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Investigation of Wet-Process Asphalt Rubber Binder Testing with Modified Dynamic Shear Rheometer Equipment: Interim Report on Screening Tests

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Investigation of Wet-Process Asphalt rubber binder Testing with Modified Dynamic Shear Rheometer

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Abstract:
In the United States, the Superpave Asphalt Binder Performance Grading (PG) system proposed by the Strategic Highway Research Program (SHRP) is the most common method used to characterize the performance-related properties of conventional and polymer-modified asphalt binders. Dynamic modulus (G*) and phase angle (δ) are the two main binder properties and they are measured using a dynamic shear rheometer (DSR) with parallel plate geometry and either a 1 mm or 2 mm gap between the plates. Since these Superpave parameters were developed for binders that do not contain additives or particulates, the California Department of Transportation (Caltrans) does not use them as asphalt rubber binder specification criteria. Instead, penetration and viscosity are used as acceptance of quality control; however, these parameters do not necessarily provide a satisfactory link between the measured binder properties and potential performance in the field over a range of operating temperatures.

In California, current specifications require that crumb rubber particles used to produce asphalt rubber binder in the "wet process" must be smaller than 2.36 mm (i.e., 100 percent passing the #8 sieve), and typically these particles vary in size between 1 mm and 2 mm. As a consequence, when the parallel plate geometry is used to test this type of binder, the larger rubber particles can contact the plates; if this occurs, the rubber particle rheology can potentially dominate the results, which in turn may not be representative of the modified binder as a whole. To address this problem, a potentially more appropriate DSR testing protocol using concentric cylinder geometry was investigated in this study to explore an alternative means of determining the performance properties of asphalt rubber binders. In the first phase of the study, documented in this technical memorandum, a series of tests were undertaken to compare the two geometries and to assess which binder properties influence the results from the testing approaches.

The interim results indicate that there is no significant difference between the concentric cylinder and parallel plate geometries in terms of the G*/sinδ after testing on a range of different binders and asphalt rubber binders with finer crumb rubber particle sizes (i.e. <250 µm). However, the correlations between results from the geometries were increasingly weaker with increasing crumb rubber particle size, indicating some potential influence of larger sizes on the results of the testing using parallel plates. The concentric cylinder geometry resulted in relatively lower values of G*/sinδ compared to samples tested with the parallel plate geometry. This difference is provisionally attributed to large rubber particles touching both plates, and to edge effects issues. The proposed alternative approach to measuring the rheological properties of asphalt rubber binder is considered feasible, and that with its use the edge effect and trimming issues can be eliminated. However, the concentric cylinder method requires a longer testing time and a larger binder sample than the parallel plate test method. The testing will be continued to develop proposed revised quality control procedures for testing asphalt rubber binders used on Caltrans projects.

Keywords:
Wet-process Rubberized Binder, Concentric Cylinder, Asphalt Rubber, Performance Grade Testing

Proposals for implementation:
None

Related documents:
None

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DISCLAIMER STATEMENT

PROJECT OBJECTIVES

The objective of this study is to recommend appropriate contract acceptance criteria for wet-process asphalt rubber binders using current Superpave PG equipment. This objective will be met by completing the following tasks:

1. Review relevant literature on the topic. Contact DSR equipment manufacturers and discuss test requirements and alternative geometries (specifically concentric cylinder [or cup-and-bob]) that can be used for these tests.
2. Collect samples of asphalt binder, crumb rubber particles, and extender oil for laboratory preparation of asphalt rubber binder. On completion of initial screening tests, identify completed and current projects where rubberized binder samples can be collected for additional testing.
3. Prepare laboratory conditioned samples for testing with a DSR.
4. Evaluate the use and ability of the alternative concentric cylinder DSR geometry to provide realistic, repeatable results for conventional, polymer-modified (PM), and tire rubber-modified (TR) binders that are comparable to results from the same tests undertaken using conventional parallel plate geometry.
5. Compare the two DSR geometries for testing asphalt rubber binder containing crumb rubber particles of various sizes.
6. Evaluate the effects of different crumb rubber particle and asphalt rubber binder properties on DSR test results.
7. Suggest contract acceptance criteria for wet-process asphalt rubber binders using a DSR with appropriate geometry.
8. Prepare a report documenting the research, with recommendations for specification language and, if required, recommendations for further research on characterizing wet-process asphalt rubber binders and correlating test results against laboratory rutting and cracking test results as well as field performance.

This technical memorandum provides an update on work completed to date on Tasks 1 through 6.

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- The staff of Anton Paar testing equipment
- The UCPRC laboratory staff

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EXECUTIVE SUMMARY

This technical memorandum documents the first phase of a study to investigate alternative test methods of measuring the performance properties of asphalt rubber binders. In the United States, the Superpave Asphalt Binder Performance Grading (PG) system proposed by the Strategic Highway Research Program (SHRP) is the most common method used to characterize the performance-related properties of conventional and polymer-modified asphalt binders. Dynamic modulus (G*) and phase angle (δ) are the two main binder properties and they are measured using a dynamic shear rheometer (DSR) with parallel plate geometry and either a 1 mm or 2 mm gap between the plates. Since these Superpave parameters were developed for binders that do not contain additives or particulates, the California Department of Transportation (Caltrans) does not use them as asphalt rubber binder specification criteria. Instead, penetration and viscosity are used as acceptance of quality control; however, these parameters do not necessarily provide a satisfactory link between the measured binder properties and potential performance in the field over a range of operating temperatures and are consequently deemed to be an insufficient measure of performance compared to the testing requirements for conventional, polymer-modified, and tire rubber-modified binders. This phase of the study consisted of preliminary testing to compare two different dynamic shear rheometer (DSR) geometries, namely parallel plate and concentric cylinder, with a view to making recommendations about adopting similar testing criteria for California-produced asphalt rubber binders to those currently used for conventional and other modified binders.

The high temperature properties of conventional and other modified binders are typically assessed through testing with a DSR that has a parallel plate geometry, with the gap size between the plates dependent on the size of any particulates in the binder. According to the AASHTO/ASTM standard, the gap size should be at least four times larger than the maximum particle size for testing an asphalt binder containing particulates. A 2.0 mm gap size is typically used for testing asphalt rubber binders and therefore the maximum size of the rubber particles should not exceed 0.5 mm (or 500 µm [#30]). However, Caltrans specifications allow crumb rubber particles of up to 2.36 mm in size (i.e., passing the #8 sieve), which is considerably larger than AASHTO/ASTM recommended maximum for a 2 mm gap size between the parallel plates. Consequently, the appropriateness of the parallel plate geometry for testing California-produced asphalt rubber binders is questioned, given that the rheology of the large rubber particles may dominate the outcome and give misleading results for the binder properties. This study therefore assessed an alternative geometry, namely the concentric cylinder, which can accommodate larger particles in the asphalt binder. The two geometries were compared using conventional, polymer-modified (PM), tire rubber-modified (TR), and asphalt rubber binders.

The results obtained from testing the same conventional, polymer-modified, and tire rubber-modified binders with concentric cylinder and parallel plate geometries in a DSR were statistically similar. The results obtained from testing the asphalt rubber binders with three different crumb rubber particle size ranges (180 µm to 250 µm, 250 µm to 425 µm, and 425 µm to 850 µm [#40 to #20, #60 to #40, and #80 to #60, respectively]), showed a strong correlation between the two testing geometries for the finer particle size ranges, but increasingly poorer correlations with increasing particle size. These poorer correlations in the larger size ranges were attributed in part to the increasing influence of the proximity of the larger rubber particles to the plates. An assessment of the effects of different crumb rubber properties on the properties of asphalt rubber binders determined using parallel plate geometry indicated that both production method (i.e., crushing at ambient or cryogenic temperatures) and whether an asphalt modifier (i.e., extender oil) was used influenced the performance properties of the asphalt rubber binder, with the use of asphalt modifiers having the largest influence.

Based on these results obtained to date, the concentric cylinder geometry is considered to be a potentially appropriate alternative geometry to parallel plates for quantifying the properties of California-produced asphalt rubber binders, and specifically for assessing the performance properties of binders containing crumb rubber particles larger than 250 µm (i.e., particles retained on the #60 sieve).

Initial results support the continuation of testing to assess the appropriateness of using the concentric cylinder geometry for measuring the performance properties of asphalt rubber binders that are produced in California using a wet process with crumb rubber particles that exceed 0.25 mm (#60 mesh) in size. This testing should be in line with the original workplan and objectives prepared for this project, and should investigate additional binder sources, the development of appropriate binder aging protocols, the development of a suitable low temperature testing method, the repeatability and reproducibility values of any proposed test methods, and the applicability of the results to the actual performance properties of mixes produced with asphalt rubber binders.

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LIST OF ABBREVIATIONS

AASHTO	American Association of State Highway and Transportation Officials
AMRL	AASHTO Materials Reference Laboratory
ANOVA	Analysis of variance
ASTM	American Society for Testing and Materials
BBR	Bending beam rheometer
Caltrans	California Department of Transportation
C_{ss}	Conversion factor
DGAC	Dense-graded asphalt concrete
DSR	Dynamic shear rheometer
G*	Dynamic modulus
HSD	Honest Significant Difference
M-E	Mechanistic empirical
PAV	Pressure aging vessel
PG	Performance grade
PM	Polymer-modified
PPRC	Partnered Pavement Research Center
RAC	Rubberized asphalt concrete
RPM	Revolutions per minute
RTFO	Rolling thin film oven
SHRP	Strategic Highway Research Program
TFO	Thin film oven
TR	Tire rubber
UCPRC	University of California Pavement Research Center
δ	Phase angle

CONVERSION FACTORS

SI* (MODERN METRIC) CONVERSION FACTORS				
APPROXIMATE CONVERSIONS TO SI UNITS				
Symbol	**When You Know**	**Multiply By**	**To Find**	**Symbol**
		LENGTH		
in	inches	25.4	Millimeters	mm
ft	feet	0.305	Meters	m
yd	yards	0.914	Meters	m
mi	miles	1.61	Kilometers	Km
		AREA		
in^2	square inches	645.2	Square millimeters	mm^2
ft^2	square feet	0.093	Square meters	m^2
yd^2	square yard	0.836	Square meters	m^2
ac	acres	0.405	Hectares	ha
mi^2	square miles	2.59	Square kilometers	km^2
		VOLUME		
fl oz	fluid ounces	29.57	Milliliters	mL
gal	gallons	3.785	Liters	L
ft^3	cubic feet	0.028	cubic meters	m^3
yd^3	cubic yards	0.765	cubic meters	m^3
		NOTE: volumes greater than 1000 L shall be shown in m^3		
		MASS		
oz	ounces	28.35	Grams	g
lb	pounds	0.454	Kilograms	kg
T	short tons (2000 lb)	0.907	megagrams (or "metric ton")	Mg (or "t")
		TEMPERATURE (exact degrees)		
°F	Fahrenheit	5 (F-32)/9 or (F-32)/1.8	Celsius	°C
		ILLUMINATION		
fc	foot-candles	10.76	Lux	lx
fl	foot-Lamberts	3.426	candela/m^2	cd/m^2
		FORCE and PRESSURE or STRESS		
lbf	poundforce	4.45	Newtons	N
lbf/in^2	poundforce per square inch	6.89	Kilopascals	kPa
APPROXIMATE CONVERSIONS FROM SI UNITS				
Symbol	**When You Know**	**Multiply By**	**To Find**	**Symbol**
		LENGTH		
mm	millimeters	0.039	Inches	in
m	meters	3.28	Feet	ft
m	meters	1.09	Yards	yd
km	kilometers	0.621	Miles	mi
		AREA		
mm^2	square millimeters	0.0016	square inches	in^2
m^2	square meters	10.764	square feet	ft^2
m^2	square meters	1.195	square yards	yd^2
ha	Hectares	2.47	Acres	ac
km^2	square kilometers	0.386	square miles	mi^2
		VOLUME		
mL	Milliliters	0.034	fluid ounces	fl oz
L	liters	0.264	Gallons	gal
m^3	cubic meters	35.314	cubic feet	ft^3
m^3	cubic meters	1.307	cubic yards	yd^3
		MASS		
g	grams	0.035	Ounces	oz
kg	kilograms	2.202	Pounds	lb
Mg (or "t")	megagrams (or "metric ton")	1.103	short tons (2000 lb)	T
		TEMPERATURE (exact degrees)		
°C	Celsius	1.8C+32	Fahrenheit	°F
		ILLUMINATION		
lx	lux	0.0929	foot-candles	fc
cd/m^2	candela/m^2	0.2919	foot-Lamberts	fl
		FORCE and PRESSURE or STRESS		
N	newtons	0.225	Poundforce	lbf
kPa	kilopascals	0.145	poundforce per square inch	lbf/in^2

*SI is the symbol for the International System of Units. Appropriate rounding should be made to comply with Section 4 of ASTM E380 (Revised March 2003)

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1. INTRODUCTION

1.1 Background

1.1.1 Use of Rubberized Asphalt Concrete

Each year the United States generates nearly 300 million scrap tires, which is equivalent to approximately one passenger car tire per person per year *(1)*. Most of these tires are dumped in landfills with the consequent environmental impacts. Another solution for their disposal is to grind the tires into crumbs and incorporate them into asphalt binder to produce rubberized asphalt concrete (RAC). The use of RAC is currently focused primarily in the states of California, Arizona, Texas, Florida, and New Jersey; however, successful, documented use *(2)* of this material has created growing interest in many other states. The maximum allowable rubber particle size differs between the different states (e.g., California and Arizona specify rubber particles passing the #8 [2.36 mm] sieve, while Florida limits the maximum size to the #30 [5 mm] sieve).

Apart from the environmental benefits of recycling tires into asphalt concrete, research has also shown that RAC, when used in overlays, has better resistance to the fatigue and reflective cracking caused by traffic and exposure to temperature extremes than conventional dense-graded asphalt concrete (DGAC). Half the thickness of RAC typically provides the same cracking life as the full thickness DGAC when it is used in overlays on cracked pavement *(3-5)*.

1.1.2 Production of Rubberized Asphalt Binders

In California, crumb rubber from scrap tires is generally added to asphalt binder in a so called "wet process." Wet-process rubberized binders can be produced either at an asphalt plant or a nearby distribution center (field blending) or at a supplier's terminal or a refinery (terminal blending). The production of field and terminal blending processes are named "asphalt rubber binder" and "tire rubber modified binder," respectively. The crumb rubber content, crumb rubber properties, types of extenders, and the digestion process differ significantly between the two processes. Consequently, the properties of the rubberized binders produced by the two processes are also very different and therefore require different approaches to define the binder properties. Tire rubber modified binders are produced with relatively small rubber particles and generally have similar characteristics to polymer-modified binders and can be characterized accordingly using existing Superpave Performance Grading (PG) procedures (i.e., AASHTO M320). In California, asphalt rubber binders are produced using larger crumb rubber particles (i.e., passing #8 [2.36 mm] sieve) and an optional asphalt modifier, which facilitates swelling of the rubber particles during blending and reaction, and ultimately decreases the viscosity of the asphalt rubber binder. Asphalt rubber binders are not homogenous blends since the rubber particles are not fully digested during production.

The Superpave PG procedures were developed for binders that do not contain additives or particulates and are therefore not considered appropriate for testing asphalt rubber binders. Consequently, alternative binder grading procedures are needed. The research discussed in this technical memorandum focuses on the development of these PG grading procedures for wet-process asphalt rubber binders.

1.1.3 Crumb Rubber Modifier Production

Crumb rubber modifier (CRM) (also known as ground tire rubber [GTR]) is produced by grinding waste tires. The two main methods used are *ambient grinding* and *cryogenic fracturing*. In the ambient grinding process, the scrap tires are cut to small pieces then shredded and ground into relatively small size crumbs at ambient temperature. The ambient grinding method results in irregular shaped rubber particles with rough surfaces. In cryogenic fracturing, the cut pieces of scrap tires are frozen with liquid nitrogen and then fractured into the small size crumbs. Cryogenic fracturing usually results in cubical shaped rubber particles with relatively smooth surfaces.

1.1.4 Current Caltrans Asphalt Rubber Binder Specifications

Current Caltrans specifications for the constituents of asphalt rubber binder, asphalt rubber binder reaction design profile, and the criteria for quality control and acceptance criteria are summarized in Table 1.1, and Table 1.2, and Table 1.3, respectively. The current Caltrans criteria for characterizing the quality of asphalt rubber binders are based on viscosity, penetration, resilient properties, and softening properties. Asphalt rubber binder properties must meet the specified limits in Table 1.3 after at least 45 minutes of reaction time between the asphalt binder and the crumb rubber.

Table 1.1: Caltrans Specifications for Asphalt Rubber Binder Constituents

Component	Characteristic	Test Method	Value
Asphalt modifier	Viscosity, m^2/s (x 10^{-6}) at 100°C	ASTM D 445	X ± 3[a]
	Flash point, Cleveland Open Cup (°C)	ASTM D 92	>207
	Asphaltenes (% by mass)	ASTM D 2007	<0.1
	Aromatics (% by mass)	ASTM D 2007	>55
Crumb rubber modifier[b]	Scrap tire crumb rubber gradation (% passing #8 sieve)	LP-10	100
	High natural rubber gradation (% passing #10 sieve)	LP-10	100
	Wire in CRM (% max.)	LP-10	0.01
	Fabric in CRM (% max.)	LP-10	0.05
	CRM particle length (in. max.)[c]	--	3/16
	CRM specific gravity[c]	CT 208	1.1 – 1.2
	Natural rubber content in high natural rubber (%)[c]	ASTM D 297	40.0 – 48.0

[a] The symbol "X" is the proposed asphalt modifier viscosity. "X" must be from 19 to 36. A change in "X" requires a new asphalt rubber binder design.
[b] CRM must be ground and granulated at ambient temperature. If steel and fiber are cryogenically separated, this must occur before grinding and granulating. If cryogenically produced, CRM particles must be large enough to be ground or granulated and not pass through the grinder or granulator.
[c] Test at mix design and for certificate of compliance

Table 1.2: Asphalt Rubber Binder Reaction Design Profile

Characteristic	Test Method	Minutes of Reaction[a,b]							Value
		45	60	90	120	240	360	1440	
Cone penetration @77°F (0.10 mm)	ASTM D 217	X				X		X	25 – 70
Resilience @ 77°F (% rebound)	ASTM D 5329	X				X		X	>18
Field softening point (°F)	ASTM D 36	X				X		X	125 – 165
Viscosity @ 375°F, (centipoise)	LP-11	X	X	X	X	X	X	X	1,500 – 4,000

[a] Six hours (360 minutes) after CRM addition, the oven temperature is reduced to 275°F for 16 hours. After the 16 hour (1,320-minutes) cool down after CRM addition, the binder is reheated to the reaction temperature expected during production for sampling and testing at 24 hours (1,440 minutes).
[b] "X" denotes required testing.

Table 1.3: Caltrans Specifications for Asphalt Rubber Binder Quality Control and Acceptance

Characteristic	Test Purpose	Test Method	Value	
			Minimum	Maximum
Cone penetration @77°F (0.10 mm)	Acceptance	ASTM D 217	25	70
Resilience @ 77°F (% rebound)	Acceptance	ASTM D 5329	18	--
Field softening point (°F)	Acceptance	ASTM D 36	125	165
Viscosity @ 375°F, (centipoise)	Quality control	LP-11	1,500	4,000

According to the ASTM D8 test method, a minimum of 15 percent CRM by weight of the asphalt binder is required to meet the definition of an asphalt rubber binder. However, Caltrans specifications require a CRM content of 20 ± 2 percent by weight of the asphalt rubber binder, of which 25 percent must be natural rubber. An asphalt modifier (extender oil) can be added at a rate of two to six percent by weight of the base asphalt binder to facilitate the reaction between the asphalt binder and rubber particles *(1)*.

Caltrans specifications also require crumb rubber particles finer than 2.36 mm (100 percent passing the #8 sieve). Cryogenic grinding is only permitted for the separation of metals and fibers, after which larger rubber particles are ground at ambient temperatures to meet the required sizes.

On each Caltrans project, asphalt rubber binder producers must propose a design and profile for the binder that will be used. The design must specify the materials including base binder, extender oil, and crumb rubber. The asphalt rubber binder profile serves as an indication of the quality of production and is not used as a performance specification. The profile illustrates the characteristics of the binder over a 24 hour (1,440 minute) interaction period. Table 1.2 shows the required properties that must be measured during a 24 hour interaction period along with their respective specified limits. Binder acceptance is checked using cone penetration, resilience, and softening point in addition to viscosity.

1.2 Problem Statements

A number of limitations to the current asphalt rubber binder specification have been identified through a review of the literature and discussions with stakeholders. These include the following:

- The current Caltrans specification for wet process asphalt rubber binders focuses mainly on measuring viscosity at the plant using a handheld rotational viscometer. While viscosity is an important parameter for the workability of the binder and ultimately of the mix, it does not directly relate to the in-service performance of the binder within a rubberized asphalt concrete mix or a rubberized asphalt surface treatment. Additionally due to the particulate phase of these binders, viscosity measurements alone lack sufficient accuracy to completely describe their complex properties.

- Although penetration grading and resilience do provide a means to evaluate the stiffness and resilience of asphalt rubber binders, the Superpave Performance Grading (PG) testing procedure moved away from these tests because they have several limitations, including the following:
  - They are empirical tests that measure the viscous and elastic properties of the binder and do not necessarily correlate with field performance.
  - The tests only measure the properties of the binder at a single intermediate temperature, and thereby fail to provide an accurate indication of the properties at typical high and low service temperatures, or the temperature susceptibility (change of stiffness with change of temperature) of the binder.
  - The tests do not address the effect of short-term aging (during mixing and compaction) and long-term aging (during field performance) on the properties of asphalt rubber binder.
- Softening point generally indicates the phase change temperature of binders and may not be sufficient for comprehensive performance-rheological characterization.
- Rheological testing using a dynamic shear rheometer (DSR) and a bending beam rheometer (BBR) are now considered standard practice for evaluating performance-related characteristics of conventional, polymer-modified, and tire rubber-modified asphalt binders. However, the standard parallel plate geometry used in the DSR test is potentially inappropriate for measuring the properties of California-produced asphalt rubber binders. When asphalt rubber binder is tested using 1 mm or 2 mm parallel plate geometry in the DSR, partially digested rubber particles can contact both the top and bottom plates and interfere with the torque and strain measurements, resulting in the rheology of the rubber particles dominating the measurement and potentially providing misleading information about the rheology of the asphalt rubber binder as a whole. A potential consequence of this misleading information is choice/use of an inappropriate binder for a given climate region. According to AASHTO T 315 (*Standard Method of Test for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer*), the gap size between the plates should be at least four times the maximum particle size to provide reliable results (i.e., an 8 mm gap, with correspondingly adjusted plate diameter, would be required for 2.0 mm [#10] crumb rubber particles). However, the maximum gap size recommended by rheologists is about 5 mm, in order to ensure a satisfactory linear shear rate between the plates. Although increasing the gap size is a potential solution for dealing with the larger rubber particle sizes, the increase can introduce other problems such as poor repeatability, unacceptable temperature gradients, difficultly in trimming the specimen, uncontrollable edge effects, and potentially misleading results. When testing with parallel plate geometry, the modulus of the asphalt binder is proportional to the sample radius to the power of four. Consequently, a two percent reduction in radius due to incorrect trimming implies a potential 16 percent reduction in the measured modulus.

To overcome these issues, there is a need for alternative test equipment and procedures that can better evaluate the performance characteristics of wet-process asphalt rubber binders using the same or similar

Superpave PG parameters as conventional, polymer-modified (PM), and tire rubber-modified (TR) binders. These alternate procedures can then be used to establish performance-based contract acceptance criteria for the production of asphalt rubber binders, which will in turn lead to more reliable performance in the field.

1.3 Project Objectives

The objective of this study is to recommend appropriate contract acceptance criteria for wet-process asphalt rubber binders using current Superpave PG equipment. This objective will be met by completing the following tasks:

1. Review relevant literature on the topic. Contact DSR equipment manufacturers and discuss test requirements and alternative geometries (specifically concentric cylinder [or cup-and-bob]) that can be used for these tests. (The literature is summarized in Chapter 2).
2. Collect samples of asphalt binder, crumb rubber particles, and extender oil for laboratory preparation of asphalt rubber binder. On completion of initial screening tests, identify completed and current projects where asphalt rubber binder samples can be collected for additional testing.
3. Prepare laboratory-conditioned samples for testing with a DSR.
4. Evaluate the use and ability of the alternative concentric cylinder DSR geometry to provide realistic, repeatable results for conventional, polymer-modified (PM), and tire rubber-modified (TR) binders that are comparable to results from the same tests using conventional parallel plate geometries. The performance of these binders is routinely measured with parallel plate geometry in terms of the Superpave PG grading system. (This testing is discussed in Chapter 3).
5. Compare the parallel plate and concentric cylinder geometries for testing asphalt rubber binder containing crumb rubber particles of various sizes. (Testing completed to date on this task is discussed in Chapter 4).
6. Evaluate the effects of different crumb rubber particle properties and asphalt rubber binder properties on DSR test results. (Testing completed to date on this task is discussed in Chapter 5).
7. Suggest contract acceptance criteria for wet-process asphalt rubber binders using a DSR with appropriate geometry.
8. Prepare a report documenting this research, with recommendations for specification language and, if required, recommendations for further research on characterizing wet-process asphalt rubber binders and correlating test results against laboratory cracking and rutting test results as well as field performance.

This technical memorandum provides an update on work completed to date on Tasks 1 through 6.

1.4 Measurement Units

Although Caltrans recently returned to the use of U.S. standard measurement units, the Superpave Performance Grading (PG) System is a metric standard and uses metric units. In this technical memorandum, both English and metric units (provided in parentheses after the English units) are provided in the general discussion. Metric units are used in the reporting of PG test results. A conversion table is provided on page xi.

2. LITERATURE REVIEW

2.1 Introduction

Discussions with binder testing equipment manufacturers, other practitioners researching asphalt binder rheology, and a review of the literature indicated that the use of a concentric cylinder (or cup-and-bob) geometry would be the most appropriate system for testing the properties of asphalt rubber binders. Although other options for testing were not excluded from the literature review, the review focused on this geometry. The literature review also focused on the potential effects of different crumb rubber modifier (CRM) properties on asphalt binder performance, with particular attention given to the method of CRM preparation, the particle size and surface area, the CRM content in the asphalt binder, and the methods used to age asphalt rubber binder before testing.

2.2 Concentric Cylinder Geometry (Cup-and-Bob)

The DSR concentric cylinder measuring system proposed for the evaluation of asphalt binders has two cylinders: the inner cylinder is called the bob and the outer cylinder is called the cup (Figure 2.1).

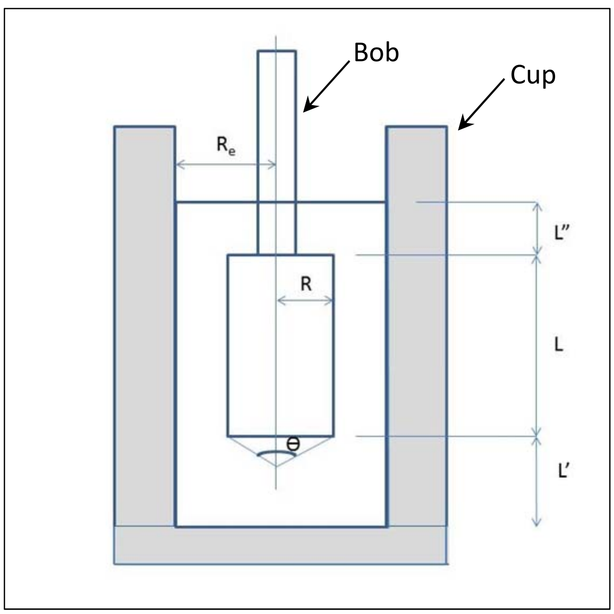

Figure 2.1: Concentric cylinder geometry.

This concentric cylinder geometry is commonly used to measure the viscosity of substances such as paints, adhesives, and various types of food that may not be homogenous or that contain particulates. However, only limited research has been undertaken on the use of concentric cylinder geometry to measure the complex shear modulus (G*) and phase angle (δ) of asphalt binders, which are the main measurement parameters used in the Superpave Performance Grading (PG) system, to assess the properties of conventional and polymer modified asphalt binders at high temperatures. A parallel plate geometry with 1 mm or 2 mm gap is used for this testing. The concentric cylinder geometry is able to accommodate a much larger gap by varying the sizes of the cup and/or the bob. The gap size between the concentric cylinders can be as high as 7 mm, rendering it more appropriate for testing wet-process rubberized binders with larger constituent particles. The shear stress and shear strain calculations used to interpret the data from the concentric cylinder geometry are shown in Equations 2.1 and 2.2 *(6)*.

$$\tau = \frac{T}{2\pi L R^2} \tag{2.1}$$

$$\gamma = \frac{\theta R_e}{(R - R_e)} \tag{2.2}$$

Where,

τ = shear stress
γ = shear strain
T = torque
L = length of the bob
Re = radius of the cup
R = radius of the bob
Θ = angular rotation of the bob

The concentric cylinder geometry is controlled by the surface area and radius of the bob and the inside surface area and radius of the cup, in a similar way to the parallel plate geometry, which is controlled by the surfaces and outside edges of the two plates. Any binder that is at the bottom of the cup or which overtops the bob can be ignored. Unlike the parallel plate geometry, which requires trimming of the sample that can lead to operator error (depending on the operator's skill level), the concentric cylinder geometry does not require trimming of the sample.

Baumgardner and D'Angelo *(7)* evaluated the concentric cylinder approach using a DSR to compare the performance grade (PG) properties of conventional, polymer-modified, and wet-process asphalt rubber binders. They concluded that the concentric cylinder geometry can provide similar results (G*/sin[δ]) to those obtained using parallel plate geometry. Cheng et al. *(8)* also investigated the G*/sin(δ) of conventional binders with concentric cylinder geometry. The results indicated a good correlation between the concentric cylinder and parallel plate geometries. However, a limited number of binders were evaluated in both studies and calibration factors between the two approaches were not discussed.

2.3 Effects of Crumb Rubber Modifier on Asphalt Binder Performance

Adding crumb rubber to conventional asphalt binders in the wet process has significant impacts on the performance-related properties of the binders, and it increases the viscosity at pumping and mixing temperatures. The complex shear modulus and the associated strain at failure of asphalt rubber binders are also higher than conventional binders at high temperatures. However, the presence of rubber in the binder has less influence on the low temperature properties *(9)* when compared to conventional binders.

2.3.1 Effect of Crumb Rubber Production Method

The chosen tire grinding method (i.e., ambient grinding versus cryogenic fracturing) influences the shape of the rubber crumbs, their texture, their surface area, and other physical properties, with ambient grinding typically producing rubber crumbs with more irregular shapes, a rougher texture, and a typically a larger surface area than cryogenically fractured rubber. As a consequence, the properties of rubberized binders can differ depending on the CRM production method used *(10)*. Binder testing results have shown that asphalt rubber binders produced with cryogenically fractured rubber have more settlement, higher temperature sensitivity, and less resistance to low temperature cracking and to drain-down compared to binders produced with rubber ground at ambient temperatures. However, it should be noted that the low temperature indicator (m-value) in the PG system was not shown to be statistically different for binders produced with rubber from the two different processes *(11)*.

2.3.2 Effect of Crumb Rubber Particle Size, Shape, and Surface Area

The particle sizes, shapes, and surface area of crumb rubber can affect the viscosity and performance-related properties of asphalt binders *(10-13)*, with different results obtained from different researchers. West *(10)*, Lee *(11)* and Kim et al. *(12)* found that crumb rubber particles with higher surface areas and more irregular shapes (i.e., those produced at ambient temperatures) tended to produce rubberized binders with higher viscosities, while Shen et al. *(13)* reported an opposite result. Lee's findings concluded that particles with higher surface areas absorb more light fractions from binders, leading to the higher viscosity of the modified binder. Shen concluded that both particle size and surface area have statistically significant effects on the viscosity of rubberized binders, with particle size having a larger impact on viscosity than surface area.

2.3.3 Effect of Crumb Rubber Content

Asphalt binders modified with higher rubber content have higher viscosities than those with lower rubber content *(10,14)*. Higher rubber contents also have significant effects on the high temperature performance (the value of $G^*/\sin\delta$) of the asphalt binders *(14)*. Lee et al. *(11)* also reported the improved fatigue resistance of mixes produced with higher crumb rubber content binders, compared to those with lower rubber contents.

2.3.4 Effect of Laboratory Aging Method

The Superpave Performance Grading system characterizes asphalt binders at three critical aging intervals. Unaged binders are tested to characterize the virgin binder prior to mixing with aggregates. Intermediate-aged binders, conditioned in a rolling thin film oven (RTFO), are tested to characterize the binder after asphalt concrete production and placement. Aged binders, conditioned in a pressure aging vessel (PAV), are tested to characterize binders that have been in service for 7 to 15 years after placement.

The high viscosities of asphalt rubber binders can have implications when conducting these performance-grading tests. In the RTFO test, the high viscosities at high temperature may result in the binders not coating the entire bottle at the start of the test, not flowing in the bottles during the test period, and/or spilling out of the bottle instead of coating it *(7)*. This coating issue defeats the original design purpose of the RTFO test, which requires that binders must keep moving in the RTFO bottle to avoid skin formation and to ensure uniform aging, representative of conditions in the asphalt plant. High viscosity rubberized binders are also difficult to scrape out of the RTFO bottle after the test is completed. Given these issues, the thin film oven (TFO) test with slight modification could potentially be considered as an alternative to the RTFO test for asphalt rubber binders.

The difference between the TFO test and the RTFO test was investigated by Jeong *(14)* and Zupanik *(15)*. Zupanick analyzed the AASHTO Materials Reference Laboratory (AMRL) database, which includes more than 2,000 TFO and RTFO tests completed in laboratories throughout the United States. Using viscosity, penetration, and weight change as the performance measures, Zupanick concluded that the TFO and RTFO tests are not interchangeable, contradicting earlier studies and industry practice. The data indicated that the RTFO test is more severe and precise than the TFO test in terms of the increase in binder viscosity. However, these results were not consistent for all of the samples, with TFO-aged samples tending to have lower viscosities than RTFO-aged samples when the original binders were softer. This was attributed to higher viscosities reducing natural convection in the TFO pan, and to skin formation on the binder during the TFO test. The study did not consider dynamic shear modulus, phase angle, or low temperature cracking resistance.

3. ASSESSMENT OF DSR TESTING GEOMETRIES

3.1 Introduction

This chapter discusses the testing of selected performance graded conventional, polymer-modified, and tire rubber-modified asphalt binders using parallel plate and concentric cylinder geometries to determine whether equivalent results can be obtained from each geometry. Before this testing could be undertaken, appropriate temperature and conversion factor calibrations needed to be developed to interpret and relate results between the two geometries.

3.1.1 Temperature Calibration and Thermal Equilibrium

The performance of asphalt binders is very sensitive to testing temperature, and therefore accurate temperature control of each measuring system is critical for testing. Each system must be calibrated appropriately to ensure that temperature control is correct.

Since each of the two measuring systems has different geometries, each one requires a different temperature calibration process. For both systems, three testing temperatures (40°C, 65°C, and 90°C) are typically calibrated to ensure accuracy. In the concentric cylinder configuration, measurements are taken at the top of the cup, at the middle of the cup close to the bob, the middle near the cup edge, and at the bottom of the cup, to check the vertical temperature gradient. This temperature gradient should not differ by more than 0.1°C from top to bottom, a value comparable to the requirements of the parallel plate testing system.

The concentric cylinder also requires significantly more binder to perform a test than the parallel plate system does. As a result, testing with the concentric cylinder takes longer to reach temperature equilibrium than the parallel plate.

For the remainder of this study, temperature calibration and thermal equilibrium were strictly controlled following the above conventions in all tests discussed in this technical memorandum.

3.1.2 Calibration of the Conversion Factor (C_{ss})

A series of laboratory tests were conducted in this part of the study to compare the results obtained from the concentric cylinder and parallel plate testing geometries. The effects of different operators, different binder types (conventional, polymer-modified, or tire rubber-modified), binder source, and aging condition (unaged, rolling thin film oven [RTFO], and in certain instances, thin film oven [TFO] aged) on complex modulus and phase angle were all investigated.

The two cylinders (i.e., the cup and the bob) used in the concentric cylinder testing system have different diameters and therefore nonlinear behavior, which is different to the parallel plate system, which uses two plates which have the same dimensions. The presence of relatively large rubber particles in the asphalt rubber binder also requires a correspondingly sized gap in the cup-and-bob geometry. Therefore, an appropriate conversion factor must be applied to relate test results obtained with the concentric cylinder geometry to the smaller gap used when testing conventional binders in the parallel plate geometry. When small gaps are used, the change in shear stresses between the cup and the bob is very small (assumed linear) and thus, the representative shear stress is the average shear stress between the cup and the bob. Because the shear stress is assumed to be linear, the conversion factor only depends on the geometric dimensions of the specific concentric cylinder configuration. In these instances, a fixed conversion factor can be used.

When using larger gap concentric cylinders, the linear assumption of shear stress between the two cylinders is no longer appropriate and binder-specific conversion factors need to be determined based on the complex viscosity, angular frequency, strain, and torque of the asphalt binders. The conversion factor for the large gap concentric cylinder can be calculated using Equation 3.1 *(Anton Paar, personal communication)*, which provides comparable results between the concentric cylinder and parallel plate geometries in terms of complex shear modulus (G*) and phase angle (δ). Calibration is required for each asphalt binder with different complex viscosity or torque values.

$$C_{ss} = \frac{\eta[\omega(\gamma/100)]}{T} \tag{3.1}$$

Where,

C_{ss} = conversion factor
η = complex visocity from parallel plate (PaS)
ω = angular frequency (rad/s)
ϒ = strain (%)
T= torque from concentric cylinder (mNm)

3.2 Test Plan

3.2.1 Testing with Binder Specific Conversion Factors

Conventional and modified binders were tested with a DSR to investigate the effects of varying conversion factors on the measurements from the concentric cylinder geometry. In this stage, the experiment was separated into the following three tasks:

- Task 1: Testing of three conventional PG 64-16 binders by three different operators with three replicates. Binders were obtained from three California-based oil refineries, namely Paramount, San Joaquin, and Valero.

- Task 2: Testing of one PG 64-28PM polymer modified and one PG 64-28TR tire rubber modified binder by three different operators and with three replicates. Binders were obtained from Paramount.
- Task 3: Testing of two conventional PG 64-16 binders, one PG 64-28PM polymer modified binder and one PG 64-28TR tire rubber modified binder, all subjected to RTFO aging. No replicates were tested in this task. The conventional binders were sourced from the San Joaquin and Valero refineries and the polymer- and tire-modified binders were sourced from the Paramount refinery. (It should be noted that TR modified binders have much smaller rubber particles (maximum size of 300 µm [#50]) than asphalt rubber binders and due to their more complete digestion are not susceptible to the problems with RTFO aging discussed in Section 2.3.4; this permits direct comparison using the two DSR configurations.)

3.2.2 Testing with Fixed Conversion Factor

Testing of a standard fluid with viscosity similar to an asphalt binder was identified as the most appropriate method of determining a representative fixed conversion factor to use for comparing the results obtained from the two testing geometries. *Cannon* certified viscosity reference standard S600 was selected to obtain this conversion factor *(Anton Paar, personal communication)*. Based on the test results, a fixed conversion factor value of 72 was selected for the testing described in this technical memorandum.

Three conventional binders (Valero PG 58-22, San Joaquin PG 64-16, and Valero PG 70-10) were assessed to investigate the effects of this fixed conversion factor. Both unaged and short-term oven aged binders were tested. Short-term aging was performed using both the RTFO and TFO in an attempt to address the issues discussed in Section 2.3.4 with regard to aging rubberized binders. Only one operator conducted the experiments (with three replicates), given that the results obtained by the three different operators in Tasks 1 through 3 were not significantly different.

3.3 Test Results

3.3.1 Testing with Binder Specific Conversion Factors

Table 3.1 presents the conversion factors determined for the asphalt binders evaluated in Tasks 1 through 3. The conversion factors were calculated from Equation 3.1 using complex viscosity measurements at 64ºC tested with both parallel plate (1 mm gap) and concentric cylinder geometries. The conversion factors were found to be different for the various evaluated asphalt binders and changed considerably with short-term aging by RTFO in some cases (e.g., PG64-16 Valero). The DSR test results are listed in Table A.1 through Table A.4 in Appendix A and summarized in the following sections.

Table 3.1: Specific Conversion Factors for the Evaluated Asphalt Binders

Asphalt Binder		Conversion Factor	
Source	**Grade**	**Original**	**RTFO Aged**
Paramount	PG 64-16	70	64
San Joaquin	PG 64-16	67	81
Valero	PG 64-16	71	50
Paramount	PG 64-28PM	80	78
Paramount	PG 64-128TR	91	81

Task 1: Conventional Binders

The boxplots of complex shear modulus (G*), phase angle (δ), and G*/sin(δ) at 64°C for the three different conventional binders are shown in Figure 3.1 through Figure 3.3. Based on these results, the complex shear moduli (G*) values appeared to be very similar between the two geometries, but with slightly different phase angles (less than 0.5°). The differences in G*/sin(δ) between the concentric cylinder and parallel plate geometries were therefore also very small. The differences in results for the three operators are shown in Figure 3.4. The results obtained by Operator #1 and Operator #2 are very close, but the results obtained by Operator #3 were slightly different for both geometries. The points in Figure 3.4 are scattered evenly for both concentric cylinder and parallel plate, indicating that the repeatability of results when using the concentric cylinder geometry is similar to that when testing with the parallel plate system.

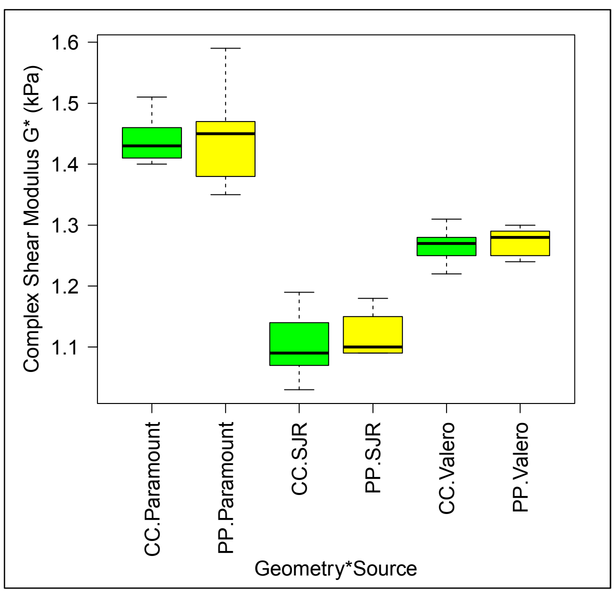

Figure 3.1: Conventional binders: G* with varied conversion factor at 64°C.
(CC = concentric cylinder, pp = parallel plate)

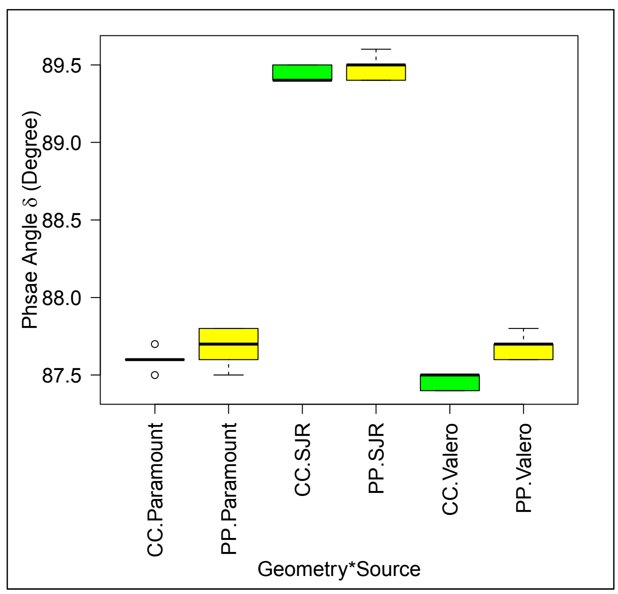

Figure 3.2: Conventional binders: δ with varied conversion factor at 64°C.
(CC = concentric cylinder, pp = parallel plate)

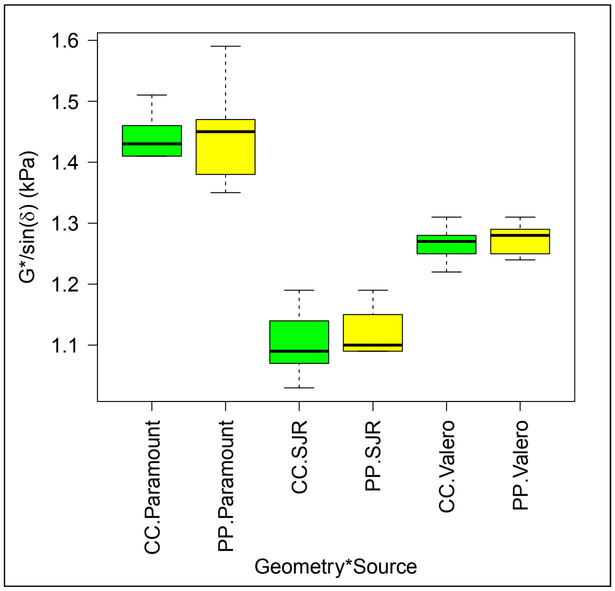

Figure 3.3: Conventional binders: G*/sin(δ) with varied conversion factor at 64°C.
(CC = concentric cylinder, pp = parallel plate)

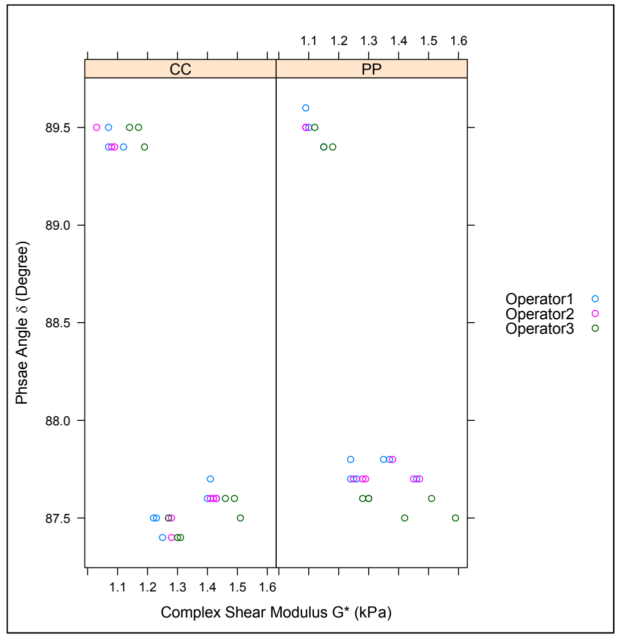

Figure 3.4: Conventional binders: G* against δ with varied conversion factor at 64°C.
(CC = concentric cylinder, pp = parallel plate)

An analysis of variance (ANOVA) was used to investigate the difference in results between the two testing geometries. G*/sin(δ) was the dependent variable and geometry was the independent variable. Binder source was selected as a blocking factor since it was not of primary interest in this study. The analysis results are shown in Table 3.2, and indicate that the measurements of G*/sin(δ) between concentric cylinder and parallel plate were not significantly different at a 95 percent confidence interval. With a varied (i.e., binder specific) conversion factor, the results obtained when using the concentric cylinder geometry were not statistically significantly different from the results obtained when using the parallel plate geometry.

Table 3.2: Conventional Binders: ANOVA Results of G*/sin(δ) with Varied Conversion Factor
(α=0.05)

Parameter	Df	Sum Sq	Mean Sq	F Value	Pr (>F)
Geometry	1	0.0006	0.0006	0.294	0.59
Source	2	0.9808	0.4904	240.582	<2e-16
Residuals	50	0.1019	0.0020	-	-

Task 2: Modified Binders

The boxplots of complex shear modulus (G*), phase angle (δ), and G*/sin(δ) at 64°C are shown in Figure 3.5 through Figure 3.7. Lower complex shear modulus was measured for the polymer-modified binder using the concentric cylinder geometry when compared to the parallel plate system, whereas the opposite trend was observed for the complex shear modulus of the tire rubber-modified binder. Higher phase angles were also recorded for both modified binders tested with the concentric cylinder geometry when compared to the parallel plate system. Trends similar to those recorded for the complex shear moduli were also recorded for G*/sin(δ) for both geometries. This was expected, given that differences in phase angle have less influence on the values of G*/sin(δ) than do differences in the complex shear modulus (G*). Results obtained by the three different operators are shown in Figure 3.8. The data points are scattered relatively evenly between the operators, with the phase angles measured with the concentric cylinder geometry slightly higher than those recorded using the parallel plate geometry.

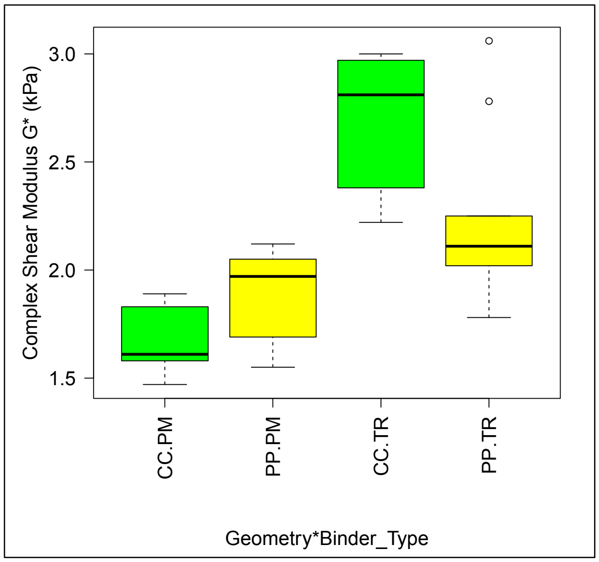

Figure 3.5: Modified binders: G* with varied conversion factor at 64°C.
(CC = concentric cylinder, pp = parallel plate)

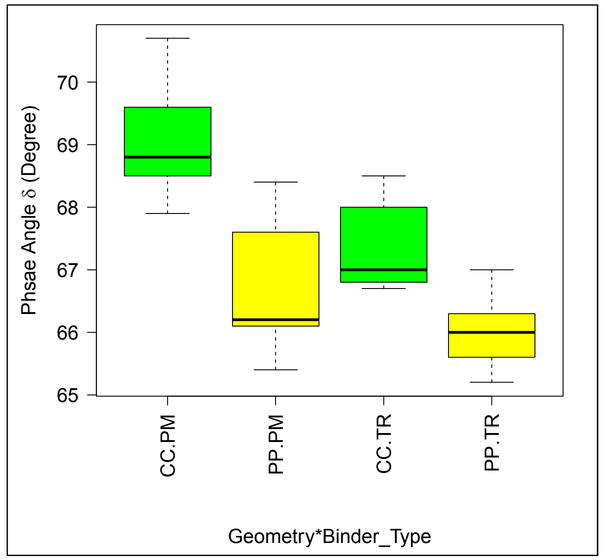

Figure 3.6: Modified binders: δ with varied conversion factor at 64°C.
(CC = concentric cylinder, pp = parallel plate)

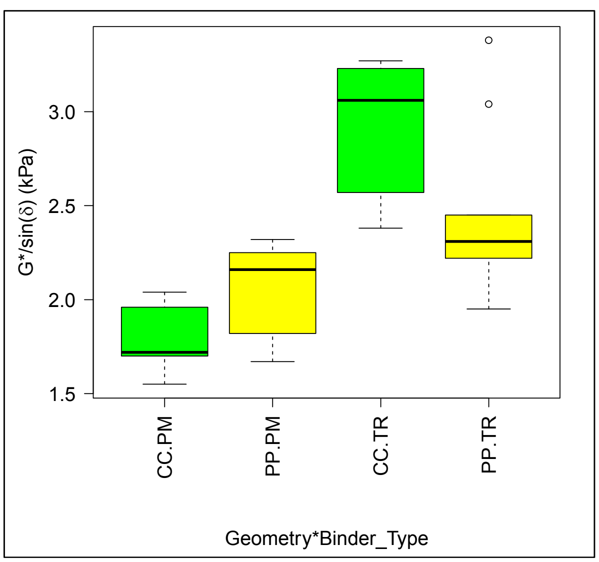

Figure 3.7: Modified binders: G*/sin(δ) with varied conversion factor at 64°C.
(CC = concentric cylinder, pp = parallel plate)

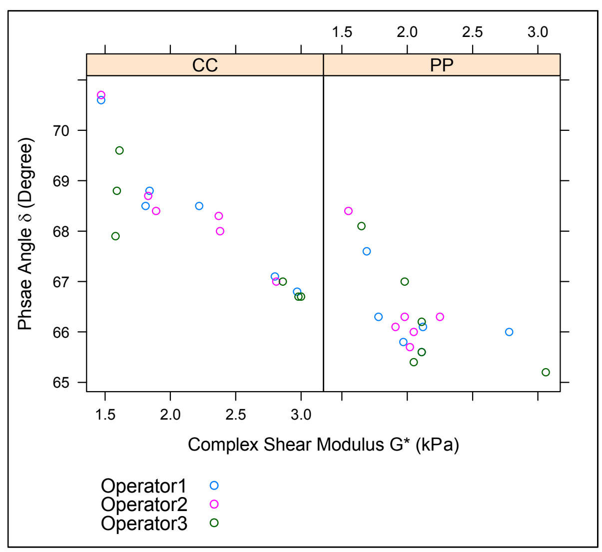

Figure 3.8: Modified binders: G* against δ with varied conversion factor at 64°C.

The ANOVA results are shown in Table 3.3. G*/sin(δ) was the dependent variable and geometry was the independent variable. Binder type was again selected as a blocking factor. The ANOVA results indicate that the measurements of G*/sin(δ) using the concentric cylinder and parallel plate geometries were not significantly different at a 95 percent confidence interval. When using a varied conversion factor, the results obtained using the concentric cylinder geometry were not statistically significantly different than those obtained when using the parallel plate system.

Table 3.3: Modified Binders: ANOVA Results of G*/sin(δ) with Varied Conversion Factor
(α=0.05)

Parameter	Df	Sum Sq	Mean Sq	F value	Pr (>F)
Geometry	1	0.106	0.106	0.751	0.393
Binder type	1	5.282	5.282	37.542	6.6e-07
Residuals	33	4.643	0.141	-	-

Task 3: RTFO-Aged Binders

The boxplots of complex shear modulus (G*), phase angle (δ), and G*/sin(δ) at 64°C for the RTFO-aged binders are shown in Figure 3.9 through Figure 3.11. The results appeared to be similar for both geometries. Both modified binders had a higher complex shear modulus than the conventional binders, as expected, despite their having the same high temperature ratings. Both modified binders also had lower phase angles compared to the conventional binders, which led to higher G*/sin(δ) values. When the results obtained by the three different operators (Figure 3.12) were compared, only one data point from Operator #1 was higher, with the rest of the data points similar among the operators.

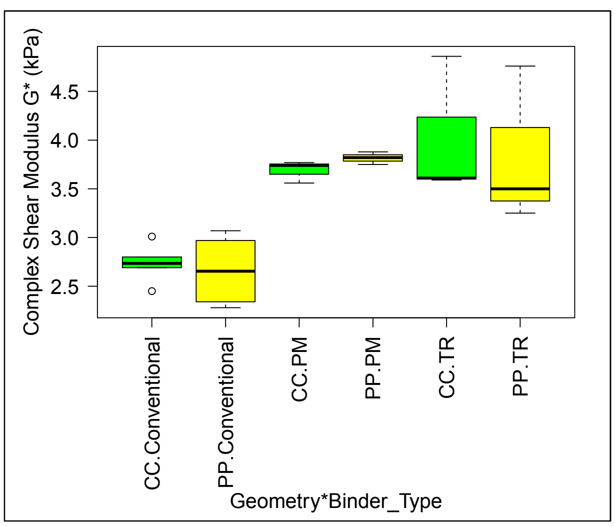

Figure 3.9: RFTO-aged binders: G* with varied conversion factor at 64°C.
(CC = concentric cylinder, pp = parallel plate)

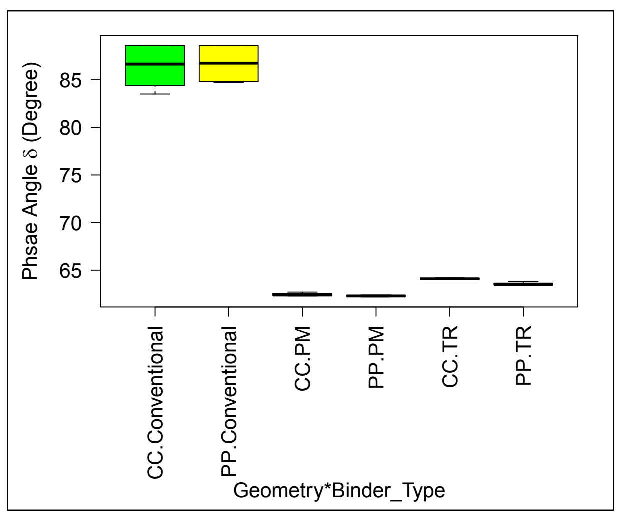

Figure 3.10: RFTO-aged binders: δ with varied conversion factor at 64°C.
(CC = concentric cylinder, pp = parallel plate)

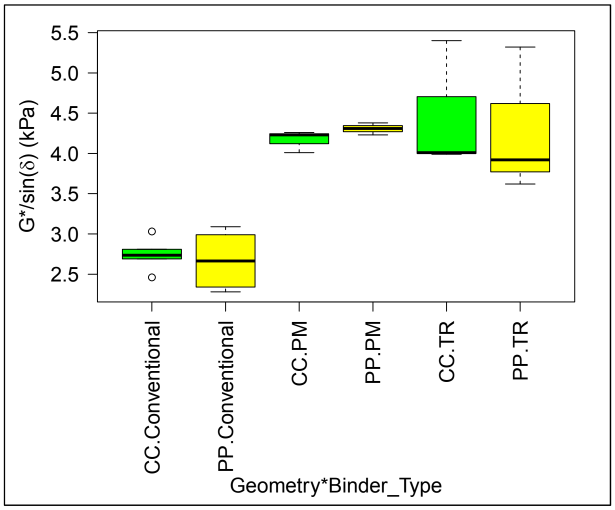

Figure 3.11: RFTO-aged binders: G*/sin(δ) with varied conversion factor at 64°C.
(CC = concentric cylinder, pp = parallel plate)

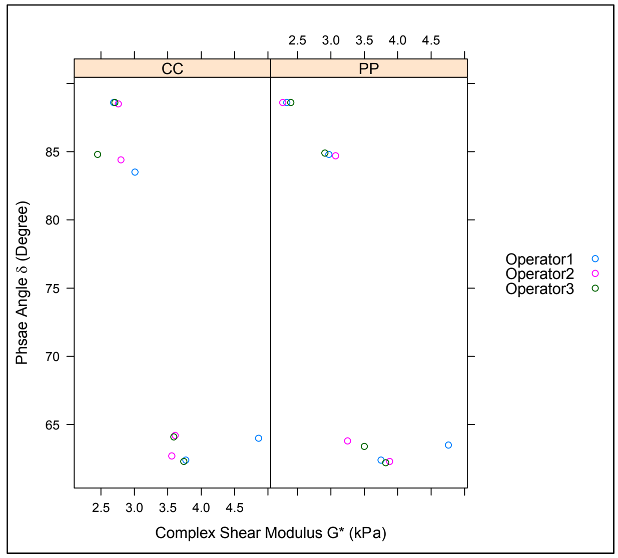

Figure 3.12: RTFO-aged binders: G* against δ with varied conversion factor at 64°C.

The ANOVA results are shown in Table 3.4. G*/sin(δ) was the dependent variable and geometry was the independent variable in the analysis. Binder type was again selected as a blocking factor. The statistical analysis indicated that G*/sin(δ) measured with the two geometries was not significantly different at a 95 percent confidence interval.

Table 3.4: RTFO-Aged Binders: ANOVA Results of G*/sin(δ) with Varied Conversion Factor
(α=0.05)

Parameter	Df	Sum Sq	Mean Sq	F Value	Pr (>F)
Geometry	1	0.013	0.013	0.064	0.802
Binder type	2	15.40	7.701	39.25	1.19e-07
Residuals	20	3.924	0.196	-	-

3.3.2 Testing with Fixed Conversion Factor

As discussed in Section 3.2.2, a fixed conversion factor of 72, determined by testing a *Cannon* certified viscosity reference standard material (S600) was used in this phase of the test. DSR test results using the parallel plate (PP) and the concentric cylinder systems are listed in Table A.4 in Appendix A and summarized below.

<u>Conventional Binders</u>

Test results for the unaged and short-term aged binders at their PG temperature (i.e., 58°C, 64°C, and 70°C) are shown in Figure 3.13 through Figure 3.15. The complex shear moduli and phase angles were similar between the two geometries. RTFO aging was found to be more severe than TFO aging on the selected binders. The test results are separated by PG grade in Figure 3.16. The measurements obtained from both geometries are close for all the tested binders.

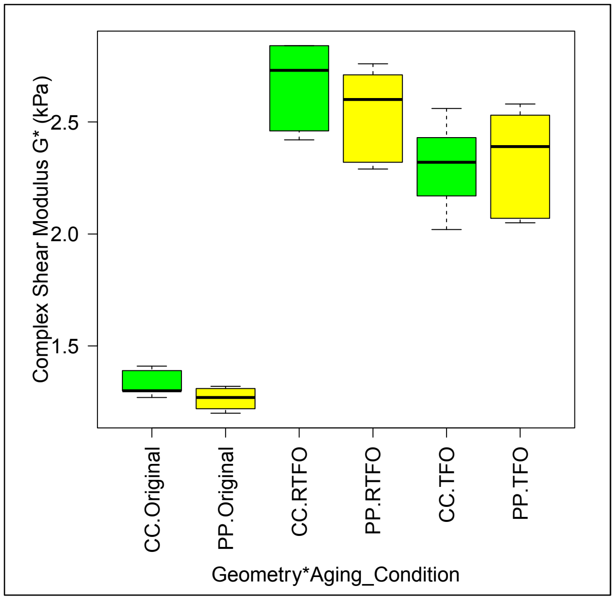

Figure 3.13: Conventional binders, unaged and aged: G* with fixed conversion factor.
(CC = concentric cylinder, pp = parallel plate)

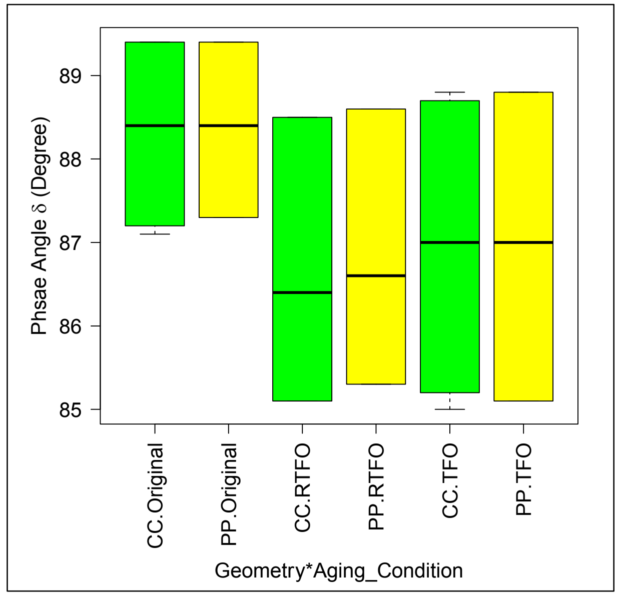

Figure 3.14: Conventional binders, unaged and aged: δ with fixed conversion factor.
(CC = concentric cylinder, pp = parallel plate)

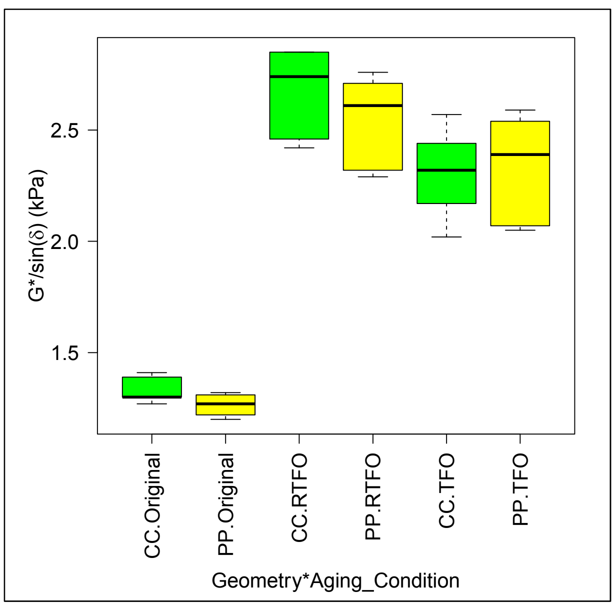

Figure 3.15: Conventional binders, unaged and aged: G*/sin(δ) with fixed conversion factor.
(CC = concentric cylinder, pp = parallel plate)

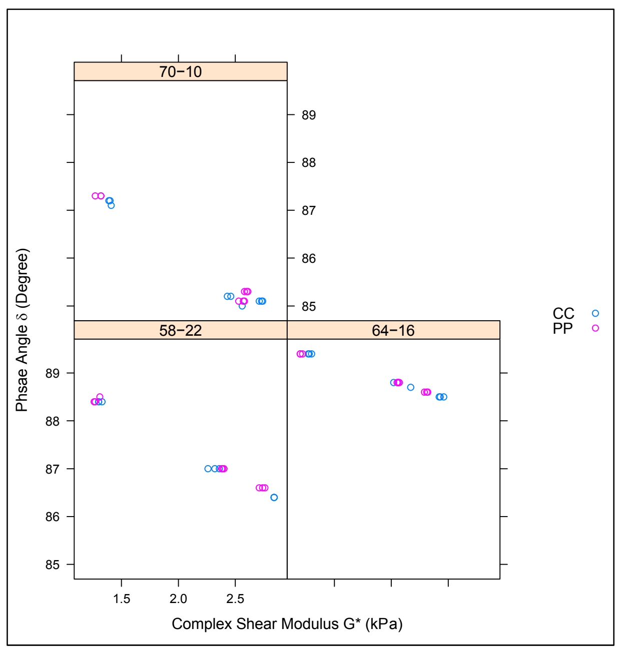

Figure 3.16: Conventional binders, unaged and aged: G* against δ with fixed conversion factor.
(CC = concentric cylinder, pp = parallel plate)

The ANOVA results are shown in Table 3.5. G*/sin(δ) was the dependent variable and geometry and aging condition were the independent variables. The results indicate that testing geometry is not a significant factor on G*/sin(δ) while aging condition is a significant factor, as expected. The results also show that the concentric cylinder geometry with a fixed conversion factor can provide results that are not statistically significantly different at a 95 percent confidence interval than those obtained using the parallel plate geometry. However, a significant difference was found between RFTO aged and TFO aged binders based on the Tukey Honest Significant Difference (HSD) parameter (Figure 3.17), with RTFO aging being more severe than TFO aging.

Table 3.5: Conventional Binders: ANOVA Results of G*/sin(δ) with Fixed Conversion Factor
(α=0.05)

Parameter	Df	Sum Sq	Mean Sq	F Value	Pr (>F)
Geometry	1	0.033	0.033	1.223	0.274
Binder type	2	17.15	8.575	315.28	2e-16
Residuals	50	1.360	0.027	-	-

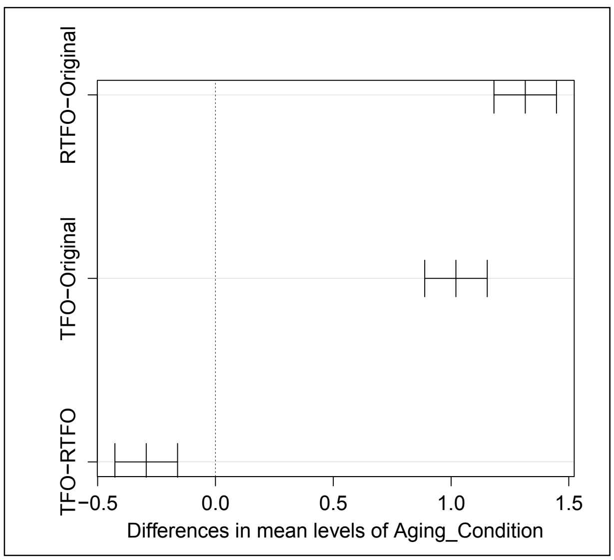

Figure 3.17: Tukey HSD with varied aging condition (95% confidence interval).

3.4 Testing Summary

Although comparatively different complex shear moduli, and consequently G*/sin(δ) values, were measured using the concentric cylinder geometry and parallel plate geometries for both unaged polymer-modified and tire rubber-modified binders, this part of the study has indicated that the results obtained from testing the same conventional, polymer-modified, and tire rubber-modified binders with concentric cylinder and parallel plate geometries in a DSR are generally not statistically different. Based on these results, the concentric cylinder geometry was considered as a potentially appropriate alternative geometry to parallel plates for quantifying the properties of asphalt rubber binders, and specifically for further comparative tests to assess the performance properties of binders containing crumb rubber particles larger than 250 µm (i.e., particles retained on the #60 sieve).

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4. TESTING ASPHALT RUBBER BINDERS

4.1 Introduction

Preliminary testing on conventional, polymer-modified, and tire rubber-modified binders using the concentric cylinder and parallel plate geometries in a dynamic shear rheometer to determine performance-related properties indicated that both geometries provided statistically similar results. Based on these results, this next part of the study was initiated to evaluate the use of the two geometries for measuring the performance properties of asphalt rubber binders produced according to Caltrans specifications, and whether the two configurations showed similar sensitivities to these variables. Three different particle size ranges were assessed, focusing on crumb rubber particles both smaller and larger than 250 µm (i.e., retained on the #60 sieve), which was identified as a key size that may influence the results of the parallel plate geometry with 2 mm gap.

4.2 Test Plan

A preliminary testing plan was developed to assess three different crumb rubber particle size ranges in wet-process asphalt rubber binders. In order to have full control over the different variables being assessed, all the binders were produced in the laboratory. The variables considered for this preliminary testing included the following:

- Binder source and grade: Paramount, PG 64-16
- Rubber content: 20 percent by weight of binder (25 percent natural rubber)
- Grinding type: ambient and cryogenic
- Extender oil: none (Type I) and four percent by weight of binder (Type II)
- Crumb rubber particle size ranges: 180 µm to 250 µm, 250 µm to 425 µm, and 425 µm to 850 µm (#40 to #20, #60 to #40, and #80 to #60, respectively)
- Aging condition: unaged

Depending on the results obtained in this part of the study, future testing will assess different binder sources, different extender oil contents, additional specific crumb rubber gradings (including gradings with particle sizes up to 2.0 mm [#10]), and different aging conditions. Repeatability and reproducibility of the two testing procedures will also be assessed. Future testing will also compare the properties of asphalt rubber binders produced in the laboratory with the properties of asphalt rubber binders produced at different asphalt plants and used on Caltrans projects.

The gap used in the concentric cylinder geometry was fixed at 6 mm and was unaltered for the tests of the binders with different particle sizes. For the parallel plate geometry testing (25 mm diameter plates) the following two different gap sizes, which were based on the crumb rubber particle sizes, were used:

- Particle size larger than 250 µm (< #60 mesh): a 2 mm gap
- Particle size smaller than 250 µm (> #60 mesh): a 1 mm gap

4.3 Binder Preparation

The asphalt rubber binders were produced by blending the individual components in a high shear mixer for 60 minutes at a temperature of 190°C (374°F). This ensured that the crumb rubber particles were appropriately swelled and had sufficient interaction with the light compounds of the asphalt binder. Blending of the crumb rubber, asphalt modifier, and asphalt binder was completed at 5,000 revolutions per minute (RPM) for the first 30 minutes, and then at 2,500 RPM for the remaining 30 minutes. This mixing process was considered to be appropriately representative of plant production for the purposes of this study. During plant production, the crumb rubber, asphalt modifier, and base binder are first mixed at high revolutions to maximize dispersion of the rubber particles, followed by mixing at slower revolutions to ensure good interaction between the rubber and the asphalt binder. The different asphalt rubber binders were produced in batches, stored in quart-size containers, and then reheated just prior to testing. In this phase of testing, comparisons were not made with plant produced asphalt rubber binders; instead the focus was on ensuring that the preparation process was consistent for all binder samples.

4.4 Test Results

The test results are listed in Table A.5 in Appendix A and summarized in Table 4.1. Plots of complex shear modulus (G*), phase angle (δ), and G*/sin(δ) are shown in Figure 4.1, Figure 4.2, and Figure 4.3, respectively. The plots show each testing point, trend lines for the results from each binder and for the combined results from the three binders, and the regression equations comparing the results from the two testing geometries for individual size ranges and for the three binders combined.

Table 4.1: Summary of Statistical Comparisons between Testing Geometries.

Particle Size Range		Correlation Between Geometries (R^2)		
µm	**#mesh**	**G* (kPa)**	**δ (°)**	**G*/sin(δ) (kPa)**
180-250	60-80	0.9973	0.9834	0.9963
250-425	40-60	0.9467	0.9621	0.9497
425-850	20-40	0.9504	0.9020	0.9490
Combined		0.9500	0.9294	0.9508

The results obtained from testing the three asphalt rubber binders, each with different maximum crumb rubber particle sizes, show a strong correlation between the two testing geometries for the finer particle size range, but increasingly poorer correlations with increasing particle size. These poorer correlations in the larger size ranges were attributed to increasing influence of the proximity of the larger rubber particles to the plates. Based on these results, the concentric cylinder geometry is considered as a potentially appropriate alternative geometry to parallel plates for quantifying the properties of asphalt rubber binders, and specifically for further comparative tests to assess the performance properties of binders containing crumb rubber particles larger than 250 µm (i.e., particles retained on the #60 sieve).

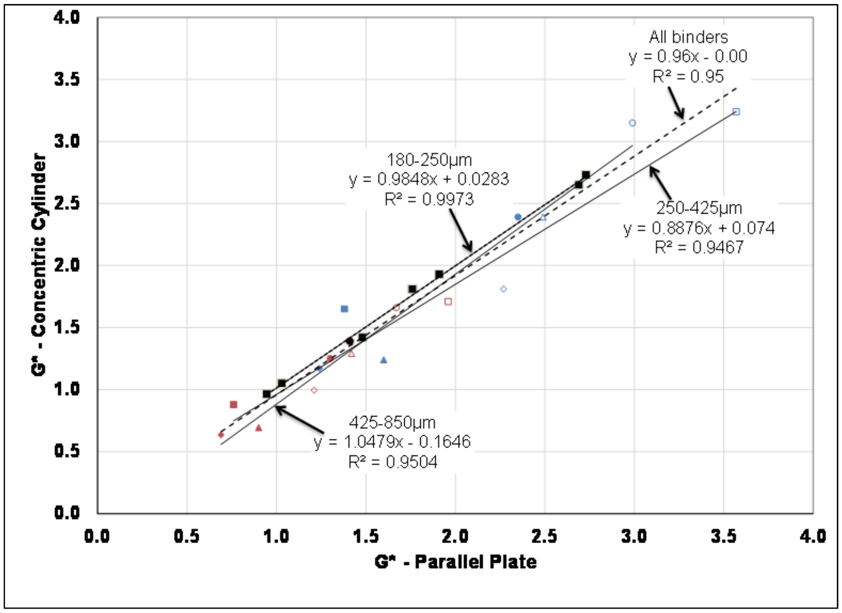

Figure 4.1: Comparison of G* results for concentric cylinder and parallel plate.

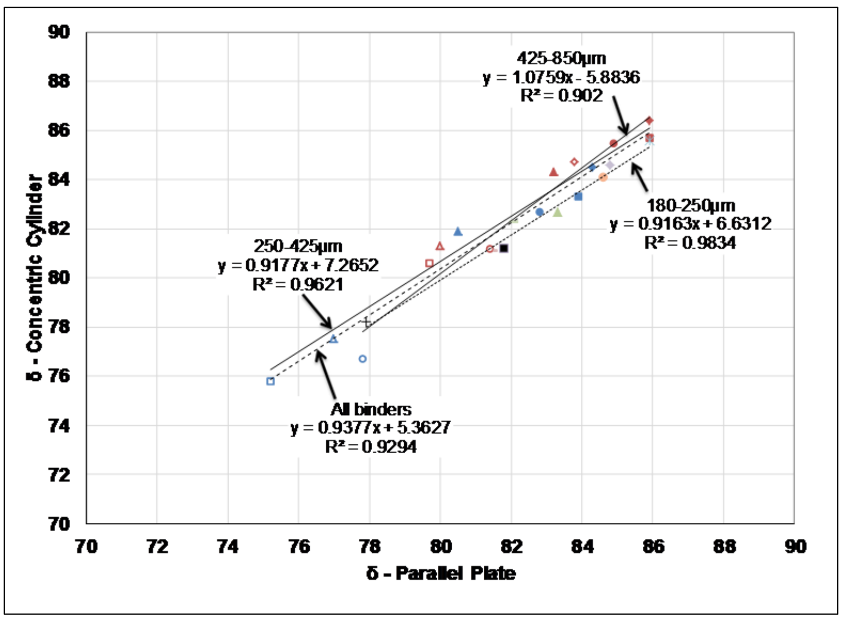

Figure 4.2: Comparison of phase angle results for concentric cylinder and parallel plate.

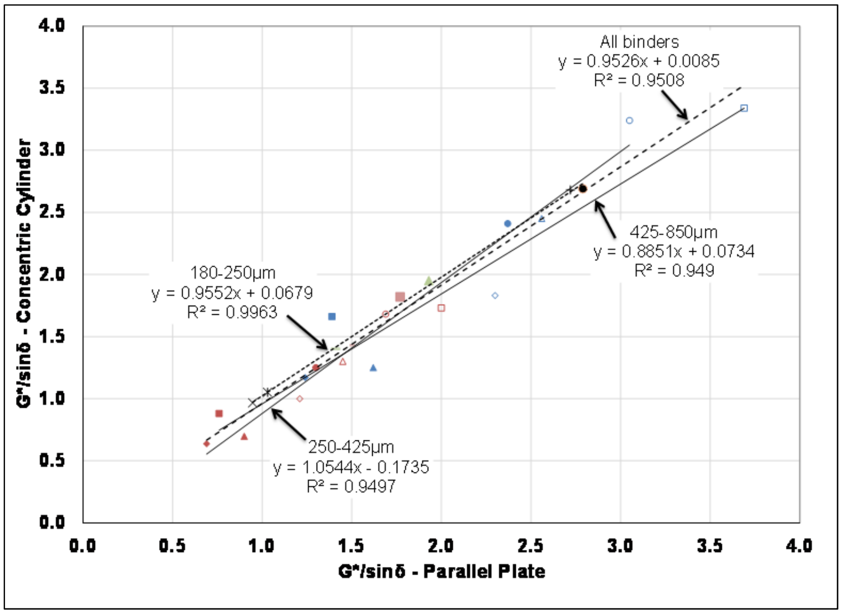

Figure 4.3: Comparison of G*/sinδ results for concentric cylinder and parallel plate.

5. EVALUATING THE EFFECTS OF RUBBER BINDER PROPERTIES

5.1 Introduction

Earlier research on rubberized asphalt has indicated that a number of factors related to the crumb rubber modifier (CRM) in the mix—the grinding method used to produce the CRM, the crumb rubber's particle size and surface area, and the crumb rubber content of the asphalt binder, as well as whether an asphalt modifier (i.e., extender oil) has been used—can all potentially influence the asphalt rubber binder's behavior and performance, and consequently that of the mix or surface treatment that includes it. This part of the study therefore focused on whether test results from the concentric cylinder and parallel plate geometries showed the same differences or similarities with respect to these variables.

5.2 Test Plan

In this part of the study, only wet-process asphalt rubber binders were tested. In order to have full control over the different variables being assessed, all binders were produced in the laboratory. The variables considered included the following:

- Binder source and grade: Paramount, PG 64-16
- Rubber content: 20 percent by weight of binder (25 percent natural rubber)
- Grinding type: ambient and cryogenic
- Extender oil: none (Type I) and four percent by weight of binder (Type II)
- Crumb rubber particle size ranges: 75 µm to 106 µm, 106 µm to 150 µm, 150 µm to 180 µm, 180 µm to 250 µm, 250 µm to 425 µm, and 425 µm to 850 µm (#40 to #20, #60 to #40, #80 to #60, #100 to #80, #140 to #100, and #200 to #140, respectively)
- Test temperatures: 76°C and 82°C (~169°F and 180°F)
- Aging condition: unaged

The surface area of the crumb rubber particles was not included as a variable in the test plan. However, the surface area of particles sampled from the products of the two different grinding types was measured and its effect on the shear modulus of the asphalt rubber binder was determined.

The test plan, summarized in Table 5.1, resulted in 24 binders being tested. To date, only testing with the parallel plate geometry has been completed. Testing with the concentric cylinder is in progress. The following two different gap sizes, based on the crumb rubber particle size, were used for testing with the parallel plates:

- Particle size larger than 250 µm (< #60 mesh): a 2 mm gap
- Particle size smaller than 250 µm (> #60 mesh): a 1 mm gap

Table 5.1: Test Plan for Assessing Rubber Particle Properties

Binder #	Grinding Type	Asphalt Modifier	CRM Size (µm)	CRM Mesh Size (#)
1	Ambient	Type I	850 – 425	#40 – 60
2			425 – 250	#60 – 40
3			250 – 180	#80 – 60
4			180 – 150	#100 – 80
5			150 – 106	#140 – 100
6			106 – 75	#200 – 140
7	Cryogenic	Type I	850 – 425	#40 – 20
8			425 – 250	#60 – 40
9			250 – 180	#80 – 60
10			180 – 150	#100 – 80
11			150 – 106	#140 – 100
12			106 – 75	#200 – 140
13	Ambient	Type II	850 – 425	#40 – 20
14			425 – 250	#60 – 40
15			250 – 180	#80 – 60
16			180 – 150	#100 – 80
17			150 – 106	#140 – 100
18			106 – 75	#200 – 140
19	Cryogenic	Type II	850 – 425	#40 – 20
20			425 – 250	#60 – 40
21			250 – 180	#80 – 60
22			180 – 150	#100 – 80
23			150 – 106	#140 – 100
24			106 – 75	#200 – 140

5.3 Binder Preparation

Asphalt rubber binders were produced as detailed in Section 4.3.

5.4 Test Results

The test results are listed in Table A.6 in Appendix A and summarized in terms of the different crumb rubber properties below.

5.4.1 Effect of Crumb Rubber Particle Size on High Temperature Grade

The true high temperatures of the performance grade (i.e., the actual high temperature grade determined as opposed to the value to the nearest 6°C used in the PG grading system) for the binders are summarized in Table 5.2 and Figure 5.1. The true grades of the binders did not show any specific trends in terms of crumb rubber particle size or whether extender oil was used. Asphalt rubber binders containing cryogenically prepared crumb rubber had a slightly higher true grade temperature compared to the binders containing crumb rubber prepared at ambient temperatures.

Table 5.2: True Grade of Laboratory-Blended Rubberized Binders

Crumb Rubber Particle Size		True Grade (°C)			
		Ambient		Cryogenic	
(µm)	(# mesh)	Type I	Type II	Type I	Type II
425 – 850	#40 – 20	84.8	78.3	87.5	80.8
250 – 425	#60 – 40	82.0	77.5	88.0	85.0
180 – 250	#80 – 60	79.9	77.5	85.4	84.6
150 – 180	#100 – 80	83.8	81.6	83.8	84.3
106 – 150	#140 – 100	83.1	80.0	85.8	84.1
75 – 106	#200 – 140	80.4	78.0	83.7	81.6

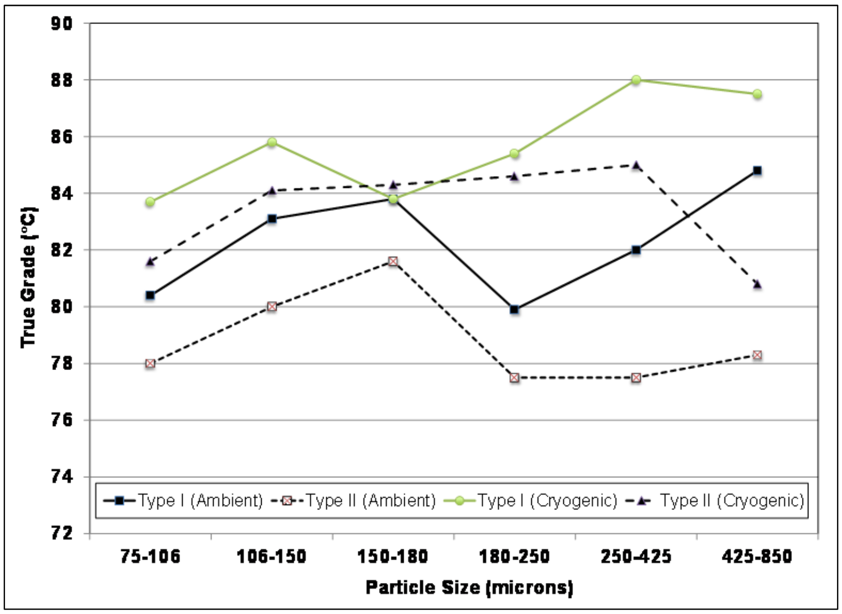

Figure 5.1: Plot of true temperature grade against crumb rubber particle size.

5.4.2 Effect of Crumb Rubber Particle Size on Shear Modulus

The G*/sin(δ) measurements at 76°C and 82°C against crumb rubber particle size for Type I and Type II asphalt binders are plotted in Figure 5.2 through Figure 5.5. Crumb rubber particles produced under both ambient and cryogenic conditions were included in the analysis. In these plots, the minimum crumb rubber particle size (in microns [µm]) was selected to represent a particle size group.

The results indicate that both the method used to produce the crumb rubber particles (i.e., grinding at ambient or cryogenic temperatures) and whether an asphalt modifier was used both influenced the shear modulus of asphalt rubber binders. Key observations include the following:

- Higher shear moduli were recorded at the lower test temperature (76°C), as expected.
- Asphalt rubber binders containing cryogenically produced crumb rubber had higher shear moduli compared to binders containing crumb rubber produced at ambient temperatures.
- There was less variation in the shear moduli determined at the higher temperature (82°C) over the range of different particle sizes compared to the shear moduli recorded at the lower temperature.

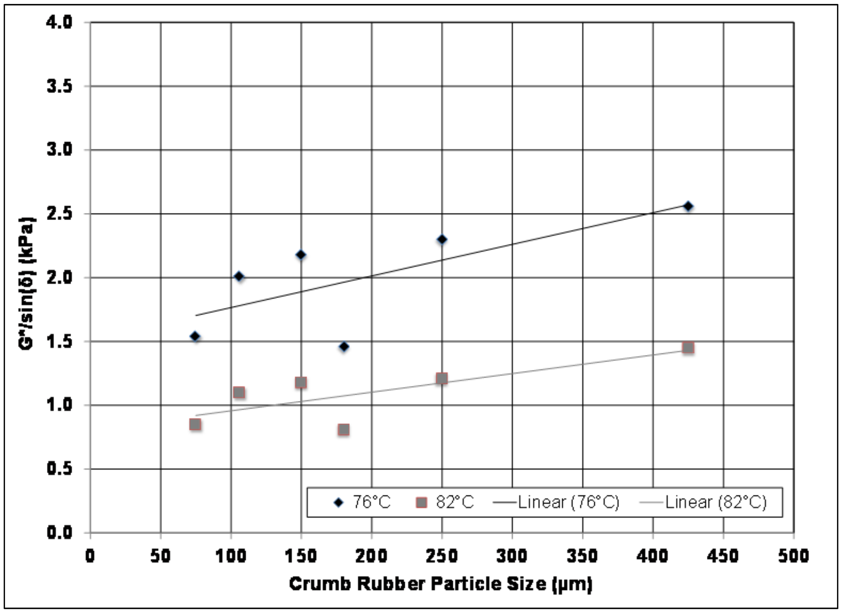

Figure 5.2: Plot of G*/sin(δ) versus particle size for Type I ambient rubber binder.

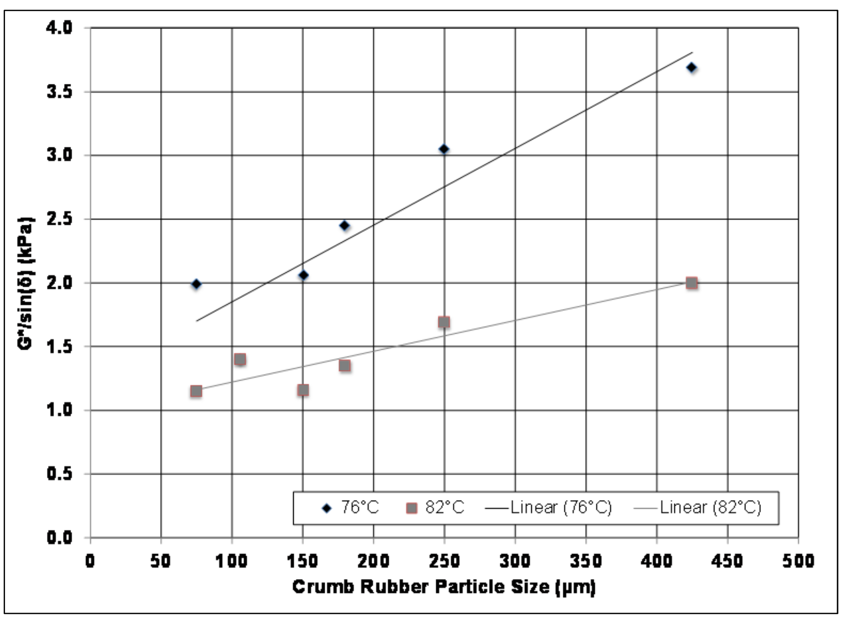

Figure 5.3: Plot of G*/sin(δ) versus particle size for Type I cryogenic rubber binder.

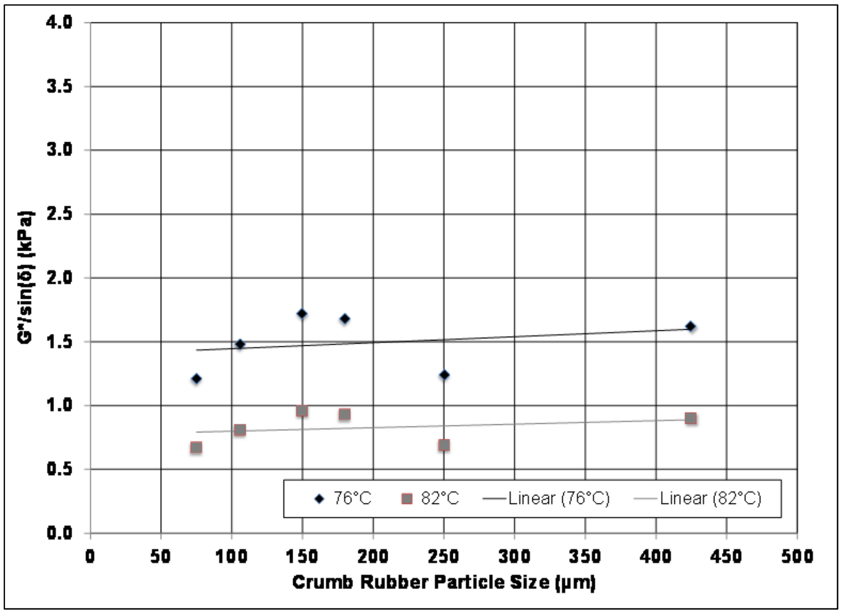

Figure 5.4: Plot of G*/sin(δ) versus particle size for Type II ambient rubber binder.

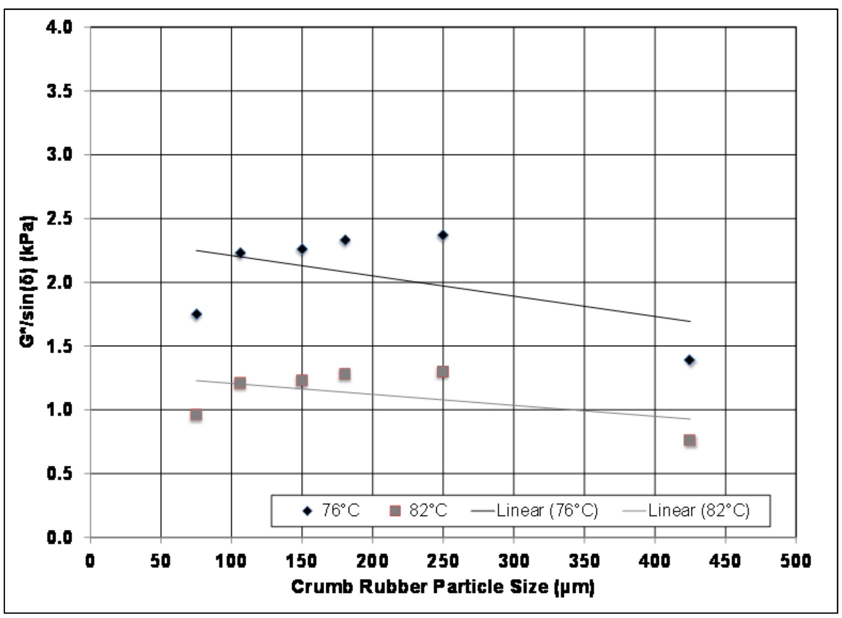

Figure 5.5: Plot of G*/sin(δ) versus particle size for Type II cryogenic rubber binder.

- Although shear modulus increased with increasing crumb rubber particle size, as expected, the differences in the shear moduli were small for the different testing variables for crumb rubber particles in the size ranges less than 200 µm compared to the differences observed for the size ranges greater than 200 µm.
- When no asphalt modifier was used (i.e., Type I binder), the shear modulus of the binder increased with increasing crumb rubber particle size at a greater rate for cryogenically produced rubber

particles compared to rubber particles produced at ambient temperatures. The difference in modulus between the 250 µm to 425 µm and 425 µm to 850 µm size ranges was significant.

- When an asphalt modifier was used (i.e., Type II binder), the difference in shear moduli among the different crumb rubber particle size ranges was small, indicating that the asphalt modifier appears to soften the rubber particles and/or enhance their digestion into the asphalt binder.
- The test results for the 425 µm to 850 µm size range of the Type II binder containing cryogenically produced crumb rubber particles were lower than expected, with a different trend than the other results in this stage of testing. Based on the results discussed in Chapter 4, this was attributed in part to the effect of particle size on the parallel plate geometry testing system, which will be confirmed after comparison when results from additional testing with the concentric cylinder geometry become available.

It is not clear at this stage of testing whether the above observations related to the larger particle sizes were attributed only to the particle size, or also to the previously noted potential limitations of the parallel plate geometry for testing large particle sizes. This will be assessed on completion of testing with the concentric cylinder geometry.

5.4.3 Effect of Crumb Rubber Particle Surface Area on Shear Modulus

The surface area of crumb rubber particles produced at ambient and cryogenic temperatures were measured by an independent accredited laboratory (*Quantachrome Instruments*). The results are listed in Table 5.3 and show that the surface area of the cryogenically produced particles was higher than that of the particles produced at ambient temperatures, indicating that particles produced in the two processes have different shapes. The difference in surface area increased with decreasing particle size, as expected, with differences between the two processes significant on particle sizes smaller than 250 µm (#60). It is worth noting that two different gasses (nitrogen and krypton) were used for the surface area measurements. Choice of gas is usually dependent on the surface roughness of the particles being evaluated. The observed differences in results could be attributed to this choice of gas.

Table 5.3: Surface Area of Rubber Particles Produced at Ambient and Cryogenic Temperatures

Crumb Rubber Particle Size		Surface Area (m^2/g)	
(µm)	(# mesh)	Ambient	Cryogenic
425 – 850	#40 – 20	0.035^{Kr}	0.039^{Kr}
250 – 425	#60 – 40	0.036^{Kr}	0.052^{Kr}
180 – 250	#80 – 60	0.077^{Kr}	0.231^{Kr}
150 – 180	#100 – 80	0.278^{Ni}	0.186^{Ni}
106 – 150	#140 – 100	0.131^{Ni}	0.245^{Ni}
75 – 106	#200 – 140	0.138^{Kr}	0.668^{Ni}

Kr : Krypton gas was used to measure surface area
Ni: Nitrogen gas was used to measure surface area

The DSR test results are summarized in Figure 5.6 through Figure 5.9 and indicate that surface area did not significantly influence the shear modulus of the asphalt rubber binder if an asphalt modifier was used (i.e., Type II binders). When no asphalt modifier was used, some influence of surface area was evident, with shear moduli dropping with increasing surface area. Other observations were consistent with the results presented in Section 5.4.2, and include the following:

- Higher shear moduli were recorded at the lower test temperature (76°C), as expected.
- Asphalt rubber binders containing cryogenically produced crumb rubber had higher shear moduli than binders containing crumb rubber produced at ambient temperatures.
- There was less variation in the shear moduli determined at the higher temperature (82°C) over the range of different particle sizes compared to the shear moduli recorded at the lower temperature.
- Although shear modulus increased with increasing crumb rubber particle size (i.e., smaller surface area), as expected, the differences in the shear moduli were again small for the different testing variables for crumb rubber particles in the size ranges less than 200 µm compared to the differences observed for the size ranges greater than 200 µm.
- When no asphalt modifier was used (i.e., Type I binder), the shear modulus of the binder again increased with increasing crumb rubber particle size (i.e., decreasing surface area) at a greater rate for cryogenically produced rubber particles compared to those for rubber particles produced at ambient temperatures. The difference in modulus between the 250 µm to 425 µm and 425 µm to 850 µm size ranges (i.e., smaller surface area) was again significant.

It is not clear at this stage of testing whether the above observations related to the larger particle sizes (i.e., smaller surface areas) were attributable only to particle size, or whether they were also attributable to the previously noted potential limitations of the parallel plate geometry for testing large particle sizes. This will also be assessed on completion of testing with the concentric cylinder geometry.

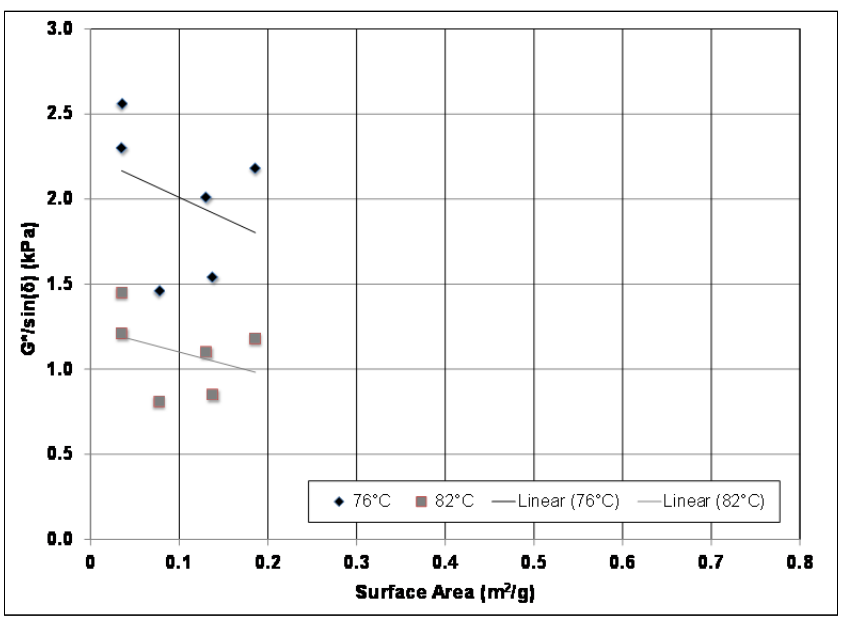

Figure 5.6: Plot of G*/sin(δ) versus surface area for Type I ambient rubber binder.

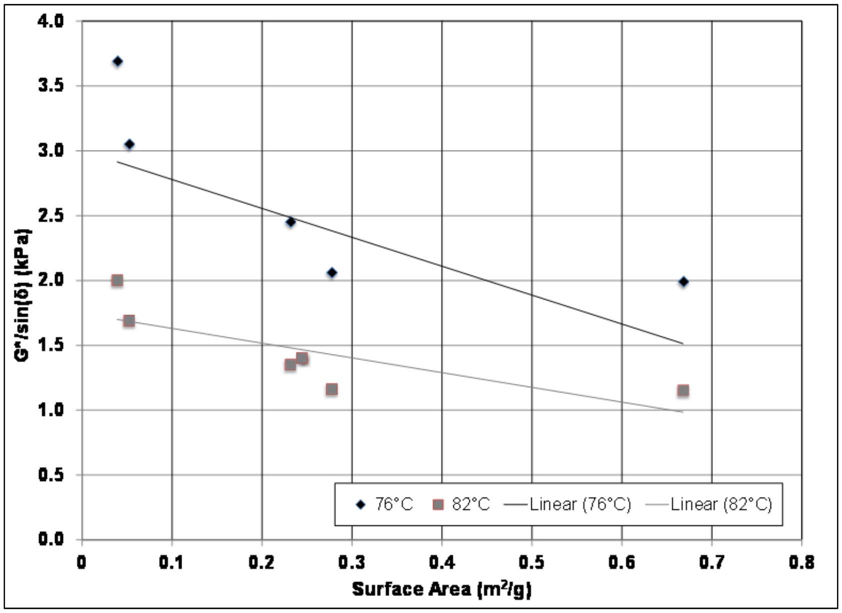

Figure 5.7: Plot of G*/sin(δ) versus surface area for Type I cryogenic rubber binder.

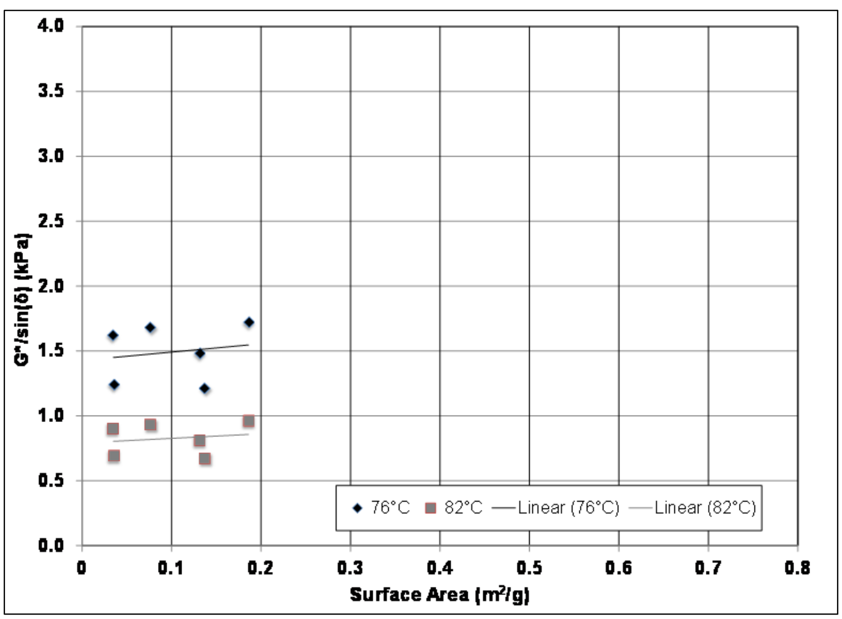

Figure 5.8: Plot of G*/sin(δ) versus surface area for Type II ambient rubber binder.

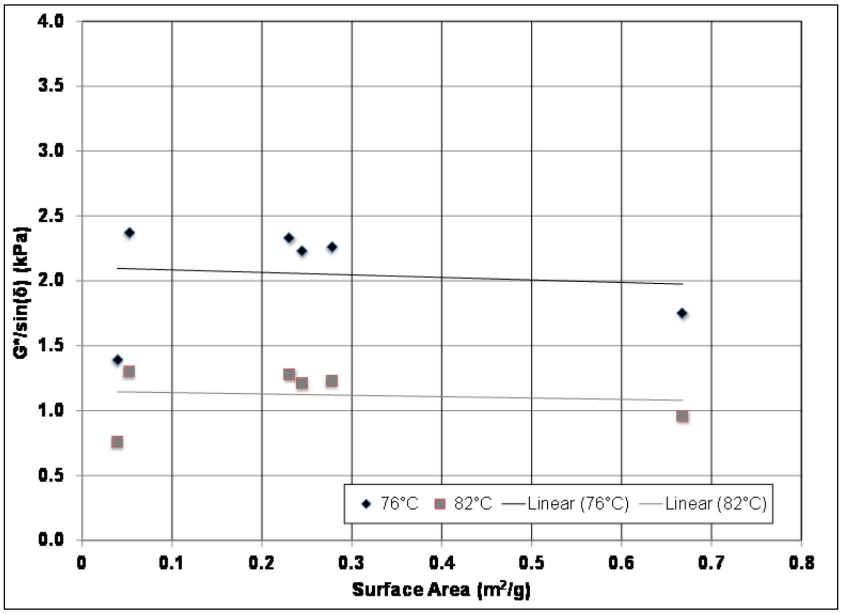

Figure 5.9: Plot of G*/sin(δ) versus surface area for Type II cryogenic rubber binder.

5.5 Testing Summary

This stage of the study covered DSR testing with parallel plate geometry to assess the influences of a selected crumb rubber production method (i.e., at ambient or cryogenic temperatures) and the use of asphalt modifiers on the performance properties of asphalt rubber binders. Testing with the concentric cylinder geometry is in progress. The results obtained to date indicate that both the production method and the use of an asphalt modifier influence the performance properties of asphalt rubber binder, with the use of asphalt modifiers having the larger influence.

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6. CONCLUSIONS AND INTERIM RECOMMENDATIONS

6.1 Project Summary

This technical memorandum documents the first phase of a study to investigate test methods for measuring the performance properties of asphalt rubber binders produced according to Caltrans spceifications. The current method of viscosity testing used by Caltrans is deemed to be an insufficient measure of performance for these types of binders compared to the testing requirements for conventional, polymer-modified, and tire rubber-modified binders. This phase of the study consisted of preliminary testing to compare two different dynamic shear rheometer (DSR) geometries, with a view to making recommendations about adopting similar testing criteria for asphalt rubber binders to supplement those currently used for conventional and other modified binders.

The high temperature properties of conventional and other modified binders are typically measured through testing with a DSR using parallel plate geometry, with the gap size between the plates dependent on the size of any particulates in the binder. A 2.0 mm gap size is considered to be the maximum appropriate gap for testing asphalt binders, provided that no particulates in the binder exceed the AASHTO/ASTM recommended maximum particle size of 0.25 mm (or 250 µm [#60]). However, Caltrans specifications allow crumb rubber particles up to 2.36 mm (i.e., passing the #8 sieve), which is considerably larger than this maximum recommended size. Consequently, the appropriateness of the parallel plate geometry for testing asphalt rubber binders is questionable, given that the rheology of the large rubber particles may dominate the DSR result and give misleading performance parameters for the binder properties. This study therefore assessed an alternative geometry, namely the concentric cylinder, which can accommodate larger particles in the asphalt binder. The two geometries were compared using conventional, polymer-modified, tire rubber-modified (terminal blend), and wet-process asphalt rubber binders. Key findings from the work completed to date include the following:

- The results obtained from testing the same conventional, polymer-modified, and tire rubber-modified binders with concentric cylinder and parallel plate geometries in a DSR were statistically similar.
- The results obtained from testing asphalt rubber binders with three different crumb rubber particle size ranges (180 µm to 250 µm, 250 µm to 425 µm, and 425 µm to 850 µm [#40 to #20, #60 to #40, and #80 to #60, respectively]), showed a strong correlation between the two testing geometries for the finer particle size ranges, but increasingly poorer correlations with increasing particle size. These poorer correlations in the larger size ranges were attributable in part to the increasing influence of the proximity of the larger rubber particles to the plates.
- Using parallel plate geometry, an assessment of the effects of different crumb rubber properties on the properties of asphalt rubber binders indicated that both production method (i.e., crushing at ambient or cryogenic temperatures) and whether an asphalt modifier was used influenced the

performance properties of the asphalt rubber binder, with the use of asphalt modifiers having the larger influence. Testing is continuing with the concentric cylinder geometry, after which the results from the two testing geometries will be compared.

6.2 Conclusions

Based on these results obtained to date, the concentric cylinder geometry is considered to be a potentially appropriate alternative geometry to parallel plates for quantifying the properties of California-produced asphalt rubber binders, and specifically for assessing the performance properties of binders containing crumb rubber particles larger than 250 µm (i.e., particles retained on the #60 sieve). Additional testing of a larger number of binders is required to confirm these initial findings. The concentric cylinder geometry requires a larger binder sample for testing and takes longer to complete the tests.

6.3 Recommendations

Initial results support the continuation of testing to assess the appropriateness of using the concentric cylinder geometry for measuring the performance properties of asphalt rubber binders that are produced according to Caltrans specifications using a wet process with crumb rubber particles that exceed 0.25 mm (#60 mesh) in size. This testing should be in line with the original workplan and objectives prepared for this project, and should investigate additional binder sources, the development of appropriate binder aging protocols, the development of a suitable low temperature testing method, the repeatability and reproducibility values of any proposed test methods, and the applicability of the results to the actual performance properties of mixes produced with asphalt rubber binders.

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APPENDIX A: TEST RESULTS

Test results from the different tests are summarized in the following tables:

- Table A.1: Test Results for Binder-Specific Conversion Factor: Operator and Binder Source
- Table A.2: Test Results for Binder-Specific Conversion Factor: Operator and Binder Type
- Table A.3: Test Results for Binder-Specific Conversion Factor: Operator and Binder Source, Type and Grade
- Table A.4: Test Results for Fixed Conversion Factor
- Table A.5: Rubberized Binder: Comparison of Concentric Cylinder and Parallel Plate
- Table A.6: Test Results for Rubberized Binder: Parallel Plate

Abbreviations in the tables are as follows:

- Binder source
  + P = Paramount
  + SJ = San Joaquin
  + V = Valero
- Binder type
  + Con = conventional
  + PM = polymer-modified
  + TR = tire rubber-modified
- Aging condition
  + Unaged
  + RTFO = Rolling thin film oven-aged
- Grinding method
  + Amb = ambient
  + Cryo = Cryogenic
- DSR geometry
  + CC = concentric cylinder
  + PP-1= parallel plate with 1 mm gap
  + PP-2 = parallel plate with 2 mm gap
- Test parameter
  + G* = Shear modulus
  + δ = Phase angle

Table A.1: Test Results for Binder-Specific Conversion Factor: Operator and Binder Source

Operator	Binder Type	PG Grade	Aging Condition	Binder Source	Geometry	G* (kPa)	δ (Degrees)	G*/sin(δ) (kPa)
1	Con	64-16	Unaged	P	CC	1.40 1.41 1.43	87.6 87.7 87.6	1.41 1.41 1.43
					PP-1	1.46 1.35 1.37	87.7 87.8 87.8	1.46 1.35 1.37
				SJ	CC	1.12 1.07 1.07	89.4 89.5 89.4	1.12 1.07 1.07
					PP-1	1.15 1.09 1.10	89.4 89.6 89.5	1.15 1.09 1.10
				V	CC	1.25 1.22 1.23	87.4 87.5 87.5	1.25 1.22 1.23
					PP-1	1.24 1.26 1.24	87.8 87.7 87.7	1.24 1.26 1.24
2	Con	64-16	Unaged	P	CC	1.42 1.43 1.41	87.6 87.6 87.6	1.42 1.43 1.41
					PP-1	1.38 1.45 1.47	87.8 87.7 87.7	1.38 1.45 1.47
				SJ	CC	1.08 1.09 1.03	89.4 89.4 89.5	1.08 1.09 1.03
					PP-1	1.09 1.09 1.09	89.5 89.5 89.5	1.09 1.09 1.09
				V	CC	1.28 1.28 1.27	87.4 87.5 87.5	1.28 1.28 1.27
					PP-1	1.29 1.25 1.28	87.7 87.7 87.7	1.29 1.25 1.28
3	Con	64-16	Unaged	P	CC	1.51 1.46 1.49	87.5 87.6 87.6	1.51 1.46 1.49
					PP-1	1.42 1.59 1.51	87.5 87.5 87.6	1.42 1.59 1.51
				SJ	CC	1.17 1.14 1.19	89.5 89.5 89.4	1.17 1.14 1.19
					PP-1	1.18 1.12 1.15	89.4 89.5 89.4	1.19 1.12 1.15
				V	CC	1.31 1.30 1.27	87.4 87.4 87.5	1.31 1.30 1.27
					PP-1	1.30 1.30 1.28	87.6 87.6 87.6	1.30 1.31 1.28

Table A.2: Test Results for Binder-Specific Conversion Factor: Operator and Binder Type

Operator	Binder Source	PG Grade	Aging Condition	Binder Type	Geometry	G* (kPa)	δ (Degrees)	G*/sin(δ) (kPa)
1	P	64-28	Unaged	PM	CC	1.47	70.6	1.56
						1.84	68.8	1.98
						1.81	68.5	1.94
					PP-1	1.69	67.6	1.82
						2.12	66.1	2.32
						1.97	65.8	2.16
				TR	CC	2.80	67.1	3.03
						2.97	66.8	3.23
						2.22	68.5	2.38
					PP-1	2.78	66.0	3.04
						2.11	65.6	2.32
						1.78	66.3	1.95
2	P	64-28	Unaged	PM	CC	1.47	70.7	1.55
						1.83	68.7	1.96
						1.89	68.4	2.04
					PP-1	1.55	68.4	1.67
						1.91	66.1	2.09
						1.98	66.3	2.17
				TR	CC	2.81	67.0	3.06
						2.37	68.3	2.55
						2.38	68.0	2.57
					PP-1	2.25	66.3	2.45
						2.05	66.0	2.25
						2.02	65.7	2.22
3	P	64-28	Unaged	P	CC	1.58	67.9	1.71
						1.61	69.6	1.72
						1.59	68.8	1.70
					PP-1	1.65	68.1	1.78
						2.05	65.4	2.25
						2.11	66.2	2.31
				SJ	CC	2.86	67.0	3.11
						2.98	66.7	3.24
						3.00	66.7	3.27
					PP-1	3.06	65.2	3.38
						2.11	65.6	2.31
						1.98	67.0	2.16

Table A.3: Test Results for Binder-Specific Conversion Factor: Operator and Binder Source, Type and Grade

Operator	Aging Condition	PG Grade	Binder Source	Binder Type	Geometry	G* (kPa)	δ (Degrees)	G*/sin(δ) (kPa)
1	RTFO	64-28	P	PM	CC	3.77	62.4	4.26
					PP-1	3.75	62.4	4.23
			P	TR	CC	4.86	64.0	5.40
					PP-1	4.76	63.5	5.32
		64-16	SJ	Con	CC	2.69	88.6	2.69
					PP-1	2.34	88.6	2.34
			V	Con	CC	3.01	83.5	3.03
					PP-1	2.97	84.8	2.99
2	RTFO	64-28	P	PM	CC	3.56	62.7	4.01
					PP-1	3.88	62.3	4.38
			P	TR	CC	3.61	64.2	4.01
					PP-1	3.25	63.8	3.62
		64-16	SJ	Con	CC	2.76	88.5	2.76
					PP-1	2.28	88.6	2.28
			V	Con	CC	2.8	84.4	2.81
					PP-1	3.07	84.7	3.09
3	RTFO	64-28	P	PM	CC	3.74	62.3	4.23
					PP-1	3.82	62.2	4.31
			P	TR	CC	3.59	64.1	3.99
					PP-1	3.5	63.4	3.92
		64-16	SJ	Con	CC	2.71	88.6	2.71
					PP-1	2.4	88.6	2.41
			V	Con	CC	2.45	84.8	2.46
					PP-1	2.91	84.9	2.92

Table A.4: Test Results for Fixed Conversion Factor

Operator	Binder Source	PG Grade	Test Temp. (°C)	Aging Condition	Geometry	G* (kPa)	δ (Degrees)	G*/sin(δ) (kPa)
1	V	70-10	70	Unaged	CC	1.40	87.2	1.40
						1.39	87.2	1.39
						1.41	87.1	1.41
					PP-1	1.32	87.3	1.32
						1.27	87.3	1.27
						1.32	87.3	1.32
				TFO	CC	2.43	85.2	2.44
						2.46	85.2	2.47
						2.56	85.0	2.57
					PP-1	2.53	85.1	2.54
						2.58	85.1	2.59
						2.57	85.1	2.58
				RTFO	CC	2.74	85.1	2.75
						2.73	85.1	2.74
						2.71	85.1	2.72
					PP-1	2.58	85.3	2.59
						2.60	85.3	2.61
						2.61	85.3	2.62
	SJ	64-16	64	Unaged	CC	1.28	89.4	1.28
						1.30	89.4	1.30
						1.27	89.4	1.27
					PP-1	1.20	89.4	1.20
						1.22	89.4	1.22
						1.20	89.4	1.20
				TFO	CC	2.02	88.8	2.02
						2.17	88.7	2.17
						2.06	88.8	2.06
					PP-1	2.07	88.8	2.07
						2.05	88.8	2.05
						2.06	88.8	2.06
				RTFO	CC	2.42	88.5	2.42
						2.46	88.5	2.46
						2.43	88.5	2.43
					PP-1	2.32	88.6	2.32
						2.31	88.6	2.31
						2.29	88.6	2.29
	V	58-22	58	Unaged	CC	1.30	88.4	1.30
						1.30	88.4	1.30
						1.33	88.4	1.33
					PP-1	1.31	88.5	1.31
						1.27	88.4	1.27
						1.26	88.4	1.26
				TFO	CC	2.32	87.0	2.32
						2.26	87.0	2.26
						2.36	87.0	2.36
					PP-1	2.39	87.0	2.39
						2.38	87.0	2.38
						2.40	87.0	2.40
				RTFO	CC	2.84	86.4	2.85
						2.84	86.4	2.85
						2.84	86.4	2.85
					PP-1	2.74	86.6	2.74
						2.76	86.6	2.76
						2.71	86.6	2.71

Table A.5: Rubberized Binder: Comparison of Concentric Cylinder and Parallel Plate

Geometry	Particle Size (µm)	Particle Size (# mesh)	Grind Method	Test Temp. (°C)	Binder Type	G* (kPa)	δ (Degrees)	G*/sin(δ) (kPa)
CC	180-250	60-80	Amb	76	I	1.81	82.7	1.82
					II	1.93	81.2	1.95
				82	I	0.96	85.6	0.97
					II	1.05	84.1	1.05
			Cryo	76	I	2.73	78.2	2.69
					II	2.65	81.1	2.68
				82	I	1.42	82.3	1.43
					II	1.39	84.6	1.40
	250-425	40-60	Amb	76	I	1.81	82.3	1.83
					II	1.17	84.5	1.17
				82	I	1.00	84.7	1.00
					II	0.64	86.4	0.64
			Cryo	76	I	3.15	76.7	3.24
					II	2.39	82.7	2.41
				82	I	1.66	81.2	1.68
					II	1.25	85.5	1.25
	425-850	20-40	Amb	76	I	2.39	77.5	2.45
					II	1.24	81.9	1.25
				82	I	1.29	81.3	1.30
					II	0.69	84.3	0.67
			Cryo	76	I	3.24	75.8	3.34
					II	1.65	83.3	1.66
				82	I	1.71	80.6	1.73
					II	0.88	85.7	0.88
PP-1	180-250	60-80	Amb	76	I	1.76	83.3	1.77
					II	1.91	81.8	1.93
				82	I	0.95	85.9	0.95
					II	1.03	84.6	1.03
			Cryo	76	I	2.73	77.9	2.79
					II	2.69	81.5	2.72
				82	I	1.48	82.1	1.49
					II	1.41	84.8	1.41
PP-2	250-425	40-60	Amb	76	I	2.27	80.0	2.30
					II	1.24	84.3	1.24
				82	I	1.21	83.8	1.21
					II	0.69	85.9	0.69
			Cryo	76	I	2.99	77.8	3.05
					II	2.35	82.8	2.37
				82	I	1.67	81.4	1.69
					II	1.30	84.9	1.30
	425-850	20-40	Amb	76	I	2.49	77.0	2.56
					II	1.60	80.5	1.62
				82	I	1.42	80.0	1.45
					II	0.90	83.2	0.90
			Cryo	76	I	3.57	75.2	3.69
					II	1.38	83.9	1.39
				82	I	1.96	79.7	2.00
					II	0.76	85.9	0.76

Table A.6: Test Results for Rubberized Binder: Parallel Plate

Binder Type	Grinding Method	Test Temp. (°C)	Geometry	Particle Size (µm)	Particle Size (# mesh)	G* (kPa)	δ (Degrees)	G*/sin(δ) (kPa)
I	Amb	76	PP-2	425-850	20-40	2.49	77.0	2.56
				250-425	40-60	2.27	80.0	2.31
			PP-1	180-250	60-80	1.76	83.8	1.46
				150-180	80-100	2.16	81.2	2.18
				106-150	100-140	1.98	80.0	2.01
				75-106	140-200	1.53	82.0	1.54
		82	PP-2	425-850	20-40	1.42	80.0	1.45
				250-425	40-60	1.21	83.8	1.21
			PP-1	180-250	60-80	0.95	85.6	0.81
				150-180	80-100	1.17	83.7	1.18
				106-150	100-140	1.10	82.6	1.10
				75-106	140-200	0.84	84.2	0.85
	Cryo	76	PP-2	425-850	20-40	3.57	75.2	3.69
				250-425	40-60	2.99	77.8	3.05
			PP-1	180-250	60-80	2.73	78.4	2.45
				150-180	80-100	2.01	76.6	2.06
				106-150	100-140	1.38	79.5	1.40
				75-106	140-200	1.93	76.8	1.99
		82	PP-2	425-850	20-40	1.96	79.7	2.00
				250-425	40-60	1.67	81.4	1.69
			PP-1	180-250	60-80	1.48	81.8	1.35
				150-180	80-100	1.14	80.3	1.16
				106-150	100-140	1.47	82.8	1.48
				75-106	140-200	1.13	79.6	1.15
II	Amb	76	PP-2	425-850	20-40	1.60	80.5	1.62
				250-425	40-60	1.24	84.3	1.24
			PP-1	180-250	60-80	1.91	81.9	1.68
				150-180	80-100	1.69	80.5	1.72
				106-150	100-140	0.81	84.7	0.81
				75-106	140-200	1.20	82.9	1.21
		82	PP-2	425-850	20-40	0.90	83.2	0.90
				250-425	40-60	0.69	85.9	0.69
			PP-1	180-250	60-80	1.03	84.0	0.93
				150-180	80-100	0.95	82.8	0.96
				106-150	100-140	2.20	81.5	2.23
				75-106	140-200	0.67	84.7	0.67
	Cryo	76	PP-2	425-850	20-40	1.38	83.9	1.39
				250-425	40-60	2.35	82.8	2.37
			PP-1	180-250	60-80	2.69	82.4	2.33
				150-180	80-100	2.24	82.3	2.26
				106-150	100-140	1.20	84.0	1.21
				75-106	140-200	1.74	83.2	1.75
		82	PP-2	425-850	20-40	0.76	85.9	0.76
				250-425	40-60	1.30	84.9	1.30
			PP-1	180-250	60-80	1.41	84.8	1.28
				150-180	80-100	1.22	84.7	1.23
				106-150	100-140	1.20	84.0	1.21
				75-106	140-200	0.95	85.2	0.96