Evaluation and Development of a Pressure Aging Vessel for Asphalt Cement

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This study evaluates a pressure aging vessel (PAV) for asphalt cement, with the specific objectives of examining the safety of the procedure and the effects of aging temperature, film thickness on aging, vertical location within the PAV, and proximity of aging asphalt to other asphalts. Four Strategic Highway Research Program (SHRP) core asphalts (AAC-1, AAD-1, AAF-1, and AAG-1) were used in this study. An interlaboratory test program to establish the variability associated with the PAV conditioning procedure was conducted. The study indicated that the PAV operated at 71°C (160°F), 2.07×10^6 pascals (300 psi) air pressure for 144 hr, effectively simulates 2 to 10 years of pavement aging depending on the method used to measure hardening, air voids in the paving mixture, average ambient temperature, and other factors. Degree of oxidative hardening of asphalt increases with increase in temperature. For a given temperature and time of exposure, degree of hardening increases with decrease in asphalt film thickness. The vertical location of asphalt in the PAV and proximity to other asphalts had no effect on the extent of hardening. Different asphalts exhibited different rates of hardening at the same conditions of temperature and time. After this work was essentially completed, the SHRP PAV conditioning protocol was modified by increasing the temperature and decreasing the time of aging. This was done to accommodate the needs of state departments of transportation for an aging test requiring no more than 24 hr.

Research efforts to develop simple, practical, and reliable procedures to simulate aging of asphalt go back to the beginning of this century. The objective of a long-term aging test for asphalt cement is to realistically simulate hardening that occurs during mixing, construction, and service of asphalt pavements. Acceleration of the oxidation reaction has been achieved using several techniques, which include (a) increased temperature, (b) decreased asphalt film thickness, (c) increased air flow, (d) increased pressure, (e) increased oxygen concentration, and (f) various combinations of these factors.

The rate of reactivity of oxygen with asphalts in pavements has been observed to depend significantly on the temperature level, which led to the various types of oven aging tests that exist today. The idea of thin film aging was first introduced to simulate the aging during plant mixing operations. Because of simplicity, low cost, and the short time required to harden asphalts, oven aging was selected by a large number of researchers to simulate long-term aging as well.

To simulate long-term aging in the shortest period of time, a number of studies were conducted to find the best technique for increasing oxygen accessibility, accelerating its reaction, and limiting volatilization of the asphalt. Increasing the temperature, reducing the film thickness, rotating the asphalt in horizontal or tilted pans or bottles, and adjusting the amount of air flow in the oven have all been used. Temperature levels have been varied between 60°C (140°F) and 163°C (325°F), time of aging varied between a few hours and a few days, and the film thickness varied between a few microns and several millimeters. As a result, a large number of procedures have been developed, some of which reasonably successfully simulate field aging.

At higher temperatures light constituents of asphalt evaporate, producing unrealistic changes in the chemistry of the oxidation reactions when compared with field-aged asphalts. In addition, the high molecular constituents are believed to dehydrogenate and a large percentage of oxygen consumed is discharged in the form of water vapor and other gases, which does not occur at normal service temperatures.

An alternative selected for application in this project was to use pressure as the main accelerating factor and lower the temperature to the level at which volatilization and dehydrogenation do not result in significant alteration of the oxidation process from that experienced in the field. The aging temperature selected was near the maximum expected pavement surface temperature. Because paving asphalts are subjected to significant oxidation and thus hardening during mixing in the plant, it appeared desirable to simulate this step in the laboratory. Therefore, all asphalts were subjected to thin film oven (TFO) aging before they were exposed to the PAV.

This report is an abridged version of the original research report prepared for SHRP as partial fulfillment of project A-002a (*1*).

OBJECTIVES

A study was performed at the Texas Transportation Institute as part of a larger study headed by the Western Research Institute (WRI) in Laramie, Wyoming, to develop a procedure using oxygen or air under pressure at realistic pavement temperatures to simulate long-term field aging of asphalt cement and associated equipment. The overall objective of this project was to evaluate a PAV for asphalt cement, modify the equipment design and procedure as necessary to optimize suitability for routine use by an asphalt specifying agency, and develop a standardizable test protocol for accelerated aging

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of asphalt cement. Specific objectives included determining the effects of aging temperature, asphalt film thickness, simultaneously aging asphalts of different grades or from different sources, and location of asphalt within the PAV. The study also examined the effect of oxidative aging on rheological properties of asphalts using test methods normally specified by state highway agencies and evaluating the repeatability and sources of variability in the test.

SCOPE OF WORK

The effects of PAV conditioning were determined from measurements of penetration and viscosity before and after the conditioning. The method is designed to predict the approximate change in properties of asphalt during the first few years of service in an asphalt concrete pavement as indicated by standard viscosity and penetration measurements. Four SHRP "core" asphalts with a wide range of aging characteristics were chosen for use in the evaluation. An interlaboratory test program (round-robin) was performed using 10 laboratories for estimating the reproducibility and repeatability of the procedure.

The initial PAV aging procedure, developed for SHRP by WRI, was a slight modification of that developed by Lee (*2*). In order to accelerate oxidative aging yet maintain kinetic effects similar to those in a pavement, the use of pure oxygen was specified. The pressure of the reaction vessel was specified to be 2.07×10^6 pascals (300 psi) but the temperature was maintained at 60°C (140°F), average maximum pavement surface temperature, for a period of 144 hr.

Early in the study, safety of the test was a concern and, after investigation, oxygen was replaced with air. During the course of the work, the recommended PAV conditioning temperature was raised by SHRP from 60°C (104°F) to 71°C (160°F). The PAV aging period remained unchanged. Most of the work reported herein was conducted at the latter temperature. The prototype test procedure and equipment design received from WRI was thoroughly evaluated.

DESCRIPTION OF EXPERIMENT

Design of Apparatus

In the early stages of the study, the asphalt aging procedure required pure oxygen to be pressurized to 2.07×10^6 pascals (300 psi). Three prototype pressure aging vessels were fabricated of stainless steel to accommodate the test program. These were designed to withstand pressures of up to 6.2×10^7 pascals (9000 psi) in case of a deflagration of the asphalt-oxygen mixture.

After careful consideration of the hazards associated with pressurized oxygen in the presence of a hydrocarbon fuel like asphalt, a study was conducted by WRI to assess the effect of oxygen concentration on the oxidation reaction of asphalt. It was found that oxygen concentration (between 20 and 100 percent) had relatively little effect on the rate of reaction; therefore, the decision was made to use pressurized air (which contains 20 percent oxygen) in the procedure. The operating parameters were initially 60 C (140 F) and 2.07×10^6 pascals pressure. After significant testing had been completed, it was recommended by WRI that the aging temperature be raised to 71°C (160°F) to increase the rate of reaction and thus simulate a longer in-service aging period.

Test Procedures

The response variables measured for each specimen before and after aging consisted of the following:

Penetration at 25°C, dmm – ASTM D 5: Test Method for Penetration of Bituminous Materials (77°F), and
Viscosity at 60°C, Pa-s – ASTM D 2171: Viscosity of Asphalts by Vacuum Capillary Viscometer (140°F)

Penetration and viscosity of (a) original asphalt, (b) TFO, and (c) TFO + PAV-aged asphalt were measured to estimate the relative age-hardening potential of the material.

Original Unaged Specimens

Asphalt cement specimens were prepared in accordance with the SHRP Materials Reference Library protocol for handling and use of asphalt cements. Physical properties (penetration and viscosity) of original asphalts were measured.

TFO-Aged Specimens

The first step in aging the asphalt specimens was to expose the specimens to the TFO test to simulate hardening in an asphalt plant. This was performed in accordance with ASTM D 1754. The asphalt properties were then measured using those same tests performed on the unaged specimens.

TFO Plus PAV-Aged Specimens

In an cases, PAV aging of asphalts was preceded by TFO aging. The pans of TFO-aged asphalts were placed in the PAV and aged according to the protocol developed. After removing the asphalts from the PAV, changes in their physical properties were measured using the same tests performed on the unaged and TFO-aged specimens.

Experimental Design

Two separate experimental programs were conducted to accomplish the goals of the work: (a) PAV Test Development and (b) Interlaboratory Test Program (Round-Robin).

PAV Test Development

Development of the PAV test encompassed items such as equipment design, variability of the aged asphalts, control of independent variables, written procedures, and personnel safety. The experimental design for evaluating and developing the PAV test included the following controlled variables and levels:

- Oxidation level: 2 levels (zero and 144 hours);
- Asphalt type: 4 levels/tests (selected SHRP core asphalts);
- Different asphalts in PAV simultaneously: 2 levels (yes, no);
- Film thickness: 3 levels 1.6 mm, 3.2 mm, and 6.4 mm or (1/16 in., 1/8 in., and 1/4 in.);
- Location in PAV: 3 levels (top, middle, bottom); and
- Aging temperature: 4 levels 54°C, 60°C, 71°C, and 77°C (or 130°F, 140°F, 160°F and 170°F).

Because the recommended test temperatures changed during the course of the work, it was necessary to conduct the experiment on a partial factorial basis. Penetration and viscosity were selected as response variables because they give a reasonable assessment of oxidative hardening of asphalts, and most state highway agencies have these capabilities.

Interlaboratory Test Program

An interlaboratory test program was conducted to establish accuracy and precision associated with the PAV conditioning procedure. The experimental design included the following controlled variables and levels:

- Number of laboratories: 10 levels;
- Asphalt type: three levels per test; and
- Number of specimens: three samples of each asphalt in the PAV simultaneously.

Asphalts Tested

In this project, four SHRP core asphalts of different grades and from different sources were chosen. Asphalts AAC-1, AAD-1, AAF-1, and AAG-1 were obtained from the SHRP Materials Reference Library in Austin, Texas (Table 1; data supplied by SHRP Contract A001).

TABLE 1 Properties of asphalts chosen for project

Asphalt Grade MRL Code	AC-5 AAC-1	AC-3 AAD-1	AC-20 AAF-1	AR-4000 AAG-1
Original Asphalt				
Viscosity				
60°C (140°F), poise	419	1055	1872	1862
135°C (275°F), Cst	179	309	243	327
Penetration, 0.1 mm				
25°C or 77°F,100 g,5 sec	133	135	55	53
4°C or 39.2F,100 g,5 sec	7	9	0	2
Ductility, cm 4°C or 39.2°,1 cm/min	137.0	150+	7.6	0.0
Softening Point(R&B), °C (°F)	43 (109)	48 (118)	50 (122)	49 (120)
Component Analysis, %				
Asphaltenes (n-heptane)	11.0	23.0	14.1	5.8
Polar Aromatics	37.4	41.3	38.3	51.2
Naphthene Aromatics	37.1	25.1	37.7	32.5
Saturates	12.9	8.6	9.6	8.5
Elemental Analysis				
Nitrogen, %	0.42	0.90	0.23	1.10
Sulfur, %	2.74	8.60	4.52	2.00
Vanadium, ppm	71	293	68	32
Nickel, ppm	40	145	27	71
Thin Film Oven Residue				
Mass Change, percent	-0.2590	-0.8102	-0.0921	-0.1799
Viscosity				
60°C (140°F), poise	1014	3420	4579	3253
135°C (275°F), Cst	239	511	472	304
Viscosity Ratio (60°C or 140°F)	2.42	3.24	2.45	1.75

Three complete repetitions of the PAV aging procedures were conducted with three of these four asphalts to determine the inherent variability of the aging procedure. These three asphalts were then used in the round-robin testing program to further analyze variability for the aging protocol.

RESULTS OF LABORATORY INVESTIGATION AND DISCUSSION

A laboratory testing program was conducted to evaluate the PAV procedure and equipment, to assess any hazards associated with the process, and to determine the allowable variation in such items as aging temperature or thickness of the asphalt film. The laboratory investigation is described in the following subsections.

Safety Investigation

As pure oxygen was used in the first artificial asphalt aging procedure, significant effort was expended to study the associated hazards. A hydrocarbon fuel like asphalt in the presence of pressurized pure oxygen presents a serious potential hazard, particularly in relatively large quantities as desired in the proposed procedure. Furthermore, extreme caution would be necessary when designing, fabricating, and operating such a vessel.

Disastrous explosions were reported when "liquid" oxygen contacted asphalt and pressure was applied on the reacted product (*3*), because of the formation of peroxides. As a part of this study, tests performed by WRI to determine if the use of gaseous oxygen under these conditions would form peroxides revealed no peroxide formation.

Chemical and engineering principles and thermodynamics were applied to determine whether detonation or deflagration would be likely to occur should a spark enter a PAV fully charged with asphalt and gaseous pure oxygen. Deflagration appeared most probable (*3,4*). Recommendations were made regarding the design of the vessel, construction materials, rapid pressure relief systems, and venting of gases or flaming liquids in case of deflagration. The PAV was hydraulically tested to withstand pressures twice the normal operating pressure. Operation behind a safety barrier such as a concrete wall or bunker was recommended.

Interviews with state highway agency and industry personnel indicated a reluctance to routinely operate test equipment with the hazards previously described. Further work at WRI indicated that replacement of oxygen with air in the PAV made little difference in the extent of oxidation of asphalts. Therefore, based on these findings, the decision was made to forego the use of pure oxygen in the PAV and use dry air instead.

Effect of Aging Temperature

In order to determine the effect of PAV aging temperature, PAV tests were performed at 54°C, 60°C, 71°C, and 77°C (130°F, 140°F, 160°F and 170°F). Pressure and aging time remained the same for all tests: 2.07×10^6 (300 psi) air pressure and 144 hr. Fifty grams of asphalt were used for all tests as prescribed by ASTM D 1754. The effects of aging temperature are depicted in Figures 1 and 2.

It should be noted that the relative degree of hardening of asphalts varies at different aging temperatures in the PAV; that is, two asphalts may age similarly at one temperature but age differently at another temperature. This phenomenon is demonstrated by the difference in slopes of the lines in Figures 1 and 2. These findings agree with previous work by Lau et al. (*5*). This fact is significant because the relative results of an aging test will be dependent on the temperature at which the test is conducted. It may be necessary, therefore, to age asphalts at different temperatures to obtain an accurate and complete assessment of susceptibility to oxidation.

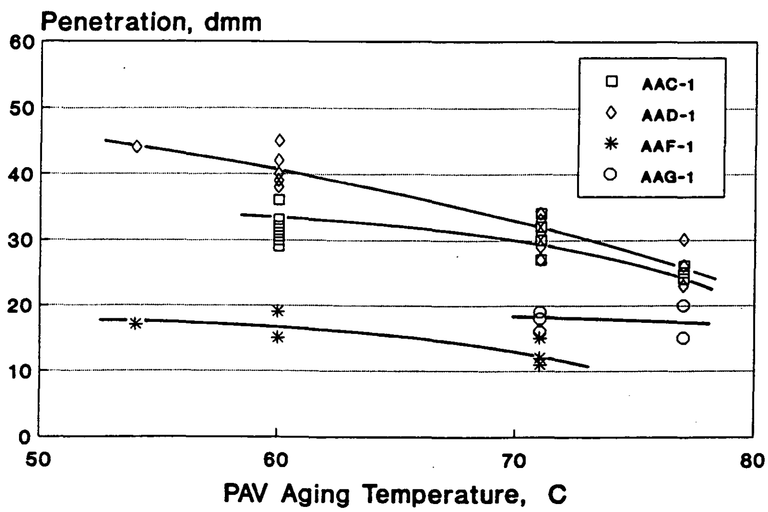

FIGURE 1 Penetration at 25°C of TFO/PAV-aged asphalts as a function of PAV temperature.

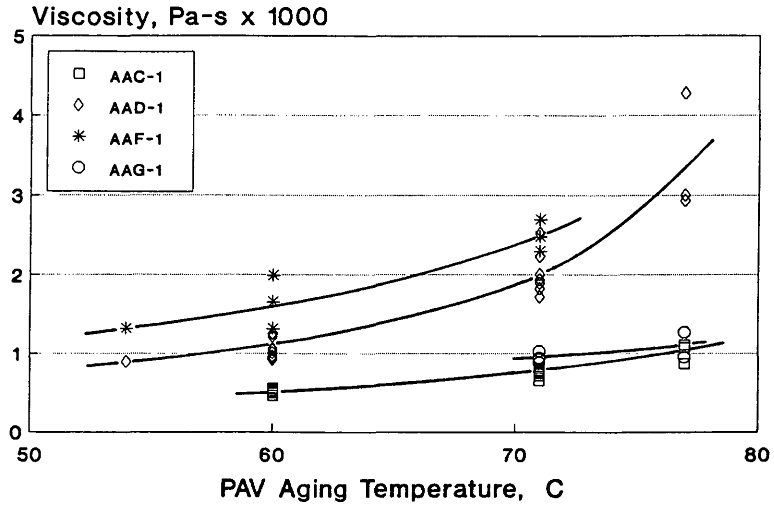

FIGURE 2 Viscosity at 60°C of TFO/PAV-aged asphalts as a function of PAV temperature.

Effect of Film Thickness on Aging

Tests were conducted to examine the effect of asphalt film thickness on aging in the TFO/PAV. The three film thicknesses chosen for this test were 1.6 mm, 3.2 mm, and 6.4 mm ($^1\!/_{16}$ in., $^1\!/_8$ in. and $^1\!/_4$ in.). Normally, 50 gm of asphalt per TFO pan was used throughout this aging study, which gives an asphalt layer thickness of approximately 3.2 mm ($^1\!/_8$ in.) in the TFO pan. In this portion of the study, one-half this amount (25 gm) and twice this amount (100 gm) were used. The pressure aging vessel (PAV) was operated at 2.07×10^6 (300 psi) air at 71°C for 144 hr. Penetration at 25°C and viscosity at 60°C were measured before and after aging. Each test included two replicates (3 separate specimens per replicate) for each of the three film thicknesses.

An almost linear relationship between asphalt film thickness and age hardening over the range of film thickness tested and at the 71°C aging temperature is shown, on the average, in Figures 3 and 4, and this relationship is asphalt dependent.

Effect of Vertical Location Within PAV

The objective of these tests was to determine if there is any effect of vertical location on a specimen within the PAV. An experiment was designed to determine if gases or vapors will stratify within the static PAV to form an oxygen-deficient atmosphere near the top or bottom that would ostensibly reduce oxidative aging in that vicinity. The PAVs used in this study would accommodate up to 10 TFO pans stacked ver-

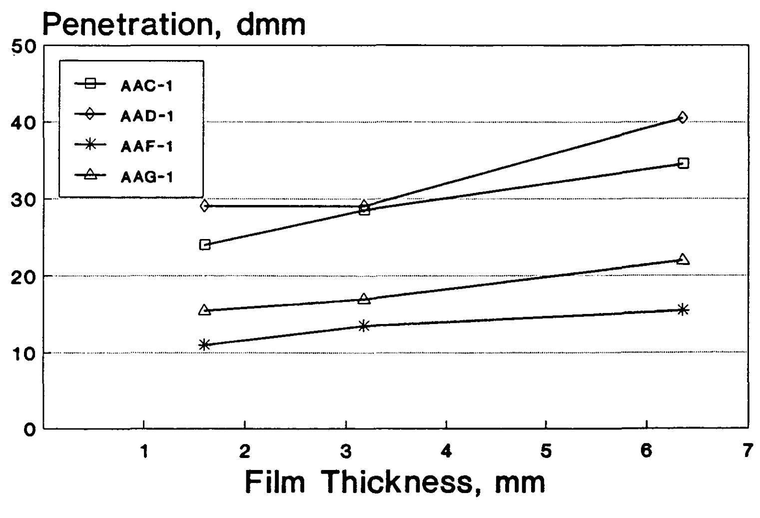

FIGURE 3 Penetration at 25°C after TFO/PAV as a function of asphalt film thickness.

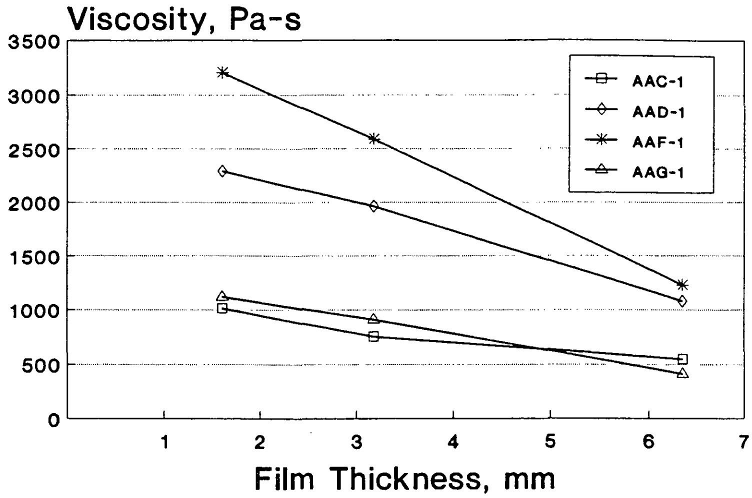

FIGURE 4 Viscosity at 60°C after TFO/PAV as a function of asphalt film thickness.

tically with about 5 mm of space between them. Pans of asphalt were placed in the upper, middle, and lower positions of the pan rack and aged as usual in the PAV. Tests were conducted at 54°C, 60°C, and 71°C (130°F, 140°F, and 160°F).

None of the tests indicated that differential aging occurred with respect to the vertical position in the PAV.

Effect of Proximity to Other Asphalts

The objective of this experiment was to determine the effect of simultaneous aging of asphalts from different sources. Tests were performed to determine whether or not hydrocarbon vapors are released from the asphalt that has been previously exposed to TFO test procedure during PAV aging at 60°C and 71°C. TFO pans filled with a known weight of dried silica gel were placed in the bottom and top pans in the PAV during separate aging tests with three identical asphalts and with three different asphalts aged simultaneously. Asphalts AAC-1, AAD-1, and AAF-1 were used. After completion of the normal aging procedure, the silica gel was removed and weighed. The silica gel was then desorbed using cyclohexane and the extracted solution was subjected to the fourier transform infrared analyzer (FTIR) and gravimetric analysis to determine whether hydrocarbons were present and, if so, to identify them as fully as possible. No asphalt components were detected in the silica gel extracts.

On the basis of this evidence, the assumption was made that no commingling would occur when asphalts from different sources or of different grades are aged simultaneously in the PAV at 71°C. It should be pointed out that only three asphalts were tested in this manner but one of them (AAD-1) was selected because of its relatively high volatile loss in the TFO test (see Table 1). Before the silica gel experiment was conceived, a series of aging tests had been initiated to determine whether commingling occurs when two or more chemically and physically different asphalts are aged simultaneously at 60°C or 71°C in a PAV. Penetration at 25°C and viscosity at 60°C were used in the evaluation. The results show that the range of values for asphalts aged individually are about the same as the range of those aged with other asphalts in the PAV, thus substantiating the silica gel experiment.

Other Findings

When conditioning asphalts in a standard thin film oven equipped with exposed heating elements at the bottom and a rotating shelf containing large pie-shaped ventilation slots, it was found that the extent of aging depended on the location of the asphalt pan on the TFO shelf. If the pan was placed over a slot, thus exposing a portion of the bottom of the pan directly to the radiation from the heating element, much more hardening of the asphalt occurred. After realizing this phenomenon fairly early in the study, all subsequent work was performed with the asphalt pans located between the slots so that no part of the pan was exposed directly to the heating element and to permit the ventilation slots to function as designed. Random placement of pans on the TFO shelf during the test will result in wide variation of aging between pans. This problem is being addressed by ASTM. Pan placement on the shelf should be specified. Heating coils should be covered to shield radiant heat from the asphalt pan.

Because of the rapid evaporation of the hot water, a system was devised using a float valve to automatically replace the lost water so as to prevent overheating of the water-heating elements. In addition, hollow plastic balls 19 mm (¾ in.) in diameter that float on the water bath were used quite successfully to reduce evaporation and heat loss.

Kandhal (*6*) measured penetration at 25°C, viscosity at 60°C, ductility at 15°C (59°F), and 5 cm/min of asphalts extracted from pavements at various times during their service lives, and compared the results with the onset of cracking. He produced plots of these values as a function of time for up to 140 months. Asphalts AAF-1 and AAG-1 have original physical properties similar to the asphalts used in his study. When penetration and ductility of these asphalts after conditioning in the PAV operated at 71°C are compared with the plots produced by Kandhal, it appears that the PAV simulates in-place pavement aging greater than 140 months. However, when viscosity of these asphalts after PAV aging at 71°C is compared with his data, the PAV appears to simulate only about 20 to 40 months of pavement aging.

When penetration and viscosity data from the TFO/PAV tests are compared with field aging data published by Lee (7), they appear to represent about 70 months (extrapolated beyond 50 months of recorded data) of aging in the field.

Additional Comments

Although not considered in this study, other related work has indicated that the TFO test is unsuitable for use with many polymer-modified asphalts. A hard crust will often form on the surface of TFO-aged modified asphalt, thus rendering it unsuitable for further testing. For these types of materials, it may be necessary to use the rolling thin film oven test (ASTM D 2872).

Since the completion of this study, the SHRP PAV conditioning protocol has again been changed. The aging temperature was increased to 100°C (212°F) and the aging time was decreased to 20 hr. A forced-air oven instead of a water bath is required to control the aging temperature of the PAV. The primary reason for these changes was a reduce the time requirements from 6 days to 1 day to accommodate the needs of the state highway agencies. These changes in the test protocol will, of course, significantly reduce the value of the test results reported herein.

INTERLABORATORY TEST PROGRAM

General

On the basis of the foregoing work, a written procedure for the TFO/PAV asphalt conditioning process was prepared. The written protocol and equipment were subjected to an interlaboratory test program, or round-robin, to examine the variability in the properties of artificially aged asphalts.

The following ten agencies participated in the round-robin test program:

Alabama Department of Transportation (Ala)
Arkansas Department of Transportation (Ark)
Colorado Department of Transportation (Col)
Florida Department of Transportation (Fla)
Georgia Department of Transportation (Geo)
Louisiana Department of Transportation (La)
Mississippi Department of Transportation (Mis)
Oklahoma Department of Transportation (Ok)
Southwestern Laboratories (SwL)
Texas Department of Transportation (Tx)

Round-robin participants received training at their respective laboratories by a representative of the research team and were provided with a written test protocol. The round-robin participants performed the tests on replicate sets of the three asphalts. The tests were conducted using the PAV operated at 71°C (160°F) and 2.07 × 10^6 pascals (300 psi) air pressure for 144 hr. Penetration and viscosity data were recorded.

Statistical Analysis

The overall mean values at each level of aging are plotted in Figures 5 and 6 to illustrate the different effects of aging on the three asphalts.

A two-factor analysis of variance (ANOVA) model with interaction was performed on the data from the various laboratories. The two factors were asphalt type (AAC-1, AAD-1 and AAG-1) and treatment (original asphalt, after TFO, and after TFO/PAV).

For the penetration data, there was a significant interaction between asphalt and treatment, meaning that the three asphalts did not respond the same for each treatment level. In order to better understand this difference in response, two analyses were conducted. The first was to compare asphalts

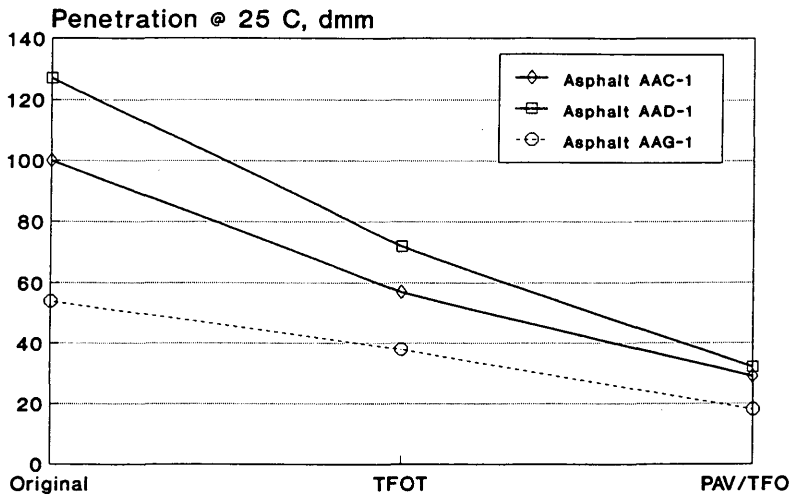

FIGURE 5 Penetration data from round-robin tests showing sensitivity of asphalts to artificial aging.

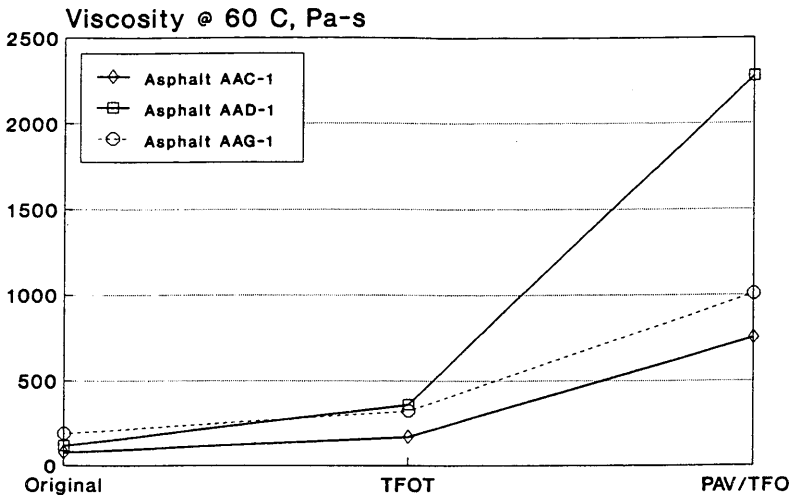

FIGURE 6 Viscosity data from round-robin tests showing sensitivity of asphalts to artificial aging.

for each treatment and the second was to compare treatments for each asphalt.

For the original asphalt, the after TFO, and the after TFO/PAV groups, there was a significant difference in the average penetration among the three asphalts, with AAD-1 being significantly greater than AAC-1, which was significantly greater than AAG-1, according to Duncan's multiple range test at the 5 percent level of significance. Similarly, for each asphalt, there was a significant difference among mean penetrations for each group, with penetration decreasing from the original to the after TFO/PAV.

An interlaboratory analysis was performed to determine if there were significant differences among penetrations measured by the different laboratories. For both the original and after TFO groups, there were no significant differences among the laboratory results for any of the three asphalts. However, after TFO/PAV, there were significant differences for all three asphalts.

For AAC-1, there is no practical difference among the laboratories. For AAD-1, Ok gave significantly higher penetration values than the other states. For AAG-1, Ark had significantly higher penetration values and Lou and Col had significantly lower penetration values than the other states.

The viscosity data yielded similar results. Analysis over all states indicated that there was a significant asphalt-treatment interaction and that mean viscosity significantly increased from original to after TFO/PAV for all three asphalts. Also, there was a significant difference among asphalts within any treatment level (i.e., mean viscosity was different in the original sample for all three asphalts and this difference remained both after TFO and after TFO/PAV).

The interlaboratory analysis for viscosity was similar to the penetration results in that the only significant difference in viscosity among laboratories occurred after TFO/PAV. There were no differences in laboratories in the original or after TFO. However, after TFO/PAV, there was a significant difference among laboratories for all three asphalts.

For AAC-1 and AAG-1, Fla gave significantly higher viscosity than the other states, and for AAG-1, Ok, Lou, and SwL gave significantly lower viscosities. AAD-1 did not show any distinct differences among the laboratory readings.

Precision

Precision of penetration and viscosity test results following TFO/PAV conditioning was computed based in the round-robin test. Criteria for judging acceptability of penetration and viscosity of an asphalt after conditioning in the TFO/PAV are provided in the following two subsections.

Penetration

The single operator coefficient of variation was 5.97 percent (1*S* percent). Thus results of two properly conducted tests by the same operator on similar material should not differ by more than 16.88 percent (*D*2*S* percent).

The multilaboratory coefficient of variation was 10.90 percent (1*S* percent). Thus results of two properly conducted tests from two different laboratories on similar material should not differ by more than 30.82 percent (*D*2*S* percent).

Viscosity

The single operator coefficient of variation was 7.09 percent (1*S* percent). Thus results of two properly conducted tests by the same operator on similar material should not differ by more than 20.05 percent (2*DS* percent).

The multilaboratory coefficient of variation was 13.79 percent (1*S* percent). Thus results of two properly conducted tests from two different laboratories on similar material should not differ by more than 39.0 percent (*D*2*S* percent).

The following data for the associated ASTM tests show that most of the variability obtained in the TFO/PAV conditioning procedure originates in the thin film oven test.

	Single Operator Precision	*Multiple Laboratory Precision*
Penetration, ASTM D5	1	4
Viscosity, ASTM D2171	7	10
TFOT, ASTM D1754 (for viscosity)	9	33

FINAL DESIGN OF EQUIPMENT

After a thorough evaluation of the PAV written procedure and the prototype equipment fabricated as a part of this study, a final equipment design was developed (*1*). This final design was formulated to accommodate commercial production and routine operations in a laboratory. All effort was given to furnish a design that is functional yet convenient, safe, and economical.

CONCLUSIONS

1. Degree of asphalt hardening caused by oxidation increases as aging temperature in the PAV increases from 54°C to 71°C (130°F to 160°F).
2. At a given aging temperature and aging time period, degree of hardening caused by oxidation increases as asphalt film thickness decreases from 6.4 mm to 1.6 mm (¼ in. to ¹⁄₁₆ in.).
3. For the asphalts tested in this study, vertical location of the asphalt in the PAV has no effect on degree of hardening at an aging temperature of 71°C (160°F).
4. For the asphalts tested and the conditions used in this study, aging asphalts simultaneously in the PAV has no effect on the extent of hardening.
5. Different asphalts exhibit different rates of hardening at the same conditions of temperature and time.
6. The relative degree of hardening of asphalts varies at different aging temperatures in the PAV; that is, two asphalts may age similarly at one temperature but age differently at another temperature.
7. On the basis of comparisons of data obtained in this study with field data obtained in other studies, in generally appears that the PAV operating conditions of 71°C (160°F), 2.07×10^6 pascals (300 psi), and 144 hr will simulate 2 to 10 years of aging in a pavement, depending on the method used to measure hardening, air voids in the paving mixture, average ambient temperature, and other factors.
8. On the basis of testing by 10 laboratories of three asphalts in an interlaboratory test series, precision of the TFO plus PAV tests were as follows:

	Single Operator	*Multi-Laboratory*
Penetration	17	31
Viscosity	20	39

REFERENCES

1. J. W. Button, M. Jawle, D. N. Little. *Evaluation and Development of a Pressure Aging Vessel for Asphalt Cement.* Texas Transportation Institute, Texas A&M University, June 1992.
2. D. Y. Lee. Development of a Laboratory Durability Test for Asphalts. In *History Research Record 231.* HRB, National Research Council, Washington, D.C., 1968, pp. 34–49.
3. L. H. Dimpfl. *Asphalt Tank Explosions, Fire Protection Manual for Hydrocarbon Processing Plants.* Chevron Research Company, Vol. 1, 1985.
4. G. E. Moffett, M. D. Pedley, N. Schmidt, R. E. Williams, D. Hirsch, and F. J. Benz. *Ignition of Nonmetallic Materials by Impact of High-Pressure Gaseous Oxygen, Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres.* ASTM STP 986, American Society for Testing and Materials, 1988.
5. C. K. Lau, K. M. Lunsford, C. J. Glover, R. R. Davison, and J. A. Bullin. *Reaction Rates and Hardening Susceptibilities as Determined from POV Aging of Asphalts* (in publication). TRB, National Research Council, Washington, D.C., Jan. 1992.
6. P. S. Kandhal. *Low-Temperature Ductility in Relation to a Pavement Performance, Low-Temperature Properties of Bituminous Materials and Compacted Bituminous Paving Mixtures* (C. R. Marek, ed.). ASTM STP 628, American Society for Testing and Materials, 1977, pp. 95–106.
7. D. Y. Lee. Asphalt Durability Correlation in Iowa. In *Highway Research Record 468,* HRB, National Research Council, Washington, D.C., 1973, pp. 43–60.