APPENDIX D. SAFT OPTIMIZATION STUDY REPORT

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1.0 OBJECTIVE AND SCOPE

The objective of the SAFT Optimization Study was to optimize the operating parameters for the commercial SAFT so that it more nearly reproduces the degree of aging that occurs in the RTFOT for neat binders. Originally it was envisioned that only minor adjustments to the prototype SAFT operating parameters would be needed; however, initial testing with the commercial SAFT showed significantly less aging than the RTFOT when operated using the prototype SAFT operating parameters. Because of the difference in aging between the commercial SAFT and prototype SAFT a more comprehensive optimization study than originally planned was undertaken.

The SAFT Optimization Study included two components. The first component was a series of tests to verify that the heat-up phase of the test does not result in significant aging of the binder. During the heat-up phase, the temperature of the binder is increased from approximately 100 °C to the testing temperature of 163 °C while nitrogen flows through the SAFT vessel. Since the starting temperature (temperature of the binder after charging the SAFT vessel) can not be accurately controlled, it is critical that the heat-up phase not contribute significantly to the degree of aging that occurs in the test. The second component of the SAFT Optimization Study was an experiment to determine the effects of impeller speed, air-flow rate, and test duration on the degree of aging measured by the high pavement temperature rheology of the binder. From this component of the SAFT Optimization Study, operating parameters for the commercial SAFT were selected. The sections that follow describe the two components of the SAFT Optimization Study in detail.

2.0 HEAT-UP EFFECTS EXPERIMENT

The first experiment in the SAFT Optimization Study was an experiment to evaluate the degree of aging that occurs during the heat-up phase of the procedure. In this study, three binders were used: Citgo PG 58-28, Citgoflex PG 82-22, and Novophalt PG 76-22. These are the same binders that were used in the Selection Study. In this experiment the binders were heated until they were sufficiently fluid to pour, the preheated SAFT vessel was charged with

250 g of binder, the vessel was assembled and inserted into the SAFT oven which was preheated to 176 °C.

Immediately after the vessel was closed the flow of nitrogen was started and the binder was allowed to reach 160°C with the oven temperature held constant at 176°C. This time period is referred to as the heat-up phase. During the heat-up phase the impeller stirs the binder at 700 rpm and nitrogen is bubbled through the binder at a flow rate of 2,000 ml/min. The temperature of the binder at the beginning of the heat-up phase is not controlled but is determined by the temperature of the binder when it is poured into the vessel and the time it takes to assemble the vessel and load it in the oven. Once the binder reaches a temperature of 160 °C the flow of nitrogen is stopped and the flow of air is started. At this point the process controller controls the oven temperature to bring the temperature of the binder to 163°C and to maintain it at 163°C throughout the conditioning period.

In order to assess the change in the properties of the asphalt binder during the heat-up period DSR measurements at 10 rad/s were made at the respective upper specification temperatures for each binder on material removed from the SAFT after completion of the heat-up phase. Replicate tests were run for each of the three binders. Initial testing showed that the SAFT trapped air bubbles in the PG 82-22; therefore, for this experiment, both the tank and the materials sampled at the end of the heat up-up period were exposed to the PAV vacuum degassing procedure to remove the trapped air before they were tested in the DSR. The procedure given in the PAV test method, AASHTO R28, was used for degassing.

Figure 1 is a plot of the binder temperature as a function of time during the heat-up phase for the three binders. As shown, the heat-up curves are similar for the three binders, particularly as the binder approaches 160 °C, the temperature where the process controller controls the remainder of the test. The DSR data from this experiment are summarized in Table 1. For a given binder all of the DSR data are within the AASHTO T315 single operator precision of 9.5 percent for testing of original binder. Because the complex modulus before and after the heat-up vary by less than the single operator precision it was concluded that the binder does not significantly stiffen during the SAFT heat-up phase.

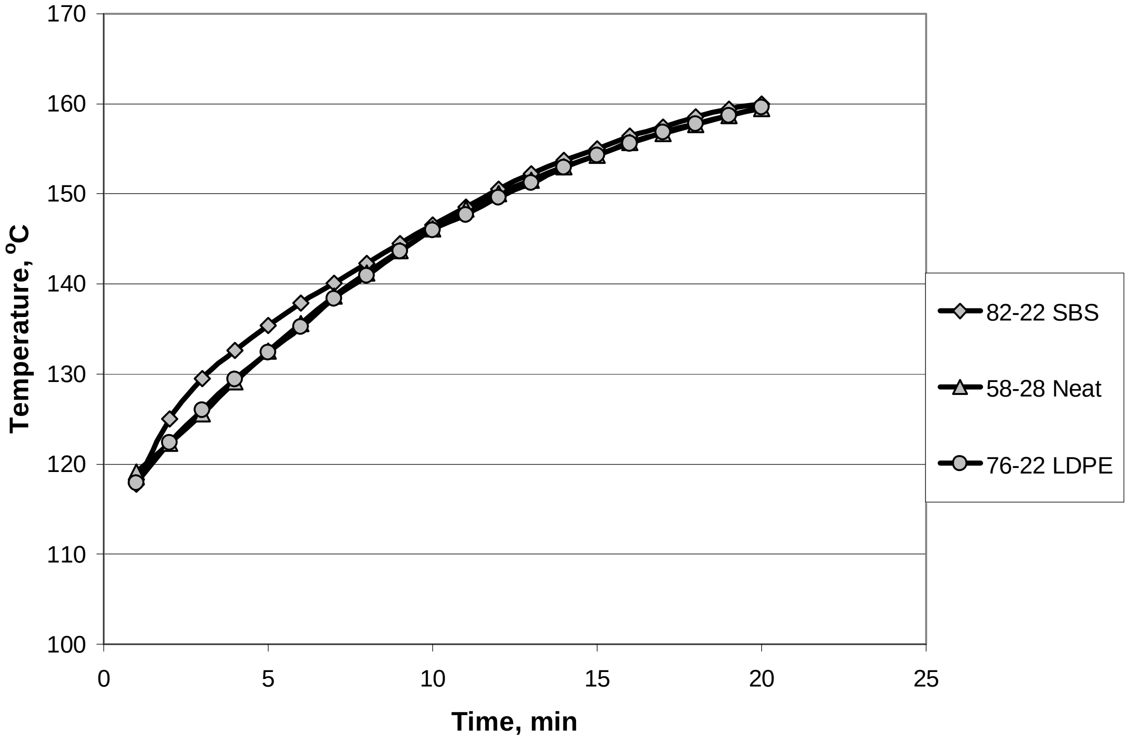

Figure 1. Binder Temperature During Heat-Up Phase of the Stirred Air Flow Test.

Table 1. High Temperature DSR Data From Heat-Up Effects Experiment.

			Tank				After Heat-Up			
Binder	Temp., °C	Rep	G*, kPa	δ, deg	G*/sinδ, kPa	Average G*/sinδ, kPa	G*, kPa	δ, deg	G*/sinδ, kPa	Average G*/sinδ, kPa
PG 58-28	58	1	1.18	87.1	1.18	1.24	1.20	87.0	1.20	1.25
		2	1.30	86.9	1.30		1.29	87.0	1.29	
PG 76-22	76	1	1.45	83.6	1.46	1.48	1.44	84.1	1.45	1.39
		2	1.50	83.3	1.51		1.33	83.9	1.34	
PG 82-22	82	1	1.54	66.3	1.68	1.71	1.54	65.9	1.69	1.71
		2	1.59	66.1	1.74		1.58	65.6	1.73	

3.0 OPERATING PARAMETERS EXPERIMENT

3.1 Materials and Methods

The purpose this experiment was to determine the effect of three operating parameters: impeller speed, air-flow rate, and conditioning time on the properties of asphalt binder aged in the commercial SAFT. Three different unmodified binders, listed in Table 2, were chosen for this experiment. The binders, which have been studied extensively elsewhere, represent a range in chemical composition, mass change, and sensitivity to short-term aging. No modified binders were included because the purpose of the experiment was to determine the SAFT operating parameters that best mimic the aging that occurs in the RTFOT for neat binders. The Verification Study included both neat and modified binders.

Table 2. Binders for the Optimization Study.

Binder/Source	Comments	PG Grade	RTFOT Mass Change (%)	Aging Index, 135°C Viscosity(2)
California Coastal (AAD-2)	SHRP core binder except AAD-1 used during SHRP	52-28	-1.058	2.86
West Texas Intermediate (AAM-1)	SHRP core asphalt	64-16	+0.122	1.98
California Valley (ABM-2)	Replacement for SHRP core binder AAG-2, except AAG-1 used during SHRP	58-16	-0.348	1.62

The testing protocol for this experiment is shown in Figure 2. The commercial SAFT fitted with the VCS-I was used during this experiment. The test procedure for the prototype SAFT was modified slightly based on the capabilities of the commercial equipment and the measurements of filter mass required for the VCS-I.

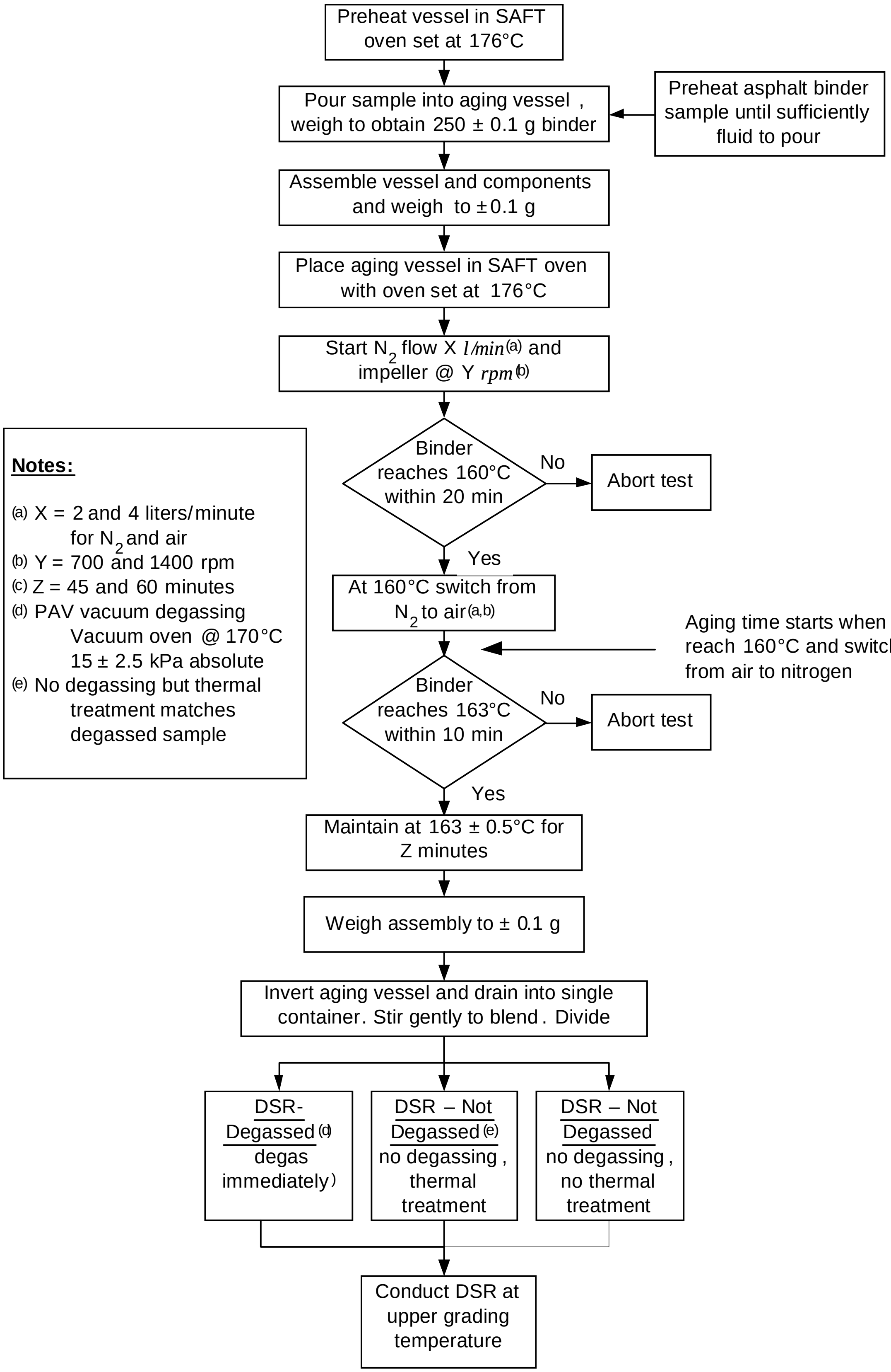

Figure 2. Testing Program for Optimization Study.

The SAFT oven was preheated to 176°C with the aging vessel in place. An asphalt binder sample was then heated in a separate laboratory oven until the binder was "sufficiently fluid to pour." With the binder at the appropriate consistency, the preheated SAFT vessel was removed from the SAFT oven and 250 ± 0.1 g of asphalt binder were poured into the tarred aging vessel. The impeller and lid were assembled on the vessel, and the assembly was then weighed ± 0.1 grams. The assembled vessel was returned to the oven immediately after pouring and weighing during which time the asphalt binder cooled to approximately 90°C . The combined weight of the assembled vessel was included in this experiment so that the total mass change could be compared to the mass of volatile compounds collected in the VCS-I condenser and filters.

As soon as the assembled vessel was reinstalled in the SAFT oven, the stirring and the flow of nitrogen was initiated. Once stirring was initiated, the temperature of the binder must reach 160 °C within 20 minutes for the test to be considered valid. The conditioning time starts when the binder reaches 160 °C at which time the flow of gas is switched from nitrogen to air. The temperature of the binder must reach 163 ± 0.5 °C within 10 minutes and must remain within this temperature tolerance for the duration of the aging. To maintain this level of control, the PID settings of the temperature controller must be adjusted to account for impeller speed and flow rate. These settings were obtained for each combination of impeller speed and flow rate through a tedious trial and error process using the Citgo PG 58-28 binder. The SAFT device as supplied was calibrated for a flow rate of 2,000 ml/min and an impeller speed of 700 rpm; the values specified for use with the prototype SAFT.

Mass change was monitored during the Operating Parameters Experiment. The mass change was monitored by weighing the assembled vessel, the condenser and the two filters before and after the test. The weighing of the vessel was included to provide a check on the mass gained by the condenser and filters.

As soon as the assembled vessel was weighed, the lid and impeller were removed. The vessel was inverted and the binder in the vessel was allowed to drain into a single container. The walls of the vessel were scraped to remove material clinging to the side of the vessel. A very small amount of binder clings to the impeller; therefore, the impeller was not scraped. The residue was stirred with a spatula to thoroughly blend the residue. After being blended, the residue was poured into four containers; three 1-oz tins and a fourth container that contained the remainder of the residue. The residue in each container was stirred gently to aid in the removal of bubbles. One of the 1-oz containers was placed in a vacuum oven for degassing. A second 1-oz container was not be degassed, but was subjected to the same thermal history as the degassed sample. The third 1-oz container was allowed to cool at room temperature.

At this point the conditioning process was completed and the binder from the three 1-oz tins was tested in the DSR at 58 °C and 10 rad/s. The test results were used as the response variable in the statistical analysis. As discussed in more detail below, the experimental design requires that only a single SAFT run be completed for each binder for the selected operating conditions.

3.2 Experiment Design

The experiment design for the Operating Parameters Experiment is presented in Table 3. It employs a Plackett-Burman design to simultaneously assess the effects of changes in impeller speed, air-flow rate, and conditioning time on the aging that occurs in the commercial SAFT. Plackett-Burman designs are often used in ruggedness testing to assess the effect of changes in multiple test parameters. These are extremely efficient designs that allow the main effects to be determined with a limited amount of testing. For the three variables included in this experiment only four test results are needed to assess the main effects. Since we are only concerned with determining the magnitude of the effects and using this data to estimate operating conditions that will reproduce the RTFOT, it is not necessary to perform replicate SAFT runs for each combination of conditions. ASTM 1169 (*1*) presents detailed information on the design and analysis of the type of experiment used here.

Table 3. Operating Parameters Experiment Design.

Binder Source	Impeller Speed (rpm)	Air Flow (l/min)	Aging Time (min)	Testing Plan
AAD-2	700	2	45	Yes
			60	-- --
		4	45	-- --
			60	Yes
	1400	2	45	-- --
			60	Yes
		4	45	Yes
			60	-- --
AAM-1	700	2	45	Yes
			60	-- --
		4	45	-- --
			60	Yes
	1400	2	45	-- --
			60	Yes
		4	45	Yes
			60	-- --
ABM-2	700	2	45	Yes
			60	-- --
		4	45	-- --
			60	Yes
	1400	2	45	-- --
			60	Yes
		4	45	Yes
			60	-- --

3.3 Analysis

This section discusses the analysis of the rheological data from the Operating Parameters Experiment. The rheological data from the Operating Parameters Experiment are summarized in Table 4. Two statistical analyses were performed on the DSR data shown in Table 4.

Table 4. DSR Data From the Operating Parameters Experiment.

Binder	Order	Operating Parameters			DSR at 58 °C											
					Degassed			Oven Treatment			Air Cooled			RTFOT, AVG		
		Speed rpm	Flow L/min	Time min	G*, kPa	δ, deg	G*/sinδ, kPa	G*, kPa	δ, Deg	G*/sinδ, kPa	G*, kPa	δ, deg	G*/sinδ, kPa	G*, kPa	δ, deg	G*/sinδ, kPa
AAD-2	1	700	4	60	1.72	81.5	1.74	1.69	81.6	1.71	1.68	81.5	1.70	2.60	79.0	2.65
AAD-2	2	,1400	4	45	4.16	74.6	4.31	4.20	74.6	4.36	4.03	75.0	4.17			
AAD-2	3	1,400	2	60	4.73	73.2	4.94	4.71	73.4	4.91	4.37	73.6	4.56			
AAD-2	4	700	2	45	1.30	83.4	1.31	1.36	83	1.37	1.32	83.0	1.33			
AAM-1	1	700	4	60	5.43	82.4	5.48	5.31	82.3	5.36	5.04	82.5	5.08	5.94	81.9	6.00
AAM-1	2	1,400	2	60	8.99	78.7	9.17	9.22	78.6	9.41	8.52	78.7	8.69			
AAM-1	3	1,400	4	45	7.37	79.9	7.49	7.57	79.9	7.69	7.03	80.0	7.14			
AAM-1	4	700	2	45	4.13	83.8	4.15	4.37	83.5	4.40	3.77	83.2	3.80			
ABM-2	1	1,400	4	45	4.39	88.8	4.39	4.27	88.8	4.27	4.44	88.8	4.44	3.14	89.5	3.14
ABM-2	2	1,400	2	60	5.24	88.5	5.24	5.04	88.5	5.04	5.39	88.5	5.39			
ABM-2	3	700	2	45	2.45	89.4	2.45	2.50	89.5	2.50	2.54	89.5	2.54			
ABM-2	4	700	4	60	2.85	89.4	2.85	2.83	89.3	2.83	2.97	89.3	2.97			

The first analysis was performed to determine if the PAV degassing procedure affects the DSR data obtained after short-term aging of neat binders in the SAFT. Recall that previous work with modified binders during the Heat-Up Effects Experiment showed that degassing was a necessary step for modified binders aged in the SAFT. For simplicity, the vacuum degassing procedure currently specified in AASHTO R28-06, *Standard Practice for Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel (PAV),* was used. This procedure exposes the binder to 170 °C for 40 ± 2 minutes. For the last 30 ± 1 minutes, the binder is exposed to vacuum with a residual pressure of 15 ± 2.5 kPa absolute. The effect of the degassing process was evaluated by performing DSR tests on three split samples of the SAFT aged material. One sample was immediately cooled at ambient temperature, a second sample was exposed to 170 °C for 40 minutes, and the third sample was exposed to the PAV degassing procedure.

The measurements of G*/sinδ from the 12 SAFT runs are shown in Table 4. Three differences in the G*/sinδ measurements were considered:

1. Difference between degassed and air cooled binder
2. Difference between air cooled binder and binder heated per the degassing procedure but without the application of vacuum (oven treatment)
3. Difference between degassed binder (degassed) and binder heated per the degassing procedure but without the application of vacuum (oven treatment)

The statistical hypothesis test used in analyzing this data is summarized below:

Null hypothesis: The difference caused by the treatments considered is zero

Alternative hypothesis: The difference caused by the treatments considered is greater than zero.

Test Statistic:

$$t = \frac{\bar{d}}{\left(\frac{s}{\sqrt{n}}\right)} \tag{1}$$

where:

t = value of t statistic
$\bar{d}$ = mean difference
s_d = standard deviation of the differences
n = number of paired differences considered

Rejection Region: Reject the null hypothesis if $t > t_{a,n-1}$.

The results of this analysis are summarized in Table 5 and include the mean difference ($\bar{d}$) its standard deviation (S_d), the calculated t statistic (T), and its percentage value (p). Low p values (bold and underlined in Table 5) indicate significant differences; therefore, it can be concluded that the degassing procedure significantly affects the rheology of neat SAFT aged binders, and that it is probably the additional exposure to high temperature that causes this additional aging. Since degassing is needed to remove entrapped air from stiff modified binders, this experiment shows that it must be included as part of the procedure for all binders, and that the degassing must be standardized.

Table 5. Summary of Degassing Effects.

	Paired Differences		
Parameter	Degassed - Air cooled	Oven Treatment - Air cooled	Degassed – Oven Treatment
$\bar{d}$	0.14	0.17	-0.03
S_d	0.23	0.34	0.14
T	2.11	1.75	-0.64
p	**<u>0.03</u>**	**<u>0.05</u>**	0.74

The second analysis that was performed was to investigate the effects of impeller speed, air-flow rate, and conditioning time on the degree of aging that occurs in the SAFT to determine the sensitivity of the SAFT aging to these parameters. Temperature was not included in the experiment because the review of the literature and research in progress indicated a strong desire to perform the short-term aging test at 163 °C, which reasonably simulates hot-mix plant operating temperatures. Figures 3, 4, and 5 show the effect of the three operational parameters on G*/sinδ measured after SAFT aging and degassing.

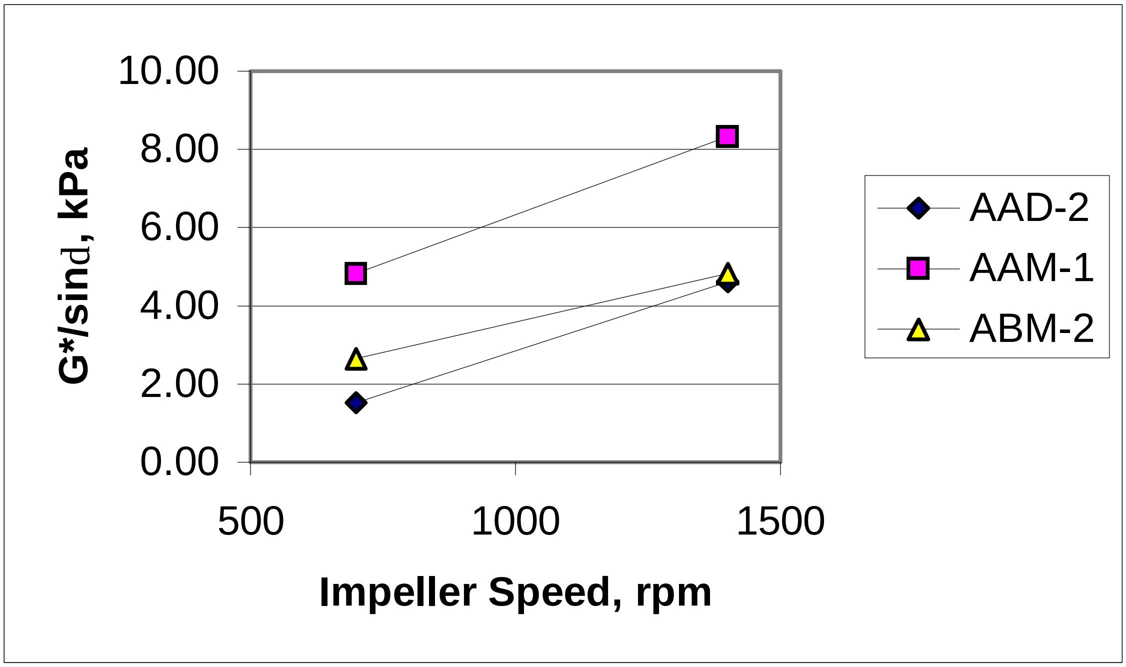

Figure 3. Effect of Impeller Speed on G*/sind for SAFT Aged Material.

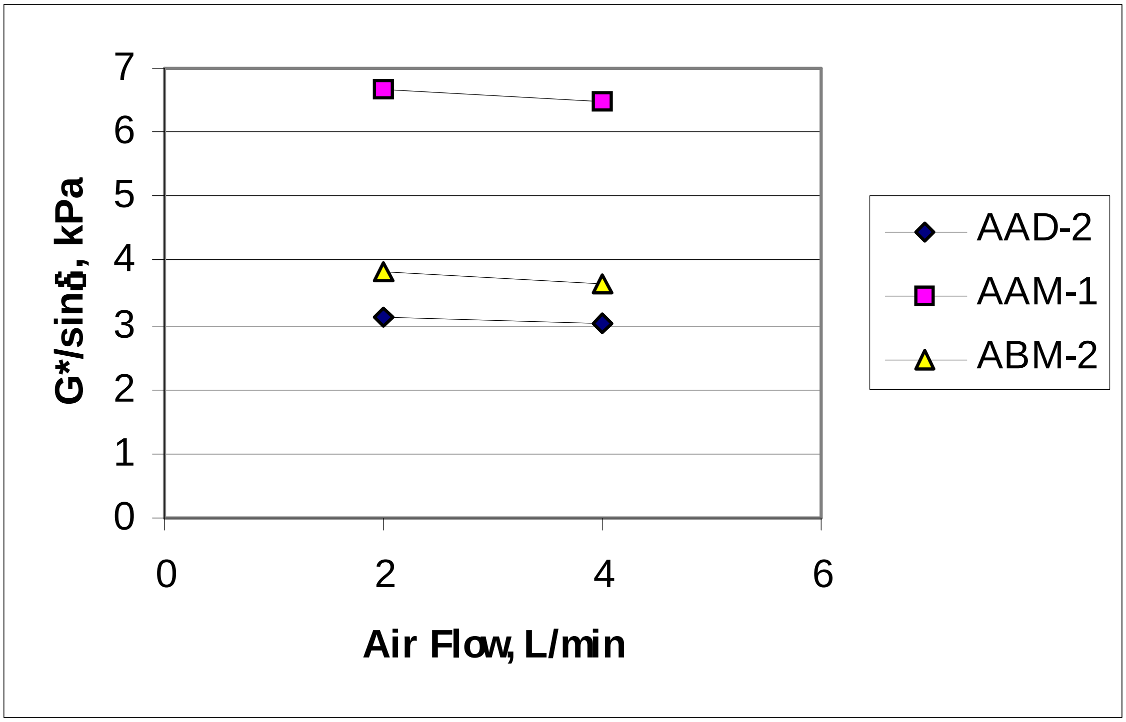

Figure 4. Effect of Air Flow on G*/sind for SAFT Aged Material.

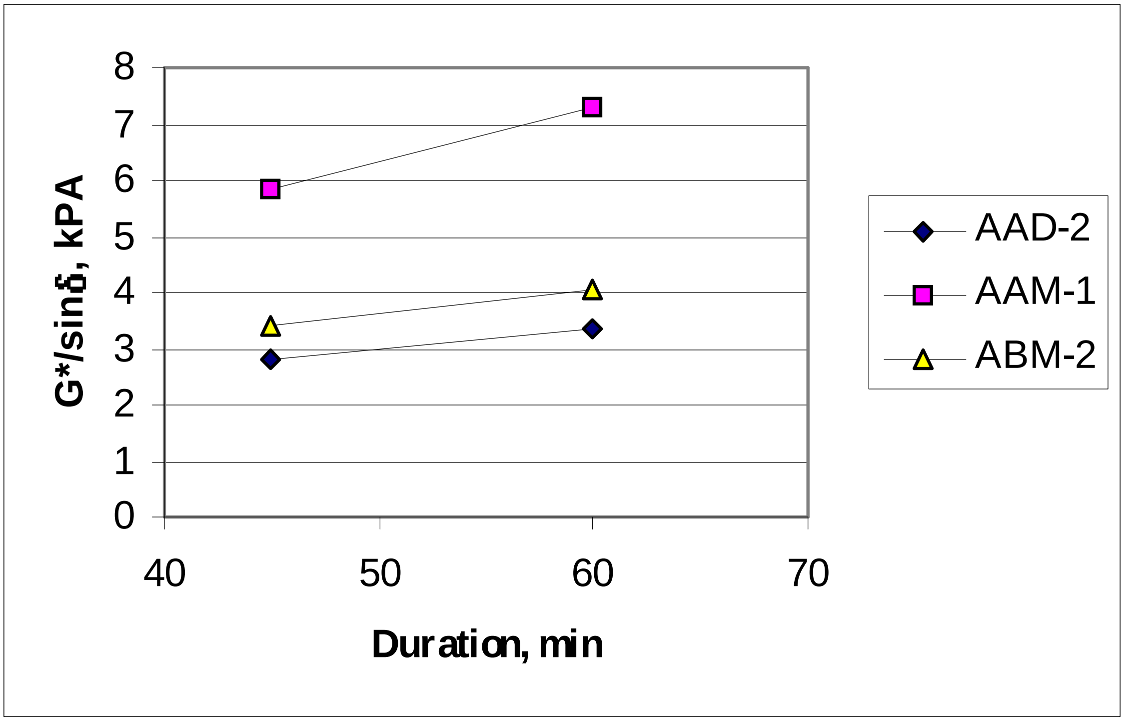

Figure 5. Effect of Conditioning Time on G*/sind for SAFT Aged Material.

From these figures it is clear that the aging in the SAFT is affected by impeller speed and conditioning time, but not by air flow rate over the ranges studied. Using the slopes in these figures, the RTFOT G*/sinδ values measured for three binders, and assuming linear relationships, the impeller speed required to reproduce RTFOT aging at the average conditioning time of 52.5 minutes and the conditioning time required to reproduce RTFOT aging at the average impeller speed of 1050 rpm can be estimated. These are summarized for the three binders in Table 6.

Table 6. Estimated SAFT Operational Parameters to Reproduce RTFOT Aging.

Binder	RTFOT G*/sinδ, kPa	Estimated Impeller Speed, for 52.5 min Conditioning, rpm	Estimated Conditioning Time for 1,050 rpm Impeller Speed, min
AAD-1	2.65	962	40
AAM-1	6.00	935	47
ABM-2	3.14	857	38
Average		918	42

Based on Table 6, it appeared that using an impeller speed of 1,000 rpm, an air flow rate of 2000 ml/min and a conditioning time of 45 minutes will result in aging in the commercial SAFT that is approximately equivalent to that which occurs in the RTFOT. Thus, the tentative operating parameters for the commercial SAFT were:

- 163 °C aging temperature.
- 2,000 ml/min air flow
- 1,000 rpm impeller speed
- 45 minute aging time
- 250 g sample mass
- Vacuum degassing per AASHTO R28 after short-term aging in the SAFT

The analysis presented above assumes linear effects. Developmental work on the VCS-II provided an opportunity to assess the tentative operating parameters. Independent rheological data (average of duplicate runs) collected during the VCS-II testing are presented in Table 8. These data show that for the operating conditions listed above for the SAFT underestimates the aging that occurs with the RTFOT in three of the four cases. Either the conditioning time or the impeller speed can be increased to increase the degree of aging in the SAFT. The magnitude of these changes were estimated to be a 100 RPM increase in impeller speed or a 5 minute increase in conditioning time to increase $G^*/\sin\delta$ by 7 percent. After careful consideration, the longer conditioning time was selected because of concern that further increases in impeller speed could result in significant quantities of binder splashing onto the lid of the SAFT with the potential for asphalt droplets to exit into the VCS. Visual observation from completed tests indicated that at 1,000 RPM no asphalt splashes on the lid while at 1,400 RPM a significant amount of asphalt splashes onto the lid.

Table 7. Rheological Properties From VCS-II Development Study.

Property	Aging Condition	Citgo 58-28	ABM-2	AAM-1	AAD-2
G*, kPa	Original	1.18	1.89	2.95	0.88
	After SAFT	2.44	3.17	4.94	2.19
	After RTFOT	2.48	3.15	5.41	2.60
Phase Angle, deg	Original	87.3	89.7	85.6	85.8
	After SAFT	83.7	89.2	82.7	79.7
	After RTFOT	84.1	89.5	80.3	79.0
G*/sinδ	Original	1.18	1.89	2.96	0.88
	After SAFT	2.45	3.17	4.98	2.23
	After RTFOT	2.49	3.15	5.49	2.65

Table 8 presents rheological data for the Citgo PG 58-28 where the conditioning time was increased from 45 minutes to 50 minutes. As shown, this change increased G*/sinδ by approximately 6.5 percent. With the new operating parameters the SAFT ages the Citgo PG 58-28 binder slightly more than the RTFOT, but based on Table 7, this increased aging should provide better agreement when a wide range of binders is considered.

Table 8. Rheological Properties for the Citgo PG 58-28 for SAFT Aging Times of 45 and 50 Minutes Compared to RTFOT.

Property	RTFOT	SAFT 45 min Aging	SAFT 50 min Aging
G*, kPa	2.48	2.44	2.65
Phase Angle, deg	84.1	83.7	83.9
G*/sinδ, kPa	2.49	2.45	2.66

The final operating parameters selected from the Operating Parameters Experiment for the commercial SAFT for use in the Verification Study were:

- 163 °C aging temperature.
- 2,000 ml/min air flow
- 1,000 rpm impeller speed

- 50 minute aging time
- 250 g sample mass
- Vacuum degassing per AASHTO R28 after short-term aging in the SAFT

Figure 6 illustrates the sequence of operations for the commercial SAFT determined from the SAFT Optimization Study. This sequence was used in the Verification Study.

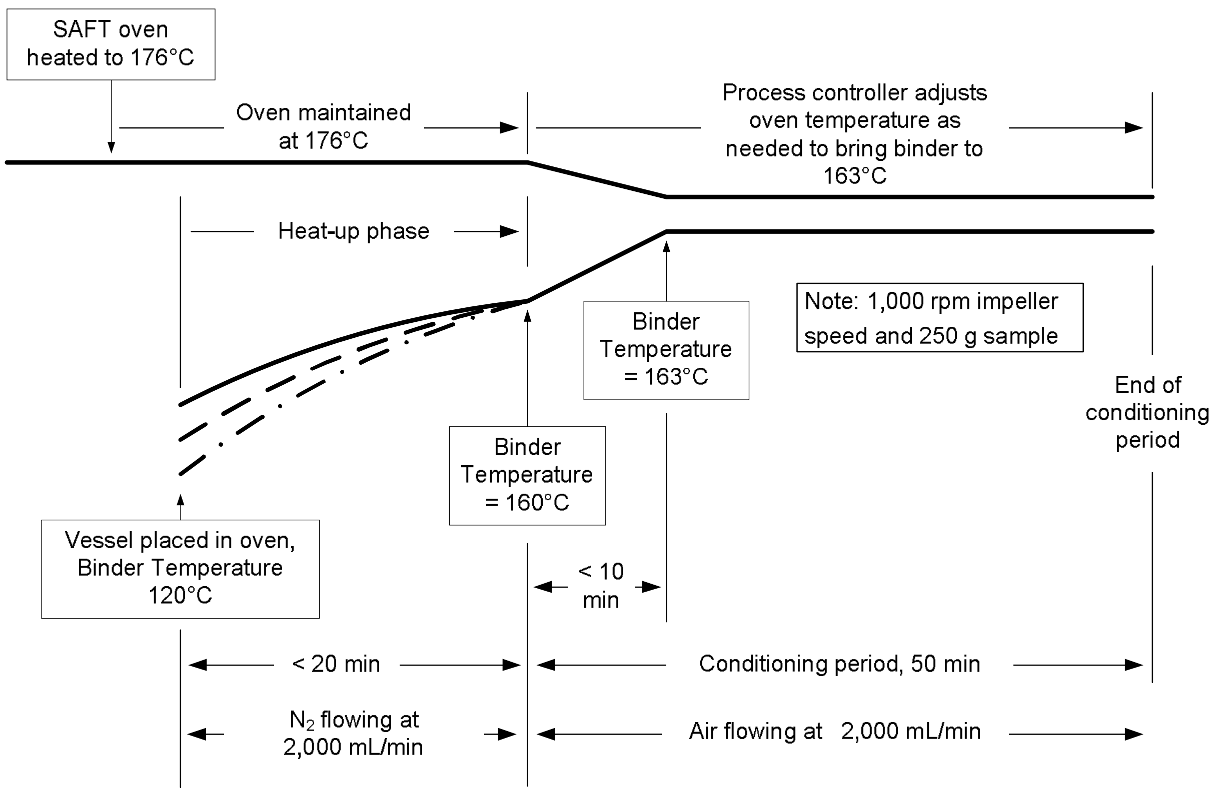

Figure 6. Sequence of operations used in final SAFT configuration,

4.0 REFERENCES

1. American Society for Testing and Materials, Designation E 1169 - 02, Standard Guide for Conducting Ruggedness Tests, Annual Book of ASTM Standards, Vol. 14.02.