INTERIM REPORT

to the

NATIONAL COOPERATIVE HIGHWAY RESEARCH PROGRAM
(NCHRP)

Project NCHRP 09-54

Long-Term Aging of Asphalt Mixtures for Performance Testing and Prediction

from

Y. Richard Kim, Ph.D., P.E., F.ASCE
Cassie Hintz, Ph.D.
Farhad Yousefi Rad, Michael Elwardany
North Carolina State University

Shane Underwood, Ph.D.
Arizona State University

Mike J. Farrar, Ronald R. Glaser
Western Research Institute

October 2013

Table of Contents

List of Figures

List of Tables

Part I: Literature Review

Introduction

The issue of oxidative aging of asphalt binder has been recognized and studied for almost a century. Hubbard and Reeve (1913) published the first results of a study that examined the effects of a year of outdoor weathering on the physical (weight, hardness, etc.) and chemical (solubility) properties of paving grade asphalt cements. Subsequent studies confirmed their basic finding that oxidation, and not volatilization alone, is responsible for changes in asphalt properties that occur due to exposure (Thurston and Knowles 1936, Van Oort 1956, Corbett and Merz 1975). Significant research also has been conducted to investigate the chemical aspects of the aging process, and excellent reviews of these studies can be found in the literature (Wright 1965, Lee and Huang 1973, Jemison et al. 1992, Petersen 2009). In some of these studies, researchers conducted sophisticated experiments and proposed conceptual, empirical and/or analytical models to explain the aging phenomenon of asphalt binder (Lunsford 1994, Liu et al. 1996, Petersen and Harnsberger 1998, Glover et al. 2008). Although a significant amount of effort has been placed on understanding the aging process of asphalt binder, relatively little work has been put forth to develop laboratory aging procedures for producing aged mixture specimens for performance testing. In addition, very little effort has been made to validate the laboratory aging methods with field measurements. Furthermore, the existing laboratory aging methods do not allow for consideration of different climates, depths, etc., which are known to impact aging significantly. A review of the pertinent literature that can be used to develop a more comprehensive method of producing aged specimens for performance testing is provided in the following sections.

1. Overview of Methods for Aging Asphalt Mixtures in the Laboratory

Several asphalt mixture laboratory aging procedures have been tried. These procedures can be broadly classified based on (a) state of material during aging (compacted specimen vs. loose mix) and (b) pressure level (oven aging vs. pressurized aging). Thus, the discussion of the laboratory aging of asphalt mixtures will focus on these two factors.

1.1. *Compacted Specimen Aging vs. Loose Mixture Aging*

1.1.1. Compacted Specimen Aging

Aging compacted samples is helpful for characterizing the performance of asphalt mixtures throughout the service life of the pavement. The standard method used to assess the long-term aging of asphalt mixtures in the United States is AASHTO R30 (2002). In this method, compacted asphalt mixture specimens are subjected to short-term aging at 135°C for four hours. Then, the short-term aged mixture is compacted. The compacted specimen is cored or sawn to the required dimensions and then placed in a forced draft oven for long-term aging. The long-term aging simulation is performed at 85°C ± 3°C for

120 ± 0.5 hours to represent five to ten years of aging in the field. Potential limitations associated with this aging method can be summarized as follows (Harrigan 2007):

- Although the mean air temperature in the United States varies with geographic region, AASHTO R30 specifies only one aging temperature to represent the long-term aging spectra.
- The aging time range recommended by this standard is too wide for design purposes, and simulation of aging at any time beyond ten years is not possible.
- The standard does not consider the effect of air void content, which is speculated to have a significant effect on oxidative aging.

As part of a SHRP project, studies were performed on the short-term and long-term aging of asphalt mixtures in the laboratory (Bell 1989, Bell et al. 1994). The outcome of these studies was standardized as AASHTO R30 for the long-term aging of compacted asphalt specimens in the laboratory. Bell et al. (1994) conducted different experiments on compacted specimens during SHRP-A-390 project. They proposed oven aging of loose mixtures at 135°C for four hours to simulate short-term aging during the mixing, storage and placement processes that take place in actual construction practice. In order to simulate long-term aging, Bell et al. conditioned asphalt specimens in the oven for different lengths of time. They conducted performance tests on laboratory long-term aged specimens and corresponding field cores. Based on the results of these trials, their recommendation for the long-term aging of compacted specimens was to age specimens at 85°C for 2, 4, and 8 days in the oven. A detailed discussion of the SHRP project trials is presented later in this document.

Brown and Scholz (2000) conducted a laboratory aging study that was similar to the Bell et al. study (1994) in that Brown and Scholz aged compacted specimens in an oven. Short-term and long-term aged specimens were evaluated based on the stiffness measurements of the compacted mixture specimens. The Brown and Scholz study results indicate that short-term aging of loose mix specimens at 135°C significantly increases the stiffness of the mixture by 9% to 24% per hour of aging. They speculated that for continuously-graded mixtures, oven aging at 135°C for two hours is representative of the aging during the mixing, storage and transport that occur during actual construction. Brown and Scholz compared the stiffness modulus ratios for laboratory-aged specimens and field cores and concluded that five days of aging compacted samples at 85°C in the oven gives reasonable results for long-term aging in the United Kingdom. They also found that four days of aging compacted samples at 85°C in the oven represents 15 years of aging in the field for mixtures in the United States (Brown and Scholz 2000).

Several other aging procedures for compacted mixture specimens have been proposed in the literature. A summary of these methods is provided in Table 1. It can be seen that the procedures vary with respect to temperature and duration, and most of the studies do not include field validation, which would provide an estimate of the level of field aging that the procedure simulates.

Table 1. Accelerated Laboratory Aging Procedures Developed for Compacted Asphalt Specimens

Aging Procedure	Temperature (°C)	Duration	Aging Equipment
AAMAS (Von Quintus et al. 1992)	2 days at 60 + 5 days at 107	7 days	Oven
TRL (Nicholls 2006)	60	48 hours	Oven
EMPA (Van den Bergh 2011)	110-120	16 hours	Oven
AASHTO R30-02 (Bell et al. 1994)	85	5 days	Oven
Nottingham University SATS (Collop et al. 2004)	85	65 hours	Air-drafted oven (saturated, 2.1 MPa)
Hayicha et al. (2003)	60	20 days	Oven
Tia et al. (1988)	60	90 days	Oven with and without UV radiation
Liverpool University	60	21 days	Oven
Mugler (1970)	163	5 hours	Oven
Hveem et al. (1963)	60	1000 hours	Oven
Martin et al. (2003)	3 days at 70 + 4 days at 80	7 days	PAV

Several problems associated with the laboratory aging of compacted specimens have been cited in the literature. Slump can occur during the long-term oven aging (LTOA) of compacted asphalt specimens, leading to a change in air void distribution (Reed 2010). Any performance results confounded by air void or volume changes cannot be trusted. To minimize slump, NCHRP 9-23 (2005) suggests wrapping specimens in a wire mesh held in place by three steel clamps to preserve the specimen from slump. However, even with the wire mesh, slump may occur and needs to be considered in this study. Another problem associated with aging compacted specimens is the development of an aging gradient through the specimen thickness. The NCHRP 9-23 project (2005) demonstrated that LTOA leads to both a radial and vertical age gradient in mixture specimens. RILEM-206-ATB indicates that changes in the size and shape of compacted samples during the aging process affect the final results and their variability (Partl et al. 2013). Therefore, selecting an appropriate sample size may help to eliminate the problems associated with the aging gradient in the mixture specimen.

A recent aging study conducted by North Carolina State University (NCSU) and the Western Research Institute (WRI) clearly demonstrates the problems associated with aging compacted specimens for performance testing. NCSU prepared and aged compacted specimens according to AASHTO R30, and the WRI evaluated the extent of aging at the surface and interior of the compacted specimens using a micro-extraction method. Prior to compaction, loose mix was short-term aged in a forced draft oven at 135°C for four hours. The long-term aging was performed in a forced draft oven at 85°C. The effects of the short- and long-term laboratory aging were measured by first splitting open a short-term aged specimen and an 8-day long-term aged specimen and removing small samples of mastic from the edge and center of the open face of each core. Asphalt from the small mastic samples was recovered and evaluated using infrared (IR) spectrometry. Figure 1 shows the extraction locations and moduli values estimated from different locations at different aging levels.

The IR spectra showed an increase in carbonyl at the center of the 8-day aged specimen compared to the center of the short-term aged specimen, which suggests that significant aging occurred during the 8-day oven aging period. Also, a carbonyl gradient from the center to the edge in the 8-day aged specimen was apparent. The observed differences in carbonyl content were further quantified by estimating the dynamic shear modulus, $|G^*|$ (at 10 rad/s and at 60°C). The asphalt at the center of the short-term aged specimen had a dynamic shear modulus value of 10.6 kPa. The dynamic shear modulus values at the center and edge of the long-term aged specimen were 17.0 kPa and 27.5 kPa, respectively. The dynamic shear modulus value of the pressurized aging vessel (PAV) -aged asphalt that was used to prepare the compacted specimens was 14.7 kPa, which is somewhat lower than the dynamic shear modulus value at the center of the long-term aged specimen, thereby suggesting that the 8-day oven aging effect is more severe than that of the standard PAV test.
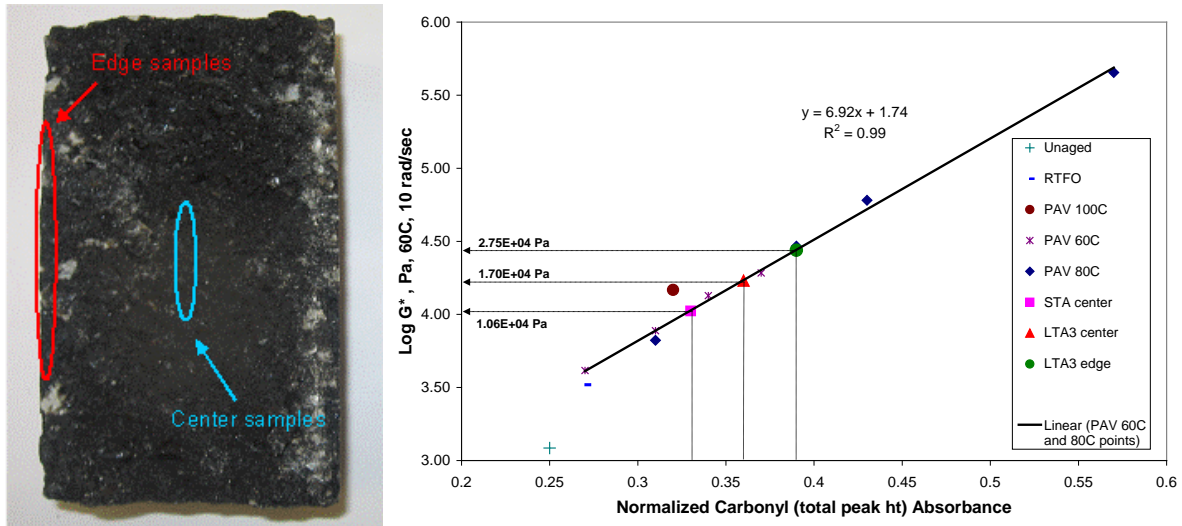
Figure 1. (a) Split core face showing locations of extraction and (b) estimated modulus values based on carbonyl content at different locations and for long-term oven-aged versus short-term oven-aged specimens.

The aging gradient observed from the aged specimens using the AASHTO R30 method makes it difficult for the general approach to be used for conditioning specimens for performance testing. The performance properties measured from the aged specimens will be the 'average' properties of the materials that have a binder modulus value difference of almost a factor of two.

1.1.2. Loose Mixture Aging

Although less common than aging compacted specimens, the laboratory aging of loose (uncompacted) asphalt mixtures also has been tried. Some studies recommend aging loose mixtures in the laboratory to simulate the aging of asphalt pavements instead of aging compacted specimens (Mollenhauer and Mouillet 2011, Van den Bergh 2011). The primary advantages of loose mixture aging over compacted specimen aging are: (1) air and heat can easily circulate inside the loose asphalt mixture, which allows for uniform aging throughout the mix; (2) problems associated with the loss of compacted specimen integrity (e.g., slump) during laboratory aging may be reduced; and (3) the rate of oxidation may increase due to a

larger area of the binder surface being exposed to oxygen. However, difficulties associated with the compaction of aged loose mixtures remains a major concern.

A review of studies that consider loose mixture aging is provided herein. The majority of the procedures proposed for loose mix aging utilize forced draft ovens at constant temperatures. However, a few procedures also consider using the binder PAV. It should be noted that the goal of a significant portion of the laboratory aging studies conducted on loose mixtures is to prepare reclaimed asphalt pavement (RAP) materials rather than to investigate compaction and subsequent performance testing. The compaction of aged materials is unnecessary for the preparation of RAP materials and, hence, any difficulty associated with highly aged loose mixtures is not a problem.

Von Quintus (1988) aged loose mixture specimens at 135°C in a forced draft oven for 8, 16, 24, and 36 hours to simulate long-term field oxidation. Von Quintus then conducted penetration and viscosity tests on the extracted and recovered asphalt binders. The results were promising, indicating similar short-term aging levels between the laboratory-aged materials and field samples taken from batching plants. However, the laboratory-aged materials exhibited considerable variability. Based on these results, Von Quintus recommended short-term aging of loose mixtures at 135°C in a forced draft oven for four hours before compaction. He also performed a study on the long-term aging of compacted asphalt mixtures in both an oven and in a pressurized oxidation vessel. The long-term aging procedures will be explained under the *Pressure Aging* section in this document.

Van den Bergh (2009) conducted an experimental program for aging loose mixes in the laboratory with the goal of replicating RAP material. In this study, several mixtures were short-term aged in an air-ventilated oven at 130°C for three hours and then long-term aged at 90°C for extended times. Binder aging was evaluated by determining the increase in the ring and ball (R&B) softening point, the dynamic shear modulus, and the carbonyl and sulfoxide indices. For the carbonyl and sulfoxide indices, a plateau was reached after seven days of long-term oven aging, but the rheological properties did not reach such a plateau after seven days. Then, these results were compared with the ones for binders extracted from seven- to ten-year-old pavement sections (Van den Bergh 2011). Based on this comparison, two procedures for producing RAP materials using a standard forced draft oven in the laboratory were recommended, as follows:

- Short-term aging at 130°C for 3 hours following long-term aging at 90°C for 168 hours (7 days).
- Short-term aging at 134°C for 4 hours following long-term aging at 85°C for 168 hours.

Because the goal of the Van den Bergh study was to produce RAP material, the compaction of the aged loose mixtures was not attempted.

Mollenhauer and Mouillet (2011) also conducted a study on the aging of loose mixtures to produce RAP materials. The properties of the binders extracted from laboratory-aged mixtures were compared with the properties of binders extracted from 11- to 12-year-old sections. The results suggest that aging asphalt loose mixtures in the PAV at 90°C with a pressure level of 2.1 MPa for 20 hours gives a comparable aging level with the oven aging of loose mixtures at 85°C for nine days. These researchers concluded that the aging time can be significantly reduced by using oxygen pressure. A summary of the comparison of the properties of the recovered binders for each aging method is shown in Table 2. Mollenhauer and

Mouillet (2011) also reported that aging at a low temperature (i.e., 60°C) is safer for mixtures that contain polymer-modified asphalt binder and produces less scattered results.

Table 2. Properties of the binders extracted and recovered after different aging methods (after Mollenhauer and Mouillet 2011)

Binder property	Aging method (temperature)	Pure binder (-1)	After mixing (0)	LTA 1 day (1)	LTA 3 days (3)	LTA 6 days (6)	LTA 9 days (9)	LTA 14 days (14)
Penetration (25 °C) [1/10 mm]	BRRC (60 °C)	36	28	28	27	-	26	23
	RILEM (85 °C)		28	-	25	23	20	-
	UV (60 °C)		27	-	-	-	-	22
	PAV (90 °C)		28	20	-	-	-	-
	PAV (100 °C)		25	-	-	2	-	-
Softening temp. $T_{R\&B}$ [°C]	BRRC (60 °C)	62	67,2	67,4	68,0	-	68,6	69,4
	RILEM (85 °C)		68,6	-	70,8	72,4	73,6	-
	UV (60 °C)		68,8	-	-	-	-	73,8
	PAV (90 °C)		66,8	72,4	-	-	-	-
	PAV (100 °C)		68,6	-	-	122	-	-

Partl et al. (2013) conducted a study (RILEM 206-ATB) with the goal of reproducing RAP via the laboratory oven aging of loose mixture specimens. Asphalt mixtures from the same component materials were produced in nine laboratories and a batching plant. A comprehensive study was performed on the binders recovered from long-term aged plant-produced and laboratory-produced mixtures. The laboratory-produced mixtures first were short-term aged for four hours at 135°C and compared with the plant-produced mixtures. The penetration and R&B softening point results obtained from the laboratory-aged mixtures showed a strong correlation with those from the plant-produced mixtures, but the plant-produced mixtures had higher dynamic shear modulus values and lower phase angles than the short-term aged laboratory-produced mixtures. The long-term aging process consisted of conditioning both the plant-produced and short-term laboratory-produced loose mixtures in the oven for 1, 2, 5, 7, and 9 days at 85°C.

The results of the Partl et al. (2013) study suggest that binder oxidation continues up to nine days of conditioning, but that the rate of oxidation decreases with an increase in the duration of the conditioning. Another significant finding is that performing extraction and recovery immediately following the laboratory aging process is important, because the binders that were extracted and recovered after a considerable amount of time were stiffer than those recovered immediately after aging. The Partl study also found that these laboratory aging procedures produce reasonable repeatability and reproducibility. The authors concluded that loose mixture aging in the laboratory to produce RAP material is an appropriate approach. Partl et al. (2013) cautioned, "However, with such a method, directly determining the mixture characteristics afterwards is not possible." Despite this comprehensive study of long-term aging of loose mixtures in the laboratory, the research team did not validate the field results.

Reed (2010) compared laboratory-aged loose mixtures to compacted specimens produced with crumb rubber-modified asphalt. Both the loose and compacted mixture samples were conditioned at 85°C for 5 and 14 days. Loose mixture aging was conducted in pans with sample heights ranging from 50 mm to 100 mm. The samples were agitated daily. Following aging, the loose mixtures were compacted and cored for testing. A significantly higher number of gyrations and higher shear stress levels were required to

compact the aged loose mix comparing to the short-term aged loose mix, suggesting that aged loose material is stiffer than aged core materials. Reed also found, however, that the dynamic modulus values of the compacted specimens following loose mix aging decreased with aging time, which is counter-intuitive. Reed (2010) concluded that the apparent loss in dynamic modulus value with an increase in aging time was caused by a lack of cohesion between the aggregate particles in the specimens that were compacted following loose mixture aging. He hypothesized that this outcome was influenced by the uniform aging of the asphalt around each aggregate particle in the laboratory-aged loose mix, as opposed to the field- and laboratory-aged compacted specimens that exhibit an aging gradient throughout the binder film (Figure 2). Reed also reported significant changes in air void content despite the miniscule dimensional changes during the long-term aging of the compacted specimens.
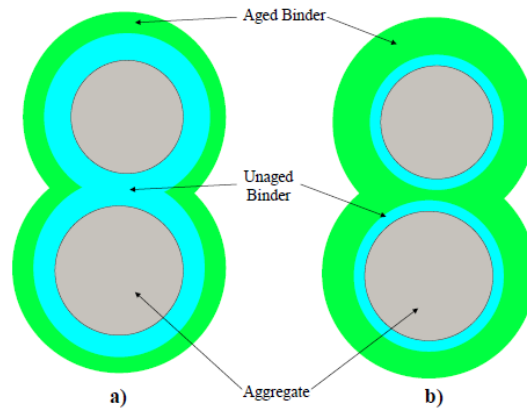
Figure 2. Schematic of binder film on aggregate particles: (a) core and (b) loose mix (after Reed 2010).

A summary of loose mix aging procedures is presented in Table 3. It should be noted that most of the studies on loose mixture aging were conducted by European researchers.

Table 3. Accelerated Laboratory Aging Procedures Developed for Loose Mixtures

Aging Procedure	Short-term Aging		Long-term Aging		Aging Equipment
	Temp. (°C)	Duration	Temp. (°C)	Duration	
Van Gooswilligen (1989)	-	-	160	16 hours	Loose hot mix in sealed tin in oven
Shell (Read and Whiteoak 2003)	Mixing Temp.	2 h	80	7 days	Oven
LCPC (Such et al. 1997)	135	4 h	100	24 h	Oven
RILEM TG5 (De la Roche et al. 2009)	135	4 h	85	7-9 days	Oven
BRRC (Belgian Road Research Center) (Piérard and Vanelstraete 2009)	135	1.5 h	60	14 days	Oven
Re-Road (Mollenhauer and Mouillet 2011)	135	4 h	90	20 h	PAV
Van den Bergh (2011)	135,130	4 h, 3 h	85, 90	7 days	Oven

Based on the results found in the literature, loose mixture aging appears to be advantageous over compacted specimen aging due to the homogeneity and efficiency of aging. However, for performance testing purposes, the aged loose mix must be compacted to the specified requirements. Previous studies of the long-term aging of loose mixtures indicate that much more effort is required to compact long-term aged loose mixtures than un-aged/short-term aged loose mixtures (Reed 2010). Very high compaction forces may cause degradation in the aggregate structure and change the mixture properties (Gatchalian et al. 2006).

Thus, compacting a highly aged loose mixture in the laboratory is problematic for developing a long-term aging procedure using loose material. A very stiff film of binder will lead to less cohesion in the compacted mix (Reed 2010), which may confound the performance test results. To avoid problems associated with compacting aged loose mixes, it has been recommended that specimens should be compacted at equiviscous temperatures based on the viscosity of the short-term aged mix (Bell 1994). This method requires obtaining the viscosity of the binder in the short-term aged and long-term aged loose mixtures to determine the temperatures required for an equivalent binder viscosity for two different aging levels. It is expected that very high (approximately 370°F) temperatures will be required for the long-term aged material to obtain the same viscosity as the reference short-term aged material. Such high temperatures can lead to emission and practicality problems in the laboratory.

The use of a warm mix additive that can allow the mixing and compaction temperature to be reduced may offer an alternative to increasing the compaction temperature. Aspha-min® zeolite is a product that can reduce the production temperature by as much as 20% without affecting mixture performance. Zeolite contains 21% water by mass that is released in the temperature range of 85°C to 182°C (185°F-360°F). A study conducted by the National Center for Asphalt Technology (NCAT) shows that adding 0.3% zeolite improves the compactability of the mixture at temperatures as low as 88°C (190°F) without sacrificing the resilient modulus and rutting resistance. This product also was used in a mix containing 20% RAP and reduced the production and compaction temperature by 19°C (35°F) while offering the same in-place density and performance. During the study, it was noted that an average 0.65% reduction in air void content occurred when zeolite was used in the asphalt mix production (Hurley and Prowell 2005).

NCHRP 9-39 to Determine Accurate Temperature for Mixing and Compaction of Hot Asphalt Mixtures (West et al. 2010)

West et al. (2010) conducted a study as part of the NCHRP 9-39 project to introduce a procedure that can determine appropriate mixing and compaction temperatures for asphalt mixtures that contain both modified and unmodified asphalt binders. Three candidate methods were evaluated in the laboratory:

(1) Candidate Method A is designed to determine the equiviscosity temperature at high shear rates, as recommended by Yildirim et al. (2000). This method is based on the fact that most modified asphalt binders exhibit shear thinning behavior. To determine the equiviscosity temperature required for compaction, the viscosity was measured in a rotational viscometer at shear rates ranging from 0.1 s[-1] to 93 s[-1] and at temperatures ranging from135°C to 165°C. The viscosity rate was fitted to the Cross-William model, and extrapolation then was used to estimate the temperatures at which the viscosity at a shear rate of 500 s[-1] is equal to 0.17 ± 0.02 Pa/s (for mixing) and 0.28 ± 0.03 Pa/s (for compaction) (Yildirim et al. 2000).

(2) Candidate Method B also is based on the equiviscosity concept but differs from Candidate Method A in that the viscosity is measured using a dynamic shear rheometer (DSR) rather than the rotational viscometer. This approach is based on the method developed by Reinke (2003). The approach uses the viscosity data measured by a DSR with 25-mm parallel plate geometry and a 500-micron gap. The viscosities of the binders are measured in constant shear mode over a range of shear stress levels at temperatures ranging from 76°C to 94°C. Steady-state viscosity of the modified binders was observed at shear rates as high as 500 s^{-1}. The viscosity results at 500 Pa shear were extrapolated to 180°C using a log viscosity versus log temperature chart. The temperature that corresponded to a viscosity of 0.17 ± 0.002 Pa.s was selected for mixing, as in Method A. However, Reinke recommended determining the temperature for compaction based on the temperature that corresponded to a viscosity of 0.35 ± 0.03 Pa.s (Reinke 2003).

(3) Candidate Method C consists of the so-called Casola method (Casola 2008). The Casola method is based on an equiphase angle concept rather than on equiviscosity. In this method, a DSR with 25-mm parallel plate geometry and 1-mm gap is used. Frequency sweep testing with frequencies ranging from 0.001 rad/s to 100 rad/s was used to generate a phase angle versus frequency mastercurve at a reference temperature of 80°C. "The binder's transition point from viscous behavior to viscoelastic behavior was represented by the Phase Angle of 86°. The frequency corresponding to this transition point was correlated to the temperatures where binders provide good aggregate coating during mixing and lubrication during compaction" (Casola 2008).

The NCAT study incorporated laboratory testing to assess the three candidate methods in order to determine appropriate mixing and compaction temperatures and also to determine the emissions potential and binder degradation at high temperatures using modified and unmodified binders. In addition to evaluating the effects of temperature on the coating of aggregate particles during mixing, the study also assessed the workability of the asphalt mixture, compactability, and properties of the asphalt mixtures at low temperatures. The following findings were obtained from the NCAT study.

Method A resulted in mix temperatures above 177°C for all the mixtures that contained modified asphalt binders; these temperatures are considered to be too high in the asphalt paving industry. On the other hand, the results obtained from the high shear rate viscosity method were very similar to the ones obtained from the traditional equiviscous method that applied a Brookfield rotational viscometer at a shear rate as low as 6.8 s^{-1} (AASHTO T 316). These results suggest that no improvements to the current procedure are resulted from Method A. Table 4 shows some typical results of the measurements performed in this study.

Table 4. Comparison between the mixing and compaction temperatures for the high shear rate viscosity method and traditional equiviscous method (after West et al. 2010)

Binder ID	True Grade	Mixing Temperature °F (°C)		Compaction Temperature °F (°C)	
		Equiviscous Method	High Shear Rate Viscosity	Equiviscous Method	High Shear Rate Viscosity
M	**85.5 -19.5**	**372 (189)**	**363 (184)**	**343 (173)**	**336 (169)**
N	**84.3 -25.5**	**433 (223)**	**433 (223)**	**401 (205)**	**401 (205)**
G	**82.5 -24.2**	**379 (193)**	**372 (189)**	**352 (178)**	**349 (176)**
H	**78.3 -26.1**	**365 (185)**	**363 (184)**	**338 (170)**	**338 (170)**
C	**75.1 -38.7**	**388 (198)**	**385 (196)**	**355 (179)**	**352 (178)**
I	71.8 -29.2	333 (167)	333 (167)	311 (155)	311 (155)
B	**69.3 -37.3**	**354 (179)**	**352 (178)**	**325 (163)**	**325 (163)**
F	67.8 -21.3	320 (160)	318 (159)	298 (148)	297 (147)
O	65.6 -29.7	318 (159)	318 (159)	293 (145)	297 (147)
K	65.3 -13.0	295 (146)	295 (146)	271 (132)	275 (135)
J	64.3 -20.7	295 (146)	295 (146)	275 (135)	273 (134)
E	60.9 -33.1	293 (145)	293 (145)	273 (134)	297 (147)
D	60.3 -31.7	295 (146)	297 (147)	275 (135)	279 (137)

Method B resulted in lower mixing and compaction temperatures compared to the traditional equiviscosity method for both the modified and unmodified binders. These results also demonstrate that the temperature difference between Methods A and B is greater for the modified binders than the unmodified binders, thereby confirming their shear thinning behavior (see Table 5).

Table 5. Comparison between the mixing and compaction temperatures for the steady shear flow viscosity method and traditional equiviscous method (after West et al. 2010)

Binder ID	True Grade	Mixing Temperature °F (°C)		Compaction Temperature °F (°C)	
		Equiviscous Method	Steady Shear Flow Viscosity	Equiviscous Method	Steady Shear Flow Viscosity
M	**85.5 -19.5**	**372 (189)**	**296 (147)**	**343 (173)**	**275 (135)**
N	**84.3 -25.5**	**433 (223)**	**337 (169)**	**401 (205)**	**311 (155)**
G	**82.5 -24.2**	**379 (193)**	**340 (171)**	**352 (178)**	**312 (156)**
H	**78.3 -26.1**	**365 (185)**	**333 (167)**	**338 (170)**	**304 (151)**
C	**75.1 -38.7**	**388 (198)**	**320 (160)**	**355 (179)**	**291 (144)**
I	71.8 -29.2	333 (167)	316 (158)	311 (155)	289 (143)
B	**69.3 -37.3**	**354 (179)**	**325 (163)**	**325 (163)**	**295 (146)**
F	67.8 -21.3	320 (160)	309 (154)	298 (148)	281 (138)
O	65.6 -29.7	318 (159)	309 (154)	293 (145)	280 (138)
K	65.3 -13.0	295 (146)	280 (138)	271 (132)	257 (125)
J	64.3 -20.7	295 (146)	289 (143)	275 (135)	263 (128)
E	60.9 -33.1	293 (145)	293 (145)	273 (134)	269 (132)
D	60.3 -31.7	295 (146)	289 (143)	275 (135)	262 (128)

Method C yielded lower mixing and compaction temperatures than the equiviscosity method for the modified binders. However, for the unmodified binders, in some cases Method C resulted in lower temperatures and in some cases higher temperatures than the equiviscosity method (see Table 6).

Table 6. Comparison between the mixing and compaction temperatures for the phase angle method and traditional equiviscous method (after West et al. 2010)

Binder ID	True Grade	Freq. at δ =86° T=80°C	Mixing Temp. °F (°C)		Compaction Temp. °F (°C)	
			Equiviscous Method	Phase Angle Method	Equiviscous Method	Phase Angle Method
M	85.5 -19.5	0.07	372 (189)	337 (169)	343 (173)	310 (154)
N	84.3 -25.5	0.03	433 (223)	341 (172)	401 (205)	313 (156)
G	82.5 -24.2	0.03	379 (193)	341 (172)	352 (178)	313 (156)
H	78.3 -26.1	0.22	365 (185)	332 (167)	338 (170)	305 (152)
C	75.1 -38.7	0.21	388 (198)	332 (167)	355 (179)	306 (152)
I	71.8 -29.2	2.98	333 (167)	320 (160)	311 (155)	296 (147)
B	69.3 -37.3	1.10	354 (179)	325 (163)	325 (163)	300 (149)
F	67.8 -21.3	75.00	320 (160)	307 (153)	298 (148)	285 (141)
O	65.6 -29.7	21.12	318 (159)	312 (156)	293 (145)	289 (143)
K	65.3 -13.0	800	295 (146)	297 (147)	271 (132)	277 (136)
J	64.3 -20.7	580	295 (146)	298 (148)	275 (135)	278 (137)
E	60.9 -33.1	37.85	293 (145)	309 (154)	273 (134)	287 (142)
D	60.3 -31.7	122.56	295 (146)	305 (152)	275 (135)	283 (139)

The NCAT study concluded that both Methods B and C provide a reasonable way to determine the temperatures for mixing and compacting mixtures, and that both candidate methods are applicable to both unmodified and modified asphalts.

1.2. *Oven Aging vs. Pressure Aging*

Two general types of aging procedures have been used to age asphalt mixtures in a laboratory setting: long-term oven aging and pressurized aging. Each type of procedure has associated advantages and disadvantages that are discussed in this report. In addition, the operating parameters for each type of procedure differ among researchers and, hence, these parameters are reviewed in the following sections.

1.2.1. Oven Aging

Long-term oven aging (LTOA) is the most common method that is used to simulate oxidative aging of asphalt mixtures in the laboratory. As discussed, the current standard procedure, AASHTO R30, consists of conditioning compacted asphalt concrete specimens in an oven at 85°C for five days. However, other oven aging procedures have been tried and are cited in the literature. Although they are somewhat similar in terms of methodology, the LTOA procedures differ in terms of temperature and duration (e.g., Bell et al. 1994, Houston et al. 2005, Reed 2010).

The SHRP project conducted an extensive evaluation of long-term aging procedures for asphalt mixtures (Bell 1989, Bell et al. 1994). LTOA was conducted at both 85°C and 100°C. The results for LTOA at 100°C were similar to those at 85°C, but the same level of hardening was achieved in less time. It was reported that the modulus measurements for most of the laboratory-aged specimens that were subjected to eight days of aging at 85°C and four days at 100°C were comparable. However, greater specimen-to-specimen variability was observed at 100°C and, therefore, the research team suggested that 85°C is preferable. Based on the field validation, Bell et al. (1994) recommended the LTOA of compacted specimens at 85°C for 2, 4, and 8 days to simulate long-term aging. The resilient modulus values for the laboratory- and field-aged cores were compared to determine the extent of aging that the laboratory procedures simulated. The resilient modulus test results demonstrated good agreement between the oxidation levels of the asphalt mixtures that were aged in the field for 14 to 19 years and the mixtures that were laboratory aged for 4 to 8 days (Bell 1994). However, Bell cautioned that in some cases, the LTOA method leads to less aging than that observed in the field. In addition, it was reported that due to some undefined factors (e.g., traffic and moisture-induced cracks) no correlation could be made between the measured modulus values of the field cores and the corresponding laboratory-aged samples. Also, no comparison was made between the binder properties of the laboratory- and field-aged mixtures.

According to Petersen (1989), aging can develop at higher temperatures for a longer time and to a higher level than at lower temperatures. Therefore, Petersen concluded that at higher temperatures, the plateau region for the aging index is reached after a longer time and at higher levels compared to lower aging temperatures (Figure 3). Bell (1989) conducted a study on the possible effects of different aging methods. He speculated that very high temperatures can cause severe aging in asphalt mixtures and may exceed the field aging level (Figure 3), which is important for evaluating oven aging methods (Bell 1989).
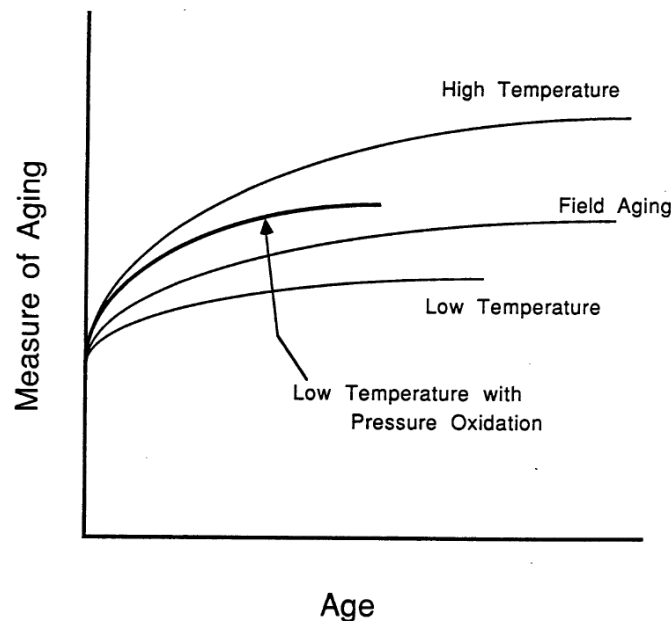
Figure 3. Possible interactive effects of temperature and pressure oxidation on aging (after Bell 1989)

As discussed, increasing the temperature of LTOA conditioning expedites aging significantly. Based on the Re-Road study conducted for producing RAP mixtures in the laboratory, conditioning loose mixture at 85°C for nine days was found to cause more severe aging than conditioning at 60°C for 14 days (the BRRC aging method), but with more scattered results (Mollenhauer and Mouillet, 2011). In addition, applying elevated temperatures as high as 100°C can expedite the aging process, but such high temperatures may cause degradation of the binders and may damage the compacted specimens (Partl et al. 2013, Mollenhauer and Mouillet 2011, Bell and Sosnovske 1994, Bell et al. 1994). Therefore, numerous aging studies recommend 85°C for aging either loose or compacted asphalt mixtures, although they may suggest different aging durations. (Tables 1 and 2 provide summaries of the oven aging procedures found in the literature.)

The summaries of compacted and loose mixture aging trials presented in Tables 1 and 2 demonstrate that LTOA requires considerable time to produce oxidation levels that correspond to field conditions near the end of a pavement's service life, thus providing the motivation to conduct trials of pressure aging to expedite the oxidation process. Despite the advantages offered by oven aging of availability, easier function, and higher capacity compared to a pressurized system, ovens differ with respect to their mechanisms of air drafting (Partl et al. 2013). This oven variability may impact the aging process of asphalt mixtures and, therefore, may contribute to a lack of repeatability between laboratories. On the other hand, PAVs are standardized equipment, thereby reducing problems associated with instrument variability.

1.2.2. Pressure Aging

As an alternative to the oven aging, pressure and forced air can be used to increase the rate of oxidation in asphalt mixture specimens. Pressure aging asphalt mixtures has been tried by several researchers for both compacted and loose mixture specimens.

Kumar and Goetz (1977) aged compacted specimens at 60°C using 'pulled air' at a constant water-head of 0.5 mm for durations varying between one and ten days. Figure 4 depicts the apparatus used in the Kumar study. Kumar evaluated the aged specimens using a nondestructive compressive test with creep loading at 21°C ± 2°C. The slope and intercept of the creep curve (creep deformation versus loading time) were used to indicate the oxidation progress. No comparisons were made between laboratory and field aging; however, the Kumar study results indicate that this method does not induce significant aging in the asphalt specimens within a reasonable period of time (i.e., 10 days). The Kumar study reports that specimen integrity was maintained during the aging procedure and recommends that oxygen be tried instead of air to increase the rate of oxidation in the test.
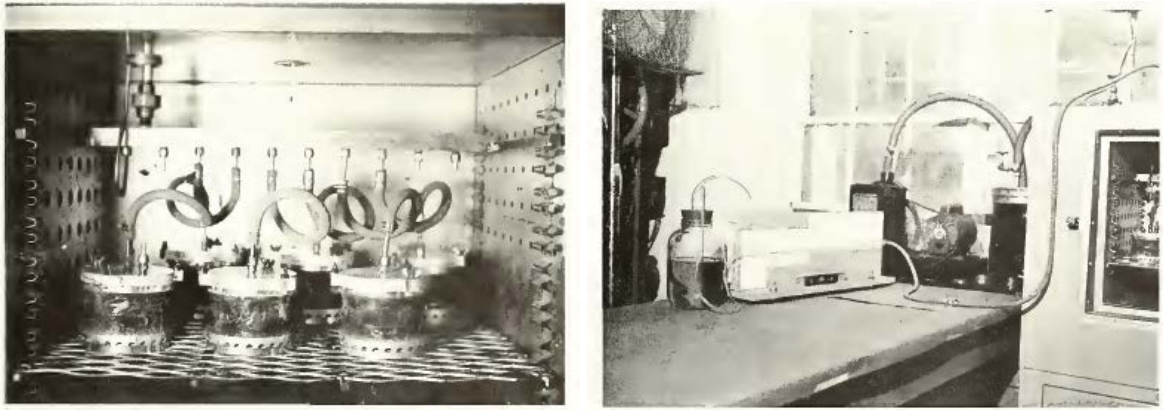
Figure 4. Kumar pressure aging procedure (after Kumar and Goetz1977)

Von Quintus et al. (1988) performed a study on LTOA and long-term pressure aging of compacted asphalt mixture specimens. The study consisted of LTOA compacted specimens at 60°C for two days following five days of conditioning at 107°C. Pressure aging was conducted at 60°C at 100 psi (690 kPa) for durations of five and ten days. Following aging, the indirect tensile strength of the aged specimens was measured and it was found that the oven-aged specimens had higher indirect tensile strength levels and lower failure strain levels than the pressure-aged samples. The authors attributed this finding to a higher aging level for the oven-aged specimens than for the pressure-aged samples. In addition, penetration and viscosity tests on the extracted and recovered binders demonstrated more severe aging in the LTOA procedure than from the pressure aging procedure. Hence, Von Quintas et al. (1988) recommended oven aging over pressure aging but also advised that a more comprehensive study and evaluation of pressure aging should be conducted.

In a similar study, Kim et al. (1986) applied pressure to laboratory-aged compacted asphalt mixture samples. Specimens were compacted to different levels of the original sample (i. e., 88%, 94%, and 100%) to study the effect of air void content on the extent of oxidation. Specimens were subjected to oxygen pressure at 100 psi (690 kPa) and at 60°C for 0, 1, 2, 3, and 5 days. The resilient modulus values and fatigue life data obtained from diametral testing were measured following the aging process. Modulus ratios (i.e., the modulus value of the aged mixture divided by the modulus value of the un-aged mixture) were compared for specimens aged for the various durations. The Kim et al. findings showed that the modulus ratios increased overall with aging, and the rate of increase was higher for the less compacted samples. Figure 5 depicts an example of measured modulus ratios for two different compaction levels. Interestingly, the modulus ratio decreases at the early aging levels. The authors attributed this outcome to the loss of cohesion in the specimens at 60°C, suggesting the potential loss of specimen integrity during the laboratory aging procedure. Figure 6 indicates that the fatigue life also increases with the aging level of the specimens.
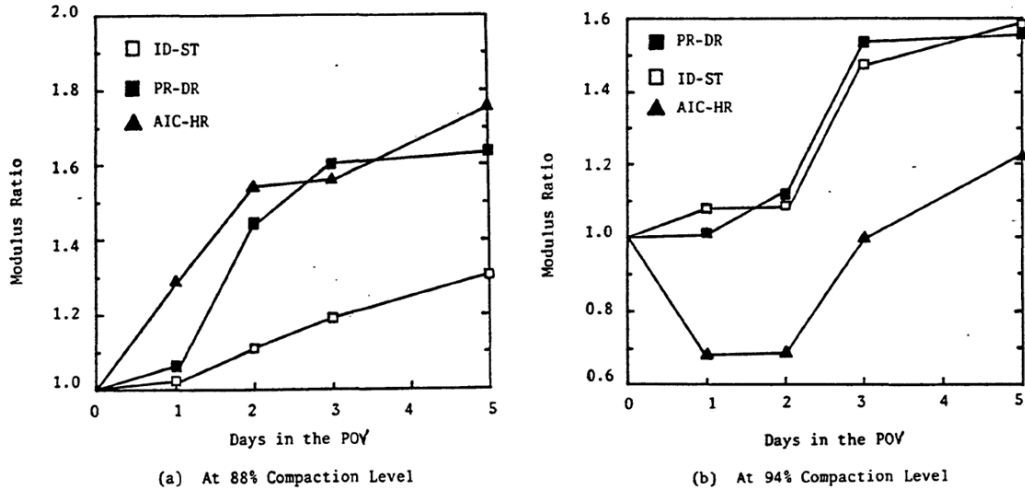
Figure 5. Aging modulus ratios for different compaction levels and three mixture types (after Kim et al. 1986)
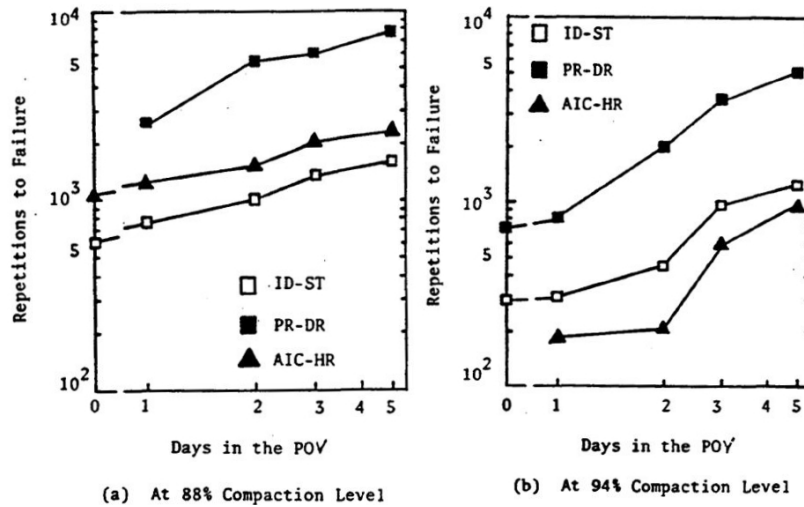
Figure 6. Fatigue life data for different compaction levels and three mixture types (after Kim et al. 1986)

Bell et al. (1994) evaluated several pressure aging systems as part of the SHRP project. they first tried 'pressure oxidation' of compacted specimens using several pressure/temperature combinations in a pressurized vessel. Compacted samples were exposed to air or oxygen for 0, 2, or 7 days at pressures of 690 kPa or 2070 kPa and at 25°C or 60°C. The results revealed an unexpected trend in the modulus of the pressure-oxidized specimens (e.g., see Figure 7). With an increase in the level of oxidation (air or oxygen pressure), the modulus value decreases. The authors attributed this finding to the destructive effect of relieving the pressure at the end of the test.

15
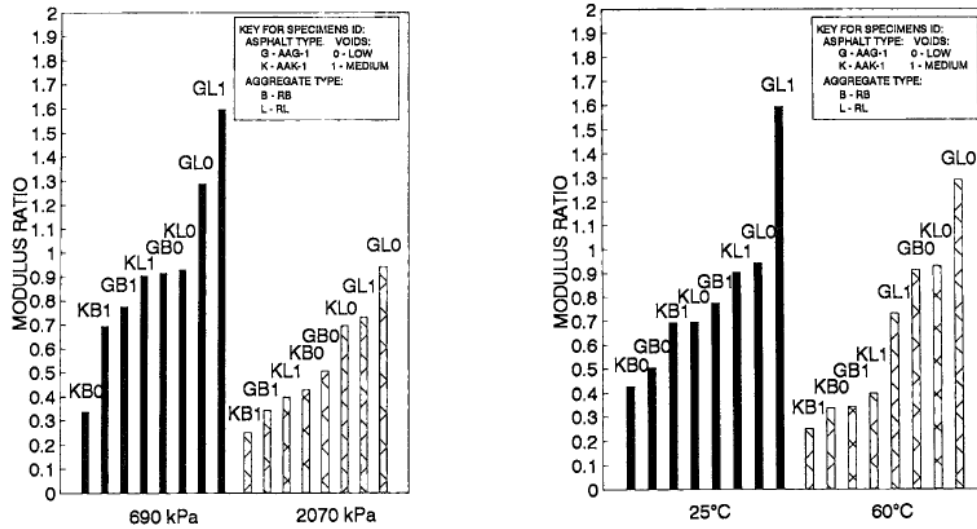
Figure 7. Effect of oxygen pressure and temperature on modulus ratio using the pressure oxidation method

This outcome prompted Bell et al. (1994) to switch to a different pressure oxidation procedure in which oxygen was passed through the asphalt specimens using a triaxial cell set-up and using a much lower pressure level than was used in previous trials. Although specimen integrity problems were reported for the pressure vessel tests, no further trials (with lower pressures or solutions for preserving the specimen during pressure release) were conducted to improve the test method, so Bell et al. switched the procedure from the pressure vessel set-up to the triaxial set-up depicted in Figure 8. The aging procedure included a flow rate of 0.11 m^3/hour (equivalent to roughly 50 psi (345 kPa)) at temperatures of both 25°C and 60°C for 1, 3, and 7 days. The resilient modulus was measured throughout conditioning in order to track the age hardening of the material. The authors concluded that the triaxial cell set-up appears "viable for realistic long-term oxidative aging, particularly since the mixture is 'supported' by a confining pressure throughout the aging process. It is also much safer than the pressure-oxidation approach, since the required pressure is much lower" (Bell et al. 1994). However, in comparing the modulus ratios for triaxial and LTOA, the latter method causes more severe aging in the compacted specimens. Note that these conclusions all were drawn from mixture test results. No tests were conducted on extracted binders, so the actual levels of oxidation achieved are unknown.

16
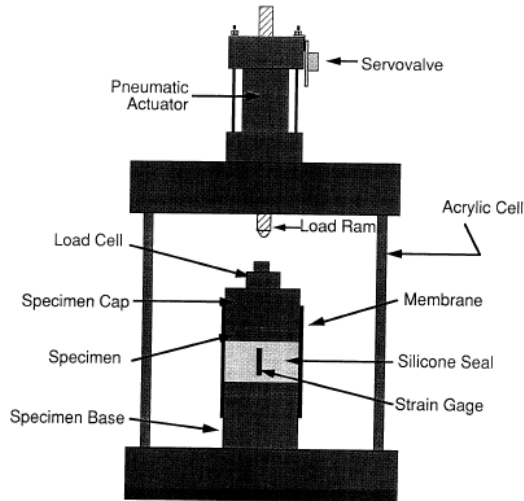
Figure 8. SHRP triaxial aging procedure (after Bell et al. 1994)

Table 7 presents the resilient modulus results for different test sections in the Bell et al. study (1994). The data indicate very severe effects of LTOA on the stiffness of the specimens, indicating that a high level of aging has occurred.

Table 7. Summary of long-term oven aging data (after Bell et al. 1994)

SAMPLE ID	ACTUAL VOIDS (%)	MODULUS (MPa)		MODULUS RATIO
		BEFORE AGING	AFTER AGING	
GLLL00O	7.38	1718	2029	1.18
GLLL10O	7.99	2346	6403	2.73
GLLL20O	7.42	2767	10674	3.86
GLLL01O	7.91	2422	2532	1.05
GLLL11O	7.87	2001	5969	2.98
GLLL21O	7.54	2270	10950	4.82
GLML01O	10.00	1615	2401	1.49
GLML11O	9.30	1656	4685	2.83
GLML21O	10.00	1780	7838	4.40
KLLL00O	6.69	2036	2263	1.11
KLLL10O	7.55	2125	3498	1.65
KLLL20O	7.23	2450	10992	4.49
KLML00O	9.20	1366	1656	1.21
KLML10O	9.50	1277	4340	3.40
KLML20O	9.10	1435	8867	6.18

Bell et al. (1994) concluded that the triaxial pressure test method provides reasonable aging results (modulus ratios). They also speculated that the aging level may be increased if 85°C is used instead of 60°C. The results of the SHRP-A-383 project are presented in Table 8.

Table 8. Summary of triaxial (oxygen) aging data (after Bell et al. 1994)

SAMPLE ID	AGING CONDITION		ACTUAL VOIDS (%)	MODULUS (MPa)		MODULUS RATIO
	PERIOD (day)	TEMP. (°C)		BEFORE AGING	AFTER AGING	
KLLL00TO	0	25	4.9	2670	2670	1.00
KLLL10TO	1	25	5.0	2870	3450	1.20
KLLL20TO	3	25	5.3	3374	3795	1.12
KLML01TO	0	60	7.3	2808	2808	1.00
KLML11TO	1	60	7.0	2843	2732	0.96
KLML21TO	3	60	7.9	2318	2967	1.28
KLML31TO	7	60	7.4	2187	3298	1.51
GLLL01TO	0	60	6.3	2905	2905	1.00
GLLL11TO	1	60	5.5	3153	3788	1.20
GLLL21TO	3	60	6.1	3174	4092	1.29
GLML00TO	0	25	7.1	2912	2912	1.00
GLML10TO	1	25	8.3	2125	2691	1.27
GLML20TO	3	25	8.1	2788	3360	1.21
GLML31TO	7	60	7.8	3091	5154	1.67
KBLL01TO	0	60	5.3	2305	2305	1.00
KBLL11TO	1	60	5.2	2415	3209	1.33
KBLL21TO	3	60	4.8	2332	3450	1.48
KBML00TO	0	25	7.8	1994	1994	1.00
KBML10TO	1	25	6.6	1932	1842	0.95
KBML20TO	3	25	7.4	2036	2208	1.08
KBML31TO	7	60	6.7	1960	3422	1.75

Korsgaard et al. (1996) aged compacted specimens in a standard binder PAV and found that mixture aging for 72 hours at 100°C and at 2.07 MPa leads to the same level of aging of extracted binders as standard PAV aging of binders. No problems associated with specimen integrity loss upon pressure release were noted.

Khalid and Walsh (2002) developed an accelerated pressure oven procedure for simulating the long-term aging of porous asphalt mixtures. This procedure forces air through compacted specimens at a rate of 3 L/min at 60°C. A rubber membrane is placed on the specimens to maintain air flow throughout the sample and minimize peripheral localization (Figure 9). This procedure is able to simulate aging to the same extent that AASHTO R30 specifications can simulate aging, although due to the low temperature used, a longer aging time (up to 25 days) was required for the Khalid and Walsh study than for the AASHTO guidelines.
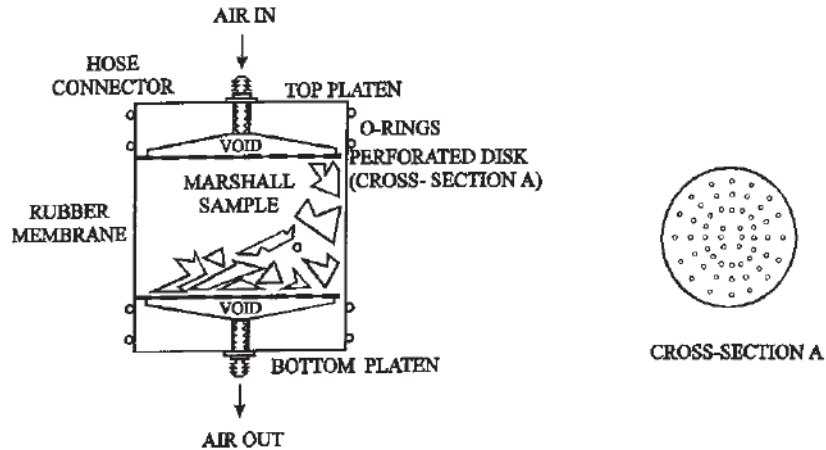
Figure 9. Khalid and Walsh pressure aging system (after Khalid and Walsh 2002)

The combined effect of oxidation and water conditioning on an asphalt mixture's performance was evaluated in a study conducted at the University of Nottingham (Airey 2003). Compacted specimens were exposed to moisture and 2.1 Mpa oxygen pressure at 85°C for 65 hours. As shown in Figure 10, the moisture conditioning was provided from condensed water dripping onto the specimen. The results showed similar rheological properties ($|G*|$ and δ) for recovered asphalt binders from both field- and laboratory-conditioned samples (Airey 2003). Nevertheless, the performance of the laboratory-conditioned mixture specimens differed from that of the field cores. It was speculated that the difference in performance of the two mixture types was caused mainly by water damage during aging. The destructive effect of water not only has an adverse effect on adhesion but also on the cohesion of the asphalt binder/asphalt mastic in the mixture (Hagos 2008, Airey 2003). Figure 10 shows the test set-up developed by Airey (2003) to determine the effects of both pressure oxidation and moisture on the performance of asphalt mixtures.
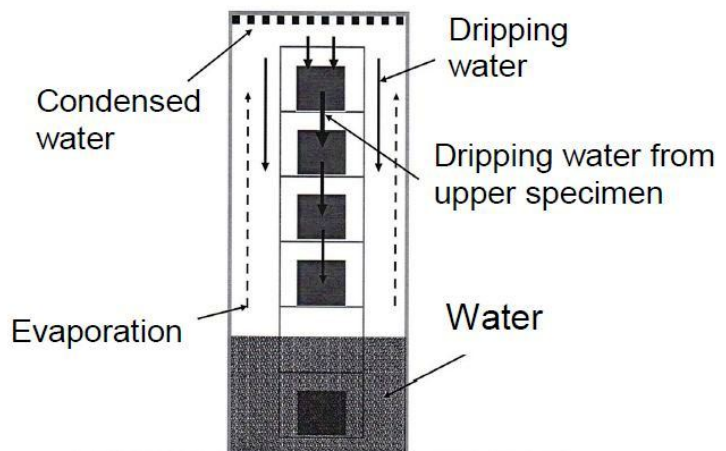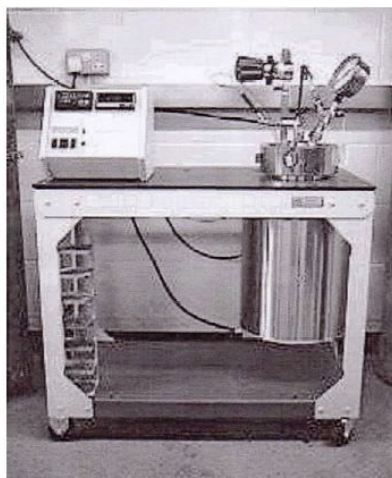
Figure 10. Test set-up for asphalt mixture pressure aging and moisture conditioning (after Airey 2003)

19

A study conducted by Hachiya et al. (2003) introduced an accelerated method for the long-term aging of asphalt concrete. The procedure consists of two stages: First, asphalt mixture beams were aged in an oven at 70°C for eight hours to induce aging due to heat; and second, for the actual aging process, the beams were placed in an oven filled with pure oxygen at 60°C for 20 days. The effect of aging on the different properties of the asphalt concrete beams was examined. Flexural tests were performed to determine the mechanical properties of the asphalt mixture, and penetration tests, the softening point and the Fraass breaking point were used to evaluate the mechanical properties of the recovered asphalt binders. The Hachiya et al. study findings suggest that oxygen aging may properly simulate aging in the field. These researchers also concluded that the top 5 mm of the surface layer experienced the most aging during the aging procedure and that aging decreases with the depth of the pavement (Hachiya et al. 2003).

Collop et al. (2004) developed a procedure to inspect the aging/moisture sensitivity of a compacted coated macadam binder course. Moisturized cored samples were subjected to 2.1 Mpa air pressure at 85°C. The mechanical properties (stiffness moduli) of the samples were compared before and after the aging process (Collop et al. 2004).

One of the main shortcomings of PAV aging compared to oven aging is the amount of material that can be aged simultaneously. According to previous studies on loose mixture aging, in order to obtain uniform aging, a uniform thin layer of loose mix should be placed in the PAV, which reduces the capacity of the instrument to a very low level (around 1 kilogram) (Partl et al. 2013). A simple calculation can show that several PAV aging tests must be performed to obtain enough aged loose mixture for one standard large compacted specimen. Considering the standard PAV capacity, for compacted specimens, just one large asphalt mixture core (Ø100 mm × 150 mm) can fit vertically inside the chamber. However, if a smaller geometry for asphalt mixture cores is used, more specimens can be aged at once. Despite this capacity problem, it is believed that the air/oxygen pressure in the PAV can expedite aging and reduce the required time to achieve a certain level of aging in the asphalt mixture specimen. However, concern remains with respect to maintaining compacted specimen integrity during pressure aging and upon pressure release, which needs to be considered carefully when assessing PAV aging of compacted mix specimens.

1.3. Summary

Generally, the advantages and disadvantages of using loose and compacted asphalt mixtures can be summarized as outlined in Table 9.

Table 9. Comparison between loose mix and compacted specimens in the aging procedure

Loose Mix	Pros:	• *Homogenous aging in the mixture* • *Higher oxidation rate than compacted mix* • *Maintenance of specimen integrity a non-issue*
	Cons:	• *Difficulties associated with compaction of aged loose mix, which limits use for producing specimens for performance testing* • *Limited amount of materials can be aged in standard PAV chamber*
Compacted Specimen	Pros:	• *Can produce aged sample for performance tests if slumping is minimized through use of wire mesh*
	Cons:	• *Slower oxidation rate than loose mix* • *Integrity of the specimens is compromised at high temperatures and pressures due to slump and cracking upon pressure release* • *Oxidation gradients exist radially and along height of the specimen*

The comparison between aging in the oven and in a pressure oxidation vessel (e.g., a PAV) is summarized in Table 10.

Table 10. Comparison between oven and PAV aging methods

Oven Aging	Pros:	• *Available and easy to perform and control* • *Large amount of material can be aged simultaneously*
	Cons:	• *High variability among ovens, especially in terms of air drafting* • *More time needed to age materials in the oven than in the PAV* • *Maintenance of compacted specimen integrity is required, especially at high temperatures*
Pressure Aging	Pros:	• *Pressure can expedite the aging process* • *More reliable than oven aging due to less instrument variability between laboratories*
	Cons:	• *Due to limited capacity of the vessel, less material can be aged in each aging cycle unless new device is developed* • *Integrity of compacted samples during and after testing is a major concern*

2. Modeling of Asphalt Binder Aging in Pavements

The current standard laboratory procedure for long-term oxidative aging of asphalt mixtures, AASHTO R30, specifies the use of a single time-temperature combination, which does not allow for consideration of various climatic conditions, pavement properties, and/or structure, which undoubtedly affect the extent of oxidation. Changes in pavement mechanical properties over time as a result of binder oxidation can affect the pavement response and, hence, can result in pavement distress. The goal of this research project is to develop a calibrated and validated procedure to simulate the long-term oxidative aging of asphalt mixtures in a pavement as a function of climate, depth and air void content. Therefore, understanding the nature of the oxidation process and consequently being able to model this process to predict the level of oxidation that occurs throughout the depth of the pavement as a function of age is essential. A model that can predict the extent of asphalt binder aging in pavements and consequently laboratory aging conditions as a function of climate, depth, and time will be developed in this study, either by refining existing models or by creating a new model.

A review of relevant information regarding the development of such a model is presented in the following sections. Key elements required for developing the aging model include binder aging index properties (AIPs), oxidation kinetics modeling, and diffusion modeling.

2.1. Aging Index Properties (AIPs)

Modeling the extent of oxidation in a pavement requires 'tracking' the property with respect to time, which is related directly to the oxidation level in the pavement. Asphalt binder is the asphalt mixture constituent that undergoes oxidative aging; hence, the oxidation of a pavement is best tracked using binder properties, because the mixture is subjected to other factors, including mechanical degradation that is caused by traffic loading, thermal-induced stress, and moisture, all of which could confound test results. In the following sections, the binder properties that are used to measure the extent of oxidation are referred to as *aging index properties* (AIPs).

Asphalt binder AIPs can be divided into two main categories: 1) chemical functional groups and 2) rheological parameters. The following sections discuss the AIPs that fall into these two categories. Chemical functional groups can be used to track the oxygen uptake of asphalt binders directly. Rheological properties are advantageous in that they do not require IR spectroscopy (Glaser et al. 2013). Underwood et al. (2010) presented laboratory aging data that suggest that the rheology and aging kinetics of asphalt binders are linked. An important advantage of the evolution of a model that is based on rheological parameters, such as the crossover modulus, as an AIP is its ability to relate to the mechanical properties of polymer-modified binders, whereas when using chemical functional groups as AIPs, the chemistry of the base binder oxidation and concurrent oxidation and degradation of the polymer is extremely complex and makes it more difficult to relate the model to mechanical properties.

2.1.1. Chemical Functional Groups

Fourier transform infrared (FTIR) spectroscopy can be used to analyze the chemical functional groups that constitute asphalt binder. The FTIR spectrometer simultaneously collects spectral data within a wide

spectral range (400 cm^{-1} to 4000 cm^{-1}). Peaks in the spectral data can be used to detect the chemical functional groups. Functional groups that contain oxygen include carbonyl and sulfoxides. Hence, changes in the concentration of carbonyls and sulfoxides can be applied to the AIPs to measure the extent of oxygen uptake in the asphalt binder (Han 2011). The extent of the oxidation reaction in asphalt binders usually is tracked by following the infrared peak at wave number 1700 cm^{-1} (1650 cm^{-1} to 1820 cm^{-1}) that typically is assigned to carbonyl (Prapaitrakul 2009, Petersen 2009, Han 2011).

The carbonyl chemical functional group has long been used to indicate the level of asphalt oxidation. The linear relationship between the log viscosity increase and the carbonyl formation during asphalt oxidation is well established (Petersen 2009). The ketone functional group is the major component of the carbonyl IR absorption region. Ketone formation sufficiently changes the polarity of the associated aromatic ring components, which leads to an increase in the asphaltene fraction, thereby increasing viscosity (Petersen 2009, Petersen et al. 2011).

The other major oxidation product that is readily identified via IR spectroscopy is the sulfoxide functional group. The effects of sulfoxides have received less consideration in the past, primarily because sulfoxides that form at high temperatures during oxidative aging conditioning thermally decompose, which made sulfoxide concentrations an unreliable, variable measure. However, an expanded and more fundamental understanding of the role of sulfoxide formation on physical properties during oxidative age hardening is presented in (Petersen et al. 2011). Figure 11 shows the relationship between the sum of ketones-plus-sulfoxides and the log viscosity increase for oxidative aging from the Petersen et al. study. The analysis shows that the alcohols that are produced have a similar effect on viscosity increase as the ketones, and because they are more polar than ketones, they have a significant effect on the increase in viscosity, especially for high sulfur asphalts. Taking into consideration that ketones and sulfoxides form at different rates for different asphalts, the ratio between the ketone and sulfoxide formation rates is highly source-dependent. For example, binders extracted from different sources will have different sulfur contents, and this parameter will change the rate of sulfoxide formation. Then, the ratio between sulfoxide formation versus ketone formation will be binder source-dependent (Petersen et al. 2011).
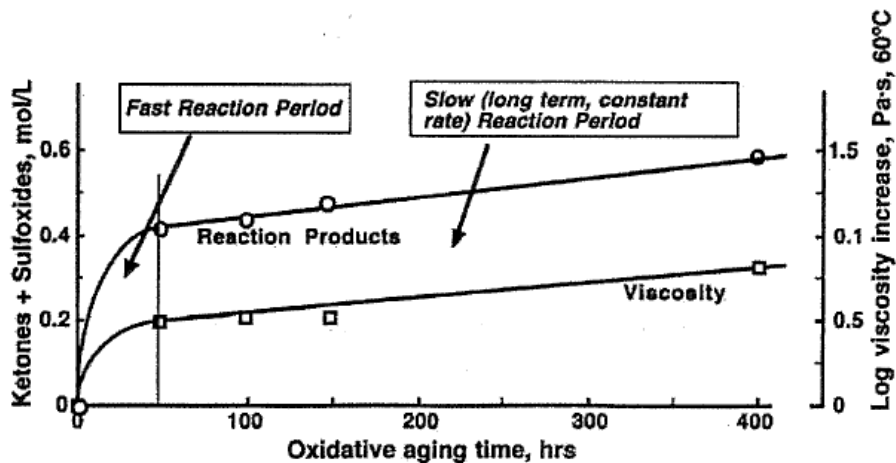
Figure 11. Kinetics of ketones plus sulfoxide formation and viscosity increase during the dual oxidation mechanism for SHRP asphalt AAB-1, PAV-aged at 80°C (Petersen et al. 2011)

2.1.2. Rheological Parameters

Although chemical groups can be used to track oxygen uptake directly, and hence, can track the oxidation in asphalt binders, FTIR spectrometry is not a standard test procedure for characterizing asphalt. Moreover, the chemical functional groups provide little indication of the implications of oxidation on the rheological properties of interest. This limitation has prompted researchers to consider the use of rheological properties as AIPs. The *limiting zero shear complex viscosity* (η_0^*), also known as *limiting viscosity*, has been used as an AIP. The limiting viscosity is defined as the plateau at low frequencies that is determined from a viscosity mastercurve. This property is independent of frequency or shear rate for unmodified binders (Han 2011). Furthermore, this property has been linked to carbonyl content for both neat and polymer-modified asphalt binders using Equation (1) (Han 2011).

$$ln(\eta_0^*) = HS.CA + m \tag{1}$$

where: η_0^* = asphalt binder limiting viscosity;
HS = hardening susceptibility;
CA = carbonyl content; and
m = log-viscosity intercept, where HS and m are asphalt binder-dependent.

The crossover modulus (G_C^*) also has been proposed as an AIP (Farrar et al. 2013). The crossover modulus is defined as the value of the dynamic modulus at the point where the storage modulus (G') and loss modulus (G'') cross (i.e., where the phase angle is 45°), as illustrated in Figure 12. If the asphalt binder is thermorheologically simple, then the crossover modulus is independent of temperature (Farrar et al. 2013).
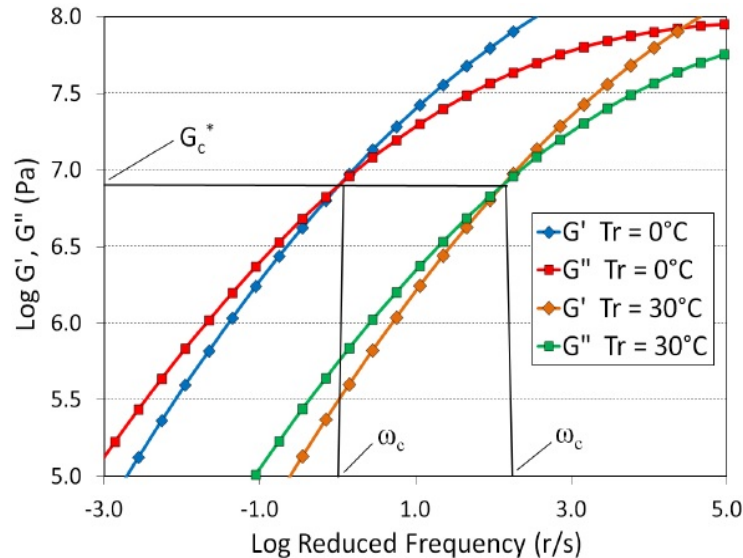
Figure 12. Crossover modulus and frequency at two reference temperatures

The WRI is investigating the linkage between the binder crossover modulus ($G_C{}^*$) and carbonyl and sulfoxide measurements made using a FTIR spectrometer. The oxygen uptake, measured as a percentage of the total oxygen, is an indication of the formation of carbonyl and sulfoxide bands and, to some extent, the bands that are related to aromatization. It has been demonstrated that oxygen uptake correlates linearly with the inverse of the log crossover modulus $\left(\frac{1}{\log G_C{}^*}\right)$, as shown in Figure 13. (Farrar et al. 2013).
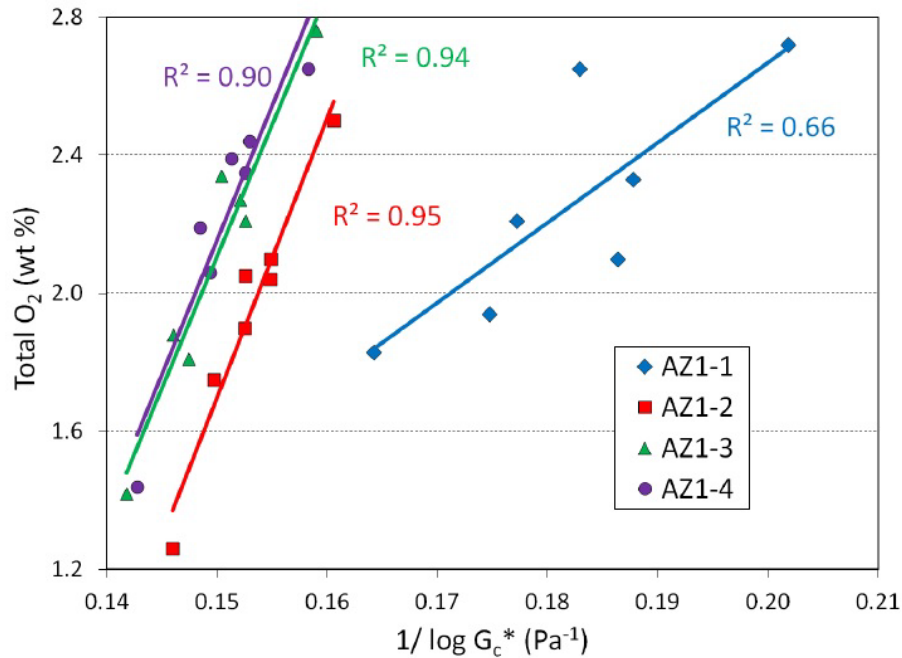
Figure 13. Observed approximate linear relationship between oxygen uptake and the inverse of the log crossover modulus for the recovered binders from loose mix collected during construction and cores collected at 4 and 9 years after construction

2.2. *Oxidation Kinetics*

All asphalt materials exhibit relatively similar kinetics, which can be described as trends in AIPs that are indicated by an initial fast reaction period, also known as *spurt*, followed by a slower reaction period that has an approximately constant rate (Petersen et al. 1996, Petersen 1998, Petersen et al. 2011, Prapaitrakul 2009, Han 2011). The hydrocarbon chemical reactions that occur during these two reaction periods are known to be fundamentally different (Petersen 1998). During the spurt, sulfoxides are the major oxidation product and cause an increase in viscosity. During the slower reaction period, ketones are the major product and drive the increase in viscosity (Petersen et al. 1996). Highly reactive hydrocarbon precursors of limited concentration react with oxygen during the fast reaction period to form hydroperoxides. Then, these hydroperoxides react primarily with the asphalt sulfides to form sulfoxides. When all of the highly reactive hydrocarbons are exhausted, the spurt is over. Then, a classical free radical hydrocarbon chain reaction takes over during the slow, approximately constant reaction period (Petersen et al. 1996). Figure 14 depicts the two oxidation reaction periods, where η_O is the original viscosity of the un-aged binder,

and η_{OT} is the viscosity after short-term oven aging, which simulates the hardening that takes place during the production of asphalt concrete (Prapaitrakul 2009, Petersen et al. 2011).
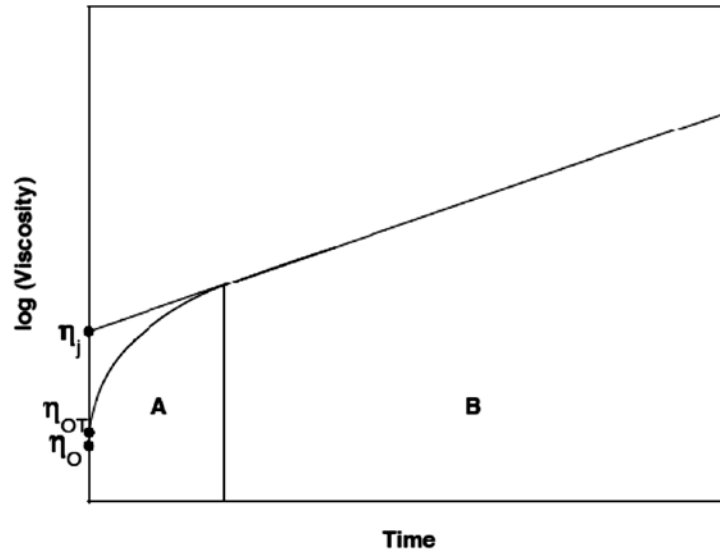
Figure 14. Dual reaction mechanism (Prapaitrakul 2009)

Asphalt binder oxidation kinetics typically is modeled using the Arrhenius expression of temperature and pressure dependency, as shown in the general form expressed in Equation (2) (Prapaitrakul 2009).

$$r = A\, P^\alpha \exp\left(\frac{-E_a}{RT}\right) \tag{2}$$

where: r = rate of the reaction;
   A = frequency (pre-exponential) factor (s^{-1});
   P = absolute oxygen pressure;
   α = reaction order with respect to oxygen pressure;
   E_a = activation energy;
   R = universal gas constant, or ideal gas constant; and
   T = temperature (Kelvin).

When carbonyl content is used as an AIP, the kinetics models reveal a wide range of binder-specific Arrhenius parameters (Petersen 2009). However, when carbonyl + sulfoxide contents were used in a recent study by the WRI to track the oxidation reaction, a simple dual mechanism model was used successfully to fit the oxidation of all 12 asphalt binders that originated from a wide variety of sources. The fitting used the same Arrhenius parameters for all 12 binders studied, with only one adjustable parameter, which was the amount of the fast reaction reactive material ($C_{fast,0}$). Equation (3) shows the model that was used to fit the isothermal data to determine the rate constants and amount of reactive

26

material for each binder studied (Glaser et al. 2013). If the Arrhenius parameters can be applied universally, a single aging trial at a single temperature may be performed to characterize the oxidation kinetics for unmodified binders (Glaser et al. 2013).

$$P = C_{fast,0}\left(1 - \frac{k_2}{k_1}\right)\left(1 - e^{-k_1 t}\right) + k_2\, C_{fast,0}\, t + P_0 \tag{3}$$

where: P = reaction products;
$C_{fast,0}$ = fast reaction reactive material;
k_1, k_2 = temperature-dependent parameters of fast and slow reaction paths, respectively;
t = time.

$$k'_1 = A_1 e^{\frac{E_{a,1}}{RT}}$$

$$k'_2 = A_2 e^{\frac{E_{a,2}}{RT}} \tag{4}$$

where: $A_{1\&2}$ = Arrhenius pre-exponential constant; and
$\frac{E_a}{R}$ = Arrhenius activation energy divided by gas constant.

2.3. *Empirical Models for Asphalt Binder Aging in Pavements*

The *global aging system* (GAS) model (Mirza and Witczak 1995) is an empirical model that allows for the prediction of the change in binder viscosity as a function of age, given the mean annual air temperature (MAAT), and also considers the aging gradient with pavement depth. The model assumes a hyperbolic aging function that predicts a decreasing rate of viscosity with an increase in age (generally consistent with the observations made for the two-stage oxidation kinetics model discussed previously), under the assumption that most age hardening occurs within the first ten years of a pavement's service life. The GAS model is summarized in Equations (5) and (6).

$$\log\log(\eta_{aged}) = \frac{\log\log(\eta_{t=0}) + At}{1 + Bt}\, F_v \tag{5}$$

$$\log\log(\eta_{aged,z}) = \frac{\eta_{aged}\,(4+E) - E \times \eta_{t=0}(1-4z)}{4(1+E \times z)}\, F_v \tag{6}$$

where: η_{aged} = aged viscosity (centipoise);
$\eta_{t=0}$ = viscosity at mix/laydown (centipoise);
A and B = are functions of the temperature and MAAT for the location of interest
        and $\eta_{t=0}$;
F_v = optional air void content adjustment factor;
t = time in months;
z = depth; and
E = function of MAAT.

The simplicity of the GAS model makes it an attractive option. However, the GAS model has been criticized for the following reasons:

1) The form of the GAS model implemented into the MEPDG does not account for aging 1.5 inches below the pavement surface (Mirza and Witczak 1995, Prapaitrakul 2009).

2) The model does not account directly for differences in asphalt binder kinetics among binder types.

3) The method used to account for air void content is based on a relatively small set of conditions, which is the reason this adjustment factor is considered optional (Mirza and Witczak 1995).

4) Prapaitrakul expressed concern over the unrecorded recovery method used to obtain binders from the field in developing the GAS model, because solvent recovery processes can leave residual solvent in the extracted binder, which leads to an effective softening and, hence, potentially leading to erroneously low viscosity values (Prapaitrakul 2009).

Despite these criticisms, the GAS model has been useful in the MEPDG and in other research studies. Its success and practical usage can be linked to its use of simple and easy-to-identify parameters.

2.4.   *Fundamental Models for Asphalt Binder Aging in Pavements*

A preliminary fundamentals-based, one-dimensional combined asphalt oxidation kinetics and diffusion model was developed at Texas A&M University. This model is referred to as the *transport model* (Lunsford 1994). Lunsford's foundational work on the transport model (1994) combines a mathematical model of asphalt oxygen diffusion and reaction kinetics, as shown in Equation (7), with Fick's law of diffusion, as presented in Equation (8).

$$\left(\frac{\partial C_{O_2}}{\partial t}\right) = -\nabla \cdot N_{O_2} - r_{O_2} \tag{7}$$

where: C_{O_2} = concentration of oxygen (mol. $/m^3$);

N_{O_2} = oxygen diffusion flux (mol.m^{-2}.s^{-1}); and

r_{O_2} = oxygen depletion rate.

$$N_{O_2} = -D_{O_2} \nabla C_{O_2} \tag{8}$$

where: N_{O_2} = chemical molar flux of oxygen (mol.m^{-2}.s^{-1});

D_{O_2} = diffusion coefficient of oxygen (m^2.s^{-1});

∇ = gradient operator; and

C_{O_2} = concentration of oxygen (mol. $/m^3$).

However, Lunsford's initial work did not include the effect of aggregate and void structure on diffusion; hence, approximate values were used as the oxygen diffusivity coefficients for the asphalt binders. In addition, the framework did not include a pavement temperature model (Prapaitrakul 2009).

Prapaitrakul et al. (2009) expanded on the work of Lunsford (1994) to overcome these deficiencies. The Prapaitrakul et al. diffusion model is based on three interlinked processes: 1) the diffusion of oxygen into the asphalt binder mastic in the pavement, 2) heat transfer into the pavement that results in temperature variations with depth and time, and 3) asphalt binder oxidation kinetics. Modeling these processes was facilitated by quantifying the air void distribution in the mixture, which dictates the availability of oxygen for the binder (Prapaitrakul et al. 2009).

Figure 15 provides a depiction of the Prapaitrakul overall approach (Prapaitrakul 2009). This improvement to the original transport model was developed in a cylindrical coordinate system that is able to capture the effects of air void structure on pavements and include those effects in the model calculations. The use of cylindrical coordinates is based on the assumption that oxygen in asphalt concrete exists in air channels that extend through the pavement depth and is replenished from the thermal cycle within the pavement that leads to excess oxygen for the oxidation reaction. The model also is based on the assumption that the partial pressure of oxygen at the pore surface equals the partial pressure of oxygen in air (approximately 0.2 atm).
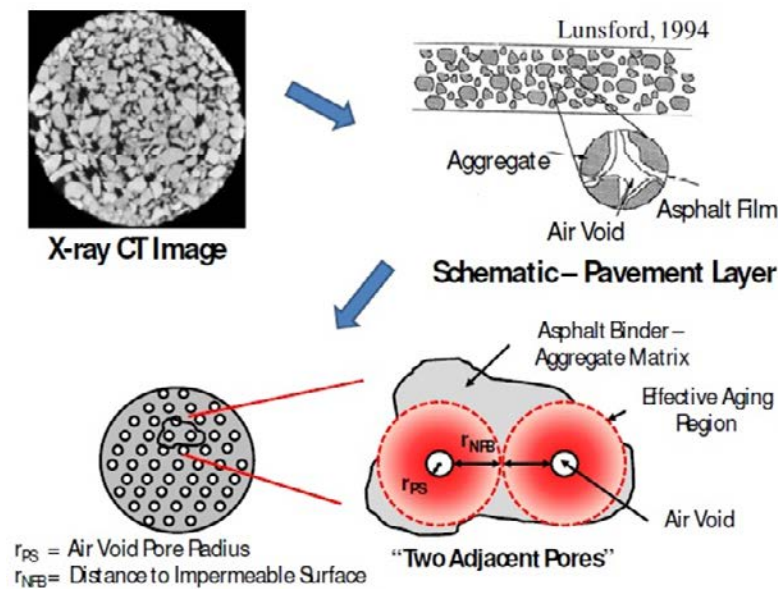
Figure 15. Texas A&M University oxygen transport model, proposed by Prapaitrakul et al. (2009)

As part of the Asphalt Research Consortium (ARC) project, a more recent study by Han (2011) built upon the work of Prapaitrakul by modeling diffusion as two interlinked steps: 1) the diffusion/flow of oxygen from the atmosphere above the pavement into the interconnected air voids in the pavement (termed *vertical transport*) and 2) the diffusion of oxygen from the air voids to the adjoining asphalt-aggregate matrix (termed *horizontal transport*) where it reacts with the asphalt binder. Equation (9) summarizes the vertical transport sub-model, which replaces the aforementioned assumption made by Prapaitrakul that oxygen partial pressure at the pore surface at any given depth equals the oxygen partial pressure in air (0.2 atm). Equation (10) expresses the sub-model for horizontal oxygen diffusion. These steps are

modeled mathematically to calculate the oxidation rate for a finite asphalt-aggregate matrix layer for each air void. Taking in consideration that Equation (9) is used for a single void geometry, this function would need to be repeated multiple times for the different air void geometries. The bulk oxidation rate for a pavement layer then is calculated as the average of the oxidation rates calculated for each air void.

$$\frac{\partial P}{\partial t} = 0.2 \, \frac{\partial P}{\partial z} + A \times r_{O_2}$$
(9)

where: P = oxygen partial pressure;

                A = proportion of modeled cross-section that is void space;

                r_{O_2} = rate of oxygen consumption; and

                z = depth along diffusion channel.

$$\frac{\partial P}{\partial t} = \frac{1}{r} \frac{\partial}{\partial r} \left(r \, D_{O_2} \frac{\partial P}{\partial r} \right) + \left(\frac{c \, R \, T}{h} \right) r_{CA}$$
(10)

where: r = diffusion distance (in cylindrical coordinate system);

                D_{O_2} = oxygen diffusivity in asphalt binder;

                c = conversion factor from oxygen partial pressure units to concentration units in asphalt binder;

                R = gas constant;

                T = absolute temperature;

                h = Henry's law constant; and

                r_{CA} = rate of carbonyl uptake.

It is should be noted that the model calculations provide a range of oxidation rates, not a single value, depending on the upper or lower limit of the oxygen partial pressure, P_{av}, in a given pore. The higher oxidation rates are based on the assumption that convective flow through the pavement pores is sufficient to maintain oxygen partial pressure at 0.2 atm. The lower rate calculation is based on the assumption that the replenishment of oxygen in the pores is achieved only via the diffusion of oxygen from the pavement surface (Asphalt Research Consortium 2011).

Figure 16 provides an overall picture of the four sub-models developed at Texas A&M University that comprise the transport model. Many of the required inputs for the sub-models are not standard test methods. For instance, X-ray computed tomography (CT) was used in this model to measure sophisticated air void structure properties, such as the air void radius and its frequency of existence within the pavement, to be used as inputs for the vertical transport sub-model. Because taking these measurements is extremely challenging, many assumptions had to be made, even with the X-ray CT. Also, FTIR spectrometry was used extensively to obtain binder-specific kinetics parameters (such as AP^{α}, E, HS, and m) as inputs for the oxidation kinetics sub-model. Thus, if this model is used in the current project, efforts will be made to develop surrogate means to obtain these inputs.

Modeling of Asphalt Binder Aging in Pavements

| Inputs for Sub-Models | Sub-Models | Outputs of Sub-Models |

- Aggregate Gradation
- Air Void Structure, Distribution, and Interconnectivity

Vertical Transport Sub-Model (Through Air Void Channels)

Amount of Oxygen available into the interconnected air voids at a given depth

- Mastic Shell Thickness/ Binder Film Thickness
- Filler Volume Fraction

Horizontal Transport Sub-Model (Through Asphalt-Aggregate Matrix)

Amount of Oxygen available into the Asphalt-Aggregate Matrix at a given location

- Oxidation and Hardening Parameters (AP^α, E, HS, and m)

Asphalt Binder Oxidation Kinetics Sub-Model

Rate of binder oxidation happens at a given location

- Site-Specific Model Parameters
- Climatic Data

Pavement Temperature Modeling (Heat Transfer Sub-Model)

Pavement Temperature at a given depth

Bulk oxidation rate for a pavement layer

Figure 16. Diagram of required inputs and resulting outputs of each sub-model developed at Texas A&M University

The asphalt binder oxidation kinetics sub-model developed at Texas A&M University does not consider the aforementioned dual oxidation kinetics; instead, it considers an initial jump assumption that allows the model to consider only the second slower reaction following the initial fast reaction (Han 2011).

The field validation of the Texas A&M University transport model was part of the ARC project. The predicted versus measured oxidation rates obtained from the field cores were compared using six pavement sites in Texas and Minnesota. The pavement sections used in this study and the corresponding results are summarized in Table 11 and Table 12 and in Figure 17. The oxidation rates measured in the field agree well with the predicted oxidation rates, with a few exceptions for newly constructed pavements where the rate of the asphalt binder oxidation is likely still governed by the initial rapid reaction (i.e., spurt). For those sections, the oxidation rates were underestimated significantly due to the use of the slower, constant reaction rate assumption used in the model (Han 2011). Despite the under-prediction of the oxidation levels in the newer pavements, in general, the fundamentals-based model provides a very good prediction of the field measurements, which suggests that the model captures the critical elements that affect asphalt binder oxidation in pavements (Asphalt Research Consortium 2011).

Table 11. Field Sites Studied in the ARC Study (Han 2011)

District (State)	Highway	Thickness (inch)	PG (modifier)	Binder Supplier	AAV (%)	Cons.	1st Coring	2nd Coring
Laredo (TX)	US277	2.5	70-22 (SBS)	Valero-C	7.27	2008	07/2008	09/2009
Lufkin (TX)	US69	2	70-22 (SBS)	Marlin	7.91	2003	02/2005	06/2008
Bryan (TX)	US290	2.5	64-22 (Un)	Eagle	12.44	2002	10/2005	08/2008
Waco (TX)	IH35 #5	3	70-22 (SBS)	Alon	5.86	2003	10/2005	08/2008
Amarillo (TX)	US54	2.5	70-28 (SBS)	Alon	7.33	1998	12/2004	07/2008
Laredo (TX)	IH35 #4	2	70-22 (SBS)	Valero-C	2.01	2007	--	06/2008
Metro Area (MN)	I-94	4.5	AC-120(Un)	--	4.81	1993	11/2004	11/2008

Table 12. Comparison of Measured and Modeled Field Oxidation Rates (Han 2011)

STATES	Site	Oxidation rate modeled (CA/year)		Bulk oxidation rate measured (CA/year)
		Maximum	Minimum	
MN	Cell 1	0.0200	0.0195	0.0256
TX	US277-LRD	0.0465	0.0451	0.0705
	US69-LFK	0.0258	0.0255	0.0370
	IH35-LRD #4	0.0384	0.0245	---
	US290-BRY	0.0651	0.0620	0.0671
	IH35-WAC #4	0.0264	0.0243	0.0340
	US54-AMR	0.1117	0.0994	0.0935
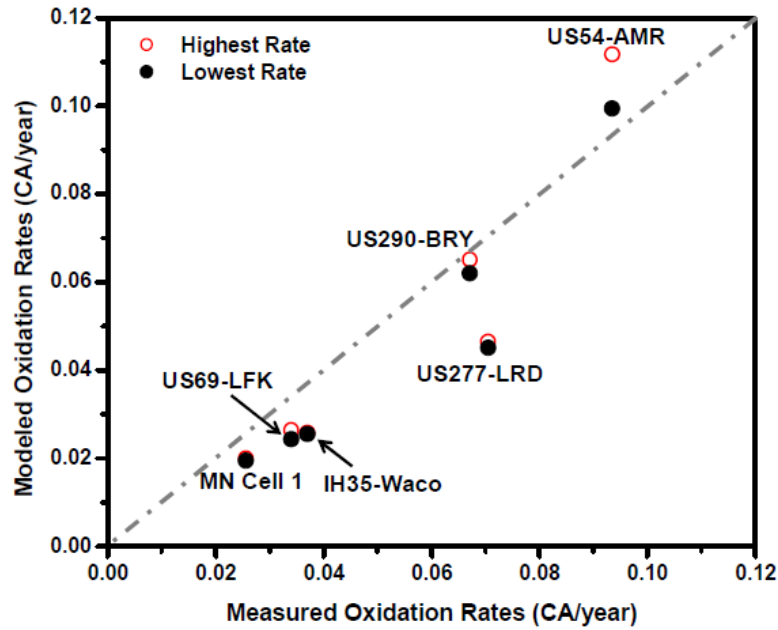
Figure 17. Graphical comparison of measured and modeled field oxidation rates (Han 2011)

The transport model developed at Texas A&M University will be considered by the research team because of the model's fundamental nature; however, as documented by Han (2011), this model is still in the initial stage of development, and the model calibration and validation steps are ongoing. Furthermore, the transport model has some additional constraints. It currently requires detailed information about air void content and distribution as well as the temperature gradient along the depth at a higher level of accuracy than can be provided by available databases, including the Enhanced Integrated Climatic Model (EICM).

2.5.   *Summary*

In summary, four candidate AIPs are discussed in the literature to be used to track the aging of asphalt binders in pavements. These parameters, which are 1) limiting viscosity, 2) crossover modulus, 3) carbonyl content, and 4) carbonyl + sulfoxide content, should be obtained from extracted and recovered binders. The first two AIPs are considered to be rheological parameters that are evaluated using the DSR, and the last two AIPs are considered to be chemical function groups obtained from FTIR spectroscopy.

The GAS model is an empirical model that allows for the prediction of changes in binder viscosity as a function of age, given the MAAT, and also considers the aging gradient with pavement depth. It has been useful in the MEPDG and in other research studies. Its success can be attributed to its use of simple and easy-to-identify parameters. However, fundamental-based studies were performed to overcome many of the criticisms of the empirical model.

AIPs are used to study asphalt binder oxidative aging kinetics. Two kinetics models have been proposed by the WRI and Texas A&M University. The WRI model is based on tracking the carbonyl + sulfoxides chemical functional group of AIPs and takes into account the dual oxidation kinetics (initial fast reaction followed by a slower reaction). Although the transport model developed at Texas A&M University is based on tracking the carbonyl chemical functional group, it assumes that a constant rate of the slower reaction occurs after the initial fast rate reaction. The field validation study for the Texas A&M University transport model shows good prediction results for old pavements but significantly under-predicts for recently constructed pavements.

In addition to the kinetics models, the fundamental-based transport model developed at Texas A&M University includes three additional sub-models to predict pavement aging. These sub-models include 1) oxygen diffusion/flow from the atmosphere into the interconnected air voids in the pavement, 2) oxygen diffusion through the asphalt-aggregate matrix, and 3) heat transfer.

These previous research efforts will be considered as a starting point for the current research to develop a kinetics/diffusion model to facilitate the development of a laboratory procedure that can simulate long-term aging in the field.

Part II: Experimental Plan

Introduction

The objective of this research is to develop a calibrated and validated procedure to simulate the long-term aging of asphalt mixtures for performance testing and prediction. The final product will be a laboratory aging procedure and associated algorithms that prescribe a set of laboratory aging conditions to represent the long-term aged state of asphalt mixtures in a pavement as a function of climate, depth, and air void content. The experimental plan hinges around the use of key asphalt binder aging index properties (AIPs) to develop a reliable long-term aging procedure that mimics field aging conditions. The candidate AIPs include carbonyl absorbance, carbonyl plus sulfoxide absorbance, the dynamic shear crossover modulus, and viscosity. This AIP-based approach has been selected because asphalt binders are affected most directly by oxidative aging, whereas mixtures are subject to other factors, including mechanical degradation that is caused by traffic loading, thermal induced stress, and moisture.

The comprehensive literature review in Task 1 and the efforts made in Task 2 of the Work Plan have resulted in the experimental plan composed of the following five primary tasks under Task 3:

- Task 3.1: Acquisition of Materials and Cores
- Task 3.2: Establishment of the Preliminary Aging Procedure for Field Materials
- Task 3.3: Development of the Optimal Long-Term Aging Protocol
- Task 3.4: Validation of the Optimal Aging Protocol
- Task 3.5: Development of Calibration Functions for Traffic, Thermal, and Moisture Conditions

The following sections describe the experimental plan developed by the research team based on the efforts made in Tasks 1 and 2. It should be noted that in the following sections, the sequencing of the tasks deviates somewhat from the sequencing of tasks put forth in the work plan. However, based on extensive discussion amongst the research team members, this approach implemented in the experimental plan has been deemed more logical for the interest of the reader and the efficient execution of the experimental plan. In order to compare the work plan and the experimental plan herein, Table 1 presents the tasks in the experimental plan as they correspond to the tasks in the approved work plan.

Table 13. Comparison of tasks in the work plan and tasks in the experimental plan

Tasks in the Work Plan	Tasks in the Experimental Plan
3.2. Establishment of Candidate Aging Procedures	3.2.1. Establishment of Candidate Aging Procedures
3.3. Determination of Aging Index Properties from Original Materials and Cores	3.2.2. Preliminary Evaluation of Selected Aging Procedure
3.4. Preliminary Evaluation of Aging Procedures and Sensitivity Study	3.3.1. Determination of AIPs from Original Binders, Laboratory-Aged Specimens, and Cores from In-Service Pavements
	3.3.2. Sensitivity Study
3.5. Development of Long-Term Aging Protocol and Field Calibration	3.3.3. Development of Optimal Aging Protocol
	3.4. Validation of the Optimal Aging Protocol
	3.5. Development of Calibration Functions to Account for Traffic, Thermal, and Moisture Conditions

Task 3.1: Acquisition of Materials and Cores

The entire experimental plan involves two groups of materials. The Group A materials are a set of laboratory-prepared materials for which relatively large quantities of materials are available but no corresponding field data are available. The main purpose of investigating these materials is to understand the aging behavior of asphalt concrete during preliminary trials of laboratory aging procedures without having to consume precious materials from the selected field sections. Also, the Group A materials allow for a systematic sensitivity study to investigate the effects of changes in the AIPs on the performance of asphalt concrete. The Group B materials are the original component materials (i.e., binder and aggregate) and field cores extracted from in-service pavements. These materials will be used to develop the long-term aging procedure and for validation and calibration. In the following, detailed descriptions of the selected materials are given.

Group A Materials

Three binders and two aggregate types are included in the Group A materials, as detailed in Table 2. Two commonly used binders, PG 58-28 and PG 64-22, and one polymer-modified binder, PG 76-16, have been selected in order to cover a wide range of aging characteristics. The limestone aggregate and PG 58-28 and PG 76-16 binders used in the Asphalt Research Consortium (ARC) study will be acquired for this study. The PG 64-22 binder and granite aggregate will be acquired from sources typically used in North Carolina.

Table 14. Summary of Group A materials

Material Source	Material	
	Aggregate	Asphalt Binder
ARC	Limestone	PG 58-28, PG 76-16
North Carolina	Granite	PG 64-22

Group B Materials/Projects

Table 15 summarizes the field projects selected for the Group B materials. Material selections from the available projects were made on the basis of available component materials and cores, geographic coverage, and age level. Also, consideration was given to include a wide range of mixture designs and constituents (e.g., reclaimed asphalt pavement (RAP), warm mix asphalt (WMA), etc.) in order to establish a comprehensive experimental plan. The agency sponsors of these proposed sites are the NCSU and Nichols Consulting Engineers (NCE).

Table 15. Selected sections for development of long-term aging protocol and field calibration

Agency	Site ID	Location	Binder / Modification	Date Built	Date Core Extracted
NCSU	FHWA ALF	Virginia	Control, Airblown, SBS-LG, CR-TB, Terpolymer	2001	2013
	Manitoba	Manitoba	Control, WMA, 40% RAP	2010	2014
	NCAT	Alabama	Control, 15% RAP, Foamed WMA, Advera WMA, 50% RAP, 50% RAP with Foamed WMA	2009	2013
NCE	LTPP SPS-1	Ohio, US-23 MP 20.9	Asphalt Cements AC-20	Nov-95	2006, 2012
	LTPP SPS-8	California	Asphalt Cements AR-4000 (AR_40 by AASHTO Designation)	Sep-99	2007
		New Mexico	Asphalt Cements AC-20	Nov-96	2006
		Texas	Asphalt Cements AC-20	Jul-96	2007
		Washington	Asphalt Cements AR-4000 (AR_40 by AASHTO Designation)	Oct-95	2006
		Wisconsin, SR-29	-	Nov-97	2005
	WesTrack (Sections: 1, 2, 3, 4, 14, 17, 18, 36, 39)	Dayton, Nevada	PG 64-22	1995	1995
					1997
					1999

Figure 18 provides a summary of the geographic coverage of the selected Group B materials/projects. It can be seen that a broad range of geographic locations spanning the United States are included in the proposed experimental plan.
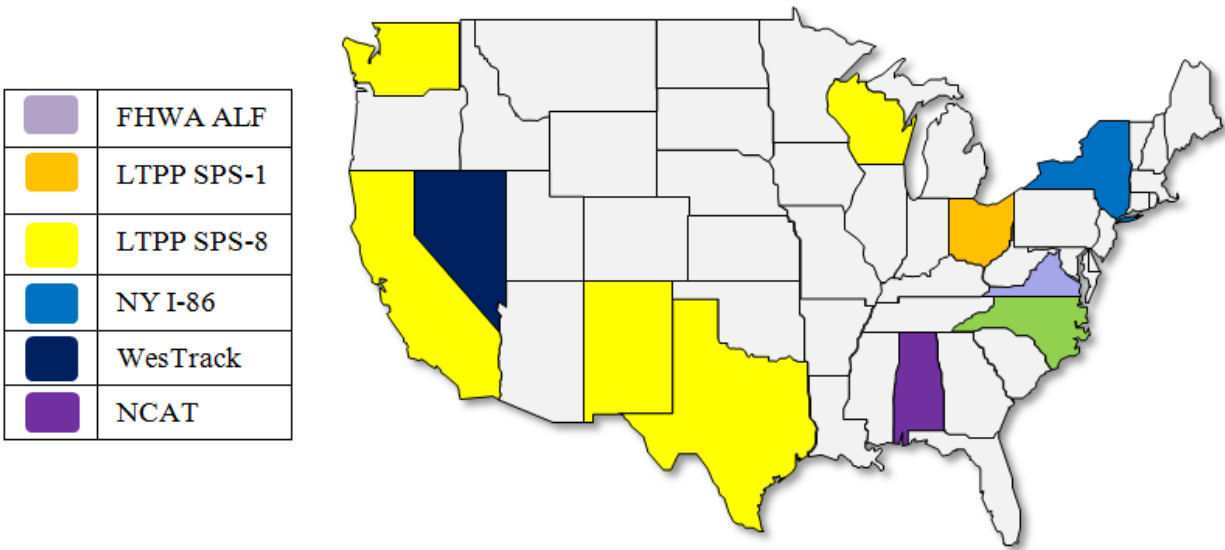
Figure 18. Locations of Group B materials/projects

Task 3.2: Establishment of the Preliminary Aging Procedure for Field Materials

Overview

In order to establish the aging procedure for the field materials, a subset of the Group A materials will be subjected to a set of long-term aging procedures. Based on the literature review, it has been decided that these preliminary procedures will include high temperature conditioning of loose mixtures and compacted specimens with and without pressure. For the compacted specimens, two geometries will be considered: standard 100 mm diameter specimens and the newly proposed 38 mm diameter small specimen geometry. The 38 mm diameter geometry was suggested by the FHWA in its forensic evaluation of ALF pavements (Mbarki et al. 2012) and further developed by the NCSU research team for the forensic study of fatigue cracking of pavements in North Carolina.

Based on the outcomes of trials using the Group A materials, decisions for the testing of the Group B materials will be made with respect to the state of the material (i.e., loose mix versus compacted mix), specimen geometry (100 mm diameter versus 38 mm diameter), pressure, temperature, and duration. In order to select the appropriate preliminary test procedure and its parameters, the selected aging procedure will be tried on a small subset of component materials from the Group B materials (four sections only) with various air void contents and binder types. Selection of the aging procedure and associated parameters will be based upon the integrity of the specimens following aging (and subsequent compaction in the case of loose mix aging), the reasonableness of the AIPs measured from the lab-aged materials compared to those from the Group B field cores, and the relative efficiency of the procedure.
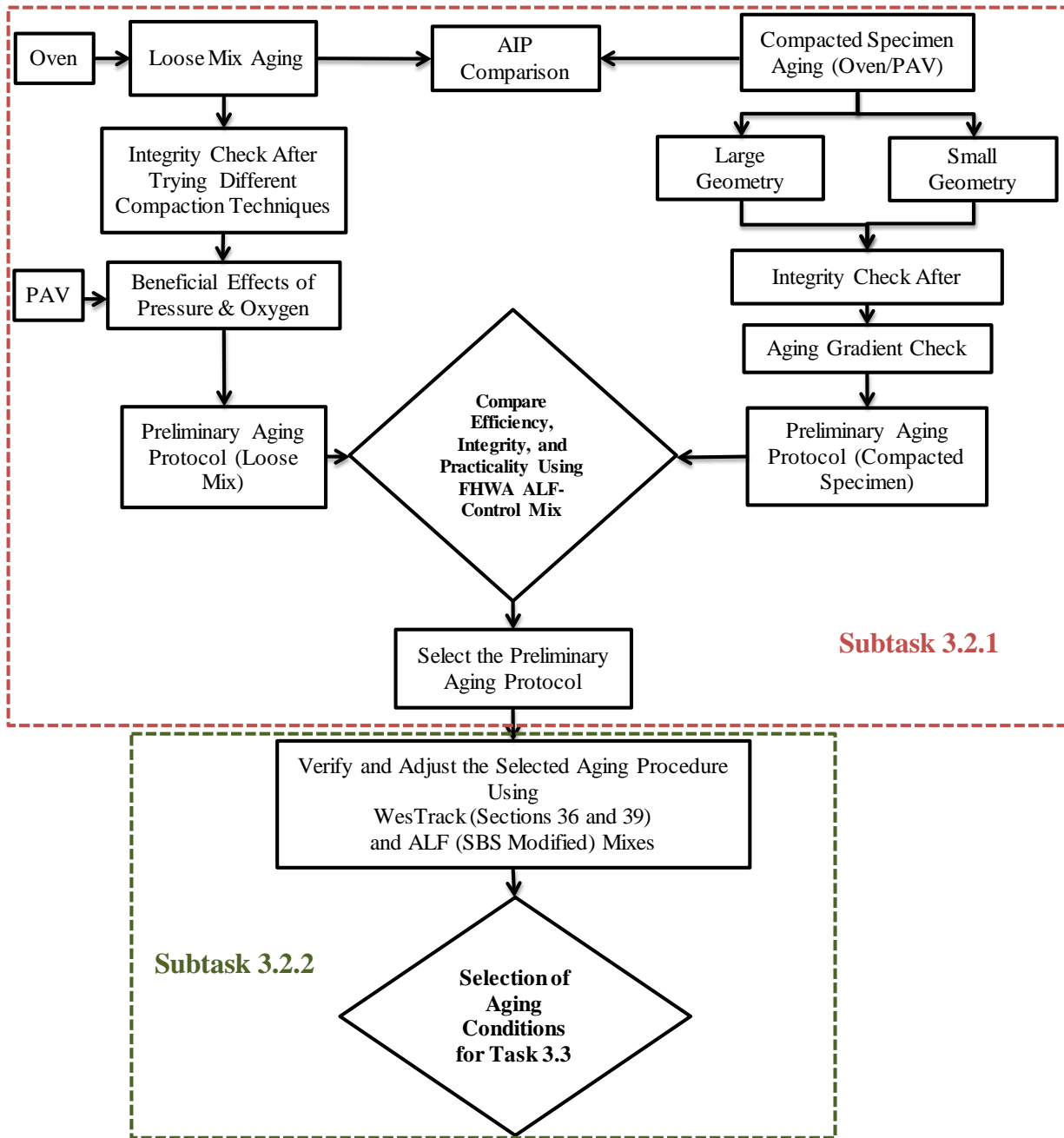
Figure 19. Flowchart for Task 3.2: Establishment of the preliminary aging procedure for field materials

Subtask 3.2.1: Establishment of Candidate Aging Procedures

Based on the literature review, two candidate aging methods have been identified: oven aging and pressure aging vessel (PAV) aging. These aging methods can be applied to both loose mix and compacted specimens. Therefore, the preliminary aging procedure will be divided into two subtasks.

Preliminary Study on Aging of Compacted Specimens

Trials for aging the compacted specimens will be made using both the oven and PAV. The current standard procedure for asphalt mixture laboratory aging, AASHTO R30, consists of aging compacted specimens in the oven at 85°C for five days. However, several concerns have emerged using this procedure. AASHTO R30 specifies a single aging procedure regardless of climate, depth, and age of interest. In addition, changes in air void content and slump during aging have been reported with the procedure (Reed 2010). To overcome these problems, the NCHRP 9-23 protocol recommends wrapping specimens in a metal wire mesh secured with three clamps to prevent the samples from slumping. However, this approach is reported to solve the slumping problem only partially (Reed 2010). Also, oven aging of compacted specimens leads to an aging gradient from the surface to the center of the specimen, which is a concern for its use in performance testing.

Potential solutions to overcome the slump and aging gradient problems associated with the AASHTO R30 protocol include either aging loose mixtures or aging compacted specimens using PAV aging and/or a small specimen geometry (Ø 38 mm) as opposed to using standard dynamic modulus test sized specimens. The preliminary experimental plan consists of conducting oven and PAV aging on specimens of both small and large geometries to provide possible solutions to the aforementioned problems. Asphalt mixture specimens will be fabricated using North Carolina component materials, as detailed in Table 1. Two temperatures of 60°C and 85°C (specified by AASHTO R30) will be used for aging the specimens in the oven. The lower temperature of 60°C is included to evaluate the effect of temperature on the integrity of the compacted specimens. In order to establish a time scale for comparing the different aging methods, cores will be aged for 0, 4, and 8 days in the oven.

The SHRP project evaluated pressure aging of compacted mixtures and, hence, those findings will be used to guide the work herein. Two pressure levels were tried in the SHRP project for PAV aging of compacted specimens: 690 kPa and 2.1 MPa, both at 60°C. It was found that the specimen air void contents increased during the PAV aging at both pressure levels, which was attributed to the release of pressure following aging. Therefore, efforts transitioned to the pressure aging in a triaxial cell at reduced pressure levels during the SHRP project. It was found that 350 kPa in the triaxial cell set-up at 60°C could be used to age compacted mixtures effectively without damaging effects. The reason for shifting from the PAV aging set-up to the triaxial cell set-up is not clear beyond the fact that reduced pressures were being tried.

Initial PAV Aging Evaluation

Based on the findings of the SHRP project, it is evident that one problem that still needs to be resolved is the damaging effects that pressure aging and the associated release of pressure can have on specimen integrity. For this reason, an initial trial of PAV aging will be conducted on a mixture produced from the Group A component materials (North Carolina). Part of this task will seek to determine the maximum

pressure level that can be used without damaging the specimen upon the release of pressure. In addition, the research team will try modifying the pressure release procedure in the PAV to mitigate damage to the specimens; this attempt will include reducing the temperature of the PAV prior to pressure release and slowing the rate of the pressure release. Based on the SHRP findings, two pressures and two temperatures will be tried initially: 300 kPa and 600 kPa at both 60°C and 85°C. Aging will be conducted for only one hour. The intent here is to check only the pressure and pressure release effects on the specimen, which will be facilitated by comparing the mixture performance properties before and after aging. This comparison will be most direct if there is minimal aging induced in the specimen.

Evaluation Criteria for Compacted Specimen Aging

After each aging trial, the following criteria will be used to assess specimen integrity:

Level 1 – Visual inspection, air void content, and dimensions of specimens. Following the PAV aging, each specimen will be inspected visually and the air void contents and dimensions will be measured and compared to the values prior to aging. If changes before and after aging are noted, no further analysis will be conducted, and it will be concluded that the pressure must be reduced and/or the pressure release procedure must be modified.

Level 2 – Mix performance testing. If the Level 1 criterion is met, dynamic modulus and simplified viscoelastic continuum damage (S-VECD) testing will be conducted. The results will be compared to the test results for the reference un-aged mixture to determine if significant changes occurred due to loss of specimen integrity during aging.

Comparison of Oven and PAV Aging

After performing the initial trials of PAV aging for the North Carolina compacted specimens and determining the pressure limits, further experiments will be conducted to determine the criteria for establishing the preliminary aging protocol and to compare/assess the effectiveness of oven versus PAV aging of the compacted specimens. For the discussion herein, *oven aging* refers to aging with zero pressure, and *PAV aging* refers to all other non-zero pressure conditions.

Table 16 details the experimental plan to assess the compacted specimen aging procedures. Factors under consideration in the plan include specimen size, aging duration, pressure, and temperature. The compacted specimens will be cored prior to long-term aging. The two cored specimen geometries will be used: standard dynamic modulus test sized specimens (100 mm diameter by 150 mm height) and the newly proposed small specimen geometry (38 mm diameter by 100 mm height). The potential advantages of using the small specimen geometry are that it provides a reduced aging gradient as well as the ability to test individual layers obtained from field cores. Furthermore, the use of small specimens would allow more specimens to be aged simultaneously in the PAV. Three pressures (high, low, and zero) were selected to determine the effect of pressure on the rate of oxidation. High and low pressure levels will be selected based on the results of the previous trials performed on the North Carolina compacted specimens. In addition, two temperatures and different durations are included to evaluate their respective effects on the aging level achieved and specimen integrity. Note that AASHTO R30 long-term aging Levels 1 and 3, which consist of 4 and 8 days of oven aging at 85°C, respectively, are included in this plan.

Evaluation Criteria for Compacted Specimen Aging

Two criteria will be used to evaluate the aging procedures presented in Table 16. These criteria are:

Level 1 – Specimen integrity for performance testing will be evaluated by visual inspection, dimension measurements, and air void contents before and after aging to determine if the specimen was damaged during the laboratory aging process. If this specimen integrity check is not deemed satisfactory, no further analysis will be conducted.

Level 2 – The aging gradient that runs through the compacted specimen needs to be checked in terms of aging level achieved. The WRI's micro-extraction and recovery procedure will be applied to the laboratory-aged specimens and compared with previously collected AIP data obtained from field cores to assess the level of aging that has been achieved for each aging trial. Although these mixtures and the AIP data from the corresponding field mixtures are inherently different, a comparison between the laboratory and field AIP values will still give an indication of the reasonableness of the level of aging that is achieved using the tried laboratory procedure. In order to assess the level of aging that is achieved and the specimen aging gradients, micro-extraction and recovery will be conducted at various specimen locations following laboratory aging. Figure 20 provides a schematic illustration that shows the locations required for micro-extraction and recovery. The recovered asphalt binder will be subjected to Fourier transform infrared spectroscopy (FTIR) and dynamic shear rheometer (DSR) testing in order to determine the AIPs.

Table 16. Trials and conditions to be used to establish the preliminary aging procedure

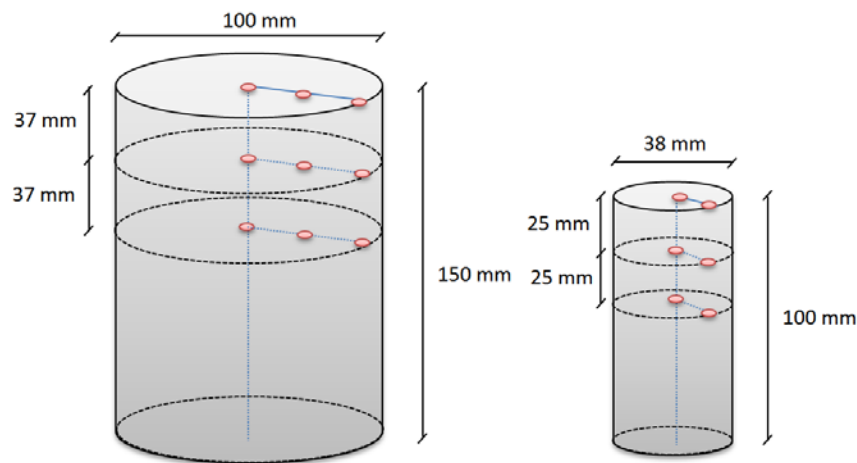| Materials State	Sample Size	Temperature	Pressure			Total Conditions
			0 (Oven)	Low	High	
Compacted Specimen	Small (Ø38 mm)	60°C	4, 8 days	2, 4 days	2, 4 days	24
		85°C	4, 8 days	2, 4 days	2, 4 days	
	Large (Ø100 mm)	60°C	4, 8 days	2, 4 days	2, 4 days	
		85°C	4, 8 days	2, 4 days	2, 4 days	

Figure 20. Schematic illustration of locations required for extraction and recovery to determine aging gradient

The outcomes of this subtask will be an assessment of the feasibility of compacted specimen aging and associated preliminary recommendations for the compacted specimen aging procedure, including specimen size, pressure (Oven if 0, and PAV if pressure other than 0 is selected), temperature, and time.

Preliminary Study on Aging of Loose Mixtures

Trials for aging the loose mixtures will be conducted using both the oven and PAV. Oven aging of loose mixtures will be performed in a standard oven at 60°C and 85°C. These temperatures were selected based on the literature regarding loose mixture aging (e.g., Mollenhauer et al. 2011, De la Roche et al. 2013). The aging of loose asphalt concrete mixtures allows air and heat to circulate easily inside the material, which causes uniform aging of the asphalt film. Loose mixture aging is believed to allow for the simulation of long-term field aging more efficiently than compacted specimen aging and to avoid problems associated with compacted specimen aging gradients.

However, a major concern with the long-term aging of loose asphalt mixtures in the oven or PAV to produce specimens for performance testing is the inability to compact the aged mixtures easily and efficiently. Previous studies of the compaction of laboratory-aged loose mixtures indicate that significantly more effort is required for compaction than for un-aged/short-term aged loose mixes (Reed 2010). Furthermore, it has been found that the increased compaction force/effort required to reach target air voids using aged loose mixtures may cause degradation in the aggregate structure and alter the mixture properties (Gatchalian 2006).

To overcome the problems of compacting aged loose mixtures, the research team will investigate various strategies to improve compactability, including the use of zeolite additives and the phase angle method that was used to determine compaction temperatures developed in the NCHRP 9-39 project (West 2010). The preliminary experimental plan for evaluating loose mixture aging consists of conducting oven aging trials using loose mixtures fabricated with North Carolina component materials. Specimens will be compacted after they have been aged. Inspections of the compacted specimens after each aging trial will be undertaken using the following evaluation criteria. If the compaction problems are overcome, aging trials will continue using both the oven and PAV test methods. The main focus of this comparison will be to determine whether PAV aging is more efficient than oven aging to age loose mixtures.

Evaluation Criteria

Level 1 – Total air void content and air void distribution. Following each trial of the different compaction techniques, each specimen will be inspected visually and the total air void content and air void distribution will be checked. The air void distribution will be studied by measuring the air void content of each slice of the Superpave gyratory compactor (SGC) specimen. Also, the SGC curves and number of gyrations will be recorded in order to control the compaction effort. Finally, digital imaging processing software will be used to analyze the internal coarse aggregate structure following compaction and to compare it to the structure of compacted, un-aged specimens to assess each specimen's integrity.

Level 2 – If the Level 1 criterion is met, the compacted specimens and loose mixtures will be aged to the same level (i.e., based on equivalent AIPs). In order to check the integrity of the specimens compacted after loose mixture aging, the dynamic modulus values and fatigue properties obtained from the direct

tension testing of the aged compacted specimens and the specimens fabricated from aged loose mixes will be compared.

In case of the failure of all the compaction techniques to meet the evaluation criteria, loose mixture aging will be aborted, and efforts will proceed with compacted specimen aging. On the other hand, if the compaction problems are overcome, the potential beneficial effects of aging loose mixtures in the PAV using pressure and/or pure oxygen will be evaluated. Table 17 shows the trials and conditions for this subtask.

Table 17. Trials and conditions to be used to establish preliminary aging procedure

Materials State	Temperature	Pressure			Total Conditions
		0 (Oven)	Low	High	
Loose Mix	60°C	2, 4, and 8 days	2, 4 days	2, 4 days	14
	85°C	2, 4, and 8 days	2, 4 days	2, 4 days	

The outcome of this task will be the determination of the feasibility of loose mixture aging. Recommendations for long-term aging of loose mixtures will be given, including guidelines for appropriate compaction techniques for aged loose mixtures.

Selection of Preliminary Aging Procedure

Subtask 3.2.1 will lead to the selection of the appropriate aging method for the next tasks. The major decision to be made in this task is whether the subsequent work will be based on the aging of loose mix specimens or compacted specimens.

Selecting the proper aging procedure requires a comparison of the efficiency, specimen integrity, and practicality of the two techniques. This selection process will be accomplished using the FHWA ALF Control mix from the Group B materials; that is, a mixture will be produced using component materials from the FHWA ALF Control section. The mixture will be aged based on the recommendations for preliminary aging protocols developed in the previous tasks for both loose mix and compacted specimens.

Further refinement of the recommendations for different factors (i.e., temperature, duration) will be made by performing performance tests on the laboratory- and field-aged specimens.

Evaluation Criteria

Level 1 – Efficiency. The relative time required for each aging protocol in order to meet the field aging level will be considered carefully.

Level 2 – Specimen Integrity. The integrity of the product that is achieved using each protocol will be evaluated visually. Further evaluations will be performed using performance tests.

- a. Aged Compacted Specimens
  - Air void content

- Dimensions/slump
  b. Specimens Fabricated from Aged Loose Mixture
    - Compactability
    - Air void content
    - Aggregate structure – image analysis
    - Performance testing – to check against performance measured from aged compacted specimens

Level 3 – Practicality. The relative cost and availability of the required equipment will be considered when selecting the appropriate aging procedure.

The outcome of this subtask is to make a selection between the two preliminary aging protocols (loose mix versus compacted specimens). Preliminary recommendations for the aging conditions (e.g., temperature, duration, and pressure level) also will be defined based on the experience gained in this subtask. In the case that the loose mix option is selected for developing the aging protocol, any necessary adjustments to the aging devices (oven/PAV) will be considered.

Subtask 3.2.2: Preliminary Evaluation of Selected Aging Procedure

The procedure selected in the previous subtask must be verified and possibly refined for use with additional mixtures, because the aggregate gradation, design air void content and binder type are believed to play an important role in this decision. Table 18 summarizes the Group B materials for consideration in this subtask.

Table 18. Materials for verification of and adjustment to the selected aging procedure

Factor Considered	Condition I	Condition II
Gradation and Air Void Effect	WesTrack Section 36 (Medium Air Voids)	WesTrack Section 39 (High Air Voids)
Binder Effect	FHWA ALF Control	FHWA ALF SBS

In order to evaluate the effects of aggregate gradation and air void levels, two mixtures from the WesTrack test site (Sections 36 and 39 in Table 19) will be considered. The air void levels in these sections have been altered systematically while keeping the other mix design parameters constant. Table 19, which presents the WesTrack experimental design, indicates that Sections 36 and 39 were prepared using optimal asphalt contents and the same (coarse) gradation but have optimal vs. high design air void contents.

The asphalt binder type also is expected to play a critical role in the aging that occurs within pavements. Thus, a systematic study that uses different binder types will be useful for this task. Two sections from the FHWA ALF project, one prepared with neat, control PG 70-22 asphalt binder and one with SBS-modified asphalt, have been selected for this purpose. Previous research at the FHWA has demonstrated that these binders exhibit significantly different oxidative aging characteristics.

Table 19. WesTrack sections and specifications

Design Air Void Content	Aggregate Gradation Designation								
	Fine			Fine Plus			Coarse		
	Design Asphalt Contents								
	Low	Opt.	High	Low	Opt.	High	Low	Opt.	High
Low		04	18		12	09/21		23 (39)	25 (55)
Medium	02	01/15	14	22	11/19	13	08 (38)	05/24 (35, 54)	07 (37)
High	03/16	17		10	20		26 (56)	06 (36)	

Shaded area denotes cells not filled

Evaluation Criteria

Specimens will be evaluated based on two criteria:

Level 1 - Specimen integrity for performance testing.

Level 2 - Comparison between the laboratory-aged specimen binder AIP and corresponding field core AIP. The laboratory-aged mixtures will be subjected to micro-extraction and recovery to determine the AIPs of the laboratory-aged specimens. These AIPs then will be compared to the corresponding field core AIP values at different field ages and depths to determine the level of aging achieved in the laboratory procedure.

Based on the above criteria, the aging procedure will be revised, if necessary, to prevent specimen damage and to produce realistic levels of aging.

The outcome of this subtask will be the selection of aging conditions (i.e., pressure, temperature, and time) for the preliminary aging procedure to be employed in Task 3.3.

Task 3.3: Development of the Optimal Long-Term Aging Protocol

Overview

The remaining Group B materials will be subjected to three aging trials of various durations. The AIPs will be measured from extracted binders taken from the laboratory-aged materials and compared to the values measured at different depths and age levels of the field cores that have been subjected to different climate conditions. Interpolation will allow for estimating the corresponding field aging condition (i.e., age level at a given depth) that has been achieved using each laboratory aging trial. Relationships will be developed between the aging conditions (such as time, temperature, pressure, etc.) used in the aging procedure and the conditions (such as depth, climatic condition, air void content, diffusivity, etc.) used for the core measurements. This work will be facilitated by a diffusion and kinetics model that will be developed either by refining an existing model or by creating a new model. The diffusion/kinetics model will predict the aging gradient along the depth of the asphalt layers as a function of climate conditions and

air void content and will allow the user to tailor the laboratory aging procedure to simulate any field condition (climate, age, depth, mix) of interest. Simultaneously, a sensitivity study will be conducted with the Group A materials to identify the degree of sensitivity of the asphalt mixture properties to changes in the AIPs of the binders, which will lead to the establishment of tolerance limits for the accuracy of the lab AIP in relationship to the target field core AIP.
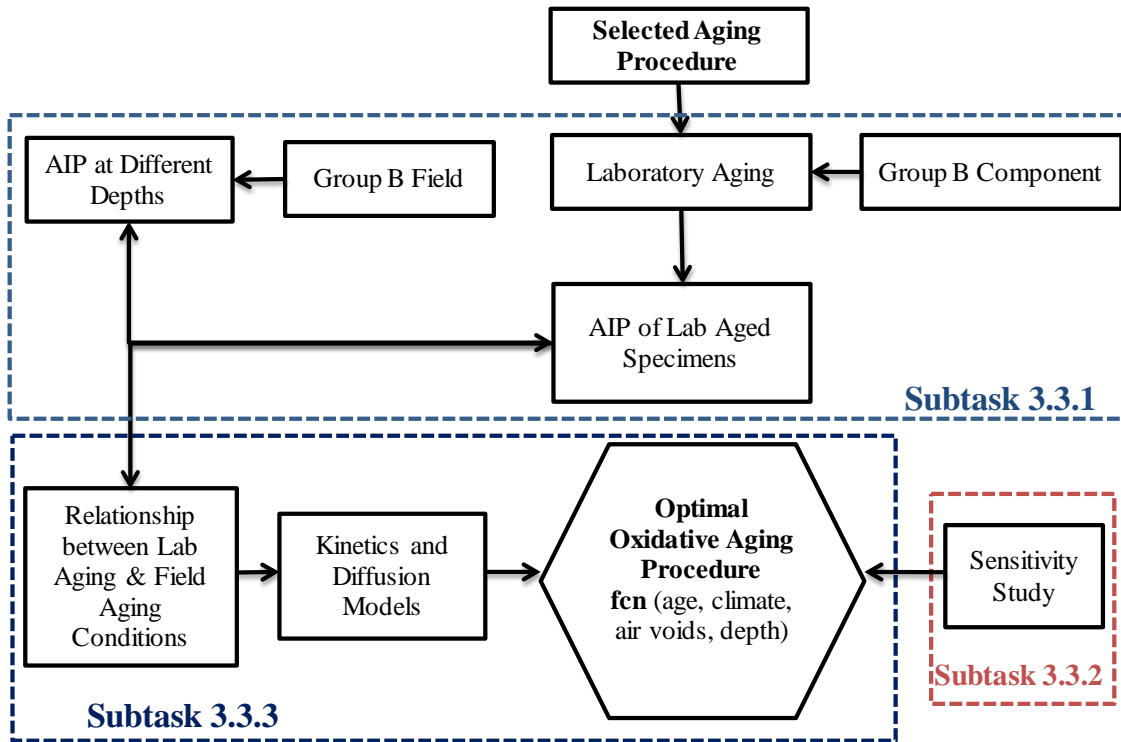
Figure 21. Flowchart for Task 3.3: Development of the optimal aging protocol

Subtask 3.3.1: Determination of AIPs from Original Binders, Laboratory-Aged Specimens, and Cores from In-Service Pavements

Cores from the Group B materials will be subjected to the WRI's micro-extraction and recovery procedure in order to determine the AIPs at varying pavement depths. Based on previous studies and ongoing studies, the following four AIPs will be used to quantify the extent of oxidation in the original project binders, in the binders extracted from the laboratory-aged materials, and in the recovered binders extracted from field cores:

- Carbonyl absorbance using infrared (IR) spectrometry
- Carbonyl + sulfoxide absorbance using IR spectrometry
- Dynamic shear crossover modulus (G_c^*) using the DSR
- Viscosity (η) using the DSR

Subtask 3.3.2: Sensitivity Study

The purpose of the sensitivity study is to identify the sensitivity of the asphalt mixture properties to changes in the asphalt binder AIPs. The findings of the sensitivity study will be used to determine the acceptable level of accuracy that is needed to match the target field AIPs using the developed laboratory aging procedure without significantly impacting the mixture performance characteristics. A partial factorial experiment is proposed that will identify the key trends and meet the study objectives. Dynamic modulus and fatigue experiments will be conducted to identify the fundamental damage behavior in accordance with the VECD method. This approach is advantageous because, as the PI and members of the ASU research team have shown, it can be used to extract the fracture properties and thermal cracking behavior (Chehab and Kim 2005) of asphaltic composites.

Table 20 outlines the research team's current plan to meet the objectives. The naming convention used in Table 20 indicates the asphalt binder type (B1, B2, or B3), the aggregate source (A1 or A2), and the gradation (G1 through G5) of the materials. The experimental plan involves creating mastic and fine aggregate matrix (FAM) in the laboratory using asphalt binders that have been pre-aged to various levels that represent a range of AIP values that have been obtained from the field cores.

Table 20. Proposed testing plan for AIP sensitivity assessment

Group	FAM Name[a]	Mastic/Binder Name[a]	Air Voids (%)	Asphalt Aging Levels (yr)	Evaluation Purpose	Experimental Description
A-1	B1A1G1	--	8	15	Interactive effects of %AV and asphalt aging	• AASHTO TP79/PP61 • S-VECD Fatigue
	B1A1G5	--	8	7		
A-2	B1A1G2	--	4	7	Aggregate skeleton effects	
	B1A1G3	--	4	7		
	B2A1G5	--	4	15		
A-3	--	B1	N/A	15	Interactive effects of physico-chemical processes	• Temperature and frequency sweep at 10°C, 20°C, 30°C, 40°C, and 54°C • Controlled strain cyclic fatigue test
	--	B2	N/A	15		
	--	B3	N/A	15		
	--	B1A1G1	N/A	7		
	--	B1A1G5	N/A	15		
	--	B2A1G2	N/A	7		
	--	B2A1G4	N/A	15		
	--	B3A1G1	N/A	7		
	--	B3A1G2	N/A	15		
	--	B1A2G1	N/A	15		
	--	B2A2G2	N/A	15		
	--	B3A2G2	N/A	15		

Note: B1 = PG 58-28, B2 = PG 64-22, B3 = PG 76-16, A1 = Limestone, A2 = Granite, G1 = Coarse, G2 = Fine, G3 = Gap, G4 = Coarse Plus, G5 = Fine Plus

Figure 22 summarizes how the sensitivity study will address the development of the aging protocol and will address critical questions, and outlines the experimental comparisons that will be used to address those questions and meet the primary goal of the project. Analysis will consist of comparisons between the mechanical properties of the materials at different AIPs, and sensitivity will be quantified using

graphical and statistical analyses of the percentage of change in the mechanical properties with respect to changes in the AIP.
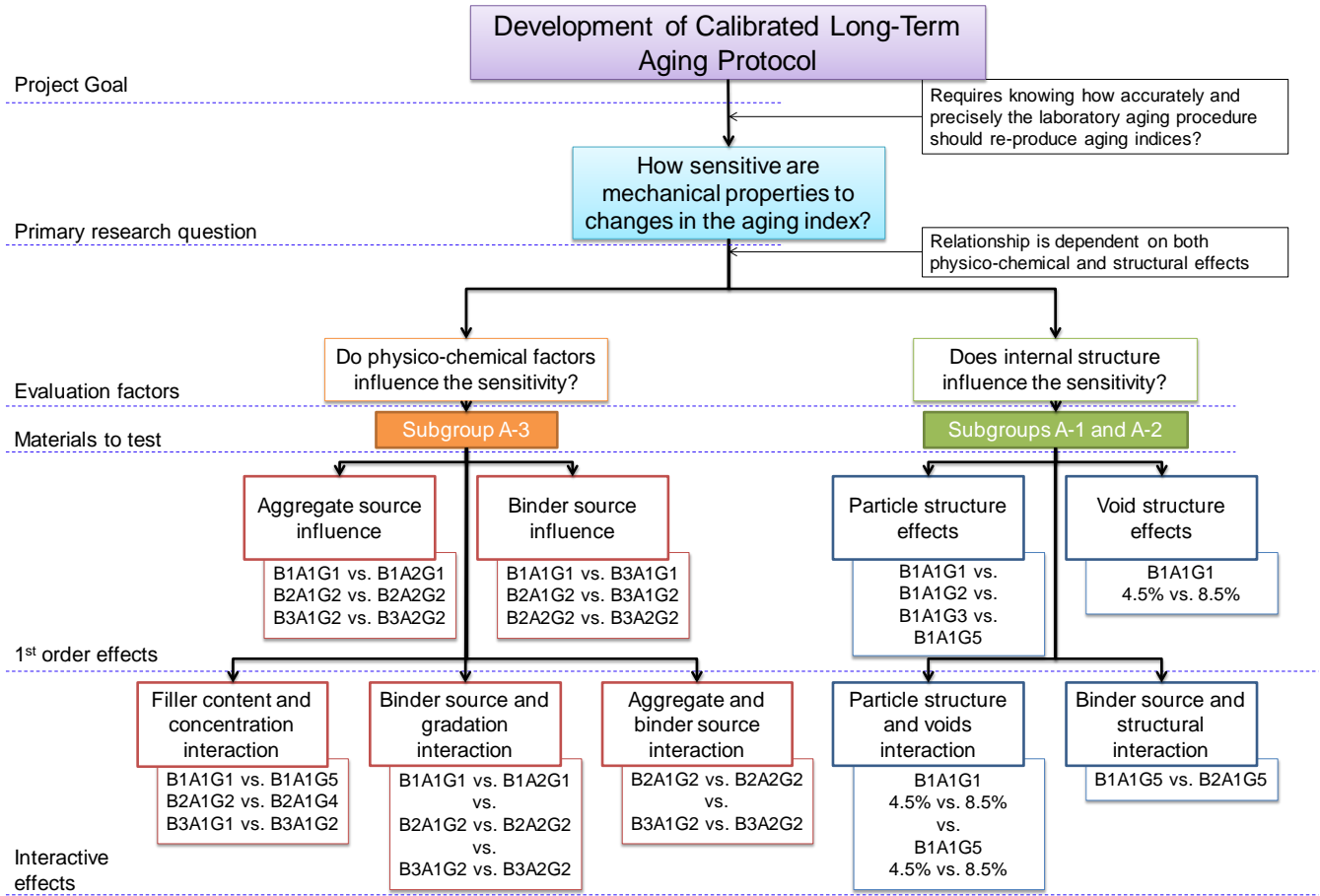
Figure 22. Summary of sensitivity study and its goals

In Subgroups A-1 and A-2, experiments will be conducted on the FAM materials for two reasons:

- These materials reflect much of the aggregate interaction effects that exist in asphalt mixtures.
- FAM samples are much smaller than asphalt mixture samples (19 mm diameter and 50 mm height versus 100 mm diameter and 150 mm or 130 mm height) and thus require substantially less asphalt binder, which will make it possible to create test samples using artificially-aged asphalt binder.

A method to create the FAM test samples in a way that represents the FAM as it exists in the asphalt mixture has been developed and verified (Underwood and Kim 2013b). Subgroup A-1 will be used to study the interactions among air void content, gradation, and aging, and Subgroup A-2 materials will be used to supplement the Subgroup A-1 findings and evaluate the structural effects more comprehensively. The final Subgroup A-3 consists of asphalt binder and asphalt mastic and is used to study the physico-chemical effects. This subgroup contains considerably more combinations than the other two subgroups

because, based on the literature review, physico-chemical interactions constitute the primary factor that contributes to sensitivity differences and aging potential within the asphalt mixture, and because the physico-chemical processes are not as well understood as the structural changes. This larger number of conditions is not expected to impose any major scheduling problems because tests with asphalt binder and mastic can be completed in considerably less time than equivalent tests on asphalt FAM or asphalt mixtures. To maximize the resources and gain the most insight possible, different gradations of the granite and limestone aggregates will be evaluated. Although the intent here is not to carry out full regressive analysis, clearly the experimental plan has been developed so that interactive effects can be examined directly. A complete interaction analysis will not be needed because the goal here is to evaluate sensitivity and not to develop a comprehensive link between the asphalt mixture and binder.

The outcome of this subtask will be the determination of tolerance limits for accuracy required in matching laboratory-aged to field core AIP values.

Subtask 3.3.3: Development of the Optimal Aging Protocol

This subtask is designed to develop an optimal long-term aging protocol that can be tailored to match the aging level of a pavement, location, and depth of interest. For this subtask, the Group B component materials and cores from the field sections will be used to prepare laboratory-compacted specimens with air void levels matching those of the field cores. Findings from the previous tasks will be used to select three aging durations for use with the selected aging protocol for the laboratory aging of the Group B materials included in Table 15.

Evaluation Criteria

The binder AIPs of the laboratory-aged mixtures will be compared to those of the cores extracted from the field at various age levels and depths. Figure 23 depicts this process. The AIP values measured from the field will give an indication of the AIP values at varying age levels and depths. The three laboratory aging trials will provide three AIP values, and a comparison with the field core AIP measurements using interpolation will allow for the estimation of the level of aging achieved for each laboratory aging trial.
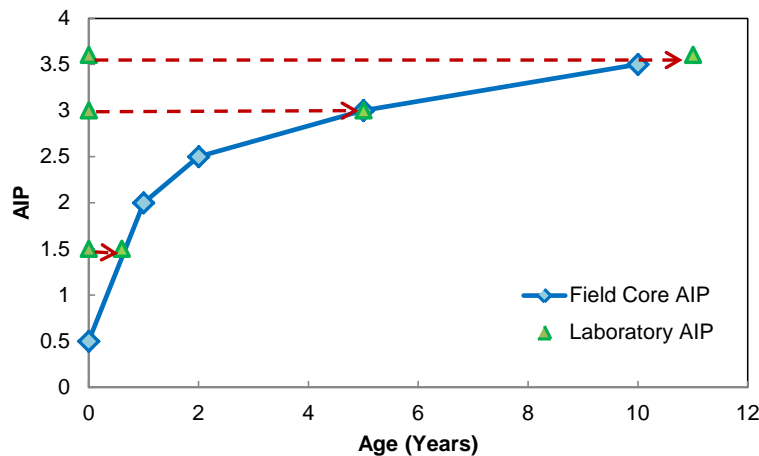

Figure 23. Depiction of proposed approach to estimate level of aging achieved in the laboratory

In addition to the laboratory aging experiments, efforts will be made to develop and calibrate a diffusion/kinetics model using the AIPs from the lab-aged and field cores. This model will be able to estimate the required duration for the developed protocol to match any desired level of aging, which will be a function of pavement age, climate, depth, air void content, and binder type.

Laboratory measurements required for the diffusion/kinetics model are summarized as follows:

1. The total air void content will be measured using a saturated surface dry method and/or the Corelok® method.

2. The percentage of accessible air voids will be evaluated using the Corelok® method.

3. Changes in rheological parameters, such as limiting viscosity and the crossover modulus, will be traced using DSR measurements of extracted and recovered asphalt binders.

4. Changes in chemical functional groups, such as carbonyl and carbonyl + sulfoxides, need to be evaluated using FTIR spectroscopy measurements on extracted and recovered asphalt binders.

Based on the proposed timeline for this project, it is estimated that the aging procedure development using the Group B materials will be accomplished over the course of 12 months and that the validation/calibration process (i.e., Task 3.4 and Task 3.5) will be conducted during the subsequent 12 months.

By the end of this task, the optimal aging procedure with corresponding calibrated diffusion/kinetics model will be developed. The completion of this task will allow for the determination of the appropriate laboratory aging procedure operating parameters to match any field condition of interest, including age, climate, and depth.

Task 3.4: Validation of the Optimal Aging Protocol

In order to validate the developed optimal long-term aging protocol, a subset of the Group B materials will be used. The sections selected for validation are specified in Table 21. For each section selected, the developed diffusion/kinetics model will be used to determine the laboratory aging operating conditions to match the level of oxidation of a corresponding field core of a specific age and depth (also a function of the geographic location of the section and the mixture air void content). The AIP values of the binders from the laboratory-aged mixtures will be compared to those from the field cores at the target age level. Based on the tolerance limit defined in the sensitivity study, the ability of the developed aging protocol to simulate the field conditions can be assessed. The flowchart for Tasks 3.4 and 3.5 are presented in Figure 24.

The outcome of this subtask will be validation of the developed long-term aging protocol.

```
┌─────────────┐        ┌─────────────┐        ┌─────────────┐
│  Group B    │        │ Group B     │        │  Group B    │
│ Component   │        │ Field Cores │        │ Component   │
│ Materials   │        └─────────────┘        │ Materials   │
└─────────────┘                               └─────────────┘
```
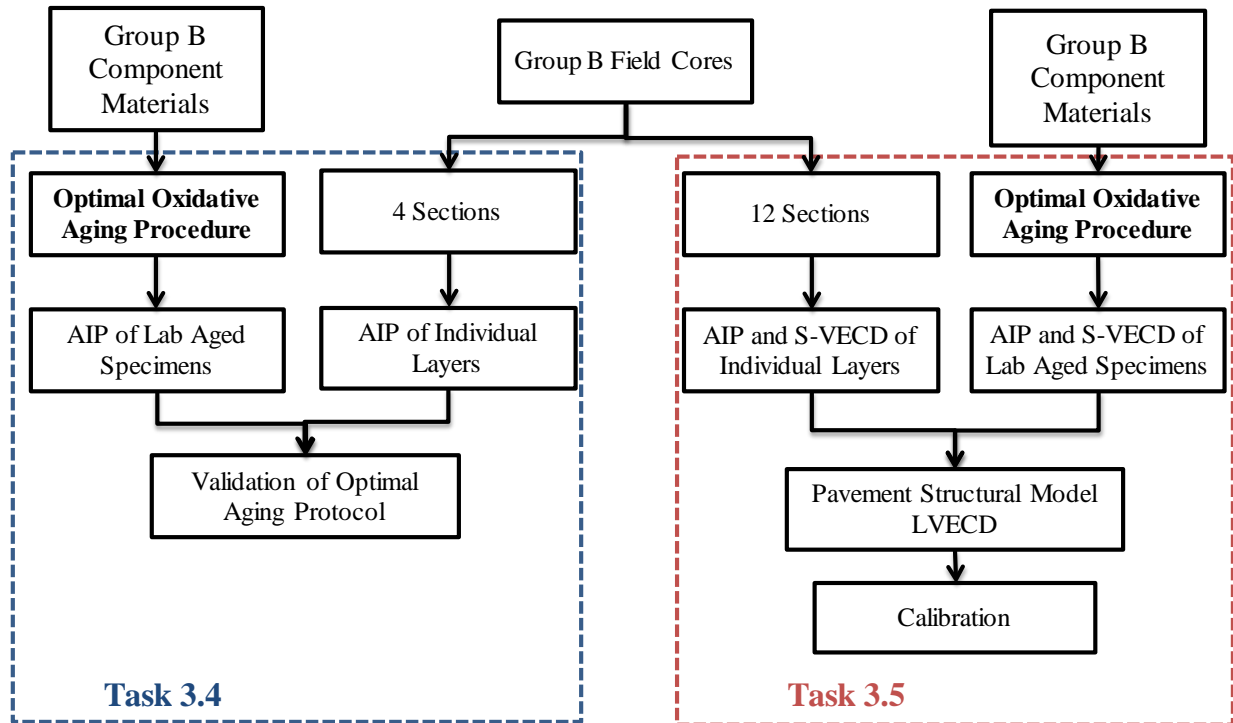
Figure 24. Flowchart for Task 3.4: Validation of the optimal long-term aging protocol and Task 3.5: Development of calibration functions for traffic, thermal, and moisture conditions

Task 3.5: Development of Calibration Functions for Traffic, Thermal, and Moisture Conditions

In order to develop a long-term aging procedure that can be used for performance testing and prediction, a set of performance tests will be conducted on the laboratory-aged materials and on the corresponding layers of the field cores using the 38 mm diameter small specimen geometry. The 38 mm specimen geometry was suggested by the Federal Highway Administration (FHWA) in its forensic evaluation of Accelerated Loading Facility (ALF) pavements (Mbarki et al. 2012) and further developed by the North Carolina State University (NCSU) research team for the forensic study of fatigue cracking of pavements in North Carolina.

The performance tests will include the dynamic modulus test and the S-VECD fatigue test. Both tests will be performed in direct tension mode. The material properties to be determined from these tests are the dynamic modulus mastercurve and the damage characteristic relationship. It is expected that the properties determined from the laboratory-aged specimens would be different from those determined from the field cores. In order to assess the effects of these differences on the pavement cracking performance, the material properties determined from each of the laboratory-aged specimens and field cores will be input to the Layered ViscoElastic analysis for Critical Distresses (LVECD) program developed by the NCSU research team as part of the FHWA DTFH61-08-H-00005 *HMA Performance-Related Specifications Based on VEPCD Models* project. Changes in the damage contours during the 20-year service life and the predicted fatigue life will be used to assess the effects of the differences in the

material properties. This assessment will allow for the development of calibration functions to account for differences other than oxidation (e.g., traffic, thermal, and moisture) in mixture performance.

Twelve Group B sections have been selected for calibration purposes, as shown in Table 21. These sections were selected not only based on the availability of component materials and extracted field cores but also to include different climatic regions and a broad range of material types.

The outcome of this subtask will be calibration functions to account for differences between field core and laboratory-aged specimens due to traffic, thermal, and moisture conditioning.

Table 21. Sections selected for validation and calibration stages

Work Task	Section ID	Date Built	Date Core Extracted
Validation/Calibration	FHWA ALF – CR-TB	2001	2013
Calibration	NCAT – Control	2009	2013
Calibration	NCAT – Advera WM	2009	2013
Calibration	NCAT – 50% RAP	2009	2013
Validation/Calibration	Ohio, US-23 MP 20.9	1995	2006, 2012
Calibration	California	1999	2007
Validation/Calibration	New Mexico	1996	2006
Validation/Calibration	Washington	1995	2006
Calibration	Wisconsin, SR-29	1997	2005
Calibration	WesTrack Section 1	1995	1995,1997,1999
Calibration	WesTrack Section 4	1995	1995,1997,1999
Calibration	WesTrack Section 17	1995	1995,1997,1999

Time Requirements

Task Description	2013			2014												2015												2016				
	10	11	12	1	2	3	4	5	6	7	8	9	10	11	12	1	2	3	4	5	6	7	8	9	10	11	12	1	2	3	4	5
	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
Panel Review of the Experimental Plan	■	■																														
Panel Meeting for the Experimental Plan		■																														
Task 3. Execution of the Approved Experimental Plan		■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■					
Task 3.1: Acquisition of Materials and Cores		■	■	■	■																											
Task 3.2: Establishment of the Preliminary Aging Procedure			■	■	■	■	■	■	■																							
Task 3.3: Development of the Optimal Long-Term Aging Procedure			■	■	■	■	■	■	■	■	■	■	■	■	■																	
Task 3.4: Validation of the Optimal Long-Term Aging Protocol																				■	■	■	■	■								
Task 3.5: Development of Calibration Functions for Thermal and Moisture																				■	■	■	■	■								
Panel Meeting for Progress Evaluation									■																							
Task 3.6: Evaluation of the Effects of Aging on Cracking in Asphalt Pavements and Development of Future Research Plan																									■	■						
Task 3.7: Development of AASHTO Standard for Long-Term Aging of Asphalt Concrete																											■	■				
Task 3.8: Cost and Benefit Analysis																											■					
Task 4. Submission of a Final Report																												■	■	■	■	
Panel Review of the Final Report																															■	■

References

AASHTO, R30. (2002). *Standard Practice for Mixture Conditioning of Hot Mix Asphalt.*

Airey, G. D. (2003). State of the Art Report on Ageing Test Methods for Bituminous Pavement. *International Journal of Pavement Engineering, Vol. 4,* (No. 3), 165-176.

Airey, G. D., Choi, Y. K., Collop, A. C., and Elliot, R. C. (2003). *Development of an Accelerated Durability Assessment for High Modulus Base (HMB) Materials.* 6th RILEM symposium PTEMB'03. pp160-166.

Asphalt Research Consortium. (2011). *Quarterly Technical Progress Report.* Western Research Institute, October - December.

Bahia, H., and Anderson, D. (1995). *The Pressure Aging Vessel (PAV): A Test to Simulate Rheological Changes Due to Field Aging.* West Conshohocken, PA: ASTM Special Technical Publication 1241, Hardin, J.C., ed. American Society for Testing and Materials.

Bell, C. A. (1989). *Summary Report on Aging of Asphalt-aggregate Systems.* No. SHRP-A-305.

Bell, C. A., and Sosnovske, D. (1994). *Aging: Binder Validation.* Strategic Reshearch Program Report no. SHRP-A-384, National Research Council. Washington, DC.

Bell, C. A., AbWahab, Y., Cristi, R. E., and Sognovske, D. (1994). *Selection of Laboratory Aging.* SHRP-A-384, Strategic Highway Research. Washington, D.C.

Bell, C. A., Wieder, A. J., and Fellin, M. J. (1994). *Laboratory Aging of Asphalt-Aggregate Mixtures:Field Validation.* SHRP-A-390, Strategic Highway Research Program, National Research Council. Washington, D.C.

Bell, C. A., Fellin, M., and Wieder, A. (1994). Field Validation of Laboratory Ageing Procedures for Asphalt Aggregate Mixtures. *Journal of the Association of Asphalt Paving Technologists*, Vol. 63, pp 45-80.

Brown, S. F., and Scholz, T. V. (2000). Development of Laboratory Protocols for the Aging of Asphalt Mixtures. *I*, pp. vol. 1, pp. 83-90. 2nd Eurasphalt and Eurobitume Congress.

Casola, J. R. (2008). *Development of a Simple Procedure for Selecting Mixing and Compaction Temperatures.* Austin, TX: Association of Modified Asphalt Producers.

Collop, A., Choi, Y., Airey, G., and Elliott, R. (2004). *Development of a Combined Aging / Moisture Sensitivity Laboratory Test.* Vienna: 4th Euroasphalt and Eurobitume Congress.

Corbett, L. W., and Merz, R. E. (1975). *Asphalt Binder Hardening in the Michigan Test Road after 18 Years of Service.* Washington, D.C., pp. 27-34.: Transportation Research Record, 544, National Research Council.

De la Roche, C., Van de Ven, M., Van den Bergh, W., Gabet, T., Dubois, V., Grenel, J., and Porot, L. (2009). *Development of a Laboratory Bituminous Mixtures Aging Protocol.* Advanced Testing and Characterization of Bituminous Materials 331.

Farrar, M. J., Turner, T. F., Planche, J. P., Schabron, J. F., and Harnsberger, P. M. (2013). *Evolution of the Crossover Modulus with Oxidative Aging.* Washington, DC: Transportation Research Board.

Gatchalian, D., Masad, E., Chowdhury, A., and Little, D. (2006). Characterization of Aggregate Resistance to Degradation in Stone Matrix Asphalt Mixtures. *Transportation research record*, 55-63.

Glaser, R. R., Schabron, J. F., Turner, T. F., Planche, J. P., Salmans, S. L., and Loveridge, J. L. (2013). *Low Temperature Oxidation Kinetics of Asphalt Binders.* Washington, DC: Transportation Research Board.

Glover, C. J., Martin, A. E., Chowdhury, A., Han, R., Prapaitrakul, N., Jin, X., and Lawrence, J. (2008). *Evaluation of Binder Aging and its Influence in Aging of Hot Mix Asphalt Concrete: Literature Review and Experimental Design.* Texas: Report 0-6009-1.

Hachiya, Y., Nomura, K., and Chen, J. (2003). *Accelerated Aging Test for Asphalt Concretes.* Zurich: 6th RILEM Symposium PTEBM.

Hagos, E. (2008). *The Effect of Aging on Binder Proporties of Porous Asphalt Concrete.* PhD DiS 'SEP'I 'tZll'0nS, Delft University of Technology, the Netherlands.

Han, R. (2011). *Improvement to a Transport Model of Asphalt Binder Oxidation in Pavements: Pavement Temperature Modeling, Oxygen Diffusivity in Asphalt Binders and Mastics, and Pavement Air Void Characterization.* Collage Station, TX: Texas A&M University.

Harrigan, E. T. (2007). *Research Results Digest 324 - Simulating the Effects of Mot Mix Asphalt Aging for Performance Testing and Pavement Structural Design.* National Cooprative Highway Research Program.

Hubbard, P., and Reeve, C. S. (1913). The Effect of Exposure on Bitumens. *Journal of Industrial and Engineering Chemistry*, 5(1), pp. 15-18.

Hurley, G. C., and Prowell, B. D. (2005). *Evaluation of Aspha-min® Zeolite for Use in Warm Mix Asphalt.* Auburn, Alabama: NCAT Report 05-04.

Hveem, F., Zube, E., and Skog, J. (1963). *Proposed New Tests and Specifications for Paving Grade Asphalt.* Proceedings of the Association of Asphalt Paving Technologists, Volume 32. AAPT, Minnesota, USA.

Jemison, H. B., Burr, B. L., Davison, R. R., Bullin, J. A., and Glover, C. J. (1992). Application and Use of the ATR, FT-IR Method to Asphalt Aging Studies. *Petroleum Science and Technology Journal*, 10(4), pp. 795-808.

Khalid, H. A., and Walsh, C. M. (2000). Relating Mix and Binder Fundamental Properties of Aged Porous Asphalt Materials. *Proceedings of the 2nd Eurasphalt and Eurobitume Congress*, *1 , pp. 398-405.*

Kim, O. K., Bell, C., Wilson, J., and Boyle, G. (1986). *Effectof Moistureand Aging on AsphaltPavement Life, Part 2 - Effectof Aging.* FHWA-OR-RD-86-01-2, Final Report to Oregon Departmentof Transportationand the Federal Highway Administration.

Korsgaard, H. C., Blumensen, J., Sundahl, J., and Gonzales, C. (1996). Accelerated Ageing of Asphalt in Pressure Ageing Vessel. *Eurasphalt and Eurobitume Congress*, (p. VOLUME 1. PAPER E&E. 4.048). Strasbourg.

Kumar, A., and Goetz, W. (1977). Asphalt Hardening as Affected by Film thickness, Voids, and Permeability in Asphaltic Mixtures. *Proceedings, Association of Asphalt Paving Technologists*, (pp. Vol. 46, 571-605.).

Lee, D. Y., and Huang, R. J. (1973). Weathering of Asphalts as Characterized by Infrared Multiple Internal Reflection Spectra. *Journal of Applied Spectroscopy*, 27(6), pp. 419-490.

Liu, M., Lunsford, K. M., Davison, R. R., Glover, C., and Bullin, J. A. (1996). The Kinetics of Carbonyl Formation in Asphalt. *AlChE Journal*, 42(4), pp. 1069-1076.

Lunsford, K. M. (1994). *The effect of temperature and pressure on laboratory oxidized asphalt films with comparison to field aging.* College Station, TX: Texas A&M University.

Mirza, M. W., and Witczak, M. W. (1995). Development of Global Aging System for Short and Long Term Aging of Asphalt Cements. *Journal of the Association of Asphalt Paving Technologists, 64*, 393-430.

Mollenhauer, K., and Mouillet, V. (2011). *Re-road – End of Life Strategies of Asphalt Pavements.* European Commission DG Research.

Mouillet, V., Durrieu, J. L., Kister, J., and Martin, D. (2003). *Development of a New Methodology for Characterization of Polymer Modified Bitumens Aging by Infrared Microspectrometry Imaging.* Zurich: The 6th International RILEM Symposium, PTEBM.

Mugler, M. (1970). *Die veranderung der Bitumeneigenschaften wabrend der Verarbeitung und in Fertigen Belag.* Route et Traffic, 6.

Nicholls, C. (2006). *Analysis of Available Data for Validation of Bitumen Tests.* Forum of European National Highway Research Laboratories, ed. Cliff Nicholls, TRL. UK: Report on Phase 1 of the BiTVal Project, Forum of European National Highway Research Laboratories.

Partl, M. N., Bahia, H. U., Canestrari, F., De la Roche, C., Di Benedetto, H., Piber, H., and Sybilski, D. (2013). *Advances in Interlaboratory Testing and Evaluation of Bituminous Materials.* RILEM. RILEM.

Petersen, J. C. (1993). Asphalt oxidation — an overview including a new model for oxidation proposing that physicochemical factors dominate the oxidation kinetics. *Fuel Sci Technol Int*, 11:57–87.

Petersen, J. C. (1998). A Dual, Sequential Mechanism for the Oxidation of Petroleum Asphalts. *Petroleum Science and Technology*.

Petersen, J. C. (2009). *A Review of the Fundamentals of Asphalt Oxidation.* Washington, D.C.: Transport Research Board.

Petersen, J. C., and Glaser, R. (2011). Asphalt Oxidation Mechanisms and the Role of Oxidation Products on Age Hardening Revisited. *12*, pp. 795 - 819. Lavoisier, Paris: Road Materials and Pavement Design.

Petersen, J. C., and Harnsberger, P. M. (1998). *Asphalt Aging: Dual Oxidation Mechanism and Its Interrelationships with Asphalt Composition and Oxidative Age Hardening.* Washington, D.C.: Transportation Research Record, 1638, National Research Council.

Petersen, J. C., Hansberger, P. M., and Robertson, R. E. (1996). Factors Affecting the Kinetics and Mechanisms of Asphalt Oxidation and the Realative Effects of Oxidation Products on Age Hardening. *Preprints of Papers, American Chemical Society, Division of Fuel Chemistry 41.CONF-960807.*

Piérard, N., and Vanelstraete, A. (2009). *Développement d'une méthode d'essai pour vieillir de manière accélérée les enrobés bitumineux en laboratoire.*

Prapaitrakul, N. (2009). *Toward and Improved Model of Asphalt binder Oxidation in Pavements.* Collage Station, TX: Texas A&M University.

Prapaitrakul, N., Han, R., Jin, X., and Glover, C. J. (2009). *A Transport Model of Asphalt Binder Oxidation in Pavements.* Road Materials and Pavement Design.

Read, J., and Whiteoak, D. (2003). *The Shell Bitumen Handbook. 5th Ed.* London: Thomas.

Reed, J. (2010). *Evaluation of the Effects of Aging on Asphalt Rubber Pavements.* Masters Thesis, PhD Dissertation., Arizona State University.

Reinke, G. (2003). Determination of Mixing and Compaction Temperature of PG Binders Using a Steady Shear Flow Test. *Presentation Made to the Superpave Binder Expert Task Group.* https://engineering.purdue.edu/~spave/old/Technical%20Info/Meetings/Binder%20ETG%20Sept%2003%20Las%20Vegas,%20NV/Reinke_MIX%20AND%20COMPACTINO%20INFO%20FOR%20ETG%209-15-03.pdf.

Such, C., Ballie, M., Lombardi, B., Migliori, F., Ramond, G., Samanos, J., and Simoncelli, J. P. (1997). *Susceptibilite au vieillissement des bitumen - Experimentation A08.* LCPC. Paris: Bitume, GNB - Groupe National.

Thurston, R., and Knowles, E. (1936). Oxygen Absorption Tests on Asphalt Constituents. *Journal of Industrial and Engineering Chemistry*, 28(1), pp88-91.

Tia, M., Ruth, B. E., Charai, C. T., Shiau, J. M., Richardson, D., and Williams, J. (1988). *Investigation of Original and In-service Asphalt Properties for the Development of Improved Specifications – Final Phase of Testing and Analysis.* Final Report, Engineering and Industrial Experiment Station. Gainesville, FL: Final Report, Engineering and Industrial Experiment Station.

Underwood, B. S., Sakhaei Far, M. S., and Kim, Y. R. (2010). *Using Limited Purchase Specification Tests to Perform Full Viscoelastic Characterization of Asphalt Binder.* ASTM Journal of Testing and Evaluation.

Van den Bergh, W. (2009). *The Development of an Artificially Aged Asphalt Mixture (in Dutch): Artificial Aged Asphalt mixture.* Gent, Belgium: In: Belgian Road Congress.

Van den Bergh, W. (2011). *The Effect of Aging on Fatigue and Healing Properties of Bituminous Mortars.* Ph.D Thesis, Delft University of Technology, Delft University.

Van Gooswilligen, G., De Bats, F., and Harrison, T. (1989). Quality of Paving Grade Bitumen - a Practical Approach in Terms of Functional Tests. *Proc. 4th Eurobitume Symp.*, pp. 290-297.

Van Oort, W. P. (1956). Durability of Asphalt – It's Aging in the Dark. *Journal of Industrial and Engineering Chemistry.*

Von Quintas, H., Scherocman, J., Kennedy, T., and Hughes, C. (1992). *Asphalt Aggregate Mixture Analysis System (AAMAS).* NCHRP. NCHRP 9-6(1).

West, R. C., Watson, D. E., Turner, P. A., and Casola, J. R. (2010). *Mixing and compaction temperatures of asphalt binders in hot-mix asphalt.* National Cooperative Highway Research Program. No. Project 9-39.

Wright, J. R. (1965). Weathering: Theoretical and Practical Aspects of Asphalt Durability. In *Chapter 8 in Bituminous Materials: Asphalts, Tars and Pitches* (p. Volume II). New York: Ed. A.J. Hoiberg. Interscience Publishers.

Yildirim, Y., Soaimanian, M., and Kennedy, T. (2000). *Mixing and Compaction Temperatures for Hot Mix Asphalt.* University of Texas, Center for Transportation Research, Research Report #1250-5.