Summary Report
on

AGING OF ASPHALT-AGGREGATE SYSTEMS

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by

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ABSTRACT

Asphalt paved roads have been used in the United States for about 100 years. They have been used in Europe since the 1850's. No doubt, pioneering pavement engineers soon realized that in the short term asphalt hardened after heating, mainly due to volatilization, and, in the long term it hardened, mainly due to oxidation.

Hardening is primarily associated with loss of volatile components in asphalt during the construction phase (short-term aging), and progressive oxidation of the in-place material in the field (long-term aging). Both factors cause an increase in viscosity of the asphalt and a consequent stiffening of the mixture. This may cause the mixture to become hard and brittle and susceptible to disintegration and cracking failures. Also, the products of oxidation may render the mixture less durable than the original mixture, in terms of wear resistance and moisture susceptibility. However, "aging" is not necessarily a negative phenomenon, since some aging may help a mixture achieve optimum properties.

Compared to research on asphalt cement, there has been little research on the aging of asphalt mixtures, and, to date, there is no standard test. Pavement engineers understand the need to model the effects of short- and long-term aging of asphalt-aggregate mixtures in structural design procedures, and while some research has addressed this need, as yet no standard procedure has emerged to address it.

This report presents a state of the art on research directed to understanding the phenomenon of aging of asphalt-aggregate mixtures. Recommendations are made for aging procedures which show promise for laboratory investigation. Test methods to evaluate aging are also considered.

At this point, extended heating procedures show the most promise for short-term aging and pressure oxidation and/or extended heating the most promise for long-term aging. Such procedures will also be considered for long-term aging, together with oxidation techniques. Promising test methods to evaluate the effects of aging of mixtures include: resilient modulus, indirect tensile test, and dynamic modulus test. Tests on recovered asphalt will also be necessary. Microviscosity and ductility tests will be considered together with chemical fractionation tests.

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DISCLAIMER

The contents of this report reflect the views of the author, who is solely responsible for the facts and accuracy of the data presented. The contents do not necessarily reflect the official view or policies of the Strategic Highway Research Program (SHRP) or SHRP's sponsors. The results reported here are not necessarily in agreement with the results of other SHRP research activities. They are reported to stimulate review and discussion within the research community. This report does not constitute a standard, specification, or regulation.

TABLE OF CONTENTS

LIST OF FIGURES

Figure

LIST OF TABLES

1.0 INTRODUCTION

1.1 Problem Definition

Asphalt paved roads have been used in the United States for about 100 years (Krchma and Gagle, 1974). They have been used in Europe since the 1850's (Croney, 1977). No doubt, pioneering pavement engineers soon realized that in the short term asphalt hardened after heating, mainly due to volatilization, and, in the long term it hardened, mainly due to oxidation. Because of the many ways asphalt can age, it seems necessary to clarify the terminology used by pavement engineers regarding hardening of asphalt and asphalt mixtures. The terms "age hardening" and "aging" are regularly used to describe the phenomenon of "hardening". The term "embrittlement" may also be used.

As implied above, hardening is primarily associated with loss of volatile components in asphalt during the construction phase, and progressive oxidation of the in-place material in the field. Both factors cause an increase in viscosity of the asphalt and a consequent stiffening of the mixture. This may cause the mixture to become excessively hard and brittle and susceptible to disintegration and cracking failures (Vallerga, 1981). Also, the products of oxidation may render the mixture less durable than the original mixture, in terms of wear resistance and moisture susceptibility (Barth, 1962). However, "aging" is not necessarily a negative phenomenon, since some aging may help a mixture achieve optimum properties. It is appropriate to note the Webster's dictionary definitions for aging which support the above discussion, viz:

- becoming old
- showing the effects or characteristics of increasing age
- acquiring a desirable quality by standing undisturbed for some time
- becoming mellow or mature
- bringing to a state fit for use or to maturity.

Since the 1930's research has continued to develop an understanding of the factors contributing to short- and long-term aging (Welborn, 1979). Much of this work has been directed toward the asphalt cement rather than the asphalt aggregate mixture, such that in 1989 we have standardized tests to determine and control the short-term aging of the neat asphalt. The present thin film oven test (TFOT) and rolling thin film oven tests (RTFOT) are an outcome of some of this research.

Compared to research on asphalt cement, there has been little research on the aging of asphalt mixtures, and, to date, there is no standard test. Pavement engineers understand the need to model the effects of short- and long-term aging of asphalt-aggregate mixtures in structural design procedures, and while some research has addressed this need, as yet no standard procedure has emerged to address it.

1.2 Purpose

The purpose of this report is to present a state of the art on research directed to understanding the phenomenon of aging of asphalt-aggregate mixtures. Short-term and long-term effects will be considered. Also included is a review of test methods that have been used to assess the effects of aging. Recommendations will be made for aging procedures and test methods which show promise for laboratory investigation. Following preliminary tests, more detailed testing will be done, which will ultimately lead to development of standard procedures for aging mixtures. Such procedures may be incorporated in a mix design or mix analysis system so that the effects of aging on the fundamental properties of asphalt mixtures can be assessed.

It is not the purpose of this report to formulate a detailed work plan for the laboratory studies that will follow. The testing required for developing standard tests and identifying limitations of such tests will be determined only after thorough study of the promising aging and test methods. Similarly, the details of mixtures to be tested will not be given here. A range of mixtures representing contemporary practice will be tested in the detailed test program. This will include mixtures designed for heavy duty pavements such as "large stone" mixtures.

1.3 Scope

As indicated above, this study will involve evaluation of methods considering the short- and long-term aging of asphalt mixtures. An extensive literature search was conducted such that the author believes that the majority of relevant studies have been considered. In doing so, it was clear that much of the literature was published 20 to 50 years ago. However, contemporary research has yielded the promising methods of aging which will be recommended for further evaluation.

1.4 Organization

After this introduction, Chapter 2 presents a literature review which concentrates on laboratory test procedures for aging asphalt and asphalt-aggregate mixtures. However, because of the need to relate laboratory aging to field aging, studies that have attempted to establish such relations are also reviewed. Similarly, since thorough evaluation of the effects of aging could include consideration of chemical properties, the final section of Chapter 2 reviews studies that have considered the relation between these properties and field performance.

Chapter 3 identifies promising aging and test methods and presents a preliminary evaluation of them. Chapter 4 discusses gaps in the knowledge regarding aging of asphalt-aggregate mixtures. Finally, Chapter 5 presents Conclusions and Recommendations. A substantial list of references is also included, along with three Appendices presenting specific details for test methods and for a proposed work plan for laboratory tests.

2.0 LITERATURE REVIEW

2.1 Introduction

A literature search was conducted through the Transportation Research Information Services (TRIS) unit of the Transportation Research Board. In addition, DIALOG, and the University of California (Berkeley) MELVYL system was used. Also, the author conducted an informal search and received many helpful suggestions from various colleagues.

The literature is extensive and, by necessity, only selected references will be cited. Table 1 presents a selection of references dealing specifically with laboratory aging and summarizes the methods used, together with the test or tests used to evaluate the extent and/or effects of aging. Discussion of the work represented in Table 1 is presented below in two sections, one relating to binder studies and another to mixture studies. Other work addressing the aging of mixtures in the field will also be reviewed, and a section is included that reviews research that has attempted to relate asphalt chemical composition to the field performance of asphalt aggregate mixtures. Before the discussion of aging and test methods, the factors influencing aging are summarized.

2.2 Factors Influencing Aging

Traxler (1961) listed five factors influencing hardening of asphalt cements in approximate order of importance, viz:

1) Oxidation
2) Volatilization
3) Time (development of internal structure on aging)
4) Polymerization induced by actinic light (free radical reactions)
5) Condensation polymerization (by heat)

He indicated that "a complete understanding of hardening will require extensive experimentation."

Table 1. Laboratory Accelerated Aging and Evaluation Methods.

Date	Investigator(s)	Aging Method	Evaluation Method
1903	Dow	18, 24 hrs, 325°F (163°C) Mixture aged for 30 min, 300°F (149°C)	Change in weight, penetration of residue Recovered asphalt - change in penetration
1937	Nicholson	Air blowing, 15 min, 425°F (229°C)	Penetration, ductility
	Raschig & Doyle	Air blowing, 15 min, 400°F (204°C)	Change in penetration
	Hubbard & Gollomb	Ottawa Sand mixture, time and temperature varied	Recovered asphalt - change in penetration
1939	Lang & Thomas	Ottawa Sand mixture, aging oven, outdoor exposure	Change in mix properties, abrasion, strength, etc.
1940	Shattuck	Mixture oven aging 30 min 325°F (163°C)	Recovered asphalt - penetration, ductility, softening point
	Lewis & Welborn	1/8-in. film oven test 5 hr, 325°F (163°C) TFOT	Change in weight, penetration, ductility
1946	Lewis & Halstead	1/8-in. film oven test 5 hr, 325°F (163°C)	Change in weight, penetration, ductility
1952	Pauls & Welborn	Ottawa Sand mixture oven aged 325°F (163°C) TFOT	Compressive strength, recovered asphalt, TFOT residue
1955	Griffin, Miles, & Penther	Shell microfilm test - 5 micron (.0002-in.) film, 2 hr, 225°F (107°C)	Viscosity before and after aging - aging index
1957	Vallerga, Monismith & Granthem	Ultraviolet and infrared weathering	Penetration Softening point Ductility
	Brown, Sparks & Smith	Rapid chilling of asphalt sample	Tensile test on asphalt sample
1958	Heithaus & Johnson	Road tests - laboratory aging - microfilm test	Recovered asphalts Microfilm Aging Index
1961	Traxler	TFOT and microfilm 15 micron (.0006-in.) film, 2 hr, 225°F (107°C)	Microviscosity at 77°F (25°C) compared
	Halstead & Zenewitz	TFOT and 15 micron film (.0006 in.) film, 2 hr, 225°F (107°C)	Microviscosity at 77°F (25°C) compared A shear rate of 0.05 sec^{-1} was used
1963	Hveem, Zube & Skog	Shell microfilm test modified - 20 micron (.0008-in.), 24 hr, 210°F (99°C) Rolling TFOT and TFOT 325°F (163°C), 50 min Cohesiograph test	Microviscosity at 77°F (25°C) before and after aging Viscosities of RTFOT, TFOT, and recovered asphalts compared
1968	Lee	TFOT at 325°F then POB at 150°F 24, 48, 96, 240 hr at 29 psig and 132 psig Asphalt and sand-asphalt mixes	Microviscosity at 77°F - limiting viscosity - time to harden to 30 megapoises - shear index Asphaltene content
1969	Schmidt & Santucci	Rolling microfilm test 20 micron (.0008-in. bottle) 210°F (99°C)	Microviscosity of residue

Table 1. Laboratory Accelerated Aging and Evaluation Methods (Cont.)

Date	Investigator(s)	Aging Method	Evaluation Method
1973	Lee	TFOT POB at 150°F and 20 atm Recovery of field aged materials	Microviscosity at 77°F Capillary viscosity at 140°F Microductility, Fraass test Asphaltene content and oxygen percentage Rostler analysis
1976	Benson	TFOT Actinic light Mixtures weathered in the field	Microviscosity at 77°F Penetration at 77°F
	Plancher, Green & Petersen	RTFOT, RMFO, Column oxidation Mixture oven aging for 5 hr @ 302°F (150°C) Lime and nontreated asphalt and mixtures	Microviscosity @ 77°F (25°C) Asphaltene determination Chemical analysis Resilient modulus
1977	Kumar & Goetz	Permeation by air at 140°F (60°C) at a head of 0.02 in. of water (0.5 mm) for 1, 2, 4, 6, 10 days	Creep Test @ 70°F ± 3° (21°C ± 2°) - repeated load conditioning - then 5 psi for 5 min Slope and intercept of creep curve used indicate progressive oxidation Ratio of slope or intercept at X days to initial slope = Durability Index
1981	Kemp & Predoehl	Actinic Light Weathering test, Rolling microfilm test, Ottawa sand mixture aging, Modified Shell Microfilm test Mixtures weathered in field	Penetration 77°F (25°C) Ductility 77°F (25°C) Resilient Modulus (M_R) Microviscosity @ 77°F Capillary Viscosity @ 140°F
	Santucci, Goodrich & Sundberg	Tilt Oven Durability test @ 235°F (113°C) for 168 hr and @ 239°F (115°C) for 100 hr	Viscosity @ 60°C and 135°C Penetration @ 4°C and 25°C Ductility @ 25°C
1983	McHattie	Extended RTFOT 100 hr, 239°F (115°C)	Penetration 77°F (25°C) Kinematic-viscosity 275°F Resilient Modulus EAL (Equivalent Axle Load) Life
1985	Edler, et al.	Weatherometer - 100 micron asphalt films, RTFOT extended to 8 hr; Pressure oxidation for 96 hr, 149°F (65°C), 300 psi Modified TFOT - 100 micron films, 24 hr	Viscosity at 113°F (45°C) by sliding plate microviscometer at a shear rate of 0.05 sec^{-1} Oxidation level - infrared spectra High molecular weight constituents
	Hugo & Kennedy	Oven age hardening of mixtures @ 100°C Ultraviolet exposure of mixtures for 54 hr and 14 days	Microviscosity @ 77°F (25°C) Shrinkage of beams of mixture
1986	Kim et al.	Pressure oxidation @ 140°F (60°C) and 100 psi 0 to 5 days Recovery of field aged materials	Capillary viscosity at 140 and 275°F Fraass and penetration tests Resilient Modulus and fatigue Corbett-Swarbrick analyses.

Table 1. Laboratory Accelerated Aging and Evaluation Methods (Cont.)

Date	Investigator(s)	Aging Method	Evaluation Method
1988	Von Quintas et al.	Short-term oven aging @ 275°F (135°C) for 8, 16, 24, & 36 hr Long-term pressure oxidation @ 135°F (60°C and 100 psi Oven aging for 2 days @ 135°F (60°C) then 5 days @ 225°F (107°C)	Resilient modulus Indirect tensile strain Creep
	Tia et al.	Convection oven aging @ 140°F (60°C) for 1, 7, 28, and 90 days Forced draft oven aging @ 140°F (60°C) for 1, 7, 28, and 90 days Ultraviolet light aging @ 140°F (60°C) for 1, 7, 28, and 90 days Aging under natural conditions for 1, 2, and 3 years	Resilient modulus Indirect tensile strength Recovered asphalt properties including viscosity @ 140°F (60°C) penetration @ 77°F (25°C) Schweyer rheometer at 77°F and 59°F (15°C) Infrared spectral analyses Corbett-Swarbrick analyses
1989	Petersen	Thin film accelerated aging test	Weight loss due to volatilization Ketone content Viscosity

In a subsequent paper Traxler (1963), listed 15 effects which may reduce the binding properties of asphalt. Table 2 is reproduced from this paper. It should be noted that the effects listed are not necessarily in order of severity and may not apply when considering asphalt mixtures. Traxler provides some experimental data to support the list of effects, however, he states clearly in the paper that "some effects have not been given experimental consideration.

More recently, Peterson (1984) has listed three major factors causing hardening of asphalt in asphalt mixtures, viz:

1) loss of oily components by volatility or absorption
2) changes in composition by reaction with atmospheric oxygen
3) molecular structuring that produces thixotropic effects (steric hardening)

It should be noted that these include four of Traxler's 15 effects.

The majority of researchers considering aging of asphalt and asphalt mixtures have limited their investigations to those factors given by Petersen. Significant developments are presented below.

2.3 Binder Studies

Table 1 shows that early work on laboratory aging procedures emphasized binder studies and, in particular, extended heating. Much of this was done by or for the Bureau of Public Roads (BPR). Many of these studies and others relating to asphalt durability were summarized by Welborn (1979).

2.3.1 Extended Heating Procedures

a) Thin Film Oven Test (TFOT)

Welborn (1984) includes a reference to Dow (1903) indicating the use of an early extended heating test. Nearly 40 years later, Lewis and Welborn (1940) introduced the TFOT for differentiating among asphalts in terms of volatility and hardening characteristics. Further work was reported by Lewis and Halstead (1946).

Table 2. Effects Which May Reduce the Binding Properties of Asphalt (after Traxler, 1963)

Effects	Influence by					Occurs		Ways to Retard
	Time	Heat	Oxygen	Sunlight	B&G Rays	At Surface	In Mass	In General, Selected Source and Process
1. Oxidation (in dark)	X	X	X	-	-	X	-	1) Inert atmosphere 2) Free radical inhibitors
2. Photooxidation (direct light)	X	X	X	X	-	X	-	1) Protection from light 2) Inert atmosphere 3) Free radical inhibitors
3. Volatilization	X	X	-	-	-	X	X	Protection from heat
4. Photooxidation (reflected light)	X	X	X	X	-	X	-	1) Proection from light 2) Inert atmosphere 3) Free radical inhibitors
5. Photo chemical (direct light)	X	X	-	X	-	X	-	1) Protection from light 2) Additives?
6. Photo chemical (reflected light)	X	X	-	X	-	X	X	1) Protection from light 2) Additives?
7. Polymerization	X	X	-	-	-	X	X	Free radical inhibitors
8. Development of an internal structure (aging) (Thixotropy)	X	-	-	-	-	X	X	1) Add dispering agents 2) Change source and processing of asphalt
9. Exudation of oil (Syneresis)	X	X	-	-	-	X	-	Reduce paraffinic content
10. Changes by nuclear energy	X	X	-	-	X	X	X	————————
11. Action of water	X	X	X	X	-	X	-	Change source and processing
12. Absorption by solid	X	X	-	-	-	X	X	Improve dispersion of asphalt
13. Adsorption of components at solid surface	X	X	-	-	-	X	-	————————
14. Chemical reactions or catalytic effects at interface	X	X	-	-	-	X	X	————————
15. Microbiological deterioration	X	X	X	-	-	X	X	Add fungistatic and bacteriostatic agents

A 50 ml sample of asphalt was heated in a 1/8 in. (3 mm, i.e. 3000 microns) film in a 5.5 in. (140 mm) diameter flat container for 5 hrs at 325°F (163°C). This test was adopted by AASHTO in 1959, and by ASTM in 1969. Residue from the TFOT was tested for penetration, ductility and softening point.

Welborn (1979) notes that the test is primarily used to predict relative changes that occur in asphalts during hot-plant mixing. Several researchers have made significant modifications to the TFOT, as will be described below. One minor change was done by Edler et al (1988), who reduced the film thickness to 0.004 in. (100 microns) and increased the exposure to 24 hrs.

b) Shell Microfilm Test

Griffin, Miles and Penther (1955) reported this test for 0.0002 in. (5 micron) film of asphalt aged for 2 hrs on glass plates. The "aging index" parameter was used to evaluated asphalts, i.e.:

$$\text{Aging Index} = \frac{\text{viscosity after aging}}{\text{viscosity before aging}} \tag{2.1}$$

Welborn (1979) notes that no data were reported correlating field and laboratory aging, but that the study indicated the importance of volatilization from the standpoint of hardening.

Hveem, Zube and Skog (1963) proposed a modification to the Shell microfilm test, by increasing the film thickness to 0.0008 in. (20 microns) and the exposure time to 24 hrs. They demonstrated indirectly a relationship between field and laboratory hardening.

Traxler (1961), and Halstead and Zenewitz (1961) also presented slight variations of the microfilm test as noted in Table 1.

c) Rolling Thin Film Oven Test (RTFOT)

The RTFOT was developed by the California Division of Highways in an effort to age asphalt in thinner films than the 1/8 in. used in the TFOT. Hveem, Zube and Skog (1963) reported the procedure. Bottles containing 35 g samples are rotated in an oven at 325°F (163°C) for 75 minutes. This causes a 0.05 in. (1.25 mm or 1250 microns) film to flow around the glass jar.

This test was adopted by ASTM in 1970 and is used by several Pacific Coast states for routine testing.

Several researchers have made slight modifications to the RTFOT, for example, Edler et al. (1985) used a time period of 8 hrs. More significant modifications are presented below.

d) Rolling Microfilm Oven Test (RMFO)

Schmidt and Santucci (1969) modified the RTFOT procedure by dissolving asphalt in benzene, coating the inside of the bottle, then allowing the benzene to evaporate and leave a 20 micron film of asphalt. This procedure was necessary to create a 0.008 in. (20 micron) film of asphalt. The asphalt is then rotated at 210°F (99°C) for 24 hrs. A disadvantage of this test is the very small amount of asphalt obtained from each bottle (0.5 g).

e) Tilt-Oven Durability Test (TODT)

Kemp and Predoehl (1981) adapted the RTFOT test by tilting the oven by 1.06° (higher at the front). The tilting prevented asphalt migration from the rotating bottles. The test is continued for 168 hrs at 235°F (113°C). Penetration, viscosity, and ductility tests were run on the residue. The comparison of data obtained with field data will be discussed in a subsequent section.

McHattie (1983) has reported a similar extension of the RTFOT. He used a temperature of 239°F (115°C) for a period of 100 hours.

Santucci et al. (1981) evaluated the TODT at the conditions noted above (168 hrs at 235°F), and at the conditions used by McHattie. They note that these latter conditions were formerly recommended by the Caltrans researchers. Santucci et al. evaluated several asphalts with both test procedures, and concluded that 168 hrs at 235°F (113°C) was more severe. They measured penetration and viscosity of the aged asphalt, and also concluded that viscosity was a more sensitive measure of changes in the TODT. An alarming point raised by these authors is that two ovens produced by different manufacturers were used in the study and generated significantly different results.

f) Thin Film Accelerated Aging Test (TFAAT)

Petersen (1989a) and his coworkers have developed a modification of the RMFO to provide a 4 g sample of asphalt; a practical size for further testing. A temperature of 235°F (113°C) was used for 72 hrs. This test was developed to complement a column oxidation procedure developed by Davis and Peterson (1966) in which asphalt is coated on Teflon particles to film thicknesses of about 15 microns and oxidatively aged in a gas chromatographic column at 266°F (130°C) for 24 hrs by passing air through the column.

As background to the test development, Petersen (1989a) notes that many asphalts exhibit volatile loss in the TFOT and RTFOT in excess of what is typically lost during the lower temperature long-term aging in the field. He notes that Corbett and Merz (1975), in analysis of asphalts used in the Michigan Test Road observed virtually no change in the asphalt saturate fraction (which contains the potentially volatile asphalt components) after 18 years of pavement service. He also notes that with regard to the TFOT and RTFOT, "the level of oxidative aging and hardening in the tests is much less than what occurs during field aging, reflecting only the aging that occurs during hot-plant mixing." With this background in mind, the TFAAT was developed to produce a representative level of volatilization and oxidation.

Figures 1 and 2 show the effect of asphalt film thickness on ketone content and weight loss of the sample. Ketones and anhydrides are formed on oxidation. Based on data from studies by Schmidt (1973) and Petersen et al. (1983), the 4 g sample was found to have a representative level of ketones after 24 hrs aging at 266°F (130°C). However, the volatile loss was excessive (Figure 2) and therefore studies on the effect of bottle opening size on volatile loss were conducted. Figure 3 shows the results and the opening size of 3 mm selected for use.

Subsequent studies were done at 235°F (113°C) for comparison with the California tilt-oven durability test (TODT). Figure 4 shows the data obtained and the period for testing of 3 days that should be used at this temperature.

Petersen (1989a) presents data comparing the aging index of asphalts aged using the TFAAT and TFOT (Table 3). He also presents TFAAT data for several asphalts (Table 4) and data for field aged asphalts (Table 5). Table 3 shows that, for the asphalt evaluated, the TFAAT is much more severe than the TFOT. Aging is described by the aging index as defined in Eq. 2.1. Comparison of the aging indices in Table 4, representing TFAAT data, and Table 5, representing field data, shows that the TFAAT causes a similar level of aging to that occurring in the field. The field data was obtained from an extensive FHWA-funded study conducted by Vallerga et al. (1970). Petersen cautions that there is a difference between the "kinetics" of oxidation in the field and in the TFAAT. He illustrates this point with data given by Zube and Skog (1969) for the Zaca-Wigmore test road (Figure 5) which shows that field oxidation and viscosity increase fall off with time in the field, whereas they do not (Figure 4) in the TFAAT. This is associated with temperature difference between the two conditions and its effect on molecular structuring and steric hardening. Petersen

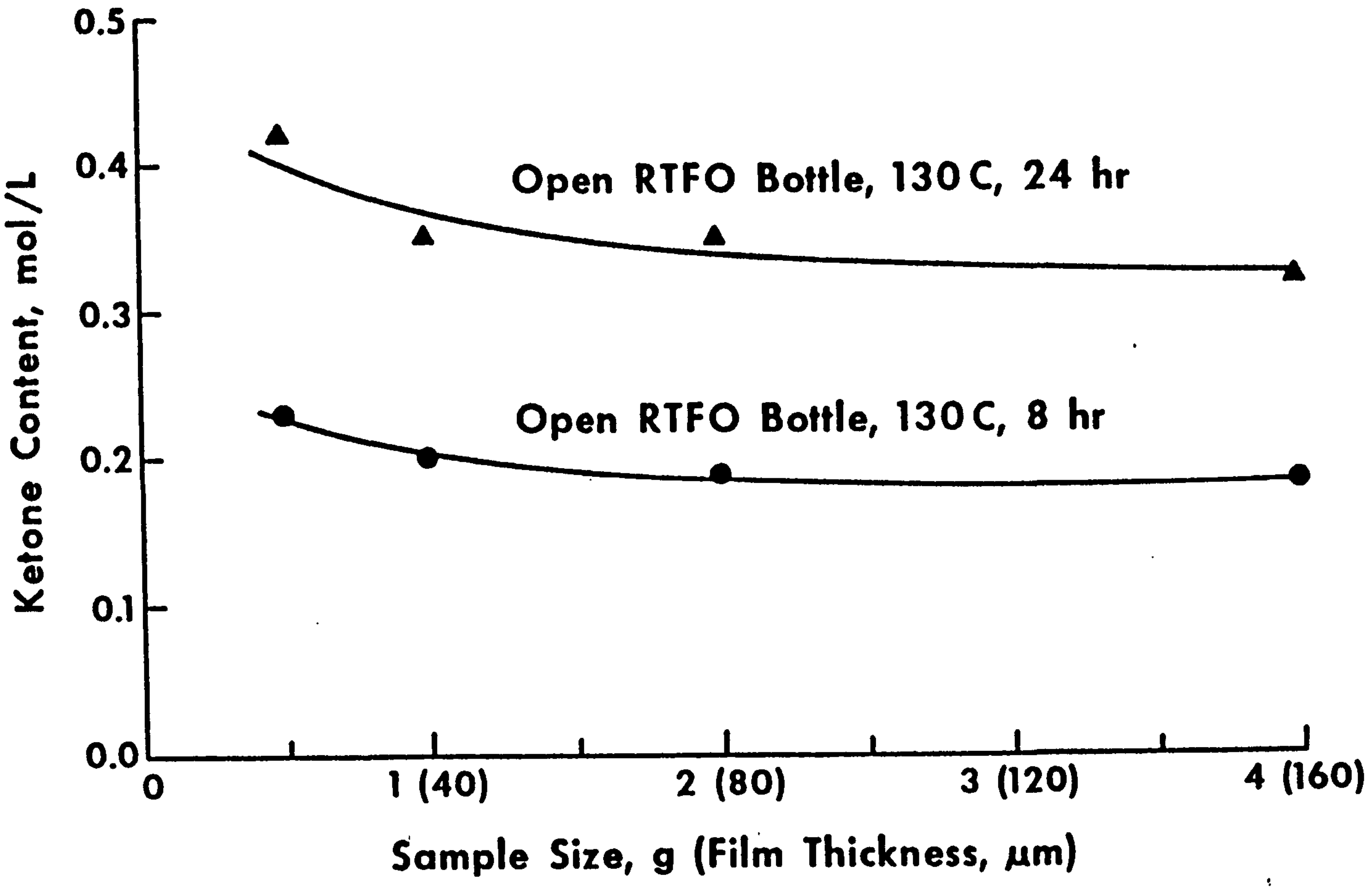


Figure 1. Effect of Asphalt Film Thickness on Level of Oxidation at 130°C (after Petersen, 1989)

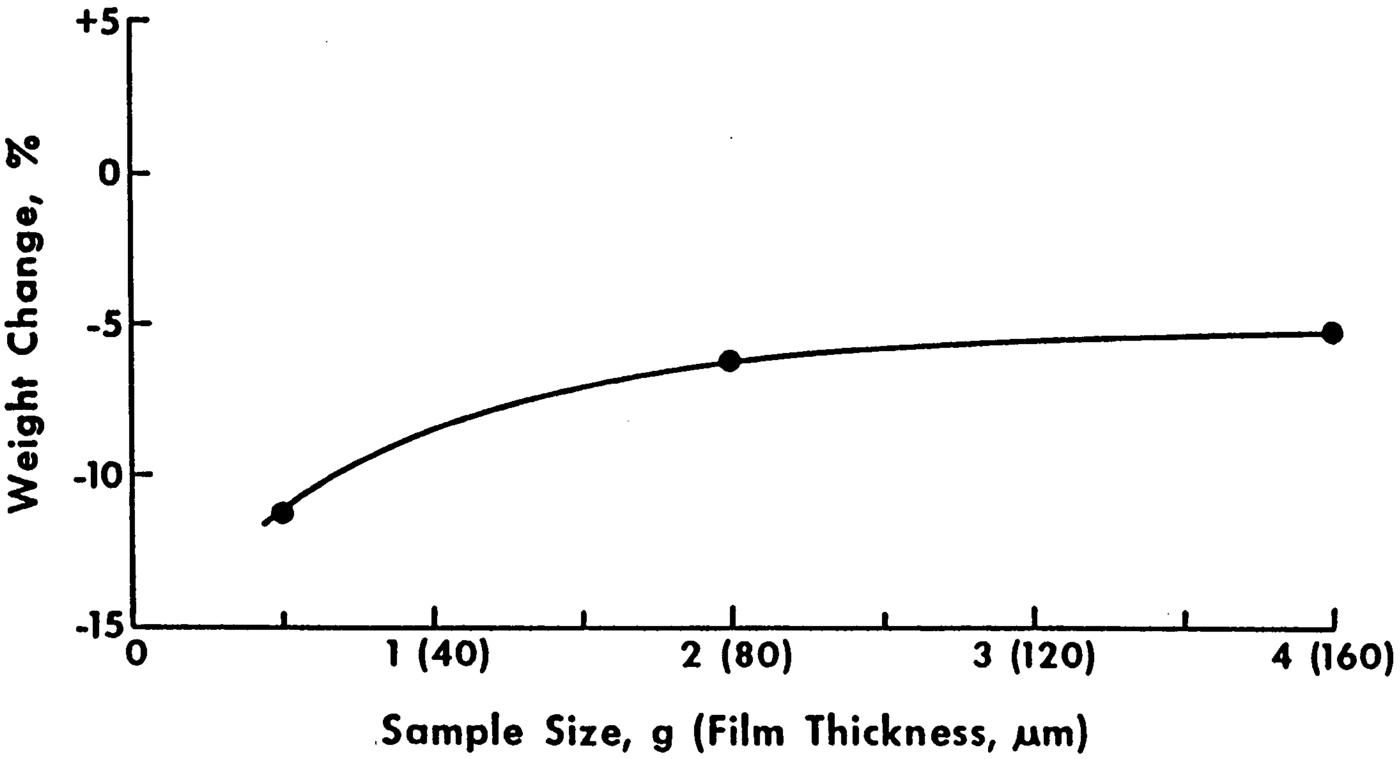


Figure 2. Effect of Asphalt Film Thickness on Weight Loss in RTFO Bottle at 130°C (after Petersen, 1989)

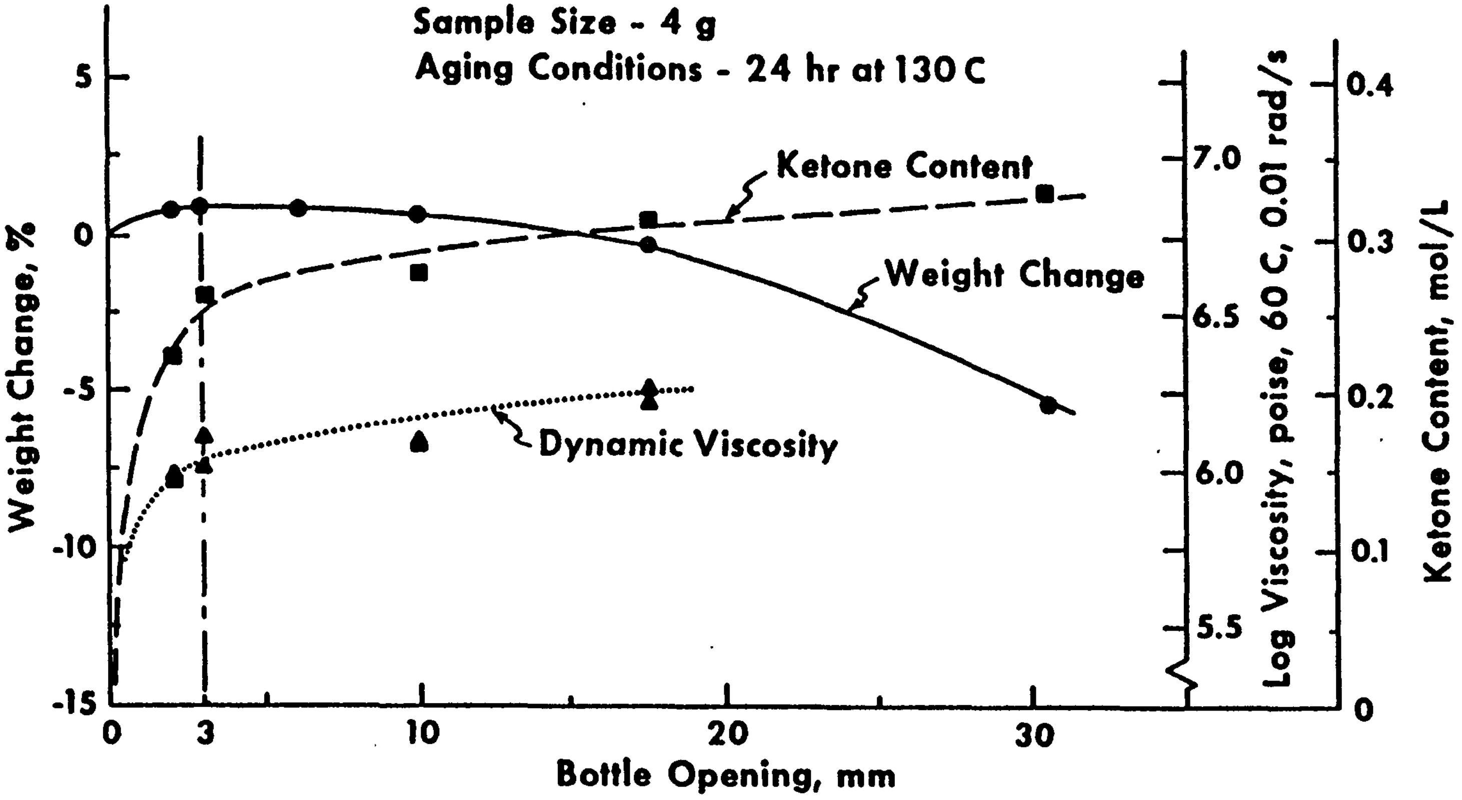


Figure 3. Effect of Bottle Opening Size on Asphalt Volatile Loss, Level of Oxidation, and Viscosity Increase at 130°C (after Petersen, 1989)

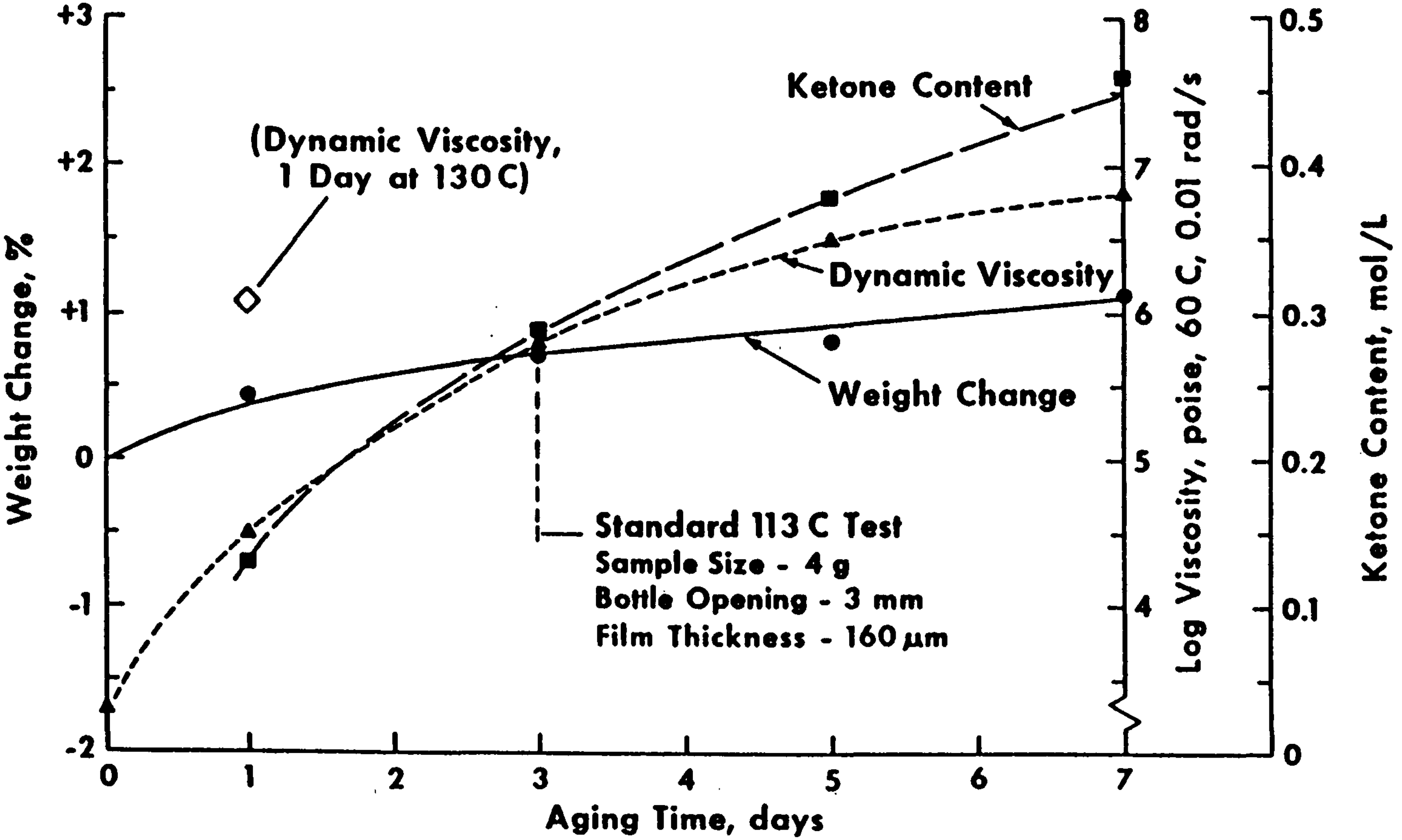


Figure 4. Effect of Aging Time on Asphalt Volatile Loss, Level of Oxidation, and Viscosity Increase at 113°C (after Petersen, 1989)

Table 3. Level of Aging of BOSCAN AC-10 Asphalt B-3051 Using Different Methods (after Petersen, 1989)

Method of Aging	Aging Index[1]	Log Aging Index
Unaged	1.0	0
TFOT	3.0	0.48
TFAAT	214.0	2.33

[1] Viscosity ratio at 140°F (60°C)

Table 4. Aging Index of AC-10 Asphalts from Different Sources Using TFAAT (after Petersen, 1989)

Asphalt Source	Grade	Aging Index[1]	Log Aging Index
California Valley	AC-10	10	1.00
Midcontinent	AC-10	32	1.51
Michigan	AC-10	74	1.87
North Slope-Maya Blend	AC-10	90	1.95
California Coastal	AC-10	134	2.13
Boscan	AC-10	214	2.33
West Texas-Maya Blend	AC-10	338	2.53

[1] Viscosity ratio at 140°F (60°C)

Table 5. Relationships Between Pavement Void Content and Viscosity of Asphalts After 11-13 Years of Pavement Service (after Petersen, 1989)

Average Initial Viscosity,[1] poise 140°F (60°C)	Average Viscosity, Recovered Asphalt,[2] poise, 140°F (60°C)	Average Void Content,[2] %	Aging Index	Log Aging Index
1.9×10^3	8.0×10^3	2	4.2	0.62
1.9×10^3	3.1×10^4	4	16	1.20
1.9×10^3	8.0×10^4	6	42	1.62
1.9×10^3	1.6×10^5	8	84	1.92
1.9×10^3	3.1×10^5	10	163	2.21

[1] Average for 18 initial asphalts
[2] From best fit line of viscosity versus void content for 53 projects

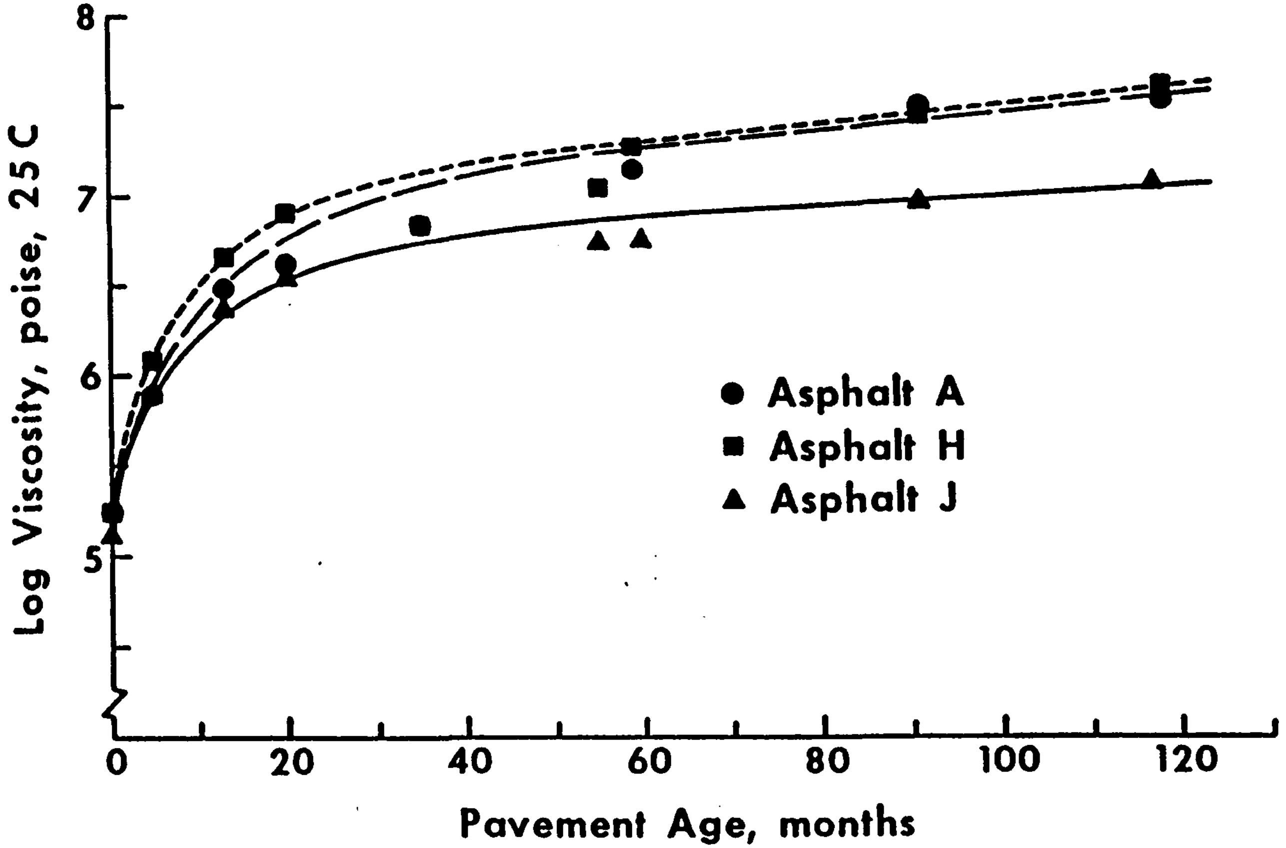


Figure 5. Viscosity Change During Pavement Service of three Zaca-Wigmore Asphalts (after Petersen, 1989)

suggests that these two phenomena significantly reduce the rate of field hardening after the first two or three years of service.

2.3.2 Oxidation Tests

a) Air Blowing

Nicholson (1937) utilized air blowing at 425°F (229°C) at a rate of 1/3 ft^3/min (0.0091 m^3/min) for 15 min. As noted in Table 1, penetration and ductility were measured before and after aging, and asphalts retaining higher values were judged superior. Another approach was to age asphalts to a penetration of 20 to 25 and those retaining highest ductility were judged to be best.

Raschig and Doyle (1937) air-blew asphalts at 400°F (204°C) at the same rate as Nicholson, and determined the change in penetration.

b) Pressure Oxidation

D.Y. Lee initiated a five-year study for the Iowa State Highway Commission in 1966 entitled, "Development of a Laboratory Durability Test for Asphalts." Lee (1968) reports on the initial developments of the study and recognizes the need to develop a two-stage test to simulate:

1) hardening during mixing
2) hardening during service life

Lee adopted the TFOT without modification to simulate the first stage and pressure oxidation for the second stage. Lee notes that other researchers had used pressure oxidation procedures and that the British Road Research Laboratory (RRL) developed a device for use with road tars (HMSO, 1962). The RRL included the Fraass brittle point as a means of evaluating the extent of aging.

Lee (1973) reported the major findings of the Iowa study and presented the following procedure for the "Iowa Durability Test" (IDT):

1) Use of BPR thin film oven test (TFOT) on the original asphalt.

2) Application of pressure-oxidation treatment to the residue from the TFOT (film thickness 1/8 in.) for up to 1000 hrs at 150°F (65°C) and a pressure of 20 atm (300 psi) of oxygen.

3) Evaluation of the physical and chemical changes in asphalt during the artificial aging process in relation to the original properties.

Lee's pressure oxygen vessels were 7.5 in. diameter by 7.5 in. high and rated to 450 psi.

The test procedures used by Lee (1973) are summarized in Figure 6. Figures 7, 8 and 9 show sample data from his study and the correlation between laboratory data and field data. Lee found that the development of aging followed a hyperbolic model as suggested by Brown et al. (1957):

$$\Delta Y = \frac{T}{a + bT} \tag{2.2}$$

where ΔY = change of property with time T or the difference between the zero-life value and the value at any subsequent time

a = constant; the value of the property at zero time

b = rate of change of the property

$1/b$ = the ultimate change (limiting value) of the property

Figures 8 and 9 show high correlation coefficients between curves developed with this model and the observed data from the laboratory and field tests. Only in the case of ductility data, was this model inadequate.

Correlation of field and laboratory data also resulted in hyperbolic relationships, implying a different rate of aging in the field, as suggested by Petersen (1989a). Figure 10 shows several master curves, one of which represents all nine projects evaluated by Lee. From this curve, Lee concludes that 46 hrs of aging with the IDT is equivalent to 60 months field aging for Iowa conditions.

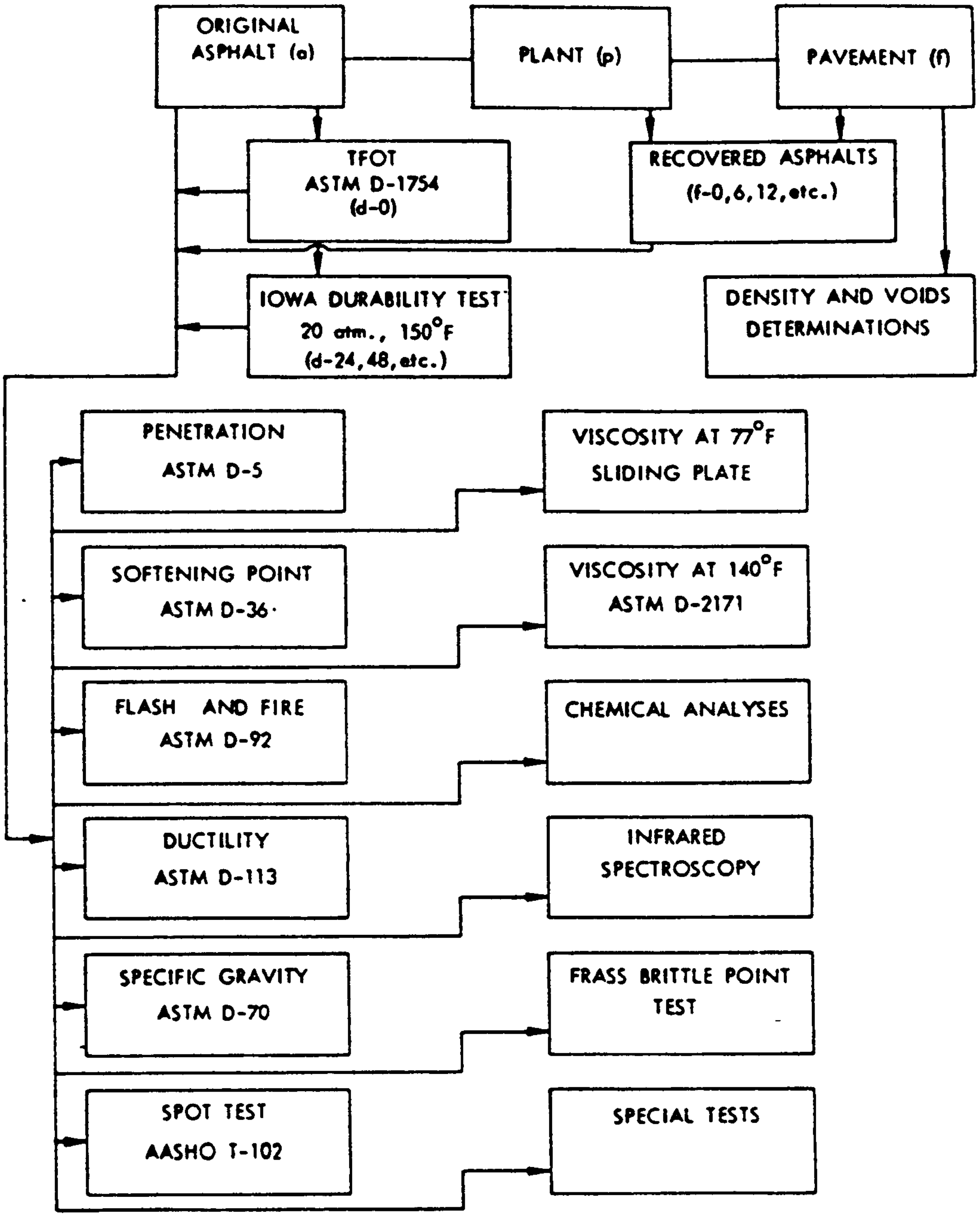


Figure 6. Flowchart of Testing Procedures (after Lee, 1973).

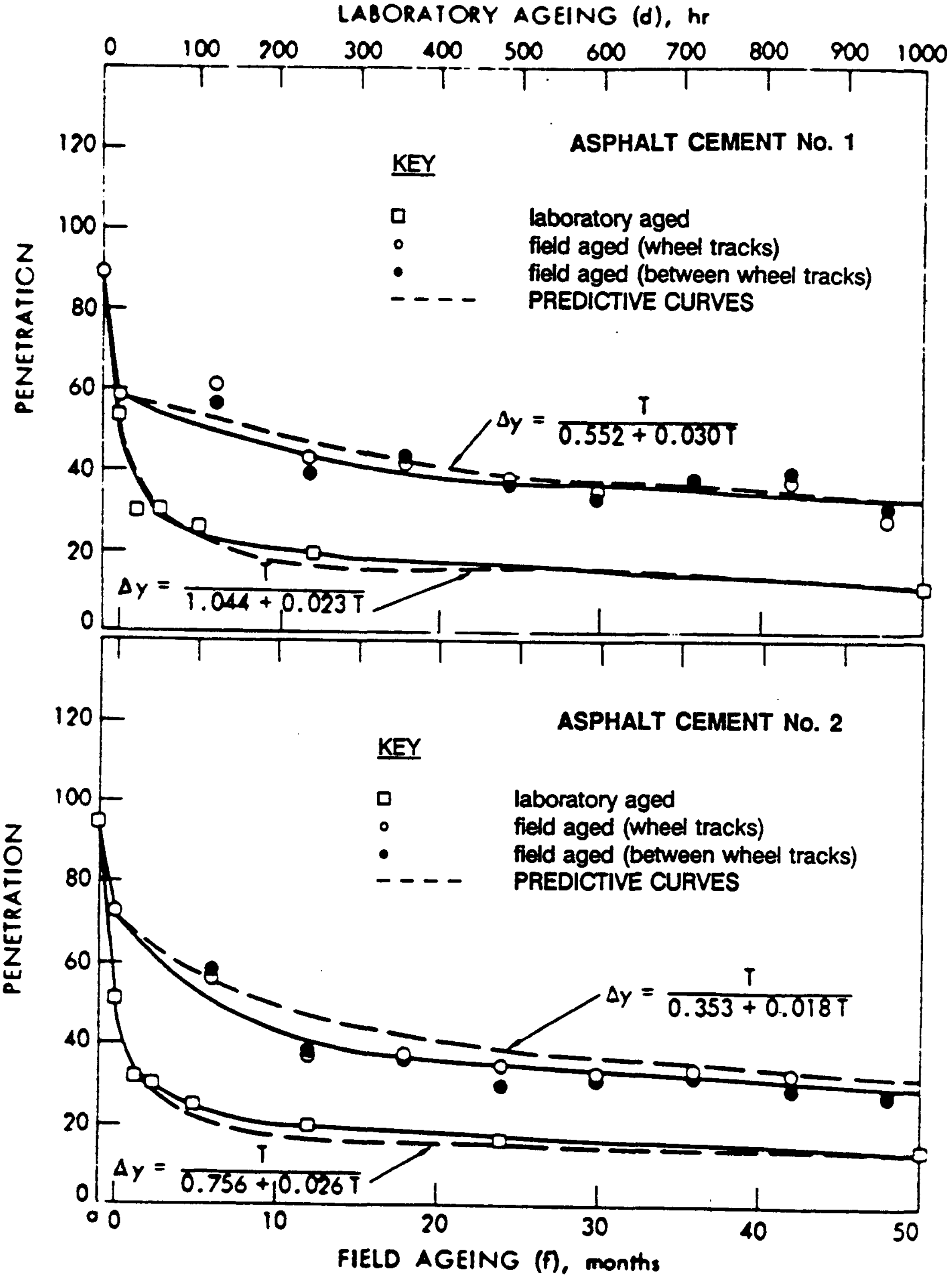


Figure 7. Penetration versus Time of Aging (after Lee, 1973)

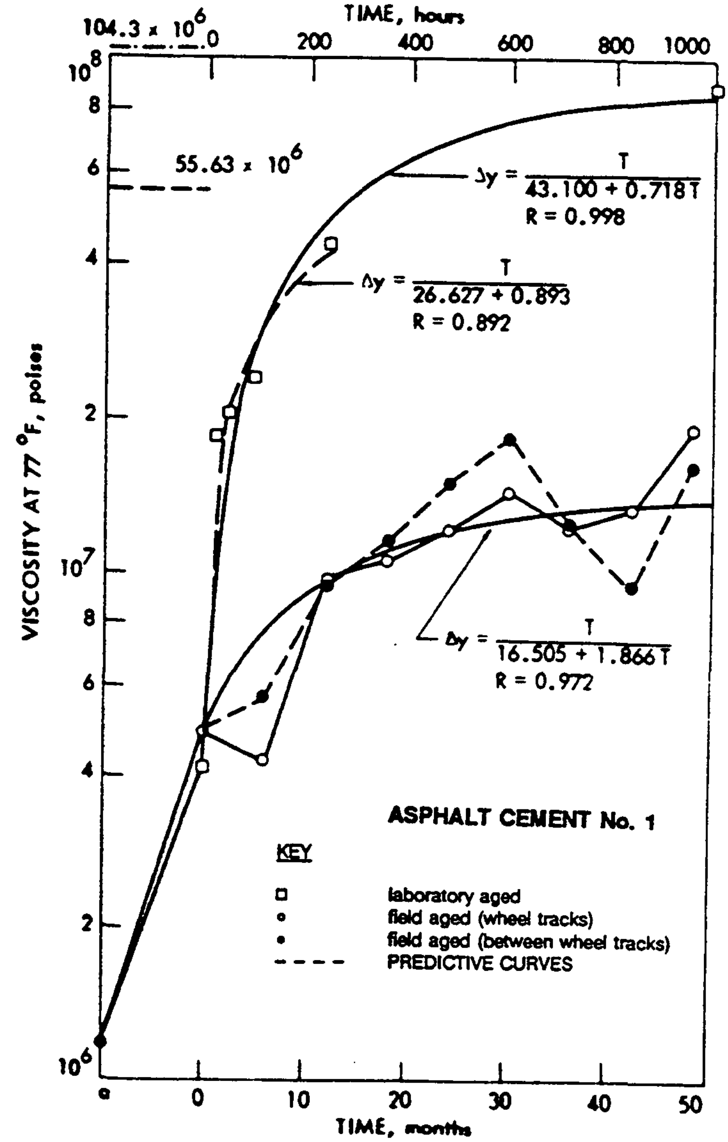


Figure 8. Viscosity at 77°F versus Time of Aging (after Lee, 1973)

Figure 4. Viscosity at 140 F versus time of aging.

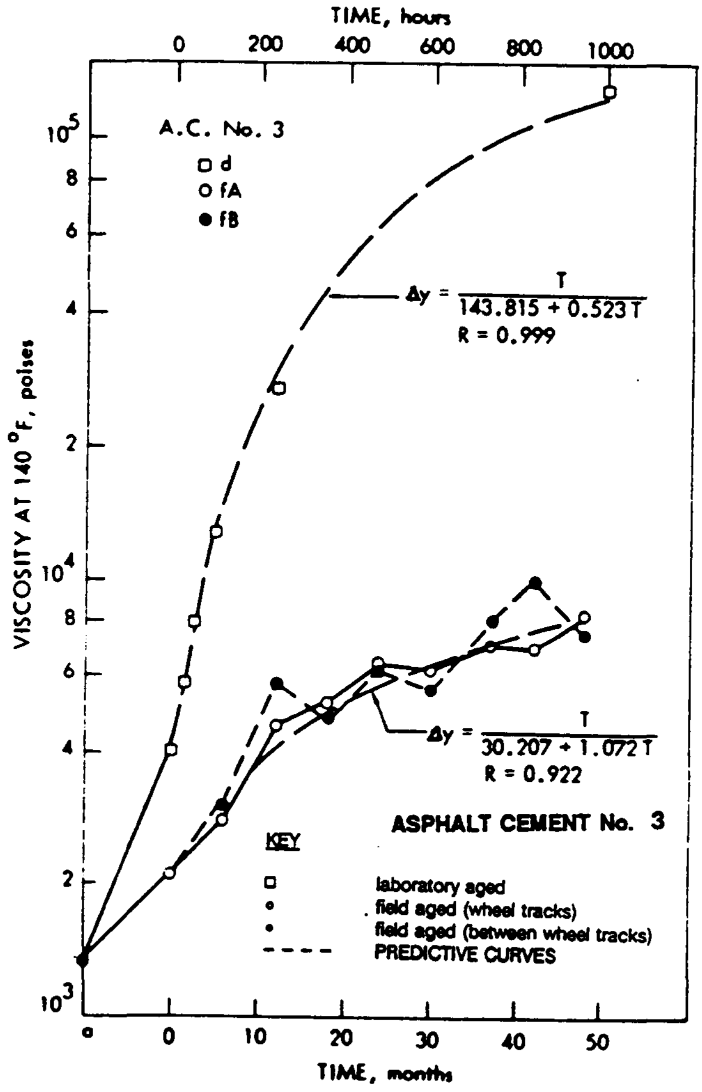


Figure 9. Viscosity at 140°F versus Time of Aging (after Lee, 1973)

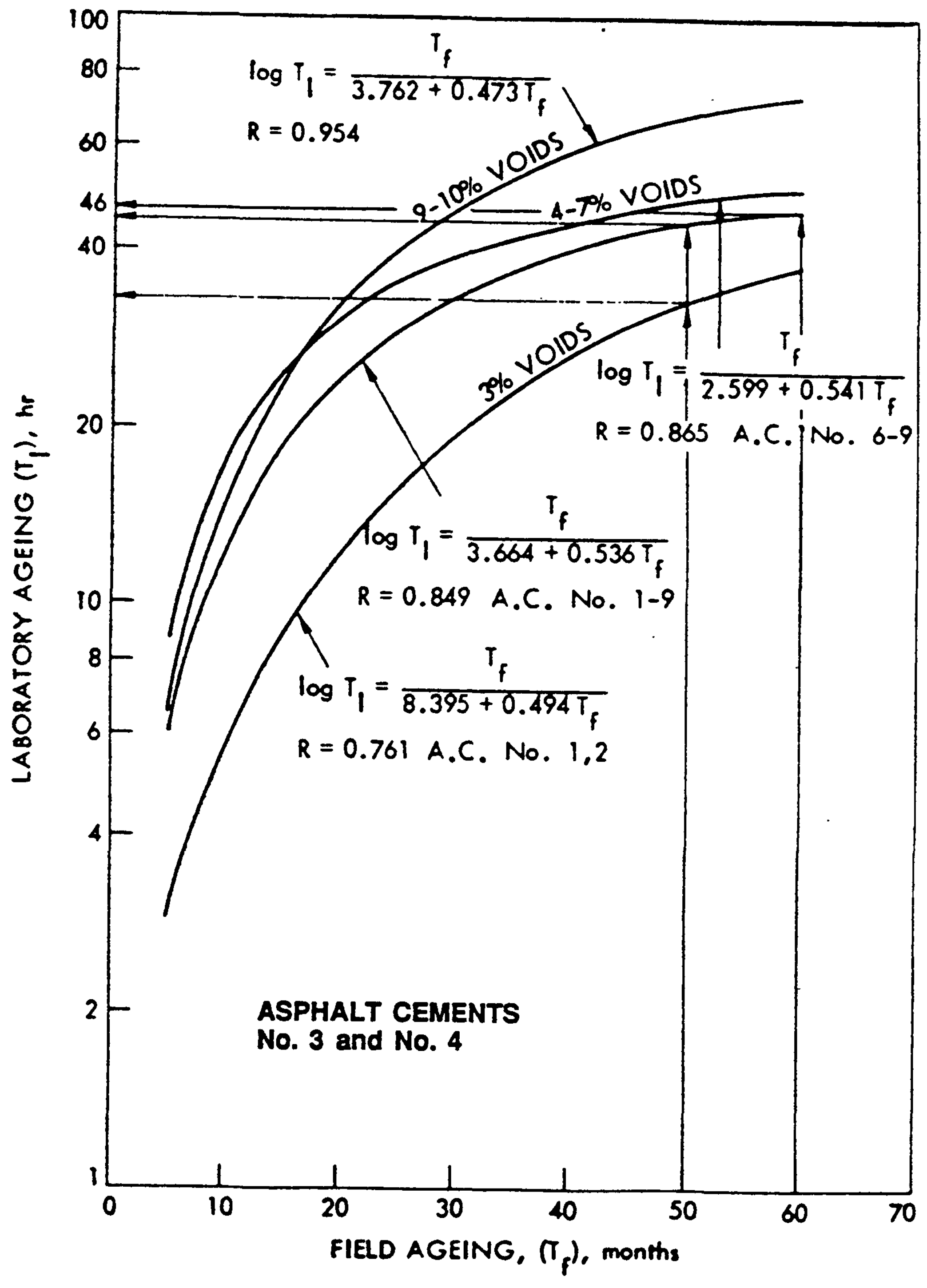


Figure 10. Time Equiivalency Correlation Curves by Voids Level (after Lee, 1973)

Edler et al. (1985) utilized a similar approach to Lee (1973) in a study to evaluate procedures to retard oxidative hardening of asphaltic surfacings in South Africa. They utilized a two-stage procedure with extended RTFOT (ERTFOT) aging done first for 8 hrs, followed by pressure oxidation at 300 psi and 149°F (65°C) for 96 hrs. Evaluation of the extent of asphalt aging was by sliding plate microviscometer at a shear rate of 0.05 sec^{-1} and at 113°F (45°C). Oxygen absorption measurements were also made, and the molecular weight composition of original and aged asphalts were determined by gel permeation chromotography. Table 6 and Figure 11 show viscosity data for lime and nontreated asphalt. Note that the curves are of a hyperbolic form, and that the levels of aging index are similar to those reported by Petersen (1989a). Extended TFOT work was also attempted on 100 micron (0.004 in.) films for 24 hrs following the ERTFOT. The viscosity levels achieved with this approach were outside the range of the microviscometer. Also, the levels of oxygen absorbed and high molecular weight fractions were higher than when the pressure oxygen treatment was used.

Kim et al. (1986) utilized pressure oxidation to age asphalt on Fraass test plaques. Asphalt samples were subjected to 100 psi oxygen pressure at 140°F (60°C) for two and five days. Figure 12 shows the data for three different asphalts. These authors note that an advantage of this aging approach is that the asphalt is aged on the "container" used for the test method to evaluate the extent of aging. The asphalt film on a Fraass plaque is 0.5 mm thick (500 microns). This is much thicker than any of the thin film tests described above. However, Figure 12 shows that the Fraass breaking point is significantly altered by pressure oxidation.

2.3.3 Ultraviolet and Infrared Light Treatment

Vallerga et al. (1957) reported on studies using both ultraviolet (UV) and infrared (IR) light to age asphalt films in TFOT containers. The ultraviolet treatment was found to be

Table 6. Effect of ERTFOT and Pressure Oxidation (POV) on Viscosity (after Edler et al., 1985).

Asphalt Penetration	Lime Added (%)	Viscosity (Pa.s)		Aging Index
		Original	ERTFOT & POV Residue	Aged / Original
60/70	0	4.37×10^3	7.50×10^5	171.6
	6	6.40×10^3	3.64×10^5	56.9
	12	1.18×10^4	4.64×10^5	39.3
80/100	0	1.91×10^3	3.44×10^5	179.8
	6	3.40×10^3	1.33×10^5	39.1
	12	5.73×10^3	1.58×10^5	27.5

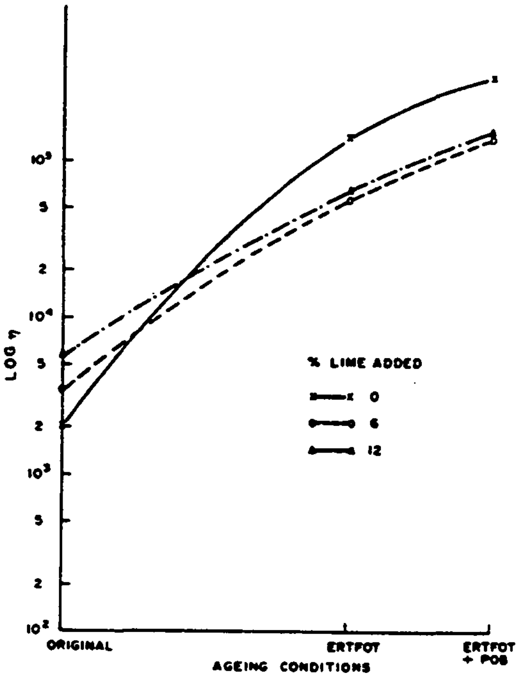


Figure 11. Effect of Lime Addition on the Viscosity of the 80/100 Pen Asphalt (after Edler, 1985)

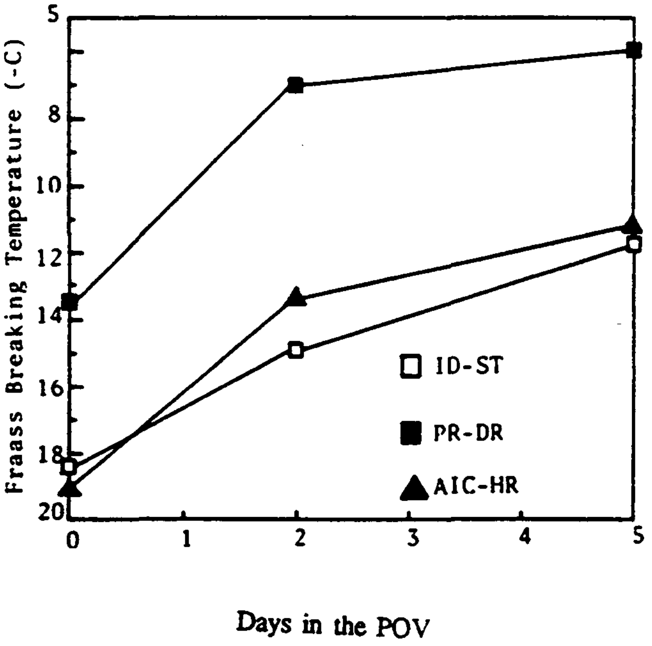


Figure 12. Effect of POV Aging on Fraass Temperature of Three Asphalt Cements (after Kim et al., 1986)

most effective in terms of changes in penetration softening point and ductility of the asphalts treated.

Traxler (1963) presents data to show the effects of "actinic" light and refers to the effect as a photochemical reaction. His data clearly shows that there is a significant effect on thin films of asphalt (3 microns) but with thicker films the effect was small.

Edler et al. (1985) used a weatherometer (ASTM, 1979) to age 0.004 in. (100 micron) films of asphalt. A temperature of 149°F (65°C) was used, and a 102 min cycle of UV, followed by 18 min of UV and water spray (300 psi). Samples were aged for a total of 32.5 hrs, 73.5 hrs, 7 days, and 14 days. The effects of the weatherometer on viscosity are shown in Table 7. It was also shown that the oxidation levels and changes in high molecular weight constituents were comparable with the other aging procedures used by these investigators.

2.3.4 Steric Hardening

Traxler (1963) identified molecular structuring (thixotropy) as one of his 15 effects (see Table 2). The result of this effect is steric hardening. He notes that this effect is mostly reversed by application of heat or mechanical working, but that a portion is permanent, and the extent to which it occurs depends on asphalt composition.

Brown et al. (1957) present some data from a study to demonstrate that more steric hardening occurs in asphalts when they are cooled slowly. The effect was demonstrated by carrying out simple tensile tests on the asphalt samples. Petersen (1984 and 1989a) has also emphasized the role of steric hardening. The discussion of the TFAAT presented above outlines Petersen's (1989a) "model" of the role of steric hardening, but as yet no test has been developed to quantify its precise role.

Table 7. Effect of the Weatherometer on Viscosity (after Edler et al., 1985).

		Viscosity (Pa.s)					Aging Index	
			Weatherometer Residue				Aged/Original	
			Hours		Days		Hours	
Asphalt Penetration	Lime Added (%)	Original	32.5	73.5	7	14	32.5	73.5
60/70	0	$4.37x10^3$	$1.35x10^5$	$6.43x10^5$	UTT*	UTT	30.9	147.1
	6	$6.40x10^3$	$1.25x10^5$	$6.58x10^5$	UTT	UTT	19.5	102.8
	12	$1.18x10^4$	$2.24x10^5$	$9.19x10^5$	UTT	UTT	19.0	77.9
80/100	0	$1.91x10^3$	$5.54x10^4$	$3.08x10^5$	UTT	UTT	29.0	161.3
	6	$3.40x10^3$	$8.40x10^4$	$2.75x10^5$	UTT	UTT	24.7	80.9
	12	$5.73x10^3$	$9.08x10^4$	$3.31x10^5$	UTT	UTT	15.8	57.8

*UTT - unable to test; residues too hard

2.4 Mixture Studies

Less work has been done with laboratory aging of mixtures than with binder studies. However, as early as 1903, Dow reported extended heating of mixtures, and the evaluation of the effects by recovery of asphalt and comparison of penetration before and after aging.

The development of an accepted procedure for extracting and recovering asphalt from mixtures (Abson, 1933) no doubt influenced subsequent research, since many of the early studies on mixture aging (see Table 1) involve tests on recovered asphalt, for example, Hubbard and Gollomb (1937) and Shattuck (1952).

Lang and Thomas (1939) used a mixture of Ottawa sand and several asphalts in their extensive study. This provided a standard "aggregate" which the authors felt would be reproducible. Several other researchers have used Ottawa sand mixtures as will be reported below. Lang and Thomas made about 12,000 mix specimens in their study. Some aging tests were done with ultraviolet light, and, some with extended exposure to heat and air. Evaluation of the extent of aging was by abrasion tests on the mixture, consistency tests and chemical composition tests on Abson recovered asphalts.

The above background shows that significant contributions to understanding mixture aging were made over 50 years ago. Subsequent contributions will be presented below.

2.4.1 Extended Heating Procedures

Pauls and Welborn (1952) exposed 2 in. by 2 in. cylinders of Ottawa sand mixture to 325°F (163°C) for various time periods. The compressive strength of the cylinders was determined. Also, the consistency of the recovered asphalt was compared with that of the original asphalt. Asphalts representing major sources produced in the mid-1930's were used in the study. The conclusions of the study included the following:

1) The hardening properties of asphalt cements can be determined either by measuring the compressive strength of laboratory oven-aged, molded

specimens, by tests on asphalt recovered from the laboratory-aged specimens or by the TFOT.

2) Because the TFOT procedure is relatively simple, it is highly valuable for predicting high temperature hardening of asphalt cements.

It should be noted that there is no suggestion that the TFOT was suitable for predicting long-term hardening due to field weathering.

Plancher et al. (1976) also used an oven aging procedure on 1 in. thick by 1-1/2 in. diameter samples (25 mm by 40 mm) as a part of a study to evaluate the effect of lime on oxidative hardening of asphalt. It was found that the resilient modulus (measured with a diametral test configuration) of lime-treated mixtures was changed less than nontreated mixtures by the aging process. It should be noted that Plancher et al. present an explanation of the chemistry of lime action. This study should also be considered with that by Edler et al. (1985), which found that lime had a considerable effect in retarding aging in asphalt samples.

Kemp and Predoehl (1981) utilized Ottawa sand mixtures in their work. A planetary oven at 140°F (60°C) was used, with various times of exposure up to 1200 hours. The asphalt was then recovered and could be subjected to "micro-tests". These authors favored the TODT since it generates a much larger quantity of asphalt.

Hugo and Kennedy (1985) describe a method of oven aging of mixture briquettes at 212°F (100°C). They note that this procedure is similar to an Australian standard (Standards Association of Australia, 1980). This procedure was carried out for 4 and 7 days in a dry atmosphere and in an atmosphere of 80% relative humidity, due to the need to assess a project located close to the ocean. Asphalt was recovered for viscosity determination from 4 in. (100 mm) samples cored from laboratory produced slabs. Table 8 shows the data obtained. Also, the samples were weighed before and after aging, and the weight loss used to indicate loss of volatiles. Finally, beam samples were cut from the slabs and the shrinkage during the aging test determined.

Table 8. Binder Viscosity—Before and After Accelerated Aging (after Hugo and Kennedy, 1985).

Mix No.	Bitumen Content %	Relative Density %	Mix Grading	Lime Content %	Viscosity of Recovered Binder at 25°C (Log Pa.S)					AI After 7 Days Dry Aging
					Before Aging	Aging 4 Days With Humidity	Aging 4 Days - Dry	Aging 7 Days With Humidity	Aging 7 Days - Dry	
1	5.5	95.2	CONT	-	5.16	5.51	5.32	5.62	5.67	3.23
2	5.5	93.0	CONT	-	5.16	5.47	5.46	5.62	5.60	2.75
3	6.5	90.4	GAP	-	5.16	5.80	5.89	5.95	6.10	8.70
4	7.0	90.4	GAP	-	5.16	5.84	5.84	5.96	5.90	5.50
5	6.0	90.6	CONT	2	5.16	5.61	5.59	5.81	5.81	4.47
6	7.0	97.5	GAP	1	5.16	5.46	5.46	5.54	5.59	2.69
7	7.0	90.5	GAP* + chips	-	5.16	5.74	5.74	5.88	5.80	4.37
8	7.0	92.7	GAP + chips	2	5.16	5.70	5.73	5.86	5.83	4.68
9	5.0	92.8	OPEN	-	5.16	5.72	5.77	5.72	5.91	5.62
10	5.0	92.5	OPEN	1.5	5.16	5.75	5.76	5.92	5.92	5.75

*Applied at 10 kg/m^2.

Von Quintas et al. (1988) have published the findings from the second phase of the study to develop an Asphalt Aggregate Mixture Analysis System (AAMAS). They investigated the use of forced-draft oven aging to simulate "production hardening", viz short-term hardening. They compared the recovered versus initial penetration and viscosity ratios for asphalts used in each of five projects, for both field and laboratory aging. The laboratory method involved oven heating loose mixture samples for periods of 8, 16, 24 and 36 hours at a temperature of 275°F (135°C). Figure 13 shows data for two of the projects and shows that similar levels of aging are obtained in the laboratory and the field, but there is also considerable scatter in the laboratory data.

Von Quintas et al. (1988) discounted the possibility of using the TFOT or RTFOT to age the asphalt first, and then prepare laboratory mixtures, because this would be time consuming. Such an approach was used by Vallerga et al. (1967).

Von Quintas et al. (1988) also investigated "long-term environmental aging". A forced draft oven was investigated with the following conditions:

1) place six compacted specimens in the oven at 140°F (60°C) for 2 days, then remove three specimens.
2) increase the temperature to 225°F (107°C) and age the remaining three specimens for 5 days.

Also, a pressure oxidation treatment was investigated, with three compacted specimens conditioned for 5 or 10 days at 140°F (60°C) and 100 psi. The data for the oven aging and oxidation treatments are shown in Table 9. It was found that indirect tensile strengths were higher for the oven-aged mixtures and failure strains were lower than for pressure oxidized mixtures implying that the oven aging was more severe. Initial and recovered asphalt properties are also shown, also indicating that the oven aging was more severe for one mixture but less severe for the other two. However, it should be noted that the data for the oxygen-treated samples appears to be transposed for Texas projects and disordered

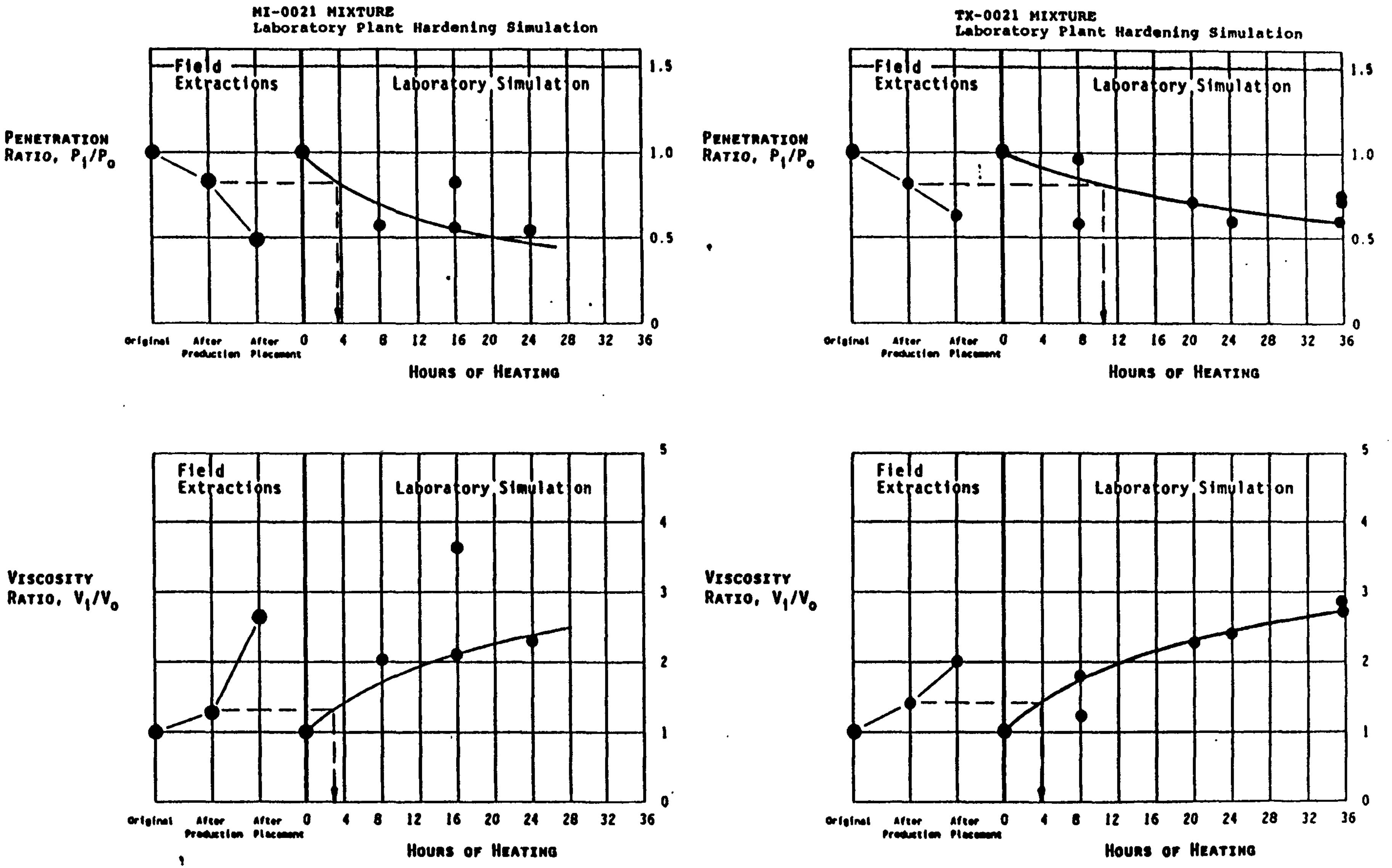


Figure 13. Change in Asphalt Properties During Construction and With Different Laboratory Heating Time Intervals for the MI-0021 and TX-0021 Mixtures (after Von Quintas et al., 1988).

Table 9. Summary of Strength Data for Specimens Conditioned Using Different Accelerated Age Hardening Techniques (after Von Quintas et al., 1988)

State/ Project	Accelerated Aging Method	No. of Days	Penetration (77°F)	Viscosity (140°F)	Indirect Tensile Strength psi	Strain at Failure mils/in.	Resilient Modulus ksi	Viscosity Aging Index
Michigan MI-0021	Unaged	0	60	2144	84	14.56	482	1.00
	Oxygen Bomb	5	63	3844	111	14.04	480	1.79
		10	43	7542	123	12.13	582	3.52
	Forced Draft Oven	2	76	2512	120	9.88	—	1.17
		7	49	5897	139	6.59	—	2.75
Texas TX-0021	Unaged	0	30	4388	129	9.01	601	1.00
	Oxygen Bomb	5	55	3488	151	8.84	738	0.79
		10	64	2822	167	5.98	683	0.64
	Forced Draft Oven	2	37	5654	200	5.11	—	1.29
		7	30	9904	241	2.69	—	2.26
Virginia VA-0621	Unaged	0	37	6000	114	7.97	758	1.00
	Oxygen Bomb	5	32	28021	128	10.23	569	4.67
		10	35	25021	121	10.66	431	4.17
	Forced Draft Oven	2	47	4401	146	5.37	—	0.73
		7	27	7910	177	2.69	—	1.32

for the Virginia project. The viscosity data for the oven aged Virginia project also appear to be disordered, and there is no modulus data for the oven aged samples.

Von Quintas et al. (1988) recommend the oven aging approach over the pressure oxidation approach. However, this author feels that more extensive research is needed before a method should be selected, particularly since the data is very limited and questionable. The future research should include some evaluation of the oxidation levels achieved by chemical tests rather than exclusively by physical tests. Temperature levels used should be carefully evaluated, since these may cause specimen disruption in some cases. Such a problem may have occurred in the AAMAS study and contributed to the unusual trends in the data. In addition, the levels of aging need to be examined closely. The viscosity data in Table 9 were used by this author to determine an aging index (see Eq. 2.1) for each aged sample and it can be seen that the highest levels achieved are low compared to those achieved in the field, as reported by Petersen (Table 5). Also, follow-up studies should be done on the five field projects utilized in the AAMAS study.

An excellent feature of the AAMAS study is their emphasis that the tensile strain at break is a better indicator of the effect of aging than the tensile strength. This is logical since the dominant effect of aging is embrittlement and failure to accommodate traffic and environmentally induced strains.

2.4.2 Oxidation Tests

Kumar and Goetz (1977) describe a study of the effects of film thickness, voids and permeability on asphalt hardening in an asphalt-aggregate mixtures. Their method of hardening the mix involved "pulling" air through a set of compacted specimens at a constant head of 0.02 in. of water. The low head was used to avoid turbulence in the air flow through the specimen. The specimens were maintained at 140°F (60°C). The test was interrupted at periods of 1, 2, 4, 6 and 10 days and the samples tested in simple creep. The creep test data were the only measures of aging in this study. There was no recovery of asphalt from the aged mixtures in this study, therefore, it is very difficult to assess the extent of aging achieved.

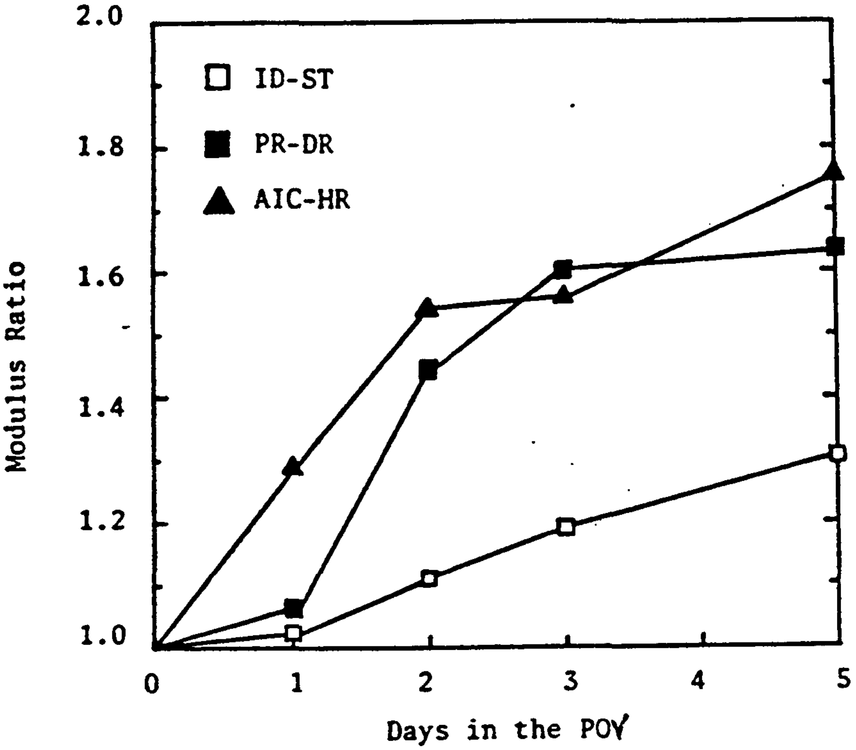
A valuable feature of the Kumar and Goetz study is the quantifying of film thickness and permeability. They evaluated open-graded and dense-graded mixtures produced with a range of air voids, permeabilities, and film thicknesses. It was concluded that for open-graded mixes, the ratio of a film thickness factor to permeability is the best predictor of resistance to hardening. However, for dense mixtures, permeability is the best predictor. It should be noted that Goode and Lufsey (1965) also concluded that permeability was a better indicator of aging susceptibility than voids content.

Kim et al. (1986) utilized pressure oxidation to age laboratory compacted samples representative of Oregon mixtures. Samples were subjected to oxygen at 100 psi and 140°F (60°C) for 0, 1, 2, 3 and 5 days. The effects of aging were evaluated by resilient modulus and fatigue life determined by the diametral test. The modulus results for the three mixtures evaluated are shown in Figure 14. Data are plotted in terms of modulus ratio, viz:

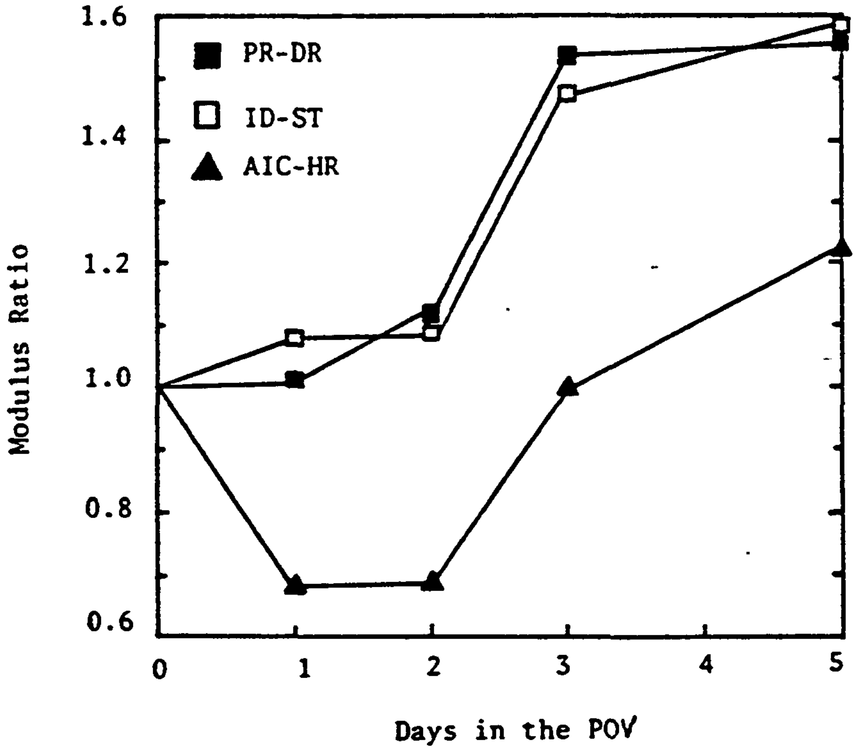
$$\text{modulus ratio} = \frac{\text{modulus of aged mixture}}{\text{modulus of unaged mixture}} \qquad (2.3)$$

Modulus ratios generally increased with aging time and more rapily for the poorly compacted mixtures (88% compaction level). However, a problem exhibited by one of the mixtures was a lowering of modulus in the early part of the aging procedure. This was attributed to a loss of cohesion in the samples at the temperature of 140°F (60°C) used in the aging procedure. Similar results were found with pressure oxidation tests done by Von Quintas et al. (1988) as shown in Table 9. This is a potential problem with any aging procedure for compacted samples, if an elevated temperature is used. Some confinement of the samples may be desirable or it may be necessary to use a lower temperature.

The fatigue data obtained by Kim et al. is shown in Figure 15 and shows a steady increase with aging with the more poorly compacted mixtures showing longer fatigue lives.

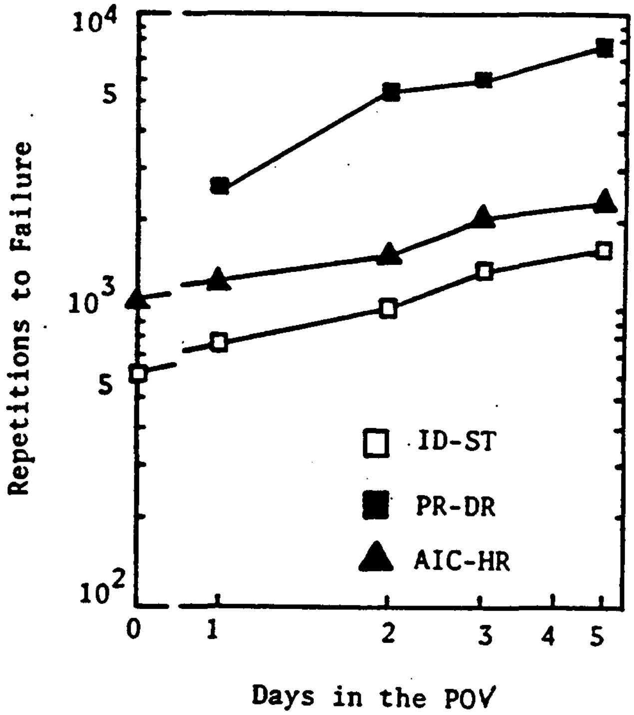


(a) At 88% Compaction Level

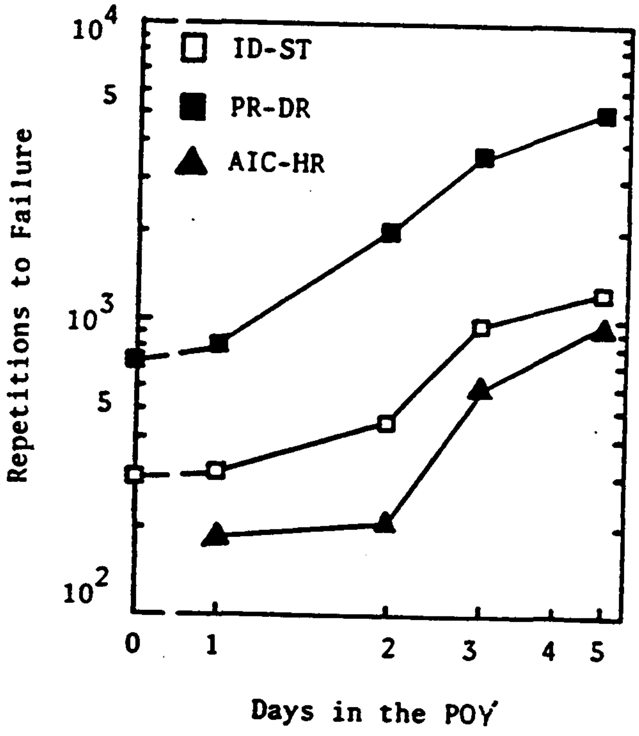


(b) At 94% Compaction Level

Figure 14. Aging Modulus Ratios for Three Asphalt Mixtures (after Kim et al., 1986)



(a) At 88% Compaction Level



(b) At 94% Compaction Level

Figure 15. Fatigue Life of Three Mixture Specimens (after Kim et al., 1986)

These tests were carried out at a fixed level of applied tensile stress and therefore, the stiffer mixes experience lower strain levels. A different trend would be expected if a fixed tensile strain had been used.

2.4.3 Ultraviolet/Infrared Treatment

Hveem, Zube and Skog (1963) presented a comprehensive description of various tests and specifications for paving grade asphalts. This included an infrared weathering test for Ottawa sand mixtures. The mixtures are produced from sand of No. 20 to No. 30 size and 2% asphalt. This gives a "statistically uniform film of asphalt" of about 5 to 7 microns (i.e. .005 to .007 mm). The mixture is tested in a "semi-compacted state". The infrared radiation was controlled to give a constant mass temperature of 140°F (60°C) and an air stream at 105°F (41°C) was maintained across the specimen.

Hveem et al. describe a calibration procedure to determine the number of hours required in the weathering test to correspond to field aging. This procedure utilized a shot abrasion test to evaluate the aged mixtures. The authors "state with some confidence" that 1000 hrs of exposure in the weathering machine is approximately equal to 5 yrs field aging.

Kemp and Predoehl (1981) also utilized an "actinic light weathering test", but do not include any data in the paper reviewed. The test was conducted with the following conditions:

1) 35°C (95°F)
2) 18 hrs
3) 1000 MW/cm^2 of 3660 Angstrom actinic radiation

The authors note that tests run with different asphalt film thicknesses indicate that the tests measure the hardening within the outer 5 microns of the asphalt film.

Hugo and Kennedy (1985) evaluated the effect of UV-radiation on mixtures obtained from laboratory prepared slabs and freshly constructed field projects. Two approaches were used. The first was similar to that used by Traxler (1963), and used 54 hours of UV exposure. The second was by use of an Atlas weatherometer for a period of 14 days. Tables 10 and 11 show the results of these tests, expressed in terms of the viscosity at 77°F (25°C) of the recovered asphalt. Note that the levels of aging index are very small compared to similar tests conducted on pure asphalt by Edler et al. (1985), see Table 7.

Tia et al. (1988) conducted an extensive laboratory study which included developing aging methods by heat and ultraviolet light. Table 1 includes a summary of the tests done. Their tests showed that similar levels of aging were achieved in mixture samples aged by either ultraviolet light or oven aging. They recommended that an improved procedure should be developed incorporating both ultraviolet light and forced draft oven heating. An operating temperature of 140°F (60°C) was recommended. In identifying ultraviolet light as a major cause of mixture aging, Tia et al. note that the resultant effect is a surface one. However, they cite data developed by Coons and Wright (1968) which showed much higher viscosities for recovered asphalt from the top quarter inch of cores than from depths of one-half inch. This data suggests that although the phenomenon occurs at the "surface", it does affect a significant depth of a mixture.

2.4.4 Steric Hardening

Hveem et al. (1963) describe a cohesiograph test to measure the "setting" quality of paving grade asphalts. The test involves making four Ottawa sand semi-cylindrical mixture specimens. Two of the specimens are tested immediately in the cohesiograph, and two after 24 hrs storage at 140°F (60°C). The cohesiograph samples are approximately 12 in. long and slender. They are extruded out of a support so that they act as a cantilever and will break into short sections at the test temperature. The "length of break" determined is the average length of the broken sections of the samples.

Hveem et al. note that if there is a difference between the two sets of samples, this reflects the tendency of an asphalt to "structure". They note that this is not substantially

Table 10. Aging Results of Cores from Newly Constructed Road Surfaces and Laboratory Prepared Briquettes Using 54 Hours UV-Radiation by Adapted TTI Method* (after Hugo and Kennedy, 1985).

	Johannesburg	Pretoria	Cape Town	Mpacha, Southwest Africa	Lab Mix No. 4	Lab Mix No. 5
Binder, %	5.9	6.8	5.6	9.6	7.0	6.0
Binder Penetration	40/50	60/70	80/100	60/70	80/100	80/100
Voids, %	13.2	2.2	4.9	11.0	12.0	13.0
Mix Composition—Reference Table No.	Semigap 15	Semigap 15	Cont. 15	Sand 15	Gap 12	Cont. 12
Viscosity $\eta_{0.05}$ Log Pa.S @ 25°C						
Surface: 0-5 mm						
Aged	6.46	6.27	5.90	5.42	6.08	6.13
Unaged	6.05	5.87	5.16	5.14	5.16	5.16
Aging Index	2.57	2.51	5.50	1.91	8.32	9.33

*Tests were done at University of Stellenbosch. Bitumen extracted from 5 mm slice off top of sample.

Table 11. Aging Results of Cores from Newly Constructed Road Surfaces and Laboratory Prepared Briquettes Using 336 Hours Atlas Weatherometer UV-Radiation* (after Hugo and Kennedy, 1985).

	Johannesburg	Pretoria	Cape Town	Mpacha, Southwest Africa	Lab Mix No. 4	Lab Mix No. 5
Binder, %	5.9	6.8	5.6	9.6	7.0	6.0
Binder Penetration	40/50	60/70	80/100	60/70	80/100	80/100
Voids, %	13.2	2.2	4.9	11.0	12.0	13.0
Mix Composition—Reference Table No.	Semigap 15	Semigap 15	Cont. 15	Sand 15	Gap 12	Cont. 12
Viscosity $\eta_{0.05}$ Log Pa.S @ 25°C						
Surface: 0–5 mm Aged	6.64	6.53	6.05	6.56	6.18	5.86
2 mm Below Surface Aged	6.18	6.25	5.93	5.67	5.48	5.47
Relative Aging Index	2.88	1.91	1.32	7.76	5.01	2.45

*Tests were done at Council for Scientific & Industrial Research Laboratories, Pretoria, by A. Jurriaanse, A. Edler, C.M. MacCarron and M.M. Hattingh. Bitumen was extracted and tested by the same method as that used for the BKS survey.

due to the 24 hr curing period, because remolding the samples reduces the 24 hr reading and, in some cases, reduces it to the original state.

The author is not aware of any other studies that have investigated steric hardening of mixes.

2.5 Relationship Between Laboratory Aging Tests and Field Performance

As noted at the beginning of this chapter, the majority of researchers have considered aging of the binder rather than that of the mixture. Throughout sections 2.3 and 2.4 very little of the information presented includes field data. However, there are notable exceptions, such as Lee (1973) who presented data such as shown in Figures 7, 8, 9, and 10. Also, Von Quintas et al. (1988) included some data relating short-term field aging to laboratory aging (see Figure 13), and Petersen (1989a) compared TFAAT aging indices with those from field data reported by Vallerga and Halstead (1971). In each of these studies, asphalt was recovered from the field, consistency data obtained and compared with laboratory aged asphalt properties. The Von Quinatas, et al. (1988) study recovered asphalt from the laboratory aged mixture.

The results from several test roads will be reviewed below, where controlled projects provide excellent data. However, there are several cases where insufficient data are available to draw meaningful conclusions. Welborn (1979) has previously done an excellent job of reviewing most of this work.

2.5.1 California Studies

a) Zaca-Wigmore Test Roads

Two test roads were constructed in 1954 and 1955, with 10 test sections using different asphalts. Several researchers have evaluated the results.

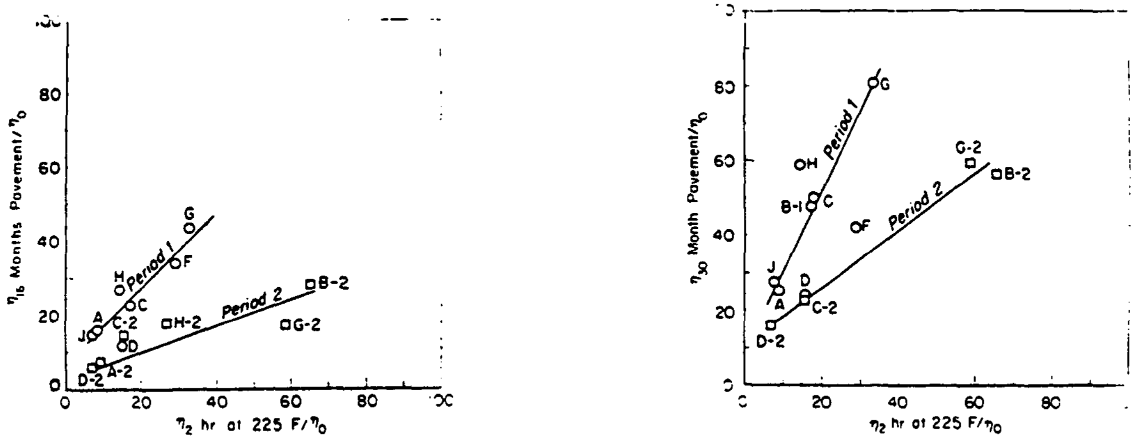
Simpson et al. (1959) compared the viscosity of asphalt recovered from various sections of the two test roads (referred to as period 1 and period 2) after 16 months and three years service with that of asphalt aged in the Shell microfilm test for two hours. Viscosity was determined at 77°F (25°C) with a sliding plate viscometer at a shear rate of 0.05 sec^{-1}. Figures 16 and 17 show selected results. Note the hyperbolic curves in Figure 17 which shows two sets of data representing the two construction periods where different procedures were used. Note that there is a definite correlation between field and laboratory data.

Zube and Skog (1969) published a final report on the test road. The final report included the following:

1) An excellent correlation was found for the hardening of asphalts during plant mixing and during the TFOT.
2) The rate of hardening under equivalent weathering and pavement conditions was influenced by the source of the asphalt. The hardening can be attributed mainly to the initial void content and rate of change in void contents during pavement life.
3) The amount of fatigue cracking appeared to be related to the consistency of the recovered asphalt as measured by penetration or viscosity. Other forms of cracking appeared to be related to the gain in shear susceptibility of the asphalt during service life. This was also indicated by loss of ductility.

This last conclusion regarding a relationship with ductility loss is also noted by Kandhal and Koehler (1984) from observations in Pennsylvania. These findings are discussed later.

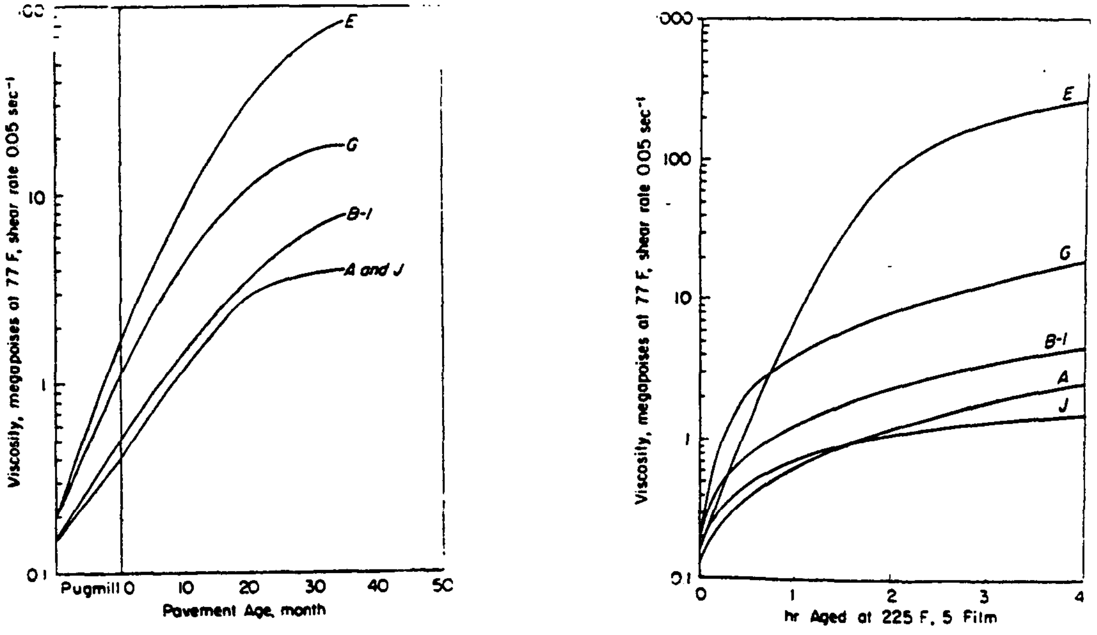
Davis and Peterson (1967) used inverse gas liquid chromatography (IGLC) to study Zaca-Wigmore asphalts. The data for phenol retention coefficient for laboratory aged asphalt correlates extremely well with performance rating for eight of the sections after 51 months service. Petersen (1984) explains that phenol retention time is a measure of the concentration of polar functional groups in the asphalt. Furthermore, the concentration of these polar groups is related to potential for



a) 16 Month Service

b) 30 Month Service

Figure 16. Pavement Aging Index Compared with Microfilm Aging Index (after Simpson et al., 1959)



a) Test Section Data

b) Microfilm Aging Test Data

Figure 17. Increase Viscosity with Time (after Simpson et al., 1959)

cracking or fracturing. Figure 18 shows the excellent correlation between phenol interaction time and performance rating.

b) Other California Studies

Following the Zaca-Wigmore test road, other test sections were installed in the 1960's. Kemp (1973) has described the relationship between laboratory durability tests and field hardening as measured by recovered viscosity at 77°F (25°C). Kemp and Predoehl (1981) indicate that these tests failed to provide consistent data due to many uncontrolled variables. The California Transportation Laboratory therefore pursued a new approach where most variables and constants would be controlled.

Kemp and Predoehl (1981) report on a study conducted between 1974 and 1980, where laboratory produced specimens 3.5 in. high by 4 in. in diameter were aged in four distinct climates in the field. Three asphalts of high, medium, and low temperature susceptibility were evaluated, with two aggregate types (absorptive and nonabsorptive) prepared at three void contents. Samples were exposed to the field conditions for 1, 2, and 4 yrs. The properties of the aged samples, and asphalt recovered from them, were compared with those obtained from laboratory tests.

Figure 19 shows data for the microviscosity at 77°F (25°C) for each asphalt recovered from the field aged samples. Note in particular the characteristic hyperbolic shape of the aging curves. The major conclusions drawn from this study were as follows:

1) High average temperature (thermal oxidation) is the most significant factor affecting the rate and amount of asphalt hardening in hot climates.
2) Void content is a contributing factor and its effect was similar among all asphalts.

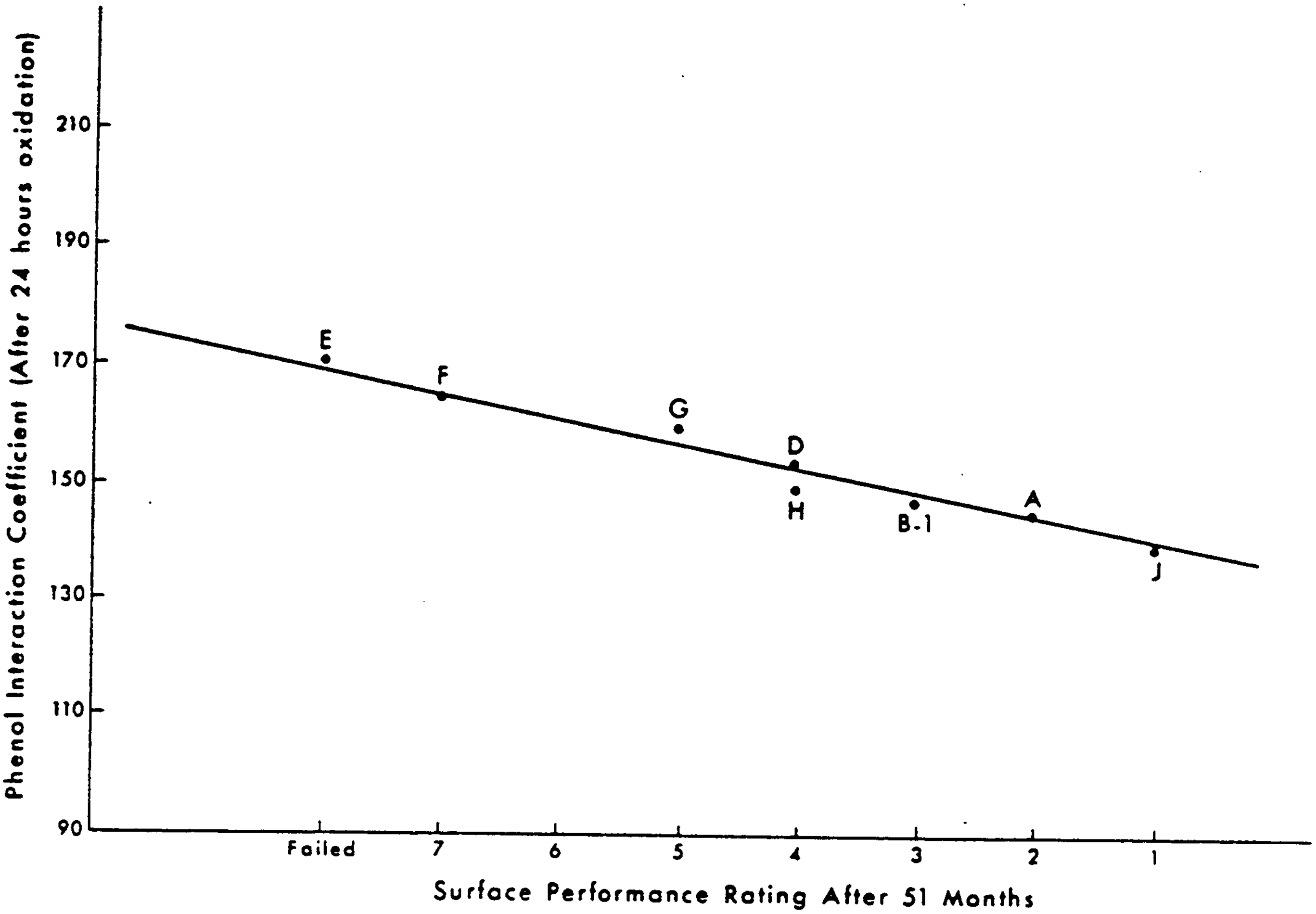
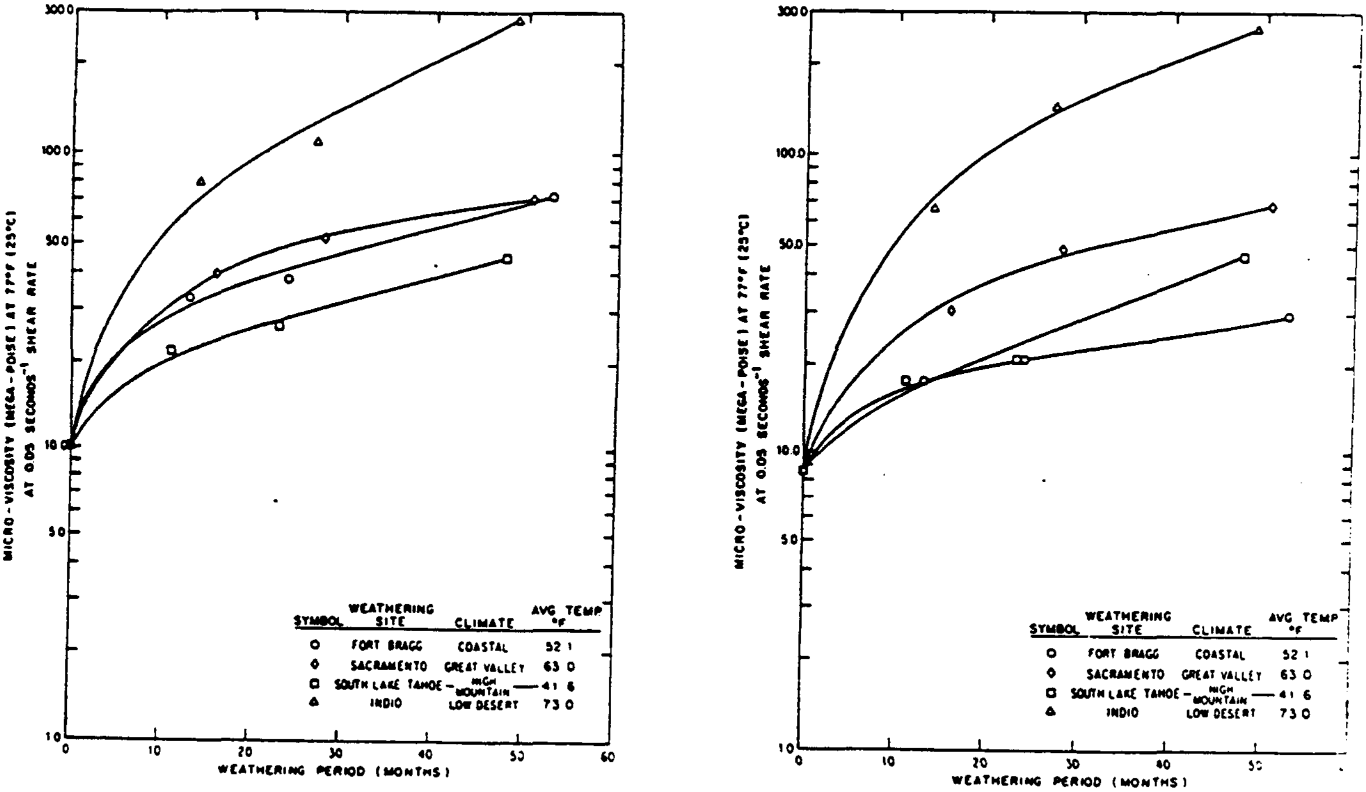
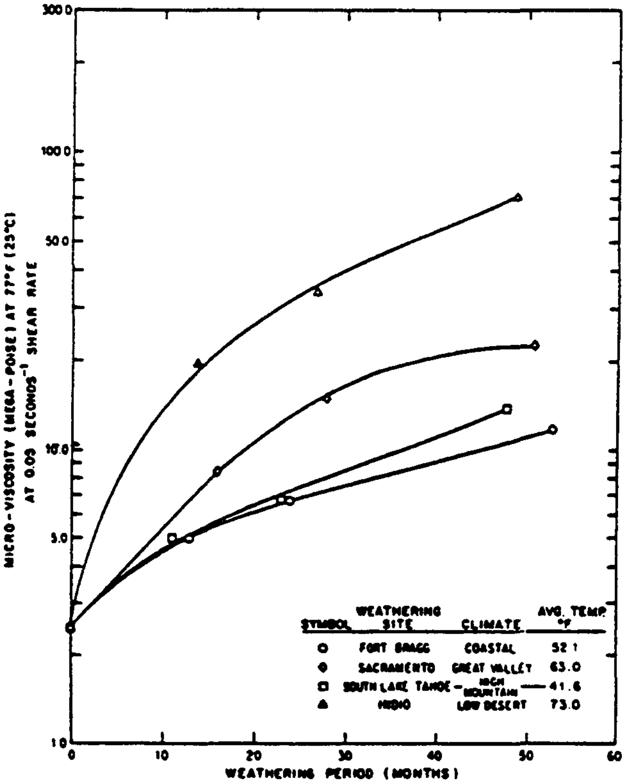


Figure 18. Relationship Between Phenol Interaction Coefficient and Pavement Surface Performance Rating (after Petersen, 1984)



a) Valley Asphalt

b) LA Basin Asphalt



c) Santa Maria Asphalt

Figure 19. Effect of Climate on Hardening (4 Climatic Sites) Combined Aggregates and Voids (after Kemp and Predoehl, 1981)

3) Aggregate absorption is a contributing factor and is more significant with more volatile asphalts.

4) The tilt oven durability test can be used to predict hot climate hardening of asphalt.

5) The following factors will improve durability:

    a) adherence to specified compaction requirements to reduce voids

    b) avoid use of absorptive aggregates

    c) use the softest grade of asphalt consistent with curing and stability constraints

    d) insulate the asphalt concrete surface with a cover such as a reflective chip seal in hot areas.

2.5.2 Michigan Test Road

Michigan State Highway Department completed construction of the test road in 1954. Six different asphalts were used in six test sections. Welborn (1979) has summarized the major findings of various researchers who have studied this test road over a period of over 25 years. In a study by Corbett and Merz (1975), the major conclusion was that, considering the age of the road and the current level of hardness of the binders, no distinction could be made among the binders used.

Corbett and Schweyer (1981) use data from the test road to illustrate some concepts regarding composition and rheology effects on age hardening of bitumen. They present Figure 20, which is presumably based on average data from the test road, and make the following points:

1) there is a two- to fourfold viscosity increase and a 30% penetration decrease by the time the pavement is a year old.

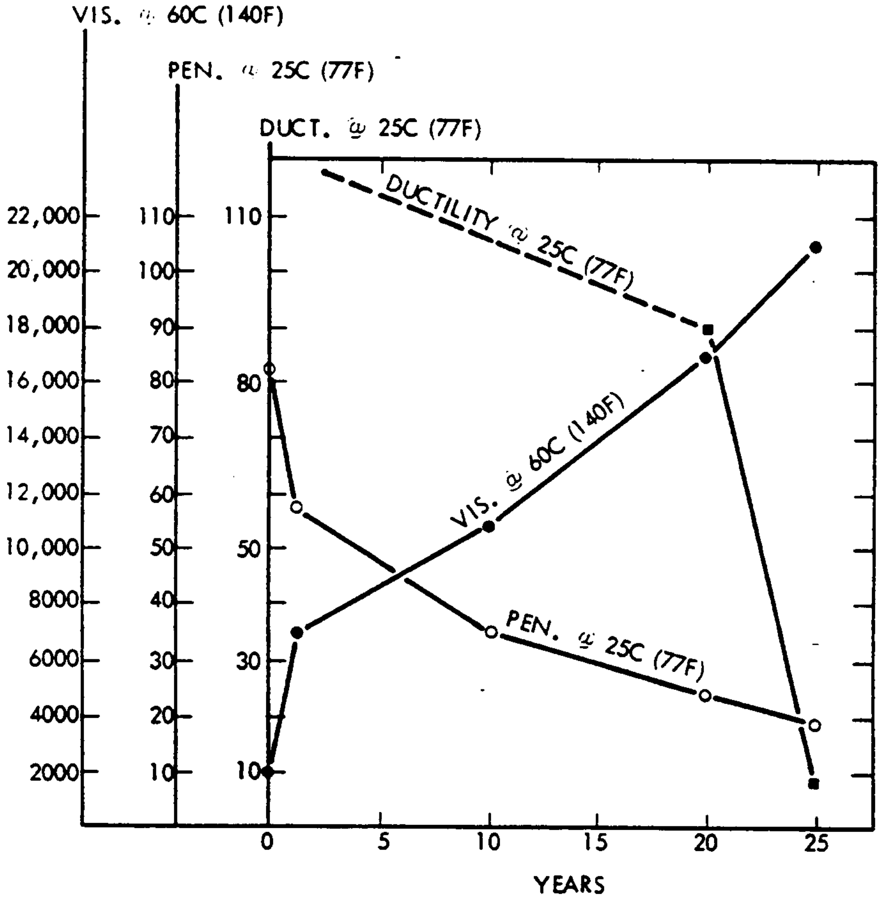


Figure 20. Age Hardening Involves Consistency Changes (after Corbett and Schweyer, 1981)

2) thereafter the viscosity level increases more slowly and typically reaches about 20,000 poises at 140°F (60°C) in about 25 years, based on an original AC-20 asphalt.

3) ductility at 77°F (25°C) is, in part, a flow test and it drops off rapidly in later life.

Goodrich (1985) has summarized data from the Michigan and other test roads. It is interesting to note that he found that the performance ratings of the Michigan test roads best correlated with:

1) percent ductility retained
2) softening point of original asphalt
3) viscosity aging ratio
   (18 yr viscosity at 140°F/original viscosity at 140°F)
4) percent asphaltenes
5) percent air voids after 52 months service.

The analysis presented by Goodrich (1985) does not clarify whether the percent asphaltenes is that measured on original asphalt, or on asphalt extracted from the test road.

2.5.3 Texas Test Roads

Gallaway (1959) reports on a study of surface treatment type pavements built in Texas in 1954. Eleven test roads were built and performance monitored. One of the conclusions given by the author is to restrict the oxidation susceptibility of asphalts in the specification requirements.

Benson (1976) addressed the relation between low temperature cracking of asphalt pavements in Texas, and asphalt hardening. Nine test sections were evaluated in detail and relationships between asphalt physical properties and performance established. Good correlations were found between the viscosity at 77°F (25°C), aging index, and air voids with performance. Laboratory mixture samples 17 in. in diameter by 2 in. deep were compacted using a Texas gyratory compactor. These laboratory samples were called "pizzas". Pizzas were aged in the field in two locations with different levels of solar radiation and precipitation. Control sets were stored in the laboratory. After three years the pizzas were cored, tested for resilient modulus, and penetration and viscosity tests, both at 77°F (25°C) were carried out on the recovered asphalt. The majority of pizzas were regular dense mix. However, a set of 15 was made using an "antihardening" additive. This was a combination of paraphenyclene diamine antiozonates and ultraviolet light inhibitors, at a treatment level of 1%. Benson also conducted the "Actinic Light Test" on asphalt samples. The test was conducted on 10 micron films with the following conditions:

1) 95°F (35°C)
2) 18 hours
3) 1000 microwatts/cm^2 of 3660 Angstrom wavelength radiation

These are identical to the conditions reported by Kemp and Predoehl (1981). The thin film oven test was also conducted. Asphalt properties after these two aging tests were compared with recovered asphalt properties. No significant correlation was established.

An excellent feature of Benson's study is the hardening models developed. These were developed for viscosity and penetration and take the form:

$$V = a\, t^{b} \tag{2.4}$$

and,

$$P = a + b \ln(t) \tag{2.5}$$

where, V = viscosity at 77°F
(megapoises, shear rate = 0.05 sec^{-1} in a sliding plate viscometer)
P = penetration at 77°F
t = time from laydown (months)
a+b = nondimensional coefficients derived from least squares regression analysis.

It should be noted that the curves for viscosity shown in Figure 21 are very similar to those obtained by Lee (1973), as presented earlier, and illustrated in Figures 7 through 10. However, Benson used an exponential model rather than a hyperbolic one such as used by Lee. Benson found better correlation coefficients with the penetration data and attributed the poorer correlation for the viscosity model to the complexity of the microviscosity test. Perhaps better correlation would have been achieved with a hyperbolic model. An example of the data is shown in Figure 21, and a collection of curves for several sites in Figure 22. The penetration model was chosen because of 14 nine-year-old pavements studied, this model fitted the data with a correlation coefficient greater than 0.95.

Benson notes that the key to applying this model is to predict the parameters "a" and "b", and that "a" is a measure of short-term hardening, since when t is equal to one month:

$$P = a + b \ln(1) = a \tag{2.6}$$

The parameter "b" relates to the curvature and represents long-term aging susceptibility. A predictive equation for "a" was developed from multiple regression analysis, yielding:

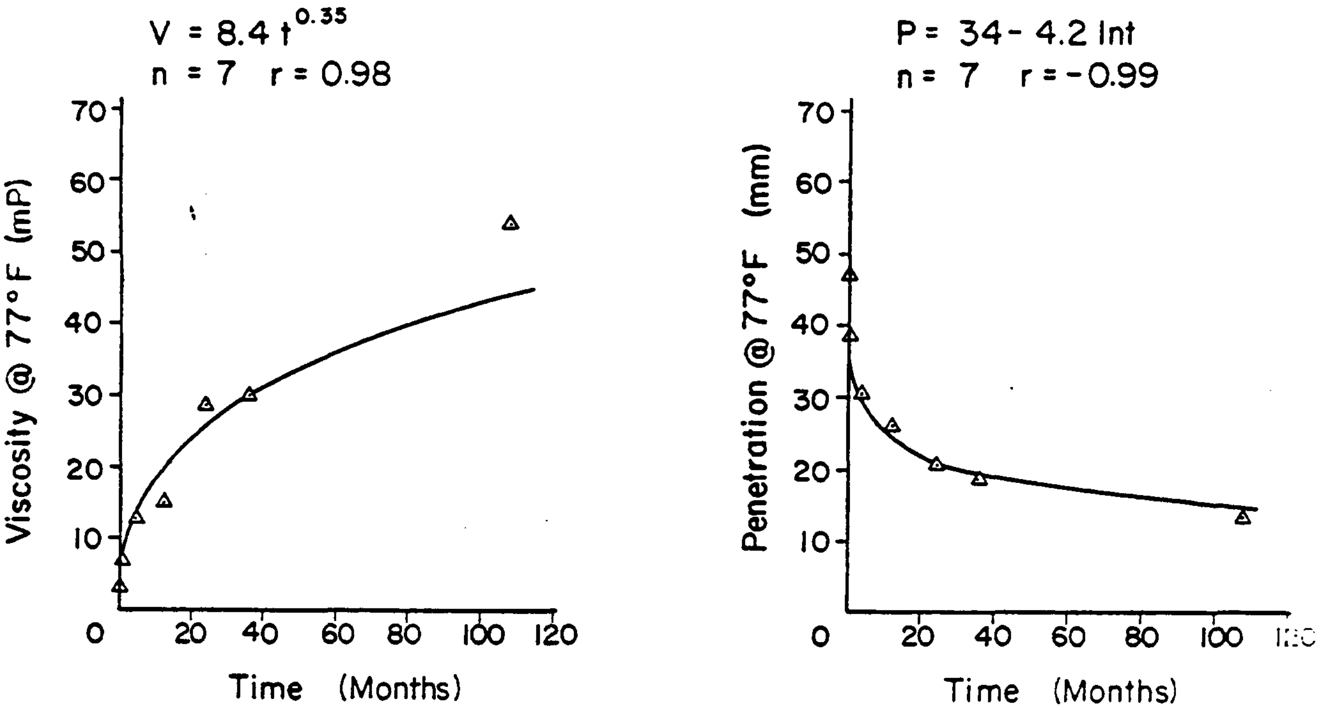
$$a = 0.52(ORP) - 2.0 \tag{2.7}$$

where, ORP = Original Penetration.

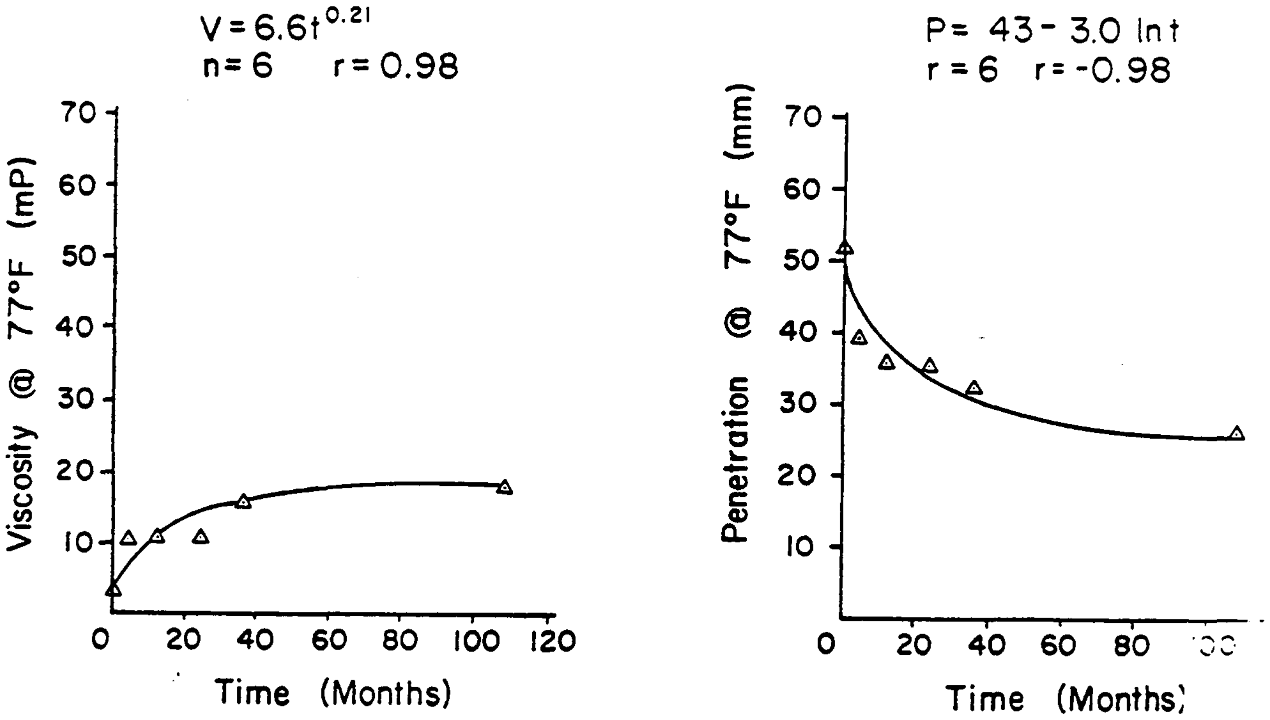
This results in "a" values of about half the original penetration. It was not possible to develop an equation for "b". However, Benson notes that this should be related to environmental factors and long-term chemical reactions.

Other major findings from Benson's study are as follows:

1) There was not a significant relationship between laboratory mix density and hardening susceptibility. However, field data did show a significant correlation between cracking, hardening, and void content.
2) The additives investigated were ineffective as hardening inhibitors.



a) Site 40



b) Site 41

Figure 21. Typical Hardening Curves for Two Different Sites (after Benson, 1976)

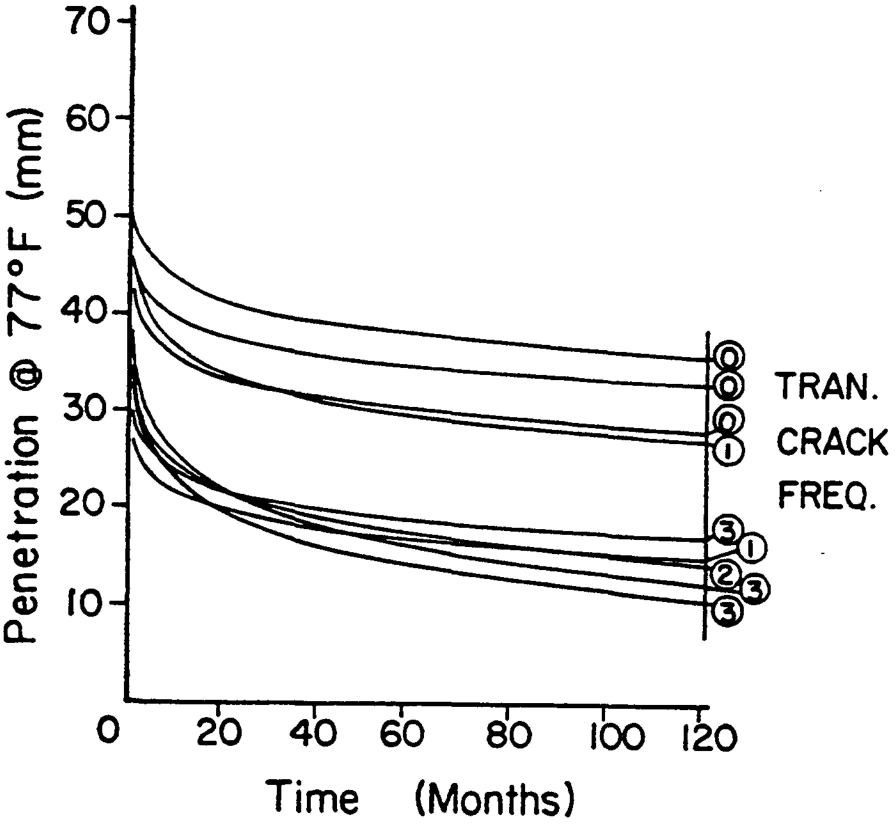


Figure 22. Penetration Hardening Curves for Nine Test Sites (after Benson, 1976)

3) The Actinic Light Test and Thin Film Oven Test were not reliable indicators of hardening susceptibility.

2.5.4 Pennsylvania Test Roads

Kandhal and Koehler (1984) reported major findings from test roads constructed in 1961, 1962, 1964, and 1976. Several test sections were constructed and their performance monitored. A very thorough set of tests was completed on original and recovered asphalts. Figure 23 shows viscosity data for recovered asphalt. The authors point out that these are hyperbolic curves, similar to those observed by Lee (1973). However, they note that low temperature ductility is an important factor, with pavements containing asphalt of low ductility showing greater tendency to crack.

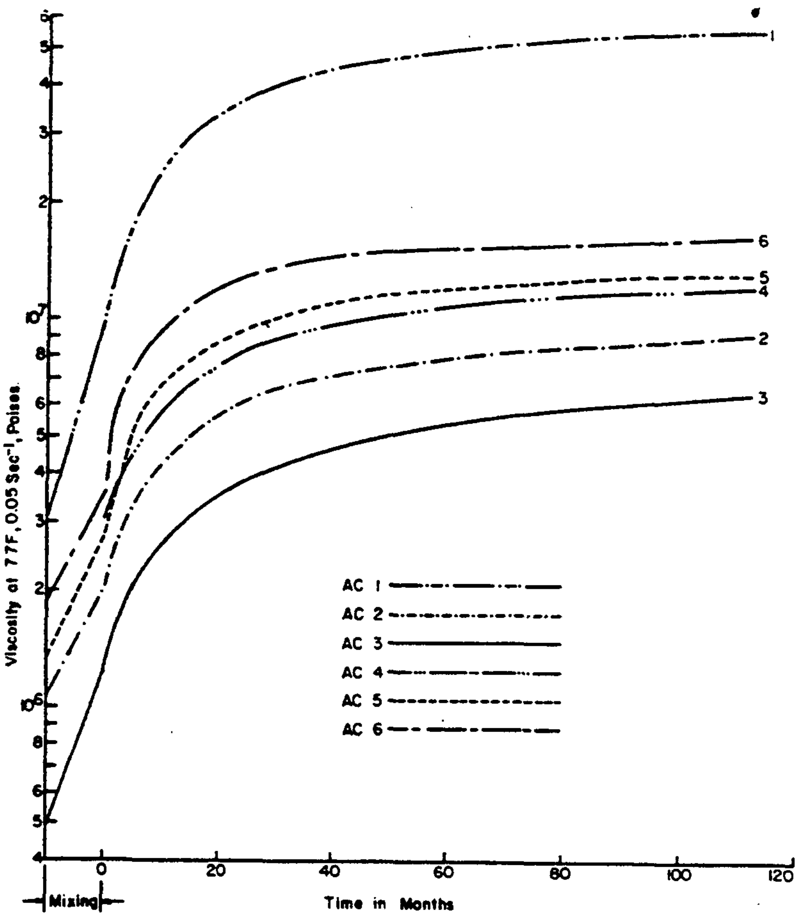
2.5.5 Iowa Study

As noted earlier, a significant study was conducted by Lee (1973) relating the properties of laboratory aged asphalts to those of recovered asphalts from nine test sections. Hyperbolic models were developed for field and laboratory data, as illustrated in Figures 7, 8, and 9. Field and laboratory data were related with "time-equivalency correlation curves" as shown in Figure 10.

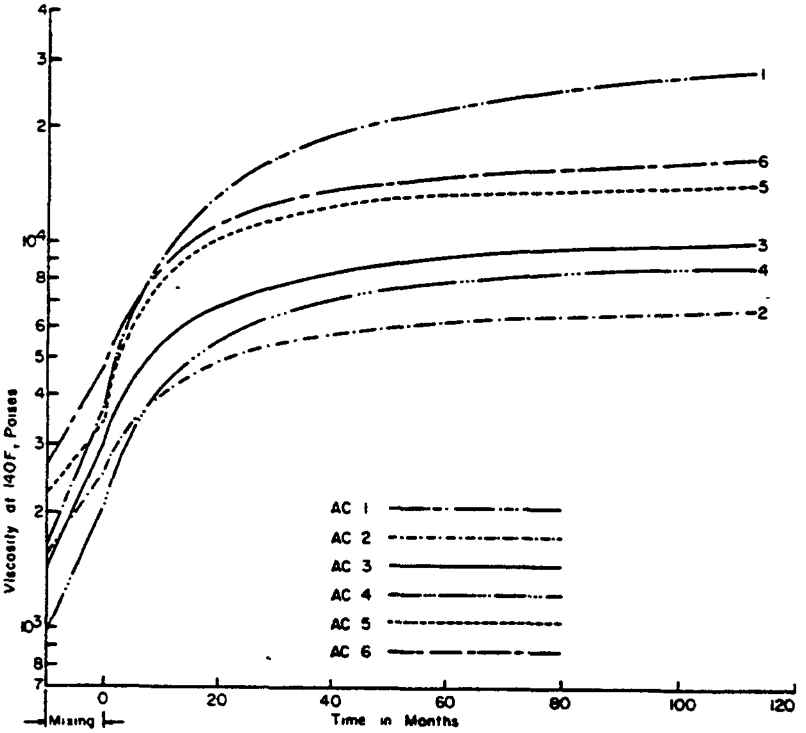
2.5.6 Oregon Study

Kim et al. (1986) attempted long-term aging of asphalt mixtures using pressure oxidation. It was shown that this approach did produce significant aging as shown in Figures 14 and 15.

Thenoux et al. (1988a) in a companion study attempted to relate asphalt properties to field performance. The properties included the common physical data as well as the Corbett-Swarbrick fractionation data. The attempt to develop relationships was frustrated by the lack of control of the field test sections. Eight sites, dispersed around the state were selected. These were nominally five to 10 years old, and asphalt had been stored in the



a) Viscosity at 77°F



b) Viscosity at 140°F

Figure 23. Viscosity versus Time Relationships, 1964 Pavements (after Kandhal and Koehler, 1984

state materials laboratory for each project. Since only the initial condition and end condition (i.e. at the time of the study) were known, no rate of change information could be established.

The Oregon study would have been more valuable had more recovered asphalt properties been obtained, and if historical pavement condition data had been available.

2.6 Relationship Between Chemical Composition and Field Performance

Many of the studies described earlier in this chapter have involved determination of the composition of an asphalt, as indicated in Table 1. Changes in composition were an indication of the extent of aging in a laboratory or field aged sample. Some studies (e.g. Vallerga and Halstead, 1971) included a comparison of a compositional parameter with the observed performance of a field project. This section presents a review of several studies that specifically address the question of whether there is a relationship between composition and field performance. An excellent paper by Goodrich et al. (1986) addresses this topic and forms the basis for what is presented below.

2.6.1 Tests for Asphalt Composition

Goodrich et al. (1986) indicated that the procedures commonly used fall into six categories as follows:

1) Fractionation by precipitation: solvent precipitation, chemical precipitation.
2) Fractionation by distillation: vacuum distillation, thermogravimetric analysis.
3) Chromatographic separation: gas chromatography, inverse gas-liquid chromatography, liquid chromatography (adsorption, ion exchange, coordination, thin layer, size exclusion).
4) Chemical analysis: spectrophotometric techniques (infrared ultraviolet, nuclear magnetic resource, X-ray fluorescence, emission, neutron activation), titrimetric and gravimetric techniques, elemental analysis.

5) Molecular weight analysis by mass spectrometry, vapor pressure osmometry, and size exclusion chromatography.

6) Indirect compositional analysis by internal dispersion stability tests.

Of these tests, fractional separation has been used most. Four types of procedure are commonly used:

1) Chemical Precipitation, e.g. the Rostler-Sternberg approach, standardized in ASTM D2006, but discontinued in 1976.

2) Solvent Fractionation, e.g. the Traxler-Schweyer approach.

3) Adsorption-Desorption Chromatography, e.g. the Clay-gel procedure, standardized in ASTM D2007, and the Corbett-Swarbrick procedure, standardized in ASTM D4124.

4) Size Exclusion Chromatography, e.g. the gel permeation chromatographic (GPC) technique standardized in ASTM D3593.

A brief description of each of these procedures is given by Goodrich et al. (1986). The first three types of procedure involve precipitating an asphaltene fraction from a solution of an asphalt in a solvent. It should be noted that the Corbett-Swarbrick approach uses a different solvent to the other methods, and that the "asphaltenes" fraction obtained by this approach will be different to that obtained by other approaches. This reinforces the point that fractional separation does not separate the asphalt into unique chemical compounds, but rather into groups of compounds of a similar type.

The GPC separation approach has been used by a number of researchers during the 1970's and 1980's. In particular, a group at Montana State University has used this technique extensively (Jennings, 1985). The asphalt is separated according to the size of molecular associations, and indeed, Jennings (1985) has likened this technique to a sieve size analysis for aggregates.

2.6.2 Value of Compositional Analyses

Goodrich et al. (1986) assessed the value of compositional analyses by fractional separation techniques. They conclude that such analyses provide a method for following changes in an asphalt, whether during laboratory studies, refinery processing, field mixing or field aging, or recycling. They also conclude that compositional parameters obtained have not correlated well with field performance, nor have ratios based on the fractions. However, they indicate that physical tests have correlated well. Goodrich et al. also emphasize a point that is frequently overlooked when considering the durability of asphalt pavements; namely, that construction, mix design, and climatic variables have an overriding influence.

The asphalt content, film thickness, air voids, and permeability are very important. As noted in an earlier section, Kumar and Goetz (1977) showed that film thickness and permeability were the main factors controlling aging of open-graded mixtures. However, for dense mixtures, permeability was the major factor. The California durability study (Kemp and Predoehl, 1981) is a clear indicator that climatic effects dominate aging of mixtures in the field. The data in Figure 19 represents three distinctly different asphalts and a wide range of air void contents but these have much less effect on aging than the climate.

With the conclusions of Goodrich et al. (1986) in mind, it should be noted that if the construction and climatic variables were similar, compositional parameters may be an indicator of potential performance among projects using different asphalts. In a thorough review of the relation of asphalt chemistry to physical properties and specifications, Halstead (1985) notes that although the value of compositional analysis has been questioned, work in South Africa reported by Jamieson and Hattingh (1970) showed reasonable correlation between compositional parameters and road performance. Halstead emphasizes that such parameters are one indication of performance, but other factors must be considered. Clearly good mix design and construction are required. Goodrich et al. (1986) note that good correlations were obtained with Australian test roads (see Dickinson, 1980), which involved some surface treatments as well as dense mixtures.

2.6.3 Significant Compositional Parameters

A simple "model" of asphalt is that it consists of a colloidal dispersion of "asphaltenes" in a dispersion medium of "maltenes" (Barth, 1962). The asphaltenes have the highest polarity and tendency to interact and associate. They are also insoluble in a nonpolar solvent such as pentane, hexane or heptane. The maltenes are soluble in such solvents. This broad distinction enables fractionation of asphalts. Figure 24 shows schematics of two of the procedure noted earlier. It may be seen that the maltenes are further separated. The four fractions obtained with the Corbett-Swarbrick procedure or the five from the Rostler-Sternberg procedure form the basis for most comparisons of composition of asphalt with performance. Three parameters derived from the fractions obtained from these procedures are described below.

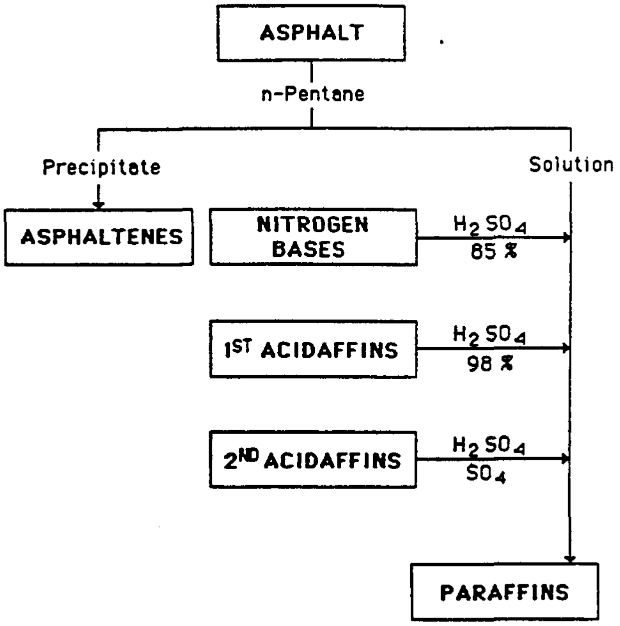
a) Rostler Durability Ratio (RDR)

The fractions obtained from the Rostler-Sternberg procedure may be used to develop a Rostler durability ratio as follows:

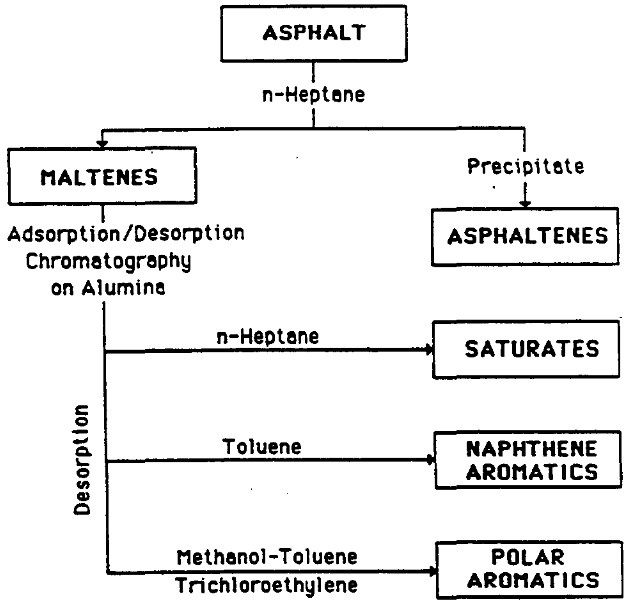
$$RDR = \frac{N + A_1}{P + A_2} \tag{2.8}$$

where N, A_1, P, A_2 are the nitrogen bases, first acidaffins, paraffins, and second acidaffins, respectively.

Rostler and White (1962) presented extensive data relating this ratio to percent weight loss from pellet abrasion tests on Ottawa Sand mixtures before and after aging. The pellet abrasion test was an adaptation of a shot abrasion test described by Hveem et al. (1963). Aging was accomplished in an oven for 7 days at 140°F (60°C). The RDR is the ratio of the most reactive fractions to the least reactive.



a) Chemical Precipitation by Rostler-Sternberg, ASTM D2006



b) Adsorption/Desorption Chromatography by Corbett-Swarbrick, ASTM D4124

Figure 24. Fractionation Procedures for Asphalt Cement

Rostler and White indicated that asphalts with RDR less than 1.14 had excellent abrasion resistance.

Vallerga and Halstead (1971) presented an overview of an extensive BPR study, which sampled more than 300 asphalts from projects 11 to 13 years old. This included Rostler-Sternberg analyses, and it was concluded that for pavements with less than 2% air voids there was a direct relationship between the RDR and aging measured by the viscosity of recovered asphalt. For higher air voids the RDR was overshadowed by other effects, but the ratio for minimum hardening appears to be in the range 1.0 to 1.4. It was significant that most of the pavements containing asphalts originally found to have high RDR values did not survive to be 11 to 13 years old.

Jamieson and Hattingh (1970) conducted an extensive study involving field projects using 18 bitumens in "premix" and evaluation of asphalts which included the Rostler-Sternberg approach. They concluded that there was good correlation between the RDR and road performance and that appropriate limits for coastal South Africa would be 1.0 to 1.7, which is a similar range to that suggested by Vallerga and Halstead (1971).

Goodrich et al. (1986) point out that the RDR does not consider the asphaltene content and that it has limited correlation with field data (overlooking the findings of Jamieson and Hattingh, 1970). They note that the Gotolski ratio does consider the asphaltenes.

b) Gotolski Ratio (GR)

Gotolski et al. (1965) proposed this as an alternative to the RDR:

$$GR = \frac{N + A_1 + A_2}{P + A} \tag{2.9}$$

where N, A_1, P and A_2 are as before and A is the percent asphaltenes.

This parameter was used by Jamieson and Hattingh (1970) and by Anderson and Dukatz (1980). The former found a range of 0.8 to 3.4 for the GR for 18 bitumens investigated and suggested limits of 1.3 to 2.6 to ensure satisfactory performance. The latter authors addressed the question of whether asphalts had changed over a 30-year period, 1950-80. They concluded that there had been statistically significant changes and that the Rostler and Gotolski parameters had increased during that time. They also indicate that high and low Rostler parameters indicate potentially poor asphalt performance, which is in agreement with Vallerga and Halstead (1971), and that the RDR is most closely associated with temperature susceptibility. Anderson and Dukatz also indicate that a high GR is an indicator of poor performance and that this ratio is more closely related to aging effects.

In discussing the Anderson and Dukatz (1980) paper, Puzinauskas states that there is no substantiation of the claim that the RDR is more closely associated with temperature susceptibility of an asphalt and the GR relates more closely to aging. Furthermore, he states that in his opinion the RDR and GR are associated with a variable reactivity of asphalts to sulfuric acid and nothing more. These comments appear to ignore the work reported by Vallerga and Halstead (1971) mentioned earlier in this section.

In a subsequent extension of the work at Pennsylvania State University, Anderson et al. (1983) concluded that, "Except for temperature susceptibility, there have been no long-term, significant changes in asphalt properties that can be identified as significant by the analytical techniques used in the project." This study included additional data to that reported in 1980 by Anderson and Dukatz. Only brief mention of the RDR is made to the effect that it "remained relatively unchanged over the period 1950-81," and there is no mention of the GR. The authors obviously reconsidered the conclusions made in 1980, and there is a clear indication in the discussion to the 1983 paper that the Rostler analysis was used because it was the only analysis used in the data available from 1950 and 1960.

c) Gaestel's Colloidal Instability Index (IC)

Brûlé et al. (1986) summarized the Gaestel Colloidal Instability Index (IC) which was defined by Gaestel et al. (1971) as follows:

$$IC = \frac{\text{Asphaltenes + Flocculants (Saturated Oils)}}{\text{Peptizers (Resins) + Solvents (Aromatic Oils)}} \tag{2.10}$$

Brûlé et al. present the following explanation of IC:

> "The higher the ratio, the more will the asphalt cement be a gel type and the lower will be its colloidal stability. Gaestel also notes that all the properties of the binder (softening point, ductility, embrittlement temperature, thermal susceptibility, elastic recovery, shearing susceptibility, etc.) vary significantly with the colloidal instability index and hence with composition."

Brûlé et al. did not develop any data for IC, because fractionation would be required, and a faster GPC technique was preferred.

Tuffour et al. (1989) presented excellent data relating IC to changes in asphalt properties due to aging. They express the index as follows:

$$IC = \frac{\text{Asphaltenes + Saturates}}{\text{Naphthene Aromatics + Polar Aromatics}} \tag{2.11}$$

Tuffour et al. carried out clay-gel compositional analyses on two sets of asphalt samples: one set recovered from cores from roads showing signs of durability and aging problems, the other set were laboratory aged using the TFOT at three temperature levels (163, 140, and 120°C) and various periods of aging. Figure 25 shows the data developed for absolute viscosity (60°C) versus IC. The authors developed regression equations which confirmed the strong correlation apparent from the figure. They conclude that high values of IC are associated with instability and that the data suggest there is a unique relationship between IC and viscosity.

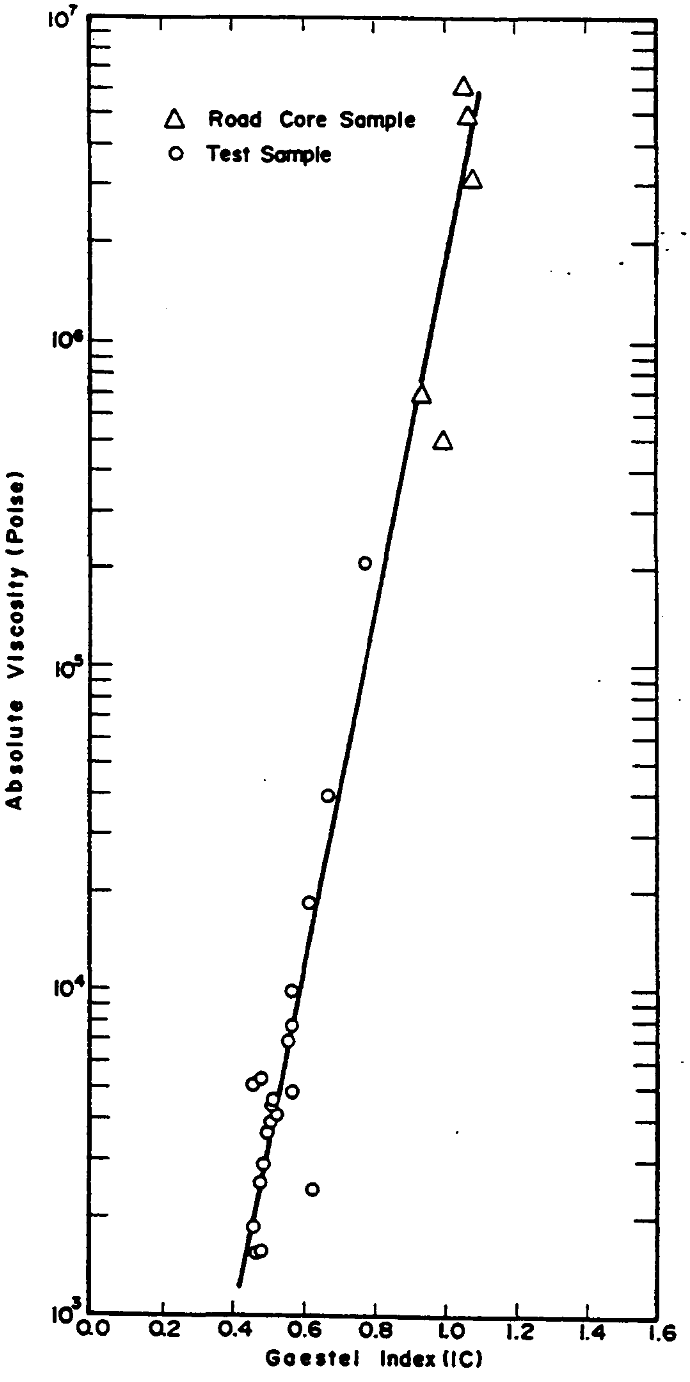


Figure 25. Relationship between Absolute Viscosity and Gaestel Index (after Tuffour et al., 1989)

2.6.4 Other Pertinent Studies

Button et al. (1984) addressed the influence of temperature susceptibility on pavement construction and performance. They conducted an extensive review of previous work, and a significant laboratory test program, which included some fractionation tests. They make a number of significant conclusions, including the following:

1) Asphalts containing less than 10% asphaltenes, particularly the softer grades, appear to have a greater probability of producing slow setting paving mixtures. However, an asphalt will manifest itself as slow setting only if the aggregate type and/or gradation is such that a critical paving mixture is produced (even through the aggregate may meet specifications) or possibly if densification of the pavement is inadequate.
2) There is no correlation between asphalt temperature susceptibility and asphaltene content. There is no relationship between asphalt temperature susceptibility and other chemical constituents of asphalts as determined by the Rostler-Sternberg analysis or the Rostler parameter.

More recently, Thenoux et al. (1988b) published data, from Corbett-Swarbrick analyses of field and laboratory aged asphalts, which showed reasonable correlation between various temperature susceptibility parameters and the compositional parameters, particularly asphaltene content. This conflict with Button et al. (1984) may be due to the difference in the two fractionation procedures, and reflects a general trend in the literature of sporadic correlations when the Rostler-Sternberg parameters are considered but generally good correlations when the Corbett-Swarbrick or clay-gel parameters are considered. Thenoux et al. (1988a) also noted that since asphalt temperature susceptibility has a significant influence on field performance, their correlation of composition with temperature susceptibility implies that composition relates to performance.

The work of Jennings and his associates at Montana State University has special significance, since it has led to widespread adoption of the GPC technique during the

1980's. Jennings (1985) reviewed the technique and stated that while GPC is not the answer to everything, it can be used to predict the cracking potential of roadways. Jennings et al. (1988) present data to support this prediction potential. They also included Corbett-Swarbrick analyses and noted that test sections that were rutting most severely had lower asphaltene contents and that resistance to cracking is associated with relatively lower concentration of naphthene aromatics. The first observation agrees with the first conclusion given above for Button et al.

3.0 PROMISING AGING AND TEST METHODS

The previous chapter outlined a variety of laboratory aging methods which have been used by various researchers. This chapter will discuss the most promising methods for aging asphalt-aggregate mixtures. It will also include test methods used to establish the effects of the aging procedure. This may include tests used to evaluate the extent of aging in field test sections as well as laboratory aged mixtures.

3.1 Promising Aging Methods

The literature review clearly shows that there are two components of aging to consider, viz:

1) short term
2) long term

The short-term component occurs during the construction phase, while the mix is hot. This is probably caused by volatilization although there may be some steric hardening and oxidation involved and volatilization of oxidation products. The long-term component occurs while the mixture is in place. A time period of about 10 years is a reasonable period of interest for this component. This long-term aging is predominantly caused by oxidation, although there may be some steric hardening involved and volatilization of oxidation products. Actinic light may serve to accelerate the aging at the surface of a pavement.

3.1.1 Short-Term Aging Methods

Candidate methods should simulate conditions during mixing, storage, and transportation. Therefore, conditions similar to the TFOT or RTFOT are appropriate. The mixture should be loose and possibly agitated continuously or periodically at a temperature of about 275°F (135°C) to simulate the precompaction phase of construction. Possibly the mix should be compacted prior to cooling to simulate field conditions.

Only two short-term methods emerged in the literature. The first is the oven aging method used by Von Quintas et al. (1988). This method is described briefly below. Other methods involved Ottawa Sand mixtures. Such mixtures are considered inappropriate for this study (e.g., Pauls and Welborn (1952), and Kemp and Predoehl (1981)) since they are not representative of typical mixtures with regard to internal voids and permeability. Two additional heating approaches are worthy of consideration, viz:

1) extended mixing
2) microwave heating

a) Oven Heating

The Von Quintas et al. (1988) procedure consisted of heating the mixture sample at 275°F (135°C) for 0, 8, 16, 25 and 36 hours, prior to compaction and cooling. The effect of temperature was also studied before adopting 275°F.

b) Extended Mixing

Since mixing is an inevitable part of the aging process, it is logical to take advantage of extending this in an aging method. An added advantage of this is that since the mix is continuously agitated, all the mix is given equal opportunity to age. It is probably best to combine this approach with oven heating.

c) Microwave Heating

Microwave ovens may provide a controlled method of extended heating. A thorough evaluation of this technique is required, since there is little knowledge regarding its use. Al-Ohaly and Terrel (1988) provide some insight regarding the mechanism of microwave effects, and this will provide a starting point for subsequent work.

3.1.2 Long-Term Aging

Candidate methods should simulated conditions in the field and emphasize oxidation. Therefore, the mixture must be compacted and aged at a moderate temperature, probably no more than 140°F (60°C). Several methods emerged from literature, viz:

1) pressure oxidation treatment
2) extended oven aging
3) infrared/ultraviolet treatment

An additional approach could be used, which would attempt to pass osygen at low temperature through a sample in a triaxial cell. This configuration has promise for evaluating the effects of other fluids, such as air, water, or water vapor.

a) Pressure Oxidation Treatment

Kim et al. (1986) applied a pressure of 100 psi at 140°F (60°C) within a pressure-safe vessel for periods of 0, 1, 2, 3, and 5 days. The complete procedure is given in Appendix A. The pressure level used should probably be raised to 300 psi, since a thorough evaluation of asphalt aging by Lee (1973) suggests that such a pressure is necessary.

Petersen (1989a and 1989b) proposes that *molecular structuring and steric* hardening have a major effect on levels of field aging. He cites data such as are shown in Figures 5, 8, 9, 19, and 23, to illustrate an aging model. This model suggests that aging progresses rapidly during the early life of a pavement (the first two to three years), and then slows considerably due to the progress of molecular structuring and steric hardening. The extent to which this occurs is dependent on the properties of the asphalt, the climate, and the mixture properties (air voids). Most of the data for field aging shows that the asphalt approaches an ultimate level of consistency, typically expressed as a viscosity. However, some field data (e.g. Figure 19) and limited laboratory data (Figures 8 and 9) suggest that the ultimate value will not be reached for some time. This may be explained by the model, in terms of lower level of structuring and steric hardening which exists due to the type of "quenching" used to prepare the samples prior to testing, and/or the temperature levels prevailing. Petersen (1989b) suggests that higher temperatures reduce the ability of an asphalt to form molecular associations, and therefore aging can continue for a longer period and/or to a higher level than at lower temperatures.

The above model has significant implications in the development of an aging method for asphalt aggregate mixtures. Figure 26 has been developed to illustrate the possible interaction of temperature and pressure oxidation with regard to aging. This illustrates the need to include temperature as a variable in evaluation of aging methods, and it is proposed to include 77°F (25°C) in the preliminary laboratory studies for this project. Further, Figure 26 illustrates that to use a temperature which is too high may lead to unrealistic levels of aging when compared to field aging. This is significant when evaluating oven aging techniques which rely on high temperatures.

Kumar and Goetz (1977) passed air through samples at 140°F (60°C). This approach using air is not regarded as suitable, since the extent of aging achieved appeared to be quite low. However, a similar configuration, using oxygen under low pressures, may be appropriate, particularly when considering cycles of aging and moisture treatment, as discussed below.

b) Extended Oven Aging

Von Quintas et al. (1988) used the following approach:

1) heat six compacted specimens in an oven at 140°F (60°C) for 2 days, then remove three.
2) increase the temperature to 225°F (107°C) and age the remaining three specimens for 5 days.

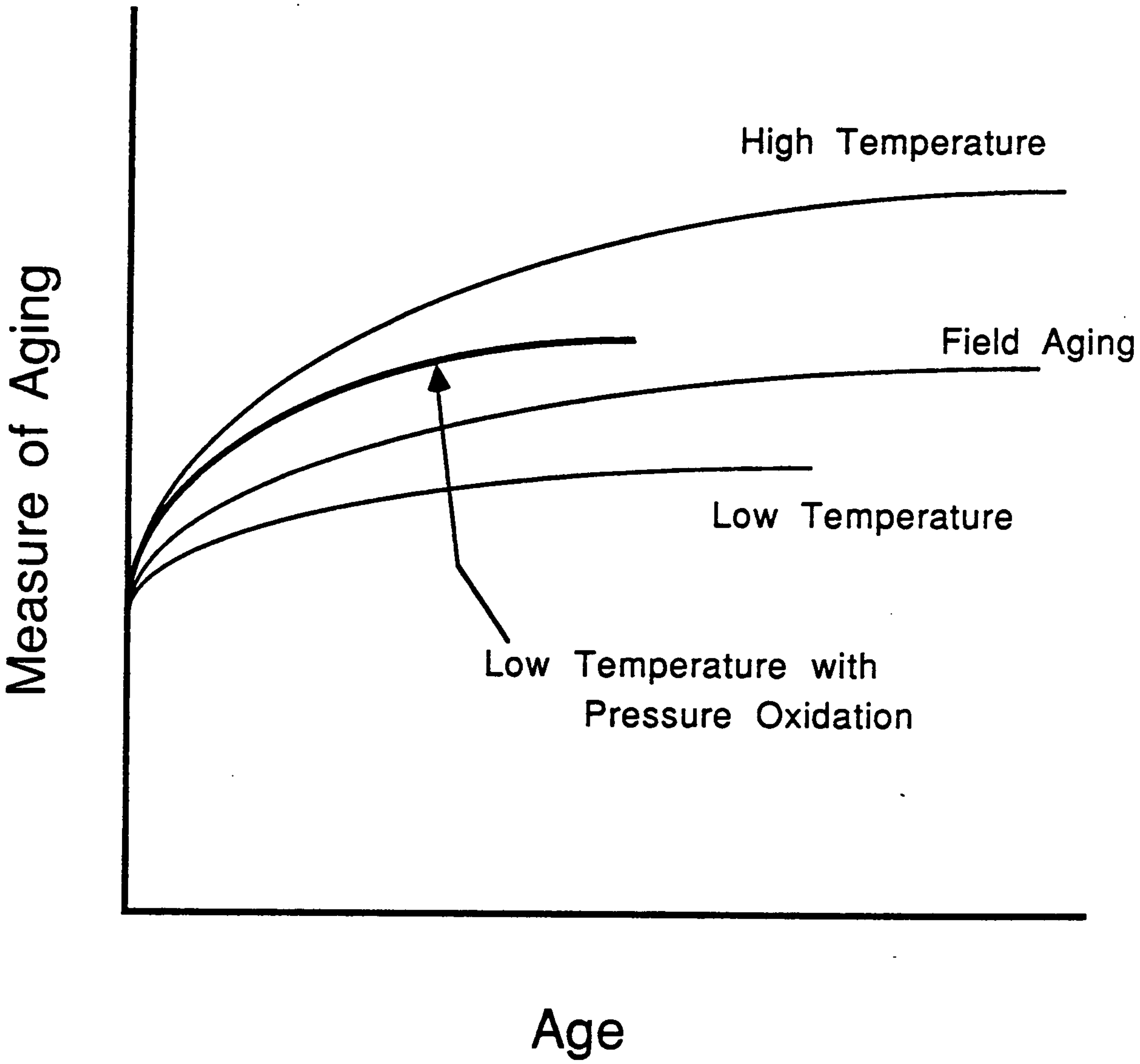


Figure 26. Possible Interactive Effects of Temperature and Pressure Oxidation on Aging

The specimens were not supported throughout this process and no collapse of specimens was detected. Hugo and Kennedy (1985) heated specimens in an oven at 212°F (100°C) for 0, 4 and 7 days. Dry and moist atmospheres were used. As suggested above, these oven aging methods are operating at higher temperatures than desirable to achieve aging similar to field aging.

Tia et al. (1988) used forced draft and conventional oven aging at 140°F (60°C). The forced draft oven showed promise for future development but clearly the effect of temperature level on the integrity of the compacted samples needs to be evaluated. This author is concerned that the internal structure of a sample may be disrupted at temperatures such as 140°C. In extreme cases the samples may "slump".

c) Ultraviolet/Infrared Treatment

Hugo and Kennedy (1985) utilized "ultraviolet" treatment. Benson (1976) used a level of "actinic radiation" apparently identical to that used in the California studies described by Kemp and Predoehl (1981). California weathering tests have been described previously by Hveem et al. (1963) as infrared treatment.

Clearly there is a need to define the difference between ultraviolet and infrared radiation and its likely effects. To this end, Appendix B has been prepared. This shows that the wavelength of light used by Benson (1976) and Kemp and Predoehl (1981) is in the ultraviolet range. In their procedure, samples were subjected to actinic light of 1000 MW/cm^2 of 3660 Angstroms (i.e. 366 nanometers) for 18 hrs at 95°F (35°C).

Vallerga et al. (1957) utilized both infrared and ultraviolet and found that more aging occurred with ultraviolet light. However, their explanation of the rationale for using infrared is in keeping with the information in Appendix B, namely that infrared causes molecular vibration of materials. This results in a radiant heating effect. It is most likely that aging of asphalt exposed to infrared is the same as if it was heated directly (Petersen, 1989b). However, ultraviolet light is recognized as having potential to promote chemical reactions, and in the case of asphalts, will do so, but only to a depth of about 3 microns (Traxler, 1963).

Tia et al. showed significant aging of compacted samples in an ultraviolet chamber, and recommended that it should be used together with forced draft oven aging, although they did not age samples with such a combination.

Ultraviolet light will be considered further in this study, possibly in combination with one or more of the other aging methods. There is potential for using an Atlas Weatherometer or similar device for this purpose.

d) Triaxial Cell Conditioning

A similar approach to that used by Kumar and Goetz (1977) but with modifications has great promise. It should be possible to "force" a fluid through a mixture specimen while it is contained in a triaxial cell. Thus the cell would act as the conditioning vessel, but would also enable measurements to be made on the specimen, such as permeability and modulus determinations. This idea is described more fully by Terrel (1989).

As noted in the Introduction, aged asphalt materials may be more moisture susceptible than unaged materials. Since asphalt surfaces are subject to alternate cycles of dry and wet conditions in the field, it is important to establish if cycles of aging and moisture treatments are more damaging than the additive effects of each treatment done separately. A method of alternatively cycling oxidation with moisture treatment will be developed. Such a procedure could be done quite easily in a modified triaxial test cell. This configuration could also be used to determine the modulus of the test specimen without its removal from the cell. The permeability can also be measured in a triaxial cell.

3.2 Promising Test Methods

A procedure using oxygen as the fluid will be compared with one using air. The effects of temperature should also be evaluated.

The Literature Review revealed that a wide variety of test methods have been used to evaluate the effects of aging of asphalt mixtures. These fall into two categories:

1) Tests on aged mixture samples
2) Tests on asphalt recovered from aged mixture samples.

3.2.1 Tests on Aged Mixture Samples

Table 1 includes the following test procedures used to evaluate aging:

1) Resilient Modulus, M_R (e.g. Kemp and Predoehl, 1985; Kim et al., 1986; Von Quintas et al., 1988; and Tia et al., 1988).
2) Fatigue (e.g. Kim et al., 1986).
3) Creep Test (e.g. Kumar and Goetz, 1977).
4) Indirect Tensile Test (strain at break) (e.g. Von Quintas et al., 1988; and, Tia et al., 1988).

In addition, a dynamic or complex modulus has promise, for reasons outlined below.

a) Resilient Modulus (M_R)

Kemp and Predoehl (1985), Kim et al. (1986), Von Quintas et al. (1988) and Tia et al. (1988) used the diametral approach to determine M_R. Kim et al. and Tia et al. used the approach described in ASTM D-4123, and presumably this was also used by Kemp and Predoehl, and Von Quintas et al. The general trend observed by all these researchers is an increase of M_R as the age of samples increases. This test can be performed quite quickly and is nondestructive.

b) Fatigue

Kim et al. conducted fatigue tests on aged samples and noted that fatigue life increased as aging progressed. As noted in the previous chapter, this is not surprising since the modulus of samples increased, and the applied stress was kept the same. Fatigue tests are potentially very time consuming and destroy the samples used.

c) Creep

Kumar and Goetz (1977) conducted creep tests at intermediate time periods as their aging test progressed. In order to establish a creep curve at each increment of aging, a test period of about 25 minutes was necesary. This is longer than the time required to conduct a modulus test.

d) Indirect Tensile Test

This test was utilized by Tia et al. (1988) and Von Quintus et al. (1988). Von Quintas et al. (1988) utilized this test and recommended the strain at break as the parameter to evaluate the extent of aging rather than the tensile strength. As a sample ages and its modulus increases, so does its strength, in the same way as its fatigue life at a particular stress level does. However, the strain at break decreases, since the sample becomes brittle as it ages. Strain at break is therefore a good representation of the deterioration in flexibility as a sample ages. Although this test is destructive, it is performed quickly.

e) Dynamic Modulus

The principles of dynamic analysis of asphalts and asphalt-aggregate mixtures were discussed by Goodrich (1988). Mamlouk and Sarofim (1988) have also described methods to obtain the modulus of asphalt mixtures. Many other researchers before them have described the concepts, for example, Coffman et al.

(1964) and Sisko and Brunstrum (1968). Figure 27 shows a typical plot of stress versus strain from a repeated loading test capable of monitoring the stress and strain pulses.

The following summary is based on the references cited above. The complex modulus (E^*) resulting from such a test is:

$$E^* = \frac{\sigma_0 \sin \omega t}{\varepsilon_0 \sin(\omega t - \phi)} \tag{3.1}$$

where, ω = frequency of applied stress or strain

ϕ = phase difference between stress and strain

t = elapsed time

σ_0 and ε_0 = maximum values of stress and strain pulses, respectively.

The absolute value of the complex modulus $|E^*|$ is commonly referred to as the dynamic modulus, and is found from:

$$|E^*| = \frac{\sigma_0}{\varepsilon_0} \tag{3.2}$$

Other parameters that may be obtained are the storage modulus (E'), the loss modulus (E"), and the loss tangent. These are defined as follows:

$$E' = |E^*| \cos \phi \tag{3.3}$$

$$E'' = |E^*| \sin \phi \tag{3.4}$$

$$\text{Loss tangent} = \tan \phi = E''/E' \tag{3.5}$$

For a test on asphalt, the dynamic viscosity ($|\eta^*|$) may be expressed as:

$$|\eta^*| = |E^*|/\omega \tag{3.6}$$

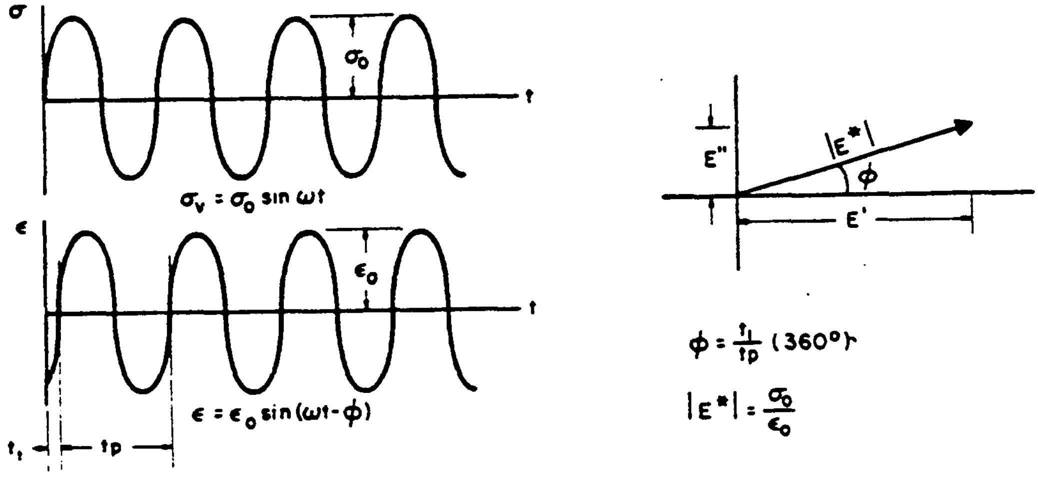


Figure 27. Dynamic Mechanical Analysis (after Coffman et al., 1964)

Goodrich (1988) notes that the loss tangent correlates well with the performance of an asphalt in mixes. For this reason, dynamic analysis will be considered in this project as having the promise for evaluating the aging effects in asphalt-aggregate mixtures.

None of the tests outlined above have shown a strong correlation with field data. However, the resilient modulus, indirect tension and dynamic modulus tests are worthy of use in subsequent studies, and perhaps the creep test, since it is somewhat analogous to viscosity tests used for asphalt cements.

3.2.2 Tests on Recovered Asphalts

Table 1 shows that numerous tests have been done on samples of asphalt recovered from laboratory and field aged mixtures. The most common are:

1) Penetration
2) Softening Point
3) Viscosity
4) Ductility
5) Corbett-Swarbrick Fractionation
6) Rostler-Sternberg Fractionation

No attempt will be made to describe these tests, since they are either currently standardized by ASTM, or have been in the past. A common element in all studies is the use of the Abson approach for recovering asphalt. Several studies utilized the sliding plate microviscometer, e.g. Hugo and Kennedy (1985), Kemp and Predoehl (1981), Benson (1976), and Simpson et al. (1959).

Another test worthy of consideration is a cone and plate viscosity determination, which requires a very small asphalt sample and is capable of operating at higher temperatures than a sliding plate device. Other tests will probably emerge once the laboratory investigations are underway, and where appropriate these will be investigated.

A hot centrifuge recovery procedure will also be considered. This latter method of recovery avoids the use of solvent extraction, but does require heating of a mixture so that small amounts of asphalt can be centrifuged from a mixture. The method has been described by Cahill, Jamieson and Sheedy (1989), and was developed for use with surface dressings.

3.3 Evaluation of Aging Methods

To establish which of the aging methods described above are the most promising for future study, a number of criteria have been selected as follows:

1) simulation of field conditions
2) simplicity of use
3) cost
4) existing experience
5) reliability
6) sensitivity to mix variables
7) other, i.e. any other relevant factors.

No attempt has been made to weight the criteria; they are all important. However, it is vital that the methods simulate field conditions and that they are viable for routine laboratory use. Hence, the first three criteria are probably more important than the others. Also, the first three criteria are the only ones for which reasonable estimates can be made. Tables 12(a) and 12(b) present an evaluation of the promising short-term and long-term methods described earlier in this chapter. Of the three short-term aging methods, extended heating and mixing of loose mixtures are the most promising. However, the use of microwave heating should not be discounted until more detailed evaluation has been carried out. Of the long-term methods, pressure oxidation and oven aging are the most promising and

Table 12. Evaluation of Aging Methods.

a) Short Term

Criterion	Oven Heating	Extended Mixing	Microwave Treatment
1. Simulation of field conditions	Good based on data from Von Quintas et al. (1988)	Simulates plant mixing	Not the same
2. Simplicity of use	Easy to use -no special equipment needs	Easy to use -could use lab mixers -or modified RTFOT	Easy to use
3. Cost of equipment	Moderate	Moderate	Moderate
4. Existing experience	Very little with mixtures	None	Very little
5. Reliability or accuracy	Not established -may require a standard oven	Not established -would require standardization of equipment	Not established
6. Sensitivity to mix variables	Not established	Not established	Not established
7. Other	Analogous with the TFOT	Analogous to RTFOT	May promote structuring

b) Long Term

Criterion	Pressure Oxygen Treatment	Oven Aging	Ultraviolet Treatment	Interaction With Moisture Conditioning
1. Simulation of field conditions	Preliminary tests show that similar levels of aging are achieved	Preliminary tests show that significant aging can be achieved -but at higher temp. than field	Difficult to assess -in service pavements are subject to heat, light, and oxidation	True representation of climatic cycles
2. Simplicity of use	Moderate -needs careful attention to safe handling of oxygen	Easy to use -no special equipment needs	Moderate -could use a weatherometer or lamps	Difficult
3. Cost of equipment	Moderate to high	Moderate	Moderate to high	Moderate to high
4. Existing experience	Very little	Very little	Little with mixtures	None
5. Reliability or accuracy	Questionable -based on data from AAMAS study	Questionable -based on data from AAMSA study	Not established	Not established
6. Sensitivity to mix variables	Preliminary tests indicate promising performance	Not established	Not established	Not established
7. Other	Good experience of several studies with asphalt indicates potential for this method	Analogous with an extended TFOT or RTFOT		

will be thoroughly evaluated in a laboratory study. However, ultraviolet treatment and interaction with moisture conditioning will be evaluated also to provide further insight.

Of the aging methods identified, the literature does not lead to a conclusion that one method is clearly superior to others, or that an as yet untried method is available. Indeed, the literature is very sparse with regard to laboratory aging of asphalt-aggregate mixtures. At the beginning of the literature search, the author did not expect to find evidence that ultraviolet light would have a significant effect. However, several researchers found a significant effect and one group (Tia, et al., 1988) strongly recommend further investigation of such an approach in conjunction with a forced draft oven. Clearly, the effects of ultraviolet light must be included in a laboratory study of promising methods.

3.4 Evaluation of Test Methods

The criteria used to evaluate test methods are similar to those for aging methods, viz:

1) comparison with field data
2) simplicity of use
3) cost
4) existing experience
5) reliability
6) sensitivity to mix variables
7) sample size
8) destructive versus nondestructive
9) other, i.e. any other relevant factors.

Tables 13(a) and 13(b) present an evaluation of the various methods for mixtures and recovered asphalts described earlier in this chapter. Of the tests on mixtures, the modulus and indirect tensile test should definitely be included in a thorough laboratory study. The dynamic modulus test shows particular promise, since it has the potential to identify the

Table 13. Evaluation of Test Methods

a) Mixture Tests

Criterion	Resilient Modulus	Indirect Tensile Test	Dynamic Modulus	Creep
1. Comparison with Field Data	Not established	Not established	Not established	Not established
2. Simplicity of Use	An established and standardized test -moderately difficult	An established test -straightforward	Not significantly different to Resilient Modulus	An established test -straightforward but takes time
3. Cost of Equipment	High	Moderate	High	Moderate
4. Existing Experience	Extensive	Extensive	Moderate	Good
5. Reliability or Accuracy*	Varies with equipment	Unknown	Varies with equipment	Unknown
6. Sensitivity to Mix Variables	Excellent	Good	Not established	Good
7. Sample Size	Varies with mode of testing	Usually 4" dia. by 2-1/2" high	Often 4" diameter by 2-1/2" high, but preferably higher	Varies with mode of testing
8. Destructive vs. Nondestructive	Nondestructive	Destructive	Nondestructive	Nondestructive
9. Other	Representative of repeating loading in the field		Has potential to establish the effect of the asphalt	Analogous to a viscosity test

* NOTE: Only the resilient modulus test is standardized by the ASTM as method D4123 (ATSM, 1988). As of 1988, no precision and bias statements have been established for this test.

b) Tests on Recovered Asphalts

Criterion	Standard Consistency Tests	Ductility	Fractionation	Microviscosity
1. Comparison with Field Data	Good for viscosity and penetration data	Good	Not well established -conflicts in the literature	Good
2. Simplicity of Use	Standard tests, easy to do	Standard test	Standard tests requiring experienced technician	Requires an experienced technician
3. Cost of Equipment	Low to moderate	Moderate	Moderate	Moderate to high
4. Existing Experience	High	Good	Good	Good
5. Reliability or Accuracy*	High	High	Doubtful with inexperienced technician	Doubtful with inexperienced technician
6. Sensitivity to Mix Variables	N/A	N/A	N/A	N/A
7. Sample Size	Requires fairly large samples -difficult to obtain	A microductility test can be used to minimize sample size needed	These methods need only a small sample	Need only a small sample
8. Destructive vs Nondestructive	N/A	N/A	N/A	N/A
9. Other			The procedures available are slow	A cone and plate procedure is a better alternate to a sliding plate approach — it can be used over a wider range of temperatures

* NOTE: The ASTM provides precision statements for most of the tests falling into the generic groups of tests covered by this table.

effect of the asphalt in an asphalt-aggregate mixture. The creep test will not be used since it is the least promising.

No attempt has been made to weigh the criteria. However, perhaps the most important criterion for mixture tests is whether the test is nondestructive. For example, the resilient modulus test, being nondestructive, enables the modulus of a sample to be obtained throughout the entire aging process to which a sample is subjected. Alternatively, the indirect tensile test requires destruction of the sample, and therefore requires a large number of duplicate samples in order to establish the incremental effects of an aging method. The first criterion is intended to indicate if data from the test has shown any correlation with field aging. As with the aging methods, viability for routine use is vital, and therefore, criteria 2 and 3 are also extremely important. Reliability and accuracy are also critical and a note is included in the table regarding test precision where the test is standardized by ASTM. In the case of mixture tests, there is insufficient data from previous research to make any evaluation based on this criterion.

For the tests on recovered asphalt, the most important criterion is probably regarding the specimen size. Tests requiring small specimens (say less than 5 g) are preferable, since the effort required to recover asphalt is minimized. An overriding concern with this category of test is the effect of the recovery process itself, and the possibility that the composition of the asphalt may change. Thenoux et al. (1988b) evaluated four alternatives of recovering asphalt, and determined the composition of the recovered asphalt by the Corbett-Swarbrick approach. It was concluded that the composition was sensitive to the recovery method used. A recovery approach which will be evaluated in this study is a hot centrifuge procedure reported by Cahill et al. (1989). This approach will still change the composition of an asphalt because of the need for heat, and it must be established whether this change is more or less than with solvent extraction and recovery.

4.0 GAPS IN THE KNOWLEDGE

The focus of the preceding chapters has been to describe methods of laboratory aging of asphalts and asphalt mixtures. Studies relating laboratory methods and field aging were also described, and methods of aging and testing mixtures in the laboratory were evaluated. Two major gaps in the knowledge are particularly evident:

1) There is little definition of the relative contribution of the various factors that cause aging.
2) There is little data relating aging of laboratory mixtures to field aging.

These will be discussed further below.

4.1 Contribution of Various Factors to Aging

In Chapter 2, three major factors identified by Peterson (1984) that contribute to aging were presented, viz:

1) loss of oily components by volatilization or absorption
2) changes in composition by reaction with atmospheric oxygen
3) molecular structuring that produces thixotropic effects (steric hardening)

In Chapters 2 and 3, aging and test methods were identified for asphalts and asphalt mixtures that take into account each of these factors. The effects of volatilization are probably the easiest to reproduce, and best defined. For example, Benson (1976) developed a model for penetration change in the early life of a mixture which shows that a typical drop in penetration is of the order of 50%. It should also be noted that a study conducted in Oregon (e.g. Lund and Wilson, 1984) suggests that a similar level of hardening is experienced when expressed as a change in viscosity.

The extent of aging resulting from oxidation is not as clearly defined as that resulting from volatilization. This is hardly surprising since it is a long-term effect with many more variables contributing to the observed performance with time. However, aging methods fall into three main groups as follows:

1) Extended heating
2) Pressure oxidation
3) Ultraviolet light treatment

These are all exaggerations of what a pavement experiences in the field, and although each type of test has been shown to produce changes in properties similar to those occurring in the field, it is not clear which is most appropriate, or what the contribution of each effect is in field aging. In other words, if possible, it should be clarified which approach, if any, most closely reproduces "oxidation".

The steric hardening phenomenon is probably the most difficult to define and reproduce. Any test which would detect a contribution to aging by this phenomenon must be a mixture test since recovery of the asphalt would destroy the associated molecular structuring (Petersen, 1984). A repeated load test on mixtures, such as a resilient modulus test, may also destroy the structuring. Therefore, a rapid "one-shot" test would be suitable, such as a creep test, or, the dynamic modulus test approach which tracks the changes in several parameters as loading progresses.

4.2 Aging of Laboratory Mixtures versus Field Aging

Several studies have shown a good correlation between properties of laboratory aged asphalt and asphalt recovered from the field, e.g. Lee (1973) and Benson (1976). However, no such data is available for mixtures, other than to suggest that property levels of laboratory aged mixtures are similar to those in the field. Clearly, a requirement of this study will be to establish the needed correlations.

One cause for concern with laboratory aging of mixtures to simulate long-term effects is that a compacted sample is required and its integrity may be affected by the elevated temperature used in the aging methods. It should be noted that of the three general methods of long-term aging, i.e. extended heating, pressure oxidation, and ultraviolet light exposure, all known applications use high temperature. In the case of the extended heating approach recommended by Von Quintas et al. (1988), the second stage of testing requires a temperature of 225°F (107°C) which could cause collapse of the sample unless it is confined in some way. The data presented by Kim et al. (1986) also suggests a problem with maintaining the integrity of a sample. This problem needs to be thoroughly investigated.

5.0 CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

Based on the review of available literature, and to some extent, on engineering judgment, the following conclusions are made.

1) Aging of asphalt mixtures occurs in essentially two phases, short-term and long-term. Short-term aging is primarily due to volatilization of the asphalt cement during construction, whereas long-term aging is due to oxidation and steric hardening in the field.
2) There is a wealth of data on laboratory aging of asphalt cements, and several studies have developed relationships between properties of laboratory aged asphalts and those recovered from in service projects. The studies on asphalt aging provide considerable guidance for development of aging methods for asphalt mixtures.
3) The most promising methods for short-term aging of mixtures are extended heating and extended mixing. Microwave heating should also be considered.
4) The most promising methods for long-term aging of mixtures are: pressure oxidation, extended oven aging, ultraviolet light treatment, and alternate aging and moisture treatment.
5) Test methods for evaluating the effects of aging fall into two groups: test on mixtures, and, tests on recovered asphalts.
6) Tests to evaluate mixtures may be divided into nondestructive and destructive groups. Nondestructive tests, in particular resilient modulus and dynamic modulus, show promise for future use. However, the indirect tension test and the resulting tensile strain at break should also be evaluated.

7) Tests requiring small specimen sizes are preferable for tests on recovered asphalt. Therefore, microductility and microviscosity tests have potential. Fractionation tests should also be included in subsequent studies. A hot centrifuge asphalt recovery method should be investigated as an alternate to solvent extraction and recovery.

5.2 Recommendations

A testing plan has been developed to enable evaluation of the promising aging and test methods. An outline of this is presented in Appendix C. The key elements of the plan are given below.

1) The following methods of aging should be investigated:
   a) Short Term
      - oven aging of loose mixtures
      - extended mixing of loose mixtures
      - microwave heating
   b) Long Term
      - pressure oxidation
      - oven aging
      - ultraviolet light treatment
      - cycles of aging and moisture
2) The following tests to evaluate laboratory aging should be investigated:
   a) Tests on Mixtures
      - Resilient Modulus
      - Indirect Tensile
      - Dynamic Modulus

b) Tests on Recovered Asphalt

- Viscosity at 25°C and 60°C
- Penetration at 25°C
- Ductility
- Corbett-Swarbrick and/or Rostler-Sternberg fractionation

3) Field data need to be obtained to demonstrate the validity of the aging methods and to calibrate them. It is recommended that on-going field studies, such as those being carried out for the AAMAS project are used for the purpose. The study team needs to establish other sources of field data. In all cases, a supply of the original materials used is required, and data for mixture and asphalt properties recovered from the pavement at regular time intervals during and after construction.

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APPENDIX A

AGING PROCEDURE USING PRESSURE OXIDATION

The modified pressure oxidation vessel, which was developed originally in England (HMSO, 1962), consists of a cylindrical pressure vessel fitted with end plates. The upper plate contains a safety rupture disc and a pressure gauge. The lower plate contains a plug valve that connects to the oxygen supply. Figure A1 shows a diagram of the pressure oxidation vessel (POV) used in the study by Kim et al. (1986).

The following are the main steps in the use of the POV:

1) Samples (asphalt mixtures or asphalt cement) are prepared.
2) The samples are placed in a POV.
3) A vacuum [26 in. (66 cm) Hg] is applied for 20 minutes.
4) The POV is filled via the valve from an oxygen cylinder to 300 psi (2070 kPa). This pressure is held for 30 minutes to ensure leak-free joints.
5) The oxygen is then disconnected and the POV is then placed in an oven maintained at 77°F (25°C) or 140°F (60°C) for a period of time such as 1, 2, 3, and 5 days.
6) At the conclusion of the test the valve is opened, the cover is removed, and the aged mixtures are cooled to room temperature prior to storage and subsequent testing.

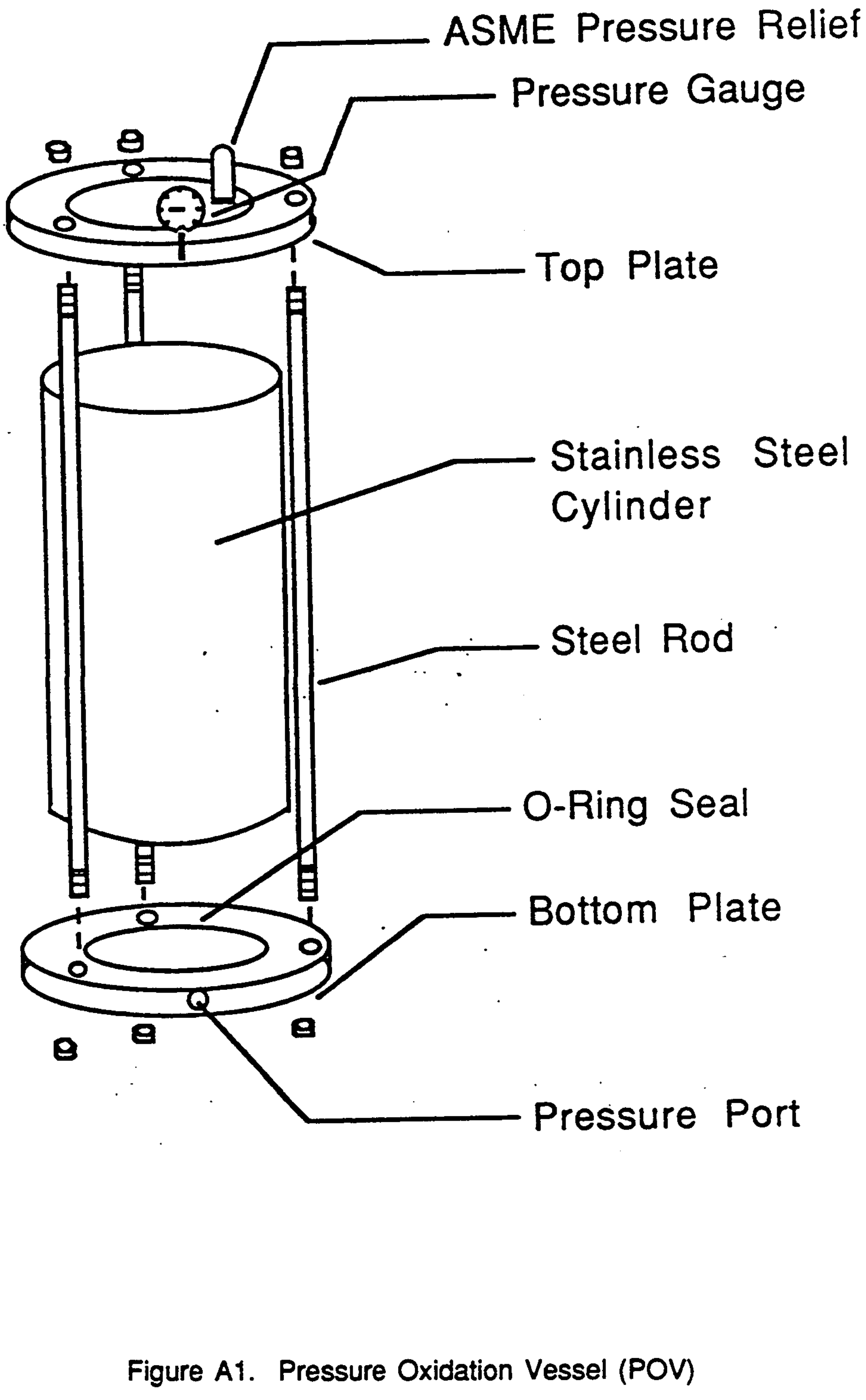

Figure A1. Pressure Oxidation Vessel (POV)

APPENDIX B

A BRIEF SUMMARY OF THE ELECTROMAGNETIC SPECTRUM COMPONENTS: ULTRAVIOLET AND INFRARED RADIATION

Ultraviolet (UV) and infrared radiation are two components of the electromagnetic spectrum. Referring to Figure B1 visible light lies between these two regions. This appendix will briefly summarize each of these types of radiation.

Ultraviolet radiation was discovered by Johann Wilhelm Ritter in 1801, while investigating the effects of light on chemical substances. From this experiment, he hypothesized that energy existed beyond the violet region of visible light spectrum (see Figure B2). The wavelength of UV radiation is longer than x-rays but shorter than visible light. Visible light and UV light are divided into regions. Ultraviolet radiation has three regions. They are:

1) Near or black light - 4000Å-3000Å (where 4000Å is the boundary)
2) Far - 3000Å-2000Å
3) Vacuum - below 2000Å

where 1Å = 1 Angstrom and 1Å = 0.10 nanometer (1x10^{-10} meters).

The chief source of UV radiation is the sun. Most of the UV generated is from the "black light" category, while less than 14% has a wavelength of less than 3000A. More than 50% of UV radiation is absorbed by the atmosphere, the majority of which is the shorter wavelength radiation. UV radiation follows the laws of reflection and refraction. It is absorbed by substances which are transparent to visible light and transmitted through quartz, fluorite and distilled water (Koller, 1989). Ozone in the atmosphere is made by the absorption of the shorter UV wavelength by oxygen. Aluminum is used as a reflecting

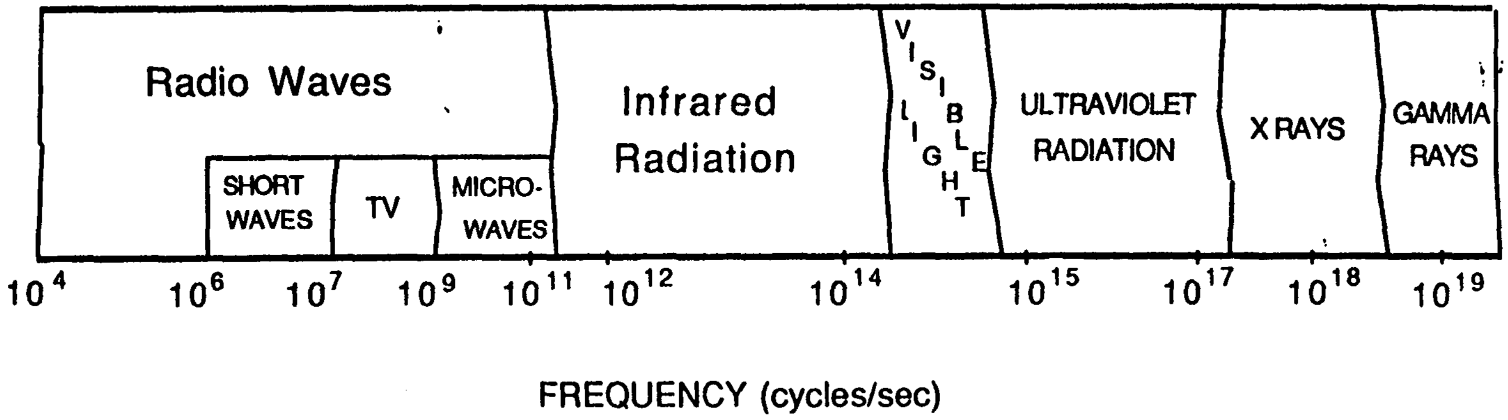

Figure B1. The Electromagnetic Spectrum (after Brady and Holum, 1981)

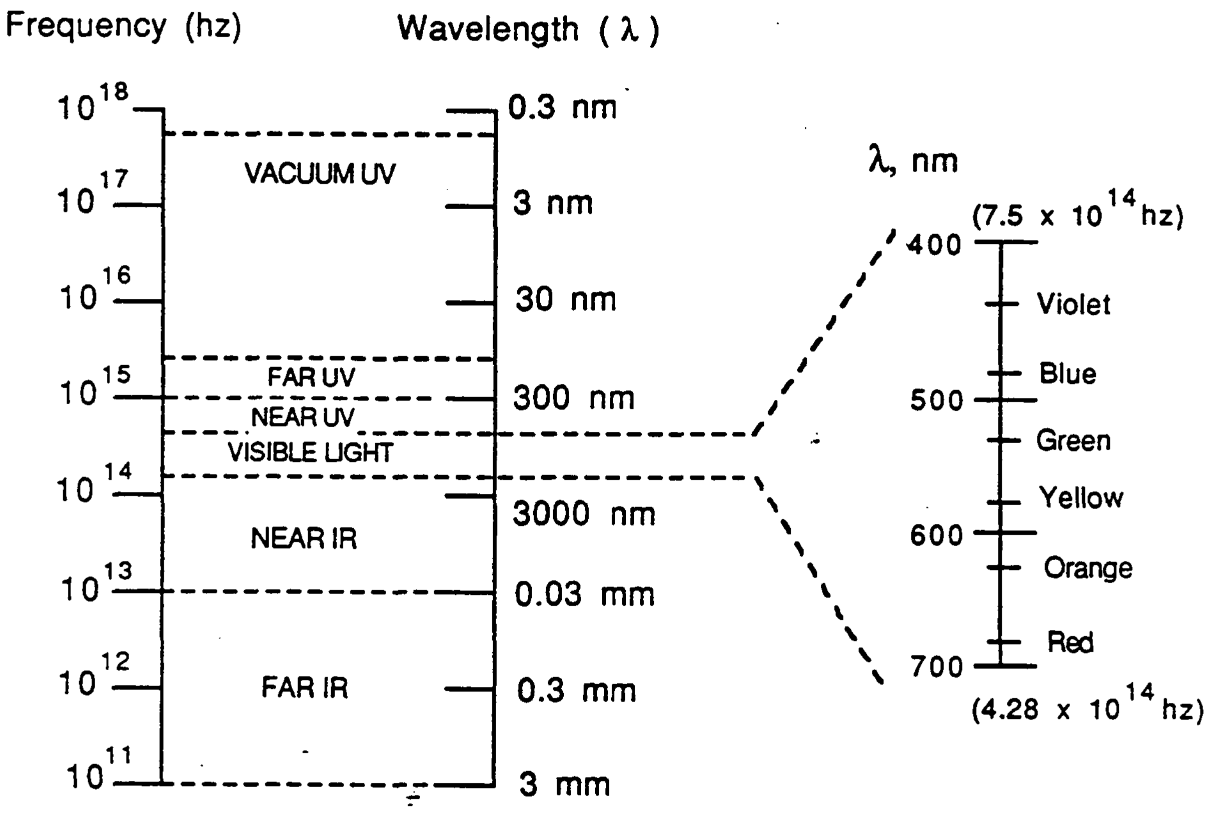

Figure B2. Spectral Regions (after Koller, 1989) device due to its high reflectivity and resistance to tarnishing. Other reflecting materials include sand, snow and water.

UV radiation "promotes chemical reaction", which is the study of photochemistry. An example of this would be the bleaching and folding of dyes due to the exposure of the sun. Its biological effects include suntans and sunburns. This radiation type is a source of vitamin D, which aids in healing wounds in children. Another unique characteristic is its ability to kill bacteria; therefore, UV lamps are used to sterilize air in hospitals, etc. UV radiation is also an efficient method in making fluorescent light.

Infrared radiation was discovered by Sir William Herschel (1738-1822) in 1800. With the aid of a thermometer, he realized that radiant heat was greatest outside the "red" region of the visible spectrum (see Figure B2). Sources of infrared radiation are incandescent solids or electrical discharges in gases. They are detected by the use of photographic plates (Andrew, 1988). Infrared radiation is composed of frequencies (a range of) which make molecules of substance vibrate internally. Because it is unique (like a set of fingerprints), this can be used to identify a compound. Also, similar to UV radiation, certain portions of the spectra are absorbed in the atmosphere. The study of infrared radiation is important because it is a method in probing atomic and molecular structure of solids.

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APPENDIX C

OUTLINE OF TEST PROGRAM

AGING OF ASPHALT-AGGREGATE SYSTEMS

The following is a summary of a laboratory test program that has been presented in a separate document for the SHRP project A-003A (see Hicks et al., 1989). It should be noted that at the time that this report is in preparation (October 1989), a subcontract is being negotiated between Oregon State University and the Research Triangle Institute (RTI), in North Carolina. It is intended that RTI conduct studies regarding the role of ultraviolet light on aging and also some fundamental studies regarding steric and oxidative aging.

1. Purpose

To evaluate the most promising aging method(s) which simulate short-term and long-term aging effects.

2. Test Program

The program to be undertaken at Oregon State University will be done in three phases as follows:

a. Preliminary Test Program
b. Expanded Test Program
c. Field Validation

Only the preliminary program will be outlined here. This program will evaluate the aging methods presented as the most promising in the body of this report. The evaluation will be done with a limited number of material and test variables. The expanded test program and field validation phases will consider more variables.

The preliminary program will be done in two groups of tests as follows:

a. Conventional Procedures

b. Modified Triaxial Procedures

Each of these is described below.

3. Conventional Procedures

Aging Methods. Based on the literature review, the following "conventional" methods will be evaluated:

Short-Term	Long-Term
• Oven Aging	• Pressure Oxidation
• Microwave Aging	• Oven Aging
• Extended Mixing	

Evaluation Methods. The tests proposed at this time are as follows:

Short-Term	Long-Term
• Resilient Modulus	• Microviscosity
• Dynamic Modulus	• Microductility
• Tensile Test	• Fractionation

Other tests may be used, such as infrared spectrometry and size exclusion chromatography on recovered asphalt.

4. Variables Considered for Conventional Aging

The same variables will be used for each of the five aging methods (short- and long-term). These are:

2 asphalts

2 aggregates

2 air void levels

2 test temperatures

3 time periods

All mixtures will be prepared using the mix design asphalt content and gradations, and standard compaction procedures for the California kneading compactor.

A 3/4 fraction of the complete factorial with no replicate tests will provide the preliminary information required. The following table presents the combinations of the variables to be used for each method:

Asphalt (AC) and Aggregate (AC) Combinations	Air Voids Low		Air Voids High	
	Temperature		Temperature	
	Level 1	Level 2	Level 1	Level 2
	Time Period		Time Period	
	a b c	a b c	a b c	a b c
AC1 + Agg 1	x x x		x x x	x x x
AC2 + Agg 1	x x x	x x x		x x x
AC1 + Agg 2	x x x	x x x		x x x
AC2 + Agg 2	x x x		x x x	x x x

For each aging method, 36 specimens will be prepared and tested according to the combinations of variables shown in the table.

5. Modified Triaxial Cell

This approach will try an untested procedure, consisting of forcing fluids to flow through a mixture specimen. At this time it is not known what pressure level will be needed. The main tests to be done will involve oxygen and air as two additional variables to those listed above. A 1/2 fraction of the complete factorial with no replicate tests will provide the preliminary information required. The following table presents the combinations of the variables to be used:

	ATMOSPHERE							
	OXYGEN				AIR			
	Air Voids Low		Air Voids Medium		Air Voids Low		Air Voids Medium	
Asphalt (AC) and Aggregate (AC) Combinations	Temp Level		Temp Level		Temp Level		Temp Level	
	1	2	1	2	1	2	1	2
	Time Period		Time Period		Time Period		Time Period	
	a b c	a b c	a b c	a b c	a b c	a b c	a b c	a b c
AC 1 + Agg 1	x x x			x x x		x x x	x x x	
AC 2 + Agg 1		x x x	x x x		x x x			x x x
AC 1 + Agg 2		x x x	x x x		x x x			x x x
AC 2 + Agg 2	x x x			x x x		x x x	x x x	

In this case, 48 specimens will be required. A secondary series of tests will be done to cycle conditioning of specimens with both water and oxygen or air. Only one combination of asphalt and aggregate will be used. The experiment design has not yet been developed for these tests.