

THIN FILM OXIDATIVE AGING AND LOW TEMPERATURE PERFORMANCE GRADING USING SMALL PLATE DYNAMIC SHEAR RHEOMETRY: AN ALTERNATIVE TO STANDARD RTFO, PAV, AND BBR

Michael J Farrar, R. William Grimes, Changping Sui, Jean Pascal Planche, Shin Che Huang, Thomas F. Turner, Ron Glaser

Western Research Institute

ABSTRACT

This report describes progress towards development of a new thin film (300 μm) short- and long-term aging test suggested as an alternative to the standard rolling thin film oven (RTFO) and pressure aging vessel (PAV) tests. The new test, referred to as the Simple Aging Test (SAT), can be applied to neat and modified bitumens, and emulsion residues. Before the Strategic Highway Research Program, there was considerable interest in static thin film aging. However, the RTFO test was adopted for short term aging because of the large amount of aged asphalt needed for the Bending Beam Rheometer (BBR) test. Since the adoption of the RTFO, it has been found problematic for aging highly modified binders. In the case of warm mix binders, RTFO testing at lower temperature to simulate a "warm" plant may limit rolling needed to uniformly age the material. Recent development of a 4-mm dia. parallel-plate dynamic shear rheometer (DSR) technique allows DSR tests to -40°C , requires only 25 mg of binder, and can estimate BBR m-value and creep stiffness. By combining this new DSR technique with the SAT, the low and intermediate temperature rheology of short and long-term aged bitumens can be determined. Also, there is sufficient aged material to perform high temperature DSR using 25 mm plates. The SAT eliminates the "rolling" issue for polymer modified and warm mix asphalts. The SAT time and temperature equivalent to reach the same aging as the standard RTFO and PAV are reported for several bitumens.

Keywords: Bitumen, Aging, Oxidation, RTFOT, Durability

1. INTRODUCTION

The oxidative age hardening that occurs during construction and laydown and during the life of hot mix pavement has a large impact on pavement performance measures such as rutting, raveling and cracking, and consequently, durability. Simulating this age hardening in the laboratory is critical in developing performance based specifications. In the present study a newly developed thin-film oxidative aging test is described that provides equivalent results to the standard rolling thin film oven (RTFO) (ASTM D2872) and Pressure Aging Vessel (PAV) (ASTM D6521) tests when applied to neat bitumen. The key development making a thin film (several hundred μm 's) aging test feasible is a recently developed dynamic shear rheometry technique that allows testing to -40°C , requires only ~ 25 mg, and includes a correction for instrument compliance.^[1]

The rolling thin film oven test (RTFOT) was developed in the early '60's by Hveem et al.^[2] and was adopted by ASTM in 1970. During the strategic highway research program (SHRP) it was selected as the standard for simulating the short term oxidative aging that occurs during construction and laydown and is currently used in the Superpave bitumen specifications.

The key advantage of the rolling thin film oven test, compared to static aging tests such as the Thin Film Oven Test (TFOT) (ASTM D 1754) is the increased surface exposure in the RTFO due to the rolling action of the glass vessels. However, the amount of rolling and exposure of fresh faces from one bitumen to another varies with the viscosity of the bitumen before and the change in viscosity that occurs during the test. The flow and mixing of bitumen in the bottles is further complicated when polymer modified. Turning down the temperature in the RTFO to simulate the oxidation that occurs during warm mix asphalt production causes similar complications.

This reduction or elimination of rolling during the RTFO test with some highly modified bitumens has been well known for a number of years.^[3] Bahia et al.^[3] attempted to resolve the problem by introducing a metal rod into the RTFO bottle to induce film spreading. Testing performed by FHWA and the South East Asphalt User Producer Group concluded that metal rods reduced the extent of aging of modified and unmodified binders and caused both types of bitumens to creep out of the bottles.^[4]

The problem of bitumens creeping out of the bottle may have been exacerbated by the metal rod, but polymer modified bitumens often creep out of the bottle without the presence of a metal rod. The reason is attributed to the Weissenberg effect (the climbing of a visco-elastic liquid up a rotating rod).^[5] It is speculated the elastic forces (stretching of the polymer chains) generated by the rotation of the glass vessels result in a positive normal force causing the bitumen to creep up the inlet sides of the bottle.

These problems with the RTFO test have led to development at WRI of a new short and long-term aging test referred to as the Simple Aging Test (SAT), which is the subject of this paper. The requirements for the SAT as an alternative to the RTFO and standard RTFO/PAV:

- 1) Match the RTFOT in terms of extent of aging on neat bitumen ... as long as the Superpave specifications are based on RTFOT results, any new test must be able to duplicate the RTFOT aging for neat bitumens.
- 2) Keep it simple, i.e., eliminate the tedious bottle scrapping and cleaning. Design the test so the short-term aged sample can be placed directly in the long term aging oven.
- 3) Produce sufficient bitumen for SHRP PG specification testing and chemical characterization.
- 4) Do not involve solvents for casting or recovery; this is particularly important for polymer modified bitumens.
- 5) Reduce the film thickness to significantly reduce the oxygen diffusion problem that is clearly present in static thin film tests such as the TFOT.
- 6) Design it so the temperature can be reduced to simulate warm mix production.
- 7) Keep the testing time at or preferably less than the current RTFO and RTFO/PAV tests.
- 8) Keep the cost low.
- 9) Design the test so that there is potential for aging emulsion residue films.

Thin film oxidative aging -- defined here as a film of several hundred microns -- would address most if not all of the above requirements, but requires a very large surface area to generate sufficient bitumen for current SHRP specification testing. In particular, the Bending Beam Rheometer (BBR) test is the sticking point because each beam requires approximately 15 g and typically several beams are required. The intermediate and high temperature SHRP tests are performed on a dynamic shear rheometer and only require several grams of bitumen.

However, as noted above, recent development of a 4-mm diameter parallel plate dynamic shear rheometer (DSR) technique allows DSR tests down to -40°C , requires only 25 mg of binder, and can be used to estimate BBR m-value and creep stiffness.^[6] The technique is discussed in more detail below. With this new DSR technique, thin film aging is

now a feasible alternative to the RTFOT and generates sufficient bitumen for low, intermediate and high temperature SHRP specification tests. Also, sufficient bitumen is generated for chemical characterization, such as:

- Fourier transform infrared spectroscopy (~200 mg)
- Gel permeation chromatography (~200 mg)
- Differential Scanning Calorimetry (~200 mg)
- SARA separations (~500 mg)

Research and new test development over the last decade has concentrated on: a) generating several hundred grams of aged bitumen, primarily for BBR testing, b) resolving issues related to the bitumen creeping out of the RTFO bottle and c) addressing the lack of rolling with some polymer modified bitumens in the RTFOT. The Rotating Cylinder Ageing test (RCAT)^[7], Modified German Rolling Flask (MGRFT)^[4], and Stirred Air Flow test (SAFT)^[8] are examples.

2. EXPERIMENTAL AND MATERIALS

2.1 Dynamic shear rheometry using 4mm diameter parallel platens

A dispute concerning the modulus of glycerol in the glassy regime (G_g) led Schröter et al.^[9] to develop a method to correct for the compliance of a dynamic shear rheometer, and its tools and platens. Sui et al.^[1] applied this compliance correction method to bitumen low temperature dynamic shear measurements (~ 5°C to -40°C) using 4 mm diameter parallel plates and a 1.7 mm gap (the technique is referred to here as simply “4 mm DSR”). The effect of the compliance correction is demonstrated in Figure 1. At low frequency (high temperature) the bitumen is compliant and the effect of the instrument compliance correction negligible. However, at high frequency (low temperature) the bitumen is no longer as compliant and the compliance correction adjusts the DSR measurements to reveal the true magnitude of the bitumen glassy modulus.

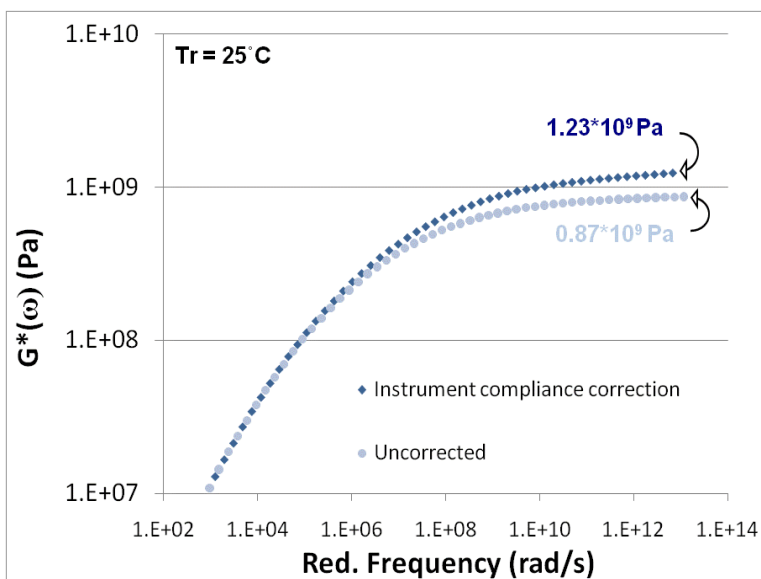


Figure 1 : Master curves of the complex shear modulus from corrected and uncorrected data for MN1-5 RTFO/PAV aged bitumen as a function of reduced frequency at a reference temperature of 25°C measured using 4 mm DSR

The test method requires only 25 mg of material (in practice about 150mg is necessary in order to allow sample trimming, etc), which is several orders of magnitude less than the amount required to fabricate a BBR beam. Also, no specimen pre-molding is needed and a relatively low temperature (60 ~ 70°C) is required to load the samples into the rheometer.

In the present study, the low-temperature rheological properties are measured with 4mm DSR. BBR m-value and creep stiffness $S(t)$ are estimated through a correlation with 4mm DSR developed by Sui et al.^[6] In the Sui et al. method, the slope and magnitude of the shear stress relaxation modulus $G(t)$ master curve at 2 hours and at the true low PG grading temperature are correlated with the corresponding $S(t)$ and m-values at 60 seconds and 10°C above the true low PG grading temperature from BBR measurements. The Sui et al. method was modified by measuring $G(t)$ slope and magnitude at 60 seconds and 10°C higher than the PG grading temperature. Intermediate and high temperature rheological properties were measured using 8 and 25mm diameter platens.

Interconversion of the storage modulus $G'(\omega)$ to relaxation modulus was performed by the approximate expression developed by Christensen.^[10]

$$G(t) \approx G'(\omega) |_{\omega=2/\pi t}$$

Dynamic shear moduli measurements were performed with Malvern Kinexus and TA ARES rotational dynamic shear rheometers (DSR). Frequency sweeps were typically performed at 15°C intervals over a temperature range of -30 to 45°C and an angular frequency range of 0.1 to 100 rad/sec. At lower temperatures the high frequency end of the sweep was reduced to 50 rad/sec.

Spinoffs or new applications of 4mm DSR technology include forensic field micro-sampling and micro-extraction for conducting pavement performance and material investigations; low and intermediate temperature rheological characterization of bitumen and recovered binders; rheological characterization of emulsion residue and crack sealant; and development of a thin-film aging test, which is the topic of this paper.

2.2 Infrared spectroscopy

Infrared analysis was performed in the transmission mode with a 1.0 mm solution cell with 50 mg bitumen per 1 ml carbon disulfide. Thirty-two scans were co-added on a Perkin Elmer Spectrum 100 operated at a resolution of 4 cm⁻¹.

2.3 Materials

Table 1 describes the bitumens used in this study. They are tank bitumens collected during construction. Three of the four binders are from a Rochester, MN comparative test site constructed in 2006. The MB bitumen is from a Manitoba warm mix test site constructed in 2010. These sites were constructed and are being monitored in a cooperative effort by the Minnesota Department of Transportation, the Manitoba Infrastructure and Transportation Department, Western Research Institute, the Asphalt Research Consortium, and the Federal Highway Administration.

Table 1 : Description: bitumens used in this study

Bitumen Binder	Description
MN1-3	Unmodified binder, PG 58 -28, Canadian blend.
MN1-4	Unmodified binder, PG 58 -28, Blend of Arab heavy, Arab medium, and Kirkuk.
MN1-5	Unmodified binder, PG 58 -28, Venezuelan blend.
MB	Unmodified binder, 150/200 pen, PG 58 – 28, Canadian blend

3. DISCUSSION AND RESULTS

3.1 Development of the SAT and comparison to RTFO and RTFO/PAV

The first point to make is the SAT is not a new idea. Bitumen thin-film aging to simulate the short and long-term oxidative aging that occurs in the field has been extensively studied over the last 70 years, a good summary of the literature has been performed by Airey.^[11]

The SAT was developed by adapting and extending the previous research on thin film aging, particularly the work performed by Glover et al.^[8,12] and Petersen et al.^[13] and employing 4mm DSR which, as discussed above, makes thin film aging a practical alternative to the RTFOT.

One of the principal goals during development of the SAT was to make the bitumen film as thin as possible to reduce diffusion effects. The SAT film thickness of 300 µm's was arrived at by trial and error in trying to prepare a uniform thin film without solvent casting. The amount of sample required for rheological and chemical testing was also a limiting factor.

The SAT aging protocols are summarized in Table 2. The table describes the short term SAT protocol for WMA as 130°C for 50 minutes in a forced draft oven. This should be considered preliminary as we are only just beginning to make the comparison between the aging that occurs with this protocol and the aging that actually occurs on a WMA project.

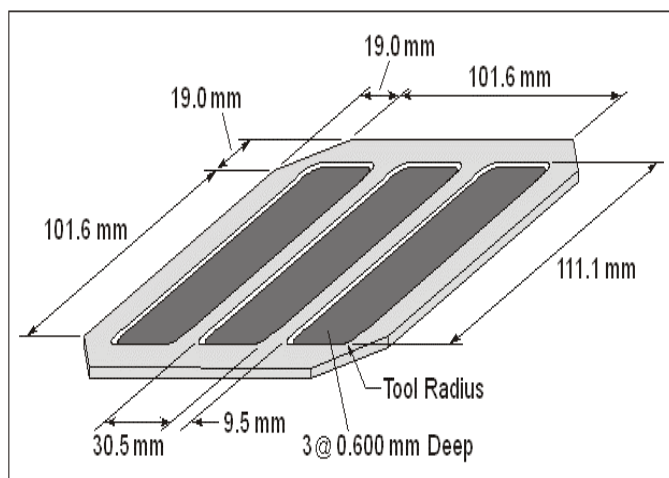
Table 2 : SAT protocol descriptions

SAT Protocol	Description	Equivalent AASHTO aging test
ST SAT HMA	Short term simple aging test 50 min. at 150°C in a forced draft oven	RTFO (ASTM D2872, AASHTO T240)
ST SAT WMA	Short term simple aging test 50 min. at 130°C in a forced draft oven	None
LT SAT PAV	Long term simple aging test in a pressure aging vessel for 8 hours at 100°C.	RTFO/PAV (ASTM D6521, AASHTO R 28)
LT SAT FDO	Long term simple aging test in a forced draft oven for 40 hours at 100°C.	~ RTFO/PAV (ASTM D6521, AASHTO R28)

Note: LT SAT PAV or LT SAT FDO samples are initially aged using either the ST SAT HMA or WMA protocols.

Exploratory infrared spectra collected near the surface and bottom of a 300 µm film after SAT short term aging suggested the presence of an oxidative gradient in terms of carbonyl and sulfoxide. Even though the film is an order of magnitude thinner than the TFOT film, the observed gradient suggests the reaction is diffusion limited, i.e. the rate of carbonyl and sulfoxide formation within in the film is dependent on the rate of oxygen transport and may affect the reaction mechanisms and concentration ratio of the various oxidation products within the film. Molecular structuring at the interface between the film and the SAT plate may also play a role in oxygen transport. The diffusion issue becomes even more complex for polymer modified bitumen. Aspects of these issues are under investigation at WRI. However, Glover^[8], in a limited investigation, considered the diffusion in an unmodified bitumen, by aging a series of thin films varying from 0.5 to 3.5 mm at different times at 163°C. Glover concluded that film thickness did not affect the mechanism of oxidation reaction and that by decreasing the thickness of the film it is possible to reduce the diffusion effects and speed up the reaction.

Figure 2 shows the latest SAT prototype for short and long-term thin film aging. There are three slots on the plate to allow aging of three separate films. Each slot requires 1.00 g of bitumen, which produces a film thickness of about 300 µm.

**Figure 2 : SAT plate dimensions**

Preparing SAT bitumen thin films for oxidative aging involves bringing a hot plate to approximately 120°C in a nitrogen saturated glove box or tent and then placing the SAT plate (with bitumen) onto the hot plate. As the bitumen temperature approaches 120°C it spreads out to roughly two-thirds of the slot surface. A small spatula is used to spread the bitumen to the remaining unwetted surface. After spreading, the bitumen is allowed to remain at temperature for several minutes to level and evenly spread out. The plate is then removed from the hot plate and allowed to return to room temperature under nitrogen.

A small hole was drilled into the side of the plate to allow insertion of a thermocouple probe to monitor the temperature in the forced draft oven. A preheated block of aluminum is below the SAT plate to rapidly bring the SAT plate to temperature when inserted into the oven. Figure 3a shows the temperature probe output from the SAT plate, performed for 50 minutes at an oven temperature of 150°C. The temperature of the SAT plate increases rapidly from room temperature to about 140°C and then gradually increases to 146°C after 50 minutes.

The short and long-term aging scheme is illustrated in Figure 3b. The plate dimensions were selected so the plate can be inserted into a standard pressure aging vessel tray. It was determined it requires approximately 40 hours in a forced

draft oven at 100°C and 0.74 atm pressure with a 300 µm film to match the standard PAV (ASTM 6521) at 20 atm using a standard PAV pan with a film thickness of approximately 3.2mm. The 40 hours is approximate because of the pressure dependency variation among bitumens.^[14] Aging the 300 µm film in a PAV at 20 atm for only 8 hours matches the level of aging of the standard PAV again using a standard PAV pan and film thickness.

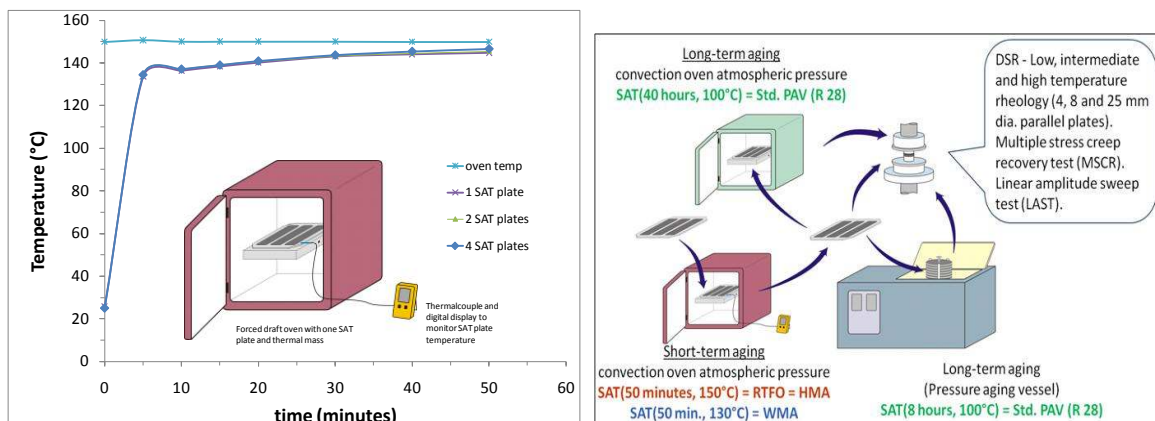


Figure 3a : SAT short-term aging temperature profile. **b:** Short and long term-aging scheme

Infrared spectroscopy was used to establish initial estimates of the time and temperature required to simulate the RTFO and RTFO/PAV extent of aging. The initial estimates were then verified with rheology. For example, Figure 4a compares the SAT, RTFO, and RTFO/PAV infrared spectra for the MB bitumen using the protocols listed in Table 2. Figure 4b shows the corresponding rheology in terms of the complex shear modulus $G^*(\omega)$. The spectra are remarkable close. Figure 5 compares the percent change in carbonyl and sulfoxides absorption for all four bitumens studied during aging. The carbonyl net absorbance was measured at 1693 cm^{-1} . Similarly, the sulfoxide net absorbance was measured at 1020 cm^{-1} .

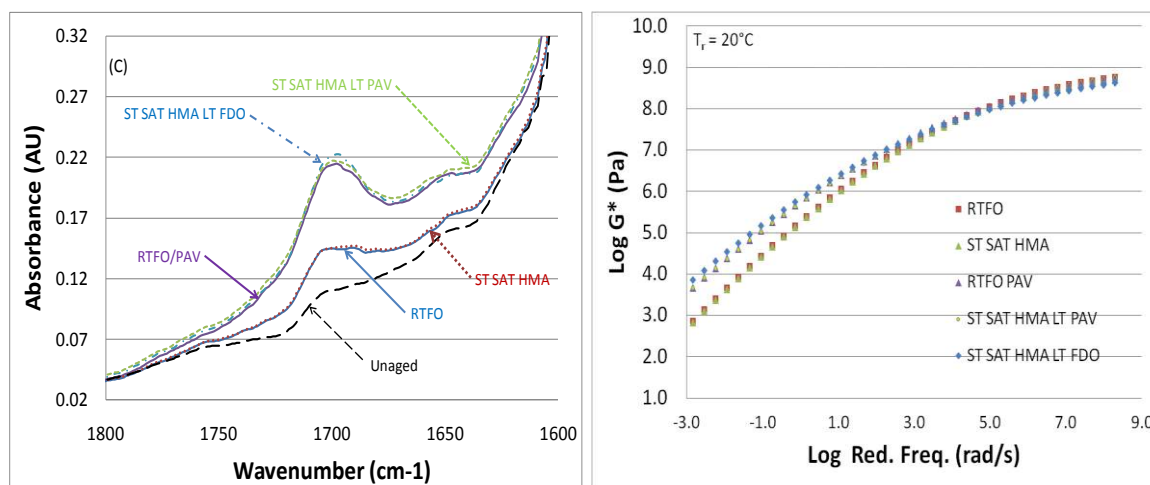


Figure 4a: Infrared Spectra MB bitumen: unaged, and short and long-term aged. **4b:** Complex shear modulus MB bitumen. Aging followed the protocols in Table 2.

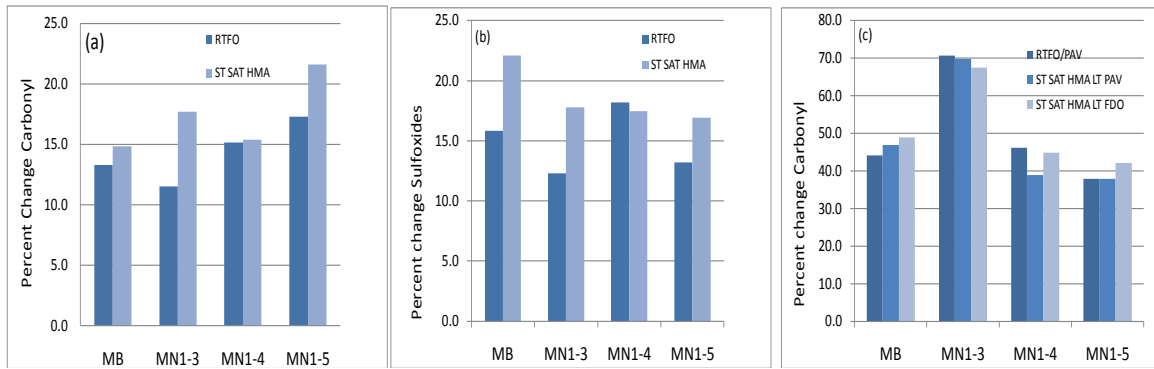


Figure 5 : a: Percent increase in carbonyl absorbance - unaged to RTFO and unaged to ST SAT HMA. b: Percent increase in sulfoxide absorbance - unaged to RTFO and unaged to ST SAT HMA. c: Percent increase in carbonyl absorbance - RTFO to RTFO/PAV, RTFO to LT PAV, and RTFO to LT FDO.

3.2 Continuous grade and rheological comparison (SAT vs. RTFO and RTFO/PAV)

The continuous grade of the RTFO, RTFO/PAV, and SAT aged binders is shown in Table 3. For both the short and long-term aging, good agreement was found between SAT aging and RTFO and RTFO/PAV aging. It appears the SAT does not significantly affect the continuous grade of bitumens studied, even the ST SAT HMA LT FDO results, which might tend to vary from the ST SAT LT PAV and RTFO/PAV conditions due to pressure dependency effects, show relatively good agreement.

Table 3 : Summary of continuous grading: RTFO, RTFO/PAV, and SAT aged binders

Bitumen	Aging Method	Continuous Grade temperature (°C)		
		Low	Intermediate	High
MB	RTFO			59.3
	ST SAT HMA			58.8
	RTFO/PAV	-34.0	13.2	
	ST SAT HMA LT PAV	-35.0	12.3	
	ST SAT HMA LT FDO	-34.0	13.6	
MN1-3	RTFO			60.5
	ST SAT HMA			61.7
	RTFO/PAV	-29.8	18.0	
	ST SAT HMA LT PAV	-30.0	16.8	
	ST SAT HMA LT FDO	-30.0	18.1	
MN1-4	RTFO			61.7
	ST SAT HMA			62.0
	RTFO/PAV	-32.0	10.9	
	ST SAT HMA LT PAV	-33.0	12.1	
	ST SAT HMA LT FDO	-33.4	11.4	
MN1-5	RTFO			58.5
	ST SAT HMA			59.2
	RTFO/PAV	-32.0	14.7	
	ST SAT HMA LT PAV	-31.9	16.9	
	ST SAT HMA LT FDO	-30.6	16.8	

Comparisons of the low (m-value), intermediate ($G^* \sin \delta$), and high temperature ($G^*/\sin \delta$) grading criteria for the SAT, RTFO and RTFO/PAV are shown in Figures 6 through 8, respectively. Only the m-value for the low temperature grading criteria is presented since the bitumens studied were predominately m-controlling. The figures include the ASTM single operator precision limits (d2s %). For example, the single operator precision for m-value reported in ASTM D6648-08 is 4.0%. The single operator precision limits represent tests on duplicate samples by the same

operator, using the same rheometer, in the same lab. Three of the four bitumens fall within the single operator precision limits at the three temperature levels, indicating the SAT and RTFO and RTFO/PAV rheology are comparable.

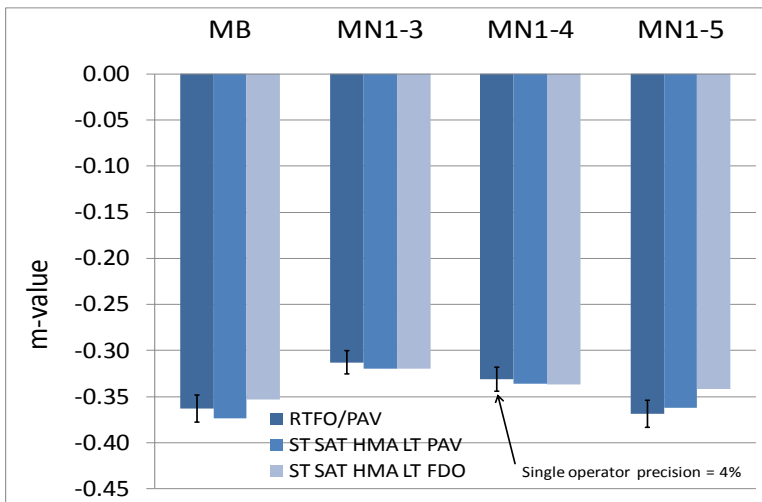


Figure 6 : Comparison of low temperature grading criteria (m-value) (-28°C): RTFO/PAV vs. SAT

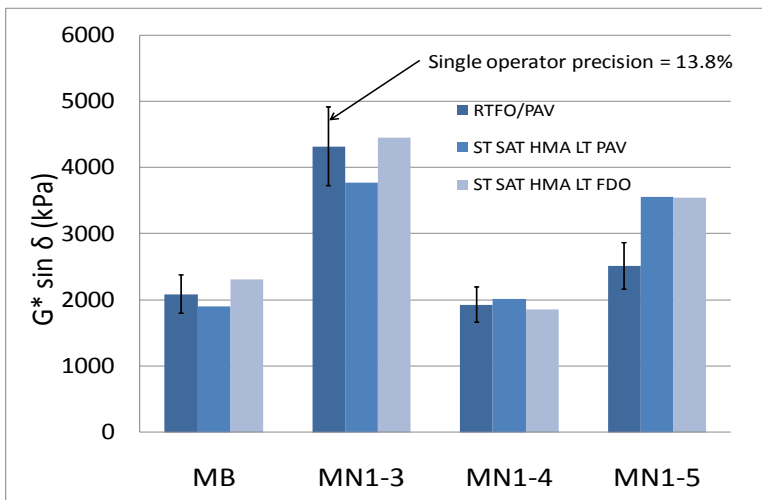


Figure 7 : Comparison of intermediate grading criteria ($G^* \sin \delta$) (19°C): RTFO/PAV vs. SAT

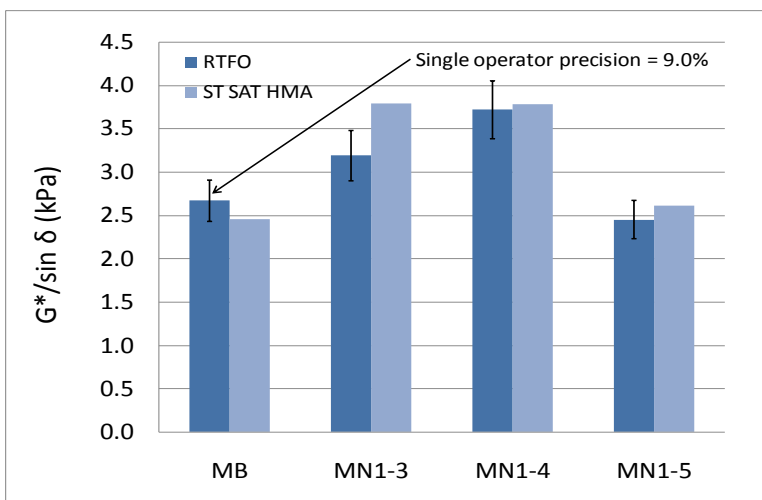


Figure 8 : Comparison of high temperature grading criteria ($G^*/\sin \delta$) (58°C): RTFO/PAV vs. SAT

4. PERCENT MASS CHANGE

Percent mass change from RTFO aging was determined in accordance with AASHTO T240. The percent mass change during short term SAT aging was determined in a similar manner i.e. the mass change of the bitumen was determined as a mass percent of the original material. A mass loss results in a negative number and a mass gain in a positive number. A mass loss is generally observed in the RTFOT, but occasionally a mass gain can occur.^[15] The percent mass change from short term SAT aging and RTFO aging are compared in Table 4. For two of the asphalts the difference in percent mass change is negligible. The other two tests suggest the SAT has a greater mass loss than the RTFO but by a relatively small amount. Additional tests are necessary to conclude the SAT is statistically different from the RTFO in terms of percent mass change.

Table 4. Percent Mass Change RTFO and ST SAT HMA

Bitumen	SAT Mass Change (%)	RTFO Mass Change (%)	Difference (RTFO – SAT)
MB	-0.43	-0.49	-0.06
MN1-3	-0.88	-0.71	0.17
MN1-4	-0.66	-0.26	0.40
MN1-5	-0.80	-0.80	0.00

5. CONCLUSION

For the neat bitumens studied, the analysis illustrates the Simple Aging Test (SAT) method is very comparable to the RTFO and RTFO/PAV in terms of rheological characteristics and to a lesser degree with carbonyl and sulfoxide generation. By combining the simple aging test (SAT) with 4mm dia. parallel plate dynamic shear rheometry the low temperature rheology and specification parameters, such as m-value and creep stiffness, can be determined. The time required to perform the short term component of the SAT is 50 minutes. This is 35 minutes less than required by the RTFOT method. Even more significant, the time required to run the long-term component of the SAT with the PAV is 8 hours -- 12 hour less than the standard PAV test. Work is in progress on applying the SAT to polymer modified bitumens, emulsion residue, recovered recycled bitumen, and to simulate the oxidative aging that occurs in a warm mix plant. Each SAT plate generates 3 g of aged bitumen, which is sufficient for low, intermediate and high temperature rheology and chemical characterization. If additional aged binder is necessary, several SAT plates can be prepared and aged simultaneously.

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