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Developing a Laboratory Protocol for Asphalt Binder Recovery

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Final Report VCTIR 15-R7

Standard Title Page - Report on Federally Funded Project

1. Report No.: FHWA/VCTIR 15-R7	2. Government Accession No.:	3. Recipient's Catalog No.:	
4. Title and Subtitle: Developing a Laboratory Protocol for Asphalt Binder Recovery		5. Report Date: October 2014	
		6. Performing Organization Code:	
7. Author(s): Stacey D. Diefenderfer, Ph.D., P.E.		8. Performing Organization Report No.: VCTIR 15-R7	
9. Performing Organization and Address: Virginia Center for Transportation Innovation and Research 530 Edgemont Road Charlottesville, VA 22903		10. Work Unit No. (TRAIS):	
		11. Contract or Grant No.: 101802	
12. Sponsoring Agencies' Name and Address: Virginia Department of Transportation Federal Highway Administration 1401 E. Broad Street 400 North 8th Street, Room 750 Richmond, VA 23219 Richmond, VA 23219-4825		13. Type of Report and Period Covered: Final	
		14. Sponsoring Agency Code:	
15. Supplementary Notes:			
16. Abstract: <p>Asphalt binder extraction and recovery are common laboratory procedures used to provide material for research and quality assurance testing. The most common methods of recovery performed today include the Abson method and the rotary evaporator (or Rotavap) method. The purpose of this study was to compare the Rotavap method proposed for use at the asphalt binder laboratory of the Virginia Center for Transportation Innovation and Research to the Abson method currently in use at the asphalt materials laboratory of the Virginia Department of Transportation Materials Division to provide for continuity in test results.</p> <p>Blank determinations were produced, recovered, and tested to provide comparative data for the two methods of recovery. In addition, base binders used in the production of the blank determinations were tested under the same conditioning protocols as those for the recovered binder to provide baseline data. Analysis of the test data indicated that using both the Rotavap and Abson methods affected the resultant binder properties; however, the impacts were similar for both methods. There were few significant differences between the test results using the two methods, and neither method was found to result in any change in the resultant binder grade of a recovered binder as compared to that of the base binder.</p> <p>Based on these findings, it was concluded that the Rotavap method provides recovered binders that are comparable to those recovered using the Abson method. It was recommended that VCTIR continue to use the Rotavap method to recover binders and proceed with efforts to become accredited by the AASHTO Materials Reference Laboratory in the use of Rotavap recovery.</p>			
17 Key Words: asphalt binder, recovery, Rotavap, performance grade		18. Distribution Statement: No restrictions. This document is available to the public through NTIS, Springfield, VA 22161.	
19. Security Classif. (of this report): Unclassified	20. Security Classif. (of this page): Unclassified	21. No. of Pages: 26	22. Price:

FINAL REPORT

DEVELOPING A LABORATORY PROTOCOL FOR ASPHALT BINDER RECOVERY

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In Cooperation with the U.S. Department of Transportation
Federal Highway Administration

Virginia Center for Transportation Innovation and Research
(A partnership of the Virginia Department of Transportation
and the University of Virginia since 1948)

Charlottesville, Virginia

October 2014
VCTIR 15-R7

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ABSTRACT

Asphalt binder extraction and recovery are common laboratory procedures used to provide material for research and quality assurance testing. The most common methods of recovery performed today include the Abson method and the rotary evaporator (or Rotavap) method. The purpose of this study was to compare the Rotavap method proposed for use at the asphalt binder laboratory of the Virginia Center for Transportation Innovation and Research to the Abson method currently in use at the asphalt materials laboratory of the Virginia Department of Transportation Materials Division to provide for continuity in test results.

Blank determinations were produced, recovered, and tested to provide comparative data for the two methods of recovery. In addition, base binders used in the production of the blank determinations were tested under the same conditioning protocols as those for the recovered binder to provide baseline data. Analysis of the test data indicated that using both the Rotavap and Abson methods affected the resultant binder properties; however, the impacts were similar for both methods. There were few significant differences between the test results using the two methods, and neither method was found to result in any change in the resultant binder grade of a recovered binder as compared to that of the base binder.

Based on these findings, it was concluded that the Rotavap method provides recovered binders that are comparable to those recovered using the Abson method. It was recommended that VCTIR continue to use the Rotavap method to recover binders and proceed with efforts to become accredited by the AASHTO Materials Reference Laboratory in the use of Rotavap recovery.

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DEVELOPING A LABORATORY PROTOCOL FOR ASPHALT BINDER RECOVERY

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INTRODUCTION

The extraction and recovery of asphalt binders from asphalt mixtures is a relatively common laboratory procedure and is used to provide material for research and quality assurance testing. However, several methods are commonly used to extract the binder from the mixture and a number of methods are used to recover the binder from the solvent used during the extraction process. The extraction processes differ in their use of heat, method of agitating the mixture, and allowable solvents. The two most common methods of asphalt recovery performed today include the Abson method, introduced in 1933, and the rotary evaporation (Rotavap) method, which became common in the mid-1970s (Burr et al., 1990).

Currently, the asphalt materials laboratory at the Virginia Department of Transportation (VDOT) Materials Division (hereinafter VDOT Materials Division laboratory) performs extraction in accordance with AASHTO T 164, Quantitative Extraction of Asphalt Binder from Hot Mix Asphalt (HMA), Method A (American Association of State Highway and Transportation Officials [AASHTO], 2012), and recovery in accordance with AASHTO T 170, Recovery of Asphalt From Solution by Abson Method (AASHTO, 2012). N-propyl bromide (nPB) (Lenium RV) is used as the solvent. The asphalt binder laboratory at the Virginia Center for Transportation Innovation and Research (VCTIR) (hereinafter VCTIR laboratory) acquired equipment to extract asphalt binders from asphalt mixtures and recover the binder from solvent using an alternative procedure. The method for this procedure is AASHTO T 164, Method A, for the extraction process and AASHTO T 319, Quantitative Extraction and Recovery of Asphalt Binder from Asphalt Mixtures (AASHTO, 2012), for the recovery process. The Rotavap method was chosen in lieu of the Abson method as greater volumes of binder solvent solution can be recovered, thus increasing productivity. In addition, it has been found that the Rotavap method is more efficient in removing solvent (Burr et al., 1993).

Through the years, a number of solvents have been used for asphalt extraction (Burr et al., 1990). Initially, carbon disulfide (CS₂) was commonly used but was phased out because of its high flammability and volatility. It was replaced with benzene when the Abson method was introduced. Chlorinated solvents, such as trichloroethylene (TCE) and 1,1,1-trichloroethane, became popular in the 1950s and 1960s. The use of benzene was phased out because of carcinogenic findings. TCE remains in use as a solvent and is the standard solvent specified by many test methods, but as a result of its health effects and environmental impacts, toluene has become a more popular alternative. Even more recently, the use of nPB was introduced as a more environmentally friendly solvent with reduced health impacts. The use of either nPB or toluene has been suggested to minimize environmental safety and health effects. The VCTIR

laboratory proposes using nPB rather than toluene, as it is less flammable. This report documents the results of a study developed to compare the recovery method proposed for use at the VCTIR laboratory to that currently used at the VDOT Materials Division laboratory to provide for continuity in test results.

PURPOSE AND SCOPE

The purpose of this study was to validate the use of the Rotavap method for recovering asphalt binders as an appropriate method to be used at the VCTIR laboratory. In addition, the study compared binders recovered by the Rotavap method and the Abson method as used by the VDOT Materials Division laboratory to determine if the binder properties determined by each method were comparable. The objective was met by collecting samples of asphalt binders, blending the binders with solvent, performing recoveries at the VCTIR and VDOT's Materials Division laboratories, testing the base and recovered binders, and comparing the results to determine if the choice of recovery method had a significant impact on the recovered binder properties.

METHODS

Binder Sampling and Specimen Preparation

Asphalt binder was sampled from various sources to provide materials for blending and recovery. Three binder performance grades (PGs) commonly used in Virginia, PG 64-22, PG 70-22, and PG 76-22, from each of two suppliers, denoted Supplier 1 and Supplier 2, were evaluated. Binders were blended with nPB solvent to produce blank determinations in accordance with AASHTO T 170 (AASHTO, 2012). Eight blank determination specimens were made for each binder evaluated. Four specimens were assigned to each laboratory for recovery.

Binder Recovery

Binder was recovered from the blank determination specimens at the VCTIR laboratory using the recovery procedure specified in AASHTO T 319 (AASHTO, 2012). Binder was recovered from the blank determination specimens at the VDOT Materials Division laboratory using the method specified in AASHTO T 170 (AASHTO, 2012). Each laboratory performed the recovery procedure on one blank determination specimen at a time. All recovery work in each laboratory was performed by the same technician to eliminate multi-operator variability. After the recovery process was performed, pairs of recovered specimens were combined to provide enough recovered binder to perform performance grading, as shown in Figure 1. Each grade and supplier combination resulted in two replicate recovered samples from each laboratory for grading.

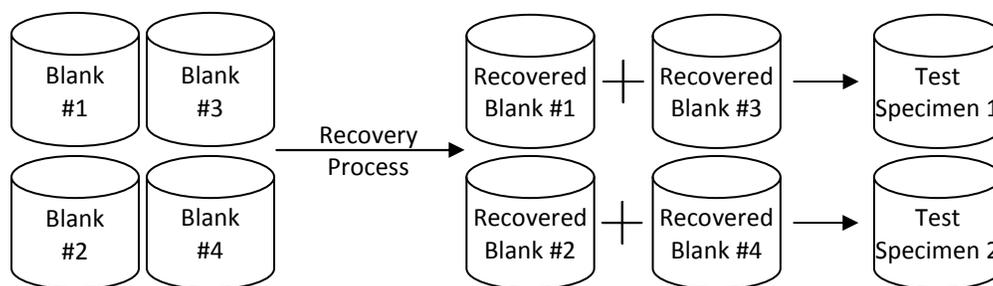


Figure 1. Single Laboratory Process for Obtaining Recovered Test Specimens From Blank Determination Specimens

Detection of Residual Solvent

Fourier transform infrared (FTIR) analysis was used to determine if any residual solvent was present in each test specimen after recovery. An Agilent 4100 Exoscan FTIR was used with an attenuated total reflectance interface to measure the infrared spectra for a specimen of solvent, which was compared with the spectra for each recovered binder specimen to verify that no residual solvent was present. Each spectrum encompassed a range of 4000 to 648 cm^{-1} wavenumbers and a resolution of 3.728 cm^{-1} .

FTIR analysis was conducted on the combined Abson-recovered test specimens in general, as most recovered blank specimens were combined in pairs after recovery when shipped to the VCTIR laboratory. FTIR analysis was conducted on each Rotavap-recovered blank specimen before combining into test specimens to determine if the recovery process was sufficient to remove all residual solvent. In addition, FTIR analysis was conducted on a sample of the nPB solvent and on each of the base binders used to prepare blank determinations. The resultant spectra were visually evaluated to determine the presence of residual solvent peaks in the recovered binders.

Binder Testing

All binder testing was performed in the VCTIR laboratory. All testing was performed by the same technician to eliminate multi-operator variability. Base binder specimens that were not exposed to solvent were tested to provide baseline information about each binder. These test results were compared with those for recovered binder specimens to assess the effect of solvent recovery on the binder properties.

During testing, because of the limited amount of recovered binder available for each specimen, rolling thin-film oven (RTFO) aging was not performed—all binders were tested in the unaged condition and then aged in the pressure aging vessel (PAV) to simulate long-term aging. This procedure follows that used for binders that are extracted and recovered from asphalt mixtures and was chosen because of the limited recovered material available for testing. The same procedure was followed for the base binder specimens that were not exposed to any solvents to provide a basis for comparison of properties. It should be noted that the base binder test data are not valid for PG determination, as the binder specimens were not exposed to short-term aging prior to long-term aging in the PAV.

The procedures described in AASHTO T 315, Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR) (AASHTO, 2012), were followed for all DSR testing. Creep stiffness and slope were evaluated in accordance with AASHTO T 313, Determining the Flexural Creep Stiffness of Asphalt Binder Using the Bending Beam Rheometer (BBR) (AASHTO, 2012). A summary of the testing performed is provided in Table 1.

Table 1. Binder Testing

Data Collected	Test Method
Unaged Binder	
$G^*/\sin \delta$, kPa G^* , kPa δ , ° Failure temperature, °C	AASHTO T 315 (AASHTO, 2012)
PAV-aged binder	
$G^* \cdot \sin \delta$, kPa G^* , kPa δ , ° Failure temperature, °C	AASHTO T 315 (AASHTO, 2012)
S , kPa m-value	AASHTO T 313 (AASHTO, 2012)

Evaluation Methods

Two comparisons were made to determine the impact of the recovery method on the binder properties.

1. *Comparisons of base binder properties and recovered binder properties.* This analysis was performed to determine if there were any differences in binder properties that may have occurred because of the dissolution in nPB and recovery of binder using the Abson or Rotavap method.
2. *Comparisons of binder properties after Abson and Rotavap recovery.* This analysis was performed to determine if use of the two methods resulted in binders having significantly different measured properties.

Analyses were performed using the paired *t*-test and, where applicable, evaluating the reproducibility of test results in accordance with AASHTO T 313 (AASHTO, 2012) and AASHTO T 315 (AASHTO, 2012). The paired *t*-test was used to evaluate the hypothesis that the difference in the mean value of test results was zero. Multi-laboratory precision or reproducibility for test results (denoted *d2s%*) was evaluated to determine if there were differences in the test results attributable to the different recovery methods. Acceptable ranges for the two test results are provided in AASHTO T 313 and AASHTO T 315 for different aging conditions. These ranges were developed using virgin binders that were split between laboratories and tested using the same procedures to determine the between-laboratory variability in test results.

In addition, the practical implications of test differences were assessed by considering if any changes in properties attributed to the recovery of the binders changed the resultant PG of the binder as determined in accordance with AASHTO M 320, Standard Specification for Performance Graded Asphalt Binder (AASHTO, 2012).

RESULTS AND DISCUSSION

Detection of Residual Solvent

Figures 2 through 7 present the collected spectra for each of the binder grade and supplier combinations. Each figure includes the reference spectrum for the nPB solvent used during extraction and a reference spectrum for the base binder used to prepare the blank determinations. It should be noted that in many cases the base binder sample was frozen when analyzed and shows a characteristic flattened peak at approximately $3,390\text{ cm}^{-1}$ that is a function of condensation on the specimen during testing. The infrared scan of the nPB solvent shows C-Cl stretching bands or peaks at 775 cm^{-1} , 838 cm^{-1} , and 1282 cm^{-1} . The scans also show a CH_2Br stretching band at 1225 cm^{-1} . These peaks were used to evaluate the presence of residual nPB solvent as they are absent in the base asphalt binder spectra.

Although the visual evaluation cannot conclusively determine that no residual solvent was present, as minimum sensitivity was not determined within the scope of this study, no solvent peaks were seen in the spectra of the recovered specimens.

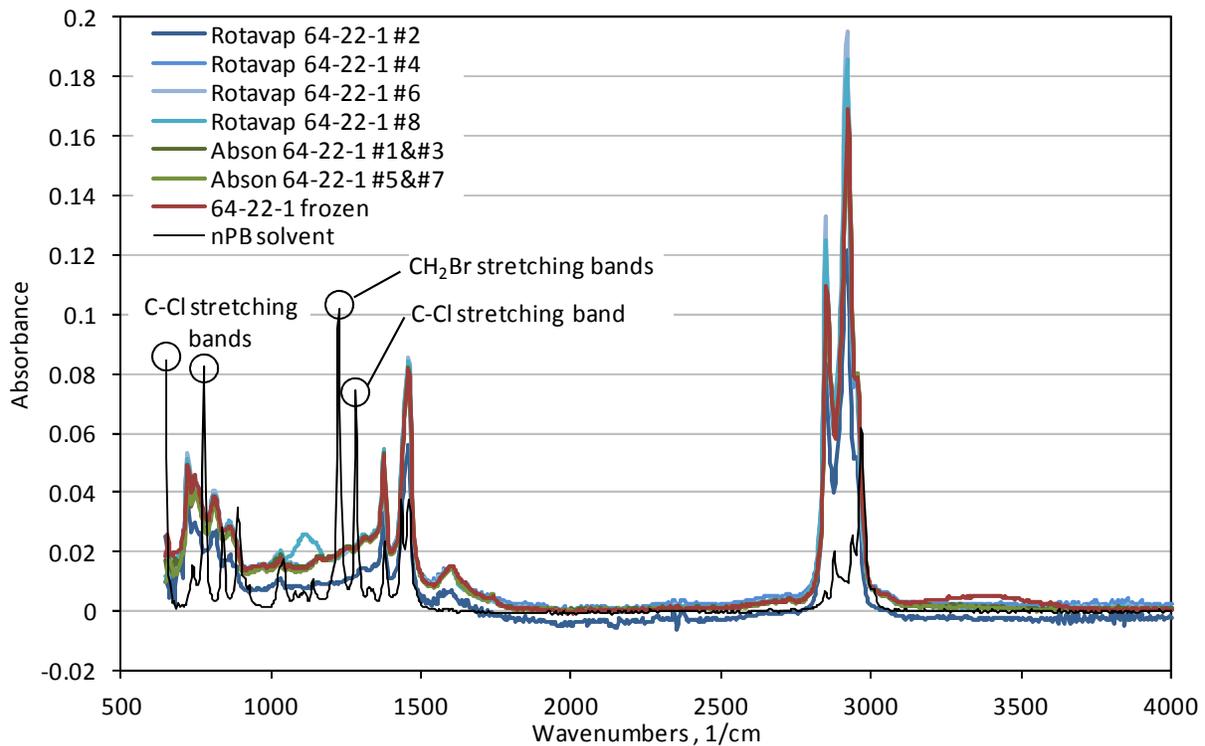


Figure 2. Spectra for PG 64-22, Supplier 1 Specimens

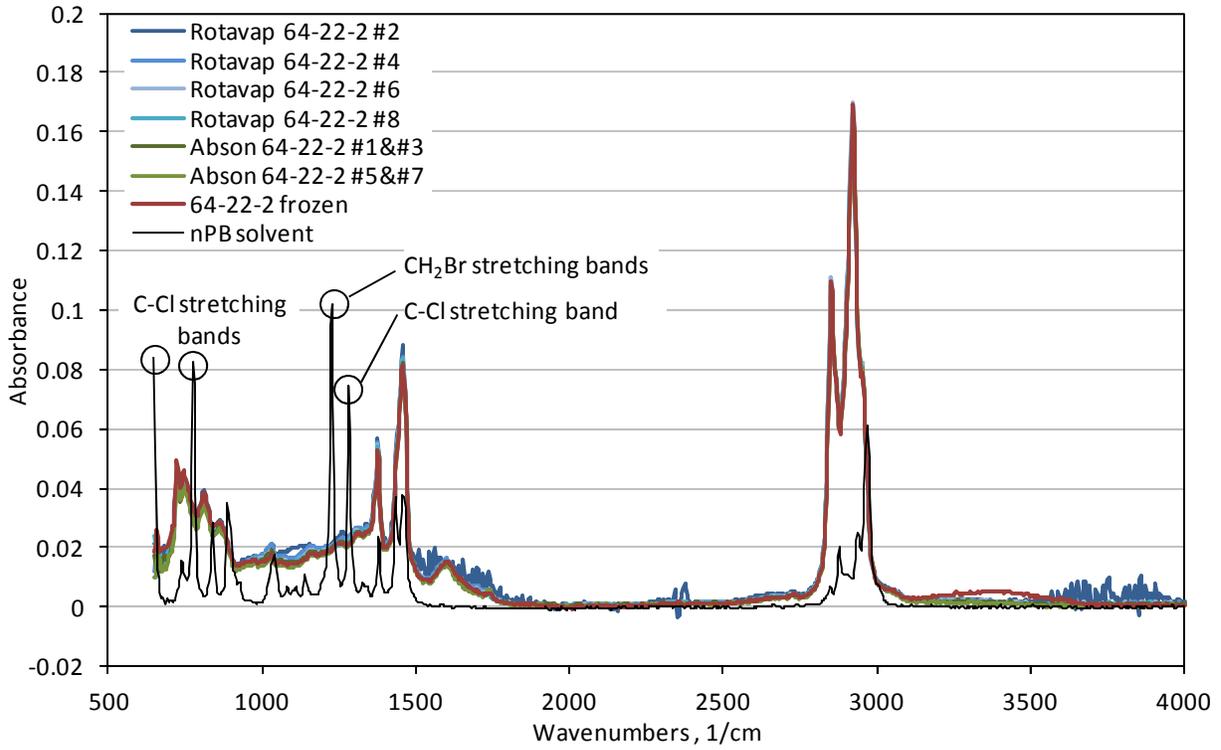


Figure 3. Spectra for PG 64-22, Supplier 2 Specimens

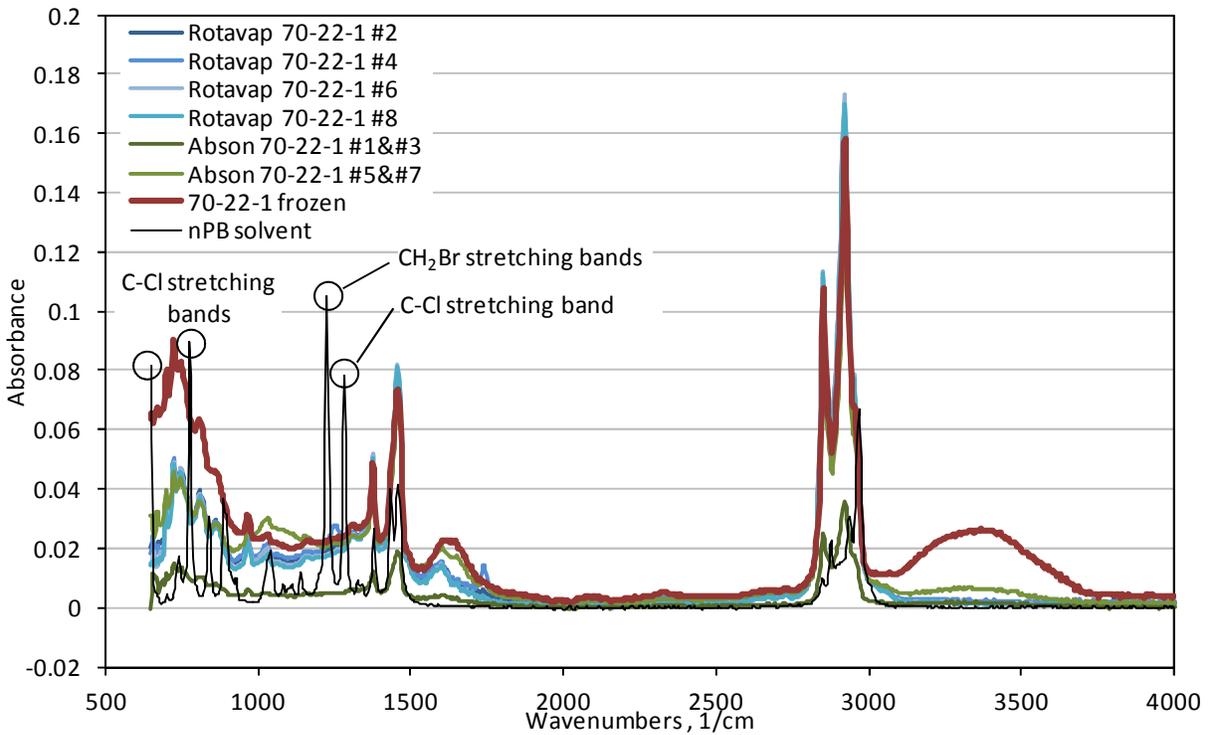


Figure 4. Spectra for PG 70-22, Supplier 1 Specimens

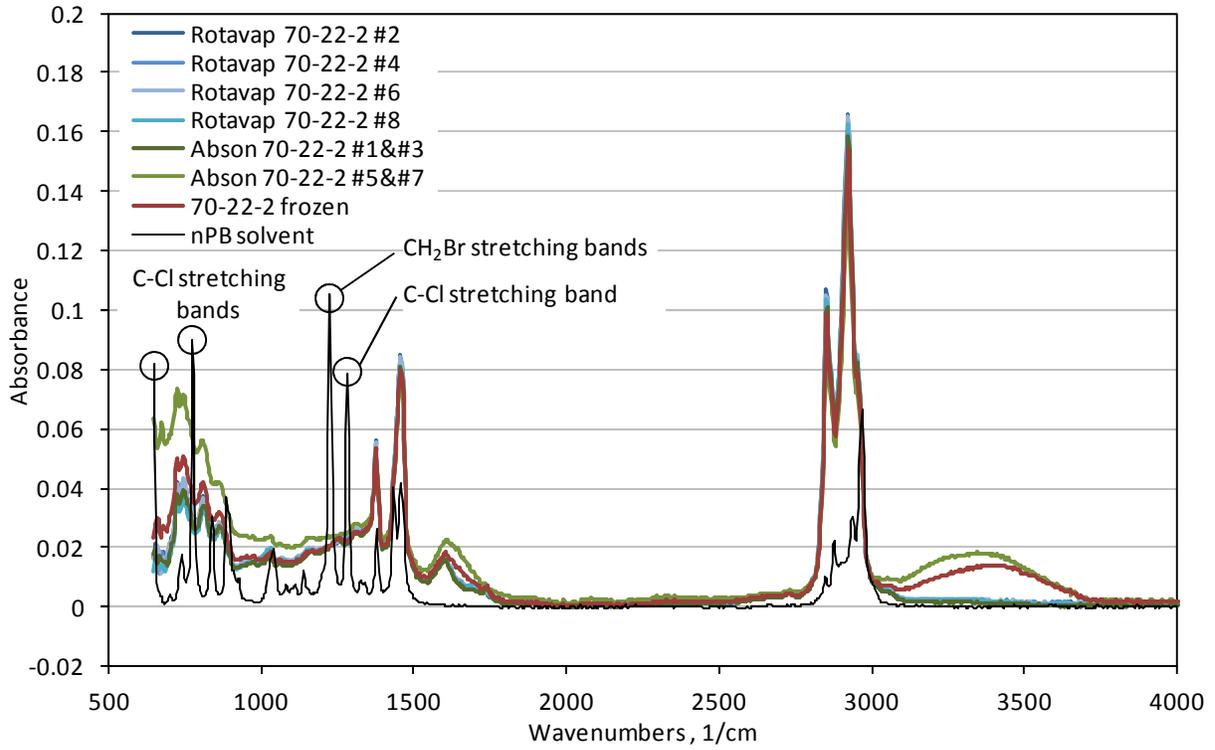


Figure 5. Spectra for PG 70-22, Supplier 2 Specimens

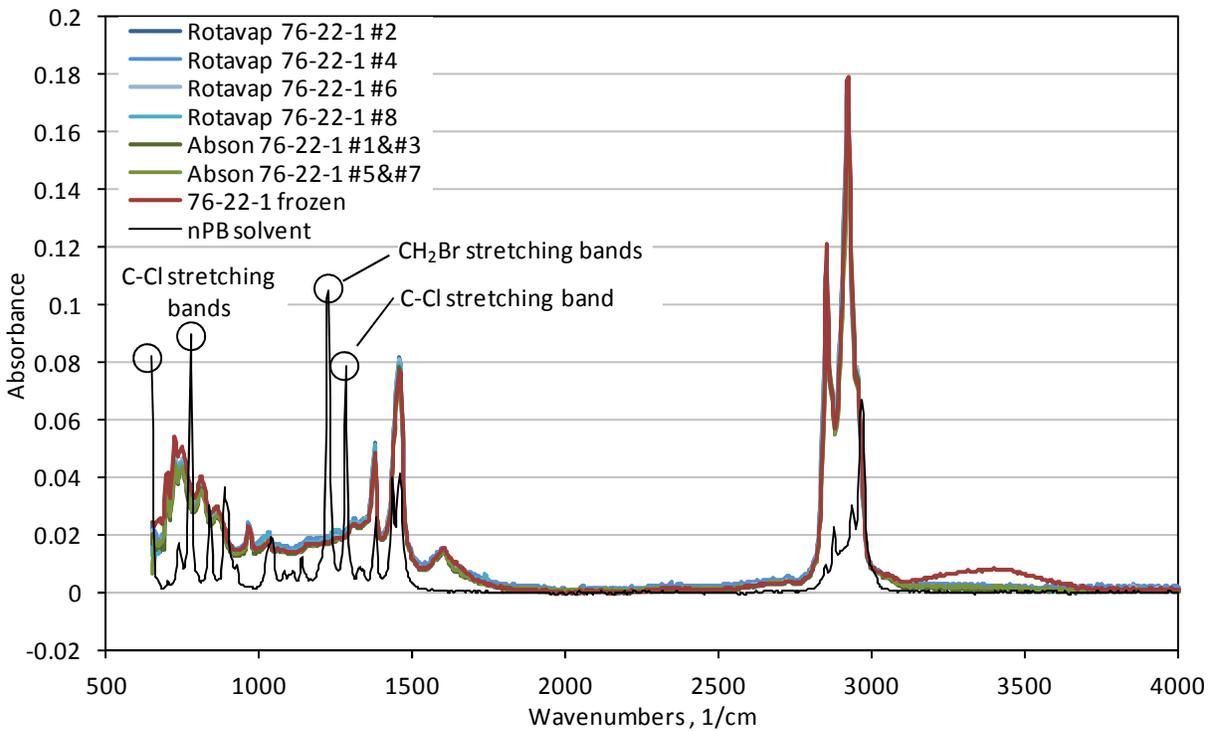


Figure 6. Spectra for PG 76-22, Supplier 1 Specimens

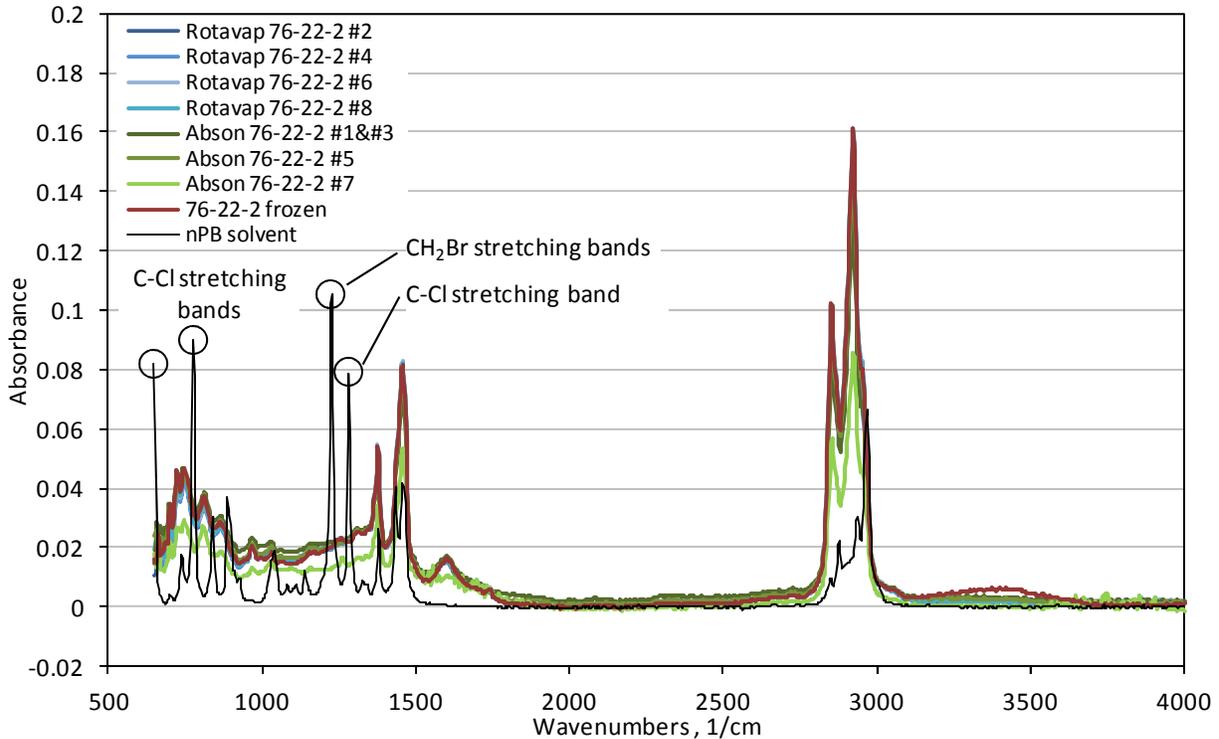


Figure 7. Spectra for PG 76-22, Supplier 2 Specimens

Comparison of Base and Recovered Binders

Comparisons were performed to determine if the properties of the recovered binder were different from those of the base binder attributable to the solvent dissolution and recovery process. These comparisons were made using the paired two-sample *t*-test and multi-laboratory precision and reproducibility evaluations. As noted previously, all binder specimens were treated in a manner such that only the method of recovery should distinguish the particular specimen sets. Details of all test results are presented in the Appendix.

Statistical Evaluation

The paired *t*-test was chosen for statistical evaluation of the data. The base binder properties were compared with the properties of the recovered binder after each recovery process. A level of significance of $\alpha = 0.05$ was used during analysis. Table 2 presents the *p*-values associated with each comparison. Unaged binder properties were evaluated in groups determined by their passing and failing properties, as determined in accordance with AASHTO M 320 (AASHTO, 2012). It can be seen that the unaged material test results appeared to be more sensitive to changes in the binder induced by recovery; the unaged results were consistent between methods. With regard to the PAV-aged material results, only the BBR test results indicated significant differences between the results for the base binder and the recovered binders; more instances of significantly different properties were found for the Abson than for the Rotavap method in the PAV-aged material test results.

Table 2. Paired *t*-Test Comparisons of Base Binder Properties and Rotavap- or Abson-Recovered Binder Properties

Test Data Evaluated	Rotavap		Abson	
	<i>t</i> -statistic	<i>p</i> -value (two-tail)	<i>t</i> -statistic	<i>p</i> -value (two-tail)
Unaged Material				
G*/sin δ, at passing grade temperature	-4.3565	0.0073	-3.7601	0.0132
G*/sin δ, at failing grade temperature	-4.6599	0.0055	-3.6823	0.0143
G*/sin δ, all test results	-5.2392	0.0003	-4.7896	0.0006
G*, at passing grade temperature	-4.0885	0.0095	-3.7439	0.0134
G*, at failing grade temperature	-0.9912	0.3671	-1.0143	0.3570
G*, all test results	-1.0187	0.3302	-1.0388	0.3212
δ, at passing grade temperature	11.0686	0.0001	12.6160	0.0001
δ, at failing grade temperature	7.2454	0.0008	8.2041	0.0004
δ, all test results	12.3745	0.0000	13.7760	2.78E-08
Failure temperature	-5.2688	0.0033	-4.2250	0.0083
PAV-Aged Material				
G*·sin δ, 19.0°C	-1.3263	0.2767	-1.0604	0.3668
G*·sin δ, 22.0°C	-0.5712	0.6078	-0.5041	0.6489
G*·sin δ, all test temperatures	-0.9838	0.3446	-0.6893	0.5038
G*, 19.0°C	-1.3189	0.2788	-0.9971	0.3922
G*, 22.0°C	-0.5584	0.6156	-0.4939	0.6553
G*, all test temperatures	-0.9548	0.3585	-0.5773	0.5744
δ, 19.0°C	0.7448	0.5104	0.2534	0.8163
δ, 22.0°C	0.5545	0.6179	0.5506	0.6203
δ, all test temperatures	1.3333	0.2072	0.6235	0.5446
Failure temperature	0.2925	0.7817	0.4239	0.6893
S, -12°C	2.5090	0.0539	2.3200	0.0681
S, -18°C	0.9786	0.3727	1.5840	0.1740
S, all test temperatures	2.5533	0.0240	3.3638	0.0051
m-value, -12°C	-0.8041	0.4578	-3.1755	0.0247
m-value, -18°C	-2.5579	0.0508	-2.9400	0.0323
m-value, all test temperatures	-2.2045	0.0461	-4.8648	0.0003

PAV = pressure aging vessel.

Values in bold text indicate *p*-values less than $\alpha = 0.05$ level of significance.

Multi-Laboratory Precision

The reproducibility of the test results, $d_{2s}\%$, was also used to assess the effects of the choice of recovery method. This method was used only for the test results obtained using AASHTO T 313 (AASHTO, 2012) and AASHTO T 315 (AASHTO, 2012), as these test methods contain precision statements. The results of the analysis are shown in Tables 3 through 5 for unaged G*/sin δ, PAV-aged G*·sin δ, and PAV-aged stiffness and m-values, respectively. The results indicated that the differences between the base binder and recovered binders were consistent between the Abson and Rotavap methods. In only 8 of 54 compared test result sets were the data not found consistently to either pass or fail with both recovery methods. All 8 sets were from the PAV-aged binder data shown in Tables 4 and 5. Of these 8 sets, 1 set was from the DSR data shown in Table 4 and 7 sets were from among the BBR stiffness and m-value test data shown in Table 5. The difference in base and recovered binder properties is not unexpected, as it is understood that the binder will undergo aging during the recovery process.

Table 3. Unaged G*/sin δ (kPa) Data Comparing Base Binder Versus Rotavap- and Abson-Recovered Binder

Binder Grade	Supplier	Test Temp.	Base	Average		d2s%		Max. d2s%	d2s% Within Allowable Range?	
				Rotavap	Abson	Rotavap	Abson		Rotavap	Abson
64-22	1	64°C	1.547	1.661	1.65	7.0	6.4	9.0	Pass	Pass
		70°C	0.7463	0.79565	0.79895	6.3	6.7	9.0	Pass	Pass
	2	64°C	1.613	2.035	1.989	22.3	20.2	9.0	Fail	Fail
		70°C	0.7676	0.9571	0.9452	21.2	20.0	9.0	Fail	Fail
70-22	1	70°C	1.251	1.471	1.41	15.7	11.7	9.0	Fail	Fail
		76°C	0.6145	0.71655	0.6951	15.0	12.1	9.0	Fail	Fail
	2	70°C	1.283	1.4245	1.4225	10.3	10.1	9.0	Fail	Fail
		76°C	0.7437	0.8236	0.82855	10.0	10.6	9.0	Fail	Fail
76-22	1	76°C	1.481	1.594	1.6	7.3	7.6	9.0	Pass	Pass
		82°C	0.7962	0.8535	0.86245	6.9	7.9	9.0	Pass	Pass
	2	76°C	1.425	1.683	1.8595	16.2	25.3	9.0	Fail	Fail
		82°C	0.8474	0.99655	1.1015	15.8	25.0	9.0	Fail	Fail

Temp. = temperature; Max. = maximum.

Values in bold text indicate d2s% values that exceeded the maximum threshold for reproducibility.

Table 4. PAV-aged G*/sin δ (kPa) Data Comparing Base Binder to Rotavap- and Abson-Recovered Binder

Binder Grade	Supplier	Test Temp.	Base	Average		d2s%		Max. d2s%	Pass?	
				Rotavap	Abson	Rotavap	Abson		Rotavap	Abson
64-22	1	19.0°C	5035	6498	6868.5	24.3	29.3	13.8	Fail	Fail
		22.0°C	3333	4375.5	4611	25.9	30.5	13.8	Fail	Fail
		25.0°C	2151	2864.5	3010.5	27.2	31.6	13.8	Fail	Fail
	2	19.0°C	5525	5463	5208.5	1.1	6.0	13.8	Pass	Pass
		22.0°C	3702	3750	3564.5	1.3	3.8	13.8	Pass	Pass
		25.0°C	2502	2517.5	2379	0.6	5.1	13.8	Pass	Pass
70-22	1	19.0°C	6053	7022	7068	14.5	15.1	13.8	Fail	Fail
		22.0°C	4061	4696	4722.5	14.2	14.7	13.8	Fail	Fail
	2	13.0°C	6391	5828	5598.5	9.4	13.5	13.8	Pass	Pass
		16.0°C	4365	3962	3808	9.8	13.9	13.8	Pass	Fail
76-22	1	19.0°C	5467	6909	6842.5	22.4	21.5	13.8	Fail	Fail
		22.0°C	3631	4659	4605.5	23.8	22.8	13.8	Fail	Fail
	2	16.0°C	7004	6660.5	6585	5.1	6.2	13.8	Pass	Pass
		19.0°C	4838	4619.5	4541	4.7	6.4	13.8	Pass	Pass

Temp. = temperature; Max. = maximum.

Values in bold text indicate d2s% values that exceeded the maximum threshold for reproducibility.

Table 5. PAV-aged Bending Beam Rheometer Data Comparing Base Binder to Rotavap- and Abson-Recovered Binder

Binder Grade	Supplier	Parameter	Base	Average		d2s%		Max. d2s%	Comparison	
				Rotavap	Abson	Rotavap	Abson		Rotavap	Abson
64-22	1	S, -12.0°C, MPa	140	118.5	114.5	17.1	20.7	7.2	Fail	Fail
		m-value, -12.0°C	0.362	0.372	0.373	2.6	3.0	2.9	Pass	Fail
		S, -18.0°C, MPa	271	280	263.5	3.2	2.8	7.2	Pass	Pass
		m-value, -18.0°C	0.312	0.316	0.312	1.3	0.0	2.9	Pass	Pass
	2	S, -24.0°C, MPa	564	534.5	524.5	5.4	7.3	7.2	Pass	Fail
		m-value, -24.0°C	0.238	0.2495	0.255	4.7	6.8	2.9	Fail	Fail
		S, -12.0°C, MPa	148	151.5	147	2.3	0.7	7.2	Pass	Pass
		m-value, -12.0°C	0.317	0.332	0.336	4.6	5.8	2.9	Fail	Fail
70-22	1	S, -18.0°C, MPa	320	327	334	2.2	4.3	7.2	Pass	Pass
		m-value, -18.0°C	0.275	0.277	0.283	0.5	2.7	2.9	Pass	Pass
		S, -12.0°C, MPa	211	198	199	6.4	5.9	7.2	Pass	Pass
		m-value, -12.0°C	0.33	0.342	0.349	3.6	5.5	2.9	Fail	Fail
	2	S, -18.0°C, MPa	492	432.5	460.5	13.2	6.7	7.2	Fail	Pass
		m-value, -18.0°C	0.275	0.275	0.285	0.0	3.4	2.9	Pass	Fail
		S, -12.0°C, MPa	76	68	67.5	11.3	12.1	7.2	Fail	Fail
		m-value, -12.0°C	0.378	0.362	0.379	4.5	0.1	2.9	Fail	Pass
76-22	1	S, -18.0°C, MPa	170	165.5	159.5	2.7	6.4	7.2	Pass	Pass
		m-value, -18.0°C	0.318	0.321	0.332	0.9	4.1	2.9	Pass	Fail
		S, -24.0°C, MPa	358	345	349.5	3.7	2.4	7.2	Pass	Pass
		m-value, -24.0°C	0.268	0.27	0.27	0.7	0.7	2.9	Pass	Pass
	2	S, -12.0°C, MPa	211	183	166	14.6	24.9	7.2	Fail	Fail
		m-value, -12.0°C	0.335	0.343	0.347	2.2	3.5	2.9	Pass	Fail
		S, -18.0°C, MPa	439	419	413.5	4.7	6.0	7.2	Pass	Pass
		m-value, -18.0°C	0.273	0.285	0.295	4.1	7.6	2.9	Fail	Fail
2	S, -12.0°C, MPa	119	115	116	3.4	2.6	7.2	Pass	Pass	
	m-value, -12.0°C	0.349	0.345	0.351	1.2	0.4	2.9	Pass	Pass	
	S, -18.0°C, MPa	256	261	253.5	1.9	1.0	7.2	Pass	Pass	
	m-value, -18.0°C	0.294	0.306	0.298	3.8	1.2	2.9	Fail	Pass	

PAV = pressure aging vessel; Max. = maximum; S = stiffness.

Values in bold text indicate d2s% values that exceeded the maximum threshold for reproducibility.

Practical Implications

Further investigation was performed to determine if the difference in measured values affected the failure temperature, and thus the PG, of the recovered binder. This analysis was performed to assess if there was an apparent risk of incorrect grade determination for binders attributable to the impact of the recovery method. It should be noted again that the evaluated binders were not subjected to RTFO aging because of material limitations and were treated as if they were recovered from asphalt mixtures, despite being blank determinations. Based on these assumptions, the failure temperatures of the base binder and recovered binders are shown in Table 6; the unaged failure temperature is indicative of the upper PG, and the PAV-aged failure temperature is the intermediate temperature. Despite the differences in test results according to statistical analysis and reproducibility as determined by $d_{2s}\%$, the failure temperatures did not vary enough to affect the PG of the recovered binders when compared to that of the base binder. This observation is important as a significant amount of quality assurance testing is performed on recovered binders, which are expected to maintain the PG required at purchase even when subjected to the varying conditions of extraction and recovery.

Table 6. Failure Temperature (°C) Data for Base Binder and Rotavap- and Abson-Recovered Binders

Binder	Supplier 1			Supplier 2			Impact on PG Determination?	
	Base Binder	Rotavap Average	Abson Average	Base Binder	Rotavap Average	Abson Average	Rotavap	Abson
Unaged PG 64-22	67.59	68.14	68.14	67.86	69.71	69.55	No	No
Unaged PG 70-22	71.89	73.22	72.91	72.74	73.88	73.80	No	No
Unaged PG 76-22	79.80	80.47	80.56	80.09	82.02	83.18	No	No
PAV-aged PG 64-22	19.08	17.95	18.34	19.74	19.71	19.35	No	No
PAV-aged PG 70-22	20.59	21.52	21.58	14.93	14.18	13.88	No	No
PAV-aged PG 76-22	22.66	21.45	21.38	18.68	18.40	18.22	No	No

PAV = pressure aging vessel.

Comparison Between Abson and Rotavap Methods

Although the previous section indicated that no difference in binder grade was to be expected as a result of the use of either recovery method, similar comparisons were performed to determine if the same was true with regard to binder properties.

These comparisons were made using the paired two-sample t -test and multi-laboratory precision and reproducibility evaluations. As noted previously, all binder specimens were treated in a manner such that only the method of recovery should distinguish the particular specimen sets.

Statistical Evaluation

As in the comparison of recovered binder and base binder properties, the paired t -test was used for statistical evaluation of the data. A level of significance of $\alpha = 0.05$ was used during analysis. Table 7 presents the p -values associated with each comparison. Unaged binder properties were evaluated in groups determined by their passing and failing grade temperatures in accordance with AASHTO M 320 (AASHTO, 2012) in addition to values across all tested

temperatures. PAV-aged binder properties were grouped by test temperature for evaluation to minimize any undue influence of temperature on results. Table 7 shows that the Rotavap and Abson methods had little impact on most test results. Significant differences were found only for phase angle measurements and for BBR m-value results. It should be noted that for performance grading purposes the phase angle is not specifically evaluated, although of course it directly influences the $G^*/\sin \delta$ and $G^* \cdot \sin \delta$ terms used to determine the PG. For the binders used in this study, the differences in phase angle did not appear to affect the determination of statistical difference for the $G^*/\sin \delta$ and $G^* \cdot \sin \delta$ values.

Table 7. Two-tailed p -value Results for Paired t -Test Comparisons of Rotavap- and Abson-Recovered Binder Properties

Test Data Evaluated	t -statistic	p -value (two-tail)
Unaged Material		
$G^*/\sin \delta$, at passing grade temperature	-0.2986	0.7772
$G^*/\sin \delta$, at failing grade temperature	-0.7942	0.4631
$G^*/\sin \delta$, all values	-0.6684	0.5177
G^* , at passing grade temperature	-0.1723	0.8700
G^* , at failing grade temperature	-1.7575	0.1392
G^* , all values	-1.6063	0.1365
δ , at passing grade temperature	5.4476	0.0028
δ , at failing grade temperature	5.9307	0.0019
δ , all values	8.0514	6.15E-06
Failure temperature	-0.2247	0.8311
PAV-aged Material		
$G^* \cdot \sin \delta$, 19°C	-0.0328	0.9754
$G^* \cdot \sin \delta$, 22°C	-0.0652	0.9521
$G^* \cdot \sin \delta$, all temperatures	0.6128	0.5506
G^* , 19°C	0.0669	0.9508
G^* , 22°C	-0.0277	0.9797
G^* , all values	0.9447	0.3635
δ , 19°C	-1.8820	0.1564
δ , 22°C	-0.5132	0.6432
δ , all values	-3.2369	0.0071
Failure temperature	0.6884	0.5218
S, -12°C	1.4399	0.2094
S, -18°C	0.0130	0.9901
S, all temperatures	0.7202	0.4842
m-value, -12°C	-2.9770	0.0309
m-value, -18°C	-1.2374	0.2709
m-value, all temperatures	-2.9405	0.0115

PAV = pressure aging vessel; S = stiffness.

Values in bold text indicate p -values less than $\alpha = 0.05$ level of significance.

Multi-Laboratory Precision

Reproducibility was used to evaluate the Rotavap and Abson methods, as the only contributing factor to their difference was the recovery method. A single operator performed all binder testing. This method was used only for the test results obtained using AASHTO T 313 (AASHTO, 2012) and AASHTO T 315 (AASHTO, 2012), as these test methods contain precision statements.

Tables 8 through 10 summarize the analysis results for unaged $G^*/\sin \delta$, PAV-aged $G^*\cdot\sin \delta$, and PAV-aged stiffness and m-values, respectively. Of the 54 comparisons made, significant differences in test results from binders recovered by the two methods were found in only seven cases. Interestingly, these differences were not consistently found for any single binder grade or supplier. In addition, these seven cases did not correlate with the cases shown in Tables 3 through 5 wherein one method compared well with the base binder result and the other did not. Such overlap was observed in only three of the seven instances of significant difference.

Practical Implications

Similar to the comparison of base binder and recovered binders, the data were evaluated to determine if the difference in measured values affected the failure temperature, and thus the PG, of the recovered binder to determine if there was an apparent risk of incorrect grade determination attributable to the effect of the recovery method. As a reminder, the evaluated binders were not subjected to RTFO aging because of material limitations and were treated as if they were recovered from asphalt mixtures, despite being blank determinations. Based on these assumptions, the failure temperatures of the recovered binders are shown in Table 11; again, the unaged failure temperature is indicative of the PG, and the PAV-aged failure temperature indicates the intermediate temperature. These results indicated that the failure temperatures did not vary enough between recovery methods to affect the PG of the recovered binders.

In summary, although there were differences found in the data that were attributable to the differences in the Abson and Rotavap methods, in this study the differences were not found to be systematic to a particular recovery method or to have a significant impact on performance grading.

SUMMARY OF FINDINGS

- *Use of the Rotavap and Abson methods significantly changed specific measured binder properties based on a comparison of recovered binder blanks and unrecovered base binder.* Typically, the recovered binder properties were slightly stiffer than the base binder properties. However, the differences did not result in changes in the PG of the recovered binder as compared to the base binder. These differences may be attributable to solvent interaction with the binder and aging during the recovery process, although these were not specifically investigated during this study.
- *Use of the Rotavap and Abson methods resulted in recovered binders having similar measured properties.* Although some significant differences were found, they were not systematic to either method and did not affect the PG of the recovered binder.
- *FTIR analysis was a reasonable method for detection of residual polymer in recovered binders.* The FTIR scans indicated that the binder recovered by the Rotavap and Abson methods matched the unprocessed binder in chemical makeup. The IR scans of the recovered binder did not indicate any of the CH_2Br or C-Cl stretching bands of the nPB solvent. However, the nPB solvent may be present at such a low concentrations that the binder peaks overtake them on the IR scan.

Table 8. Unaged $G^*/\sin \delta$ (kPa) Data Comparing Rotavap and Abson Methods

Binder Grade	Supplier	Test Temp.	Rotavap			Abson			d2s%	Max. d2s%	Result
			Rep. 1	Rep. 2	Average	Rep. 1	Rep. 2	Average			
64-22	1	64°C	1.651	1.671	1.661	1.689	1.611	1.650	0.66	6.4	Equal
		70°C	0.791	0.800	0.796	0.817	0.781	0.799	0.41	6.4	Equal
	2	64°C	1.922	2.148	2.035	2.050	1.928	1.989	2.29	6.4	Equal
		70°C	0.908	1.006	0.957	0.970	0.920	0.945	1.25	6.4	Equal
70-22	1	70°C	1.443	1.499	1.471	1.384	1.436	1.410	4.23	6.4	Equal
		76°C	0.703	0.730	0.717	0.690	0.700	0.695	3.04	6.4	Equal
	2	70°C	1.406	1.443	1.425	1.227	1.618	1.423	0.14	6.4	Equal
		76°C	0.808	0.839	0.824	0.717	0.940	0.829	0.60	6.4	Equal
76-22	1	76°C	1.530	1.658	1.594	1.638	1.562	1.600	0.38	6.4	Equal
		82°C	0.819	0.888	0.854	0.882	0.843	0.862	1.04	6.4	Equal
	2	76°C	1.641	1.725	1.683	1.770	1.949	1.860	9.96	6.4	Not Equal
		82°C	0.972	1.021	0.997	1.052	1.151	1.102	10.00	6.4	Not Equal

Temp. = temperature; Rep. = replicate; Max. = maximum.

Values in bold text indicate values exceeding the maximum d2s%.

Table 9. PAV-aged $G^*\sin \delta$ (kPa) Data Comparing Rotavap and Abson Methods

Binder Grade	Supplier	Test Temp.	Rotavap			Abson			d2s%	Max. d2s%	Result
			Rep. 1	Rep. 2	Average	Rep. 1	Rep. 2	Average			
64-22	1	19.0°C	6538	6458	6498	6711	7026	6868.5	5.54	13.8	Equal
		22.0°C	4375	4376	4375.5	4484	4738	4611	5.24	13.8	Equal
		25.0°C	2858	2871	2864.5	2918	3103	3010.5	4.97	13.8	Equal
	2	19.0°C	5645	5281	5463	5308	5109	5208.5	4.77	13.8	Equal
		22.0°C	3871	3629	3750	3638	3491	3564.5	5.07	13.8	Equal
		25.0°C	2597	2438	2517.5	2436	2322	2379	5.66	13.8	Equal
70-22	1	19.0°C	7025	7019	7022	7194	6942	7068	0.65	13.8	Equal
		22.0°C	4711	4681	4696	4813	4632	4722.5	0.56	13.8	Equal
	2	13.0°C	6066	5590	5828	5696	5501	5598.5	4.02	13.8	Equal
		16.0°C	4130	3794	3962	3868	3748	3808	3.96	13.8	Equal
76-22	1	19.0°C	6958	6860	6909	6870	6815	6842.5	0.97	13.8	Equal
		22.0°C	4703	4615	4659	4596	4615	4605.5	1.15	13.8	Equal
	2	16.0°C	6918	6403	6660.5	6402	6768	6585	1.14	13.8	Equal
		19.0°C	4788	4451	4619.5	4418	4664	4541	1.71	13.8	Equal

Temp. = temperature; Rep. = replicate; Max. = maximum.

Values in bold text indicate values exceeding the maximum d2s%.

Table 10. PAV-aged Bending Beam Rheometer Data Comparing Rotavap and Abson Methods

Binder Grade	Supplier	Parameter	Rotavap			Abson			d2s%	Max. d2s%	Result	
			Rep. 1	Rep. 2	Average	Rep. 1	Rep. 2	Average				
64-22	1	S, -12.0°C, MPa	113	124	118.5	118	111	114.5	3.43	7.2	Equal	
		m-value, -12.0°C	0.379	0.364	0.3715	0.378	0.368	0.373	0.40	2.9	Equal	
	S, -18.0°C, MPa	267	293	280	256	271	263.5	6.07	7.2	Equal		
	m-value, -18.0°C	0.322	0.31	0.316	0.312	0.312	0.312	1.27	2.9	Equal		
	S, -24.0°C, MPa	503	566	534.5	525	524	524.5	1.89	7.2	Equal		
	m-value, -24.0°C	0.259	0.24	0.2495	0.259	0.251	0.255	2.18	2.9	Equal		
	2	S, -12.0°C, MPa	151	152	151.5	150	144	147	3.02	7.2	Equal	
		m-value, -12.0°C	0.331	0.333	0.332	0.337	0.335	0.336	1.20	2.9	Equal	
S, -18.0°C, MPa		327	327	327	339	329	334	2.12	7.2	Equal		
m-value, -18.0°C		0.279	0.274	0.2765	0.284	0.281	0.2825	2.15	2.9	Equal		
70-22	1	S, -12.0°C, MPa	199	197	198	198	200	199	0.50	7.2	Equal	
		m-value, -12.0°C	0.339	0.345	0.342	0.346	0.352	0.349	2.03	2.9	Equal	
		S, -18.0°C, MPa	417	448	432.5	451	470	460.5	6.27	7.2	Equal	
		m-value, -18.0°C	0.264	0.286	0.275	0.284	0.285	0.2845	3.40	2.9	Not Equal	
	2	S, -12.0°C, MPa	69	67	68	67	68	67.5	0.74	7.2	Equal	
		m-value, -12.0°C	0.356	0.367	0.3615	0.389	0.368	0.3785	4.59	2.9	Not Equal	
		S, -18.0°C, MPa	170	161	165.5	160	159	159.5	3.69	7.2	Equal	
		m-value, -18.0°C	0.317	0.325	0.321	0.335	0.328	0.3315	3.22	2.9	Not Equal	
	S, -24.0°C, MPa	334	356	345	352	347	349.5	1.30	7.2	Equal		
	m-value, -24.0°C	0.261	0.279	0.27	0.274	0.266	0.27	0.00	2.9	Equal		
	76-22	1	S, -12.0°C, MPa	181	185	183	168	164	166	9.74	7.2	Not Equal
			m-value, -12.0°C	0.346	0.339	0.3425	0.343	0.351	0.347	1.31	2.9	Equal
S, -18.0°C, MPa			417	421	419	415	412	413.5	1.32	7.2	Equal	
m-value, -18.0°C			0.283	0.286	0.2845	0.289	0.301	0.295	3.62	2.9	Not Equal	
2		S, -12.0°C, MPa	114	116	115	117	115	116	0.87	7.2	Equal	
		m-value, -12.0°C	0.342	0.348	0.345	0.349	0.352	0.3505	1.58	2.9	Equal	
		S, -18.0°C, MPa	267	255	261	249	258	253.5	2.92	7.2	Equal	
		m-value, -18.0°C	0.307	0.304	0.3055	0.29	0.305	0.2975	2.65	2.9	Equal	

PAV = pressure aging vessel; Rep. = replicate; Max. = maximum; S = stiffness.

Values in bold text indicate values exceeding the maximum d2s%.

Table 11. Failure Temperature (°C) for Rotavap- and Abson-Recovered Binders

Binder	Supplier 1		Supplier 2		Difference in PG Determination?
	Rotavap Average	Abson Average	Rotavap Average	Abson Average	
Unaged PG 64-22	68.14	68.14	69.71	69.55	No
Unaged PG 70-22	73.22	72.91	73.88	73.80	No
Unaged PG 76-22	80.47	80.56	82.02	83.18	No
PAV-aged PG 64-22	17.95	18.34	19.71	19.35	No
PAV-aged PG 70-22	21.52	21.58	14.18	13.88	No
PAV-aged PG 76-22	21.45	21.38	18.40	18.22	No

PAV = pressure aging vessel.

CONCLUSIONS

- *The Rotavap method as performed in the VCTIR laboratory provides recovered binders that are comparable to those recovered using the Abson method as performed in the VDOT Materials Division laboratory. When used to recover binder blank specimens, use of these methods resulted in recovered binder materials with differences in measured properties from those of the base binder material; however, these differences did not affect the resultant PG determined for the recovered binders.*
- *FTIR analysis is a useful means to investigate the chemical makeup of binders and to evaluate the presence of residual nPB solvent in recovered binder.*

RECOMMENDATIONS

1. *VCTIR should continue to use the Rotavap method to recover binders. This method was found comparable to the Abson method as used by VDOT's Materials Division and is suitable for both research and quality assurance evaluation as necessary.*
2. *VCTIR should become accredited by the AASHTO Materials Reference Laboratory in the use of the Rotavap method. Accreditation is necessary to demonstrate and maintain competence in testing; if it is necessary to perform quality assurance or acceptance testing at VCTIR, this accreditation is required.*
3. *VCTIR should implement routine use of the FTIR to verify the complete removal of solvent as part of the Rotavap binder recovery procedure. Although this study indicated that residual solvent was not present in the samples evaluated, continued verification should be performed and a sensitivity analysis should be conducted to determine the minimum detectable amount of solvent present in samples.*

BENEFITS AND IMPLEMENTATION PROSPECTS

VCTIR has invested in equipment to perform asphalt binder recovery that is widely used in the asphalt industry and that will provide optimum results for research testing. The development and validation of a standard methodology to perform binder recovery have resulted in the production of reliable recovery results for research that are also compatible with those results obtained by the VDOT Materials Division laboratory during production testing. This capability allows for the development and enhancement of additional research projects that will no longer require the assistance of the VDOT Materials Division laboratory for extraction and recovery services, thus reducing research-related demands on the laboratory. In addition, upon accreditation, VCTIR will be qualified to serve as a reserve testing facility for the VDOT Materials Division for the extraction and recovery of binders.

ACKNOWLEDGMENTS

The author thanks Todd Rorrer and Angela Cable of VDOT's Materials Division and Troy Deeds, Donnie Dodds, and Kim Snead of VCTIR for their outstanding efforts in sample preparation, recovery, and testing. Thomas Rockhold and Jessica Lewis of VDOT's Materials Division assisted in FTIR spectra interpretation, and their help is greatly appreciated. The assistance of Jim Gillespie of VCTIR in reviewing the final report is also appreciated. Appreciation is also extended to Linda Evans of VCTIR for her editorial assistance. The author is also appreciative to the project review panel, Bill Bailey, Todd Rorrer, and Tommy Schinkel, of VDOT; Ben Bowers, of VCTIR; and Alex Appeageyi, formerly of VCTIR, for their assistance.

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APPENDIX
BINDER TEST RESULTS

Table A1. Binder Test Results for PG 64-22 Specimens

Binder	Supplier 1					Supplier 2				
	Base Binder	Rotavap		Abson		Base Binder	Rotavap		Abson	
		Rep. 1	Rep. 2	Rep. 1	Rep. 2		Rep. 1	Rep. 2	Rep. 1	Rep. 2
Unaged binder										
G*/sin δ, 64°C, kPa	1.547	1.651	1.671	1.689	1.611	1.613	1.922	2.148	2.05	1.928
G*/sin δ, 70°C, kPa	0.7463	0.7912	0.8001	0.8166	0.7813	0.7676	0.9082	1.006	0.9702	0.9202
G*, 64°C, kPa	1.543	1.645	1.666	1.681	1.604	1.611	1.917	2.141	2.044	1.923
G*, 70°C, kPa	745.4	789.9	798.8	815	779.9	767	907.1	1.005	968.8	919
δ, 64°C, °	85.9	85.27	85.24	84.78	84.99	86.6	85.83	85.62	85.41	85.68
δ, 70°C, °	87.26	86.77	86.76	86.36	86.53	87.77	87.23	87.05	86.9	87.1
Failure temperature, °C	67.59	68.09	68.18	68.33	67.95	67.86	69.23	70.19	69.76	69.33
PAV-aged Binder										
G*·sin δ, 19.0°C, kPa	5035	6538	6458	6711	7026	5525	5645	5281	5308	5109
G*·sin δ, 22.0°C, kPa	3333	4375	4376	4484	4738	3702	3871	3629	3638	3491
G*·sin δ, 25.0°C, kPa	2151	2858	2871	2918	3103	2502	2597	2438	2436	2322
G*, 19.0°C, MPa	6.907	9.289	9.230	9.448	10.060	8.031	8.251	7.7030	7.735	7.431
G*, 22.0°C, MPa	4.356	5.909	5.938	6.004	6.444	5.076	5.359	5.020	5.031	4.816
G*, 25.0°C, MPa	2.693	3.691	3.718	3.737	4.030	3.281	3.425	3.213	3.209	3.049
δ, 19.0°C, °	46.8	44.74	44.4	45.26	44.31	43.47	43.17	43.28	43.34	43.43
δ, 22.0°C, °	49.92	47.77	47.47	48.32	47.33	46.83	46.25	46.29	46.31	46.46
δ, 25.0°C, °	53.01	50.73	50.56	51.35	50.35	49.86	49.31	49.36	49.38	49.59
Failure temperature, °C	19.08	17.97	17.93	18.15	18.53	19.74	19.97	19.45	19.49	19.2
S, -12°C, MPa	140	113	124	118	111	148	151	152	150	144
m-value, -12°C	0.362	0.379	0.364	0.378	0.368	0.317	0.331	0.333	0.337	0.335
S, -18°C, MPa	271	267	293	256	271	320	327	327	339	329
m-value, -18°C	0.312	0.322	0.31	0.312	0.312	0.275	0.279	0.274	0.284	0.281
S, -24°C, MPa	564	503	566	525	524					
m-value, -24°C	0.238	0.259	0.24	0.259	0.251					

Rep. = replicate; PAV = pressure aging vessel; S = stiffness.

Table A2. Binder Test Results for PG 70-22 Specimens

Binder	Supplier 1					Supplier 2				
	Base	Rotavap		Abson		Base	Rotavap		Abson	
	Binder	Rep. 1	Rep. 2	Rep. 1	Rep. 2	Binder	Rep. 1	Rep. 2	Rep. 1	Rep. 2
Unaged binder										
G*/sin δ , 70°C, kPa	1.251	1.443	1.499	1.384	1.436	1.283	1.406	1.443	1.227	1.618
G*/sin δ , 76°C, kPa	0.6145	0.7031	0.73	0.6903	0.6999	0.7437	0.8082	0.839	0.7167	0.9404
G*, 70°C, kPa	1.248	1.438	1.494	1.378	1.43	1.197	1.303	1.335	1.137	1.49
G*, 76°C, kPa	0.6139	0.7020	0.7288	0.6891	0.6986	0.7017	0.7583	0.7856	0.6734	0.8771
δ , 70°C, °	86.21	85.30	85.13	85.08	84.95	68.89	67.91	67.65	68.00	67.07
δ , 76°C, °	87.51	86.81	86.66	86.56	86.5	70.65	69.75	69.45	70.00	68.86
Failure temperature, °C	71.89	73.06	73.38	72.80	73.02	72.74	73.69	74.06	72.28	75.32
PAV-aged binder										
G*·sin δ , 19.0°C, kPa	6053	7025	7019	7194	6942	6391	6066	5590	5696	5501
G*·sin δ , 22.0°C, kPa	4061	4711	4681	4813	4632	4365	4130	3794	3868	3748
G*, 19.0°C, MPa	8.500	9.783	9.698	9.978	9.609	9.522	9.018	8.272	8.389	8.124
G*, 22.0°C, MPa	5.415	6.243	6.155	6.360	6.108	6.201	5.834	5.338	5.422	5.270
δ , 19.0°C, °	45.41	45.90	46.37	46.13	46.26	41.99	42.27	42.52	42.76	42.61
δ , 22.0°C, °	48.59	49.00	49.51	49.18	49.32	44.74	45.06	45.30	45.51	45.34
Failure temperature, °C	20.44	21.52	21.51	21.72	21.43	14.93	14.50	13.86	14.01	13.75
S, -12°C, MPa	211	199	197	198	200	76	69	67	67	68
m-value, -12°C	0.33	0.339	0.345	0.346	0.352	0.378	0.356	0.367	0.389	0.368
S, -18°C, MPa	492	417	448	451	470	170	170	161	160	159
m-value, -18°C	0.275	0.264	0.286	0.284	0.285	0.318	0.317	0.325	0.335	0.328
S, -24°C, MPa,						358	334	356	352	347
m-value, -24°C						0.268	0.261	0.279	0.274	0.266

Rep. = replicate; PAV = pressure aging vessel; S = stiffness.

Table A3. Binder Test Results for PG 76-22 Specimens

Binder	Supplier 1					Supplier 2				
	Base	Rotavap		Abson		Base	Rotavap		Abson	
	Binder	Rep. 1	Rep. 2	Rep. 1	Rep. 2	binder	Rep. 1	Rep. 2	Rep. 1	Rep. 2
Unaged binder										
G*/sin δ, 76°C, kPa	1.481	1.53	1.658	1.638	1.562	1.425	1.641	1.725	1.77	1.949
G*/sin δ, 82°C, kPa	0.7962	0.8189	0.8881	0.8816	0.8433	0.8474	0.9721	1.021	1.052	1.151
G*, 76°C, kPa	1.443	1.485	1.608	1.585	1.512	1.318	1.506	1.576	1.618	1.778
G*, 82°C, kPa	782.1	0.803	869.8	863.1	825.4	0.7917	0.9023	0.9466	0.9729	1.062
δ, 76°C, °	76.88	76.1	75.82	75.34	75.45	67.59	66.58	66.05	66.05	65.82
δ, 82°C, °	79.22	78.7	78.37	78.24	78.17	69.1	68.16	67.6	67.6	67.32
Failure temperature, °C	79.8	80.08	80.86	80.78	80.34	80.09	81.68	82.35	82.65	83.7
PAV-aged binder										
G*·sin δ, 19.0°C, kPa	-	6958	6860	6870	6815	7004	6918	6403	6402	6768
G*·sin δ, 22.0°C, kPa	5467	4703	4615	4596	4615	4838	4788	4451	4418	4664
G*, 19.0°C, MPa	-	9.885	9.730	4.704	4.756	10.570	10.410	9.720	9.540	10.100
G*, 22.0°C, MPa	7.472	6.370	6.234	6.190	6.301	6.929	6.833	6.433	6.250	6.606
δ, 19.0°C, °	-	44.74	44.84	45.07	44.31	41.51	41.67	41.2	42.15	42.08
δ, 22.0°C, °	47.03	47.59	47.76	47.95	47.09	44.29	44.49	43.86	44.98	44.92
Failure temperature, °C	22.66	21.5	21.39	21.37	21.38	18.68	18.75	18.05	18	18.44
S, -12°C, MPa	211	181	185	168	164	119	114	116	117	115
m-value, -12°C	0.335	0.346	0.339	0.343	0.351	0.349	0.342	0.348	0.349	0.352
S, -18°C, MPa	439	417	421	415	412	256	267	255	249	258
m-value, -18°C	0.273	0.283	0.286	0.289	0.301	0.294	0.307	0.304	0.29	0.305

Rep. = replicate; PAV = pressure aging vessel; S = stiffness.