



(a) Ligament definition



(b) Test samples and ductility instrument



(c) Typical DENT test results

Figure 10. FHWA DENT Test (*28*).

Binder Test Results and Analysis

For each test, at least two replicates were used, and the average value is reported. Table 15 documents all of the test results of the five asphalt binders under five different tests.

Table 15. Phase I Test Results of the Five Asphalt Binders.

Asphalt Binder		PG64-22	PG64-28	PG64-34	PG 70-22	PG76-22
Binder PG Grading	Continuous Grade	69.85-24.96	68.06-29.27	68.14-34.39	74.60-24.83	84.46-22
	True Grade at Intermediate Temperature (°C)	20.32	16.815	11.14	23.045	9.7
	Overall Ranking	**D**	**C**	**B**	**E**	**A**
TXDOT Elastic Recovery	Method A(0s Holding)	28.71%	74.19%	89.50%	56.78%	69.41%
	Overall Ranking	**E**	**B**	**A**	**D**	**C**
Multiple Stress Creep Recovery RTFO	Percent Recovery-100Pa	2.0%	41.6%	75.3%	37.8%	83.8%
	Percent Recovery-3,200 Pa	0.6%	24.0%	65.8%	25.5%	78.6%
	Percent Difference between Average Recovery Values	70.0%	42.3%	12.6%	32.5%	6.2%
	Overall Ranking	**E**	**D**	**B**	**C**	**A**
LAS	Parameter a	2.18E+07	2.43E+07	1.24E+07	5.94E+07	9.61E+07
	Parameter b	-5.31	-4.94	-4.99	-5.88	-6.54
	Fatigue life at γ_{max}=2.5%	168,810	262,320	1,276,157	270,786	240,382
	Fatigue life at γ_{max}=5%	4,268	8,530	40,155	4,587	2,586
	Overall Ranking	**D**	**B**	**A**	**C**	**E**
DENT	CTOD(mm)	12	27	69	16	14
	Overall Ranking	**E**	**B**	**A**	**C**	**D**

Based on the results in Table 15, asphalt binders are ranked from *A* to *E*, with *A* referring to the best and *E* being the last one.

- **G*sinδ ranking**: Table 15 shows the true intermediate temperature, which corresponds to 5000 kPa. Generally, the higher the intermediate temperature, the poorer fatigue resistance of the asphalt binder. Surprisingly, PG76-22 binder is the best, followed by PG64-34, PG64-28, and PG64-22. PG70-22 is ranked the worst.
- **Elastic recovery ranking**: PG64-34 has the highest elastic recovery and is ranked *A*, followed by PG64-28, PG76-22, and PG70-22. The PG64-22 binder has the smallest elastic recovery. The ranking clearly validates the effectiveness of the elastic recovery test in differentiating the modified form the conventional (nonmodified) binders.
- **MSCR ranking**: Similarly, the PG76-22 binder having the highest recovery under both stress levels, is the best and ranked *A*. The PG64-34, having high recovery, is ranked *B*, followed by PG70-22 and PG64-28. The PG64-22 binder has the lowest recovery and is ranked last.
- **LAS ranking**: Based on the fatigue lives of the five binders listed in Table 15, it can be observed that the PG64-34 is the best binder, followed by PG64-28 and PG70-22. PG76-22 has a higher fatigue life in the low strain level (i.e. 2.5%) than PG64-22, but the trend is reserved at the higher strain level (i.e 5%). Apparently, the fatigue life of PG76-22 has a significant change at the two strain levels (from 240,382 at 2.5% strain to 2,586 at 5% strain). When taking into account fatigue life changes with strain levels, PG76-22 is ranked as *E*, because an asphalt binder needs to have consistent fatigue performance at all strain levels.
- **DENT ranking**: The ranking for the six binders is:

PG64-34>PG64-28>PG70-22> PG76-22>PG64-22.

Apparently, the asphalt binders are ranked differently under different binder tests. To verify the correct ranking and identify the best binder fracture test, the cracking resistances of asphalt mixes with these five binders were evaluated, and detailed information is presented in the following sections.

ASPHALT MIXES, CRACKING TEST, RESULTS, AND ANALYSIS

Asphalt Mixes

The exactly same three mixes used in the MSCR evaluation (Chapter 2) were employed here to identify a simple asphalt binder fracture test. Three types of aggregates (limestone, crushed gravel, and granite) and the five binders (PG64-22, PG64-28, PG64-34, PG70-22, and PG76-22) were mixed together, and a total of 15 mixes were tested under the Overlay Test. More detailed information can be found in Chapter 2.

Overlay Test

The Overlay Test (OT) was used to evaluate the cracking resistance of the asphalt mixes. The OT was performed following Tex-248-F: *Test Procedure for Overlay Test* (*31*). Five trimmed specimens from each mixture targeting an air void of 7 % ± 1% were prepared. Before testing, individual OT specimens were conditioned in an environmental chamber with a target temperature of 77°F (25°C). The sliding block applied tension in a cyclic triangular waveform to a constant maximum displacement of 0.025 inch (0.6 mm). The sliding block reached the maximum displacement and then returned to its initial position in 10 sec. The time, displacement, and load corresponding to a certain number of loading cycles were recorded during the tests. The number of cycles to failure is determined for each specimen when the maximum load reaches 7 percent of the initial maximum load recorded in the first cycle. The average of the OT cycles of five specimens is reported. The larger the OT cycles, the better cracking resistance is.

Results and Analysis

Figure 11 shows the OT results of the 15 mixes. It can be clearly seen that the PG64-34 binder has the best cracking resistance, followed by PG64-28. It seems that PG64-22 and PG70-22 binders have similar performance. The PG76-22 binder has the smallest OT cycles and is ranked the last.

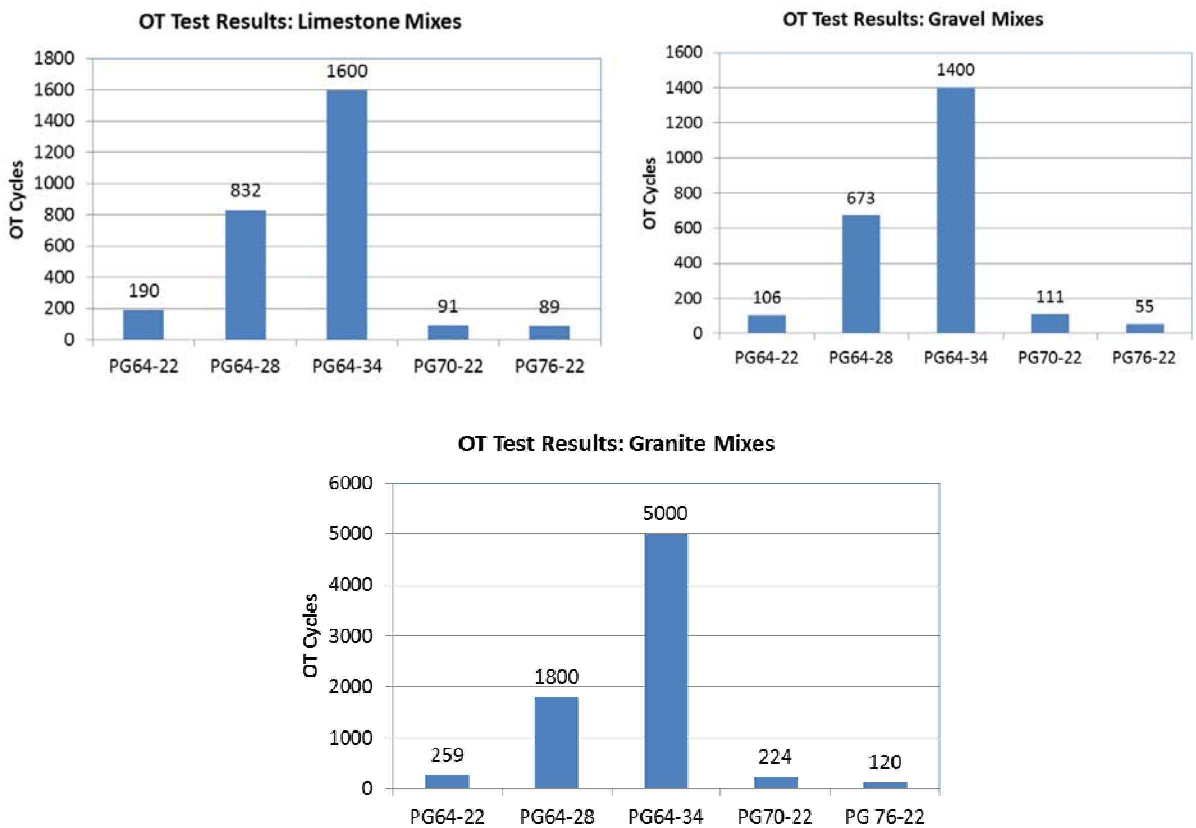


Figure 11. OT Test Results of 15 Asphalt Mixes.

DISCUSSION ON THE BEST BINDER FATIGUE TEST

Comparing the binder test results (Table 15) with the asphalt mix test results (Figure 11), it can be clearly seen that the rankings based on PG intermediate temperature, TxDOT's elastic recovery test, MSCR test have no good correlation with the ranking based on asphalt mix cracking test. Both the LAS and DENT tests clearly differentiated the PG64-34 and PG64-28 binders from the rest binders and provide very similar ranking to the asphalt mix cracking test data. Although neither one shows exactly the same ranking as that of asphalt mix cracking test, both the LAS and DENT tests can be used for characterizing cracking resistance of asphalt binders. Considering the test equipment requirement of both the LAS and DENT tests, the DSR-based LAS test is recommended for asphalt binder fracture test, because the ductility test equipment required by the DENT test is not often used in TxDOT and contractors' laboratories.

SUMMARY AND FINDINGS

The current PG binder specification uses parameter G*sinδ to quantify asphalt binder fatigue resistance, which has long been known to be the weakest part of the specification. The purpose of this study was to identify a better asphalt binder fracture test. Five asphalt binder tests were evaluated and compared with the OT cracking test. Basically, three findings are identified in this study:

- This study further confirms the poor relationship between the parameter G*sinδ and the binder fatigue resistance.
- Neither the MSCR nor the elastic recovery test shows good correlation with the asphalt mix OT cracking test.
- Both the LAST and the DENT tests provide similar ranking as that of asphalt mix OT cracking test. Considering the test equipment requirements of both the LAS and DENT tests, the DSR-based LAS test is recommended for asphalt binder fracture test, since the DSR has been widely used in last 20 years and laboratory technicians and researchers are very familiar with it.

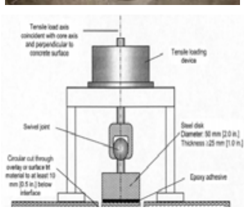
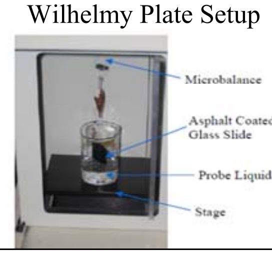
Obviously, these findings are based on laboratory test results only. Further field validation is definitely needed. Additionally, one needs always to keep in mind that "the binder alone does not determine fatigue response in the pavement structure. Mix characteristics as well as the pavement structure itself and the environment within which it is located have a significant role in determining pavement performance" (*18*).

CHAPTER 4
ASPHALT BINDER ADHESION TEST

INTRODUCTION

Asphalt binder adhesive property has influence on moisture damage of asphalt mixes. It is ideal to identify/develop an asphalt binder test to directly characterize asphalt binder adhesive property, although other factors including aggregate types, asphalt mix plant production, field construction, etc. may have more impact on moisture damage of asphalt mixes. Currently, three types of laboratory tests have been used to evaluate asphalt binder adhesive property, as presented in Table 16. This chapter describes all three types of tests and discusses the practicality for routine use. Detailed information is described in the following sections.

Table 16. Asphalt Binder Adhesion Tests.

Item	Direct Tensile Bond Test	Dynamic Mechanical Analyzer (DMA) Adhesion Test	Surface Energy Test
Test equipment	Direct tensile bond test apparatus  	Advanced DSR  	Universal Sorption Device  Wilhelmy Plate Setup 
Adhesion parameter	Bond strength ratio (wet/dry)	Fatigue life ratio (wet/dry)	Surface energy
Testing time	Short (within minutes)	Long (within hours)	Very long (Binder surface energy: 3 hours; Aggregates surface energy: 10 days)
Test complexity	Simple	Complex	Complex
Correlation to field moisture damage	No well established	Very limited data showed good correlation.	Very limited data showed good correlation.
Variability	Unknown	Unknown	Unknown

DIRECTION TENSILE BOND TEST, RESULTS, AND DISCUSSION

ASTM C 1583: Direct Tensile Bond Test (*32*) shown in Figure 12 was originally developed for use as an indicator of the adequacy of concrete surface preparation before applying a repair or overlay material. It is performed on the surface of the overlay material and determines the bond strength to the substrate or the tensile strength of either the overlay or substrate, whichever is weaker. When the test is performed on the surface of the material applied to the substrate, the measured strength is controlled by the failure mechanism requiring the least stress (Figure 12). Figure 13 contains a photo of the test device being used for the laboratory evaluation.

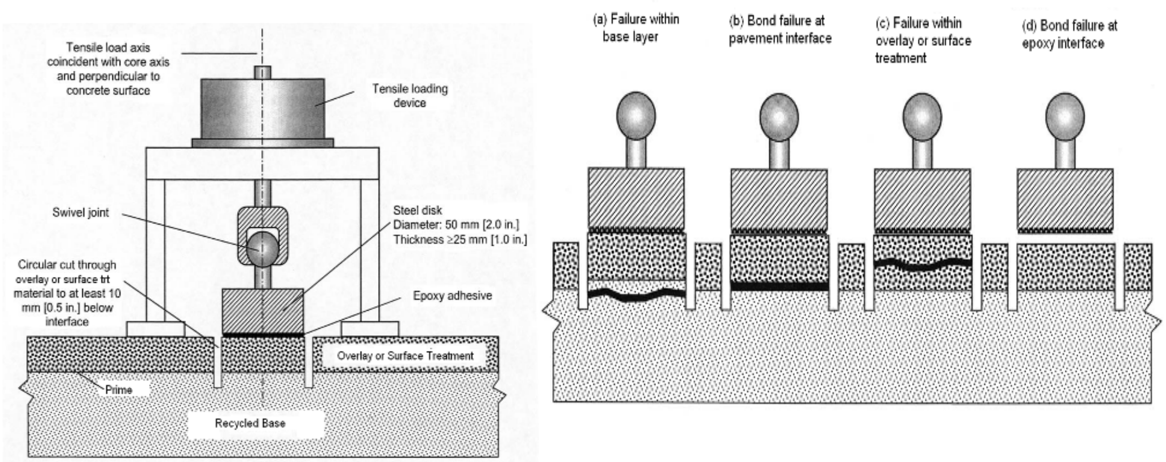


Figure 12. Schematic of Direct Tensile Bond Test and Possible Failure Mode.

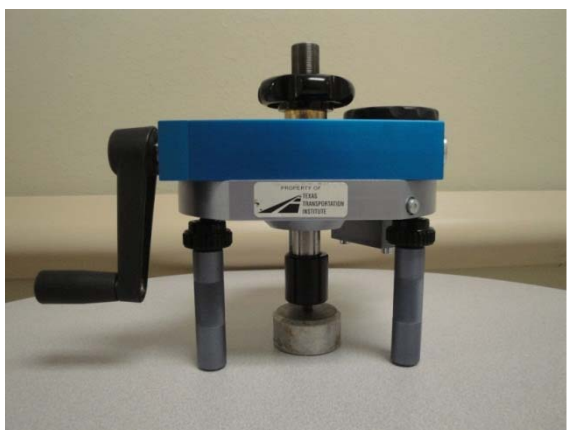


Figure 13. Photo of Direct Tensile Bond Test Apparatus.

The research team tried to use this test apparatus to measure the tensile strength of asphalt binders. It is ideal to measure the bond strength between asphalt binder and aggregates. However, a previous study on testing adhesive failure of cracking sealants under a previous research project 0-5457 (*33*) clearly indicated that it is very difficult (if not impossible) to get consistent bond strength between asphalt binders and aggregates, not only because aggregate surface varies among the same type of aggregates, but even within large aggregate, surface characteristics also change from location to location. Therefore, an alumina block rather than aggregates was used for this study so that the test results among different binders can be compared with each other. Figure 14 shows the whole process of specimen preparation and testing.

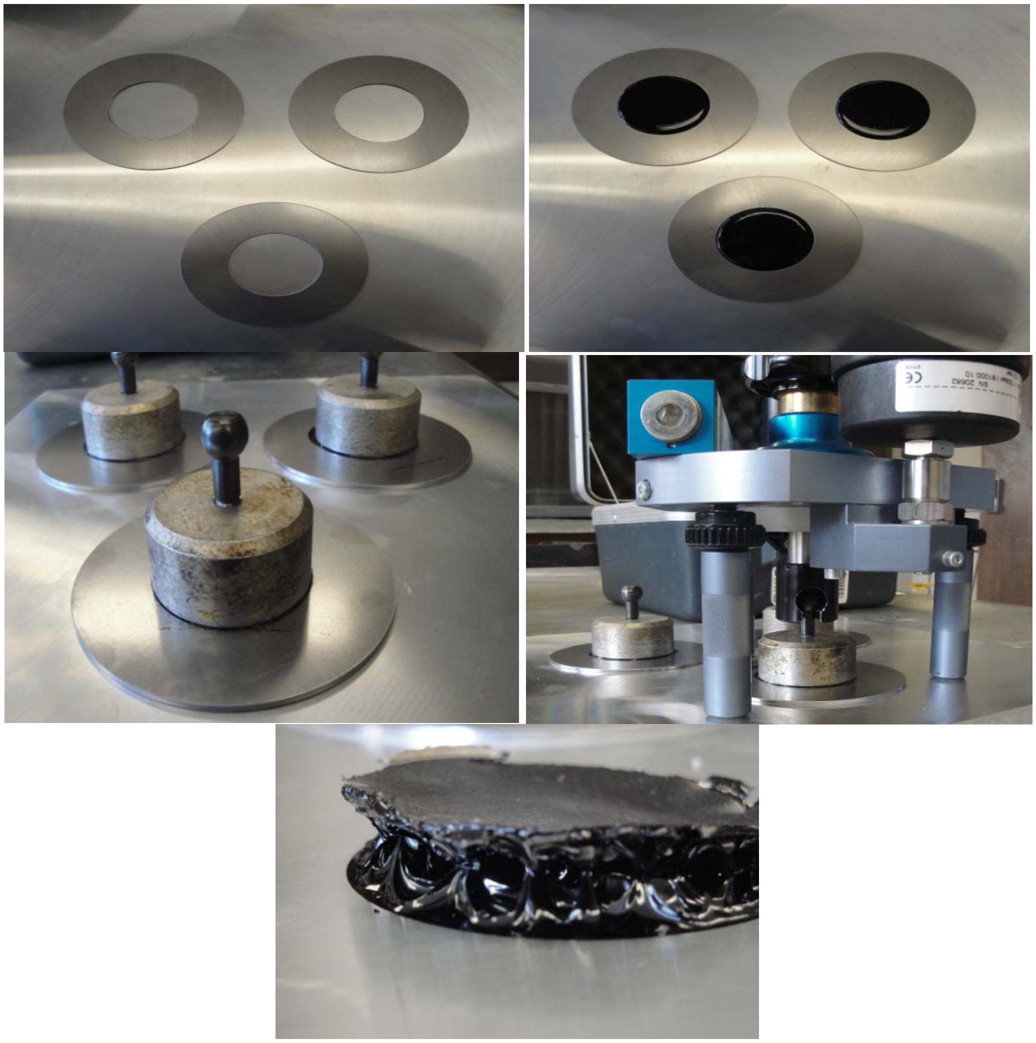


Figure 14. Whole Process of the Direct Tensile Bond Test for Asphalt Binders.

Initially it was thought that the bond strength test is a very simple test. Actually it turned out that the bond strength test for asphalt binders is a very complex one. Three problems have been observed, as described below:

- The bond strength depends on temperature and pulling rate, since asphalt binders are viscoelastic materials. The test temperature was controlled, but it is impossible to manually apply the same pulling rate for each test. Therefore, the bond strengths measured among different replicates for the same binder varied significantly.
- The bond strength and failure mode depend on the thickness of asphalt film. The same binder showed completely different failure modes when varying the thickness of the specimen.
- The direct tensile bond test apparatus has a limited load cell. For most asphalt binders at room temperature, neither adhesive nor cohesive failure can be observed. When the test temperature was reduced, the bond strength was easy to reach the limit of the load cell without any failure observed.

After many trials, the researchers concluded that the direct tensile bond test apparatus used in this subtask is not suitable to measure the bond strength of asphalt binders.

PNEUMATIC ADHESION TENSILE TEST, RESULTS, AND DISCUSSION

Recently another type of direct tension bond test has been developed and marketed with the name of PATTI (Pneumatic Adhesion Tensile Testing Instrument). Compared to the direction tension bond strength test shown in Figure 12, the biggest advantage of PATTI is that the same controlled pulling rate can be applied to each test specimen. Therefore, the test results are relatively comparable. Figure 15 shows the PATTI and the whole test process. The fastest pulling rate was used for all the tests performed. Adhesive failure (Figure 15) was observed on all binders except the very soft PG64-34 original binder. The tensile strength is reported as the end of the PATTI test.

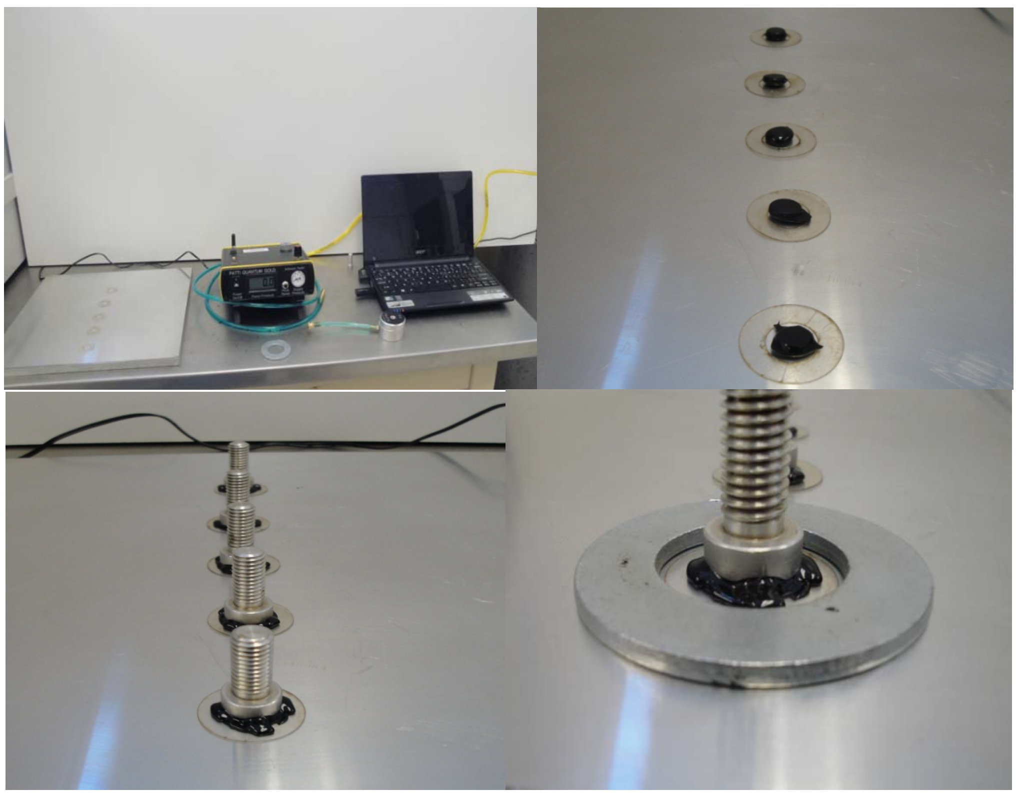


Figure 15. PATTI Test.

Figure 15. PATTI Test (continued).

Figures 16 and 17 show the PATTI test results of five asphalt binders at original and rolling thin film oven-aged conditions, respectively. The following three observations are made from Figures 16 and 17:

- Different binders show different tensile strength. As expected, stiffer asphalt binders apparently have higher tensile strength. As shown in Figure 16, PG76-22 binder has the highest tensile strength, while PG64-34 original binder has the lowest value. Note that the PG64-34 original binder did not show adhesive failure.
- Except for the softest binder, PG64-34, RTFO aging reduces the tensile bonding strength, which is not unexpected, since PG64-34 original binder is too soft to have adhesive failure.
- Adhesive property of different asphalt binders has different reaction to RTFO aging. The PG76-22 original binder has the highest tensile strength, but its tensile strength has the most dramatic change. Therefore, binder adhesion property should be evaluated at both original and RTFO aged conditions.

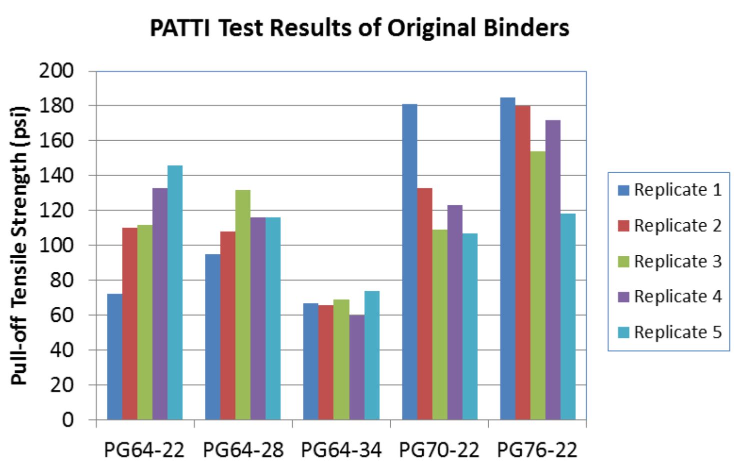


Figure 16. PATTI Test Results of Five Original Asphalt Binders.

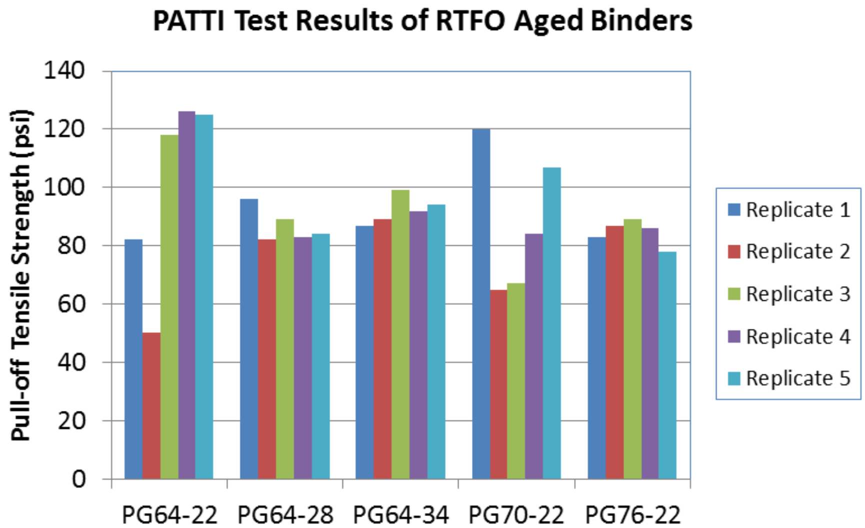


Figure 17. PATTI Test Results of Five RTFO-Aged Asphalt Binders.

DMA TEST, RESULTS, AND DISCUSSION

A typical asphalt concrete mixture exhibits two distinct phases: a portion of relatively coarse aggregate particles and a portion of asphalt mastic, as shown in Figure 18. Asphalt mastic is comprised of asphalt binder, fine aggregates (smaller than about No. 16 sieve size), and air voids. It has been observed through the HWTT that moisture damage is often related to fine aggregates. So it is reasonable to use asphalt mastic for evaluating potential moisture damage of the whole asphalt concrete mixture as well. The following sections describe the DMA tests performed in this project and associated results.

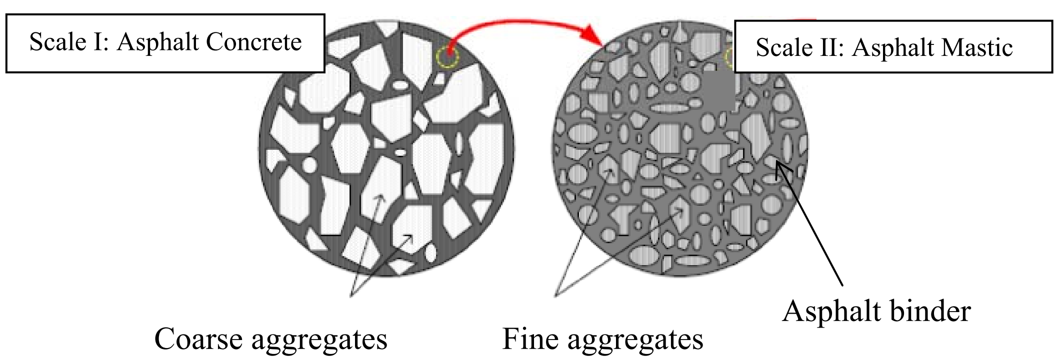


Figure 18. Schematic View of Asphalt Concrete and Asphalt Mastic.

DMA Test Materials

Two fine aggregates: limestone from the FM973 job and granite from the FM521 job, were selected for the DMA torsion test. Additionally, five binders, including PG64-22, PG70-22, PG76-22, PG64-28, and PG64-34, were chosen to blend with the two aggregates to make DMA specimens.

DMA Specimen Preparation and Test Setup

The DMA specimen preparation followed the procedure proposed by Dr. Emad Kassem, Advanced Characterization of Infrastructure Materials Laboratory (*34*). Basically, the design methodology of DMA mixture is to obtain a representative sample of the fine matrix of a complete asphalt mixture. For this reason, a previously established asphalt mix design is required for this process (*35*). The design procedure considers the granular material of the asphalt mixture passing the No. 16 sieve (1.18 mm). The percent of asphalt is estimated by calculating the amount of binder that is expected to cover the total granular particles (coarse and fine aggregates). Just the amount of binder absorbed by the coarse aggregates (larger than 1.18 mm) is used on the fine graded asphalt mix design.

The first step in the preparation of the specimens consists of mixing and compacting, using the Superpave Gyratory Compactor, to obtain a 150 mm diameter cylindrical sample with an approximate height of 85 mm. This procedure is similar to the one used to prepare regular asphalt mix specimens. The upper and lower parts of the cylinders are sawed in order to produce a new cylinder of 150 mm diameter by 50 mm height. This compacted sample is cored in small DMA cylindrical specimens of 12 mm in diameter by 50 mm in height. Each specimen is properly labeled and prepared for testing. Two methods of test-specimen preparation are herein considered: 1) when testing on dry condition, and 2) when testing on specimens that have been subjected to a moisture conditioning process. Figure 19 shows the DMA specimen preparation and test setup.

Figure 19. DMA Specimen Preparation and Test Setup.

DMA Test

The DMA test conducted in this study was a torsion (or oscillation) test with a controlled strain of 0.25. The DMA test was performed at a frequency of 10 Hz and a temperature of 86°F (30°C). Note that the same strain level, frequency, and test temperature were used for testing all DMA specimens.

Initially, it was thought that DMA test may be used as a fatigue test. However, such high test temperature became a big problem for the DMA test being used as a fatigue test. The fatigue cracking test is normally performed at a temperature below 77°F (25°C), and 68°F is the temperature most often used for the fatigue test. The main reason for using 86°F in this study was due to the limitation of the DMA machine. Asphalt mix stiffness is very sensitive to temperature. At the test temperature of 68°F and below, the DMA machine has problems reaching the selected strain level, which makes the DMA fatigue test last impractically long. Even at 86°F, the fine mix with PG76-22 binder could not reach the strain of 0.25 under the

DMA test, so no reliable test results were obtained. Therefore, the DMA test actually is not a typical fatigue test, and precaution should be exercised when using DMA as a fatigue testing.

Additionally, when the test was run at the relatively high temperature of 86°F with a strain of 0.25, the DMA test still needs 20 hr to obtain a full fatigue curve for a regular PG64-22 binder. For the soft binders, PG64-28 and PG64-34, no fatigue failure was observed even after 20 hr of testing. The testing was terminated, and no result was obtained for binders: PG64-28 and PG64-34. In summary, the original plan was to evaluate five asphalt binders: PG64-22, PG70-22, PG76-22, PG64-28, and PG64-34. Only two binders, PG64-22 and PG70-22, have the final results due to limitation of DMA test itself.

DMA Test Results and Discussion

Table 17 shows the DMA test results. For asphalt binder PG64-22, the DMA test results make sense. Fine mix at the wet condition has less fatigue life than dry condition, and as expected, moisture has more impact on granite aggregates compared to limestone aggregates. However, this is not the case for fine mix with PG70-22 binder. The fine mix with PG70-22 binder at the wet condition has a higher fatigue life than that at the dry condition. As mentioned previously, The PG70-22 binder is much stiffer than the PG64-22 binder. The DMA machine compliance may influence the final test results.

Based on the test results obtained from this study and the experience of running the DMA test, the research team believes that the DMA test cannot be used as a routine fatigue test. It may be used for evaluating potential moisture damage for different types of aggregates, but it also depends on the stiffness of asphalt binders. It is much easier to directly run the Hamburg wheel tracking test to evaluate moisture damage of asphalt mixes when considering the tedious specimen preparation and long testing time.

Table 17. DMA Test Results at the Temperature of 86°F and a Strain of 0.25.

Aggregates	Binder	Test Conditions	Fatigue Life (cycles)	Average Fatigue Life (cycles)
Limestone	PG64-22	Dry	248855	273991
			291524	
			281596	
		Wet	179570	271894
			437151	
			198960	
Granite	PG64-22	Dry	121909	178326
			234508	
			178561	
		Wet	62344	68827
			62617	
			81522	
	PG70-22	Dry	444237	353546
			345187	
			271214	
		Wet	463738	556718
			580779	
			625637	

SURFACE ENERGY TEST

Surface energy is used to assess the adhesive bond between asphalt binders and aggregates in dry and wet conditions. Surface energy is often divided into three components: 1) the nonpolar component also referred to as the Lifshitz-van der Waals (LW) component, 2) the Lewis acid component, and 3) the Lewis base component. The total surface energy is obtained by combining these components as follows:

$$\gamma = \gamma^{LW} + 2\sqrt{\gamma^{+}\gamma^{-}}$$

where γ is the total surface energy of the material, γ^{LW} is the LW component, γ^{+} is the Lewis acid component, and γ^{-} is the Lewis base component. Surface energy components of binders were measured using Wilhelmy Plate Method in this study, which are based on dynamic contact angles of probe liquid on sample surface. Table 18 lists the measured component and calculated surface energy.

Since the DMA test did not succeed in this study, the measured surface energy could not be effectively evaluated.

Table 18. Surface Energy of Asphalt Binders.

Asphalt	Surface Energy Components				Standard Deviation		
	LW	Acid	Base	Total	sigma LW	Acid	Base
PG64-22	17.60	0.31	1.63	19.03	0.29	0.06	0.37
PG64-28	33.54	0.00	0.05	33.54	0.54	0.00	0.05
PG64-34	19.71	0.00	3.48	19.71	0.78	0.00	0.41
PG70-22	17.82	0.00	17.33	18.12	0.68	0.01	0.90
PG76-22	24.09	0.00	2.87	24.14	0.49	0.00	0.50

SUMMARY AND CONCLUSIONS

This chapter evaluated four types of laboratory tests for measuring asphalt binder adhesive property. It was found that the PATTI test is the only promising test for evaluating adhesive properties of asphalt binders. All three other tests, the pull-off test, DMA, and surface energy test, were not successful in this study. More research is needed in this area.

CHAPTER 5
SUMMARY AND CONCLUSIONS

This report documents the research work on asphalt binder rutting test, fatigue test, and adhesion test. A variety of new development in each area has been made in the past. This project evaluated all new tests available for both modified and non-modified asphalt binders. Based on the results presented in this report, the following conclusions are offered.

- Asphalt mix rutting test results showed that that the MSCR test and associated specification works better than the current G*/sin δ based PG specification, especially for those highly modified asphalt binders (such as PG64-34). MSCR Round Robin results among five laboratories clearly indicated that both $J_{nr0.1}$ and $J_{nr3.2}$ results are very repeatable and reproducible. The $R_{3.2}$ results are acceptable in terms of repeatability and reproducibility, but both J_{nrdiff} and $R_{0.1}$ have pretty high variability. Since J_{nrdiff} is one of the parameters for grading asphalt binder, TxDOT should exercise caution when grading the slightly modified asphalt binders (such as PG64-28) using the MSCR specification.
- This study further confirms the poor relationship between the parameter G*sinδ and the binder fatigue resistance. Neither the MSCR nor the elastic recovery test shows good correlation with the asphalt mix OT cracking test. Both the LAST and the DENT tests provide similar ranking as that of asphalt mix OT cracking test. Considering the test equipment requirements of both the LAS and DENT tests, the DSR-based LAS test is recommended for asphalt binder fracture test, since the DSR has been widely used in last 20 years and laboratory technicians and researchers are very familiar with it.
- Four types of laboratory tests for measuring asphalt binder adhesive property were evaluated. It was found that the PATTI test is the only promising test for evaluating adhesive properties of asphalt binders. All other three tests, the pull-off test, DMA, and surface energy test, were not successful in this study for evaluating asphalt binder adhesion property.

Obviously, these findings are based on laboratory test results only, and further field validation is definitely needed. Additionally, one needs always to keep in mind that the binder alone does not determine rutting, fatigue cracking, and moisture damage of asphalt pavements. Mix characteristics as well as the pavement structure itself, traffic, and the environment within which it is located have a significant role in determining pavement performance.

REFERENCES

1. American Association of State Highway and Transportation Officials. *Standard Specification for Performance Graded Asphalt Binders* M320, 2006.
2. Dongre, R., J. and D'Angelo., "*Evaluation of Different Parameters for Superpave High Temperature Binder Specification Based on Rutting Performance in the Accelerated Loading Facility at FHWA*," Transportation Research Record, TRB, 2003.
3. Stuart, K., W. Mogawer, and P. Romero. (1999). *Validation of Asphalt Binder and Mixture Test That Measured Rutting Susceptibility Using the Accelerated Loading Facility*. Interim Report October 1993-October 1999. Turner Fairbank Highway Research Center, Federal Highway Administration, McLean, VA.
4. D'Angelo, J., R. Dongre. "Superpave Binder Specifications and their Performance Relationship to Modified Binders," Proceeding, Canadian Technical Asphalt Association, November 2002.
5. Sybilski, D. Zero-Shear Viscosity of Bituminous Binder and Its Relation to Bituminous Mixtures Rutting Resistance, *Journal of Transportation Research Record* 1535, 1996, pp. 15-21.
6. Bouldin, M. G., R. Dongre, and J. D'Angelo, J. "Proposed Refinement to The Superpave High Temperature Specification parameter for Performance Graded Binders," *Journal of Transportation Research Record 1766*, 2001, pp. 40-47.
7. Shenoy, A. "Refinement of the Superpave Specification Parameter for Performance Grading of Asphalt," Journal of Transportation Engineering, Vol. 127, No. 5, 2001, pp. 357- 362.
8. Phillips, M. C. and C. Robertus. "Binder Rheology and Asphaltic Pavement Permanent Deformation; The Zero-shear Viscosity," 1st Eurasphalt and Eurobitume Congress, Strasbourg, 1996, Paper No. 5.134.
9. Desmazes, C., M. Lecomte, D. Lesueur, and M. Phillips. "A Protocol for Reliable Measurement of Zero-shear-viscosity in Order to Evaluate Antirutting Performance of Binders," *Proceedings of 2nd Eurasphalt & Eurobitume Congress*, Barcelona, Spain, Book 1, 2000, pp. 202-211.
10. Ferry, J. D., *Viscoelastic Properties of Polymers*, Third Edition, John Wiley and Sons Inc., New York, 1980.
11. Bahia, H. U., D. L. Hanson, M. Zeng, H. Zhai, M. A. Khatri, and R. M. Anderson. *Characterization of Modified Asphalt Binders in Superpave Mix Design*, Report No. 459, National Cooperative Highway Research Program, Washington, D.C., 2001.
12. Dongré, R. N., and J. A. D'Angelo. "Development of a high temperature performance-based specification in the United States." 10th International Conference on Asphalt Pavements, Quebec, Canada, 2006.
13. D' Angleo, J., R. Q. Kluttz, N. Raj, K. Stephens, and L. Zanzotto, Revision of the Superpave High Temperature Binder Specification: the Multiple Stress Creep and Recovery Test, *Journal of the Association of Asphalt Paving Technologists*, Vol. 76, 2007.

14. D' Angleo, J. and N. Raj, Practical Use of the MSCR Test: Characterization of SBS Dispersion and Other Additives in PMA Binders, *Journal of Transportation Research Record* 2126, 2009, pp. 73-82.

15. H. Bahia, *MARC Program Research Update*, Presentation at the 12th annual meeting of the association of modified asphalt producers in Kansas City, Missouri on February 15-17, 2011.

16. ASTM E691-11 *Standard Practice for Conducting an Inter laboratory Study to Determine the Precision of a Test Method.*

17. TxDOT Designation: TEX-242-F. Hamburg Wheel-Tracking Test. Construction Division, Texas Department of Transportation, 2009.

18. Deacon, J. A., J. T. Harvey, A. Tayebali, and C. L. Monismith. "Influence of Binder Loss Modulus on the Fatigue Performance of Asphalt Concrete Pavements." *Journal of Association of Asphalt Paving Technologists*, Vol. 66, 1997, pp. 633-668.

19. Bahia, H. U., D. L. Hanson, M. Zeng, H. Zhai, M. A. Khatri, and R. M. Anderson, *Characterization of Modified Asphalt Binders in Superpave Mix Design*, Report No. 459, National Cooperative Highway Research Program, Washington, D.C., 2001.

20. Bahia, H. U., H. Zhai, M. Zeng, Y. Hu, and P. Turner. Development of Binder Specification Parameters Based on Characterization of Damage Behavior. *Journal of the Association of Asphalt Paving Technologists,* Vol. 70, 2002, pp. 442-470.

21. Andriescu, A., S. A. Am. Hesp, and J. S. Youtcheff, "Essential and Plastic Works of Ductile Fracture in Asphalt Binders," *Journal of Transportation Research Record 1875*, 2004.

22. Tsai, B. and C. L. Monismith, Influence of Asphalt Binder Properties on the Fatigue Performance of Asphalt Concrete Pavements, *Journal of Association of Asphalt Paving Technologists*, Vol. 74, 2005, pp. 733-790.

23. Andersen, D. A., M. Y. Le Hir, M. Marasteanu, J. P. Planche, D. Martin, and G. Gaauthier, "Evaluation of fatigue criteria for asphalt binder." *Journal of Transportation Research Record 1766*, Transportation Research Board, Washington, D.C., 2001, pp. 48-56.

24. Johnson, C.M. and H.U. Bahia. Evaluation of an Accelerated Procedure for Fatigue Characterization of Asphalt Binders. Submitted for publication in Road Materials and Pavement Design. 2010.

25. Hintz, C., R. Velasquez, C. Johnson, and H. Bahia, Modification and Validation of the Linear Amplitude Sweep Test for Binder Fatigue Specification, *Transportation Research Board*, Washington, D.C., 2011.

26. Hintz, C. R. Velasquez, Z. Li, and H. Bahia, Effect of Oxidative Aging on Binder Fatigue Performance, *Journal of the Association of Asphalt Paving Technologists*, Tampa, FL, Vol. 80, 2011.

27. Andriescu, A., S. A. M. Hesp, and J. S. Youtcheff, "Essential and Plastic Works of Ductile Fracture in Asphalt Binders," *Journal of Transportation Research Record 1875*, 2004.

28. Gibson, N., X. Qi, A. Shenoy, G. Al-Khateeb, M. E. Kutay, A. Andriescu, K. Stuart, J. Youtcheff, and T. Harman, *Full-Scale Accelerated Performance Testing for Superpave and Structural Validation-Transportation Pooled Fund Study TPF-5(019) and SPR-2(174) Accelerated Pavement Testing of Crumb Rubber Modified Asphalt Pavements*, Federal Highway Administration, McLean, VA, August 2010.

29. Kim, Y., D. N. Little, and R. L. Lytton, "Use of dynamic mechanical analysis DMA to evaluate the fatigue and healing potential of asphalt binders in sand asphalt mixtures." *Journal of the Association of Asphalt Paving Technologists*, Vol. 71, 2002, pp.176-206.

30. Castelo Branco, V., E. Masad, A. Bhasin, and D. Little. Fatigue Analysis of Asphalt Mixtures Independent of Mode of Loading," *Transportation Research Record 2057, Journal of the Transportation Research Board,* 2008, pp. 149-156.

31. TxDOT Designation: Tex-248-F. Overlay Test. Construction Division, Texas Department of Transportation, 2009.

32. ASTM C1583 "*Standard Test Method for Tensile Strength of Concrete Surfaces and the Bond Strength or Tensile Strength of Concrete Repair and Overlay Materials by Direct Tension (Pull-off Method)*," 2013.

33. Zhou, F., S. Hu, and T. Scullion, Preliminary Results of Repeatability and Sensitivity Study on Overlay Tester for Crack Sealants, FHWA/TX-07/0-5457-1, Texas Transportation Institute, College Station, TX, 2007.

34. Kassem, E., Preparation and Testing of Fine Graded Asphalt Mixes by Means of the Dynamic Mechanical Analyzer (DMA), ACIM Lab DMA Procedure, 2011.

35. Sousa, P., E. Kassem, E. Masad, and D. Little, "New Design Method of Fine Aggregates Mixtures and Automated Method for Analysis of Dynamic Mechanical Characterization Data." The 90th Annual Meeting, Transportation Research Board, Washington, D.C., January 2011.

APPENDIX A
ASTM STANDARD E 691-11

This section describes ASTM E 691-11, “Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method,” which can be used to determine the precision of a test method. The standard specifies a procedure to develop a precision statement based on within-laboratory repeatability and between-laboratory reproducibility. The procedure follows three basic steps as described below:

- Planning the inter-laboratory study.
- Guiding the testing phase of the study.
- Analyzing the test result data.

PLANNING THE INTERLABORATORY STUDY

Important decisions regarding the design of the ILS are made at this stage: the number of laboratories to include in the study, the type of materials to test, and decisions regarding the test results, for example. According to the ASTM standard, an ILS should include no more than 30 and no less than 6 different laboratories.

The reason behind considering a maximum number of laboratories is to reduce the detrimental effects of the laboratories that produce bad results. Only laboratories having proper facilities, good testing equipment, and trained technicians should participate in the study. Proper training should be given to the laboratory technicians regarding the test method to be analyzed.

The decision regarding the number of laboratory results must be based on the accuracy in estimating the measure of repeatability. The number of results should be kept to a minimum because of time and cost considerations.

GUIDING THE TESTING PHASE OF THE STUDY

This phase of the ILS involves preparing the test specimens, distributing them to the various laboratories, keeping track of the testing progress, and checking the data collected from all the laboratories. Evaluators should prepare 50 percent more material than needed. The specimens should be randomly selected and sent to each laboratory. At regular intervals, the progress should be checked and the final data sheets from all the laboratories should be collected and reviewed for any irregularities.

ANALYZING THE TEST RESULTS

The main tasks of this phase are to:

- Determine whether the collected data are consistent enough to form the basis for a test method precision statement.
- Investigate and act on any data considered inconsistent.
- Obtain the precision statistics on which the precision statement can be based.

Consistency verification of the test results is important because the presence of outliers may lead to invalidation of the analysis. A simple one-way analysis of variance can check data consistency. For ease of analyzing the data, the results are represented in the form of a table where each row contains data from one laboratory for 3 binders and each column contains the data obtained from all laboratories for a certain parameter.

The data are then divided into cell statistics, intermediate statistics, precision statistics, and consistency statistics, as described in the following paragraphs.

Cell Statistics

- cell average, $\bar{x}$ – calculate the cell average for each laboratory using the following equation:

$$\bar{x} = \sum_{1}^{n} x/n \tag{1}$$

where:
$\bar{x}$ = the average of the test results in one cell.
x = the individual test results in one cell.
n = the number of test results per cell.

- Cell standard deviation, s – calculate the standard deviation of the test results in each cell using the following equation:

$$s = \sqrt{\sum_{1}^{n} (x - \bar{x})^2/(n-1)} \tag{2}$$

Intermediate Statistics

- Average of the cell averages, $\bar{\bar{x}}$ – calculate the average of all the cell averages for the one material using the equation below:

$$\bar{\bar{x}} = \sum_{1}^{n} \bar{x}/p \tag{3}$$

where:
$\bar{\bar{x}}$ = the average of the cell averages for one material.
$\bar{x}$ = the individual cell averages.
p = the number of laboratories in the ILS.

- Cell deviation, d –calculate cell deviation by subtracting the average of the cell averages from the cell average:

$$d = \bar{x} - \bar{\bar{x}} \tag{4}$$

- Standard deviation of the cell averages, $s_{\bar{x}}$ – standard deviation is calculated as follows:

$$s_{\bar{x}} = \sqrt{\sum_{1}^{p} d^2/(p-1)} \tag{5}$$

Precision Statistics

The fundamental precision statistics are the repeatability standard deviation and the reproducibility standard deviation.

- Repeatability standard deviation, s_r – calculate this statistic using the following equation:

$$s_r = \sqrt{\sum_{1}^{p} s^2/p} \tag{6}$$

where:
s_r = the repeatability standard deviation.
s = the cell standard deviation.
p = the number of laboratories.

- Reproducibility standard deviation, s_R– In this case, the larger of the values obtained from the equation below or the value of s_r is considered as reproducibility standard deviation:

$$s_R = \sqrt{(s_{\bar{x}})^2 + (s_r)^2(n-1)/n} \tag{7}$$

Consistency Statistics

- For each cell, the value of h is calculated using the equation given below:

$$h = d/s_{\bar{x}} \tag{8}$$

where:
h = the between-laboratory consistency statistic.
d = the cell deviation.
$s_{\bar{x}}$ = the standard deviation of the cell averages.

- For each cell, the value of k is calculated using the equation given below:

$$k = s/s_r \tag{9}$$

where:
k = within-laboratory consistency statistic.
s = the cell standard deviation for one laboratory.
s_r = the repeatability standard deviation of the material.

To facilitate easy representation, bar charts are prepared from the calculated values of h and k. The bar charts are prepared in two ways:

- Materials grouped by laboratory.
- Laboratories grouped by materials.

The critical values for 0.5 percent significance level for both h and k are recommended based on experience. When 1 percent significance level was used, most of the cells were flagged, and when 0.1 percent was used very few cells were flagged. The values that exceed the critical values are marked in the Appendix B tables.

In the plots by laboratory, there are usually three general patterns for the h plot:

- All laboratories have both positive and negative h values.
- The h values for individual laboratories are either positive or negative, and the number of negative laboratories equals the number of positive laboratories.
- One laboratory shows all positive h values while the other laboratories show all negative h values.

The first two patterns indicate that there is no variation in the test procedure and there is no need of any investigation, while the last type suggests the need for an investigation.

In the case of the k plots by laboratory, a k value from one of the laboratories either too small or too large when compared to those from the other laboratories suggests the need for an investigation. Small k values indicate some error in measurement, and high k values indicate a large variation in data.

The plots by material are necessary to compare the plots by laboratory type when the values of h and k for the plot by laboratory are close to the critical values. If the values of h and k for one laboratory are considerably different from the values for other laboratories then an investigation of the offset laboratory is suggested.

Once the data are analyzed and the flawed cells are identified, a detailed investigation must be conducted to determine the reason for the variation in the results. Retesting of the materials is an option, and h and k values will be determined again. With the corrected data, the 95 percent repeatability (r) and reproducibility (R) limits can be determined using the following equations:

$$r = 2.8\, s_r \tag{10}$$

$$R = 2.8\, s_R \tag{11}$$

APPENDIX B
DETAILED LABORATORY TEST RESULTS AND ANALYSIS

Table B-1. Initial Preparation of J_{nr100} for Binder A.

Initial Preparation of J_{nr100} Test Result Data for Binder A												
Lab	Test Results, x							$\bar{x}$	s	d	h	k
	1	2	3	4	5	6	7					
1	4.263	4.201	4.191	4.124	4.218	3.998	4.023	4.14543	0.10119	0.08797	0.45	0.91
2	4.026	4.095	4.114	4.115	4.124	4.135	4.251	4.12276	0.06689	0.06531	0.33	0.60
3	3.816	3.764	3.746	3.870	3.607	3.789	3.760	3.76464	0.08136	-0.29281	-1.48	0.73
4	4.265	4.073	4.522	4.254	4.154	4.076	4.035	4.19699	0.16931	0.13953	0.71	1.51
5	4.400	4.400	4.200	4.200	4.200	4.000	4.100	4.21429	0.14639	0.12546	0.68	1.22
	$\bar{\bar{x}}$	$s_{\bar{x}}$	s_r	s_R								
	4.089	0.185	0.120	0.216								

Table B-2. Initial Preparation of J_{nr3200} for Binder A.

Initial Preparation of J_{nr3200} Test Result Data for Binder A												
Lab	Test Results, x							$\bar{x}$	s	d	h	k
	1	2	3	4	5	6	7					
1	5.389	5.130	5.235	5.109	5.270	5.101	5.206	5.20571	0.10379	0.24537	0.83	0.79
2	4.836	4.974	4.946	5.013	4.977	5.186	5.126	5.00837	0.11608	0.04802	0.16	0.88
3	4.570	4.545	4.525	4.592	4.347	4.596	4.546	4.53137	0.08521	-0.42897	-1.44	0.64
4	5.169	4.938	5.475	5.174	5.008	4.987	4.921	5.09591	0.19586	0.13557	0.46	1.48
5	5.400	5.500	5.200	5.200	5.100	4.900	5.000	5.185714	0.21157	0.18030	0.652	1.40
	$\bar{\bar{x}}$	$s_{\bar{x}}$	s_r	s_R								
	5.005	0.276	0.151	0.310								

Table B-3. Initial Preparation of R_{100} for Binder A.

Initial Preparation of R_{100} Test Result Data for Binder A												
Lab	Test Results, x							$\bar{x}$	s	d	h	k
	1	2	3	4	5	6	7					
1	6.549	6.888	6.697	6.874	6.686	7.579	10.680	7.42186	1.47502	0.16838	0.21	1.56
2	7.110	7.813	7.153	8.036	7.817	9.079	6.535	7.64870	0.81933	0.39522	0.48	0.87
3	7.861	7.950	7.960	6.510	8.167	8.356	8.383	7.88394	0.63911	0.63047	0.77	0.68
4	5.772	6.156	5.467	6.019	5.478	7.047	6.477	6.05940	0.56735	-1.19407	-1.46	0.60
5	7.040	6.470	7.000	6.890	6.920	7.270	7.250	6.97714	0.26856	-0.22107	-0.31	0.31
	$\bar{\bar{x}}$	$s_{\bar{x}}$	s_r	s_R								
	7.198	0.719	0.854	1.069								

Table B-4. Initial Preparation of R_{3200} for Binder A.

Initial Preparation of R_{3200} Test Result Data for Binder A

Lab	Test Results, x							$\bar{x}$	s	d	h	k
	1	2	3	4	5	6	7					
1	1.132	1.326	1.263	1.359	1.349	1.344	1.174	1.27814	0.09185	-0.08973	-0.75	1.49
2	1.395	1.353	1.353	1.360	1.350	1.353	1.323	1.35501	0.02110	-0.01286	-0.11	0.34
3	1.505	1.518	1.550	1.505	1.655	1.530	1.519	1.54045	0.05309	0.17258	1.44	0.86
4	1.275	1.357	1.192	1.263	1.313	1.335	1.350	1.29788	0.05893	-0.06999	-0.59	0.96
5	1.250	1.160	1.310	1.350	1.340	1.450	1.390	1.321429	0.094592	-0.03716	-0.35	1.362
	$\bar{\bar{x}}$	$s_{\bar{x}}$	s_r	s_R								
	1.359	0.106	0.069	0.124								

Table B-5. Initial Preparation of J_{nrdiff} for Binder A.

Initial Preparation of J_{nrdiff} Test Result Data for Binder A

Lab	Test Results, x							$\bar{x}$	s	d	h	k
	1	2	3	4	5	6	7					
1	26.410	22.110	24.910	23.910	24.920	27.590	29.420	25.61000	2.42152	3.38728	1.46	1.50
2	20.115	21.485	20.223	21.829	20.701	25.425	20.570	21.47842	1.85124	-0.74430	-0.32	1.15
3	19.741	20.744	20.799	18.636	20.527	21.280	20.901	20.37529	0.90011	-1.84743	-0.80	0.56
4	21.191	21.252	21.062	21.621	20.560	22.336	21.967	21.42716	0.59541	-0.79556	-0.34	0.37
5	23.900	22.900	23.300	21.800	21.700	21.500	22.800	22.55714	0.90895	0.26754	0.13	0.61
	$\bar{\bar{x}}$	$s_{\bar{x}}$	s_r	s_R								
	22.290	2.010	1.502	2.444								

Table B-6. Initial Preparation of J_{nr100} for Binder B.

Initial Preparation of J_{nr100} Test Result Data for Binder B

Lab	Test Results, x							$\bar{x}$	s	d	h	k
	1	2	3	4	5	6	7					
1	1.231	1.228	1.230	1.150	1.164	1.163	1.200	1.19514	0.03570	-0.03624	-0.75	1.08
2	1.262	1.285	1.299	1.252	1.253	1.212	1.270	1.26177	0.02778	0.03038	0.63	0.84
3	1.231	1.162	1.203	1.194	1.151	1.170	1.190	1.18579	0.02729	-0.04560	-0.95	0.82
4	1.240	1.358	1.250	1.256	1.297	1.293	1.285	1.28284	0.04018	0.05146	1.07	1.21
5	1.300	1.300	1.300	1.300	1.300	1.300	1.300	1.30000	0.00000	0.054892	1.06	0.00
	$\bar{\bar{x}}$	$s_{\bar{x}}$	s_r	s_R								
	1.245	0.052	0.030	0.059								

Table B-7. Initial Preparation of J_{nr3200} for Binder B.

Initial Preparation of J_{nr3200} Test Result Data for Binder B

Lab	Test Results, x 1	2	3	4	5	6	7	$\bar{x}$	s	d	h	k
1	1.400	1.457	1.423	1.327	1.421	1.356	1.367	1.39300	0.04515	-0.00434	-0.08	1.16
2	1.419	1.452	1.445	1.407	1.405	1.358	1.426	1.41614	0.03123	0.01881	0.33	0.80
3	1.372	1.290	1.339	1.320	1.288	1.326	1.318	1.32177	0.02886	-0.07556	-1.32	0.74
4	1.411	1.548	1.415	1.432	1.478	1.465	1.460	1.45843	0.04686	0.06109	1.07	1.21
5	1.400	1.500	1.400	1.500	1.400	1.500	1.500	1.45714	0.05345	0.04784	0.85	1.267
	$\bar{\bar{x}}$	$s_{\bar{x}}$	s_r	s_R								
	1.409	0.056	0.042	0.069								

Table B-8. Initial Preparation of R_{100} for Binder B.

Initial Preparation of R_{100} Test Result Data for Binder B

Lab	Test Results, x 1	2	3	4	5	6	7	$\bar{x}$	s	d	h	k
1	11.410	11.960	12.230	11.290	14.450	13.230	13.110	12.52571	1.13428	0.84231	0.79	1.60
2	12.015	11.291	11.506	11.064	12.152	12.399	11.392	11.68852	0.49919	0.00511	0.00	0.70
3	11.966	12.191	12.284	11.737	12.883	13.422	11.918	12.34285	0.60162	0.65945	0.62	0.85
4	10.583	9.968	10.355	10.462	10.299	9.725	9.844	10.17654	0.32954	-1.50687	-1.41	0.47
5	11.540	11.050	11.420	10.830	11.370	10.800	11.560	11.22429	0.32603	-0.36730	-0.39	0.50
	$\bar{\bar{x}}$	$s_{\bar{x}}$	s_r	s_R								
	11.592	0.947	0.650	1.122								

Table B-9. Initial Preparation of R_{3200} for Binder B.

Initial Preparation of R_{3200} Test Result Data for Binder B

Lab	Test Results, x 1	2	3	4	5	6	7	$\bar{x}$	s	d	h	k
1	4.415	4.166	4.037	5.137	5.084	5.182	5.194	4.74500	0.51746	-0.16623	-0.54	1.80
2	4.899	4.712	4.919	4.959	4.995	5.153	4.871	4.92964	0.13350	0.01842	0.06	0.47
3	5.209	5.350	5.303	5.293	5.506	5.299	5.366	5.33251	0.09154	0.42129	1.38	0.32
4	4.897	4.329	4.813	4.713	4.552	4.555	4.605	4.63774	0.18891	-0.27348	-0.89	0.66
5	4.620	4.480	4.680	4.520	4.640	4.480	4.530	4.56429	0.08121	-0.27755	-0.90	0.31
	$\bar{\bar{x}}$	$s_{\bar{x}}$	s_r	s_R								
	4.842	0.307	0.259	0.390								

Table B-10. Initial Preparation of J_{nrdiff} for Binder B.

Initial Preparation of J_{nrdiff} Test Result Data for Binder B												
Lab	Test Results, x							$\bar{x}$	s	d	h	k
	1	2	3	4	5	6	7					
1	13.690	18.650	15.690	15.350	22.090	16.650	13.910	16.57571	2.95623	3.08300	1.37	1.87
2	12.476	13.020	11.265	12.387	12.114	12.050	12.339	12.23588	0.53194	-1.25684	-0.56	0.34
3	11.401	11.032	11.365	10.582	11.876	13.314	10.748	11.47401	0.92038	-2.01871	-0.90	0.58
4	13.792	13.934	13.247	14.038	13.886	13.330	13.569	13.68527	0.30810	0.19255	0.09	0.20
5	15.200	13.900	14.300	13.700	13.900	13.200	15.900	14.30000	0.93630	0.64583	0.33	0.64
	$\bar{\bar{x}}$	$s_{\bar{x}}$	s_r	s_R								
	13.654	1.982	1.472	2.406								

Table B-11. Initial Preparation of J_{nr100} for Binder C.

Initial Preparation of J_{nr100} Test Result Data for Binder C												
Lab	Test Results, x							$\bar{x}$	s	d	h	k
	1	2	3	4	5	6	7					
1	0.132	0.132	0.128	0.131	0.131	0.133	0.129	0.13083	0.00177	-0.00052	-0.04	0.26
2	0.137	0.144	0.147	0.141	0.141	0.142	0.121	0.13886	0.00858	0.00751	0.62	1.25
3	0.115	0.112	0.100	0.116	0.122	0.119	0.118	0.11453	0.00705	-0.01682	-1.39	1.02
4	0.133	0.137	0.143	0.152	0.151	0.139	0.133	0.14119	0.00793	0.00983	0.82	1.15
5	0.130	0.140	0.140	0.130	0.130	0.130	0.150	0.13571	0.00787	0.00349	0.33	1.11
	$\bar{\bar{x}}$	$s_{\bar{x}}$	s_r	s_R								
	0.132	0.011	0.007	0.012								

Table B-12. Initial Preparation of J_{nr3200} for Binder C.

Initial Preparation of J_{nr3200} Test Result Data for Binder C												
Lab	Test Results, x							$\bar{x}$	s	d	h	k
	1	2	3	4	5	6	7					
1	0.147	0.141	0.140	0.133	0.133	0.136	0.137	0.13827	0.00500	0.00154	0.10	0.73
2	0.135	0.146	0.146	0.137	0.144	0.134	0.136	0.13964	0.00547	0.00291	0.19	0.80
3	0.116	0.113	0.102	0.118	0.126	0.122	0.118	0.11639	0.00770	-0.02034	-1.35	1.13
4	0.149	0.145	0.150	0.162	0.167	0.148	0.147	0.15262	0.00848	0.01589	1.06	1.24
5	0.140	0.140	0.150	0.140	0.140	0.140	0.140	0.14143	0.00378	0.00376	0.29	0.60
	$\bar{\bar{x}}$	$s_{\bar{x}}$	s_r	s_R								
	0.138	0.013	0.006	0.014								

Table B-13. Initial Preparation of R_{100} for Binder C.

Initial Preparation of R_{100} Test Result Data for Binder C												
Lab	Test Results, x							$\bar{x}$	s	d	h	k
	1	2	3	4	5	6	7					
1	89.490	89.300	89.460	88.990	88.780	88.540	87.850	88.91571	0.58802	-0.66948	-0.92	1.27
2	89.444	89.085	89.314	89.230	89.193	89.176	90.767	89.45848	0.58804	-0.12671	-0.17	1.27
3	90.718	90.734	91.142	90.665	90.096	90.543	90.416	90.61648	0.32116	1.03129	1.42	0.69
4	89.568	89.563	89.157	88.957	89.165	89.392	89.649	89.35010	0.26148	-0.23510	-0.32	0.56
5	89.850	88.560	89.310	89.790	89.280	89.610	88.070	89.21000	0.66370	-0.30015	-0.46	1.30
	$\bar{\bar{x}}$	$s_{\bar{x}}$	s_r	s_R								
	89.510	0.651	0.511	0.805								

Table B-14. Initial Preparation of R_{3200} for Binder C.

Initial Preparation of R_{3200} Test Result Data for Binder C												
Lab	Test Results, x							$\bar{x}$	s	d	h	k
	1	2	3	4	5	6	7					
1	88.560	88.600	88.940	88.930	88.870	88.540	87.600	88.57714	0.46529	-0.77437	-0.84	1.21
2	89.616	88.930	89.106	89.702	89.152	89.800	89.559	89.40921	0.33953	0.05769	0.06	0.88
3	90.751	90.811	91.179	90.658	89.961	90.473	90.528	90.62289	0.37242	1.27138	1.39	0.97
4	88.929	89.109	88.925	88.421	88.215	88.821	89.157	88.79682	0.35169	-0.55469	-0.60	0.91
5	89.030	89.100	88.670	88.900	89.100	89.030	88.960	88.97000	0.15055	-0.30521	-0.38	0.43
	$\bar{\bar{x}}$	$s_{\bar{x}}$	s_r	s_R								
	89.275	0.813	0.351	0.876								

Table B-15. Initial Preparation of J_{nrdiff} for Binder C.

Initial Preparation of J_{nrdiff} Test Result Data for Binder C												
Lab	Test Results, x							$\bar{x}$	s	d	h	k
	1	2	3	4	5	6	7					
1	11.670	6.753	9.439	0.988	1.563	2.695	6.787	5.69933	4.08771	1.63913	0.47	1.07
2	-1.623	1.181	-0.210	-2.966	2.164	-5.552	12.652	0.80647	5.82941	-3.25373	-0.94	1.52
3	1.245	0.761	1.411	1.642	3.254	2.387	0.524	1.60348	0.94686	-2.45671	-0.71	0.25
4	11.795	5.661	5.218	6.743	10.354	6.657	10.492	8.13150	2.66545	4.07131	1.18	0.70
5	10.500	-2.800	8.300	10.800	3.700	8.100	-5.700	4.70000	6.59267	0.51184	0.17	1.46
	$\bar{\bar{x}}$	$s_{\bar{x}}$	s_r	s_R								
	4.188	3.009	4.520	5.154								

Table B-16. h Value for J_{nr100}.

Table J_{nr100} -h[A]			
Laboratory	A	B	C
1	0.31	-0.96	-0.13
2	0.18	0.32	0.62
3	-1.75	-1.15	-1.67
4	0.58	0.73	0.84
5	0.68	1.06	0.33
[A] critical value=1.74			

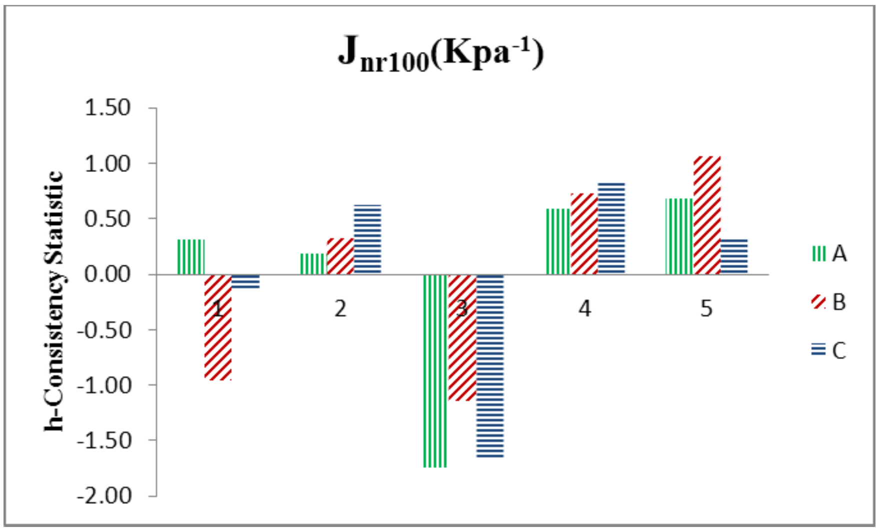


Figure B-1. h Value for J_{nr100} -- Materials within Laboratories.

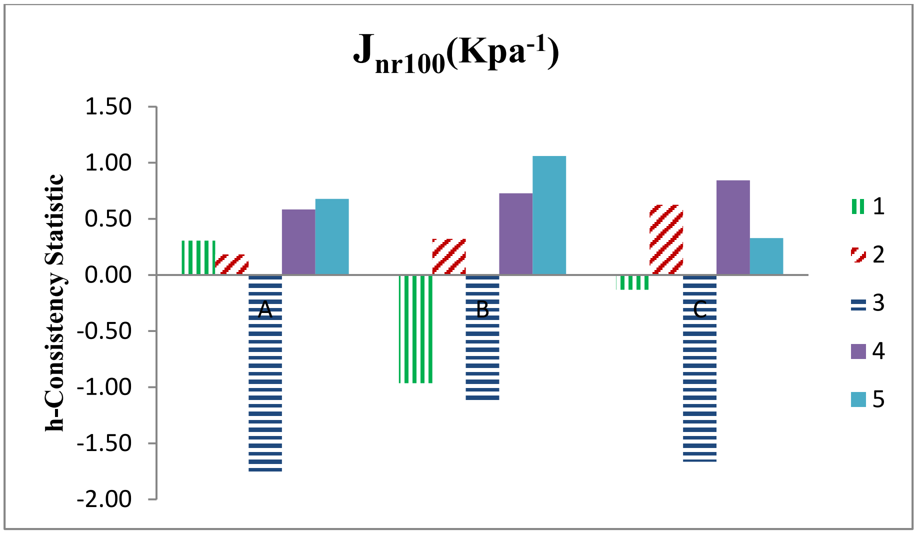


Figure B-2. h Value for J_{nr100} -- Laboratories within Materials.

Table B-17. h Value for J_{nr3200}.

Table	J_{nr3200} -h[A]		
Laboratory	A	B	C
1	0.72	-0.29	0.05
2	0.01	0.12	0.15
3	-1.72	-1.55	-1.61
4	0.33	0.87	1.13
5	0.65	0.85	0.29
[A] critical value=1.74			

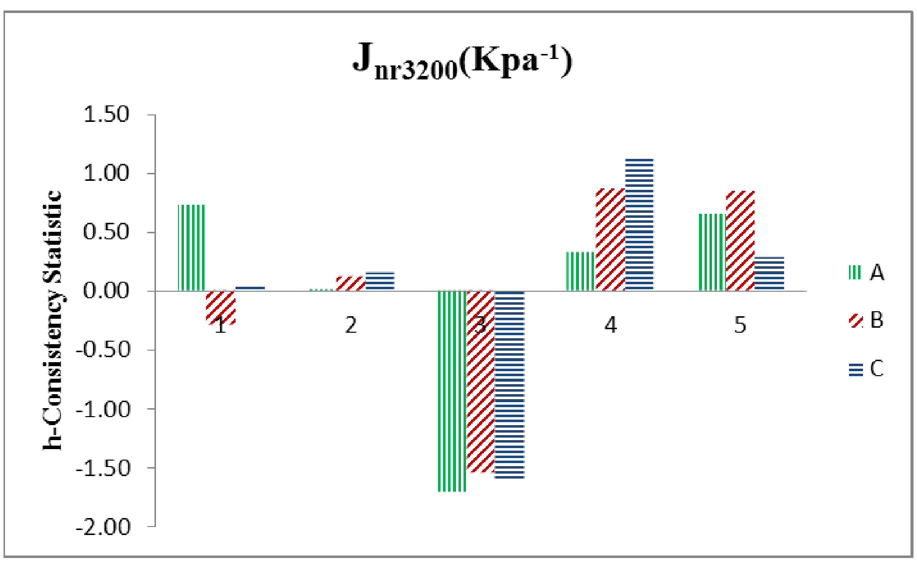


Figure B-3. h Value for J_{nr3200} -- Materials within Laboratories.

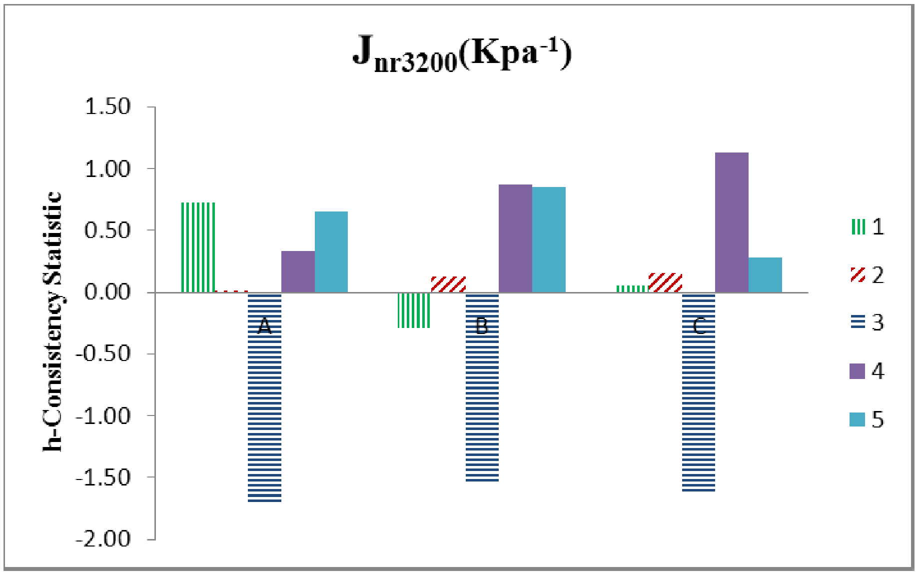


Figure B-4. h Value for J_{3200} -- Laboratories within Materials.

Table B-18. h Value for R_{100}.

Table R_{100} -h[A]			
Laboratory	A	B	C
1	0.31	0.99	-0.91
2	0.63	0.10	-0.08
3	0.95	0.79	1.70
4	-1.58	-1.49	-0.25
5	-0.31	-0.39	-0.46
[A] critical value=1.74			

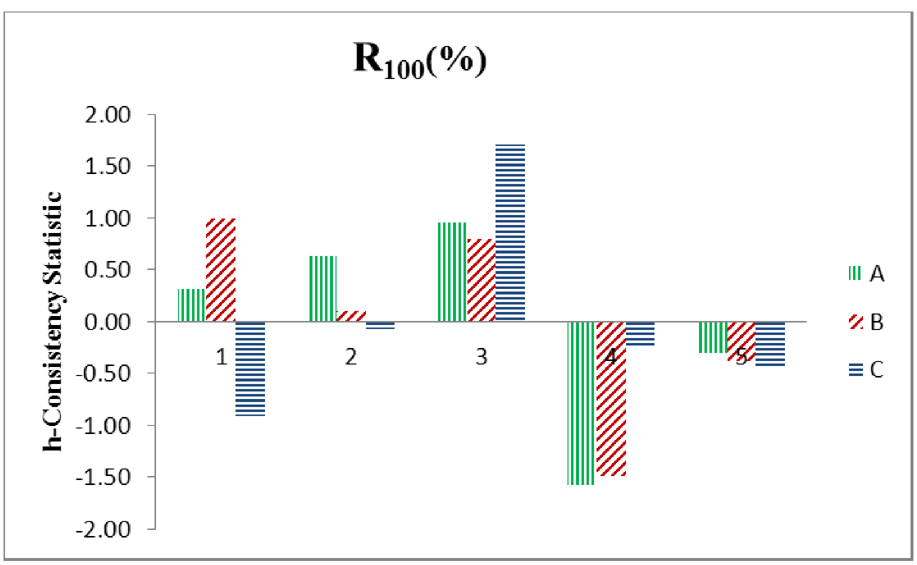


Figure B-5. h Value for R_{100} -- Materials within Laboratories.

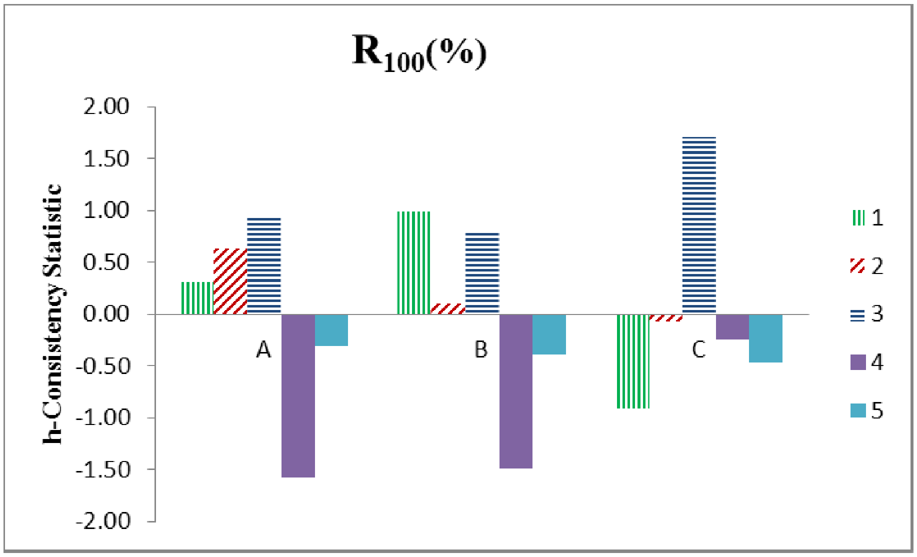


Figure B-6. h Value for R_{100} -- Laboratories within Materials.

Table B-19. h Value for R_{3200}.

Table R_{3200} -h[A]			
Laboratory	A	B	C
1	-0.76	-0.32	-0.86
2	-0.03	0.29	0.16
3	1.72	1.60	1.66
4	-0.57	-0.67	-0.59
5	-0.35	-0.90	-0.38
[A] critical value=1.74			

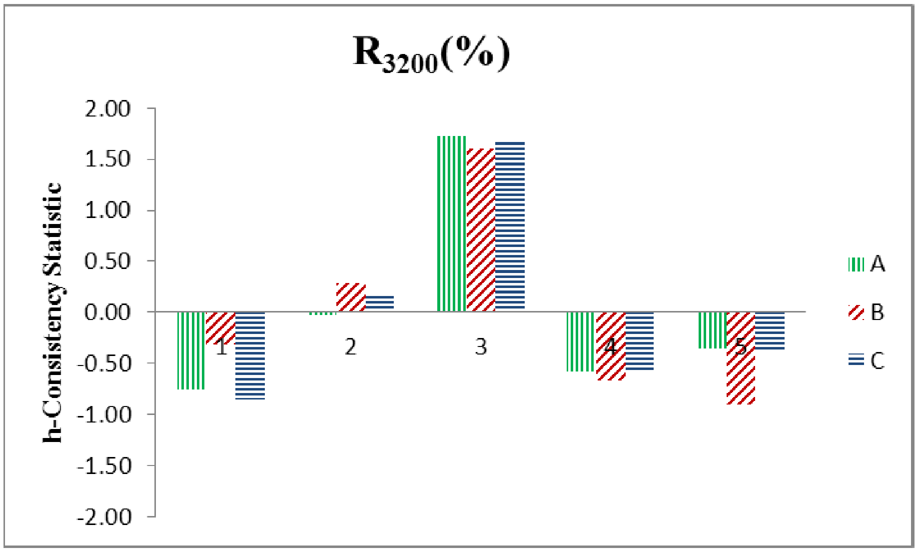


Figure B-7. h Value for R_{3200} -- Materials within Laboratories.

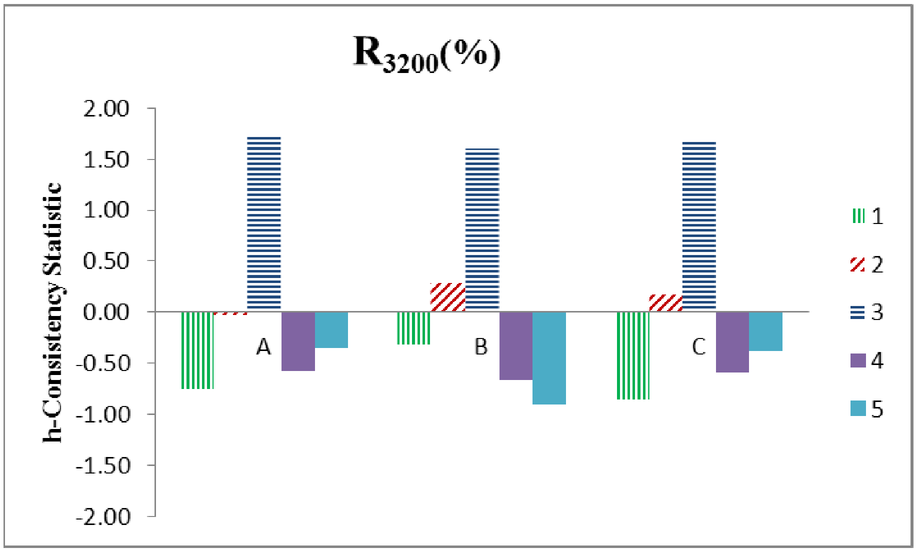


Figure B-8. h Value for R_{3200} -- Laboratories within Materials.

Table B-20. h Value for J_{nrdiff}.

Table J_{nrdiff} -h[A]			
Laboratory	A	B	C
1	1.65	1.47	0.50
2	-0.40	-0.72	-1.12
3	-0.95	-1.10	-0.86
4	-0.43	0.02	1.31
5	0.13	0.33	0.17
[A] critical value=1.74			

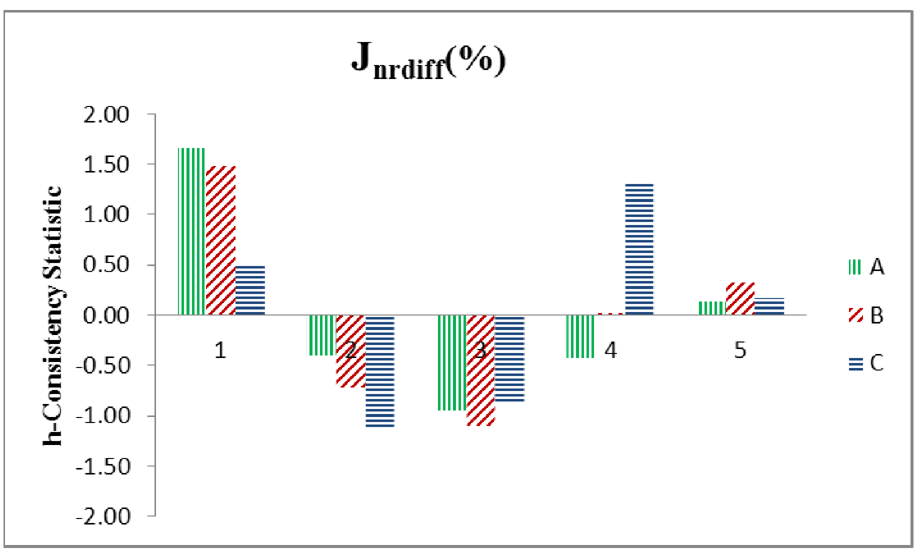


Figure B-9. h Value for J_{nrdiff} -- Materials within Laboratories.

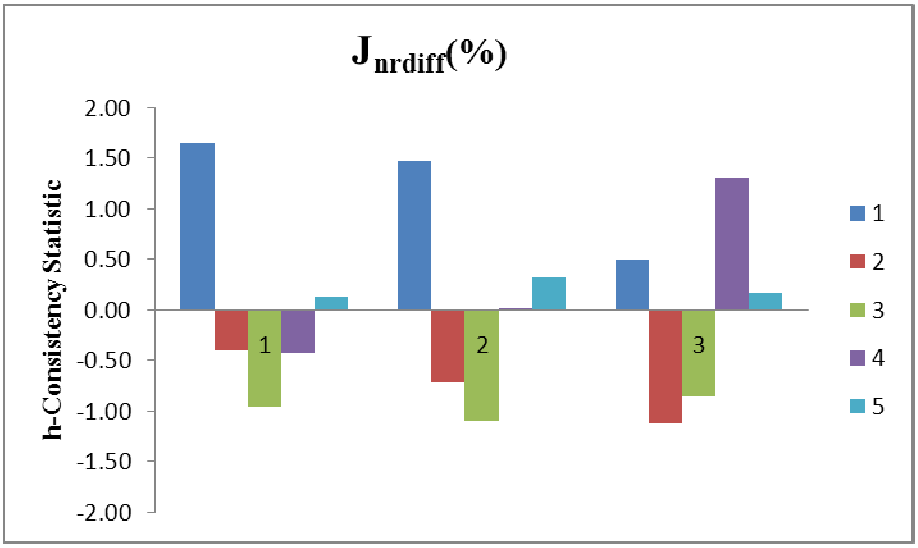


Figure B-10. h Value for J_{nrdiff} -- Laboratories within Materials.

Table B-21. k Value for J_{nr100}.

Table J_{nr100} -k[A]			
Laboratory	A	B	C
1	0.85	1.20	0.25
2	0.56	0.94	1.21
3	0.68	0.92	0.99
4	1.42	1.35	1.12
5	1.22	0.00	1.11
[A] critical value=1.60			

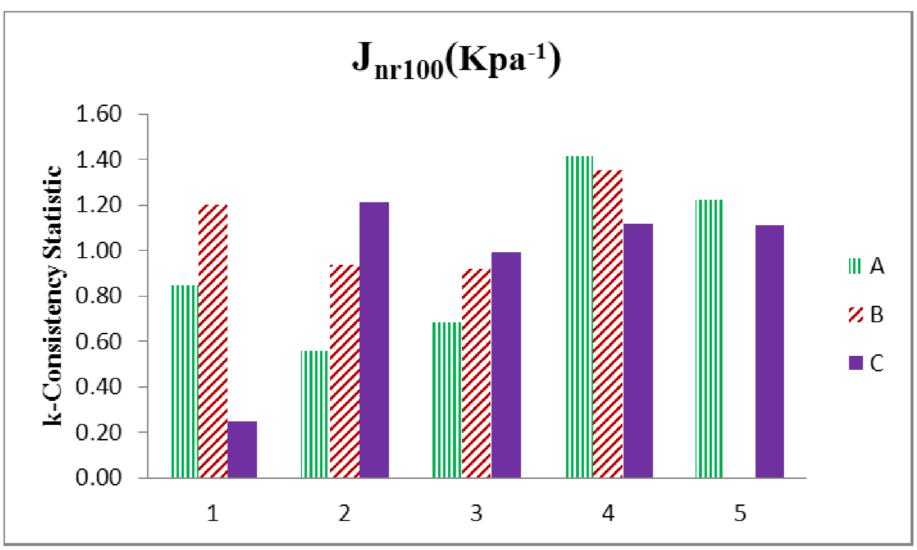


Figure B-11. k Value for J_{nr100} -- Materials within Laboratories.

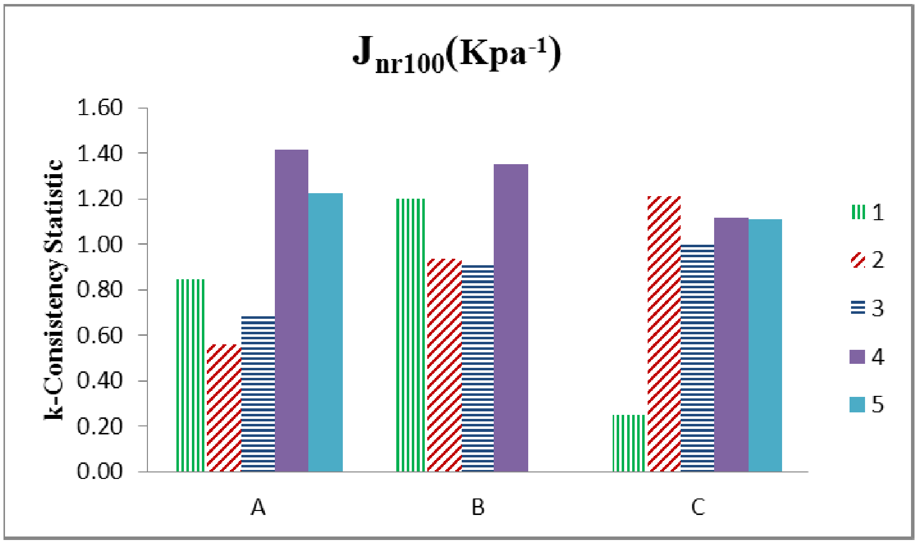


Figure B-12. k Value for J_{nr100} -- Laboratories within Materials.

Table B-22. k Value for J_{nr3200}.

Table	J_{nr3200} -k[A]		
Laboratory	A	B	C
1	0.69	1.07	0.79
2	0.77	0.74	0.86
3	0.56	0.68	1.22
4	1.29	1.11	1.34
5	1.40	1.27	0.60
[A] critical value=1.60			

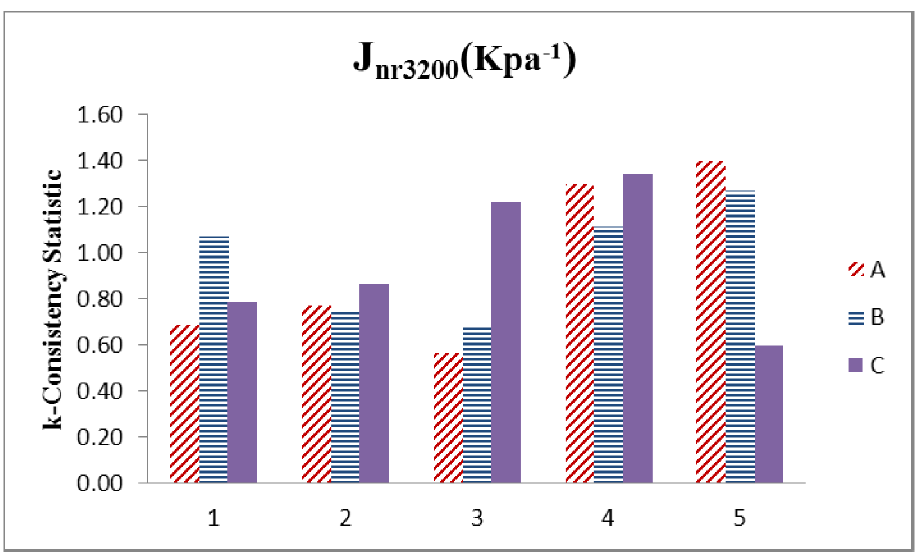


Figure B-13. k Value for J_{nr3200} -- Materials within Laboratories.

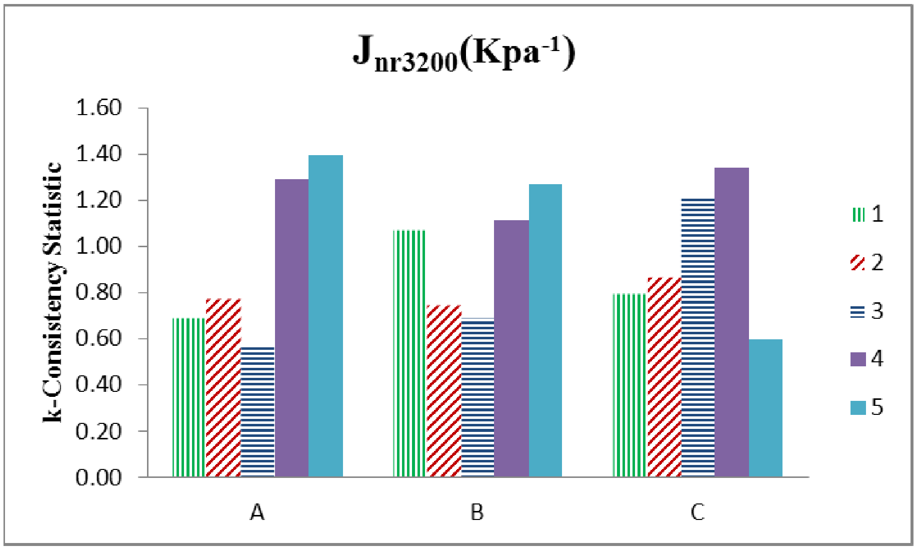


Figure B-14. k Value for J_{nr3200} -- Laboratories within Materials.

Table B-23. k Value for R_{100}.

Table	R_{100} -k[A]		
Laboratory	A	B	C
1	1.73	1.74	1.15
2	0.96	0.77	1.15
3	0.75	0.93	0.63
4	0.66	0.51	0.51
5	0.31	0.50	1.30
[A] critical value=1.60			

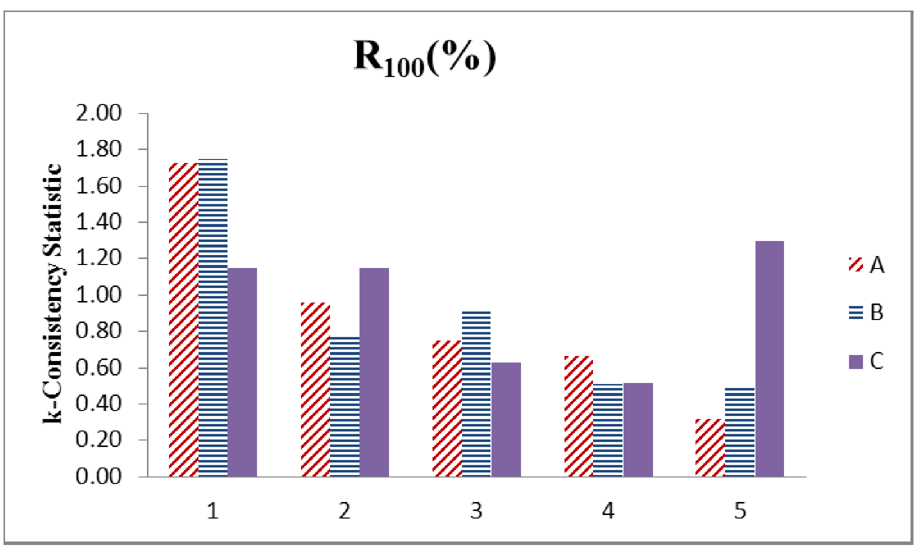


Figure B-15. k Value for R_{100} -- Materials within Laboratories.

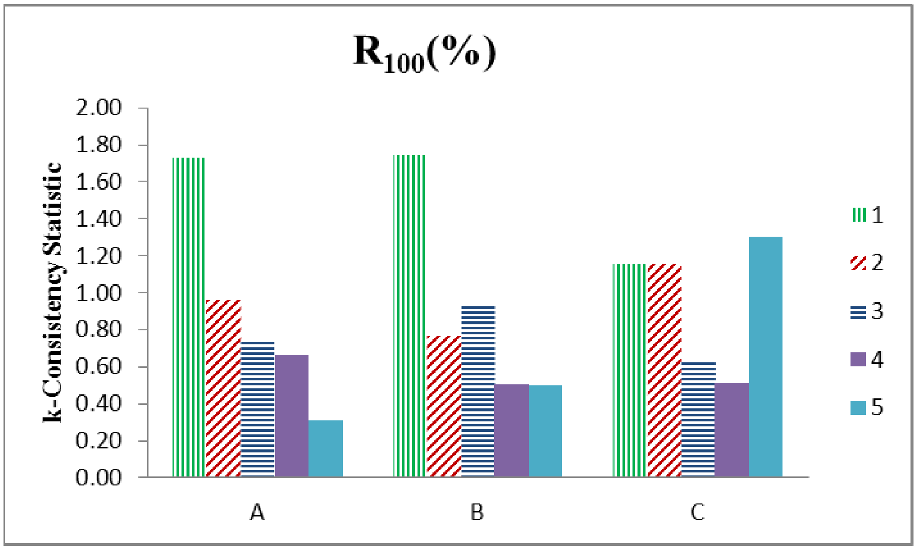


Figure B-16. k Value for R_{100} -- Laboratories within Materials.

Table B-24. k Value for R_{3200}.

Table	R_{3200} -k[A]		
Laboratory	A	B	C
1	1.32	2.00	1.32
2	0.30	0.51	0.97
3	0.76	0.35	1.06
4	0.85	0.73	1.00
5	1.36	0.31	0.43
[A] critical value=1.60			

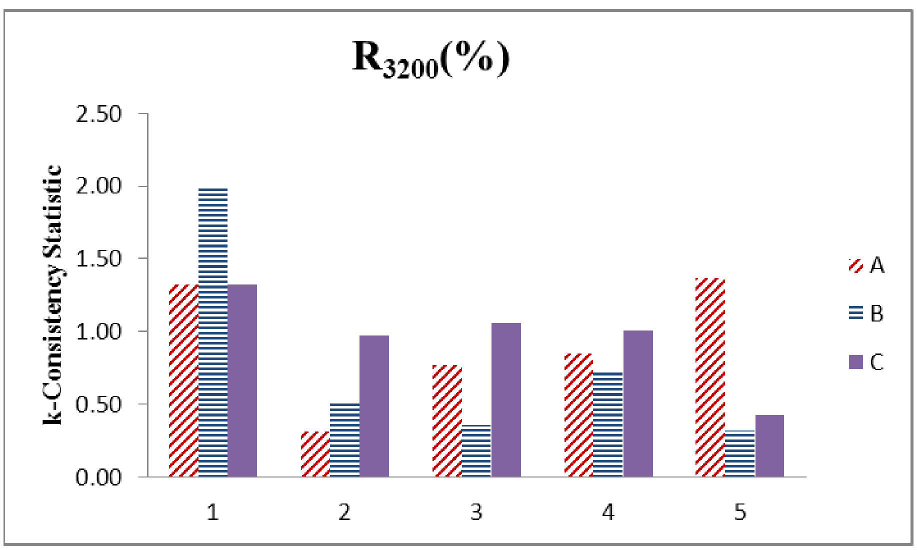


Figure B-17. k Value for R_{3200} -- Materials within Laboratories.

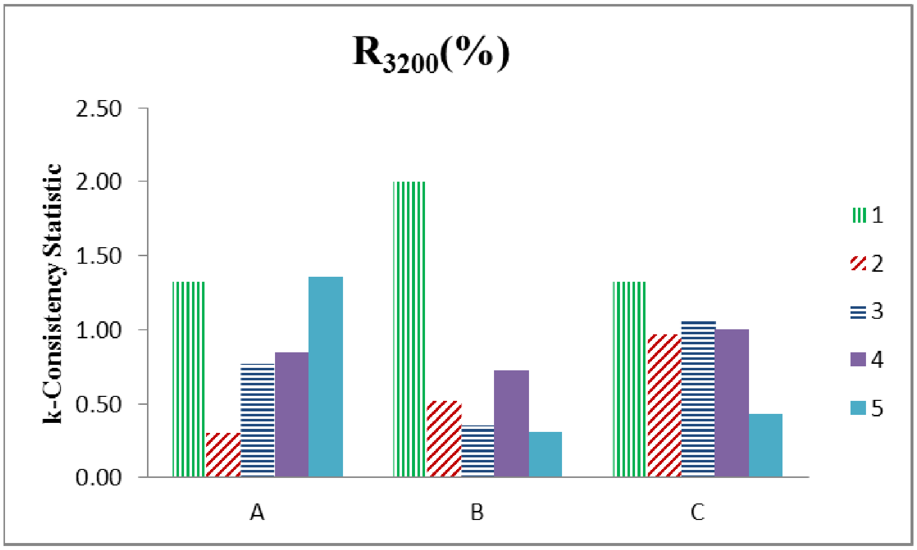


Figure B-18. k Value for R_{3200} -- Laboratories within Materials.

Table B-25. k Value for J_{nrdiff}.

Table J_{nrdiff} -k[A]			
Laboratory	A	B	C
1	1.61	2.01	0.90
2	1.23	0.36	1.29
3	0.60	0.63	0.21
4	0.40	0.21	0.59
5	0.61	0.64	1.46
[A] critical value=1.60			

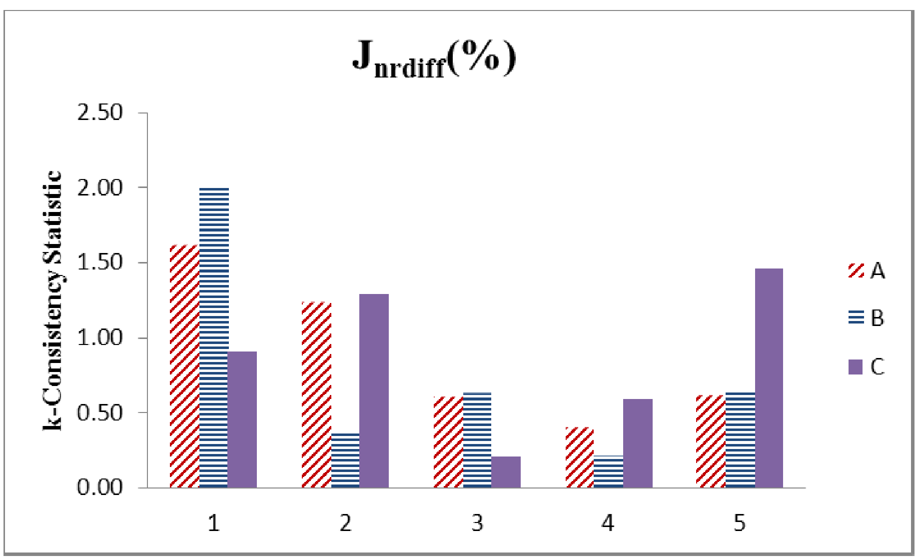


Figure B-19. k Value for J_{nrdiff} -- Materials within Laboratories.

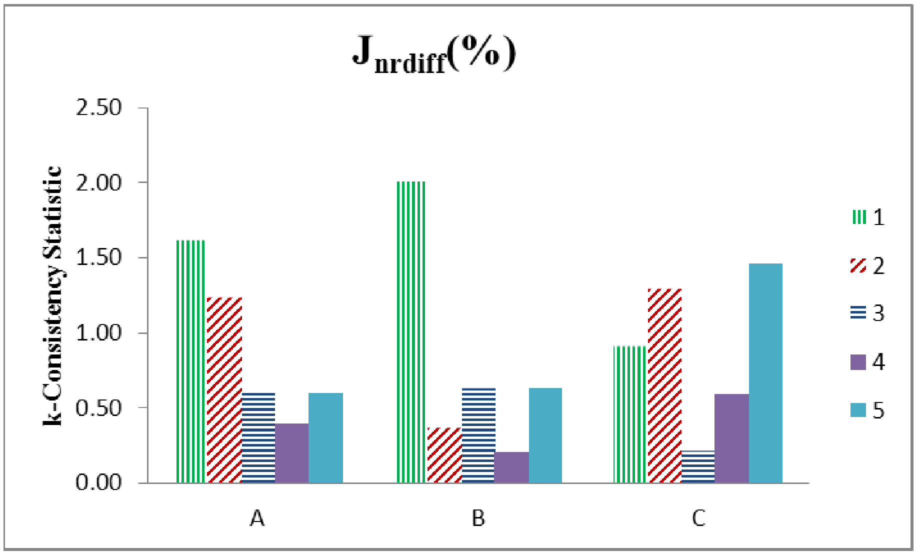


Figure B-20. k Value for J_{nrdiff} -- Laboratories within Materials.