

## An Abstract of the Thesis of

Daniel A. Sosnovske for the degree of Master of Science in Civil Engineering presented on December 20, 1994. Title: Laboratory Evaluation of Aging for Asphalt-Aggregate Mixtures.

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Abstract Approved: \_\_\_\_\_  
Chris A. Bell

This research was conducted as part of the Strategic Highway Research Program (SHRP) A-003A contract at Oregon State University to validate the findings of SHRP contracts A-002A and A-003B with regard to aging of asphalt-aggregate mixtures. One short-term and four long-term aging methods were used to simulate aging of asphalt-aggregate mixes in the field. Four aggregates and eight asphalts for a total of 32 different material combinations were tested using different aging methods. Results of the aging studies are compared with the A-002A and A-003B studies of asphalt binder or asphalt mixed with fine aggregate. This research concludes that aging of asphalt mixes cannot be predicted by tests on asphalt binder alone since results show that aggregates have considerable influence on aging.

Laboratory Evaluation of Aging for Asphalt-Aggregate Mixtures

by

Daniel A. Sosnovske

A THESIS

submitted to

Oregon State University

in partial fulfillment of  
the requirements for the  
degree of

Master of Science

Completed December 20, 1994  
Commencement June 1995

Master of Science thesis of Daniel A. Sosnovske presented on December 20, 1994

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## **Acknowledgements**

The research described herein was supported by the Strategic Highway Research Program (SHRP). SHRP is a unit of the National Research Council that was authorized by Section 128 of the Surface Transportation and Uniform Relocation Assistance Act of 1987.

I would like to express my appreciation to Dr. Chris Bell and Dr. Gary Hicks. Without their guidance and support this report would not have been completed. And also, to the other members of the OSU SHRP Research Team, whose support and encouragement made this project possible.

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# **LABORATORY EVALUATION OF AGING FOR ASPHALT-AGGREGATE MIXTURES**

## **1.0 Introduction**

### **1.1 Background**

The development of laboratory aging procedures for asphalt-aggregate mixes as a part of project A-003A of the Strategic Highway Research Program (SHRP) has been described previously by Bell et al. (1992a). The validation of these procedures is described in another report by Bell et al. (1992b).

The procedure developed for short-term aging involves heating the loose mix for 4 h at 135°C (275°F) in a forced draft oven, prior to compaction of laboratory specimens. This simulates the aging of the mix in the field during the construction process while it is in an uncompacted condition.

Alternate procedures have been developed for long-term aging of mixes. These simulate the aging of compacted mix for service periods of several years. The following approaches have been found to be appropriate:

- a) Long-term oven aging (LTOA) of compacted specimens in a forced draft oven.
- b) Low pressure oxidation (LPO) of compacted specimens in a triaxial cell by passing oxygen through the specimens.

Various combinations of temperature and time have been evaluated (Bell et al. 1992a).

The effects of aging were evaluated by resilient modulus at 25°C (77°F) using diametral (indirect tension) and triaxial compression modes of testing. Tensile strength tests were performed on specimens once all other data have been collected. A selection of specimens were subjected to a Dynamic Mechanical Analysis (DMA) testing program at different temperatures and loading frequencies.

## 1.2 Purpose

The purpose of this report is to describe a laboratory study designed to compare the results of aging of asphalt (done by the SHRP A-002A contractor) with the results of aging of asphalt-aggregate mixes (done by the A-003A contractor).

## 1.3 Scope

Following a description of the hypothesis for aging of asphalts (developed by the A-002A contractor), the experiment design for the laboratory test program is presented. The results of the test program and their analysis, including comparison with the A-002A results, are presented prior to the conclusions arising from the study. A series of appendices are included describing the aging procedures and the test methods used to evaluate aging.

## **2.0 Hypothesis for Aging of Asphalt**

The information presented in this chapter is included to satisfy a requirement of the research contract. All of the information presented in this chapter was developed by researchers at institutions other than OSU. The rankings presented in this chapter are hypothetical rankings based on chemical composition tests of the asphalt cements. The results in Chapter 5 are compared to basic asphalt viscosity data rather than the rankings presented in this chapter. The viscosity tests more closely resemble the physical testing mode used in this research program.

The asphalt contractors A-002A and A-003B were asked to rank the SHRP asphalts in terms of expected performance based on chemical and/or physical properties of the asphalt cements. Both Western Research Institute (WRI) and Pennsylvania State University (PSU) provided this information for 16 of the SHRP asphalts. These rankings, together with similar ones provided by contractor A-003B for water sensitivity, were to be validated by A-003A using simulative tests. This chapter summarizes the rankings put forth by the A-002A and A-003B contractors.

Since aging plays a role in the permanent deformation, fatigue, and thermal cracking of mixes, a summary of the hypothesis for each of these performance characteristics is presented below, as well as for aging and water sensitivity.

### **2.1 Chemical Properties - Western Research Institute**

The following is the ranking of expected performance of SHRP asphalts in terms of their chemical properties (Peterson et al. 1994). (Similar information is provided in Robertson, 1994). WRI has emphasized that the structural characteristics are primarily related to the physical (viscoelastic) properties and that

any given set of physical properties may be achieved by substantially different chemical compositions. Rankings by distress type are discussed, and a schematic illustrating the relationship among chemical and physical properties and pavement performance is shown in Figure 2.1.

### 2.1.1 Permanent Deformation

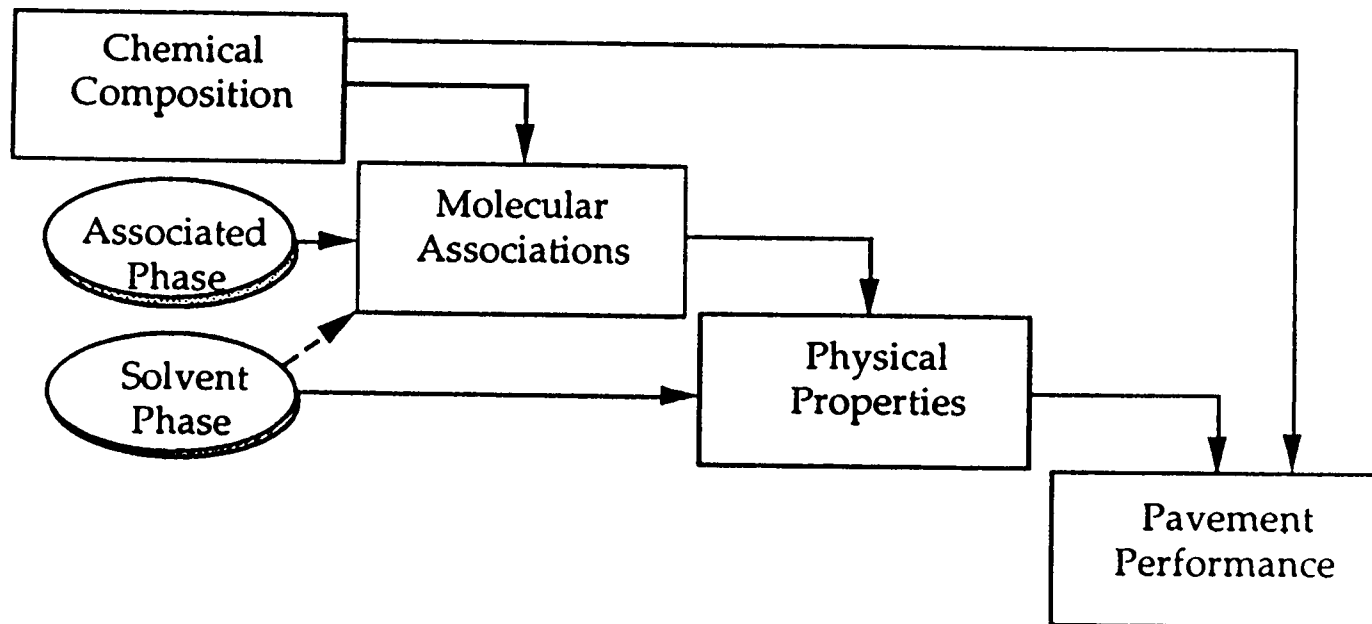
Permanent deformation of asphalt-aggregate mixes can be affected by asphalt type or by aggregate type and mix characteristics. It generally occurs at high temperatures because of shear stresses in the upper part of the pavement surface.

Size exclusion chromatography (SEC) fraction I/fraction II ratios have been found to be strongly related to permanent deformation. These ratios are evaluated on asphalts that have not experienced any long-term aging. The SEC ratio is the ratio of the weight of the non-fluorescent components (I) which appear to assemble into an elastic matrix to the weight of the fluorescent materials (II) which form the dispersing phase.

Figure 2.2 shows a plot of the SEC II fraction versus tan delta at 25°C (77°F). Table 2.1 is a ranking of the SHRP asphalts in terms of their resistance to permanent deformation.

### 2.1.2 Low Temperature Cracking

Thermal cracking in asphalt concrete requires two obvious situations 1) contraction (shrinkage) of some or all components of the concrete and 2) stiffening of the mix to a level that viscous flow to relieve strain caused by contraction cannot occur at a rate high enough to relieve the strain. Since the aggregate has effectively no viscous flow (at any service temperature), the ability to flow and



**Figure 2.1. Chemistry — physical property — performance relationships**

hence avoid cracking must be totally within the viscoelastic binder. However, aggregates may have various coefficients of thermal expansion, and hence contribute differentially to the overall contraction of pavement. So variation in aggregate may have an effect on the overall problem of thermal cracking.

Presuming that the binder is totally responsible for the creep properties, some chemical property or set of properties of the binder is (are) responsible for the variation in flow properties among different asphalts.

**Table 2.1. Ranking of high temperature permanent deformation and rutting by SEC-tan delta**

<b>Asphalt Type</b>	<b>Expected Performance</b>
AAM-1	Excellent Resistance
AAK-1	"
AAE	"
AAS-1	Very Good Resistance
AAH	"
AAD-1	"
AAB-1	"
AAW	"
AAJ	"
AAA-1	Good Resistance
AAN	"
AAX	
AAF-1	Fair Resistance
AAC-1	"
	"
AAZ	
AAV	Poor Resistance
	"
AAG-1	
ABD	Little or No Resistance
	"

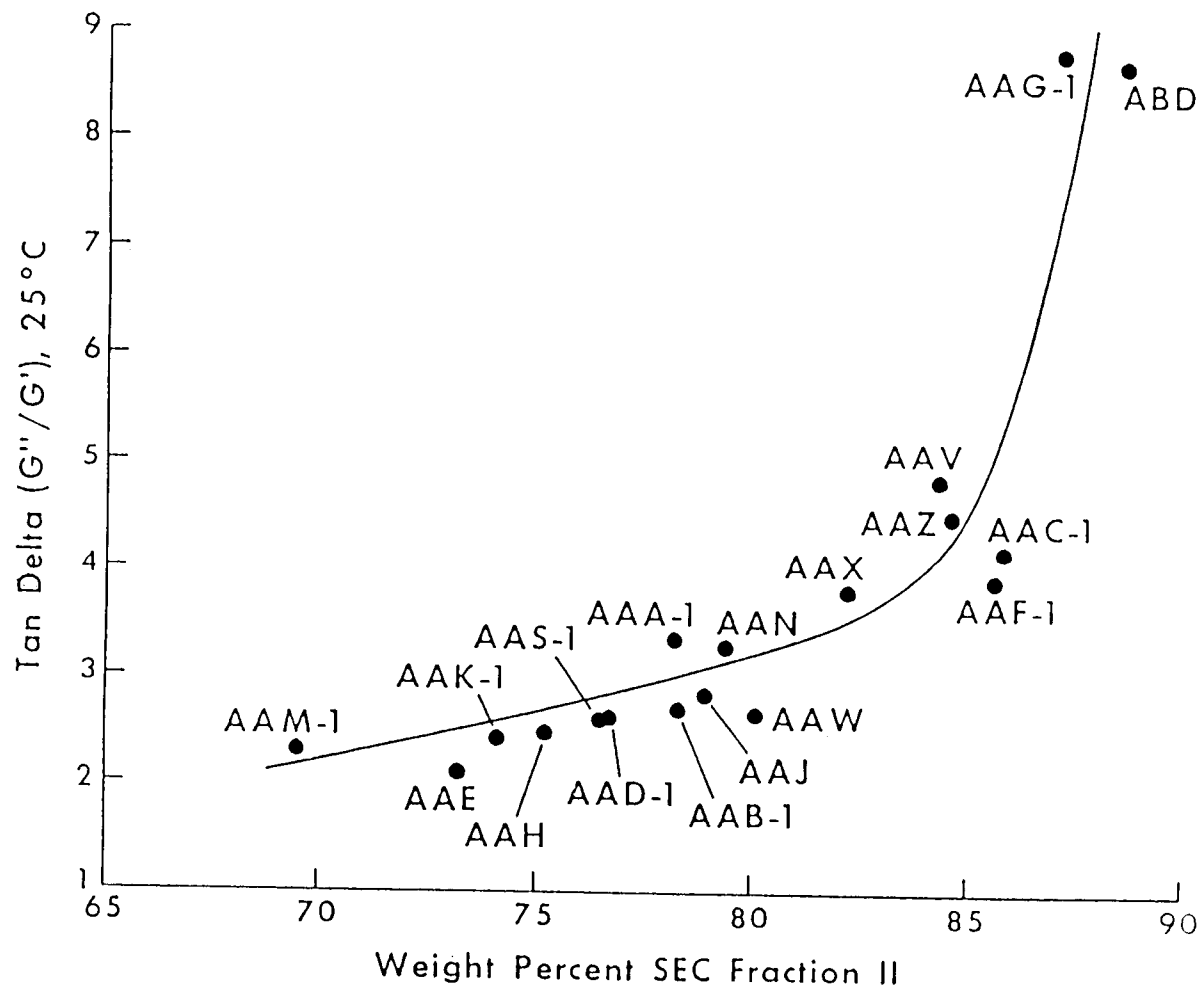


Figure 2.2. Relationship between weight percent sec fraction II and trans delta of SHRP asphalts

Viscous flow implies that the elastic matrix is not involved, especially considering the small amount of change involved. For example, a 1 in. (2.54 cm) crack every 50 feet (15.25 m) amounts to a 0.17 percent contraction. Elasticity should accommodate this amount of volume change. Further, thermodynamic data suggest that most organization of the polar matrix has been achieved at moderate and higher temperatures. A transition of the neutral materials to a glass with an accompanying contraction, similar to crystallization of non-polar materials, is a more likely cause. The stiffness, or rigidity, of homologous pseudocrystalline materials generally increases with molecular weight. The relative amounts may also have an effect, but ion exchange chromatography (IEC) experiments for the core asphalts show for the neutral range from 51 to 60 percent, relatively similar amounts. The supercritical fluid chromatography (SFC) profiles for the IEC neutrals of core asphalts are significantly different and show that the molecular weight profiles of the neutral components differ substantially. Chemical structural variations among neutrals may affect rigidity of a pseudocrystalline phase also, but no such detailed information on neutral components has been acquired systematically. An indication of structural effects is being sought at present using Fourier transformation infrared (FTIR) radiation.

The ranking of six of the eight core asphalts in table 2.2 is based on the molecular weight average by SEC. Although not shown in table 2.2, asphalt AAM-1 has a maximum at 68 carbons, but is an odd case with a very uniform and broad molecular weight range. Typically, such broad molecular weight mixes do not tend to (for a pseudocrystalline phases) and would not be expected to harden disproportionately as its carbon number might indicate. The presumption, based on the unique profile of AAM-1 neutral, is that it would show intermediate low temperature cracking properties because of the broad profile.

**Table 2.2. Ranking of SHRP asphalts in terms of their resistance to low temperature cracking**

Asphalt	Carbon Number at the Max of SFC Peak	Ranking
AAA-1	32	Least likely to low temp. crack
AAD-1	32	"
AAK-1	32	"
AAG	42	Moderate probability to low temp. crack
AAB	43	"
AAF	48	Most likely to low temp. crack

### 2.1.3 Fatigue Cracking

Fatigue cracking is typically associated with aged pavement, although it can occur in relatively new pavements. In relatively new pavement fatigue cracking, to any extent that it occurs, should be somewhat the opposite of rutting. That is, asphalts that have the smallest amounts of SEC II should have the greatest propensity to crack under stress of traffic. The matrix developed by a large SEC I would be expected to be too rigid. This is highly speculative and the best ranking available is the reverse order of the early rutting list. (Table 2.3)

Problems of fatigue cracking in aged asphalts are more likely than fatigue cracking in unaged binders. Based on Figure 2.2 the rankings were developed for 3 temperature regimes for the 8 core asphalts that are shown in Table 2.3. Each ranking also has a qualitative descriptor.

#### 2.1.4 Aging

Aging of asphalt is a complicated set of events involving oxidation at the molecular level and the additional chemical feature of structuring at the intermolecular level. The primary chemical species formed are ketones and sulfoxides. More severe oxidation produces carboxylic anhydrides and small amounts of other highly oxidized species. Structuring appears to quench further oxidative aging. The rate of aging decreases with time at any given temperature. As temperature is increased, the amount of structuring decreases, and reactivity increases. The resulting oxidation rate increases and the level of quenching decreases. At very extended times (severe oxidation aging), at least one of the core asphalts (AAK-1) shows extreme hardening, but it is not yet clear that this is still within normal road service conditions. It is speculated that the unique behavior of AAK-1 results from its extraordinary vanadium content. This suggests that vanadium may be a viable catalyst to oxidize asphalts rapidly, and hence provide a usable test method that is much faster than thin film oven/pressure aging vessel (TFO-PAV) for evaluation of oxidative aging.

In Figure 2.3, the aging index (AI) profiles for the 8 core asphalts are shown as a function of temperature between 60°C and 80°C (140° and 176°F). Note that the oxidative age hardening spreads dramatically over this 20°C (36°F) range. Note, too, that 176°F (80°C) was chosen as an upper limit. [Perhaps this should be 185°F (85°C).] At 140°F (60°C) the aging rates for all asphalts have diminished very significantly suggesting that below 55-60°C (131°-140°F) oxidative aging may be relatively insignificant.

Thus far, TFO-PAV is the chosen method to develop into a specification method for oxidative aging.

**Table 2.3. Resistance of aged asphalt to fatigue cracking**

<b>Moderate and Cool Climate 140°F and below surface temperatures</b>	<b>Intermediate Climate 155-165°F surface temperatures</b>	<b>Hot Climate 175°F higher surface temperatures</b>
AAA-1 Very resistant to fatigue cracking	AAG-1 Very resistant	AAG-1 Resistant to fatigue cracking
AAB-1 "		AAC-1 "
AAG-1 "	AAC-1 Resistant	
	AAB-1 "	AAB-1 Moderate susceptibility
AAK-1 Resistant to fatigue cracking	AAA-1 "	AAA-1 "
AAF-1 "	AAK-1 "	
AAC-1 "	AAF-1 "	AAF-1 Significant susceptibility
AAM-1 "		AAK-1 "
	AAM Moderate susceptibility fatigue cracking	AAM-1 "
AAD-1 Fair resistance to fatigue cracking	AAD "	
		AAD-1 Very susceptible to fatigue cracking

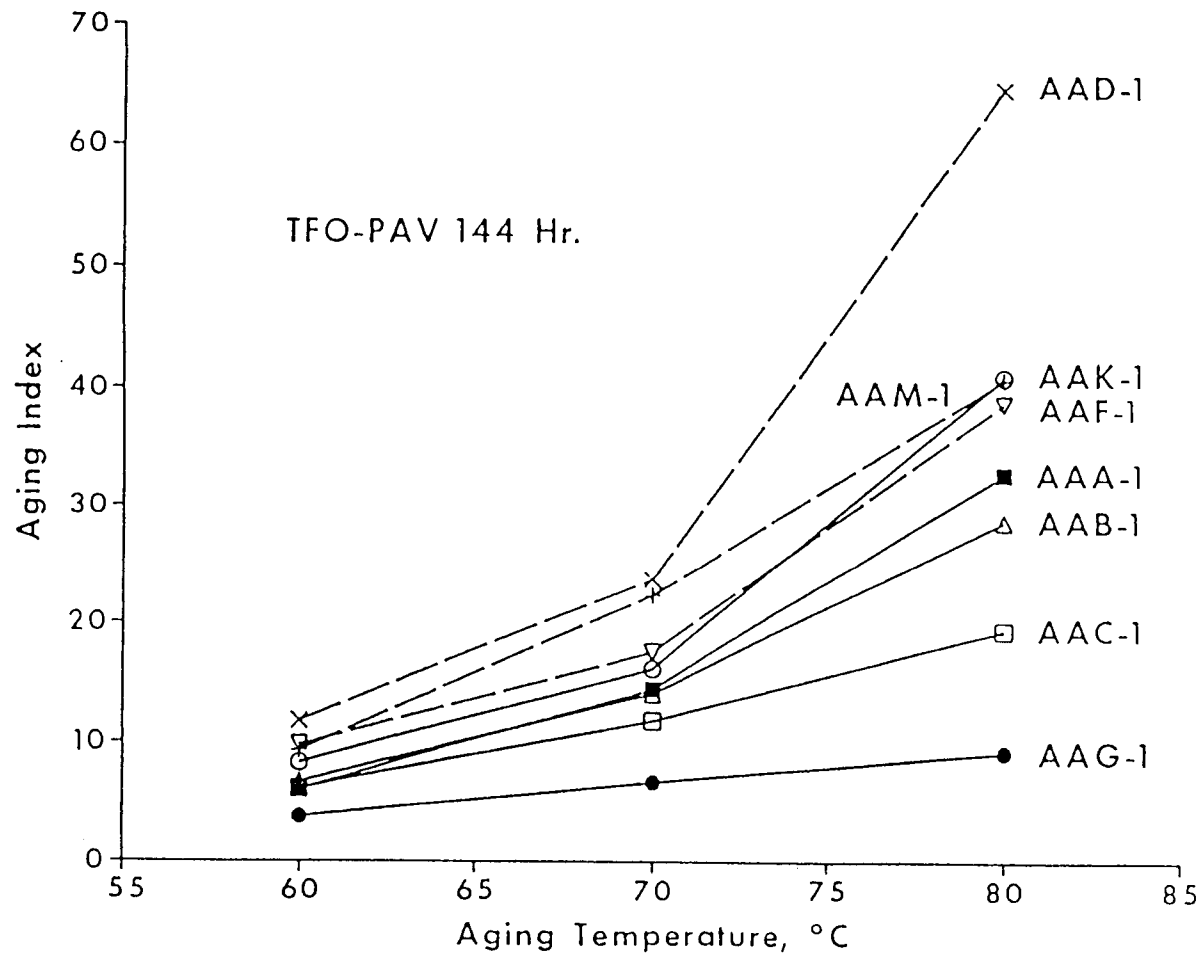


Figure 2.3. Aging indices at three temperatures of SHRP core asphalts

### 2.1.5 Moisture Damage (Loss of Adhesion)

Numerous studies and A-003B in particular have demonstrated that moisture damage causing loss of adhesion is primarily associated with aggregate. Classification of moisture damage susceptibility from the chemistry of only the binder is probably a minor effect at best. None the less, a highly speculative classification is shown in Table 2.4 based on the carbonyl content, with emphasis on the free acid content, as determined by FTIR. Note that aging affects the 8 core asphalts differently.

## 2.2 Physical Properties - Pennsylvania State University (PSU)

The Penn State rankings (see Robertson, 1994) consider three distress modes: low-temperature thermal shrinkage cracking, load associated fatigue, and rutting caused by plastic deformation in the upper layers of the hot-mix asphalt concrete. The actual rankings are presented in the attached set of tables. In some instances, where multiple parameters were selected for the ranking, the better understanding of the failure mechanisms and better models were developed by the other SHRP research programs.

**Table 2.4. Ranks of moisture damage resistance by IR of functional group analysis (limited to asphalt; aggregate not considered)**

New Material		Aged Material	
AAF-1	Good (no order established)	AAB-1	Good (in order as shown)
AAB-1	"	AAM-1	"
AAM-1	"	AAC-1	"
AAA-1	"	AAF-1	"
AAD-1 Intermediate - Good		AAD-1 Intermediate - Good	
AAK-1	"	AAA-1 Intermediate	
AAG-1 Fair - Poor		AAG-1 Poor	
ABD	Poor	ABD	Poor

For the performance parameters given in tables 2.5-2.10, a score has been assigned to each asphalt. This score is a value from 1 to 8, and is based on the parameter value for the selected asphalt and the observed range for that parameter. When more than one parameter has been given, an average score has been calculated for that test or failure mode. The total score given in the attached tables is based on this average, and has also been calculated on a scale of from 1 to 8. In all cases, a lower score is associated with better performance in the test or failure mode under consideration. Also provided in the attached tables is a rating, G (good), M (moderate), or P (poor). These ratings were assigned based on the total score; for total scores below 2.5, a "G" rating has been assigned, for total scores above 6.5, a "P" rating has been given. All intermediate scores have been given an "M" rating. These letter ratings are meant to assist in identifying asphalts showing extreme behavior in the various tests and failure modes.

### 2.2.1 Load Associated Fatigue Cracking

The mechanism that is responsible for fatigue cracking is not clear. At temperatures somewhat above 0°C (32°F), the asphalt cement behaves in a ductile manner and cracks do not propagate in a brittle manner. At lower temperatures the asphalt behaves in a brittle manner. Thus, the mechanism must be different in the region of ductile and brittle failure. Consequently, the binder properties that correlate with field behavior are probably different in the different flow regimes.

It is not clear at which temperature fatigue occurs in the field. If cumulative damage, in terms of a reduced modulus, etc., is the criterion, then a more inclusive failure than pure fatigue is implied. Thus, performance may be a function of multiple mechanisms and the ranking may not depend on a single parameter. To further complicate the matter, the ranking criterion, in the brittle or viscoelastic response region, must be different for controlled stress and controlled strain. Both modes of testing are included in the validation program:

- 1) Laboratory — Flexural Beam Fatigue, 20°C (68°F). At this temperature it was assumed that viscous deformation in the asphalt is the mechanism that is responsible for crack formation and propagation. If this is the case, the viscous component of the modulus ( $G_v$ ) at 20°C (68°F) should correlate with the laboratory fatigue test; fatigue performance should in this case improve with increasing values of the viscous modulus. The ranking is given in Table 2.5.

A further definition of the fatigue test should probably be included in the ranking: controlled stress, controlled strain, or controlled energy. These distinctions were not made in the rankings.

- 2) Laboratory — Flexural Beam Fatigue, 0°C (32°F). At this temperature, the asphalt can crack in a brittle manner with a brittle or viscoelastic response. Therefore, classic fatigue parameters for the asphalt cements should be related to the bending beam fatigue tests conducted on the mixes. Therefore, asphalt cement fatigue parameters ( $N_f$ ) and a measure of the stiffness at 0°C (32°F) are included in the ranking:
- a.  $N_f$ , Number of Cycles to Failure in Bending Beam Test
  - b.  $C^*$ , Complex Modulus @ 0°C (32°F)

The ranking is given in Table 2.6.

- 3) Laboratory — Slab on Elastic Foundation, 20°C (68°F). In this test the passing wheel probably creates shear stresses in the slab that are the primary cause of rutting and crack formation. At this temperature, plastic deformation in the mix is the primary cause of both the cracks and the ruts. Therefore, the viscous modulus ( $G_v$ , Viscous Modulus @ 20°C (68°F)) has been used in the ranking, a ranking of the asphalts is given in Table 2.7.
- 4) Field — Classic fatigue failure. As discussed above, this will only occur when the asphalt cement within a paving mix behaves in a brittle or highly viscoelastic manner. The gel point temperature, at a frequency of 10 rad/s, has been chosen as an estimate of the brittle-ductile transition temperature under traffic loading conditions. The higher this temperature, the greater should be the potential for fatigue in the field. The width (standard deviation) of the relaxation spectrum is related to the fracture toughness of the asphalt;

toughness, and fatigue performance, should generally increase with increasing spectrum width.

- a.  $T_{gp}$ , Temperature @ Gel Point
- b.  $S(\tau)$ , Width of Relaxation Spectrum

A ranking of the asphalts is given in Table 2.8.

**Table 2.5. Ranking of asphalts in load associated fatigue bending beam at 20°C (68°F)**

Asphalt	Log $G_v$ at 20°C (68°F) Pa	Score	Rating
AAA-1	6.37	8.0	P
AAB-1	6.99	4.5	M
AAC-1	6.99	4.5	M
AAD-1	6.66	6.4	M
AAF-1	7.44	2.1	G
AAG-1	6.98	4.6	M
AAK-1	7.26	3.1	M
AAM-1	7.63	1.0	G

**Table 2.6 Ranking of asphalts in load associated fatigue bending beam at 0°C (32°F)**

Asphalt	Nf @ 0°C (32°F) Cycles	Score	Log $G^*$ @ 0°C (32°F) Pa	Score	Average Score	Total Score	Rating
AAA-1	15064	8.0	7.403	1.0	4.5	4.0	M
AAB-1	22879	4.4	7.640	2.8	3.6	2.4	G
AAC-1	20147	5.6	7.934	5.1	5.4	5.5	M
AAD-1	23630	4.0	7.474	1.6	2.8	1.0	G
AAF-1	30115	1.0	8.047	6.0	3.5	2.2	G
AAG-1	20088	5.7	8.304	8.0	6.8	8.0	P
AAK-1	18537	6.4	7.749	3.7	5.0	4.9	M
AAM-1	26358	2.7	7.910	4.9	3.8	2.8	M

**Table 2.7. Ranking of asphalts in load associated fatigue wheel tracking test at 20°C (68°F)**

Asphalt	$G_v @ 20^\circ(68^\circ\text{F})$ Pa	Score	Rating
AAA-1	6.37	8.0	P
AAB-1	6.99	4.5	M
AAC-1	6.99	4.5	M
AAD-1	6.66	6.4	M
AAF-1	7.44	2.1	G
AAG-1	6.98	4.6	M
AAK-1	7.26	3.1	M
AAM-1	7.63	1.0	G

**Table 2.8. Ranking of asphalts in load associated fatigue field**

Asphalt	$T_w$ C	Score	$N_f @ 0^\circ\text{C}$ Cycles	Score	Average Score	Total Score	Rating
AAA-1	1	1.0	15064	8.0	4.5	5.2	M
AAB-1	7	4.5	22879	4.4	4.4	5.0	M
AAC-1	8	5.1	20147	5.6	5.4	7.3	P
AAD-1	2	1.6	23630	4.0	2.8	1.0	G
AAF-1	11	6.8	30115	1.0	3.9	3.7	K
AAG-1	9	5.7	20088	5.7	5.7	8.0	P
AAK-1	5	3.3	18537	6.4	4.9	6.0	M
AAM-1	13	8.0	26350	2.7	5.4	7.3	P

### 2.2.2 Rutting in Upper Layers of Hot-Mix Asphalt

Rutting is the result of accumulated permanent deformation in the mix; this should be related to the viscous deformation within the binder. Therefore, the viscous modulus has been used to rank the asphalts according to resistance to

rutting, both for the wheel tracking test and for the field. Rutting will, of course, also depend on the temperatures to which the paving mix is subjected; 45°C (113°F) was tentatively selected as a representative critical temperature for permanent deformation in the field. The viscous modulus ( $G_v$ ) at the test temperature is used for ranking the asphalts for performance in both the wheel tracking test and the field (Table 2.9).

**Table 2.9. Ranking of asphalts in rutting in hot-mix asphalt wheel tracking test at 5°C (41°F)**

Asphalt	Log $G_v$ @ 20°C (68°F) Pa	Score	Rating
AAA-1	6.37	8.0	P
AAB-1	6.99	4.5	M
AAC-1	6.99	4.5	M
AAD-1	6.66	6.4	M
AAG-1	7.44	2.1	G
AAG-1	6.98	4.6	M
AAK-1	7.26	3.1	M
AAM-1	7.63	1.0	G

**Table 2.10. Ranking of SHRP tank asphalts for resistance to low temperature cracking as indicated by A-002A (Robertson et al. 1994)**

Asphalt Type	Temperature @ S(t) = 200 MPa @ 2 h, °C	Ultimate Strain @ -26 °C, 2 h, %	Overall Rank 1 = Best
AAA-1	-31	3.1	1
AAB-1	-28	1.7	12
AAC-1	-25	1.5	15
AAE-1	-29	2.1	8
AAF-1	-21	1.2	20
AAG-1	-18	0.8	25
AAH-1	-32	2.1	6
AAJ-1	-25	1.5	15
AAK-1	-27	1.7	12
AAL-1	-30	2.8	3
AAM-1	-24	1.5	16
AAN-1	-24	1.5	15
AAO-1	-28	2.1	9
AAP-1	-27	2.2	9
AAQ-1	-24	1.1	18
AAR-1	-26	1.7	13
AAS-1	-27	2.0	11
AAT-1	-23	1.6	16
AAU-1	-23	1.7	16
AAV-1	-25	1.2	17
AAW-1	-22	1.6	17
AAX-1	-20	1.1	21
AAZ-1	-28	1.8	11
AAZ-1	-20	1.2	21
ABA-1	-29	2.4	7
ABC-1	-30	2.2	8
ABD-1	-15	0.5	28

### 2.2.3 Low-Temperature Thermal Shrinkage Cracking

The selected parameters for predicting thermal shrinkage cracking are the same for the restrained tensile strength test and for field conditions. The glass transition temperature has been selected as an indicator of the temperature at which stresses within the asphalt will begin to accumulate and the strain capacity will

decrease during cooling. The energy to failure at one percent strain has been included in these rankings as an indicator of the fracture toughness of the asphalt cement. The third parameter used in the rankings is the ratio of flexural stiffness at  $-15^{\circ}\text{C}$  ( $5^{\circ}\text{F}$ ) and 2 min after 96 h conditioning at the test temperature, to the value found after only 2 h conditioning. This has been chosen as an indicator of the potential for physical hardening. The ranking of the asphalts (Table 2.10) is based on the following properties:

1.  $T_g$ , Glass Transition Temperature
2. Energy to Failure at One Percent Strain
3. Physical Hardening at  $-15^{\circ}\text{C}$  ( $5^{\circ}\text{F}$ )

#### 2.2.4 Moisture Damage

Physical property tests are not available for ranking the moisture sensitivity of the asphalts.

#### 2.2.5 Aging

The Penn State researchers recommend that ranking the performance after aging is best measured by testing the asphalt cement and mixes for the specific performance-related parameters. Thus, it is recommended that fatigue, etc., tests be conducted on aged mixes. Correlations with a single point measurement of stiffness are not expected.

## 2.3 Auburn University - A003B

The following paragraphs are extracted from the A-003B executive summary to the project's final report (Curtis, Ensley, and Epps, 1993).

An important element of the SHRP A-003B research was to investigate the chemical and physical processes that govern adhesion and absorption. Many different investigations were undertaken to achieve that goal. Some of them were exploratory in nature while others were much more extensive. These studies laid the groundwork for the two major products from this contract: the adhesion and stripping models and the net adsorption test.

The initial asphalt-aggregate model that was proposed, postulated the adherence of asphalt at the asphalt-aggregate interface, followed by the development of a structured interphase region. Beyond the interphase was the bulk asphalt. A new understanding of asphalt-aggregate interactions has emerged from the work of SHRP A-003B in conjunction with research results from other SHRP contractors. During hot mix processing, asphalt components contact and adhere to the interfacial surface of the aggregate with the more polar constituents, those compounds containing heteroatoms of sulfur, nitrogen or oxygen, being most competitive for the active sites on the surface. Several different methods of measuring the energy of adsorption indicate that physisorption rather than chemisorption is occurring. This interaction can result from electrostatic, dipole-dipole, or Van der Waals interactions. Asphalt once contacted to the aggregate remains stationary; no net migration of polar constituents to the surface from the bulk asphalt is apparent. Some surface diffusion may occur, though, as the mix softens on a hot summer day.

### 2.3.1 Adhesion

Effect of Chemistry. Aggregate chemistry plays a key role in adhesion. Each aggregate of a given mineralogical type with a specific history has a unique surface chemistry. The electrokinetic

properties as well as the electron donating and accepting abilities of the aggregate vary according to the active metal species on the aggregate surface. The active sites on the aggregate surface that have been postulated from observed behavior have been confirmed by autoradiography. These active sites promote adsorption of asphaltic components. The covering of those active sites by nonpolar hydrocarbons completely masks their activity. Dust coatings naturally occurring on aggregate surfaces can change the chemistry of adhesion and result in weak bonding between the dust and aggregate surface, leading to attrition of the bonding forces that help maintain the pavement.

Evaluation of asphalt-aggregate interactions shows that the aggregate chemistry is much more influential than the asphalt composition for both adhesion and sensitivity to water. Large differences were observed in the amount of asphalt adsorbed and the amount of asphalt retained after exposure to water with both siliceous and calcareous aggregates. Although the asphalt compositional factors have a smaller effect, some differences in the amounts adsorbed and retained on a specific aggregate were observed.

No chemical or thermodynamic evidence has been found in this study for the development of a structured interphasal region. Aging experiments that determined the oxidatively aged products in asphalt at the interface and in the 125 $\mu$ m region beyond the interface showed no differentiation in type or concentration of asphalt oxidative products. Heats of interaction only showed an initial release of energy corresponding to the heat released upon initial contact between asphalt and aggregate. No long term energy release was observed, indicating the lack of structuring. Autoradiography also showed no evidence of structuring.

Hence, the asphalt-aggregate mix can be visualized as a system in which large, small and fine aggregate particles are coated with asphalt. The active sites on the particle attract the most polar and bondable asphaltic species upon initial contact with little or no diffusion of asphaltic species after the mix is cooled. Each asphalt molecule is contacted with an aggregate or an asphalt molecule in contact with or close to an aggregate surface. The fines that compose 5 to 8 percent of the aggregate are interspersed with the asphalt forming a mastic, a medium in which it is difficult to distinguish between asphalt and aggregate.

Aging. The aging of asphalt in a road pavement occurs in the presence of aggregate so that is natural to evaluate the asphalt aging process with aggregate present. The research in SHRP A-003B evaluated the chemistry of the aging process in terms of the production of carbonyls, including both ketones and carboxylic acids, and sulfoxides. Sulfoxide production is largely dependent upon the amount of sulfur present in the asphalt. The aggregate chemistry of a granite and a limestone had no effect on the production of these particular functional groups. However, other changes that may have occurred in the asphalt were not measured. In road pavements the ostensible measure of aging is viscosity. Recent research in SHRP contracts A-002A and A-003A suggests that the presence of aggregate decreases the viscosity of asphalt compared to bulk asphalt for equivalent aging times. This difference in viscosity may be caused by the aggregate particles binding some of the oxidative functional groups formed and, thereby, prevents the formation of viscosity building species.

Water Sensitivity. Stripping of asphalt from aggregate stems from the intrusion of water into the asphalt-aggregate system. The modes of failure are many and dependent upon the character of the system. The most important modes of failure are:

- diffusion of water through the asphalt film;
- entry of water through cracks in the asphalt film;
- separation of the bond at the interface;
- failure within the asphalt where soluble components are removed; and
- cohesive failure within the aggregate.

If the water-proofing layer of asphalt surrounding an aggregate particle is continuous, then water can penetrate the system by diffusing through the asphalt film removing along the way those asphaltic components that are solubilized. If cracks occur in the film, then water can intrude to the asphalt-aggregate interface, causing failure at or near the interface. The failure can be interfacial or cohesive either in the asphalt or in the aggregate. Reduction in water damage can be attained through modifying the aggregate surface through silylation or the addition of antistripping agents. However, complete covering of the particle by an asphalt

film should decrease the quantity of water reaching the aggregate and reduce the deleterious effect of water on the aggregate.

Building of roads with low air voids or good drainage may be most influential in reducing water damage, by limiting the exposure of the asphalt-aggregate bond to water.

Resilience of Asphalt-aggregate Bonds. Adhesion between an asphalt-aggregate pair can be promoted or inhibited by processing and environmental factors. Researchers in SHRP A-003B evaluated the effect of pH on the asphalt-aggregate bond. High pH which resulted in a very basic medium was detrimental to most asphalt-aggregate bonds; however, treatment at somewhat lower but still basic pH did not affect the bond substantially. Curing at elevated temperatures after mixing promoted adhesion in some asphalt-aggregate pairs. A test involving the factors of increased pH and curing, incorporated into the modified Lottman (AASHTO-283) test, has been suggested as a means of differentiating among asphalt-aggregate combinations. Those particular asphalt-aggregate combinations that do not perform well under chemical preconditioning (high pH) or curing can be treated with additives, either liquid antistripping agents or lime, to improve their performance. Retesting the treated mix under the stringent pH conditions offers a means of determining the effectiveness of the treatment.

### 2.3.2 Products From the A-003B Research

Two major products were produced from SHRP A-003B. The adhesion and stripping models and the net adsorption test.

The net adsorption test provides a method for determining the affinity of an asphalt-aggregate pair and its sensitivity to water. This test provides a method for selecting asphalt-aggregate pairs and determining their compatibility. The test is composed of two steps. First, asphalt is adsorbed onto aggregate from toluene solution, the amount of asphalt remaining in solution is measured, and the amount of asphalt adsorbed to the aggregate is determined. Second, water is introduced into the system, asphalt is desorbed from the aggregate surface, the asphalt present in the solution is measured, and the amount remaining on the aggregate surface is calculated. The amount of asphalt remaining on the surface after the desorption step

is termed net adsorption. The net adsorption offers a direct means of comparing the affinity of different asphalt-aggregate pairs. The test is relatively fast, reliable and readily performed. The net adsorption test predicts the behavior of the SHRP aggregates quite well when using the known field performance of the aggregate within its state as the criterion.

For this study, the eleven material reference library (MRL) aggregates were tested with three different aged asphalts: AAD-1, AAK-1, and AAM-1. Although the asphalts differed quite substantially in their chemical composition and characteristics, for a given aggregate the differences in the asphalt initial adsorption behavior were quite small. Based on the amount adsorbed, the asphalts ranked best to worst by net adsorption;  $AAD-1 \geq AAK-1 > AAM-1$  for most aggregates, with AAD-1 and AAK-1 occasionally changing positions.

The adsorption behavior of the siliceous aggregates before and after water desorption varied considerably. Two aggregates, RA-granite and RJ-gravel, showed consistently low adsorption and were quite sensitive to water regardless of the asphalt used. Aggregates, RB-granite, RE-gravel, and RG-sandstone, showed similar behavior in their initial asphalt adsorption for the three asphalts; however, RE-gravel tended to show a higher sensitivity to water and an increased amount of asphalt desorbed compared to the other aggregates. The two siliceous aggregates that gave the largest amounts of asphalt adsorption, regardless of asphalt, were RH-greywacke and RK-basalt. Both of these aggregates also had low sensitivity to water.

The MRL limestones used in this study included RC, a highly absorptive limestone, RD, a nonabsorptive limestone, and RF, a calcareous sandstone, a limestone with other types of minerals present. The initial adsorption behavior of the three asphalts was similar on the three limestones and ranked the aggregates  $RC > RF > RD$ . However, the moisture sensitivities of these limestones seemed somewhat asphalt dependent, with AAM-1 showing more sensitivity to water than either AAD-1 or AAK-1. RF-limestone yielded a considerably higher amount of desorbed asphalt than did either RC or RD-limestones. Even though the amount of adsorption and desorption that occurred varied somewhat from asphalt to asphalt for a given aggregate the net adsorption, defined as the amount of asphalt remaining on the aggregate, was similar as shown

in Figure 2.4. The net adsorption ranking of the siliceous aggregates for all three asphalts was RK-basalt > RH-greywacke  $\approx$  RL-gravel > RB-granite > RE-gravel > RG-sandstone > RJ-gravel > RA-granite. The net adsorption of the limestone aggregates ranked as RC > RD  $\approx$  RF.

Additional limestone samples were obtained by SHRP and used in the net adsorption test. The net adsorption for the limestones is given in Figure 2.4. The net adsorption ranking of the limestones for all three asphalts was R7 > R3  $\geq$  R8 > R6 > R1 > R5 > R2. The average net adsorption ranged from a high of 1.27 mg/g on R7 to a low of 0.34 mg/g on R2.

State highway officials from the respective states from which the limestones were obtained provided a classification of the limestones as stripping or nonstripping according to their observed behavior. The behavior of R1, R2 and R7 limestones in the net adsorption test did not agree with that indicated by the state highway officials. The reasons for these discrepancies are not known at this time; however, some obvious possibilities include the fact that only a very narrow size fraction of the aggregate was used and because the aggregate was prewashed.

The aggregate properties predominated in the net adsorption test, showing a stronger influence than the asphalt on the initial amount of asphalt adsorbed, the amount of asphalt desorbed by water, and the amount of asphalt remaining, the net adsorption.

The net adsorption test offers an effective means of evaluating the affinity and water sensitivity of an asphalt-aggregate pair. The test enables prediction of those aggregate materials which will achieve a substantial asphalt coating and maintain that coating in the presence of water. The test also provides a means of predicting which asphalts would be susceptible to water. For the net adsorption test to represent most accurately the aggregates used in road paving, the recommended sample is a minus No. 4 fraction of unwashed aggregate.

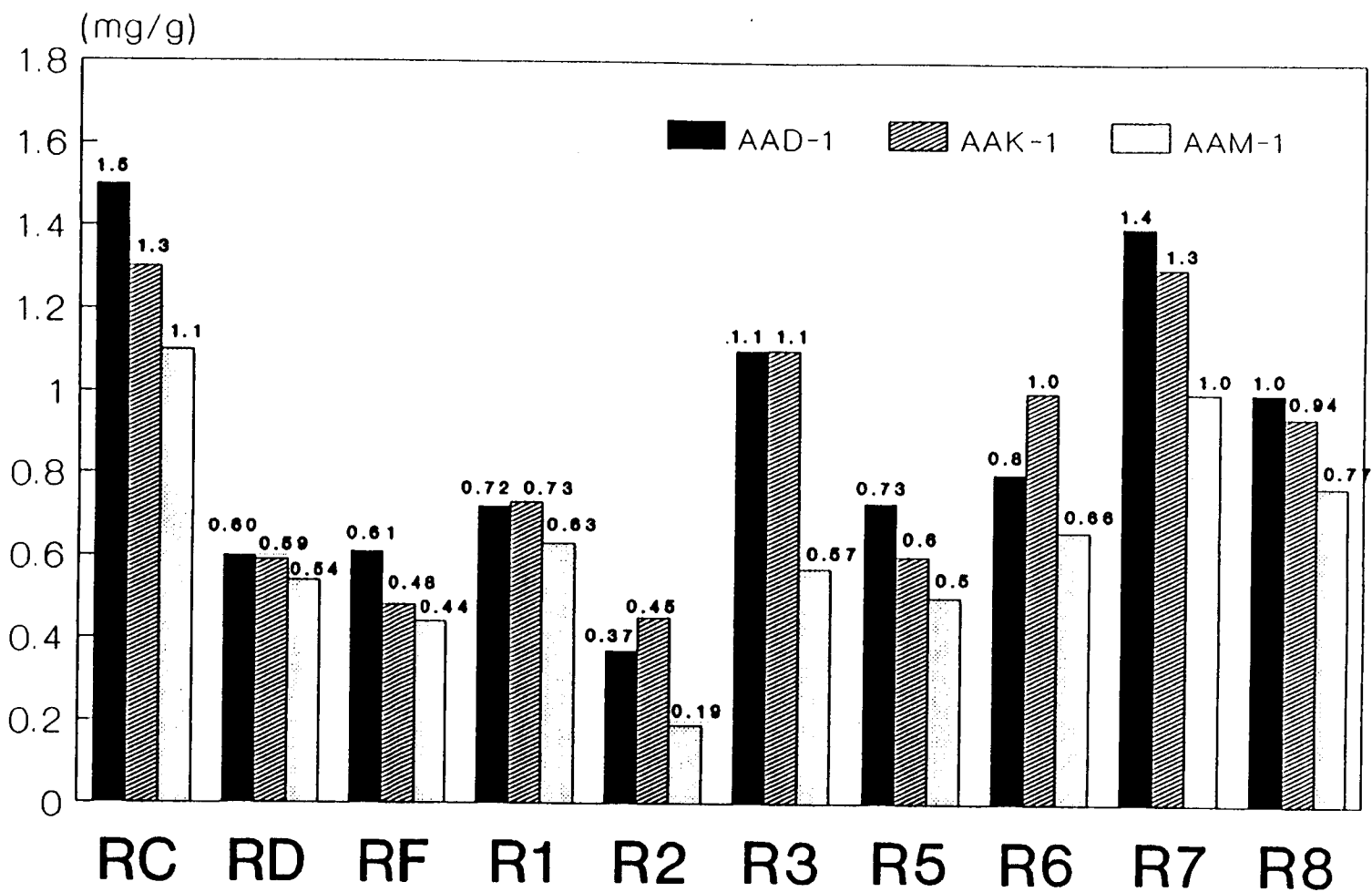


Figure 2.4. Net adsorption of asphalts on various limestones

## **3.0 Experiment Design**

### **3.1 Variables**

As mentioned earlier, the experiment design included eight different asphalt types and four different aggregates. All specimens to be long-term aged were first short-term aged at 135°C (275°F) for four hours before compaction. Four different long-term aging processes were examined: low pressure oxidation (LPO) at 60° and 85°C (140° and 185°F), long-term oven aging (LTOA) at 85°C (185°F), for 5 days, and LTOA at 100°C (212°F) for 2 days. See table 3.1 for the LPO variables and table 3.2 for the LTOA variables.

### **3.2 Materials**

The materials used for this testing program were selected from the materials stored at the Materials Reference Library (MRL).

The aggregates used represent a broad range of aggregate characteristics from a high-absorption crushed limestone to a river run gravel. Similarly, the asphalts used spanned a broad range of asphalt grades. Table 3.3 briefly describes the materials used.

### 3.3 Aging Methods

#### 3.3.1 No Aging

Three specimens were prepared at the time of mixing to represent an "unaged" condition. Their preparation was the same as for other specimens except that they were not cured for four hours at 135°C (275°F). As soon as mixing was complete the specimens were placed in an oven and brought to the proper equiviscous temperature ( $665 \pm 80$  centi-Stokes). The specimens were then compacted using a California Kneading Compactor.

**Table 3.1. Low pressure aging experiment design**

No. of Asphalts	8
No. of Aggregates	4
No. of Asphalt Contents	1
No. of Air Voids	1
<b>Test Conditions</b>	
Temperature:    Short-Term Long-Term	1, 275°F (135°C) 2, 140-185°F (60 and 85°C)
<b>Aging Periods</b>	
None (datum)	1
Short-Term and Long-Term	1
<b>Total Tests</b>	
No Aging (unaged)	32
Short-Term and Long-Term	64
Replication of Unaged	32
Replication of Short-Term and Long-Term	64
<b>TOTAL</b>	<b>192</b>

**Table 3.2. Long-term oven aging experiment design**

No. of Asphalts	8
No. of Aggregates	4
No. of Asphalt Contents	1
No. of Void Contents	1
<b>Test Conditions</b>	
Temperature:    Short-Term Long-Term	1, 275°F (135°C) 2, 185-212°F (85°C and 100°C)
<b>Aging Periods</b>	
None (datum)	1
Short-Term and Long-Term	1
<b>Total Tests</b>	
No aging	32
Short-Term and Long-Term	64
Replication of short-term and long-term	64
<b>TOTAL</b>	<b>160</b>

### 3.3.2 Short-Term Aging

The short-term aging method used in this test program was developed at Oregon State University under the SHRP A-003A test development program. The procedure is described in detail in Appendix A. The method employed consists of curing mix samples in a forced draft oven at 135°C (275°F) for a period of 4 h prior to compaction. After the curing period, the samples are brought to an equiviscous temperature and compacted using a California Kneading Compactor.

**Table 3.3. Materials used**

Aggregate		Asphalt	
Code	Description	Code	Grade
RC	Limestone (high absorption)	AAA-1	150/200
RD	Limestone (low absorption)	AAB-1	AC-10
RH	Greywacke	AAC-1	AC-8
RJ	Conglomerate	AAD-1	AR-4000
		AAF-1	AC-20
		AAG-1	AR-4000
		AAK-1	AC-30
		AAM-1	AC-20

### 3.3.3 Low Pressure Oxidation

Low pressure oxidation is an aging procedure to simulate long-term aging. The procedure is carried out on compacted specimens after they have been short-term aged. After the specimen is loaded into the cell a confining pressure is applied to keep the membrane tight to the specimen. Once the confining pressure is achieved, oxygen flow is started through the specimen at a rate of 4 standard cubic feet per hour (SCFH) [0.113 cubic meter/h ( $\text{m}^3/\text{h}$ )]. When the oxygen rate has been adjusted, the cell is placed in a water bath that has been preheated to the conditioning temperature, 60° or 85°C (140 or 185°F). The cell is left in the conditioning bath for a period of 5 days, at which time it is extracted from the bath and left to cool to room temperature. The specimens are then removed from the cell and allowed to stand for at least 24 h before testing.

### 3.3.4 Long-Term Oven Aging

Long-term oven aging is also a procedure to simulate long-term aging. The procedure is carried out on compacted specimens after they have been short-term aged. The procedure is described in detail in Appendix C. The specimens are placed in a forced draft oven, pre-heated to 85°C (185°F), and left for a period of 5 days. Alternately, a temperature of 100°C (212°F) and a period of 2 days is used. After the aging period, the oven is turned off and left to cool to room temperature. The specimens are removed from the oven when this temperature is achieved and prepared to be tested at least 24 h after removal from the oven.

## 3.4 Evaluation Methods

### 3.4.1 Resilient Modulus

The resilient modulus is determined at 25°C (77°F) using diametral (indirect tension) (ASTM D4123) and triaxial compression modes of testing with a 0.1-second loading time at a frequency of 1.0 Hz. A constant strain level of 100 microstrain is maintained throughout the test.

### 3.4.2 Dynamic Modulus

Several specimens were subjected to a thorough dynamic modulus evaluation at temperatures of 0°C (32°F), 25°C (77°F), and 40°C (140°F). Eleven loading frequencies ranging from 15 to 0.01 Hz were used. This testing, called dynamic mechanical analysis (DMA), takes approximately eight hours per specimen; therefore, it was not possible to test all specimens with this

procedure. A thorough explanation of the DMA procedure and typical results are presented in Appendix F.

From the limited evaluation performed on the materials for this project, it was apparent that DMA is a valid procedure for characterizing the aging potential of asphalt-aggregate mixtures. However, due to the time required to perform the test and reduce the data, a more through evaluation of the materials available was not pursued.

### 3.4.3 Tensile Strength Test

The tensile strength test is performed after all modulus testing has been completed. A deformation rate of 50 mm (2 in.) per minute is used, with the load and deformation of the specimen monitored continuously until failure occurs. The strains at yield and failure are considered significant, as well as the strength. The broken portions of the specimens may be used to obtain recovered asphalt. Appendix D describes the tensile test and presents data from a small sample of specimens.

## 4.0 Results

### 4.1 Resilient Modulus Data

The results of the resilient modulus test for both diametral and triaxial modes of testing are summarized in Tables 4.1 through 4.4 for each aggregate type. These data are shown graphically in Appendix D, where aged and unaged modulus values are presented.

#### 4.1.1 Short-Term Aging Results

The modulus ratios from diametral testing are shown in Figure 4.1 for each of the four aggregates, with the asphalts shown in ranking order in each case. The data used to generate these figures are presented in Tables 4.1 through 4.4. The asphalt showing the greatest aging (in terms of modulus change) has the highest ratio in each case. The ratios have been developed using a procedure to adjust the modulus values to correspond to the same air void content. This procedure is described in Section 4.1.3.

#### 4.1.2 Long-Term Aging Results

The modulus ratios from diametral testing of the long-term aged specimens are shown in Figure 4.2 through 4.5. These are shown in a format similar to Figure 4.1, with rankings based on the ratio of long-term aged modulus to unaged modulus. As with the short-term aging results, the modulus values were adjusted as described below. The data used to generate these tables are also presented in Tables 4.1 through 4.4.

### 4.1.3 Adjustment of Modulus Data

To analyze the effects of short-term and long-term aging on asphalt-aggregate mixes a method of creating an aging ratio was needed. To create this ratio a measure of the unaged modulus was needed to compare with the aged specimens. At the time of mixing in the laboratory, 3 specimens, in addition to those needed for long term aging, were prepared and compacted as soon as they could be brought to the proper compaction temperature. These specimens were said to be in an "unaged" condition and were tested for resilient modulus. In all but a few cases, the unaged specimens were found to have a different air void level than the short-term aged specimens. This prompted a need to adjust the modulus values of the short-term aged specimens to correspond to the same air void level as the unaged specimens.

To achieve this adjustment, an average slope was determined from the modulus versus air voids for the unaged specimens over the data set, for diametral modulus only. With this slope and with values for the average modulus and air void level for each combination of materials, an equation for the unaged modulus at any void level could be determined. From this equation, an adjusted unaged modulus could be calculated for each short-term aged specimen, which was then used in calculating the short-term and long-term aging ratios.

Table 4.1. Modulus data for aggregate RC

Asphalt	Aging Method	% Air Voids	Modulus Values			
			Diametral		Triaxial	
			Before	After	Before	After
AAA	LPO 85	8.2	211	572	295	805
AAA	LPO 85	8.4	193	504	350	802
AAA	LPO 60	8.0	233	367	434	600
AAA	LPO 60	8.1	270	414	373	442
AAA	LTOA 85	9.5	225	405	357	780
AAA	LTOA 85	8.7	221	412	295	583
AAA	LTOA 100	9.0	219	475	270	570
AAA	LTOA 100	8.6	216	499	295	455
AAA	NONE	8.0	152	--	230	--
AAA	NONE	8.8	153	--	225	--
AAA	NONE	7.9	164	--	236	--
AAB	LPO 85	8.4	299	638	517	1041
AAB	LPO 85	9.2	317	438	419	635
AAB	LPO 60	8.3	364	525	420	621
AAB	LPO 60	8.3	300	644	379	1041
AAB	LTOA 85	8.9	305	606	395	875
AAB	LTOA 85	9.3	339	614	500	956
AAB	LTOA 100	8.3	378	694	426	698
AAB	LTOA 100	9.7	286	618	533	958
AAB	NONE	8.8	216	--	385	--
AAB	NONE	7.8	207	--	421	--
AAB	NONE	8.2	249	--	467	--
AAC	LPO 85	8.4	329	715	574	1052
AAC	LPO 85	9.4	398	750	440	844
AAC	LPO 60	9.3	348	520	579	879
AAC	LPO 60	10.2	339	460	384	667
AAC	LTOA 85	9.1	345	561	690	889
AAC	LTOA 85	9.3	377	600	407	787
AAC	LTOA 100	9.4	335	557	409	697
AAC	LTOA 100	8.9	343	623	435	643
AAC	NONE	9.1	236	--	325	--
AAC	NONE	9.3	235	--	277	--
AAC	NONE	8.2	249	--	315	--
AAD	LPO 85	9.3	286	645	274	970
AAD	LPO 85	8.8	293	694	380	950
AAD	LPO 60	9.6	321	450	399	850
AAD	LPO 60	9.0	257	394	432	711
AAD	LTOA 85	8.9	324	615	391	1101
AAD	LTOA 85	9.4	309	616	491	882
AAD	LTOA 100	9.3	225	611	379	775
AAD	LTOA 100	9.0	269	695	344	539
AAD	NONE	8.2	202	--	279	--
AAD	NONE	8.1	208	--	277	--
AAD	NONE	8.5	182	--	275	--

\*F = 1.8(°C) + 32

Note: All modulus data are reported in ksi.

## KEY:

NONE = No aging.  
 LPO 60 = Low-Pressure Oxidation, 60°C/5 days.  
 LPO 85 = Low-Pressure Oxidation, 85°C/5 days.  
 LTOA 85 = Long-Term Oven Aging, 85°C/5 days.  
 LTOA 100 = Long-Term Oven Aging, 100°C/2 days.

Table 4.1 (continued). Modulus data for aggregate RC

Asphalt	Aging Method	% Air Voids	Modulus Values			
			Diametral		Triaxial	
			Before	After	Before	After
AAF	LPO 85	9.3	650	891	861	1384
AAF	LPO 85	8.8	687	996	864	1275
AAF	LPO 60	7.8	636	898	1113	1345
AAF	LPO 60	9.4	621	896	1323	1305
AAF	LTOA 85	9.0	612	943	980	1205
AAF	LTOA 85	9.0	701	842	1103	1573
AAF	LTOA 100	9.1	558	1004	823	1124
AAF	LTOA 100	9.7	590	1016	999	1357
AAF	NONE	9.0	507	--	779	--
AAF	NONE	9.9	428	--	550	--
AAF	NONE	9.1	458	--	851	--
AAG	LPO 85	10.9	652	983	853	1262
AAG	LPO 85	10.6	606	1038	684	1141
AAG	LPO 60	10.2	682	840	701	1000
AAG	LPO 60	10.7	744	881	851	1134
AAG	LTOA 85	10.9	714	1004	928	1191
AAG	LTOA 85	11.2	656	819	1024	1520
AAG	LTOA 100	10.2	614	1030	918	1245
AAG	LTOA 100	10.9	587	939	921	1113
AAG	NONE	11.0	450	--	658	--
AAG	NONE	9.9	523	--	734	--
AAG	NONE	9.6	476	--	804	--
AAK	LPO 85	7.9	555	974	671	1430
AAK	LPO 85	8.5	572	1000	655	1740
AAK	LPO 60	9.2	497	644	644	992
AAK	LPO 60	9.3	427	577	574	866
AAK	LTOA 85	7.9	563	827	834	1367
AAK	LTOA 85	9.2	451	713	614	993
AAK	LTOA 100	9.6	544	1019	607	1068
AAK	LTOA 100	8.6	502	1049	662	1260
AAK	NONE	9.2	345	--	413	--
AAK	NONE	8.0	450	--	579	--
AAK	NONE	8.1	429	--	578	--
AAM	LPO 85	8.9	470	763	436	1006
AAM	LPO 85	8.1	445	840	641	1110
AAM	LPO 60	8.0	421	580	577	796
AAM	LPO 60	8.6	405	602	558	850
AAM	LTOA 85	8.5	446	796	510	897
AAM	LTOA 85	9.0	456	747	488	910
AAM	LTOA 100	9.2	404	750	552	816
AAM	LTOA 100	8.5	450	787	537	818
AAM	NONE	8.3	332	--	453	--
AAM	NONE	9.0	303	--	358	--
AAM	NONE	7.9	346	--	442	--

°F = 1.8(°C) + 32

Note: All modulus data are reported in ksi.

## KEY:

NONE = No aging.

LPO 60 = Low-Pressure Oxidation, 60°C/5 days.

LPO 85 = Low-Pressure Oxidation, 85°C/5 days.

LTOA 85 = Long-Term Oven Aging, 85°C/5 days.

LTOA 100 = Long-Term Oven Aging, 100°C/2 days.

Table 4.2. Modulus data for aggregate RD

Asphalt	Aging Method	% Air Voids	Modulus Values			
			Diametral		Triaxial	
			Before	After	Before	After
AAA	LPO 85	8.2	211	572	295	805
AAA	LPO 85	8.4	193	504	350	802
AAA	LPO 60	8	233	367	434	600
AAA	LPO 60	8.1	270	414	373	442
AAA	LTOA 85	9.5	225	405	357	780
AAA	LTOA 85	8.7	221	412	295	583
AAA	LTOA 100	9	219	475	270	570
AAA	LTOA 100	8.6	216	499	295	455
AAA	NONE	8	152	--	230	--
AAA	NONE	8.8	153	--	225	--
AAA	NONE	7.9	164	--	236	--
AAB	LPO 85	8.6	356	627	320	541
AAB	LPO 85	7.2	400	632	475	539
AAB	LPO 60	8.9	414	456	450	535
AAB	LPO 60	8.4	380	506	489	696
AAB	LTOA 85	8.7	390	502	465	755
AAB	LTOA 85	8.5	528	582	578	780
AAB	LTOA 100	7.4	509	603	589	631
AAB	LTOA 100	7.5	444	642	411	588
AAB	NONE	8.4	233	--	353	--
AAB	NONE	7.6	306	--	399	--
AAB	NONE	7.6	302	--	314	--
AAC	LPO 85	8.3	419	657	614	950
AAC	LPO 85	8.2	467	671	498	884
AAC	LPO 60	6.9	486	630	762	886
AAC	LPO 60	8.1	526	628	761	741
AAC	LTOA 85	7.1	435	532	519	726
AAC	LTOA 85	7.4	456	600	644	782
AAC	LTOA 100	7.8	451	522	403	679
AAC	LTOA 100	7.3	496	658	647	732
AAC	NONE	7.9	304	--	506	--
AAC	NONE	7.1	291	--	464	--
AAC	NONE	7.5	319	--	505	--
AAD	LPO 85	8.6	321	584	383	893
AAD	LPO 85	8.2	334	633	432	966
AAD	LPO 60	8.5	325	463	425	845
AAD	LPO 60	8.2	362	450	352	698
AAD	LTOA 85	7.8	356	578	472	689
AAD	LTOA 85	8.4	393	611	410	679
AAD	LTOA 100	9.3	341	515	398	670
AAD	LTOA 100	9	395	544	438	441
AAD	NONE	8.1	250	--	227	--
AAD	NONE	6.9	253	--	298	--
AAD	NONE	7	262	--	286	--

°F = 1.8(°C) + 32

Note: All modulus data are reported in ksi.

## KEY:

- NONE = No aging.  
 LPO 60 = Low-Pressure Oxidation, 60°C/5 days.  
 LPO 85 = Low-Pressure Oxidation, 85°C/5 days.  
 LTOA 85 = Long-Term Oven Aging, 85°C/5 days.  
 LTOA 100 = Long-Term Oven Aging, 100°C/2 days.

Table 4.2 (continued). Modulus data for aggregate RD

Asphalt	Aging Method	% Air Voids	Modulus Values			
			Diametral		Triaxial	
			Before	After	Before	After
AAF	LPO 85	8.9	795	1193	763	1393
AAF	LPO 85	8.9	857	1244	1009	1818
AAF	LPO 60	9	703	1034	998	1588
AAF	LPO 60	8.6	704	862	806	1359
AAF	LTOA 85	9.2	807	1072	1066	1342
AAF	LTOA 85	8.3	786	1068	1036	1538
AAF	LTOA 100	8.9	754	1100	871	919
AAF	LTOA 100	8.9	706	1119	1127	1796
AAF	NONE	9.6	493	--	609	--
AAF	NONE	8.9	526	--	700	--
AAF	NONE	8.8	564	--	850	--
AAG	LPO 85	8.6	991	1147	1194	1588
AAG	LPO 85	8.8	1101	1162	1380	2298
AAG	LPO 60	7.7	1002	1312	1178	1570
AAG	LPO 60	8.7	854	1201	1162	1598
AAG	LTOA 85	8.5	917	1108	1264	1617
AAG	LTOA 85	8.4	893	1161	1186	1277
AAG	LTOA 100	8.4	791	1015	1116	1266
AAG	LTOA 100	8.5	745	1105	1215	1272
AAG	NONE	8	608	--	1040	--
AAG	NONE	8.4	551	--	733	--
AAG	NONE	8	552	--	975	--
AAK	LPO 85	7.8	544	977	507	1039
AAK	LPO 85	8.2	545	782	672	1065
AAK	LPO 60	8	538	721	556	745
AAK	LPO 60	8	567	804	638	1104
AAK	LTOA 85	7.6	527	761	690	1062
AAK	LTOA 85	8.8	336	650	302	1120
AAK	LTOA 100	7.7	507	900	646	842
AAK	LTOA 100	7.2	516	890	723	1066
AAK	NONE	9.3	343	--	391	--
AAK	NONE	8.3	482	--	436	--
AAK	NONE	7.7	493	--	536	--
AAM	LPO 85	8.8	437	629	536	793
AAM	LPO 85	8.2	509	703	556	668
AAM	LPO 60	8.3	406	571	605	882
AAM	LPO 60	8.3	446	616	476	807
AAM	LTOA 85	7.3	458	638	510	807
AAM	LTOA 85	8	459	710	593	809
AAM	LTOA 100	8.2	410	648	546	696
AAM	LTOA 100	8.6	458	639	518	840
AAM	NONE	5.5	438	--	485	--
AAM	NONE	8.6	407	--	391	--
AAM	NONE	7.9	518	--	469	--

°F = 1.8(°C) + 32

Note: All modulus data are reported in ksi.

## KEY:

NONE = No aging.

LPO 60 = Low-Pressure Oxidation, 60°C/5 days.

LPO 85 = Low-Pressure Oxidation, 85°C/5 days.

LTOA 85 = Long-Term Oven Aging, 85°C/5 days.

LTOA 100 = Long-Term Oven Aging, 100°C/2 days.

Table 4.3. Modulus data for aggregate RH

Asphalt	Aging Method	% Air Voids	Modulus Values			
			Diametral		Triaxial	
			Before	After	Before	After
AAA	LPO 85	8.2	211	572	295	805
AAA	LPO 85	8.4	193	504	350	802
AAA	LPO 60	8	233	367	434	600
AAA	LPO 60	8.1	270	414	373	442
AAA	LTOA 85	9.5	225	405	357	780
AAA	LTOA 85	8.7	221	412	295	583
AAA	LTOA 100	9	219	475	270	570
AAA	LTOA 100	8.6	216	499	295	455
AAA	NONE	8	152	--	230	--
AAA	NONE	8.8	153	--	225	--
AAA	NONE	7.9	164	--	236	--
AAB	LPO 85	8.8	311	479	281	541
AAB	LPO 85	10.6	244	385	275	539
AAB	LPO 60	8.5	276	490	306	605
AAB	LPO 60	8.9	256	330	356	539
AAB	LTOA 85	8.8	313	419	351	567
AAB	LTOA 85	8.4	289	445	363	655
AAB	LTOA 100	7.6	360	454	564	562
AAB	LTOA 100	8	348	451	425	434
AAB	NONE	8.8	160	--	165	--
AAB	NONE	7.8	191	--	260	--
AAB	NONE	7.5	216	--	305	--
AAC	LPO 85	8.3	290	505	271	589
AAC	LPO 85	8.5	313	487	288	520
AAC	LPO 60	8.4	264	374	242	373
AAC	LPO 60	7.8	307	375	310	449
AAC	LTOA 85	8.8	286	403	319	507
AAC	LTOA 85	8.4	272	387	364	439
AAC	LTOA 100	6.8	419	453	493	521
AAC	LTOA 100	6.8	413	455	618	548
AAC	NONE	7.5	176	--	200	--
AAC	NONE	7.7	163	--	220	--
AAC	NONE	8	161	--	210	--
AAD	LPO 85	6.3	252	553	272	573
AAD	LPO 85	8.4	317	616	401	826
AAD	LPO 60	8.9	229	316	295	522
AAD	LPO 60	7.3	261	309	237	408
AAD	LTOA 85	8	227	385	317	613
AAD	LTOA 85	7.8	278	435	184	283
AAD	LTOA 100	6.6	256	348	307	513
AAD	LTOA 100	6.9	240	390	261	567
AAD	NONE	6.2	197	--	167	--
AAD	NONE	6.9	162	--	240	--
AAD	NONE	5.6	174	--	255	--

°F = 1.8(°C) + 32

Note: All modulus data are reported in ksi.

## KEY:

NONE = No aging.  
 LPO 60 = Low-Pressure Oxidation, 60°C/5 days.  
 LPO 85 = Low-Pressure Oxidation, 85°C/5 days.  
 LTOA 85 = Long-Term Oven Aging, 85°C/5 days.  
 LTOA 100 = Long-Term Oven Aging, 100°C/2 days.

Table 4.3 (continued). Modulus data for aggregate RH

Asphalt	Aging Method	% Air Voids	Modulus Values			
			Diametral		Triaxial	
			Before	After	Before	After
AAF	LPO 85	6.9	677	982	656	1206
AAF	LPO 85	8	864	1089	1158	1705
AAF	LPO 60	7.4	889	1041	874	896
AAF	LPO 60	8	816	903	790	986
AAF	LTOA 85	6.6	776	918	720	1128
AAF	LTOA 85	7.2	762	862	742	1260
AAF	LTOA 100	7.5	775	855	787	1004
AAF	LTOA 100	7.5	700	935	689	932
AAF	NONE	7.2	617	--	855	--
AAF	NONE	7.2	603	--	665	--
AAF	NONE	6.5	673	--	864	--
AAG	LPO 85	9.4	643	912	615	1133
AAG	LPO 85	10.3	610	886	627	1020
AAG	LPO 60	10.2	624	964	925	1102
AAG	LPO 60	10.1	617	837	967	1034
AAG	LTOA 85	8.9	858	1260	982	1303
AAG	LTOA 85	8.4	727	1001	1012	1246
AAG	LTOA 100	--	--	--	--	--
AAG	LTOA 100	--	--	--	--	--
AAG	NONE	8.9	483	--	641	--
AAG	NONE	8.5	511	--	709	--
AAG	NONE	8.6	602	--	663	--
AAK	LPO 85	8.5	506	735	593	904
AAK	LPO 85	8.2	430	700	594	904
AAK	LPO 60	8.8	453	592	607	845
AAK	LPO 60	8.1	400	543	453	710
AAK	LTOA 85	7.6	502	571	517	847
AAK	LTOA 85	8.3	421	453	453	764
AAK	LTOA 100	8	371	646	753	1018
AAK	LTOA 100	7.1	443	626	531	667
AAK	NONE	7.5	250	--	353	--
AAK	NONE	6.9	274	--	303	--
AAK	NONE	6.8	277	--	377	--
AAM	LPO 85	6.8	432	563	430	747
AAM	LPO 85	7.4	382	606	583	818
AAM	LPO 60	7.1	408	521	537	721
AAM	LPO 60	7.2	365	467	530	620
AAM	LTOA 85	6.6	411	479	500	705
AAM	LTOA 85	6.5	411	545	485	779
AAM	LTOA 100	7.1	416	560	467	541
AAM	LTOA 100	7	429	576	517	546
AAM	NONE	5.8	319	--	478	--
AAM	NONE	5.1	349	--	624	--
AAM	NONE	4.6	338	--	666	--

°F = 1.8(°C) + 32

Note: All modulus data are reported in ksi.

## KEY:

- NONE = No aging.  
 LPO 60 = Low-Pressure Oxidation, 60°C/5 days.  
 LPO 85 = Low-Pressure Oxidation, 85°C/5 days.  
 LTOA 85 = Long-Term Oven Aging, 85°C/5 days.  
 LTOA 100 = Long-Term Oven Aging, 100°C/2 days.

Table 4.4. Modulus data for aggregate RJ

Asphalt	Aging Method	% Air Voids	Modulus Values			
			Diametral		Triaxial	
			Before	After	Before	After
AAA	LPO 85	8.2	211	572	295	805
AAA	LPO 85	8.4	193	504	350	802
AAA	LPO 60	8	233	367	434	600
AAA	LPO 60	8.1	270	414	373	442
AAA	LTOA 85	9.5	225	405	357	780
AAA	LTOA 85	8.7	221	412	295	583
AAA	LTOA 100	9	219	475	270	570
AAA	LTOA 100	8.6	216	499	295	455
AAA	NONE	8	152	--	230	--
AAA	NONE	8.8	153	--	225	--
AAA	NONE	7.9	164	--	236	--
AAB	LPO 85	8.7	277	398	357	556
AAB	LPO 85	9	318	521	357	578
AAB	LPO 60	8.8	325	426	284	480
AAB	LPO 60	9.4	292	376	286	588
AAB	LTOA 85	8.6	293	431	344	536
AAB	LTOA 85	9.1	292	455	494	521
AAB	LTOA 100	8.2	335	451	324	536
AAB	LTOA 100	8.2	328	460	373	650
AAB	NONE	7.9	196	--	247	--
AAB	NONE	8.2	209	--	253	--
AAB	NONE	7.5	231	--	235	--
AAC	LPO 85	8.6	267	490	341	843
AAC	LPO 85	7.6	405	594	464	604
AAC	LPO 60	7.8	392	493	478	534
AAC	LPO 60	6.7	440	558	582	651
AAC	LTOA 85	7.2	405	480	439	595
AAC	LTOA 85	8	326	457	589	689
AAC	LTOA 100	8.2	350	431	379	585
AAC	LTOA 100	8.4	345	453	500	636
AAC	NONE	6.4	326	--	376	--
AAC	NONE	6.8	238	--	355	--
AAC	NONE	7	245	--	365	--
AAD	LPO 85	7.7	259	502	445	795
AAD	LPO 85	7.9	265	507	343	780
AAD	LPO 60	7.6	262	375	434	581
AAD	LPO 60	8	299	452	296	548
AAD	LTOA 85	8.4	271	491	420	708
AAD	LTOA 85	7.5	285	476	283	439
AAD	LTOA 100	8.6	317	496	308	651
AAD	LTOA 100	9.2	326	571	481	790
AAD	NONE	7.1	149	--	205	--
AAD	NONE	7.6	136	--	192	--
AAD	NONE	7.6	154	--	214	--

°F = 1.8(°C) + 32

Note: All modulus data are reported in ksi.

KEY:

NONE = No aging.

LPO 60 = Low-Pressure Oxidation, 60°C/5 days.

LPO 85 = Low-Pressure Oxidation, 85°C/5 days.

LTOA 85 = Long-Term Oven Aging, 85°C/5 days.

LTOA 100 = Long-Term Oven Aging, 100°C/2 days.

Table 4.4 (continued). Modulus data for aggregate RJ

Asphalt	Aging Method	% Air Voids	Modulus Values			
			Diametral		Triaxial	
			Before	After	Before	After
AAF	LPO 85	8.7	635	1001	802	1186
AAF	LPO 85	8.7	752	1062	798	1025
AAF	LPO 60	7.6	673	849	756	951
AAF	LPO 60	8.9	706	871	926	1117
AAF	LTOA 85	8.3	677	884	988	1123
AAF	LTOA 85	8.4	779	1006	809	988
AAF	LTOA 100	8.4	681	961	711	1251
AAF	LTOA 100	9	712	1061	736	937
AAF	NONE	9	558	--	668	--
AAF	NONE	8.4	575	--	723	--
AAF	NONE	7.8	567	--	802	--
AAG	LPO 85	7.9	620	895	745	1465
AAG	LPO 85	8.1	735	1006	771	1341
AAG	LPO 60	8.1	812	914	853	1268
AAG	LPO 60	8.2	675	810	760	1030
AAG	LTOA 85	7.9	673	785	822	1324
AAG	LTOA 85	7.4	722	857	885	1349
AAG	LTOA 100	8.9	598	821	717	1010
AAG	LTOA 100	7.9	698	939	986	1116
AAG	NONE	7.5	527	--	657	--
AAG	NONE	7.1	535	--	563	--
AAG	NONE	7.2	581	--	640	--
AAK	LPO 85	9.1	403	660	674	1057
AAK	LPO 85	8.4	419	712	512	1066
AAK	LPO 60	9.2	408	574	499	824
AAK	LPO 60	8.5	463	665	460	656
AAK	LTOA 85	8.3	533	862	551	808
AAK	LTOA 85	9.3	562	928	771	1022
AAK	LTOA 100	9.7	354	586	520	808
AAK	LTOA 100	9	450	737	692	972
AAK	NONE	7.9	309	--	473	--
AAK	NONE	7.8	340	--	421	--
AAK	NONE	7.7	347	--	460	--
AAM	LPO 85	7.2	370	548	347	652
AAM	LPO 85	8.2	344	492	602	792
AAM	LPO 60	7.9	367	504	598	734
AAM	LPO 60	7.3	394	529	452	621
AAM	LTOA 85	8.1	437	558	604	813
AAM	LTOA 85	8.3	385	479	480	717
AAM	LTOA 100	7.6	410	442	510	492
AAM	LTOA 100	7.5	356	491	436	519
AAM	NONE	7.3	312	--	422	--
AAM	NONE	6.8	323	--	393	--
AAM	NONE	6.6	343	--	355	--

°F = 1.8(°C) + 32

Note: All modulus data are reported in ksi.

KEY:

NONE = No aging.  
 LPO 60 = Low-Pressure Oxidation, 60°C/5 days.  
 LPO 85 = Low-Pressure Oxidation, 85°C/5 days.  
 LTOA 85 = Long-Term Oven Aging, 85°C/5 days.  
 LTOA 100 = Long-Term Oven Aging, 100°C/2 days.

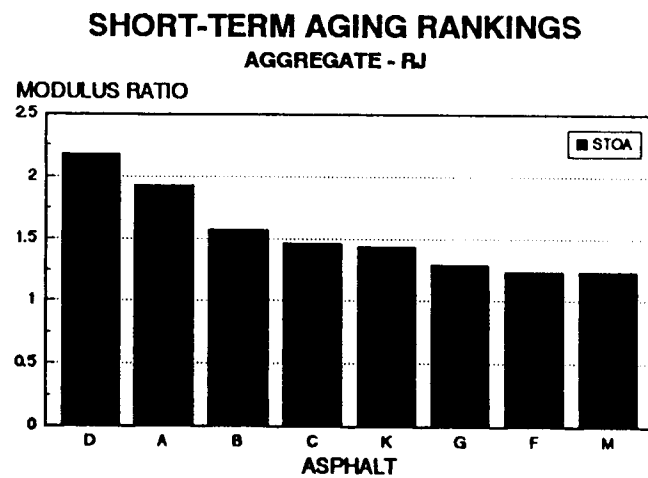
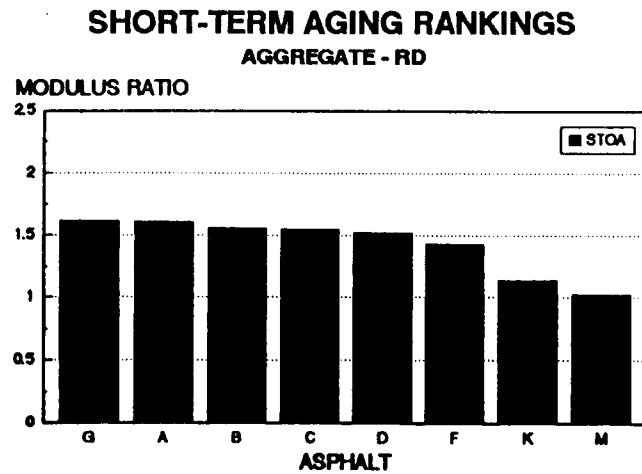
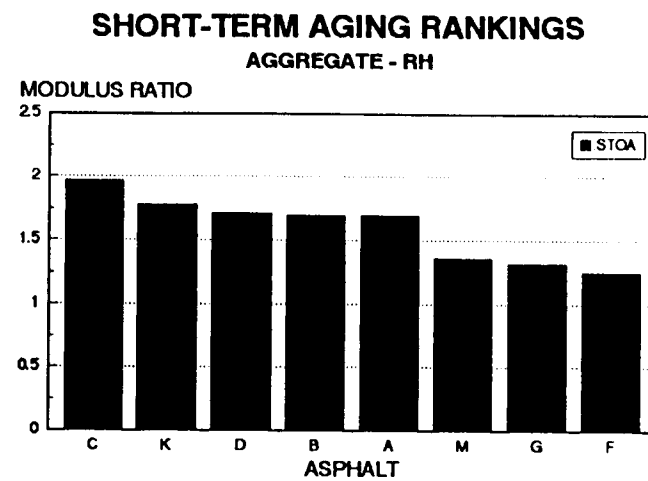
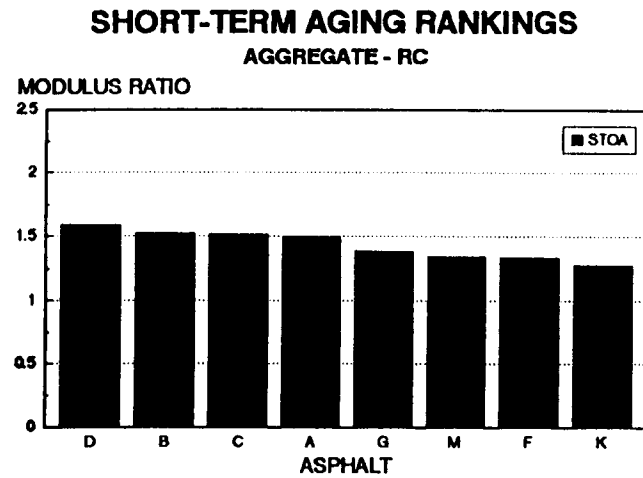


Figure 4.1. Short-term aging: rankings based on diametral modulus

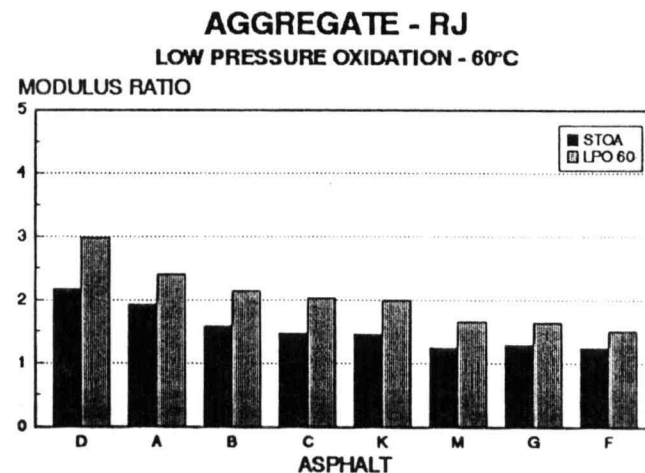
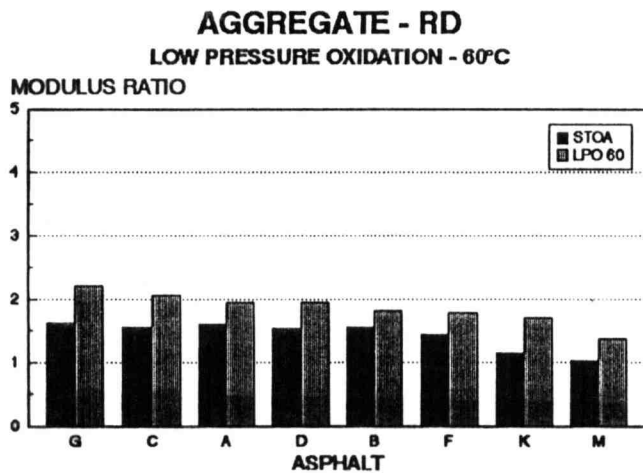
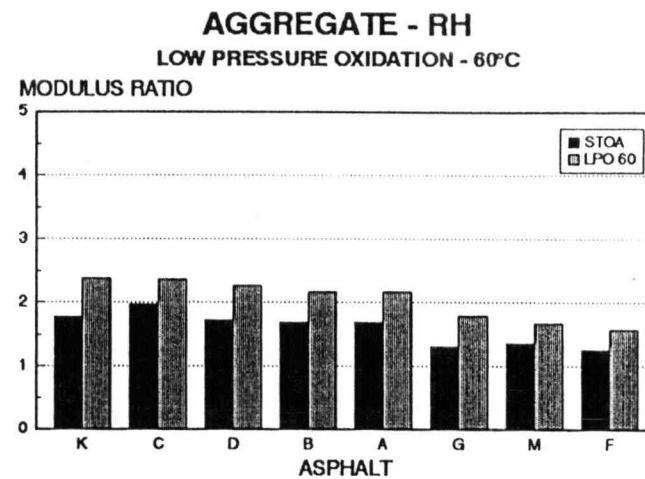
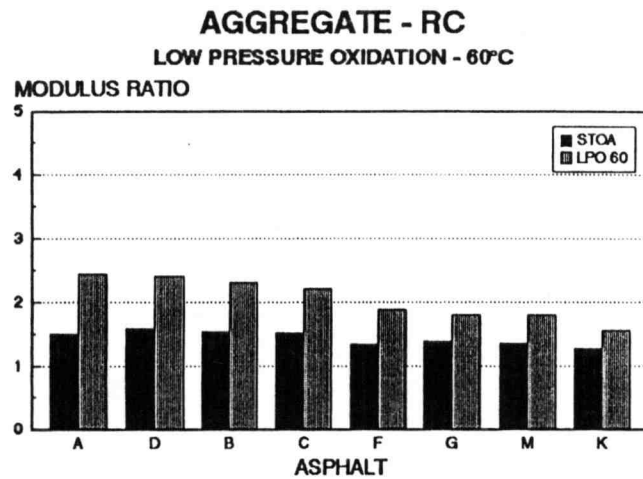


Figure 4.2. Long-term aging by low pressure oxidation at 60°C: rankings based on diametral modulus

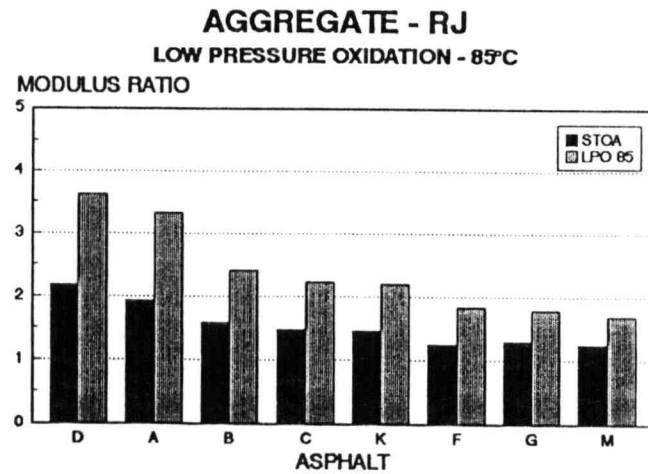
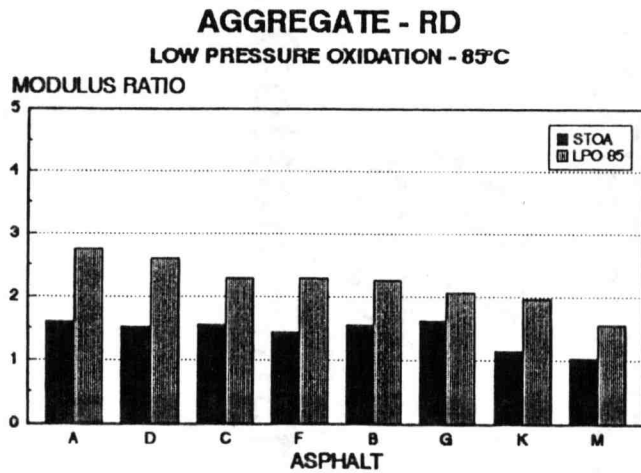
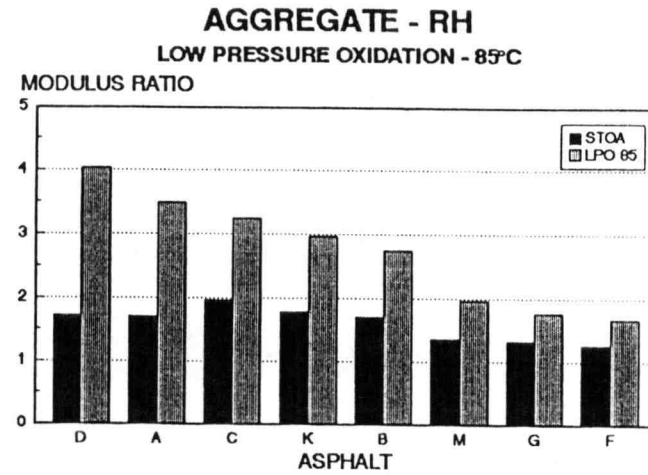
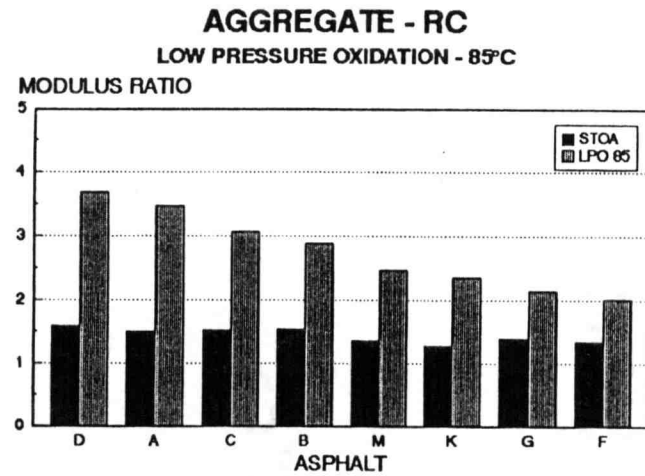


Figure 4.3. Long-term aging by low pressure oxidation at 85°C: rankings based on diametral modulus

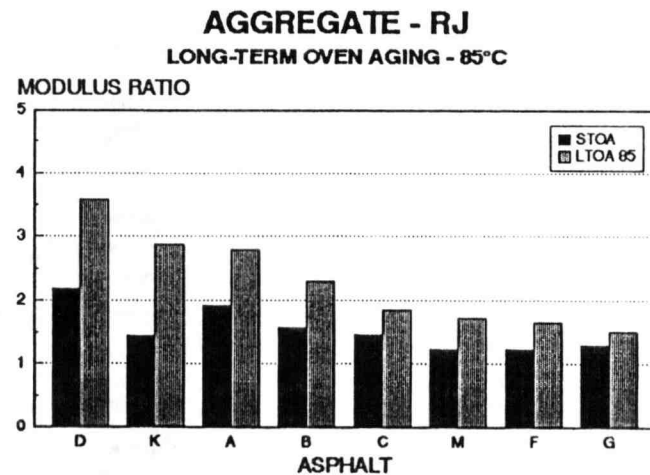
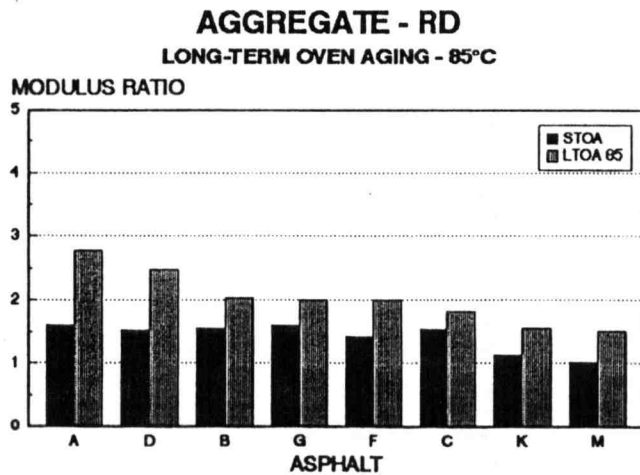
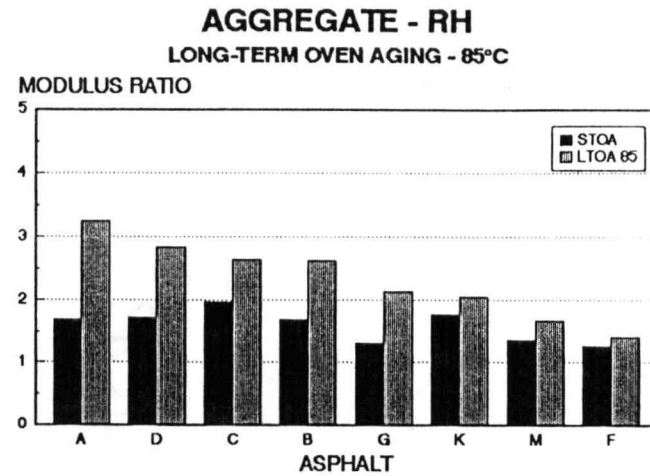
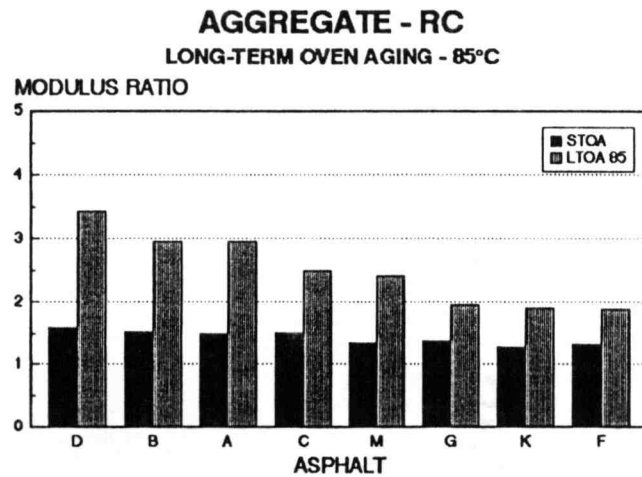


Figure 4.4. Long-term aging by long-term oven aging at 85°C: based on diametral modulus

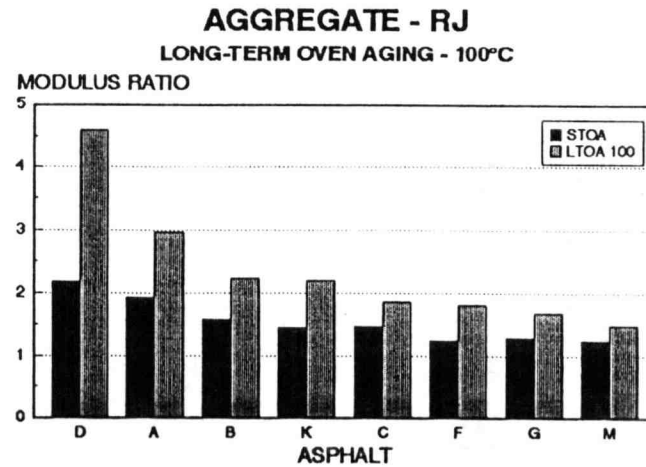
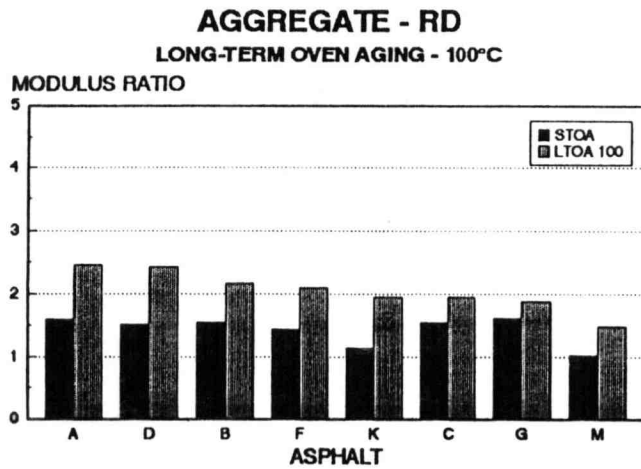
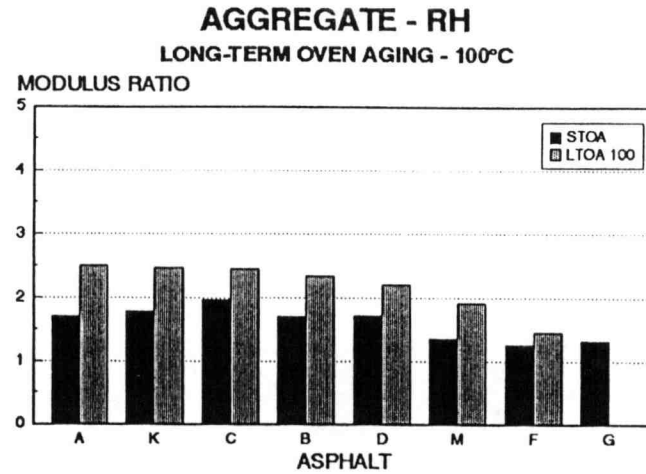
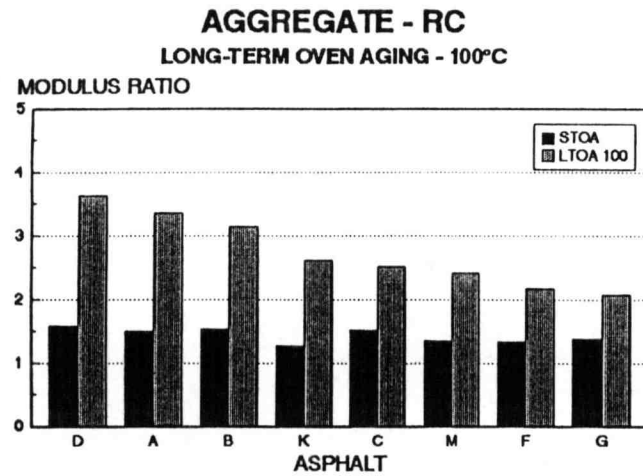


Figure 4.5. Long-term aging by long-term oven aging at 100°C: based on diametral modulus

## **5.0 Analysis of Results**

### **5.1 Statistical Analysis**

Statistical analysis of the short-term and long-term aging data was completed using the General Linear Model (GLM) procedure in the SAS/STAT software package produced by the SAS Institute, Inc. Specifically, the GLM procedure was used to run analysis of variance (ANOVA) and to rank the asphalts in terms of their aging susceptibility using the Waller-Duncan ranking method.

#### **5.1.1 Data Description**

The data were analyzed by aggregate group. The only source variable used was the asphalt group. Prior to statistical analysis, all of the data had been normalized to the same air void content to eliminate that influence on the data.

The dependent variable considered was either the short-term or long-term aging ratio. The short-term ratio is defined as the resilient modulus determined after the short-term aging process divided by the adjusted baseline modulus for that particular specimen. The long-term ratio is the resilient modulus value taken after a particular long-term aging procedure divided by the adjusted baseline modulus for that specimen.

#### **5.1.2 Waller Groupings**

The ANOVA F statistic tells us that there are statistically significant differences in the means, although it does not identify which ones are different. The Waller method is a way of determining which means in a group are

statistically different than the others. The Waller-Duncan method was first proposed by Waller and Duncan (1969) and makes use of a different set of assumptions than most of the other methods of ranking groups by multiple comparison. The Waller-Duncan method minimizes the Bayes risk under additive loss rather than control the type I error rates as do most of the other methods. The Waller-Duncan method is less conservative in grouping means together than most of the other methods, allowing more distinct groups to be formed within the analysis.

## 5.2 Short-Term Aging of Asphalt-Aggregate Mixes

The data presented in Figure 4.1 suggest that mix aging susceptibility is aggregate dependent. However, the effect of the asphalt is more significant. The rankings of the eight asphalts based on short term aging (Figure 4.1) vary with aggregate type. In particular, asphalt AAK-1 moves around in the rankings, showing relatively little aging with basic aggregates (RC and RD) and relatively high aging with acidic aggregates (RH and RJ).

The observed aging phenomena appears to be related to the adhesion of the asphalt and aggregate. A hypothesis is that the greater the adhesion, the greater the mitigation of aging. It should be noted that there is not a statistically significant difference between all asphalts, rather, for a particular aggregate, two or more asphalts show a similar degree of aging. This is illustrated in Table 5.1, which shows numerical rankings corresponding to the short-term aging rankings shown in Figure 4.1. The asphalts within the bracketed areas are groups of statistically similar aging ratios as determined by Waller groupings. When groupings are examined, it can be seen that only asphalt AAM-1 is consistently in the lowest group, and that only asphalt AAD-1 is consistently in the upper group.

### 5.2.1 Comparison of Mix Aging by Short-Term and Long-Term Aging Methods

The numerical rankings of aging presented in Tables 5.1 through 5.5 are summarized in Table 5.6. Comparison of the short-term aging rankings with the long-term aging rankings shows that small movements in the rankings are common. However, using the short-term rankings as a datum, only a few asphalts move more than two places in the rankings, as Table 5.6 shows. These comparisons imply that the low pressure oxidation aging procedure relates more closely to the short-term aging rankings than does the long-term oven aging procedure. This may be because of the greater potential for a specimen to be damaged in the long-term oven aging procedure. This could be the cause of the greater variability in the long-term oven aging specimens, particularly for the 100°C (212°F) procedure. It should be noted that the short-term aging rankings are based on data from eight specimens, while those for each set of long-term aged specimens are based on data from only two specimens. Hence, more variability in the data is expected for long-term aging.

## 5.3 Long-Term Aging of Asphalt-Aggregate Mixes

The data for long-term aging (Figure 4.2 through 4.5) support those for short-term aging, i.e., they also suggest that aging is aggregate-dependent as well as asphalt-dependent. Tables 5.2 through 5.5 present the rankings numerically and show which groups of asphalt are statistically similar, again using Waller groupings. Note that there appears to be greater differentiation among asphalts following long-term aging than after short-term aging, and that this differentiation becomes more pronounced with the severity of the aging procedure.

## 5.4 Comparison of Mix Aging by Short-Term and Long-Term Aging Methods

The numerical rankings of aging presented in Tables 5.1 through 5.5 are summarized in Table 5.6. Comparison of the short-term aging rankings to the long-term aging rankings shows that small movements in the rankings are common. However, using the short-term rankings as a datum, only a few asphalts move more than two places in the rankings, as Table 5.6 shows. These comparisons imply that the low pressure oxidation technique relates more closely to the short-term oven aging ranking than does the long-term oven aging procedure. This may be because of the higher potential for a specimen to be damaged in the long-term oven aging procedure. This causes more variability in the long-term oven aged specimens. It should also be noted that the short-term aging rankings are based on data from eight specimens, those for each set of long-term aged specimens are based on data from two specimens. Hence, more variability in the data is expected for long-term aging.

## 5.5 Comparison of Mix Aging With Asphalt Aging

Aging of asphalt cement has been carried out by the SHRP A-002A contractor. Data for original (tank), thin film oven (TFO) aged, and pressure aging vessel (PAV) aged asphalt have been presented in several A-002A reports. These routine data have been summarized by Christensen and Anderson (1992). As with the mix aging data, the asphalt aging data can be used to calculate an aging ratio based on the aged viscosity at 60°C (140°F) compared to the original viscosity at 60°C (140°F). The asphalts can then be ranked in order of aging susceptibility. Table 5.7 shows the routine asphalt data and the calculated viscosity ratios.

**Table 5.1. Short-term rankings by aggregate**

Aggregate	Ranking														
RC	D	>	B	>	C	>	A	>	G	>	M	>	F	>	K
	1.59		1.53		1.52		1.58		1.39		1.35		1.34		1.28
RD	G	>	A	>	B	>	C	>	D	>	F	>	K	>	M
	1.62		1.61		1.56		1.55		1.53		1.44		1.14		1.03
RH	C	>	K	>	D	>	B	>	A	>	M	>	G	>	F
	1.97		1.78		1.72		1.70		1.70		1.36		1.32		1.26
RJ	D	>	A	>	B	>	C	>	K	>	G	>	F	>	M
	2.18		1.93		1.58		1.47		1.45		1.30		1.24		1.24

Note: Waller groupings of statistically similar behavior are identified with brackets.

**Table 5.2. Long-term aging by low pressure oxidation at 60°C: rankings by aggregate**

Aggregate	Ranking												
RC	A	>	D	>	B	>	C	>	F	>	G	>	M > K
	2.44		2.40		2.31		2.21		1.88		1.80		1.80 1.56
	<div> <div></div> <div></div> <div></div> <div></div> <div></div> <div></div> <div></div> <div></div> <div></div> <div></div> <div></div> <div></div> <div></div> <div></div> </div>												
RD	G	>	C	>	A	>	D	>	B	>	F	>	K > M
	2.21		2.07		1.95		1.95		1.82		1.78		1.70 1.37
	<div> <div></div> <div></div> <div></div> <div></div> <div></div> <div></div> <div></div> <div></div> <div></div> <div></div> <div></div> <div></div> <div></div> <div></div> </div>												
RH	K	>	C	>	D	>	B	>	A	>	G	>	M > F
	2.38		2.36		2.26		2.17		2.17		1.79		1.67 1.58
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RJ	D	>	A	>	B	>	C	>	K	>	M	>	G > F
	2.99		2.40		2.14		2.03		1.99		1.66		1.63 1.51
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Note: Waller groupings of statistically similar behavior are identified with brackets.

**Table 5.3. Long-term aging by low pressure oxidation at 85°C: rankings by aggregate**

Aggregate	Ranking											
RC	D	>	A	>	C	>	B	>	M	>	K	>
	3.69		3.47		3.07		2.88		2.47		2.36	
RD	A	>	D	>	C	>	F	>	B	>	G	>
	2.76		2.61		2.30		2.29		2.26		2.07	
RH	D	>	A	>	C	>	K	>	B	>	M	>
	4.03		3.49		3.24		2.97		2.75		1.97	
RJ	D	>	A	>	B	>	C	>	K	>	F	>
	3.63		3.32		2.40		2.23		2.20		1.84	

Note: Waller groupings of statistically similar behavior are identified with brackets.

**Table 5.4. Long-term oven aging at 85°C: rankings by aggregate**

Aggregate	Ranking											
RC	D	>	B	>	A	>	C	>	M	>	G	>
	3.43		2.95		2.95		2.49		2.41		1.96	
RD	A	>	D	>	B	>	G	>	F	>	C	>
	2.78		2.48		2.04		2.01		2.00		1.83	
RH	A	>	D	>	C	>	B	>	G	>	K	>
	3.26		2.84		2.65		2.63		2.13		2.05	
RJ	D	>	K	>	A	>	B	>	C	>	M	>
	3.58		2.88		2.80		2.31		1.86		1.73	

Note: Waller groupings of statistically similar behavior are identified with brackets.

**Table 5.5. Long-term oven aging at 100°C: rankings by aggregate**

Aggregate	Ranking												
RC	D	>	A	>	B	>	K	>	C	>	M	>	F > G
	3.63		3.36		3.14		2.62		2.52		2.42		2.18 2.07
		>	D	>	B	>	F	>	K	>	C	>	G > M
	2.46		2.42		2.16		2.09		1.95		1.95		1.88 1.49
RH	A	>	K	>	C	>	B	>	D	>	M	>	F > G
	2.51		2.48		2.45		2.34		2.21		1.91		1.45
RJ	D	>	A	>	B	>	K	>	C	>	F	>	G > M
	4.59		2.97		2.22		2.19		1.86		1.81		1.68 1.49

Note: Waller groupings of statistically similar behavior are identified with brackets.

**Table 5.6 Ranking of Asphalt for Each Aggregate Based on Diametral Modulus Ratios and Aging Method**

	Short-Term Oven Aging Aggregate				Low Pressure Oxidation at 60°C				Low Pressure Oxidation at 85°C				Long-Term Oven Aging at 85°C				Long-Term Oven Aging at 100°C			
	RC	RD	RH	RJ	RC	RD	RH	RJ	RC	RD	RH	RJ	RC	RD	RH	RJ	RC	RD	RH	RJ
Worst	D	G	C	D	A	G	K	D	D	A	D	D	D	A	A <sup>4</sup> <sub>1</sub>	D	D	A	A <sup>4</sup> <sub>1</sub>	D
	B	A	K	A	D	C	C	A	A	D <sup>3</sup> <sub>1</sub>	A <sup>3</sup> <sub>1</sub>	A	B	D <sup>3</sup> <sub>1</sub>	D	K <sup>3</sup> <sub>1</sub>	A	D <sup>3</sup> <sub>1</sub>	K	A
	C	B	D	B	B	A	D	B	C	C	C	B	A	B	C	A	B	B	C	B
	A	C	B	C	C	D	B	C	B	F	K	C	C	G <sup>1</sup> <sub>3</sub>	B	B	K <sup>4</sup> <sub>1</sub>	F	B	K
	G	D	A	K	F	B	A	K	M	B	B	K	M	F	G	C	C	K	D	C
	M	F	M	G	G	F	G	M	K	G <sup>1</sup> <sub>5</sub>	M	F	G	C	K <sup>1</sup> <sub>3</sub>	M	M	C	M	F
	F	K	G	F	M	K	M	G	G	K	G	G	K	K	M	F	F	G <sup>1</sup> <sub>6</sub>	F	G
	K	M	F	M	K	M	F	F	F	M	F	M	F	M	F	G	G <sup>1</sup> <sub>3</sub>	M		M
Best																				

**Key:** A shaded block indicates an asphalt that changes more than two rankings relative to the short-term aging rankings. The arrow and adjacent number indicate the number of places moved and the direction.

### 5.5.1 Short-Term Aging

Table 5.8 shows rankings for mixes based on short-term aging and for asphalts based on TFO aging. It should be noted that TFO aging is analogous to short-term mix aging, and that (as with the mix rankings) the differences between some asphalts are not statistically significant. Nevertheless, it is clear that there is little relationship between the mix rankings and the asphalt rankings. The major similarity is that asphalt AAM-1 is one of the two "best" asphalts in both mix and asphalt short-term aging. A major difference is that asphalt AAK-1 is ranked one of the two "worst" from asphalt TFO aging and among the two "best" if STOA aging with aggregates RC and RD is considered.

### 5.5.2 Long-Term Aging

Table 5.9 shows the rankings for mixes based on long-term aging by low pressure oxidation at 85°C (185°F), and rankings for asphalt developed from the data reported by Christensen and Anderson (1992). Also summarized are rankings developed from data reported by Robertson et al. (1991b) for asphalt recovered from "mixes" of single size fine aggregate and from asphalt subjected to pressure aging.

As with the short-term aging comparisons, there is little similarity between the rankings for long-term aging of mixes and those for asphalt alone. In fact, there is even less similarity, since asphalt AAM-1 appears to have more susceptibility to long-term aging in the PAV than it does in the TFO (relative to the other asphalts), as shown by its movement in the rankings.

There is more similarity between the rankings based on mix aging and those based on the data for fine aggregate mixes developed by the A-003A contractor. However, the rankings are different as indicated in Table 5.8.

**Table 5.7. Summary of routine test data for asphalt alone**

	Asphalt							
	AAA-1 (150/200)	AAB-1 (AC-10)	AAC-1 (AC-8)	AAD-1 (AR-4000)	AAF-1 (AC-20)	AAG-1 (AR-4000)	AAK-1 (AC-30)	AAM-1 (AC-20)
<b>ORIGINAL ASPHALT</b>								
Viscosity (60°C) (Poises)	900	1120	710	1140	1750	1950	3320	2040
<b>AGED ASPHALT (THIN FILM OVEN TEST)</b>								
Viscosity (60°C) (Poises)	2080	2620	1780	3690	4560	3490	10240	4490
Viscosity Ratio (60°C TFO Aged/Original)	2.31	2.34	2.51	3.24	2.61	1.79	3.08	2.20
<b>LONG-TERM AGED (PAV)</b>								
Viscosity (60°C) (Poises)	5380	7110	5170	12000	16250	8140	27300	17150
Viscosity Ratio (60°C, PAV Aged/Original)	5.98	6.35	7.28	10.53	9.29	4.17	8.22	8.41

**Table 5.8. Comparison of rankings for short-term aging mixtures and asphalt alone**

Ranking of Asphalt						
	A-003A <sup>1</sup>				A-002A <sup>2</sup>	
	Aggregate RC	Aggregate RD	Aggregate RH	Aggregate RJ	Average of A-003A Rankings	No Aggregate
Worst	D	G	C	D	D	D
	B	A	K	A	A	K
	C	B	D	B	C	F
	A	C	B	C	B	C
	G	D	A	K	K	B
	M	F	M	G	G	A
	F	K	G	F	F	M
Best	K	M	F	M	M	G

<sup>1</sup> Based on short-term aging ratios from diametral modulus.<sup>2</sup> Based on data reported by Christensen and Anderson (1992).

**Table 5.9. Comparison of rankings for long-term aging of mixtures and asphalts**

Ranking of Asphalts									
	A-003A <sup>1</sup>				A-002A <sup>2</sup>	A-002A <sup>3</sup>		A-002A <sup>4</sup>	Average of A-003A Rankings
	Aggregate RC	Aggregate RD	Aggregate RH	Aggregate RJ	No Aggregate	Aggregate RD	Aggregate RJ	Aggregated RD	
Worst	D	A	D	D	D	F	D	F	D
	A	D	C	A	F	M <sub>1</sub> <sup>6</sup>	B	M <sub>1</sub> <sup>6</sup>	A
	C	C	A	B	M	D	F <sub>1</sub> <sup>3</sup>	C	C
	B	F	C	C	K	C	C	D	B
	M	B	K	K	C	A <sub>3</sub> <sup>4</sup>	M <sub>1</sub> <sup>3</sup>	G	K
	K	G	M	F	B	K	A <sub>4</sub> <sup>4</sup>	A <sub>4</sub> <sup>4</sup>	F
	G	K	G	G	A	G	K	B <sub>3</sub> <sup>4</sup>	G
Best	F	M	F	M	G	B <sub>4</sub> <sup>4</sup>	G	K	M

<sup>1</sup> Based on long-term aging ratios from diametral modulus for low pressure oxidation aging.

<sup>2</sup> Based on data reported by Christensen and Anderson (1992) for TFO-PAV aging.

<sup>3</sup> Reported in 4th Quarterly Report, 1991, based on PAV aging at 60°C for 144 hours. Prior short-term aging.

<sup>4</sup> Reported in 4th Quarterly Report, 1991. Asphalt alone was subjected to TFO aging prior to mixing and PAV aging.

## 5.6 General Discussion

The differences in rankings between mixes and asphalt, based on either short-term or long-term aging data indicate the need for mix testing to evaluate the aging susceptibility of a mix. Clearly, the aging of the asphalt alone, or in a fine aggregate mix, is not an indicator of how a mix will age. This need is caused by the influence of the aggregate on mix aging, which appears to be related to the chemical interaction of the aggregate and the asphalt. This interaction may be related to adhesion; the greater the adhesion, the greater the mitigation of aging. The mix aging rankings given in Tables 5.7 and 5.8 confirm this hypothesis, since the rankings are similar for two "basic" aggregates (RC and RD) and for the two "acidic" aggregates (RH and RJ). Some of the asphalts rank similarly regardless of the aggregate types, while others (such as AAG-1 and AAK-1) behave very differently depending on the aggregate types. It is known that asphalt AAG-1 was lime treated in the refining process and it is therefore rational that it would exhibit good adhesion and reduce aging tendency with the acidic aggregates (RH and RJ), as the short-term aging data indicate (Table 5.7). However, the rankings of asphalt AAG-1 for long-term aging do not appear to be influenced by aggregate type! Asphalt AAK-1 is known to be unusually chemically active. For instance, its high aging tendency when subjected to TFO aging is attributable to its reactivity. This reactivity appears to mitigate its tendency to age when in a mix, particularly with basic aggregates.

Tensile test data and dynamic modulus data using frequency sweep testing (dynamic mechanical analysis, DMA) for a subset of the 32 mixes used are presented in Appendices E and F, respectively. As mentioned in the appendices, both the tensile strength test and the DMA procedure are able to differentiate between the aging methods and different mixes. The tensile strength test is not as sensitive to aging changes as the resilient modulus test. The data obtained from DMA testing are much more difficult to interpret than the resilient modulus data.

For these reasons, resilient modulus was the measurement tool used most extensively in this analysis.

A major drawback of the comparison included here is the use of resilient modulus values determined at one loading time and at one temperature to generate rankings of aging susceptibility. The use of DMA parameters which describe behavior over a wide range of conditions is much more desirable. Similarly, the use of viscosity at 60°C (140°F) to generate rankings of asphalt aging susceptibility is much less desirable than considering asphalt DMA parameters, such as those developed by the A-002A contractor. Time constraints have not permitted comparisons of mixes and asphalt DMA parameters.

Another problem with the comparisons made herein is the difference in the definition of an "unaged" condition for mixes and asphalt. For mixes, the "unaged" condition can only be defined by tests on laboratory specimens that are compacted immediately after mixing. Inevitably, the mixing process "ages" the asphalt, perhaps by a substantial amount. This is in contrast to the "unaged" condition for the asphalt which is defined by tests on the original asphalts and which represents a true unaged condition.

## 6.0 Conclusions and Recommendations

### 6.1 Conclusions

The following conclusions may be drawn from the results of this study:

- 1) The aging of asphalt-aggregate mixes is influenced by both the asphalt and the aggregate.
- 2) Aging of the asphalt alone and subsequent testing does not appear to be an adequate means of predicting mix performance because of the apparent mitigating effect aggregate has on aging.
- 3) The aging of certain asphalts is strongly mitigated by some aggregates but not by others. This appears to be related to the strength of the chemical bonding (adhesion) between the asphalt and aggregate.
- 4) The short-term aging procedure produces a change in resilient modulus of up to a factor of two. For a particular aggregate, there is not a statistically significant difference in the aging of certain asphalts. The eight asphalts investigated typically fell into three groups, i.e., those with high, medium, and low aging susceptibility.
- 5) The three long-term aging methods produced somewhat different rankings of aging susceptibility compared to the short-term aging procedure and to each other. This is partially attributable to variability in the materials, aging processes, and testing. However, it appears that the short-term aging procedure does not enable prediction of long-term aging.
- 6) The low pressure oxidation long-term aging procedure causes the most aging and less variability in the aging susceptibility rankings relative to the short-term aging rankings.

## 6.2 Recommendations

Based on the results of this study and the companion test selection and field validation test program, the following recommendation are made:

- 1) Oven aging of loose mix at 135°C (275°F) is recommended for short-term aging. An aging period of 4 h appears to be appropriate.
- 2) Oven aging of compacted mixes should be adopted for long-term aging of dense mixes. A temperature of 85°C (185°F) is most appropriate for period of 5 days. It may be possible to use a temperature of 100°C (212°F) for 2 days. However, use of such a high temperature may cause damage to specimens.
- 3) A low pressure oxidation (triaxial cell) technique is recommended for long-terms of open graded mixes or dense graded mixes using soft grades of asphalt. A temperature of 85°C (185°F) is most appropriate for a period of 5 days. It may be possible to use a temperature of 100°C (212°F) for 2 days. However, use of such a high temperature may cause damage to specimens.

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## **Appendices**

## **Appendix A**

### **Short-Term Aging of Asphalt Concrete**

SHRP #1025

#### **Standard Practice for**

### **Short-Term Aging of Asphalt Concrete Mixes**

AASHTO DESIGNATION: T ###-YY  
(ASTM DESIGNATION: D ####-YY)

#### **A.1 Scope**

**1.1** This standard is used to simulate the short-term aging of asphalt concrete mixes. Short-term aging considers the aging undergone by asphalt concrete mixes during field plant mixing operations.

**1.2** This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

**1.3** The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

#### **A.2 Referenced documents**

##### **2.1 AASHTO Documents:**

M ###	Performance Graded Asphalt Binders
R 11	Practice for Indicating Which Places of Figures are to be Considered Significant in Specifying Limiting Values
T 2	Method of Sampling Aggregates
T 27	Method for Sieve Analysis of Fine and Coarse Aggregates
T 40	Method of Sampling Bituminous Materials
T 201	Method for Kinematic Viscosity of Asphalts

## **2.2 ASTM Documents:**

- D 8 Standard Definitions of Terms Relating to Materials for Roads and Pavements
- E 1 Specification for Thermometers

## **A.3 Terminology**

**3.1 Desired Mixing Temperature** - the target temperature for mixing asphalt binder and aggregate in the laboratory. The desired mixing temperature selected should be equivalent to the anticipated field plant mixing temperature. If field mixing temperatures are unknown, select a temperature which corresponds to a kinematic viscosity of  $170 \pm 20$  cS for the asphalt binder which is used.

**3.2** Definitions for many terms common to asphalt are found in the following documents:

- 3.2.1** Standard Definitions D 8
- 3.2.2** Performance Graded Asphalt Binder M ###
- 3.2.3** Kinematic Viscosity of Asphalts T 201

## **A.4 Summary of practice**

**4.1** A mix of aggregate and asphalt binder is aged in a forced draft oven for 4 h at 135°C (275°F). The oven aging is designed to simulate the aging the mix will undergo during field plant mixing operations.

## **A.5 Significance and use**

**5.1** The short-term aging practice simulates the aging asphalt concrete mixes undergo during field plant mixing operations.

**5.2** The properties and performance of asphalt concrete mixes may be more accurately predicted by using aged test samples.

## A.6 Apparatus

**6.1 Aging Test System** - A system which consists of a forced draft oven which possesses the requirements specified in Table A.1.

**Table A.1. Minimum aging test system requirements**

	Range, °C	Resolution, °C	Accuracy, °C
Temperature Measurement	10 - 260	< 1	± 1
Temperature Control	25 - 250	< 0.1	± 0.1

**6.2 Oven** - Any oven which is thermostatically controlled and capable of being set to maintain any desired temperature from room temperature to 260°C (500°F). The oven shall be used for heating aggregates, asphalt binders or laboratory equipment.

**6.3 Mixing Apparatus** - Any type of mechanical mixer which: 1) can be maintained at the required mixing temperatures, 2) will provide a well coated, homogenous mix of the required amount of asphalt concrete in the allowable time, and 3) allows essentially all of the mix to be recovered.

### **6.4 Miscellaneous Apparatus:**

**6.4.1** One metal oven pan for heating aggregates

**6.4.2** One shallow metal oven pan for heating uncompacted asphalt concrete mixes

**6.4.3** Thermometers having a range from 50° to 260°C (122° to 500°F) and conforming to the requirements for ASTM Thermometer \_\_\_\_\_ as prescribed in E.1

**6.4.4** Metal spatula or spoon

**6.4.5** Oven gloves

## A.7. Hazards

**7.1 Warning** - This test method involves the handling of hot asphalt binder, aggregate and asphalt concrete mixes which can cause severe burns if allowed to contact skin. Proper precautions must be taken to avoid burns.

## **A.8 Sampling**

**8.1** The asphalt binder shall be sampled in accordance with T 40.

**8.2** The aggregate shall be sampled and tested in accordance with T 2 and T 27, respectively.

## **A.9 Specimen preparation**

**9.1** Preheat the aggregate for a minimum of 2 h at the desired mixing temperature. The amount of aggregate preheated should be sufficient to give a mix specimen of the desired size.

**9.2** Preheat the asphalt binder for 1 to 2 h at the desired mixing temperature. The amount of asphalt binder preheated shall be of sufficient quantity to obtain the desired asphalt binder content to be tested. Asphalt binders heated for more than 2 h should be discarded.

**9.3** Mix the heated aggregate and asphalt binder at the desired asphalt content.

## **A.10 Procedure**

**10.1** Place the mix on the baking pan and place the mix and pan in the forced draft oven for  $4\text{ h} \pm 5\text{ min}$  at a temperature of  $135^{\circ} \pm 1^{\circ}\text{C}$  ( $275^{\circ} \pm 1.8^{\circ}\text{F}$ ). The mix should be spread to an even thickness over an appropriate area such that the rate of spread should be  $21\text{--}22\text{ kg/m}^2$  ( $2.2\text{ g/cm}^2$ ).

**10.2** Stir the mix every hour to maintain uniform aging.

**10.3** After 4 h, remove the mix from the forced draft oven. The aged mix is now ready for further conditioning or testing as required.

## **A.11 Report**

**11.1** Report the following information:

**11.1.1** Asphalt Binder Grade

**11.1.2** Asphalt Binder Content - in percent to the nearest 0.1 percent

- 11.1.3      Aggregate Type and Gradation**
- 11.1.4      Mixing Temperature - in °C to the nearest 1°C**
- 11.1.5      Aging Temperature - in °C to the nearest 1°C**
- 11.1.6      Aging Duration - in min to the nearest 1 min**

## **A.12. Keywords**

**12.1** Asphalt concrete, bituminous mixes, bituminous paving mixes, aging, asphalt concrete aging, short-term aging.

## Appendix B

### Long-Term Aging of Asphalt Concrete Mixes by Low Pressure Oxidation

SHRP #1030

Standard Practice for

### Long-Term Aging of Asphalt Concrete Mixes by Low Pressure Oxidation

AASHTO DESIGNATION: T ###-YY  
(ASTM DESIGNATION: D ####-YY)

#### B.1 Scope

1.1 This standard is used to simulate the long-term aging of asphalt concrete mixes. Long-term aging considers the total aging undergone by compacted asphalt concrete mixes during a service life of 5 to 10 years.

1.2 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1.3 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

#### B.2 Referenced documents

##### 2.1 AASHTO Documents:

M ###	Specification for Performance Graded Asphalt Binders
R 11	Practice for Indicating Which Places of Figures are to be Considered Significant in Specifying Limiting Values
T 27	Method for Sieve Analysis of Fine and Coarse Aggregates

T 164	Method for Quantitative Extraction of Bitumen from Paving Mixes
T 168	Method of Sampling Bituminous Paving Mixes
T 201	Method for Kinematic Viscosity of Asphalts
T 269	Method for Percent Air Voids in Compacted Dense and Open Bituminous Paving Mixes SW: See previous note.
T ###	Practice for Short Term Aging of Asphalt Concrete Mixes
T ###	Method for Preparation of Asphalt Concrete Specimens by Means of the SHRP Gyratory Compactor
T ###	Method for Preparation of Asphalt Concrete Specimens by Means of the Rolling Wheel Compactor

## 2.2 ASTM Documents:

D 8	Standard Definitions of Terms Relating to Materials for Roads and Pavements
D 3549	Method for Thickness or Height of Compacted Bituminous Paving Mix Specimens
E 1	Specification for Thermometers

## B.3 Terminology

**3.1 Desired Compaction Temperature** - The target temperature for compacting asphalt concrete mixes in the laboratory. The desired compaction temperature selected should be equivalent to the anticipated field compaction temperature. If field compaction temperatures are unknown, select a compaction temperature which corresponds to a kinematic viscosity of  $665 \pm 80$  cS for the asphalt binder which is used.

**3.2** Definitions for many terms common to asphalt are found in the following documents:

- 3.2.1 Standard Definitions D 8
- 3.2.2 Performance Graded Asphalt Binder M ###
- 3.2.3 Short Term Aging of Asphalt Concrete Mixes T ###
- 3.2.4 Kinematic Viscosity of Asphalts T 201

## B.4 Summary of practice

**4.1** A compacted mix of aggregate and asphalt binder is aged in a low pressure oxidation cell for 5 days at 85°C (185°F). The oven aging is designed to simulate the total aging the compacted mixture will undergo during a 5 to 10 year service life after field placement and compaction.

## B.5 Significance and use

**5.1** The long-term aging practice simulates the in-service aging of asphalt concrete mixes after field placement and compaction.

**5.2** The properties and performance of asphalt concrete mixes and pavements may be more accurately predicted by using aged test samples.

## B.6 Apparatus

**6.1 Aging Test System** - A low pressure oxidation cell and oxygen supply system which are capable of passing oxygen at a constant flow rate through a compacted asphalt concrete specimen and meet the requirements specified in Table B.1. The oxygen supply system (0 to 690 kPa (0 to 100 psi)) shall be equipped with a suitable pressure regulator and gage. The low pressure oxidation cell shall be equipped with the following:

**Table B.1. Minimum aging test system requirements**

	Range	Resolution	Accuracy
Oxygen Flow Control	1-10 scfh	<0.5 scfh	± 0.5 scfh
Oxygen Flow Measurement	1-10 scfh	<0.5 scfh	±0.25 scfh
Oxygen Pressure Measurement	0-600 psi	20 psi	10 psi
Confining Pressure Control	0-100	2 psi	± 1 psi
Confining Pressure Measurement	0-100 psi	2 psi	± 1 psi

**6.1.1 Load Frame Assembly** - as shown in Figure B.1. The load frame assembly shall possess equipment capable of measuring the confining pressure within the cell and providing and measuring oxygen flow to the test specimen.

**6.1.2 Specimen End Platens** - as shown in Figure B.2. The top specimen end platen shall be equipped with connections to provide for the flow of oxygen through the test specimen.

**6.1.3 Perforated Teflon Disks** - as shown in Figure B.3.

**6.2 Oven** - Any oven which is thermostatically controlled and capable of being set to maintain any desired temperature from room temperature to 260°C (500°F). The oven is used for heating aggregates, asphalt binder, asphalt concrete mixes or laboratory equipment.

**6.3 Water Bath** - A water bath which is at least 457 mm (18 in.) deep and is thermostatically controlled so as to maintain the bath at  $85 \pm 1^\circ\text{C}$  ( $185^\circ \pm 1.8^\circ\text{F}$ ). The tank requires a perforated false bottom or to be equipped with a shelf for supporting specimens 51 mm (2 in.) above the bottom of the bath. The water bath should also be equipped such that a constant flow of water is available to replenish any water loss from evaporation. This will maintain a constant water level.

#### **6.4 Miscellaneous Apparatus :**

- 6.4.1** One shallow metal oven pan/sheet about 305 by 457 mm (12 by 18 in.) for heating uncompacted asphalt concrete mixes
- 6.4.2** Thermometers having a range from 50° to 260°C (122° to 500°F) and conforming to the requirements for ASTM Thermometer \_\_\_\_ as prescribed in E 1
- 6.4.3** Waterproof marking sticks for identifying specimens
- 6.4.4** Paper labeling tags
- 6.4.5** Metal spatula or spoon
- 6.4.6** Oven gloves
- 6.4.7** 36 cm (14 in) long by 38 mm wide strip of butcher paper
- 6.4.8** 38 mm (1.5 in) of 102 mm (4 in) diameter rubber membrane
- 6.4.9** 152 mm (6 in) of 102 mm (4 in) diameter rubber membrane
- 6.4.10** One specimen holder similar to that shown in Figure B.4
- 6.4.11** Two 102 mm (4 in) by 1/8 in thick O-Rings

## **B.7 Materials**

**7.1** The following materials are required:

- 7.1.1** Oxygen for Aging Test System
- 7.1.2** Clear rubber silicone

## **B.8 Hazards**

**8.1 Warning** - This test method involves the handling of hot asphalt binder, aggregate and asphalt concrete mixes which can cause severe burns if allowed to contact skin. Proper precautions must be taken to avoid burns.

## **B.9 Sampling**

**9.1** The asphalt concrete mixes shall be sampled in accordance with T 168, or shall consist of specimens which have sampled and short-term aged in accordance with T ###

**9.2** Compacted roadway samples shall have a cut specimen size which is  $102 \pm 6$  mm ( $4 \pm 0.25$  in.) in diameter by  $152 \pm 6$  mm ( $6 \pm 0.25$  in.) in height.

## **B.10 Specimen preparation**

### **10.1 Compacting Laboratory Mix Samples**

**10.1.1** Heat the asphalt concrete to the desired compaction temperature.

**10.1.2** Compact an amount of asphalt concrete mix sufficient to give the desired specimen size in accordance with T ###.

**NOTE 1** - Compact a sufficient amount of material to ensure that the final specimen size after 9.1.4 is  $102 \pm 6$  mm in diameter by  $152 \pm 6$  mm in height.

**10.1.3** Cool the compacted specimen to  $60^{\circ} \pm 1^{\circ}\text{C}$  ( $140^{\circ} \pm 1.8^{\circ}\text{F}$ ).

**10.1.4** After cooling the specimen, apply a static load at a rate of 16,000 lb/min to a maximum of 5715 kg (12,600 lb), then release the load at the same rate. This procedure is to level the ends of the specimen.

**10.1.5** After cooling the specimen to room temperature overnight, extrude the specimen from the compaction mold.

### **10.2 Sealing Compacted Laboratory and Roadway Specimens**

**10.2.1** Place the specimens in a specimen holder and apply a

sufficient bead of silicone around the circumference of the specimen at mid height. Apply a large enough bead to uniformly cover a 38 mm (1.5 in.) strip of the specimen at mid height. Cover the bead with the 38-mm length of cylindrical rubber membrane and mold the encapsulated silicone to a uniform thickness with your fingers. Allow the specimen to stand at room temperature, overnight or longer, until the silicone is dry.

- 10.3.1** After the silicone has dried, cover the exposed portions (i.e. portions not covered with the rubber membrane) of the specimen with 2 strips of butcher paper.

**NOTE 2** - Covering the exposed portions of the specimen is extremely important as large air voids or sharp edges may cause the rubber membrane to rupture under confining pressures at high temperatures. If the rubber membrane ruptures during testing, the specimen should be discarded.

## **B.11 Procedure**

**11.1** Place the 152-mm length of cylindrical rubber membrane around the specimen. Place one O-ring around each end of the membrane to hold it in place over the specimen.

**11.2** Place a perforated teflon disk on top of the grooved surface of the bottom end platen.

**11.3** Place the specimen vertically on top of the teflon disk and bottom end platen.

**11.4** Place a perforated teflon disk on top of the specimen and place the top end platen on top of the disk.

**11.5** Place the specimen and platen assembly within the load frame and place the walls of the pressure vessel over the specimen.

**11.6** Connect the oxygen tubes between the top end platen and the top plate of the load frame. With the top plate of the load frame in place tighten the screws until the cell is sealed.

**11.7** Turn on the confining pressure within the cell and then turn on the oxygen flow. Stabilize the oxygen flow at  $32 \pm \text{___ cm}^3/\text{s}$  ( $4 \pm 0.5 \text{ ft}^3/\text{h}$ ) and monitor the corresponding pressure. Monitor and adjust the confining pressure until it is 34 to 69 kPa (5 to 10 psi) greater than the oxygen pressure.

**11.8** Place the entire cell in a  $85 \pm 1^{\circ}\text{C}$  ( $185^{\circ} \pm 1.8^{\circ}\text{F}$ ) bath for 5 days  $\pm 0.5$  h. Periodically monitor the oxygen flow to ensure that there is a continuous supply.

**11.9** After 5 days, turn off the oxygen flow and release the confining pressure. Remove the cell from the water bath and allow the entire assembly to cool to  $25^{\circ}\text{C}$  ( $77^{\circ}\text{F}$ ).

**11.10** Remove the specimen from the cell. Remove the rubber membranes and silicone from the specimen. The aged specimen is now ready for further testing as required.

## **B.12 Report**

**12.1** Report the following information:

**12.1.1 Asphalt Binder Grade**

**12.1.2 Asphalt Binder Content** - in percent to the nearest 0.1 percent

**12.1.3 Aggregate Type and Gradation**

**12.1.4 Short-Term Aging Conditions** - the following information as applicable:

**12.1.4.1 Plant Mixing Temperature** - in  $^{\circ}\text{C}$  to the nearest  $1^{\circ}\text{C}$

**12.1.4.2 Laboratory Mixing Temperature** - in  $^{\circ}\text{C}$  to the nearest  $1^{\circ}\text{C}$

**12.1.4.3 Short-Term Aging Temperature in Laboratory** - in  $^{\circ}\text{C}$  to the nearest  $1^{\circ}\text{C}$

**12.1.4.4 Short-Term Aging Duration in Laboratory** - in min to the nearest 1 min

**12.1.5 Compaction Temperature** - in  $^{\circ}\text{C}$  to the nearest  $1^{\circ}\text{C}$

**12.1.6 Compacted Specimen Height** - in mm to the nearest 1 mm

**12.1.7 Compacted Specimen Diameter** - in mm to the nearest 1 mm

**12.1.8 Compacted Specimen Density** - in  $\text{kg/m}^2$  to the nearest 1  $\text{kg/m}^2$

**12.1.9 Compacted Specimen Air Voids** - in percent to the nearest 0.1 percent

**12.1.10 Long-Term Aging Oxygen Flow** - in  $\text{cm}^3/\text{s}$  to the nearest 236  $\text{cm}^3/\text{s}$

**12.1.11 Long-Term Aging Oxygen Pressure** - in kPa to the nearest 69 kPa (10 psi)

**12.1.12 Long-Term Aging Confining Pressure** - in Kpa to the nearest 6.9 kPa (1 psi)

**12.1.13 Long-Term Aging Duration** - in min to the nearest 1 min

**12.1.14 Long-Term Aging Bath Temperature** - in  $^{\circ}\text{C}$  to the nearest  $1^{\circ}\text{C}$

## **B.13 Keywords**

**13.1** Asphalt concrete, bituminous mixes, bituminous paving mixes, aging, long-term aging, asphalt concrete aging, low pressure oxidation cell.

## Appendix C

### Long-Term Aging of Asphalt Concrete Mixes by Long-Term Oven Aging

SHRP #1031

Standard Practice for

### Long-Term Aging of Asphalt Concrete Mixes by Long-Term Oven Aging

AASHTO DESIGNATION: T ###-YY  
(ASTM DESIGNATION: D ####-YY)

#### C.1 Scope

1.1 This standard is used to simulate the long-term aging of asphalt concrete mixes. Long-term aging considers the total aging undergone by compacted asphalt concrete mixes during a service life of 5 to 10 years.

1.2 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1.3 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

#### C.2 Referenced documents

##### 2.1 AASHTO Documents:

M ###	Specification for Performance Graded Asphalt Binders
R 11	Practice for Indicating Which Places of Figures are to be Considered Significant in Specifying Limiting Values
T 27	Method for Sieve Analysis of Fine and Coarse Aggregates
T 164	Method for Quantitative Extraction of Bitumen from Paving Mixes
T 168	Method of Sampling Bituminous Paving Mixes

T 201	Method for Kinematic Viscosity of Asphalts
T 269	Method for Percent Air Voids in Compacted Dense and Open Bituminous Paving Mixes
T ###	Practice for Short-Term Aging of Asphalt Concrete Mixes
T ###	Method for Preparation of Asphalt Concrete Specimens by Means of the SHRP Gyratory Compactor
T ###	Method for Preparation of Asphalt Concrete Specimens by Means of the Rolling Wheel Compactor

## **2.2 ASTM Documents:**

D 8	Standard Definitions of Terms Relating to Materials for Roads and Pavements
D 3549	Method for Thickness or Height of Compacted Bituminous Paving Mix Specimens
E 1	Specification for Thermometers

## **C.3 Terminology**

**3.1 Desired Compaction Temperature** - The target temperature for compacting asphalt concrete mixes in the laboratory. The desired compaction temperature selected should be equivalent to the anticipated field compaction temperature. If field compaction temperatures are unknown, select a compaction temperature which corresponds to a kinematic viscosity of  $665 \pm 80$  cS for the asphalt binder which is used.

**3.2** Definitions for many terms common to asphalt are found in the following documents:

- 3.2.1** Standard Definitions D 8
- 3.2.2** Performance Graded Asphalt Binder M ###
- 3.2.3** Short-Term Aging of Asphalt Concrete Mixes T ###
- 3.2.4** Kinematic Viscosity of Asphalts T 201

## **C.4 Summary of practice**

**4.1** A compacted mix of aggregate and asphalt binder is aged in a forced draft oven for 5 days at 85°C (185°F). The oven aging is designed to simulate the total aging the compacted mix will undergo during a 5 to 10 year service life after field placement and compaction.

## C.5 Significance and use

**5.1** The long-term aging practice simulates the in service aging of asphalt concrete mixes after field placement and compaction.

**5.2** The properties and performance of asphalt concrete mixes and pavements may be more accurately predicted by using aged test samples.

## C.6 Apparatus

**6.1 Aging Test System** - A system which consists of a forced draft oven which possesses the requirements specified in Table C.1.

**Table C.1. Minimum aging test system requirements**

	Range, °C	Resolution, °C	Accuracy, °C
Temperature Measurement	10 - 260	< 1	± 1
Temperature Control	25 - 250	< 0.1	± 0.1

**6.2 Oven** - Any oven which is thermostatically controlled and capable of being set to maintain any desired temperature from room temperature to 260°C (500°F). The oven shall be used for heating aggregates, asphalt binders or lab equipment.

### **6.3 Miscellaneous Apparatus :**

**6.3.1** One shallow metal oven pan for heating uncompacted asphalt concrete mixes

**6.3.2** Thermometers having a range from 50 to 260°C (122° to 500°F) and conforming to the requirements for ASTM Thermometer \_\_\_\_\_ as prescribed in E 1

**6.3.3** Metal spatula or spoon

**6.3.4** Oven gloves

## C.7 Hazards

**7.1 Warning** - This test method involves the handling of hot asphalt binder, aggregate and asphalt concrete mixes which can cause severe burns if allowed to contact skin. Proper precautions must be taken to avoid burns.

## C.8 Sampling

**8.1** Field asphalt concrete mixes shall be sampled in accordance with T 168. Laboratory prepared asphalt concrete mixes shall be prepared and short-term aged in accordance with T-###.

**8.2** Compacted roadway samples shall have a cut specimen size which is  $102 \pm 6$  mm ( $4 \pm 0.25$  in.) in diameter by  $152 \pm 6$  mm ( $6 \pm 0.25$  in.) in height.

## C.9 Specimen preparation

### 9.1 Uncompacted Laboratory Mix Samples

**9.1.1** Heat the asphalt concrete to the desired compaction temperature.

**9.1.2** Compact a sufficient amount of mix to give the desired specimen size in accordance with T ###.

**NOTE 1** - Compact a sufficient amount of material to ensure that the final specimen size after 9.1.4 is  $102 \pm 6$  mm in diameter by  $152 \pm 6$  mm in height.

**9.1.3** Cool the compacted specimen to  $60^\circ \pm 1^\circ\text{C}$  ( $140^\circ \pm 1.8^\circ\text{F}$ ) in an oven set at  $60^\circ\text{C}$  ( $140^\circ\text{F}$ ). This will take approximately 2 h for a specimen that is  $102 \pm 6$  mm in diameter by  $152 \pm 6$  mm in height.

**9.1.4** After cooling the specimen, apply a static load at a rate of 16,000 lb/min to a maximum value of 5715 kg (12,600 lb), then release the load at the same rate. This procedure is to level the ends of the specimen.

**9.1.5** After cooling the specimen at room temperature overnight, extrude the specimen from the compaction mold.

## C.10 Procedure

**10.1** Place the compacted specimen on a rack in the forced draft oven for  $120 \pm 0.5$  h at a temperature of  $85^\circ \pm 1^\circ\text{C}$  ( $185^\circ\text{C} \pm 1.8^\circ\text{F}$ ).

**10.2** After 120 h, turn the oven off, open the doors and allow the specimen to cool to room temperature. Do not touch or remove the specimen until it has cooled to room temperature. It will take approximately overnight to cool a specimen that is  $102 \pm 6$  mm in diameter by  $152 \pm 6$  mm in height.

**10.3** After cooling to room temperature, remove the specimen from the oven. The aged specimen is now ready for testing as required.

## **C.11 Report**

**11.1** Report the following information:

**11.1.1 Asphalt Binder Grade**

**11.1.2 Asphalt Binder Content** - in percent to the nearest 0.1 percent

**11.1.3 Aggregate Type and Gradation**

**11.1.4 Short-Term Aging Conditions** - the following information as applicable:

**11.1.4.1 Plant Mixing Temperature** - in °C to the nearest 1°C

**11.1.4.2 Laboratory Mixing Temperature** - in °C to the nearest 1°C

**11.1.4.3 Short-Term Aging Temperature in Laboratory** - in °C to the nearest 1°C

**11.1.4.4 Short-Term Aging Duration in Laboratory** - in min to the nearest 1 min

**11.1.5 Compaction Temperature** - in °C to the nearest 1°C

**11.1.6 Compacted Specimen Height** - in mm to the nearest 1 mm

**11.1.7 Compacted Specimen Diameter** - in mm to the nearest 1 mm

**11.1.8 Compacted Specimen Density** - in kg/m<sup>2</sup> to the nearest 1 kg/m<sup>2</sup>

**11.1.9 Compacted Specimen Air Voids** - in percent to the nearest 0.1 percent

**11.1.10 Long-Term Aging Temperature** - in °C to the nearest 1°C

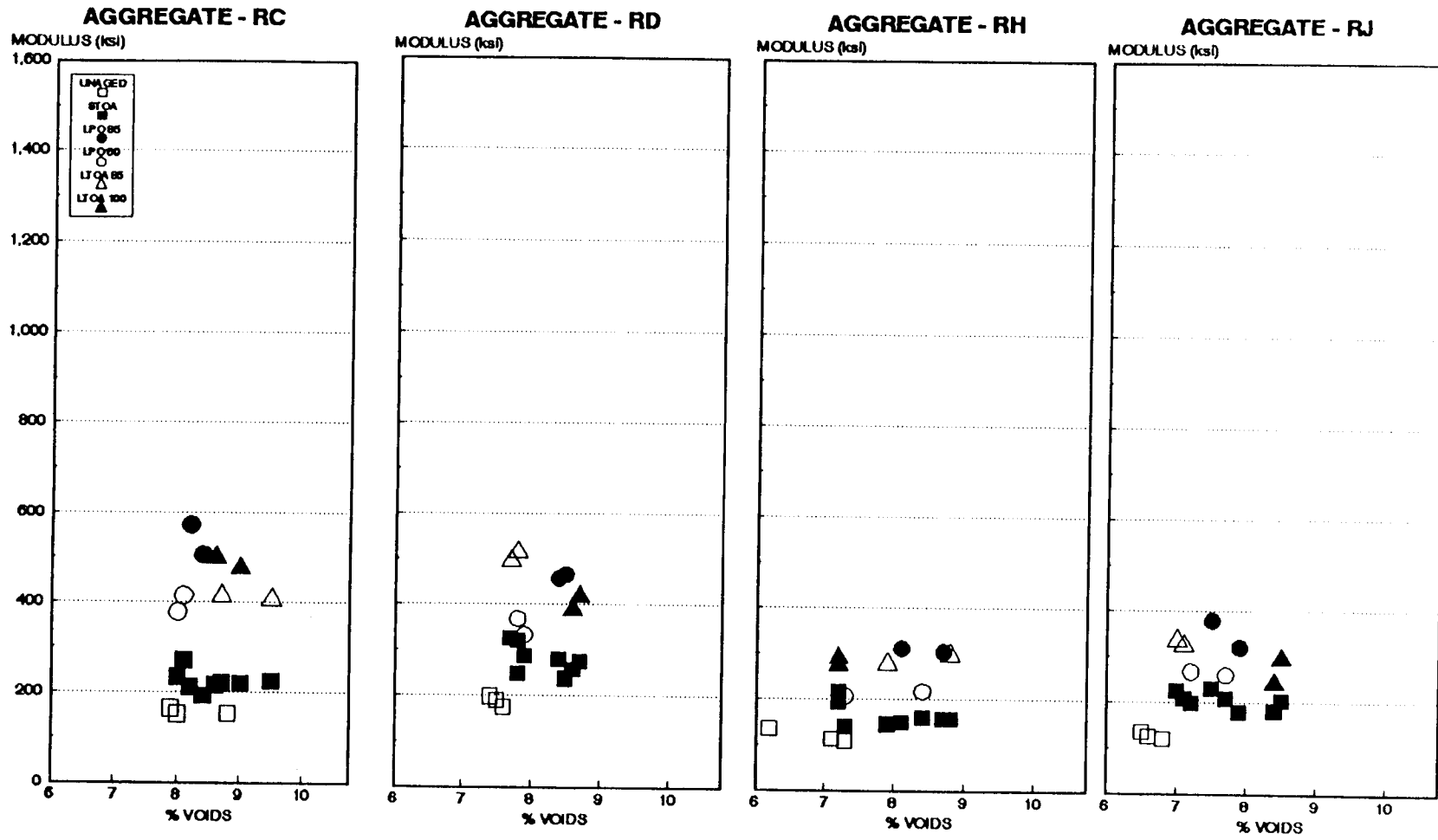
**11.1.11 Long-Term Aging Duration** - in min to the nearest 1 min

## **C.12 Keywords**

**12.1** Asphalt concrete, bituminous mixes, bituminous paving mixes, aging, long-term aging, asphalt concrete aging.

## **Appendix D**

### **Detailed Resilient Modulus Results**



**Figure D.1 Diametral Modulus: Long-Term Oven and Low-Pressure Oxidation Aging with Short-Term Oven Aging--Asphalt AAA-1.**

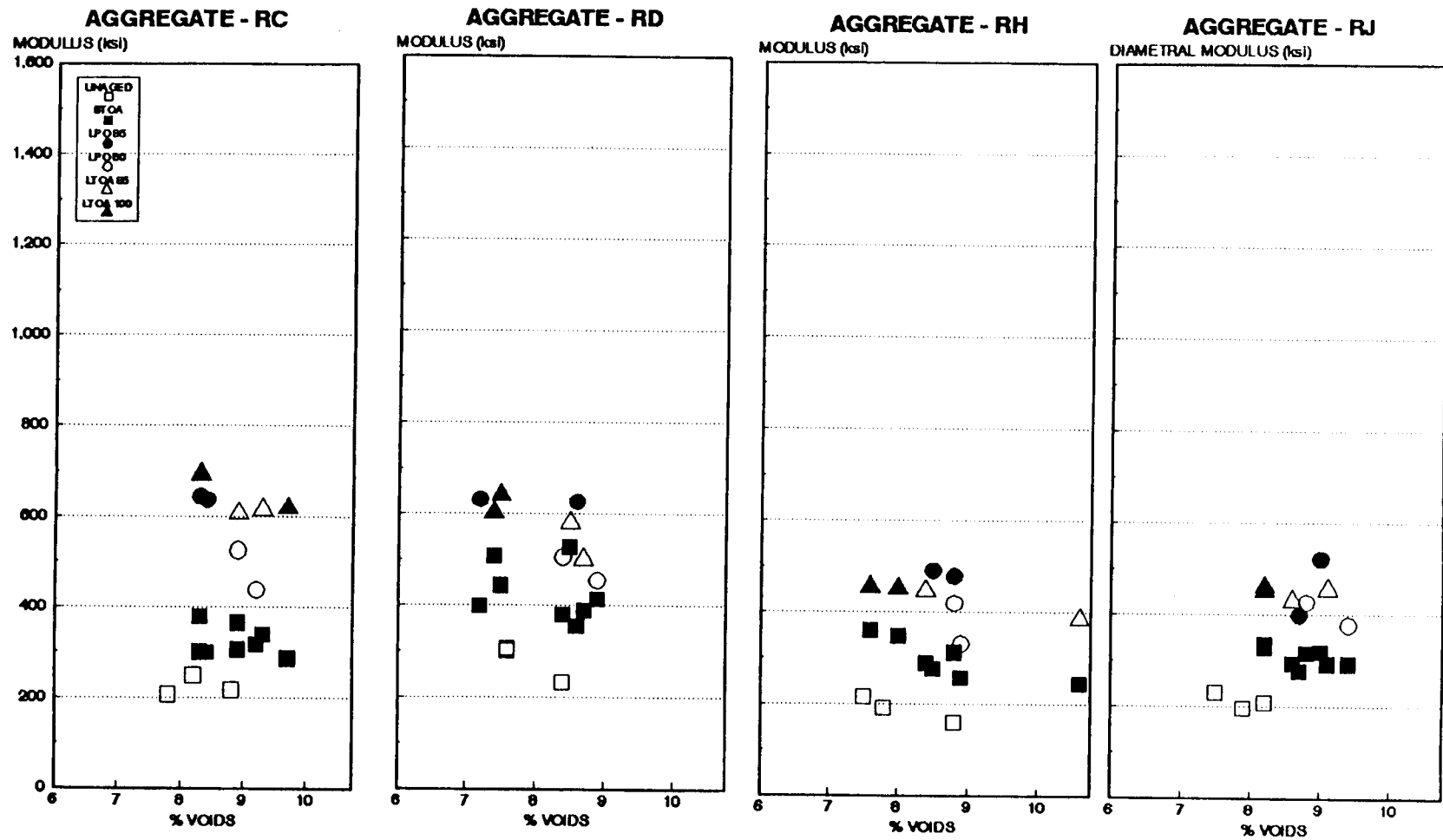


Figure D.2 Diametral Modulus: Long-Term Oven and Low-Pressure Oxidation Aging with Short-Term Oven Aging--Asphalt AAB-1.

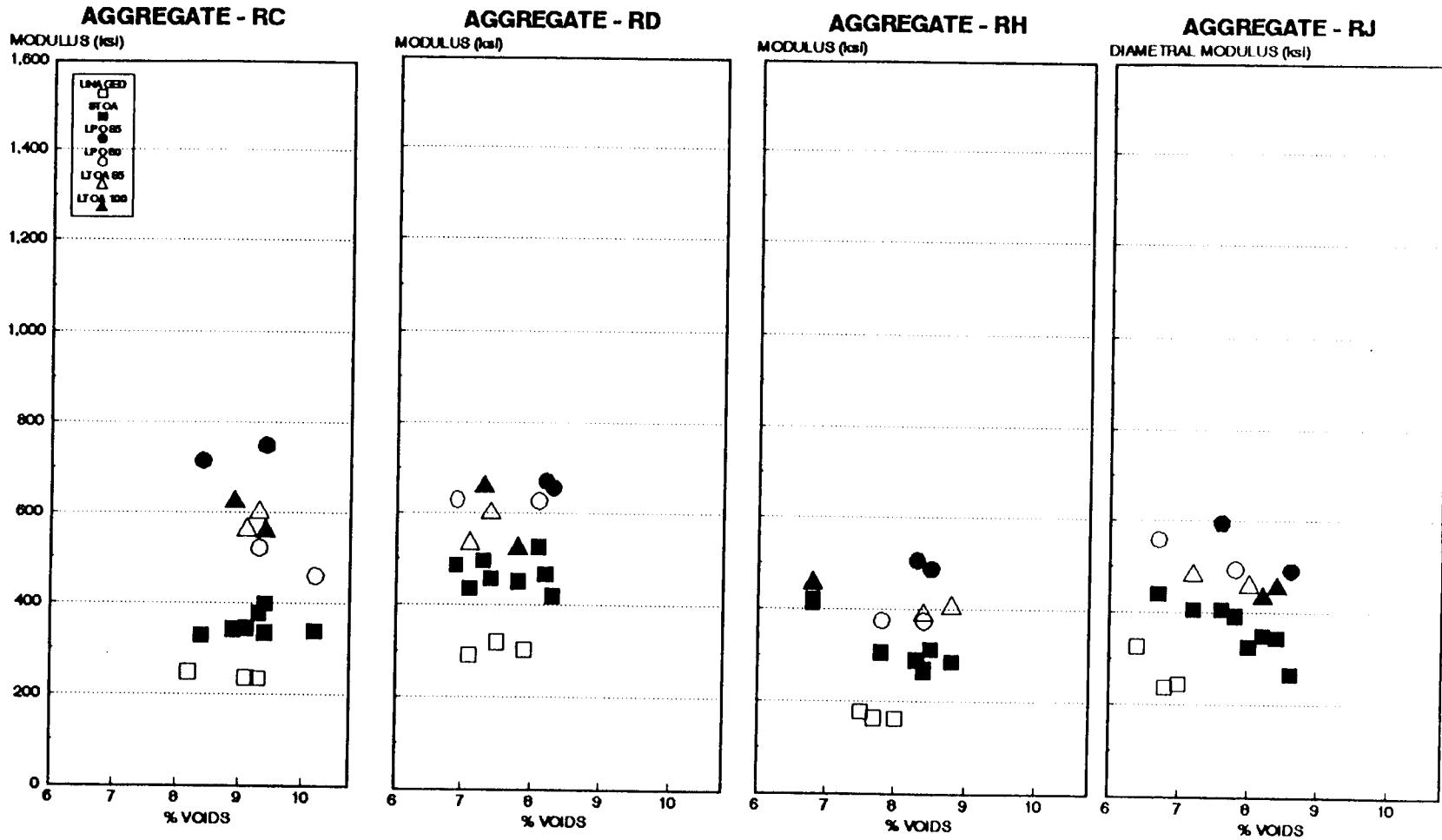


Figure D.3 Diametral Modulus: Long-Term Oven and Low-Pressure Oxidation Aging with Short-Term Oven Aging--Asphalt AAC-1.

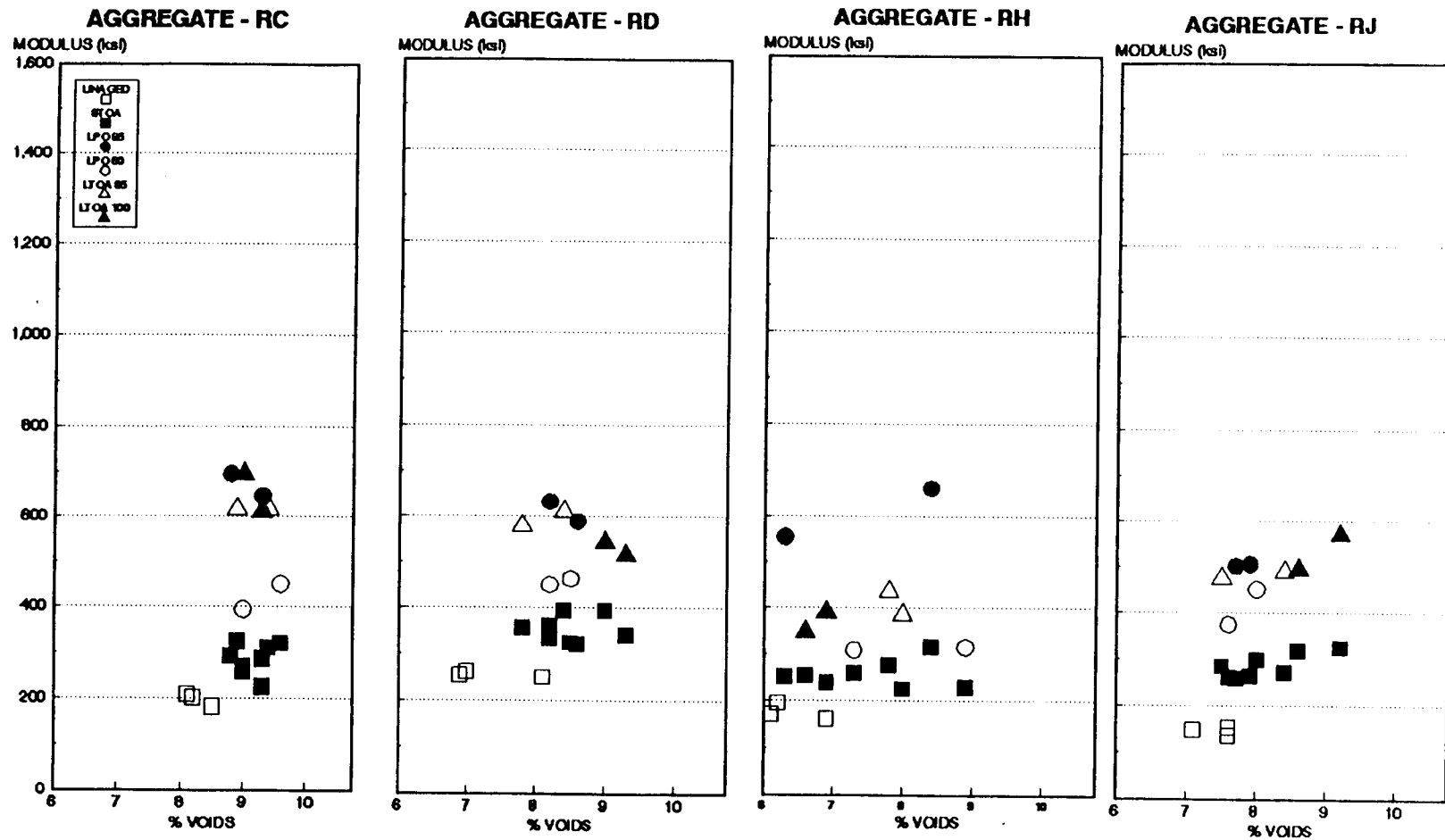
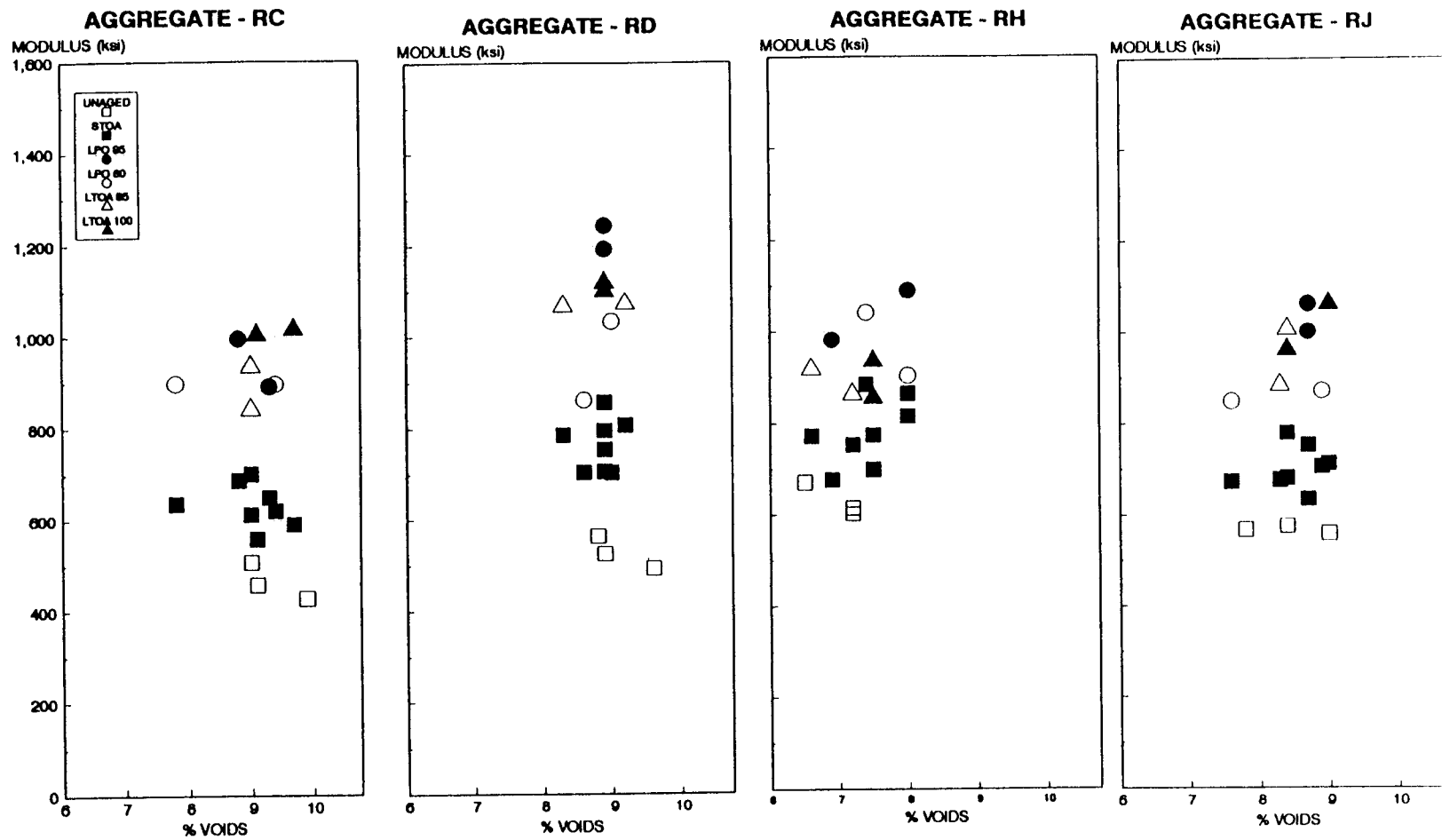


Figure D.4 Diametral Modulus: Long-Term Oven and Low-Pressure Oxidation Aging with Short-Term Oven Aging--Asphalt AAD-1.



**Figure D.5 Diametral Modulus: Long-Term Oven and Low-Pressure Oxidation Aging with Short-Term Oven Aging--Asphalt AAF-1.**

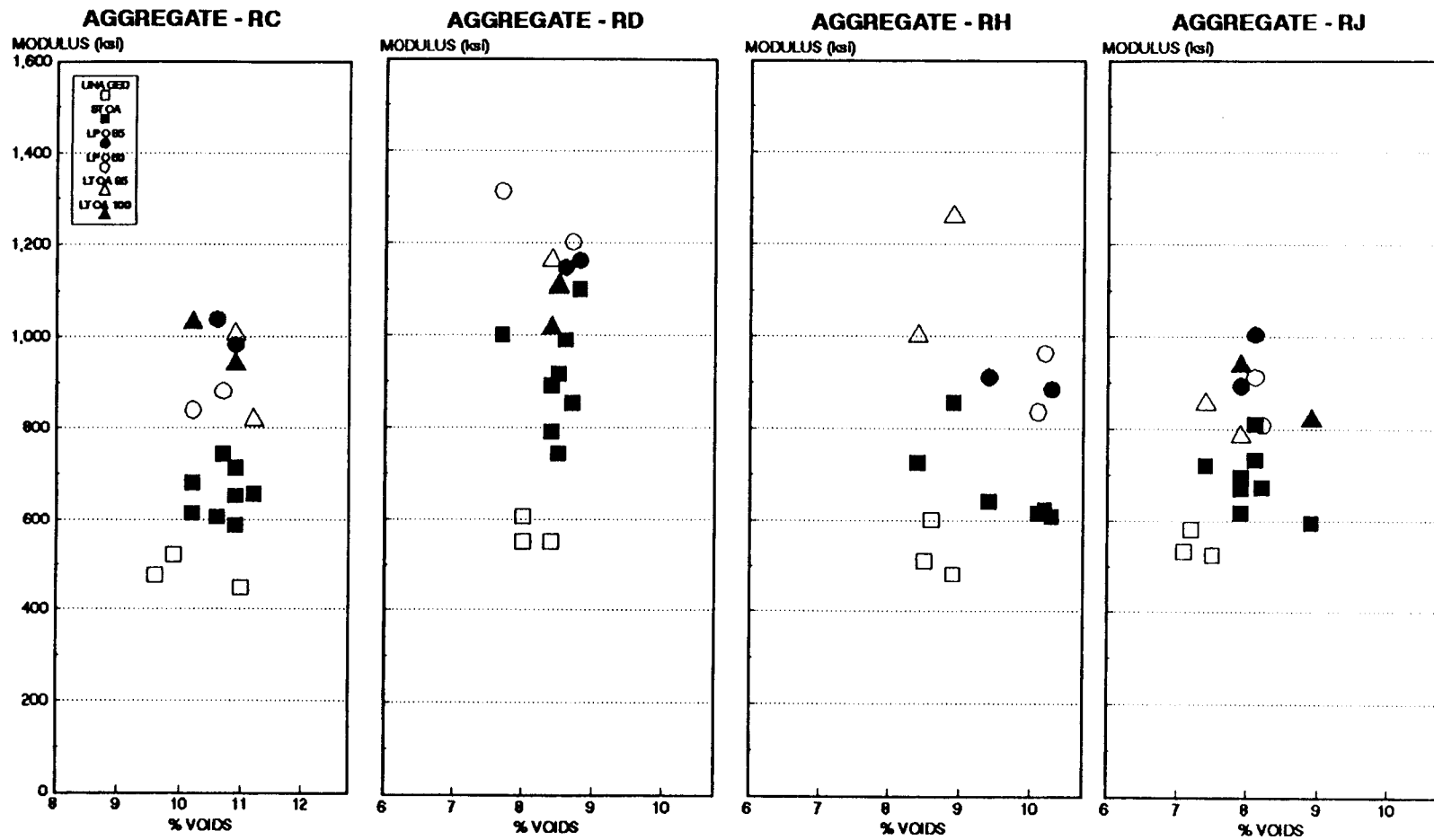


Figure D.6 Diametral Modulus: Long-Term Oven and Low-Pressure Oxidation Aging with Short-Term Oven Aging--Asphalt AAG-1.

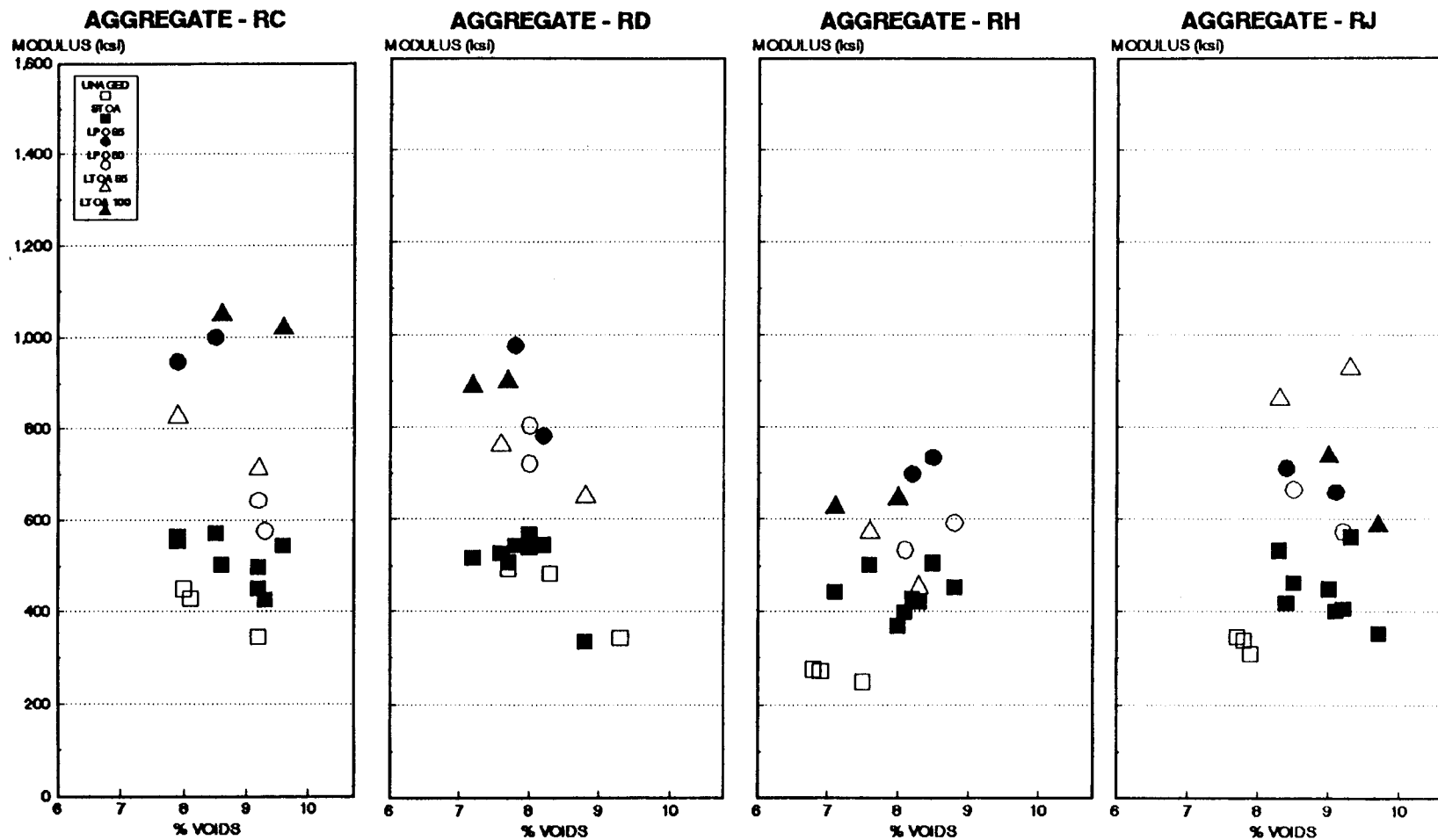


Figure D.7 Diametral Modulus: Long-Term Oven and Low-Pressure Oxidation Aging with Short-Term Oven Aging--Asphalt AAK-1.

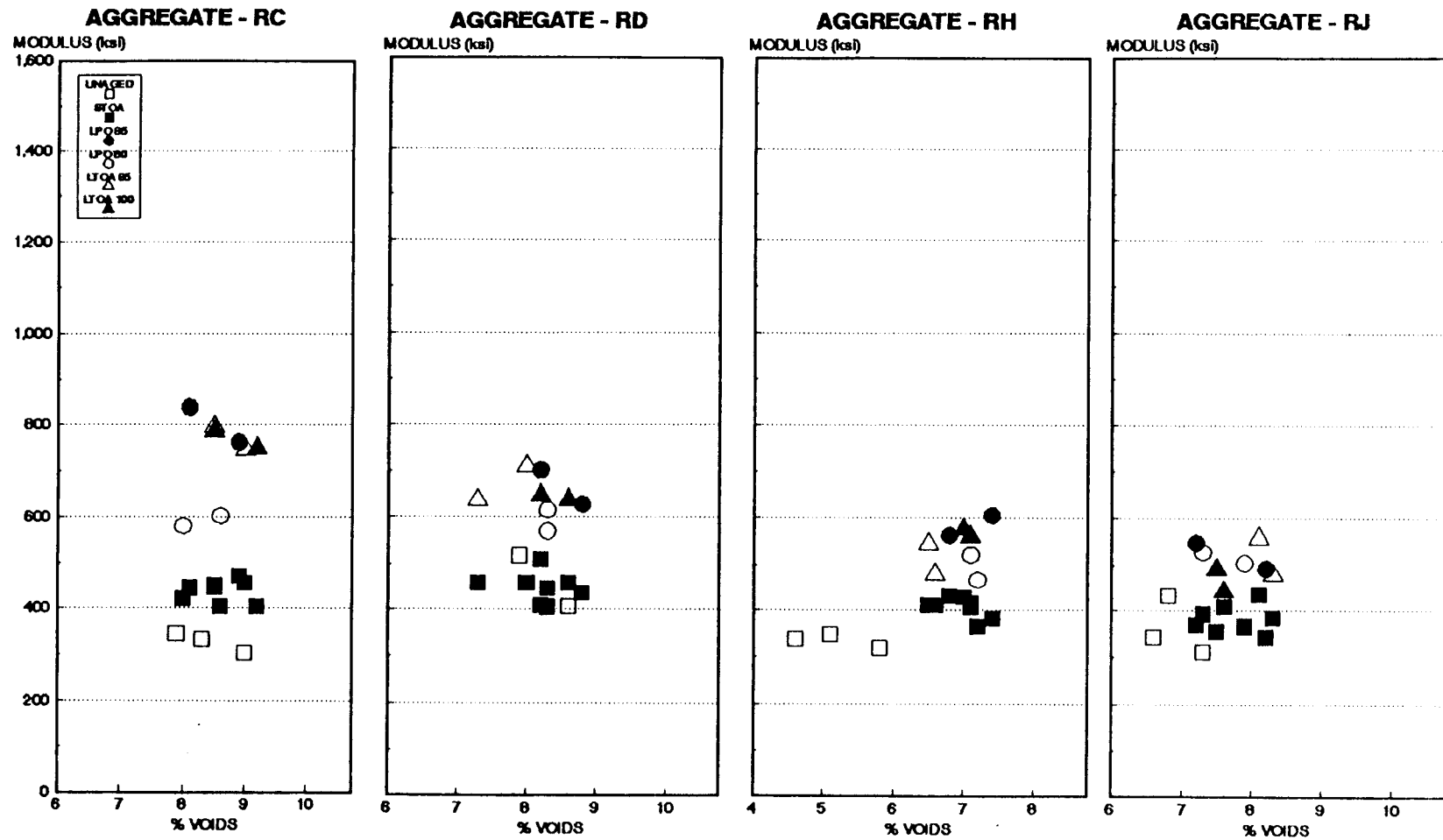


Figure D.8 Diametral Modulus: Long-Term Oven and Low-Pressure Oxidation Aging with Short-Term Oven Aging--Asphalt AAM-1.

## Appendix E

### Tensile Strength Test Results

#### Test Procedure

The indirect tensile strength test is a test to determine the maximum strength of an asphalt concrete specimen. During the test, the specimen is loaded in the vertical diametral plane as for the diametral resilient modulus test. The specimen is loaded to failure at a set deformation rate of 50 mm (2 in.) per minute. The measurements recorded during the test are load and deformation in the horizontal direction. The ultimate load and the horizontal deformation or strain level at the maximum load are reported. From the ultimate load the tensile strength can be calculated from:

$$\text{Tensile Strength} = \frac{2 P_{ult}}{\pi t D}$$

where:

$P_{ult}$  = ultimate load  
 $t$  = Specimen thickness, and  
 $D$  = Specimen diameter

## Materials

A mini study was completed using materials from the SHRP A-002A validation program. A selection of specimens from the materials used to conduct a dynamic mechanical analysis (Appendix H) study were used for the tensile strength analysis.

Specimens were selected from a group of six asphalt-aggregate combinations. Aggregate RC and RH along with asphalts AAD-1, AAF-1 and AAM-1.

One specimen from each of the following aging levels was chosen to be tested: unaged, low pressure oxidation at 60° and 85°C (140° and 185°F), and long-term oven aging at 85° and 100°C (185° and 212°F). This resulted in 5 specimens from each asphalt aggregate group for a total of 30 specimens.

## Results

Table G.1 shows a summary of the data collected for each specimen. Tensile strength, strain at yield, and diametral resilient modulus are the parameters that will be evaluated here.

As shown in Tables 2-4, the diametral resilient modulus ranks the asphalts, in order of most severe aging, AAD-1 > AAM-1 > AAF-1 in all cases. The rankings of the asphalts according to the tensile strength are AAD-1 > AAF-1 > AAM-1 in all but one case. These rankings are different than the rankings based on modulus. The rankings based on the strain at yield have no distinct pattern of rankings.

## Discussion

It should be noted that this study was conducted with only one specimen at each conditioning level. It would be more useful to have more specimens tested at each level so that a more thorough statistical analysis could be run. Table 1 shows that the values for tensile strength for asphalts AAF-1 and AAM-1 appear to be rather close in all cases. With a more thorough statistical analysis, these differences may be shown to be not significantly different; the analysis would show that only asphalt AAD-1 is different from both AAF-1 and AAM-1.

Asphalts AAF-1 and AAM-1 are both classified as AC-20 asphalts. The data collected from the tensile strength test show that these two asphalts are very similar in tensile strength ratios. This would tend to show that the tensile strength test is not as sensitive as the resilient modulus test to the aging condition or, maybe to the asphalt-aggregate interaction.

## Conclusions

1. The tensile strength test can use aging ratios to distinguish between different asphalts mixed with the same aggregate.
2. The tensile strength test may not be sensitive enough to rank asphalts according to aging susceptibility.
3. More specimens should be tested so that a more thorough statistical analysis may be performed.

**Table E.1. Tensile strength test results**

Aging	Sample ID	Thickness (in)	Air Voids (%)	Tensile Strength (psi)	Final Modulus (ksi)	Strain at Yield ( $\mu$ -strain)
LPO 85	1DCMSL	4.608	9.3	196	645	2085
LPO 60	3DCMSL	4.615	9.6	135.5	450	2073
LTOA 85	5DCMSL	4.587	8.9	181	615	1926
LTOA 100	7DCMSL	4.626	9.3	156.5	611	2147
BASE	11DCMB	4.573	8.5	87.9	182	3067
LPO 85	1FCMSL	4.593	9.3	259.4	891	1141
LPO 60	3FCMSL	4.509	7.8	264	898	1423
LTOA 85	5FCMSL	4.585	9	233.4	943	2208
LTOA 100	7FCMSL	4.576	9.1	220.1	1004	810
BASE	11FCMB	4.591	9.1	185.5	458	1914
LPO 85	1MCMSL	4.588	8.9	163.6	763	1298
LPO 60	3MCMSL	4.55	8	157.8	580	1055
LTOA 85	5MCMSL	4.583	8.5	156.4	796	1704
LTOA 100	7MCMSL	4.626	9.2	140.9	750	1177
BASE	11MCMB	4.568	7.9	120.4	346	2624

Aging	Sample ID	Thickness (in)	Air Voids (%)	Tensile Strength (psi)	Final Modulus (ksi)	Strain at Yield ( $\mu$ -strain)
LPO 85	1DHMSL	4.091	6.3	185.1	553	2564
LPO 60	3DHMSL	4.294	8.9	127.1	316	3853
LTOA 85	5DHMSL	4.277	8	138.5	385	4638
LTOA 100	7DHMSL	4.209	6.6	131.1	348	2539
BASE	11DHMB	4.198	5.6	100.8	174	5521
LPO 85	1FHMSL	4.217	6.9	288.8	982	1937
LPO 60	3FHMSL	4.214	7.4	264.9	1014	1802
LTOA 85	5FHMSL	4.18	6.6	259.7	918	1975
LTOA 100	7FHMSL	4.236	7.5	230	855	1377
BASE	11FHMB	4.188	6.5	215	673	3299
LPO 85	1MHMSL	4.207	6.8	169.8	563	1890
LPO 60	3MHMSL	4.222	7.1	149.5	521	2810
LTOA 85	5MHMSL	4.219	6.6	154.6	479	2932
LTOA 100	7MHMSL	4.252	7.1	146.5	560	2847
BASE	11MHMB	4.145	4.6	140.4	338	4675

## **Appendix F**

### **Dynamic Mechanical Analysis**

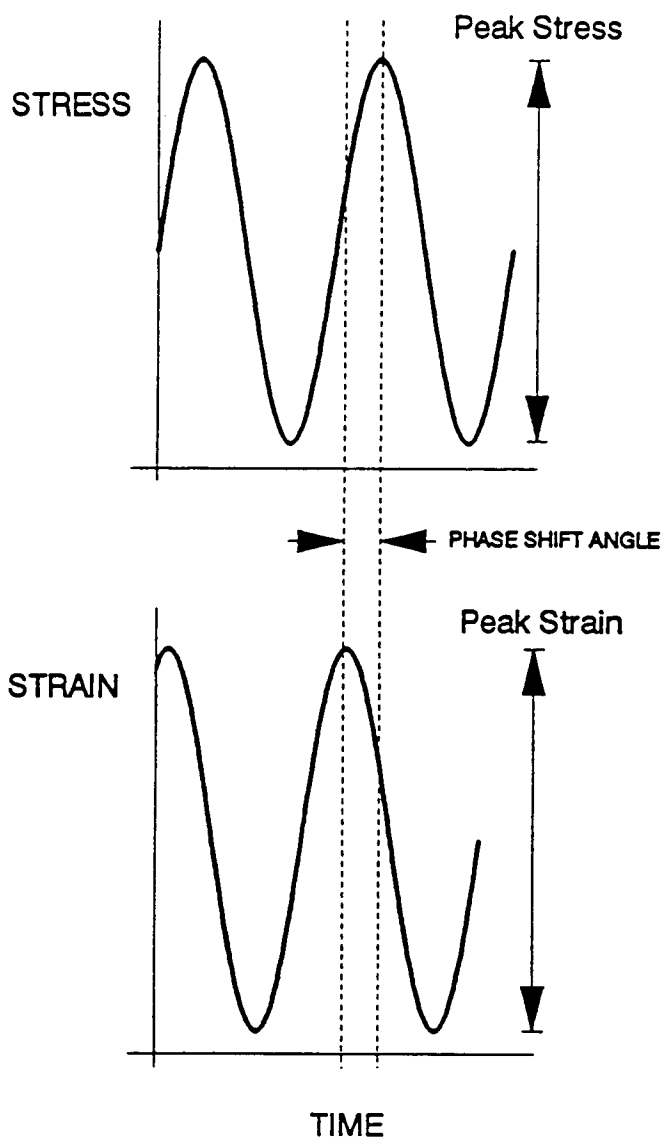
#### **Test Method**

Dynamic mechanical analysis (DMA) and other methods of rheological testing have been used to characterize the mechanical behavior of asphalt binders and asphalt-aggregate mixes (Christensen and Anderson 1992; Tayebali et al. 1990 and 1991; and Goodrich 1991). The concept of DMA has been described by Coffman et al. (1964) and by Sisko and Brunstrum (1968). DMA can characterize the linear viscoelastic behavior of asphalt binders and mixes by using a time-temperature superposition method. This is described by the time-dependent response (transformed or master curve) and the temperature-dependent response (shift factors curve) (Christensen and Anderson 1992). The responses measured by DMA in a triaxial mode of testing are complex modulus ( $E^*$ ), storage modulus ( $E'$ ), loss modulus ( $E''$ ), and loss tangent ( $\tan \delta$ ), as Figure F.1 shows. Papazian (1962) indicated that these dynamic moduli can provide insights into the time dependence of a material's response and can explain a material's behavior under varying loading rates and durations. For this reason, DMA was used to investigate the change in viscoelastic behavior of asphalt-aggregate mixes after accelerated laboratory aging.

#### **Test Procedures**

DMA was performed using a modified triaxial mode of testing. A repeated axial load, with no confining pressure, was applied to a specimen using a method similar to the standard test method for dynamic modulus of asphalt mixes (ASTM D3497-79). The repeated load was a sinusoidal wave form applied with a sequence of frequencies from 15 to 0.01 Hz at temperatures from 0 to 40°C (32° to 104°F). The frequency sweep was performed from the highest frequency to the lowest frequency, beginning with the coldest temperature and proceeding to the warmer temperatures. Load and vertical deformation were monitored during the test. Load was measured by a load cell at the bottom of the specimen. Vertical deformation was measured by two linear voltage differential transducers (LVDTs) attached to the side of the specimen with a set of yokes (Figure F.2).

The yokes were separated by four 2 in. spacers before they were glued with superglue to the specimen. The glue was allowed to set for 10 min at room temperature [25°C (77°F)] before the specimen was cooled to 0°C (32°F) in an environmental cabinet. A specimen with an imbedded thermocouple was also installed in the cabinet as a control specimen. When the control specimen reached 0°C (32°F), the other specimens in the environmental cabinet were ready for testing. A frequency sweep on a specimen takes about 25 min for each temperature.



### COMPLEX MODULUS

$$|E^*| = \frac{\text{Peak Stress}}{\text{Peak Strain}}$$

### STORAGE MODULUS

$$E' = |E^*| \cos \delta$$

### LOSS MODULUS

$$E'' = |E^*| \sin \delta$$

### LOSS TANGENT

$$\tan \delta = E''/E'$$

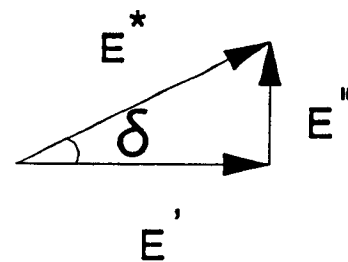


Figure F.1. Dynamic mechanical analysis (Goodrich 1991)

After the test at 0°C (32°F) was completed, the specimen was placed in an environmental cabinet set at 25°C (77°F). The control specimen was again used to monitor the temperature of the specimens in the environmental cabinet. As before, once the control specimen reached the next test temperature, the other specimens were ready for testing.

During the test program, the load cell and LVDTs were calibrated at various temperatures. It was found that the calibration factors were constant within the range of testing temperatures.

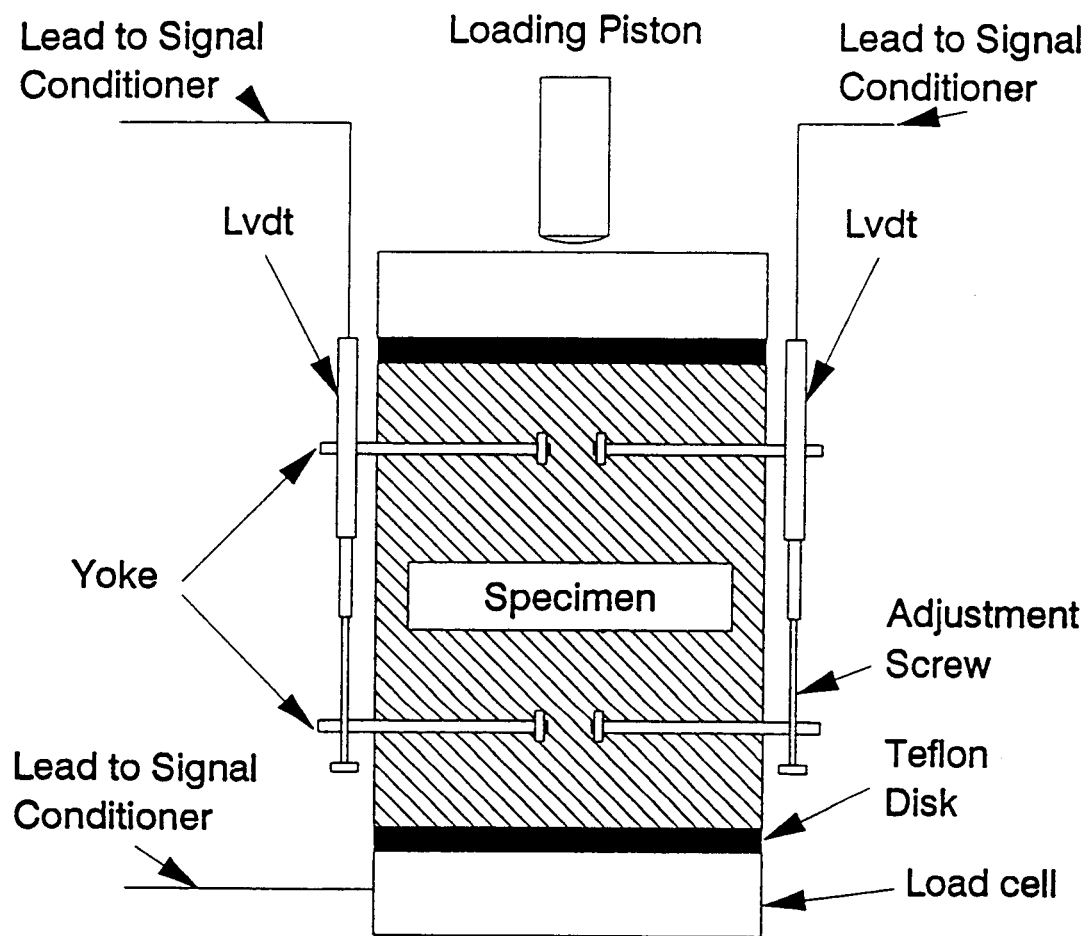
This test was non-destructive with the total recoverable deformation limited to 200  $\mu$ -inches at both the lowest frequency (0.01 Hz) and highest test temperature [40°C (104°F)]. The test was performed by adjusting the load to produce a recoverable strain of 25  $\mu$ -strain at 1 Hz. The stress required to induce the 25  $\mu$ -strain at 1 Hz was used as the applied stress throughout the frequency sweep test. This ensured that the strain level did not exceed 100  $\mu$ -strain at any other frequency or temperature. A procedure to control the strain at, for example, 100  $\mu$ -strain, would be preferable but more difficult to achieve.

The test is performed on an MTS servo-hydraulic semi-closed loop control system. The data acquisition and analysis is performed on a high-speed personal computer. The computer software controls the MTS machine during the frequency sweep and saves the data to a file. The data was then processed to generate the dynamic moduli and phase angles.

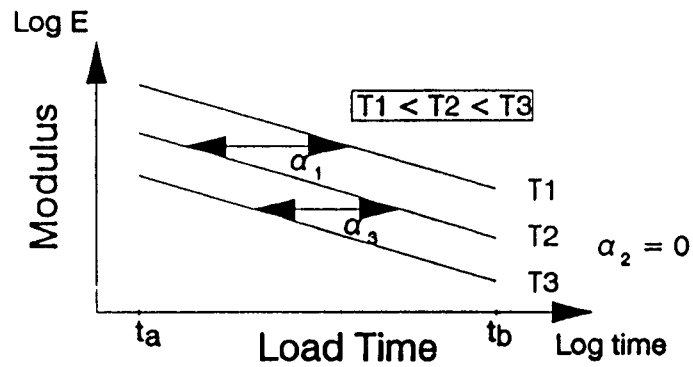
## Test analysis

The fundamental material responses obtained from DMA, characterizing the viscoelastic behavior of the materials as a function of frequency (loading time) and temperature, are the dynamic moduli: the complex modulus, the storage modulus, and the loss modulus. The loss tangent is calculated from the ratio of the storage modulus to the loss modulus. The dynamic moduli results are transformed to a standard temperature, in this case 25°C (77°F), by using the time-temperature superposition principle to create a master curve. The general process of transforming data to develop the master curve is illustrated in Figure F.3 (Stephanos 1990).

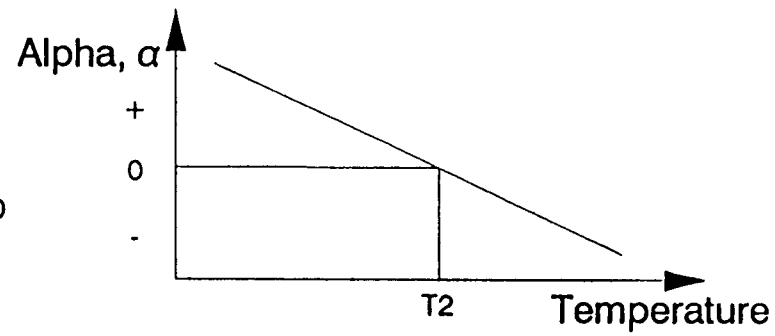
Figure F.4 shows a master curve constructed from DMA performed on a short-term aged specimen. Data collected at 0°C (32°F) has been shifted to the right into higher frequency ranges while data at 40°C (104°F) has been shifted to the left into lower frequency ranges. The points thus line up to make a smooth sigmoidal curve that includes frequencies outside the original test range. Figure F.5 shows a plot of phase shift versus temperature for the data illustrated in Figure



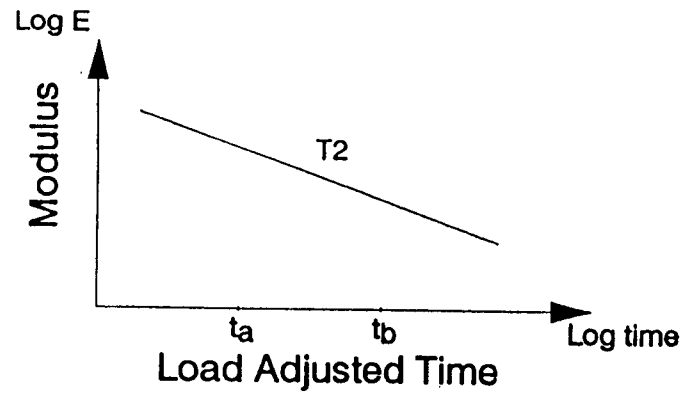
**Figure F.2. Unconfined triaxial modulus test**



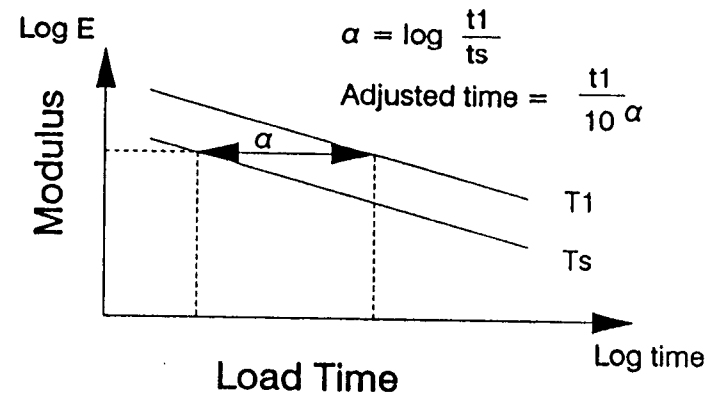
a. Modulus versus time in log-log space



b. Alpha versus temperature



c. Master curve in log-log space



d. Calculation of adjusted time

Figure F.3. Procedure for transforming modulus data to the master stiffness curve (Stephanos 1990).

F.4. Phase shift curves are unique for each specimen because they describe the amount of shift for each specimen at each test temperature. They are used to produce transformed plots for each DMA parameter, e.g., complex modulus or phase angle, for any temperature within the range tested. In this study, transformations to 25°C (77°F) were used.

## **Experiment Design**

The DMA procedure was run on a subset of the materials used in the validation of the A-002A hypothesis. Six of the material combinations had all unaged, short-term aged, and long-term aged specimens subjected to the DMA procedure. Another 18 material combinations subjected only the unaged specimens and the 100°C (212°F) for 2 days long-term aged specimens to the DMA procedure.

The specimens were tested before and after the long-term aging procedure for all of the tested combinations. This data, along with the data from the unaged specimens, will allow aging ratios to be determined based on the different DMA parameters. These ratios can then be used to rank the asphalts in order as to their aging susceptibility. These rankings can then be compared with the rankings based on the diametral resilient modulus and with the rankings from the A-002A testing.

## **Materials**

The six combinations for which all the specimens were tested were combinations of aggregates RC and RH mixed with asphalts AAD-1, AAF-1, and AAM-1. The other combinations that were only partially tested used aggregates RC, RD, RH, and RJ in combination with asphalts AAA-1, AAD-1, AAF-1, AAG-1, AAK-1, and AAM-1.

All of the specimens in this study were compacted with the California Kneeding Compactor to a target void level of 8 percent. Most of the specimens were within  $\pm 1$  percent of the target. The specimens were 4 in. in diameter by approximately 4 in. tall.

## **Results**

The data presented here are obtained from tests on aggregates RC and RH mixed with asphalts AAD-1, AAF-1, and AAM-1. Tests on other mix combinations have not yet been completed. The test specimens were compacted at a nominal target air voids of 8 percent. The percent air voids for each specimen are shown in Table F.1.

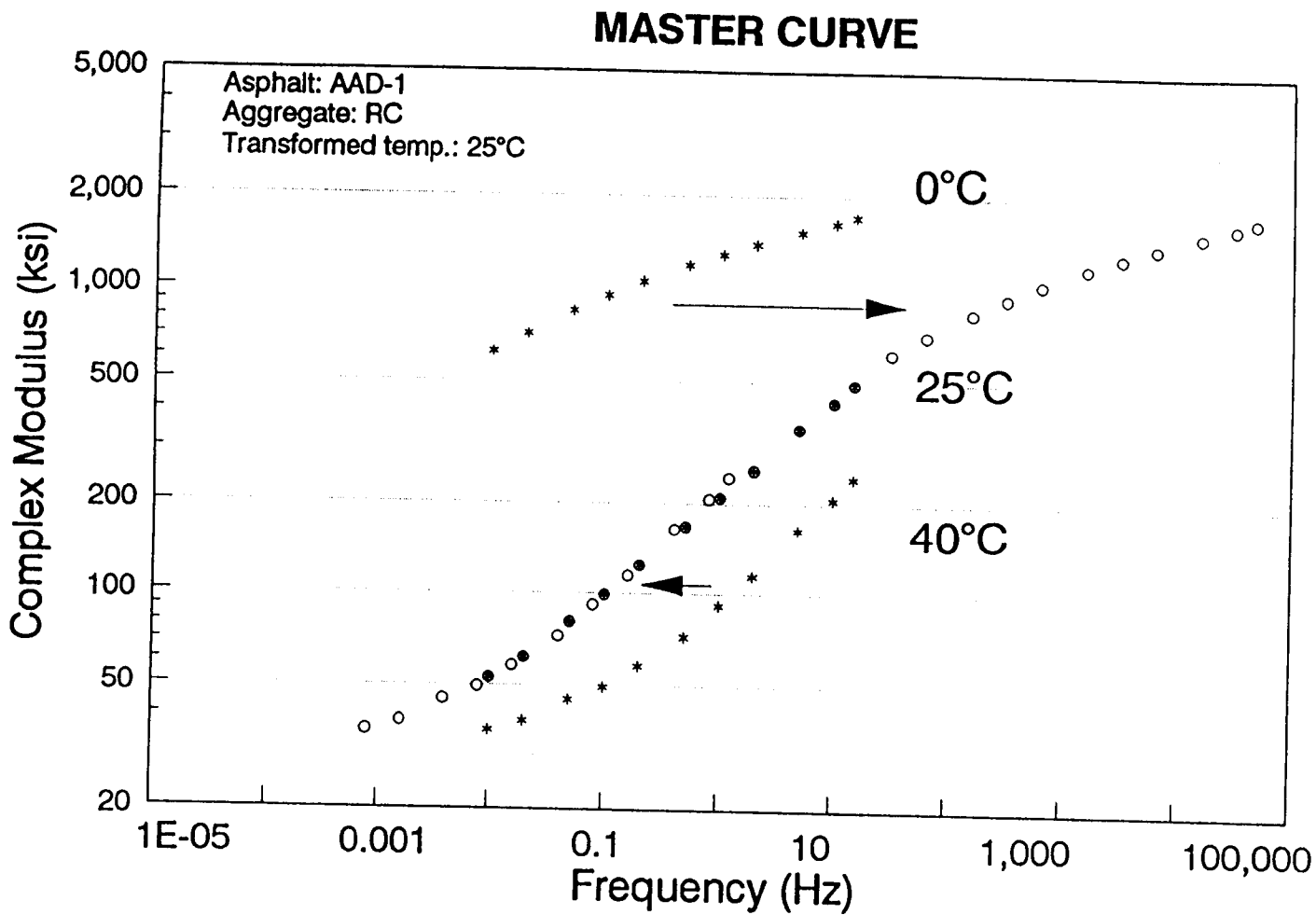


Figure F.4. Master curve for short-term oven aged specimen

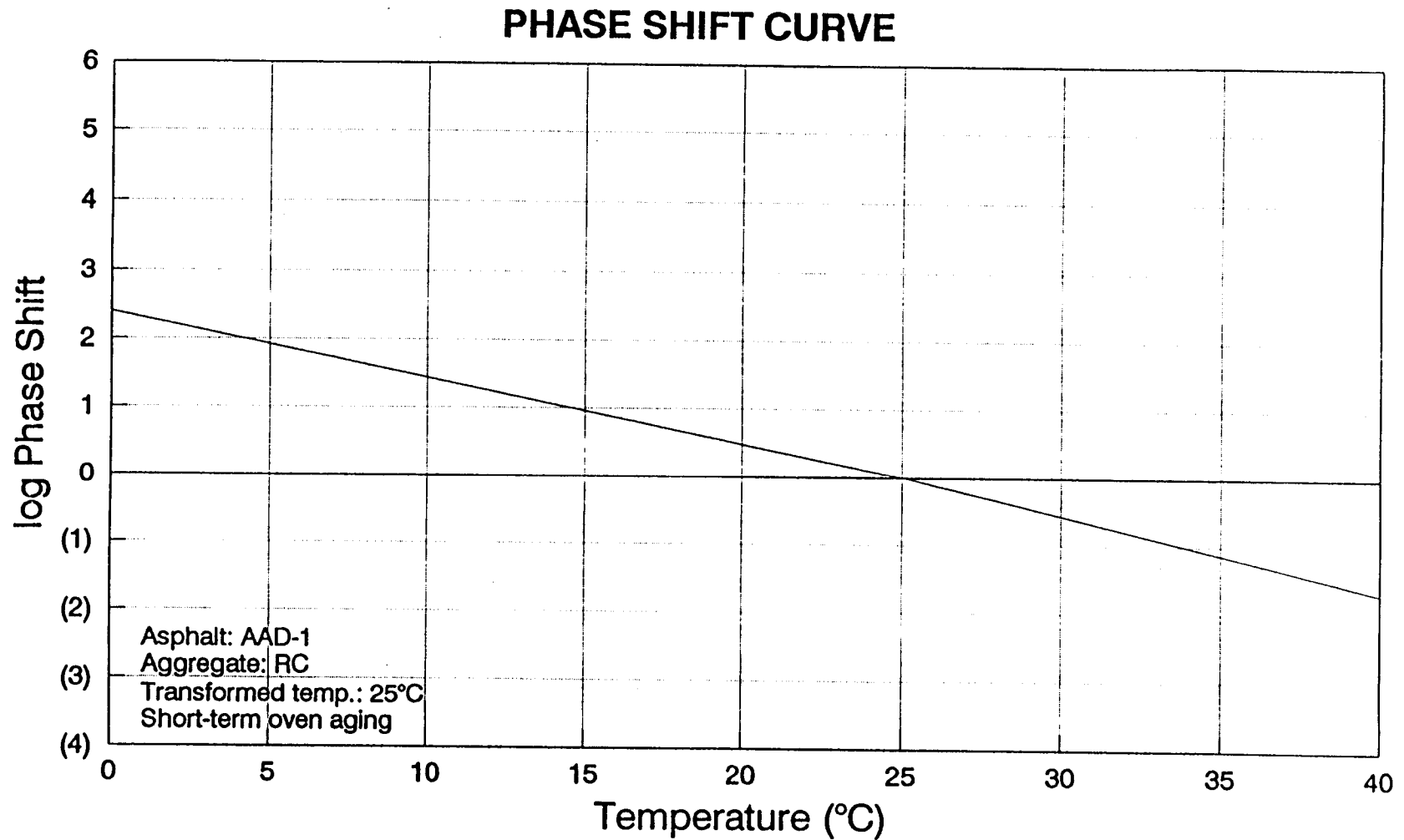


Figure F.5. Phase shift curve for short-term aged specimen

Figures F.6 through F.12 are complex modulus versus frequency master curves and phase angle curves for unaged, short-term oven aged, and long-term aged specimens. The plots for unaged specimens were obtained from DMA performed on three specimens. The plots for short-term oven aged specimens were obtained from tests performed on the same pair of specimens which were subsequently long-term aged and retested. Since four long-term aging methods were evaluated, a total of eight specimens were aged for each asphalt-aggregate combination. Only data for long-term aging at 85°C (185°F) is shown here for conciseness.

Table F.2 shows the diametral resilient modulus aging ratios for each aging treatment. The aging ratio is defined as the ratio of the diametral resilient modulus of the aged specimen to the unaged specimen. The aging ratios are plotted in Figure F.13 to show the diametral resilient modulus rankings of asphalt-aggregate combinations for each aging treatment. The complex moduli at frequencies of 0.001, 1, and 1000 Hz are shown in Table F.3. These values are obtained from the master curves transformed at 25°C (77°F). Table F.4 shows the DMA complex modulus ratios of aged specimens to unaged specimens for each aging treatment, collected at frequencies of 0.001, 1, and 1000 Hz. Figure F.14 shows the complex modulus rankings of the asphalt-aggregate combinations.

## Discussion

Typical data obtained by Christensen and Anderson (1992) from DMA results on asphalt binders are shown in Figure F.15. A master curve for a typical asphalt binder shows that the complex modulus approaches a limiting elastic value at high frequency at about 1 GPa (145 ksi). The modulus decreases monotonically as the frequency is reduced. The curve at very low frequencies usually slopes at a 1:1 ratio, which indicates that viscous flow has been reached.

Master curves of complex modulus versus frequency for asphalt mixes were constructed which showed similar trends to the master curves for asphalt binders at high frequencies. The master curves show that the complex modulus at high frequencies generally approaches a limiting value of about 5,000 ksi after long-term aging treatments. As the frequency is reduced, the complex modulus decreases from the highest frequency to the lowest frequency following the "S" curve. It appears as though each complex modulus curve could be modeled by a linear portion in the middle frequencies, showing viscous response, and by curved portions at high and low frequencies, where elastic behavior is approached. The tendency towards elastic behavior at low frequencies is very apparent for the unaged specimen and indicates that the aggregate is dominating the response. However, for the short-term aged specimens, the complex moduli are higher and the elastic response is less evident at low frequencies. The complex moduli are even higher for long-term aging curves,

Table F.1. Percent air voids for each asphalt-aggregate combination

Aggregate	Asphalt	Specimen #	Percent Void
RH	AAD-1	1	6.3
		2	8.4
		3	8.9
		4	7.3
		5	8.0
		6	7.8
		7	6.6
		8	6.9
		9	6.2
		10	6.9
		11	5.6
RH	AAF-1	1	6.9
		2	8.0
		3	7.4
		4	8.0
		5	6.6
		6	7.2
		7	7.5
		8	7.5
		9	7.2
		10	7.2
		11	6.5
RH	AAM-1	1	6.8
		2	7.4
		3	7.1
		4	7.2
		5	6.6
		6	6.5
		7	7.1
		8	7.0
		9	5.8
		10	5.1
		11	4.6
RC	AAD-1	1	9.3
		2	8.8
		3	9.6
		4	9.0
		5	8.9
		6	9.4
		7	9.3
		8	9.0
		9	8.2
		10	8.1
		11	8.5
RC	AAF-1	1	9.3
		2	8.8
		3	7.8
		4	9.4
		5	9.0
		6	9.0
		7	9.1
		8	9.7
		9	9.0
		10	9.9
		11	9.1
RC	AAM-1	1	8.9
		2	8.1
		3	8.0
		4	8.6
		5	8.5
		6	9.0
		7	9.2
		8	8.5
		9	8.3
		10	9.0
		11	7.9

**Table F.2. Resilient modulus ratio for short-term and long-term aging**

Aggregate	Asphalt	Aging Ratio			
		Short-Term Oven	Long-Term Oven @ 100°C	Long-Term Oven @ 85°C	Low Pressure Oxidation @ 85°C
RC	AAD-1	1.59	3.69	3.43	3.63
	AAF-1	1.34	2.01	1.90	2.18
	AAM-1	1.35	2.47	2.41	2.42
RH	AAD-1	1.72	4.03	2.84	2.21
	AAF-1	1.26	1.67	1.41	1.45
	AAM-1	1.36	1.97	1.67	1.91

**Table F.3. Complex modulus (ksi) data selected at frequencies, 0.001, 1, 1000 Hz**

Aggregate	Asphalt	Frequency	Complex Modulus (ksi)				
			Unaged	Short-term Oven @ 135°C	Long-Term Oven @ 100°C	Long-Term Oven @ 85°C	Low Pressure Oxidation @ 85°C
RC	AAD-1	0.001	42	50	105	155	195
		1	280	330	670	1020	1190
		1000	1400	1620	2180	2900	3300
	AAF-1	0.001	69	100	180	210	330
		1	710	890	1250	1450	1850
		1000	2320	2650	3050	3300	4000
	AAM-1	0.001	50	85	160	130	215
		1	470	610	960	800	1200
		1000	1450	1950	2350	2100	2700
RH	AAD-1	0.001	35	42	65	48	55
		1	190	280	520	340	430
		1000	1680	2000	2420	2050	2150
	AAF-1	0.001	60	70	120	140	170
		1	740	890	1000	1100	1300
		1000	2750	3000	3100	3200	3400
	AAM-1	0.001	50	68	70	79	125
		1	495	550	650	770	1080
		1000	1950	2200	2350	2500	3050

Table F.4. Complex modulus ratio selected at frequencies, 0.001, 1, 1000 Hz

Aggregate	Asphalt	Frequency	Complex Modulus Ratio*				
			Unaged	Short-term Oven @ 135°C	Long-Term Oven @ 100°C	Long-Term Oven @ 85°C	Low Pressure Oxidation @ 85°C
RC	AAD-1	0.001	1.0	1.2	2.5	3.7	4.6
		1	1.0	1.2	2.4	3.6	4.3
		1000	1.0	1.2	1.6	2.1	2.4
	AAF-1	0.001	1.0	1.4	2.6	3.0	4.8
		1	1.0	1.3	1.8	2.0	2.6
		1000	1.0	1.1	1.3	1.4	1.7
	AAM-1	0.001	1.0	1.4	1.4	1.6	2.5
		1	1.0	1.1	1.3	1.6	2.2
		1000	1.0	1.1	1.2	1.3	1.6
RH	AAD-1	0.001	1.0	1.2	1.9	1.4	1.6
		1	1.0	1.5	2.7	1.8	2.3
		1000	1.0	1.2	1.4	1.2	1.3
	AAF-1	0.001	1.0	1.2	2.0	2.3	2.8
		1	1.0	1.2	1.4	1.5	1.8
		1000	1.0	1.1	1.1	1.2	1.2
	AAM-1	0.001	1.0	1.7	3.2	2.6	4.3
		1	1.0	1.3	2.0	1.7	2.6
		1000	1.0	1.3	1.6	1.4	1.9

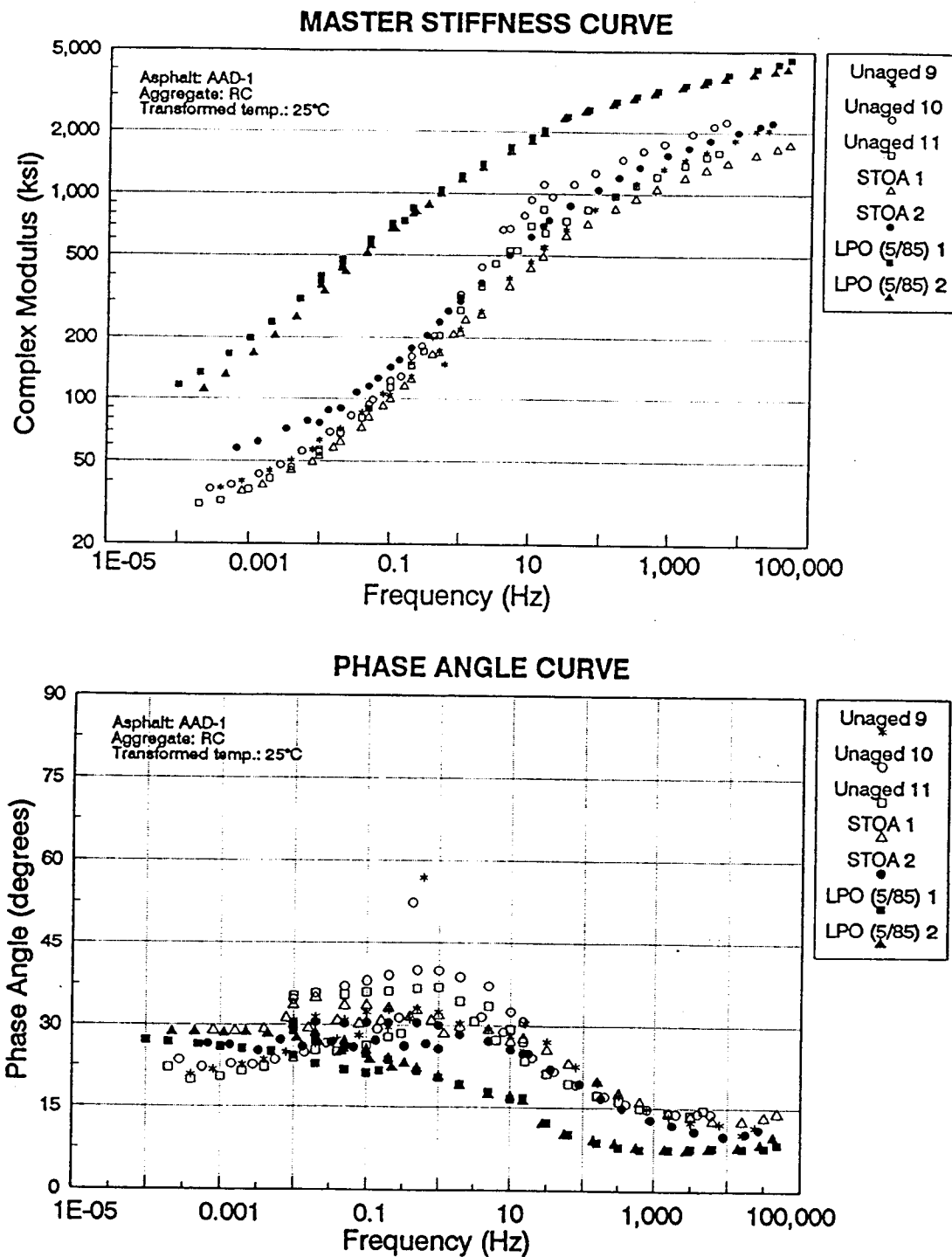


Figure F.6. Experimental data and regression data for master curve and phase angle curve

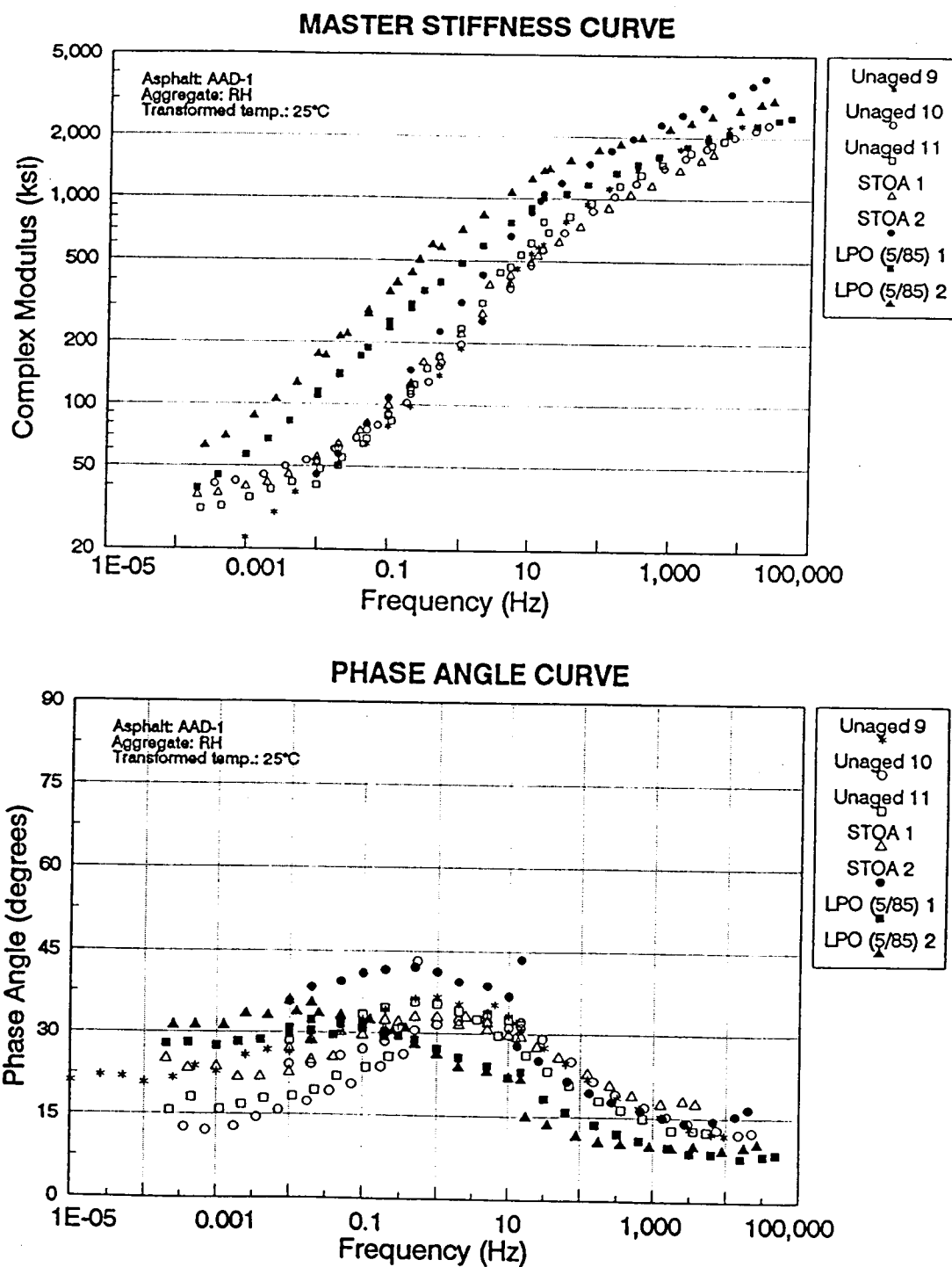


Figure F.7. Master curve and phase angle curve for asphalt AAD-1 and aggregate RC

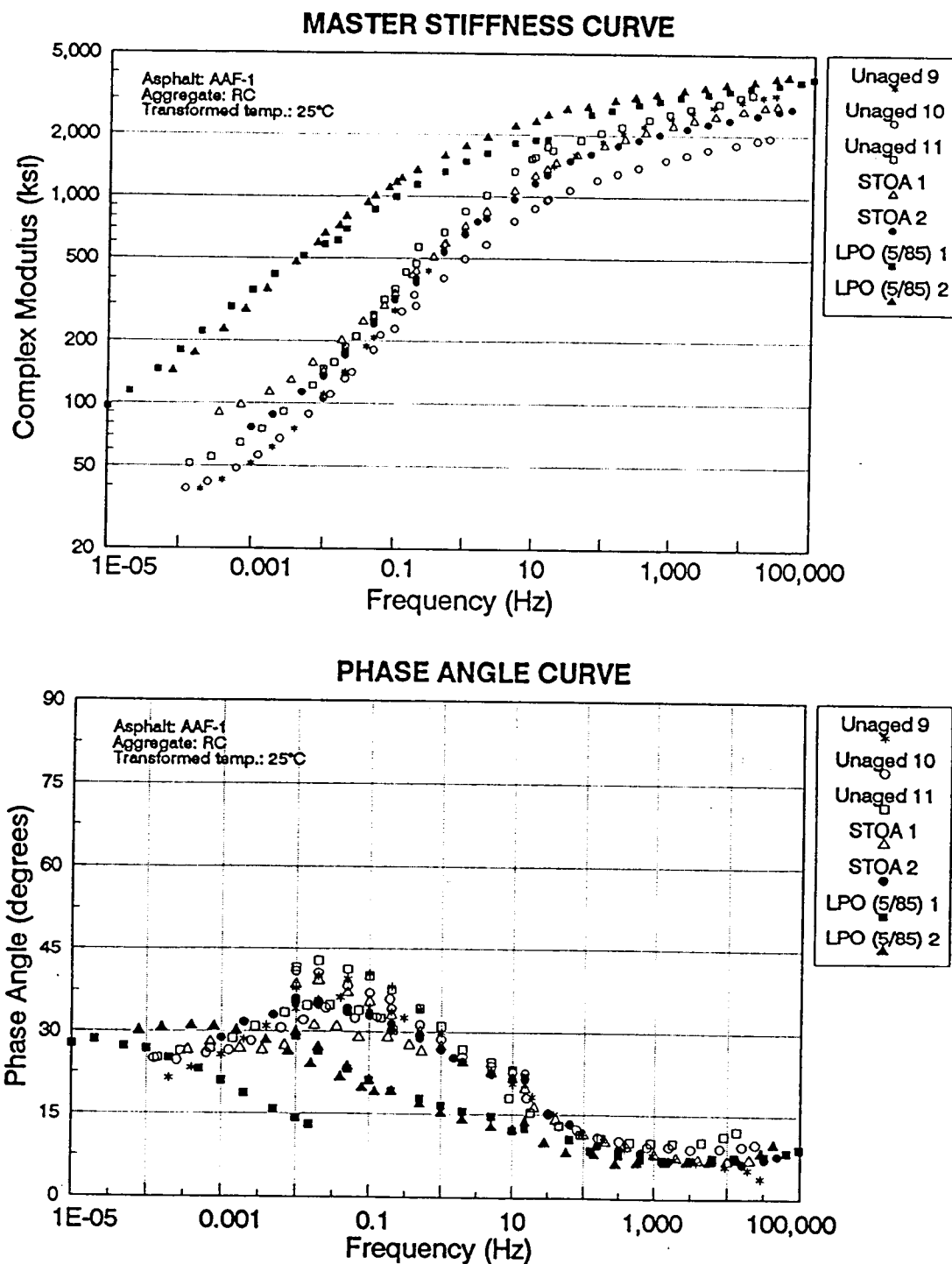


Figure F.8. Master curve and phase angle curve for asphalt AAD-1 and aggregate RH

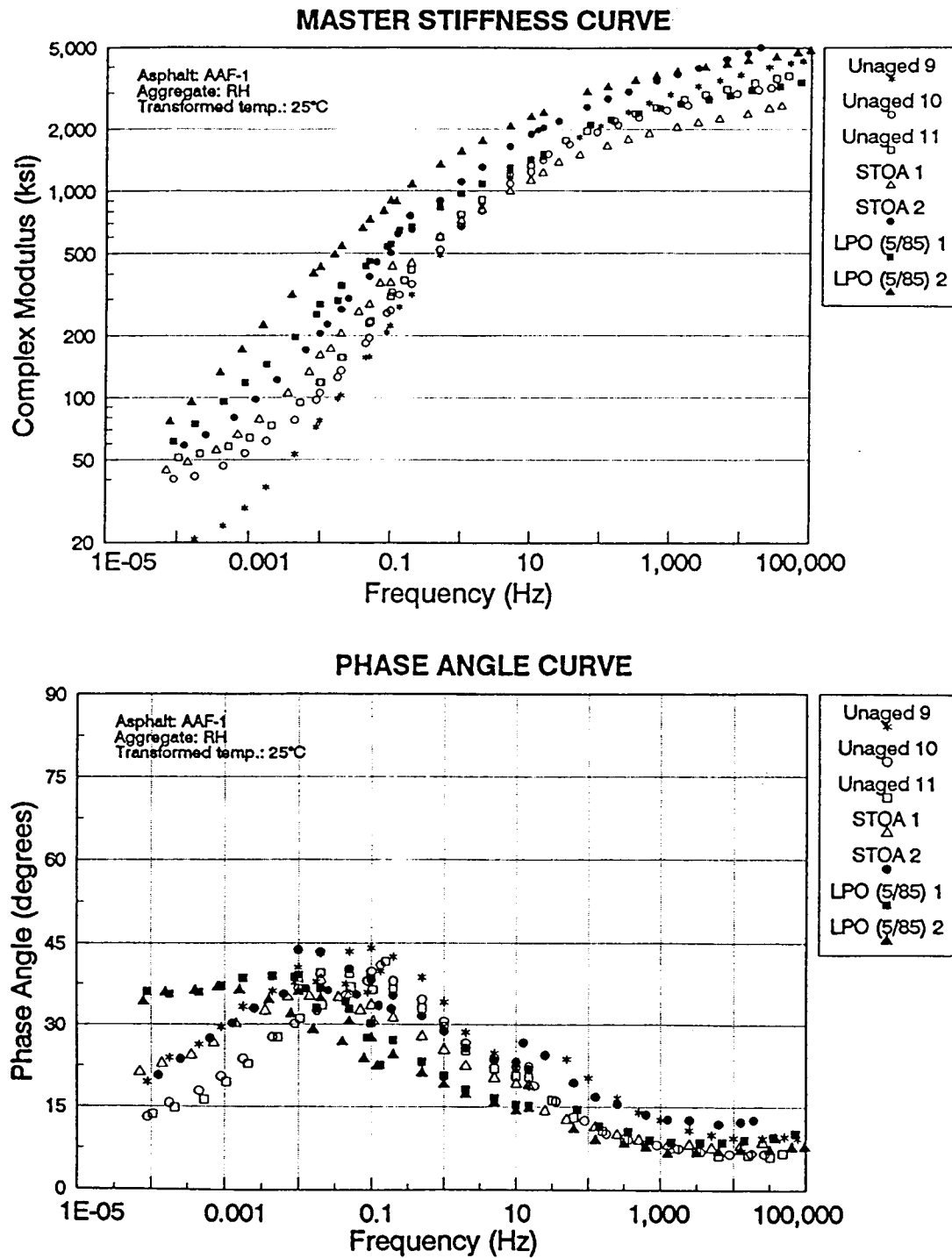


Figure F.9. Master curve and phase angle curve for asphalt AAF-1 and aggregate RC

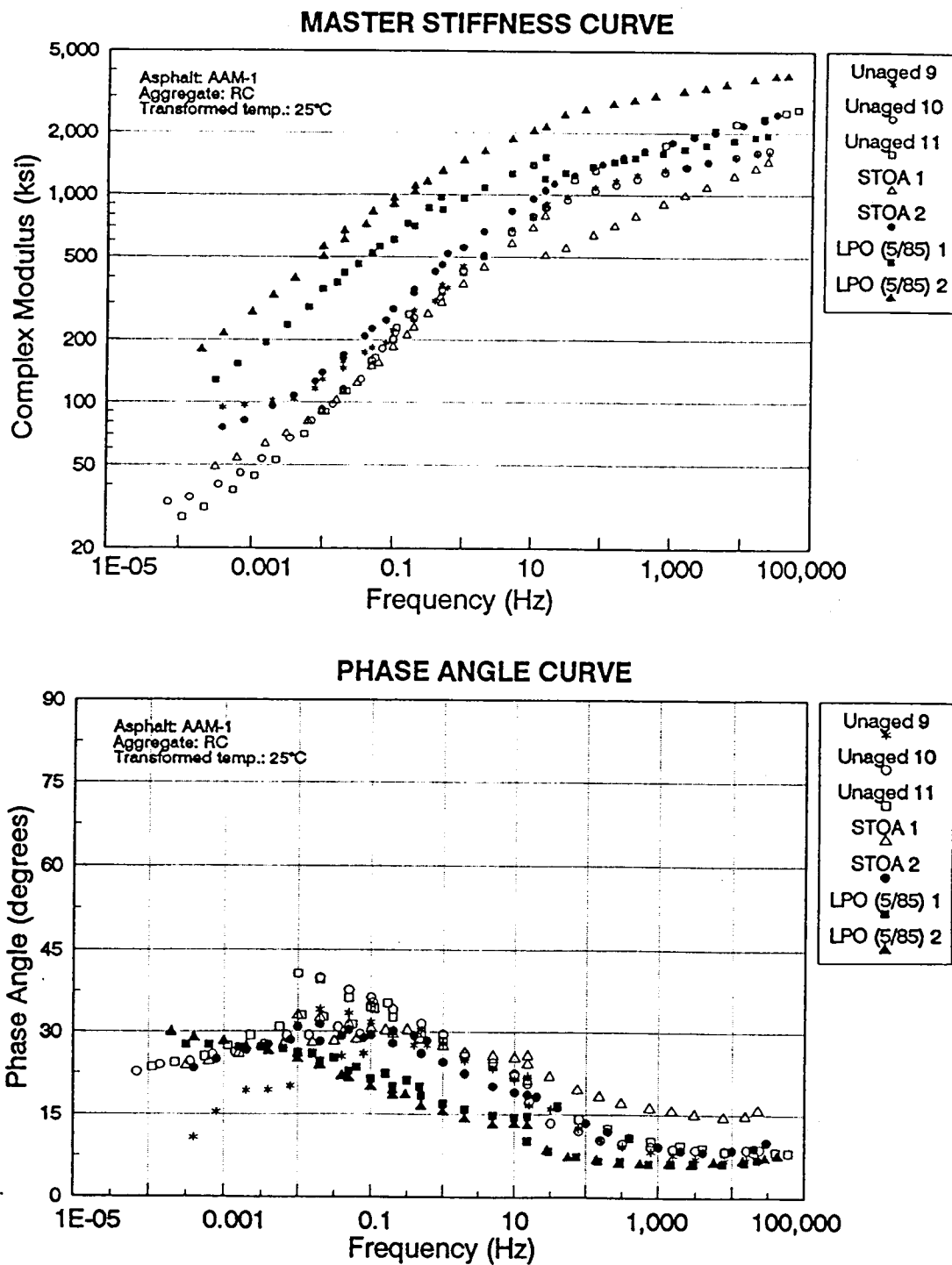


Figure F.10. Master curve and phase angle curve for asphalt AAF-1 and aggregate RH

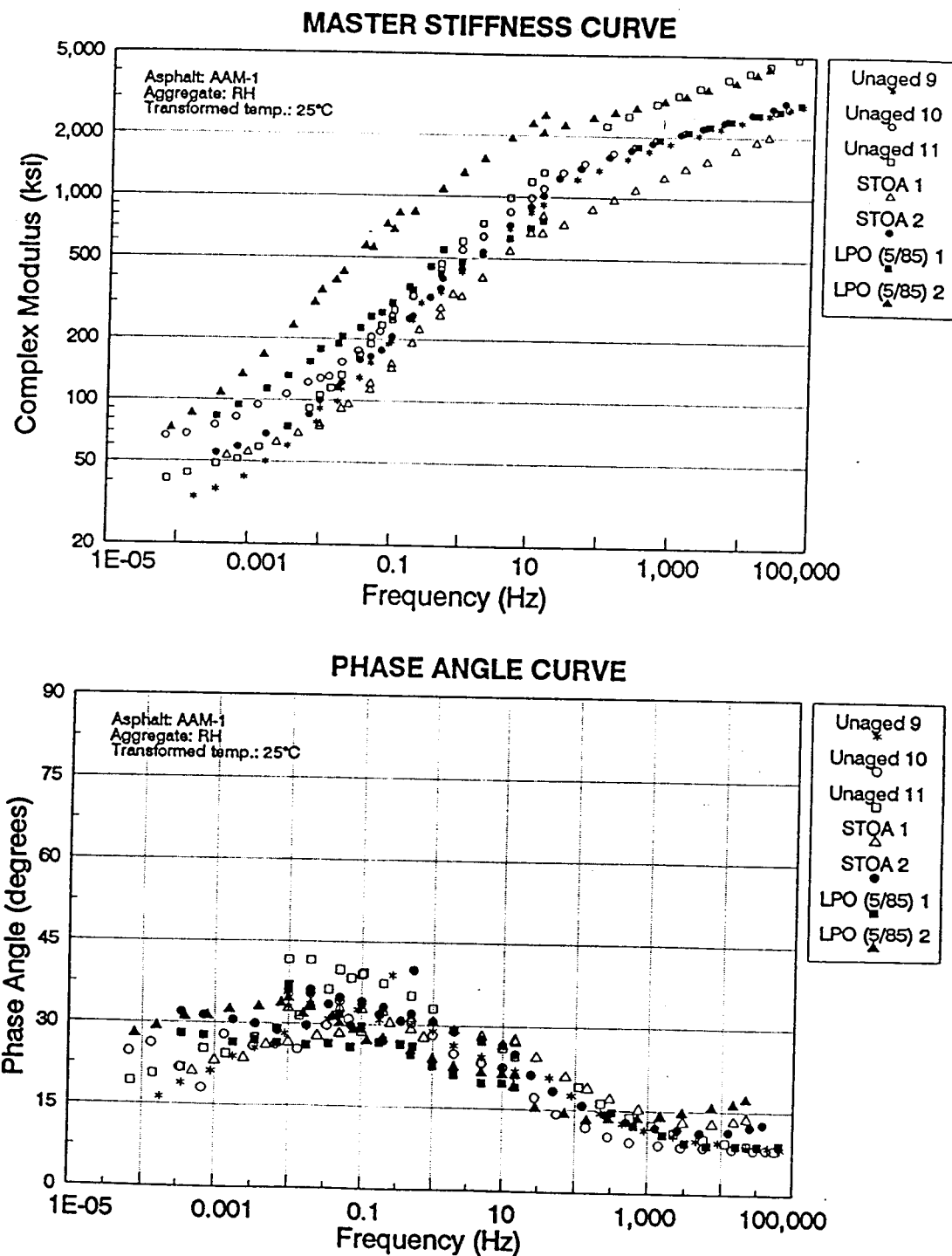


Figure F.11. Master curve and phase angle curve for asphalt AAM-1 and aggregate RC

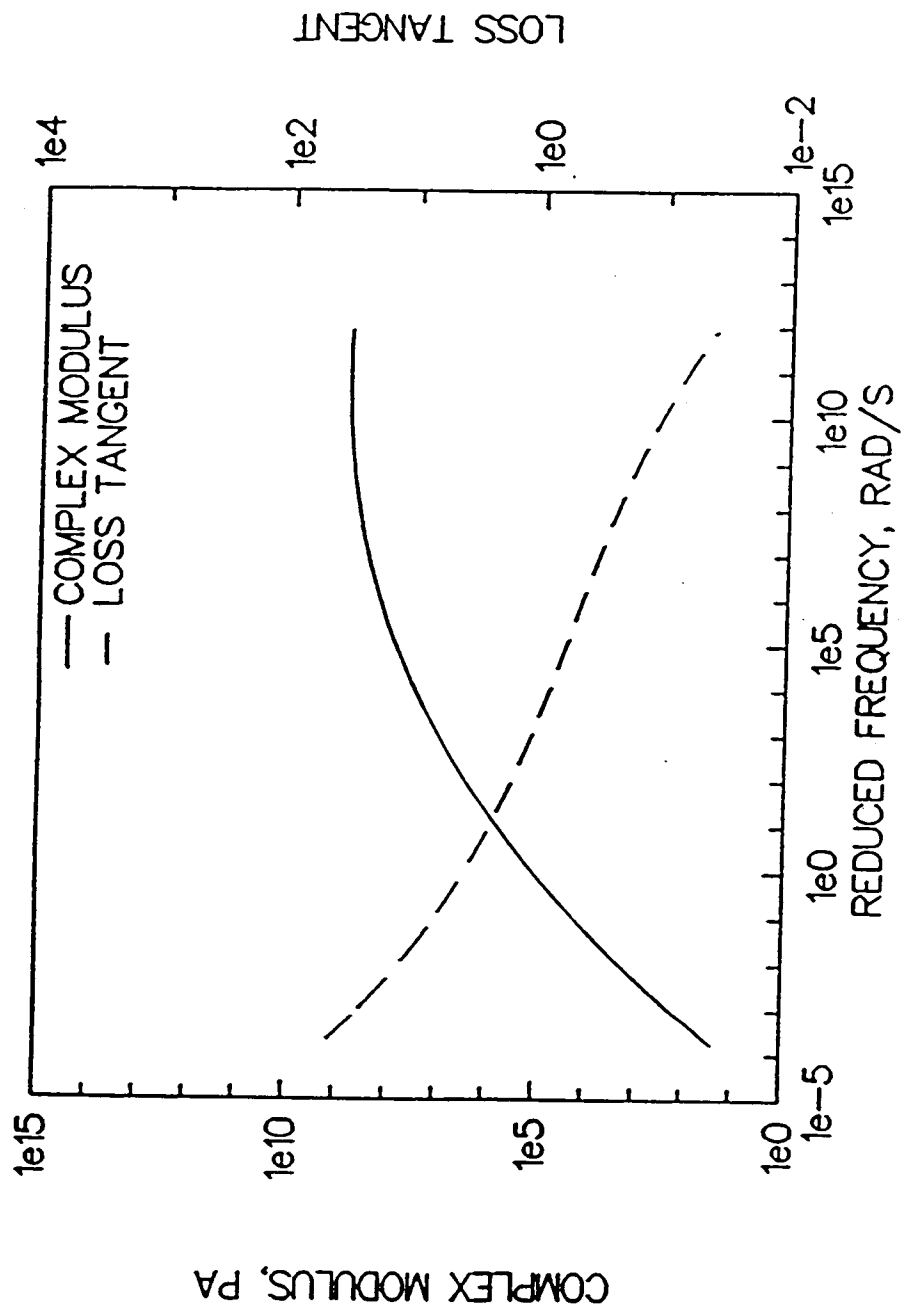


Figure F.12. Master curve for asphalt AAB-1 (after Christensen et al. 1992)

depending on the type of treatment, and have similar trends to the short-term oven aging curves.

The complex modulus data suggest that the unaged specimens at low frequencies, where the test temperature is high, correspond to the lower limit of viscous (flowing) behavior of the asphalt-aggregate mix and that they approach the conditions where the aggregate tends to dominate the material response. After undergoing short-term oven aging, the complex modulus of the asphalt-aggregate mixes increased. At low frequencies and medium test temperature, the master curves slope to a 1:1 ratio which indicates that the mixes are undergoing viscous response. Similarly, the master curves for long-term aged specimens show the same trend with higher complex moduli at lower frequencies. At high frequencies and low test temperature, the master curve starts to approach an elastic asymptote as the asphalt stiffness approaches elastic behavior.

Plots of phase angle versus frequency show that the curves peak in the frequency range of 0.01 to 1 Hz. Similar peaks are found in the data presented for modified asphalt mixes (Tayebali et al., 1991). This confirms that at an intermediate temperature or frequency, the asphalt-aggregate mix is more viscous than at high or low frequencies where either the asphalt or aggregate dominates the elastic response.

The peak phase angles for unaged, short-term aged, and long-term aged specimens are less than  $45^\circ$ . After short-term aging, the phase angle peak is lower and shifted to the left, indicating that the specimen is stiffer and that the modulus has increased. Similarly, the phase angle peak for long-term aged specimens is flatter and lower than the short-term phase angle peak depending on the aging treatment. The long-term peak is shifted even more to the left, which shows that the specimen has become even stiffer. Hence, the phase angle curves are very good indicators of mixes becoming more viscous with aging. It may be feasible to determine limiting values of phase angle maxima or minima in order to control cracking of asphalt pavements.

At high temperature or low frequency, the combinations with aggregate RH have a lower complex modulus, and at low temperature or high frequency have a higher complex modulus than the combinations with aggregate RC. The RH combinations also have higher phase angle values that peaked at higher frequencies. Phase angles that are too high might be associated with pavement rutting. These high phase angles indicate that these mixes are more susceptible to modulus change with either frequency or temperature.

The plots of master stiffness curve show that mixes with aggregate RC have a greater change in complex modulus after short-term and long-term agings, especially at high temperature (or low frequency), than mixes with aggregate RH.

The mixes become stiffer in the low frequencies region, which suggests that the mixes have undergone more aging after being long-term oven aged for 5 days at 85°C. Similarly, the phase angle curves show that the phase angle peaks for RC mixes shifted more to the left than for RH mixes. This shows that the viscous component of RC mixes changed more than that of RH mixes after short-term and long-term aging treatments. The mixes with aggregate RH have higher peak phase angles than mixes with aggregate RC, which suggests that RH mixes are more viscous.

At high frequency (or low temperature), the phase angle is small, indicating that the asphalt behaves like an elastic material. This is known as the "glassy" region where various types of molecules mobilities are "frozen-in" (Lazan 1968). As the frequency decreases (or the temperature increases), the phase angle reaches a maximum. The asphalt behaves like an elastic material at high frequency, gradually changes to a viscoelastic material and continues to change into a viscous material as the frequency decreases (or the temperature increases). After the phase angle maximum, the phase angle gradually decreases to a minimum where the mix is again in the viscoelastic phase, even though the asphalt viscosity continues to decrease. The aggregate appears to dominate the mix property at this point.

The complex moduli at 0.001, 1, and 1000 Hz were obtained from the master curve plots of unaged, short-term and long-term agings. The ratios were calculated by dividing the aged complex modulus to the unaged complex modulus. These ratios were compared to the ratios calculated using diametral resilient modulus data. It was found that the DMA ranking plots at 1 Hz have the same rankings as those plotted for the diametral resilient modulus for all aging treatments. However, the plots at 0.001 and 1000 Hz vary for each treatment. These suggest that DMA can indicate the behavior of each asphalt-aggregate mix at different test temperature in terms of each mix's susceptibility to aging.