Selection of Laboratory Aging Procedures for Asphalt-Aggregate Mixtures

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Abstract

Two major effects dominate aging of asphalt-aggregate mixtures:

1. loss of volatile components and oxidation in the construction phase (short-term aging); and
2. progressive oxidation of the in-place mixture in the field (long-term aging).

Other factors may contribute to aging. In particular, molecular structuring may occur over a long period of time, resulting in steric hardening. Actinic light, primarily in the ultraviolet range, also has an effect, particularly in desert-like climates.

Aging results in hardening (stiffening) of a mixture, which alters its performance. This may be beneficial since stiffer mixtures have improved load distribution properties and are more resistant to permanent deformation. However, aging may also result in embrittlement (increased tendency to crack and ravel) and loss of durability in terms of wear resistance and moisture susceptibility.

This report presents the results of a preliminary investigation to evaluate aging methods for asphalt-aggregate mixtures. Short-term methods included oven aging and extended mixing, while long-term methods included oven aging and oxygen enrichment. The effects of temperature and aging duration are presented. Test specimens were fabricated from two asphalts and two aggregates, representing extreme property levels. The four mixture combinations were prepared at two levels of permeability representing good and moderate compaction conditions. The effects of aging were determined using the diametral resilient modulus test.

Following this preliminary investigation, oven aging of loose mixture for 4 h at 135°C (275°F) is recommended as a short-term aging procedure for future use. For long-term aging, both oven aging and low-pressure oxidation are recommended for future use. For dense mixtures using stiff binders, oven aging for 5 days at 85°C (185°F) is recommended. For open-graded mixtures, or those with soft binders, low-pressure oxidation for 5 days at 85°C (185°F) is recommended. This procedure is more difficult to carry out than oven aging but has the benefit that it supports the specimens during the aging procedure.
Executive Summary

Two major effects dominate aging of asphalt-aggregate mixtures:

1. loss of volatile components and oxidation in the construction phase (short-term aging); and

2. progressive oxidation of the in-place mixture in the field (long-term aging).

Other factors may contribute to aging. In particular, molecular structuring may occur over a long period of time, resulting in steric hardening. Actinic light, primarily in the ultraviolet range, also has an effect, particularly in desert-like climates.

Aging may result in hardening (stiffening) of a mixture, altering its performance. This may be beneficial since stiffer mixtures have improved load distribution properties and are more resistant to permanent deformation. However, aging may also result in embrittlement (increased tendency to crack and ravel) and loss of durability in terms of wear resistance and moisture susceptibility.

This report presents the results of preliminary tests to evaluate aging methods for asphalt-aggregate mixtures. Short-term methods include oven aging and extended mixing, while long-term methods include oven aging and oxygen enrichment. The effects of temperature and aging duration are presented. Test specimens were fabricated from two asphalts and two aggregates, representing extreme property levels. The four mixture combinations were prepared at two levels of permeability representing good and moderate compaction conditions. The effects of aging were determined using the diametral resilient modulus test.

Based on the work done to date in this study, significant conclusions have been reached.

Both short-term oven aging and extended mixing procedures for loose mixtures can cause a fourfold increase in the resilient modulus of some mixtures. Extended mixing appears to produce more uniform aging than does oven aging of loose mixtures. However, oven aging is more viable where productivity is a concern, since several samples can be treated in one oven. Long-term oven aging of compacted-mixture samples at 107°C (225°F) can cause a sixfold increase in the resilient modulus of some mixtures. However, this approach is not
recommended, since the use of high temperatures is potentially damaging to specimens. However, use of a lower temperature of 85°C (185°F) appears to be viable. The results from the pressure oxidation test program for both oxygen and compressed air show a general trend of decreasing modulus with increasing treatment severity. This is a trend contrary to that anticipated and is attributed to disruption of the sample when the gas pressure is relieved. Modifications to the test procedure may improve this situation, but a low-pressure technique is preferable. Triaxial cell aging is an alternative method of oxygen enrichment. Increases of 50 to 100 percent in resilient modulus were observed in this study with this approach using a temperature of 60°C (140°F). This technique, together with long-term oven aging, is recommended for further development.

Several recommendations are made with regard to further development of aging methods. Oven aging of loose mixtures at 135°C (275°F) is recommended for short-term aging. An aging period of 4 h appears to be appropriate. A low-pressure oxidation (triaxial cell) technique is an alternative to long-term oven aging for long-term aging of compacted samples. Temperatures of 60° and 85°C (140° and 185°F) will be used in the expanded test program until a clear understanding of temperature effect is obtained. Oven aging of compacted mixtures at 85°C (185°F) is the recommended approach for long-term aging of dense mixtures. The expanded test program will include an extensive series of tests at 85°C (185°F) in order to compare the oven-aging technique with the low-pressure technique for a wide range of variables.

The results of the expanded test program referred to above are reported by Bell and Sosnovske (1992) in a companion report. A wider range of mixtures was investigated in this expanded study, using the aging methods recommended above. An additional field validation is reported by Bell, Wieder, and Fellin (1992). This study compares the aging of mixtures in the field with that of laboratory-aged mixtures. Both of these companion studies support the recommendations emerging from this study.
1

Introduction

1.1 Background

With regard to asphalt mixtures, aging is associated with the phenomenon of hardening. Two other terms commonly used are “age hardening” and “embrittlement.” The aging process occurs in two stages: short term and long term. Short-term aging occurs during the construction phase and is primarily due to oxidation and to the loss of volatile components while a mix is hot. Long-term aging is primarily due to the progressive oxidation of a mixture while it is in service.

The majority of previous work has investigated the effects of aging on asphalt cements rather than mixtures (Bell 1990), and to date no standard procedure exists for aging mixtures. A major objective of this study is to develop standard laboratory procedures that simulate aging in the field.

1.2 Scope

This report presents the results of a preliminary study of laboratory aging procedures for asphalt-aggregate mixtures. This study was followed by an expanded test program described in a companion report by Bell and Sosnovske (1992). An overview of the aging methods is given in this report, along with an outline of the test procedures that were used to evaluate the effects of different aging methods. Tables summarizing the data are included for readers interested in the specific property levels that were measured. The authors have not completed a detailed statistical evaluation of the data; therefore, the evaluation presented here is based on general trends presented in a series of figures. More detailed statistical evaluation for tests on a much wider range of mixtures is presented by Bell and Sosnovske (1992). Also, a rigorous field validation study is described in another companion report by Bell, Wieder, and Fellin (1992), which includes a detailed statistical evaluation.
1.3 Role of Aging in Specifications

It is essential that short-term aging of mixtures in the field be controlled and that long-term aging not be excessive. While it is important to note that short-term aging in the field can be controlled through mixture specifications and by appropriate quality assurance, long-term aging is more uncertain.

1.3.1 Short-Term Aging

Alternative approaches in the mixture specifications are

1. Setting limits of stiffness achieved after a short-term aging procedure relative to an unaged condition. A factor of 2 may be a suitable upper limit; preliminary studies suggest this might cause a level of asphalt aging similar to that in the thin film oven test.

2. Establishing design levels of fatigue, rutting, or thermal cracking performance for the beginning of the life of a project and ensuring that these levels are met. Some projects may require only that permanent deformation is controlled; specific requirements will depend on the pavement structure.

1.3.2 Long-Term Aging

The extent of long-term aging will depend on the amount of short-term aging that occurs. Mixtures that undergo relatively little short-term aging are more susceptible to long-term aging. Hence, a specification for long-term aging is meaningful only if the short-term aging in the field is controlled. As with short-term aging, alternative approaches exist:

1. Setting limits relative to an unaged condition. A lower limit is necessary to ensure adequate deformation resistance, and an upper limit to ensure adequate fatigue and thermal cracking resistance. These limits could be established by engineering judgment. An example would be to limit the aging to a factor of between 3 and 4.

2. Establishing design levels for fatigue, etc., after a period of 10 years (i.e., long-term aging) and ensuring that these levels are met. This is analogous with the second approach for short-term aging.

3. Use models for aging, stiffness, fatigue, deformation, and thermal cracking to estimate limits for aging on a project-by-project basis. The process is illustrated in Figure 1.1 for a thin pavement, where it is likely that only fatigue will need to be considered. The aging model shown is for long-term aging only, since our only concern is with the service life of the project.
Figure 1.1. Setting aging limits from pavement design considerations — thin asphalt layers
1.3.3 Quality Assurance

It is imperative that short-term aging be controlled in the field. It can be controlled. Thus, it is possible to ensure a known product at the start of the service life of a pavement. A simple approach would be to compact as-delivered mixture specimens with the same device used for the short-term laboratory specimens. The stiffness (or other properties) of the field mix would be compared with the unaged and short-term-aged laboratory mix. Stiffness of 50 to 100 percent of the lab-derived short-term aging ratio could be required.

1.4 Role of Aging in MiDAS

Two distinct phases of aging in the service life of an asphalt mixture are short-term aging during the construction phase of an asphalt pavement and long-term aging during the service life. Short-term aging begins at the mixing plant and ends when the compacted pavement has cooled; long-term aging proceeds thereafter. Both phases of aging must be considered in the mix design and analysis system (MiDAS) as outlined below.

1.4.1 Short-Term Aging

Evaluation of a mixture representative of its initial service condition must be done on short-term aged specimens. The recommended procedure for short-term aging is to age loose mixture in a forced-draft oven for 4 h at 135°C (275°F). This represents an average level. In reality (unless new specifications result in closer control than currently exists), mixtures may undergo very little aging or considerably more aging.

The first role of short-term aging in a rigorous MiDAS should be to evaluate the short-term aging susceptibility of a mixture by comparing the mechanical properties of unaged specimens and short-term aged specimens. This comparison will enable estimation of effects of little or excessive aging. Stiffness modulus measurements will be sufficient to estimate these effects.

A second and equally significant role is to enable specimens to be prepared for the accelerated performance tests, i.e., fatigue, rutting, and thermal cracking. Current mix design procedures neglect to do this. It is vital that a MiDAS test specimens in a condition representative of the initial service condition to establish that the initial field performance will meet the project requirements.
1.4.2 Long-Term Aging

As with short-term aging, it is vital that a MiDAS evaluate the likely performance of a mixture in an aged condition representative of a significant level of long-term aging. The recommended procedure for long-term aging is to age compacted-mixture specimens in a forced-draft oven for 5 days at 85°C (185°F). The process could possibly be accelerated to 2 days by using 100°C (212°F), although tests completed in the expanded test program (Bell and Sosnovske 1992) and field validation study (Bell, Wieder, and Fellin 1992) suggest that this could lead to damage of the specimens. A low-pressure oxidation technique is an alternative to oven aging and may prove to be more appropriate for open-graded mixtures or those with soft grades of asphalt.

The role of long-term aging is to enable representative specimens to be subjected to fatigue, rutting, and thermal cracking tests. By considering the performance of short-term and long-term aged specimens, it will be possible to establish whether the material will function in a pavement for a reasonable service life.

It may be sufficient to consider only long-term aged specimens when evaluating thermal cracking potential. However, for fatigue and permanent deformation, both short-term and long-term aging should be used to establish probable limits of performance. For instance, a mixture evaluated for rutting potential using only short-term aged specimens may appear unsatisfactory, but could exhibit little rutting potential when long-term aged.
Experiment Design

A detailed laboratory test program has been presented in a separate document for SHRP Project A-003A (Hicks, Monismith, and Painter 1989). A major objective of the study reported here was to evaluate the most promising aging method(s) that simulate short- and long-term aging effects. Three phases were undertaken: (1) preliminary test program, (2) expanded test program, and (3) field validation. Only the preliminary program will be described here. As noted in the previous chapter, the results of the other two test programs are presented in two companion reports by Bell et al. The preliminary program involved a limited number of materials and test variables. The expanded test program and field validation phases considered more materials and test variables and served to develop further those methods found to be the most appropriate in the preliminary program.

2.1 Aging Methods

The preliminary program involved two groups of aging procedures, as shown below:

<table>
<thead>
<tr>
<th>Short-Term Aging</th>
<th>Long-Term Aging</th>
</tr>
</thead>
<tbody>
<tr>
<td>Forced-draft</td>
<td>Forced-draft</td>
</tr>
<tr>
<td>oven aging</td>
<td>oven aging</td>
</tr>
<tr>
<td>Extended mixing</td>
<td>Pressure oxidation</td>
</tr>
<tr>
<td></td>
<td>Triaxial cell aging</td>
</tr>
</tbody>
</table>

The short-term methods involved conditioning loose mixtures, while the long-term methods involved conditioning compacted samples. More details are given for each method in subsequent sections.
2.2 Evaluation Methods

The tests used to evaluate the effects of each aging method included the following:

**Mixture Tests**
- Resilient modulus
- Dynamic modulus
- Tensile test

**Asphalt Tests**
- Rheology tests

Other tests were considered for use in the future, including infrared spectroscopy and size exclusion chromatography on recovered asphalt. However, because of time constraints, these tests were not used. Similarly, rheology tests on recovered asphalt were not used to the extent planned because of equipment problems and time constraints. More details are given for each group of tests in a subsequent section.

2.3 Variables Considered for Oven Aging and Extended Mixing

The same variables were selected for each of the aging methods shown in Table 2.1. All of the mixtures were prepared using the mix design asphalt content and gradations, and standard compaction procedures for the California kneading compactor were used. The program tested ¾ of the complete factorial with no replicate tests. For each aging method, 36 specimens were prepared and tested according to the combinations of variables shown in Table 2.1. Two asphalts with substantially different properties, designated with SHRP codes AAK-1 and AAG-1, were used. Likewise, two distinctly different aggregates (SHRP codes RB and RL) were used. As shown in Table 2.1, the two asphalts and two aggregates resulted in a total of four mixtures to be tested.

2.4 Variables Used for Pressure Oxidation

For the pressure oxidation tests, oxygen and compressed air were used at pressures of 690 or 2070 kPa (100 and 300 psi) to provide oxygen enrichment. Therefore, a ¼-factorial experiment requiring 48 specimens was designed, as shown in Table 2.2.

2.5 Variables Used for Triaxial Cell Aging (Low-Pressure Oxidation)

The triaxial cell approach consisted of forcing either oxygen or air through a mixture specimen, thus providing oxygen enrichment. A ½-factorial experiment requiring 48 specimens with no duplicate tests was used, as shown in Table 2.3.
Table 2.1. Variables used in oven aging and extended mixing programs

<table>
<thead>
<tr>
<th>Asphalt and Aggregate Combinations</th>
<th>Low Air Voids (~4%)</th>
<th>Medium Air Voids (~8%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Temperature</td>
<td>Time Period</td>
</tr>
<tr>
<td></td>
<td>Level 1</td>
<td>Level 2</td>
</tr>
<tr>
<td></td>
<td>a b c</td>
<td>a b c</td>
</tr>
<tr>
<td>RL + AAK-1</td>
<td>X X X</td>
<td>X X X</td>
</tr>
<tr>
<td>RL + AAG-1</td>
<td>X X X</td>
<td>X X X</td>
</tr>
<tr>
<td>RB + AAK-1</td>
<td>X X X</td>
<td>X X X</td>
</tr>
<tr>
<td>RB + AAG-1</td>
<td>X X X</td>
<td>X X X</td>
</tr>
</tbody>
</table>

Note: a, b, and c represent different aging periods.
Table 2.2. Variables used in the pressure oxidation program

<table>
<thead>
<tr>
<th>Atmospheric Conditions</th>
<th>Low Pressure</th>
<th>High Pressure</th>
<th>Low Pressure</th>
<th>High Pressure</th>
</tr>
</thead>
<tbody>
<tr>
<td>True Oxygen</td>
<td>abc</td>
<td>abc</td>
<td>abc</td>
<td>abc</td>
</tr>
<tr>
<td>False Oxygen</td>
<td>abc</td>
<td>abc</td>
<td>abc</td>
<td>abc</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>60</th>
<th>25</th>
<th>60</th>
<th>25</th>
</tr>
</thead>
</table>
| Asphalt and Aggregate Combinations
| RL + AAK-1            | X--          | -X-           | --X          | ---           |
| RL + AAG-1            | ---          | --X           | -X-          | X--           |
| RB + AAK-1            | ---          | --X           | -X-          | --X           |
| RB + AAG-1            | X--          | -X-           | ---          | --X           |

Note: a, b, and c represent different aging periods.
<table>
<thead>
<tr>
<th>Asphalt and Aggregate Combinations</th>
<th>Oxygen</th>
<th>Air</th>
<th>Atmosphere</th>
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<td></td>
<td></td>
<td>Air Voids</td>
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<td>Low</td>
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<td>Air Voids</td>
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<td>Medium</td>
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<td>Temperature (°C)</td>
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<tr>
<td></td>
<td>60</td>
<td>60</td>
<td></td>
</tr>
<tr>
<td>Time Period</td>
<td>abc</td>
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</tr>
<tr>
<td>RL + AAK-1</td>
<td>XXX</td>
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<td>XXX</td>
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<tr>
<td>RL + AAG-1</td>
<td>XXX</td>
<td>XXX</td>
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<tr>
<td>RL + AAK-1</td>
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<tr>
<td>RL + AAG-1</td>
<td>XXX</td>
<td>XXX</td>
<td>XXX</td>
</tr>
</tbody>
</table>

Note: a, b, and c represent different aging periods.
Sample Preparation, Aging, and Test Procedures

3.1 Preparation

The preliminary test program used two asphalts (SHRP codes AAG-1 and AAK-1) with extreme characteristics. The aggregates (SHRP codes RB and RL) used were a crushed granite and a chert gravel, respectively. Mixing and compaction followed protocols (Appendix A) established by the SHRP A-003A study team based on the method used to prepare Hveem samples (ASTM D 1560-81a and D 1561-81a). The mixture’s physical properties measured were bulk specific gravity, maximum theoretical specific gravity, and permeability. The procedures used are described in Appendix B, section B.1 through B.3. In addition, the modulus and tensile properties of the mixtures were determined using procedures outlined below and in detail in Appendix B, sections B.4 through B.7. An outline of tests for the recovered asphalt is also given below. The tensile strength and tensile strain at yield were measured by an indirect tensile test. The properties of the original and recovered asphalt were measured by a steady-state rotational viscometer for this phase of the study. A dynamic mode of testing was intended for the expanded test program, but because of time and equipment constraints, this testing was not done.

3.2 Short-Term Aging Procedures

The short-term aging portion of this investigation involved aging mixtures in an uncompacted state in order to simulate the precompaction phase of the construction process. Short-term oven aging used a forced-draft oven for durations of 0, 6, or 15 h at temperatures of 135° or 163°C (275° or 325°F). The aged mix was then compacted at either 1725 or 3450 kPa (250 or 500 psi) compactive effort by a kneading compactor in order to attain target void levels of approximately 8 and 4 percent, respectively. The actual void levels obtained depended on the asphalt-aggregate combination used and varied from
the target levels. The bulk specific gravity, permeability, resilient modulus, and tensile properties were determined for all of the samples.

The extended mixing program used a modified rolling thin film oven (RTFO) test. An attachment to the RTFO drum enables loose mixtures to be rolled, thus extending the mixing time. Samples were mixed using the standard procedure and were then subjected to 0, 10, 120, or 360 min of mixing at either 135° or 163°C (275° or 325°F). The aged mix was then compacted at either 1725 or 3450 kPa (250 or 500 psi) to attain the two void levels. The bulk specific gravity, permeability, resilient modulus, and tensile properties were determined for all of the samples.

3.3 Long-Term Aging Procedures

The oven aging method used forced-draft ovens in order to ensure that the temperature was constant throughout the oven. The aging method used was essentially the same as that used in the Asphalt-Aggregate Mixture Analysis Study (AAMAS) study (Von Quintus et al. 1988). Compacted samples were preconditioned for two days at either 40° or 60°C (104° or 140°F) to ensure the samples’ stability. The specimens were then aged for 0, 2, or 7 days at 107°C (225°F), after which the “before-aging” and “after-aging” characteristics were determined.

The pressure-oxidation vessel used both oxygen and compressed air. The compacted samples were exposed to one of the environments for 0, 2, or 7 days at 690 or 2070 kPa (100 or 300 psi) and 25° or 60°C (77° or 140°F). The preconditioning step performed with the oven-aging method was not required for this procedure. As with the other aging methods, tests were performed on both the unaged and aged samples to determine the effects of aging.

The triaxial cell aging approach involved conditioning a sample while it was positioned in the triaxial test cell shown in Figure 3.1. For this phase of the study, conventional kneading compacted samples were used [10.2 cm diameter by 6.4 cm high (4 in. diameter by 2½ in. high)]. Oxygen or air was passed through the sample, and the resilient modulus was determined at various times during the conditioning process. A flow rate of 0.11 cubic m per h (4 cubic ft per h) was used, which required a pressure of about 345 kPa (50 psi). Tests were run at 25° and 60°C (77° and 140°F).

3.4 Test Procedures

The resilient modulus was usually determined using an indirect tensile testing mode (ASTM D 4123). A triaxial testing mode was also used where convenient, as in the case of the triaxial aging approach. The dynamic modulus was determined in the expanded test...
Figure 3.1. Triaxial aging process
program with a modified triaxial mode of testing, where the loading frequency is varied and the phase lag between the applied load pulse and the strain response is determined. The tangent and loss moduli are obtained at different frequencies so that a thorough characterization of mixture samples can be given. This approach is analogous to the dynamic mechanical analysis of asphalt cement samples.

The modulus tests are nondestructive and yield information on the elasticity (and plasticity in the case of the dynamic test) of the mixture sample. The tensile test is destructive and, therefore, was not done until all of the modulus data had been collected. The tensile test was done at a deformation rate of 5 cm per min (2 in. per min). The load and deformation of the sample are monitored during the test, enabling the strength and the strain at yield to be determined. These data indicate the brittleness of the sample. Von Quintus et al. (1988) suggested that the strain at yield was an indicator of the aging achieved in a mixture sample. Following the tensile test, the mixture portions may be used to obtain recovered asphalt samples.

Only limited tests on the recovered asphalt were done in the preliminary test program, and the data obtained will not be reported or discussed in this report because of its limited nature. The tests that were done included size exclusion chromatography, infrared spectroscopy, and rheological tests. The rheology was defined with a plate-to-plate rotational viscometer, the which was also used in a steady-state mode during the preliminary test program.
4

Results

4.1 Resilient Modulus Data

Tables 4.1 through 4.7 show the complete set of data collected for short-term oven aging, extended mixing, long-term oven aging, pressure oxidation, and triaxial aging, respectively. The majority of the figures presented here plot resilient modulus ratio versus time. The modulus ratio is defined as:

\[
\frac{\text{resilient modulus after aging}}{\text{resilient modulus before aging}}
\]

The majority of the procedure evaluations are based on the calculated modulus ratios. Evaluations based on absolute values of modulus or on the results of tensile tests are not presented, since they result in the same conclusions.

The resilient modulus data for the short-term oven-aging test program and the extended-mixing test program are shown in Figures 4.1 and 4.2. All of the figures use a three-character code to indicate the asphalt-aggregate combination used. For example, KB0 represents asphalt AAK with aggregate RB prepared at a low-air voids level (code 0 in Tables 4.1 through 4.7). Similarly, KB1 represents the same asphalt-aggregate combination with a higher air void level (code 1 in Tables 4.1 through 4.7).

Figures 4.3 and 4.4 show resilient modulus data for the long-term oven aging tests. Note that the modulus ratios shown for 0 days of aging are greater than 1.0 because these samples were conditioned at 40° or 60°C (104° or 140°F), increasing their modulus slightly. Figures 4.5 through 4.8 show resilient modulus data for the pressure oxidation tests with oxygen, and Figures 4.9 through 4.12 show resilient modulus data for the tests with compressed air.
Table 4.1. Summary of short-term oven aging data

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KEY FOR SAMPLE IDENTIFICATION

| K | ASPHALT | K=AAK-1 | G=AAG-1 |
| L | AGGREGATE | L=RL | B=RB |
| L | VOIDS | L=LOW | M=MEDIUM |
| S | SHORT TERM | S=SHORT TERM |
| 0 | AGING PERIOD | 0=6/4 HOURS | 1=15/8 HOURS |
| 0 | AGING TEMP. | 0=LOW (135°C) | 1=HIGH (163°C) |
| O | AGING TYPE | O=SHORT TERM OVEN AGING |

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Table 4.2. Summary of extended mixing aging data

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<th>MODULUS (MPa) BEFORE</th>
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<th>MODULUS RATIO</th>
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**KEY FOR SAMPLE IDENTIFICATION**

| K | Asphalt | K=Ak-1 G=Ag-1 |
| L | Aggregate | L=RL B=RB |
| L | Voids | L=Low M=MEDIUM |
| S | Short Term | S=Short Term |
| 0 | Aging Period | 0=10 Min. 1=120 Min. 2=360 Min. |
| 0 | Aging Temp. | 0=Low (135°C) 1=High (163°C) |
| E | Aging Type | E=Extended Mixing |
Table 4.3. Summary of long-term oven-aging data

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<th>MODULUS (MPa)</th>
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(1) No data recorded due to either operator or equipment error.
(2) No data recorded.
## Table 4.4. Summary of pressure oxidation vessel (oxygen) data

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0 = AGING PRESSURE 0 = ROOM PRESSURE, 1 = 690 KPA, 3 = 2070 KPA
P = AGING TYPE       P = PRESSURE OXIDATION AGING
## Table 4.5. Summary of pressure oxidation vessel (air) data

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**NOTES AND KEY FOR SAMPLE IDENTIFICATION:**

- * Due to an operator error this data was lost.

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TO or TA AGING TYPE TO=OXYGEN TA=AIR
Table 4.7. Summary of triaxial aging (air) data

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NOTES AND KEY FOR SAMPLE IDENTIFICATION:

* Due to an operator error this data was lost.

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28
Figure 4.1. Short-term oven aging results
Figure 4.2. Extended mixing results

(a) Asphalt AAK-1 and aggregate RB

(b) Asphalt AAK-1 and aggregate RL

(c) Asphalt AAG-1 and aggregate RB

(d) Asphalt AAG-1 and aggregate RL
Figure 4.3. Long-term oven aging results for asphalt AAK-1

Figure 4.4. Long-term oven aging results for asphalt AAG-1
Figure 4.5. Effect of pressure-pressure oxidation with oxygen

Figure 4.6. Effect of temperature-pressure oxidation with oxygen

Figure 4.7. Effect of aging period-pressure oxidation with oxygen

Figure 4.8. Effect of voids-pressure oxidation with oxygen
Figure 4.9. Effect of pressure-pressure oxidation with compressed air

Figure 4.10. Effect of temperature-pressure oxidation with compressed air

Figure 4.11. Effect of aging period-pressure oxidation with compressed air

Figure 4.12. Effect of voids-pressure oxidation with compressed air
Tensile test data were obtained for all the short-term and long-term tests and tend to follow the resilient modulus data in terms of ranking aging procedures. Figures 4.13 and 4.14 show tensile test data for the pressure oxidation with oxygen tests, as well as the effect of pressure on tensile strength and strain at yield, respectively.

Figures 4.15a and 4.15b show modulus ratios for the portion of the triaxial aging test program using oxygen at 25° and 60°C (77° and 140°F). Data for the portion of the test program using compressed air are not shown, since very little aging was observed with this approach.
Figure 4.13. Effect of oxygen pressure on tensile strength

Figure 4.14. Effect of oxygen pressure on tensile strain
Figure 4.15. Modulus ratios for triaxial aging with oxygen
5

Discussion of Results

5.1 Short-Term Oven Aging

The data from the short-term oven aging tests (Figure 4.1) show that significant aging occurs, as indicated by an increase in modulus with aging time. When a temperature of 163°C (325°F) is used, the modulus ratio for samples aged for 15 h is lower than the ratio for samples aged for 6 h. This disparity was attributed to severe aging of the asphalt film in the coated mixture and inability to compact these samples adequately after aging. Table 4.1 shows that the air void levels of these samples were much higher than the levels of both unaged samples and samples aged at 135°C (275°F). Compaction of all samples was done at 120°C (248°F), as was done with the unaged samples. It is more appropriate to use an equiviscous compaction temperature in future studies using this aging method. Furthermore, subsequent short-term oven aging will be done only at a temperature of 135°C (275°F), with a maximum time of 4 h as recommended by Von Quintus et al. (1988). One advantage of this approach is that several trays of material can be aged at the same time.

5.2 Extended Mixing

The data from the extended mixing tests (Figure 4.2) show that aging increases with aging time, as indicated by increasing modulus ratio. Similar levels of modulus ratio increase were achieved in these tests and the oven aging tests. Although these tests were successful in achieving significant aging, in order to be viable for production testing, several ovens would be needed, or significant modifications would need to be made to the rolling thin film oven.
More investigation is necessary to understand the effects of oxygen on the asphalt-aggregate mixtures. However, it seems likely that subjecting the samples to a gas at higher pressure disrupts the integrity of the sample, thus reducing the modulus. The disruption probably occurs when pressure is released at the end of the test. An approach that uses low pressure or confines the sample, or both, is preferable.

5.5 Pressure-Oxidation Vessel Using Compressed Air

The pressure-oxidation vessel using compressed air produced similar results to the pressure-oxidation vessel using oxygen. The results are shown in Figures 4.9 through 4.12. Samples subjected to the extreme conditions of 60°C (140°F) and 2070 kPa (300 psi) for seven days experienced the greatest deterioration.

5.6 Triaxial Aging (Low-Pressure Oxidation)

The data shown in Figure 4.15 indicate that moderate increases in aging are achieved with this technique at 25° and 60°C (77° and 140°F), as indicated by the increasing resilient modulus ratio. It is anticipated that considerably more aging will occur if the exposure time is increased to seven days for example, or if the temperature is increased to, say, 85°C (185°F). It appears that this method is viable for realistic long-term oxidative aging, particularly since the mixture is “supported” by a confining pressure throughout the aging process. It is also much safer than the pressure-oxidation approach, since the required pressure is much lower.

This approach was thoroughly evaluated in the expanded test program (Bell and Sosnovske 1992) at 60° and 85°C (140° and 185°F) and is recommended for use with mixtures that may be susceptible to damage using the oven-aging technique, e.g. open-graded mixtures or dense-graded mixtures using soft binders. A temperature of 85°C (185°F) and a period of five days is recommended.
Conclusions

Based on the work done in this study, the following conclusions can be made:

1. Both short-term oven aging and extended mixing procedures for loose mixtures can cause a fourfold increase in resilient modulus in some mixtures.

2. Extended mixing appears to produce more uniform aging in the mix than oven aging. However, oven aging is more viable where productivity is a concern, since several samples can be treated in one oven.

3. Subsequent development of the short-term oven-aging approach will include an adjustment to the maximum exposure time and possibly the adoption of an equiviscous temperature for compaction.

4. Long-term oven aging of compacted-mixture samples at 107°C (225°F) can cause a sixfold increase in resilient modulus in some mixtures. However, the use of such high temperatures is unrealistic for long-term aging and may cause damage to specimens. A temperature of 85°C (185°F) is more appropriate.

5. The results from the pressure oxidation test program for both oxygen and compressed air show a general trend of decreasing modulus with increasing severity of treatment. This is a trend contrary to that anticipated and is attributed to disruption of the sample when the gas pressure is relieved. Modifications to the test procedure may improve this situation, but a low-pressure technique is preferable.

6. The triaxial cell (low-pressure oxidation) aging approach is an alternative method of oxygen enrichment. Increases of 50 to 100 percent in resilient modulus were observed with this approach at a temperature of 60°C (140°F). This technique merits further development at a higher temperature, i.e., 85°C (185°F).
Recommendations

Based on the results of this study and the companion expanded and field validation test programs, the following recommendations are made:

1. Oven-aging of loose mixtures at 135°C (275°F) is recommended for short-term aging. An aging period of 4 h appears to be appropriate.

2. Oven-aging compacted mixtures should be adopted for long-term aging of dense mixtures. A temperature of 85°C (185°F) is most appropriate for a period of five days. A temperature of 100°C (212°F) for two days may be possible; however, such a high temperature may cause damage to specimens.

3. A low-pressure oxidation (triaxial cell) technique is recommended for long-term aging of open-graded mixtures or dense-graded mixtures using soft grades of asphalt. A temperature of 85°C (185°F) is recommended for a period of five days.
References


Appendix A

Specimen Preparation

A.1 Introduction

Specific procedures must be followed in the preparation of asphalt-concrete specimens to be used in the research efforts for SHRP to facilitate uniformity and consistency within the mixes. Uniformity and consistency are imperative; otherwise, the data obtained from tests on the specimens will have little or no statistical significance. In short, sample preparation plays an integral role in the SHRP research efforts.

To achieve uniform and consistent mixes, a set of procedures have been established to aid researchers in sample preparation. These procedures, described below, consist of the following:

1. batching of aggregates to the mix design criteria;
2. mixing the batched aggregate with asphalt;
3. compacting the asphalt-aggregate mixture; and
4. extruding the compacted mixture.

A.2 Batching Procedure

To achieve the appropriate mix gradation, the aggregates need to be batched according to their size. That is, the mix gradation is composed of quantities of aggregate, separated by size, that are combined (batched) to meet the mix criteria. The procedure to batch aggregates is as follows:

1. Obtain the following materials and equipment:
   - the aggregate type to be batched (aggregates are stored in the loft of Merryfield 111).
   - a balance accurate to 0.1 grams.
• a bread pan (located in the mix design area).
• paper sacks to store the batched aggregate (one bag for each sample to be batched).
• a scoop (located in Cabinet N16 in the northeast corner of Apperson 103).

2. Arrange the buckets of aggregate in a semicircle on the bench in the northeast corner of Merryfield 111. Place the balance in the center of the semicircle.

3. Plug in the balance (if necessary), turn it on, and tare the bread pan.

4. Beginning with the largest size of aggregate (e.g., $1 \times \frac{3}{4} \text{ in.}$), scoop out some of the aggregate from its bucket and place it in the bread pan. Adjust the quantity of aggregate so that the aggregate mass corresponds to the batch masses as dictated by the mix criteria.

5. Repeat step 4 for each aggregate size until all sizes have been included in the batch.

   **NOTE:** Place the various sizes of aggregate in separate and distinctive piles so that material can be removed in case you “overshoot” the mass for a specific size.

6. Transfer the batched aggregate to one of the paper bags and label the bag accordingly. (Include, for example: aggregate type, asphalt type to be used, date, researcher’s [your] name, and other appropriate information.)

7. Repeat steps 4 through 6 until all of the batches have been prepared.

8. Return all materials and equipment to their appropriate places. Clean the batching area.

### A.3 Mixing Procedure

Once aggregates have been batched to the gradation specified by the mix design, the next step in the sample preparation procedure is to mix the aggregate with asphalt. Before mixing can begin, however, the mixing equipment must be prepared.
A.3.1 Preparation for Mixing

The following steps must be completed before mixing can begin:

1. Set all of the ovens in the mix design area to the 170±20 cS temperature of the asphalt to be used at least 6 h prior to mixing. The 170±20 cS temperature is 142°C (288°F) for the AR4000 oven and 162°C (324°F) for the AC30 oven.

2. Place all mixing equipment and tools in the ovens at least 4 h prior to mixing. These items consist of
   - mixing bowls with lids and scrapers.
   - at least two spatulas and the scraper spoon.
   - cake pans (enough for the number of samples being prepared).

3. Place the aggregate in the oven at least 4 h prior to mixing.

4. Place the asphalt in the oven approximately 2 h prior to mixing. The lid to the can should remain loosely in place. Stir the asphalt periodically throughout the heating process to ensure uniform heating and to prevent burning. Asphalt that has been at its equiviscous temperature for 3.5 h or more and asphalt that has been burned should not be used and should be discarded.

5. Set one of the large floor-mount forced-draft ovens to 60°C (140°F).

Once these preparations have been completed and the preheating time has elapsed, the samples are ready to be mixed.

**IMPORTANT:** Although the above preparations are presumably sufficient to preheat the tools, equipment, aggregate, and asphalt, it is necessary to ensure that preheating has actually occurred before mixing actually begins.

When mixing is about to begin, remove the asphalt from the oven and place it in the mantle to allow better temperature control of the asphalt.

A.3.2 Mixing

When the tools, equipment, aggregate, and asphalt are at the appropriate mixing temperature (the 170±20 cS temperature of the asphalt), mixing proceeds as follows:

1. Remove one mixing bowl set (bowl, lid, and scraper) from the oven. Remove the lid, leaving the scraper in place.
2. Remove one bag of aggregate from the oven. Carefully place it in the bowl, taking care not to lose the fines.

3. Rotate the scraper rapidly back and forth by hand, making sure that the scraper is at the bottom of the bowl.

4. Place the bowl containing the scraper and aggregate on the balance and tare the balance.

5. Carefully add the appropriate amount of asphalt to within ±0.1 grams of the target amount, taking special care not to overshoot the target amount. Do not take too much time when adding the asphalt.

6. Place the asphalt back in the mantle.

7. Remove the bowl from the balance. Place the lid back on the bowl, allowing the scraper shaft to protrude through the hole in the center of the lid.

8. Transfer the bowl to the mixer, note the time on your watch (or use a stopwatch), and begin mixing.

9. After 1 min of mixing, stop the mixer, remove the bowl, and remove the lid from the bowl. Using a spatula, scrape any unmixed asphalt off the scraper and spade it into the mix. Record the actual mixing time.

10. Scrape any material left on the spatula into the bowl. Rotate the scraper by hand to make sure that it is at the bottom of the bowl. Replace the bowl’s lid, again allowing the scraper shaft to protrude through the hole in the center of the lid.

11. Place the bowl in the mixer and resume mixing for 3 more min. Record the actual mixing time.

12. Remove the bowl from the mixer and transfer it to the workbench. Measure and record the temperature of the mix.

13. Remove a cake pan from the oven and place it next to the bowl.

14. Remove the bowl’s lid. Using a spatula, scrape all material from the lid’s tines into the cake pan. Repeat this step for the scraper.

15. Dump the remaining mix from the bowl into the cake pan. Use the scraper spoon to scrape out all remaining material from the bowl.
16. Shake the cake pan back and forth to ensure that the mix is uniformly deep. Label the pan appropriately and place it in the floor-mount forced-draft oven.

17. Reassemble the mixing bowl. Put the bowl and the other tools back into the oven.

**NOTE:** Rotate the mixing bowls so that the bowl last used has sufficient time to reheat.

18. Repeat steps 1 through 17 until all of the mixes have been prepared.

19. Clean up and return all tools and equipment!

20. Cure the mixes for 15 h at 60°C (140°F).

21. While the mixes are curing, set the ovens to the compaction temperature, 120°C (248°F).

### A.4 Compaction Procedure

Once the mixes have been batched, mixed, and allowed to cure for 15 h at 60°C (140°F), the next step is to compact the mix. As with mixing, several preliminary preparations need to be accomplished before compaction can begin.

#### A.4.1 Preparation for Compaction

The following preparations must precede compaction:

1. Set all of the ovens in the mix design area to 120°C (248°F) (This already should have been done at the end of the mixing procedure.)

2. Place all mixing tools and equipment into the ovens at least 4 h prior to compaction. These items consist of:
   - compaction molds and Marshall mold bases (enough for the number of specimens being prepared).
   - mold funnel.
   - two tamping rods.
   - trough and scraper.

3. Remove the mixes from the 60°C (140°F) oven 2 h prior to compaction and place them in the 120°C (248°F) oven.
4. Set up the kneading compactor as follows:

- Turn on the compactor.
- Turn on the heater for the tamping foot.
- Set the pressures to be applied by the tamping foot:
  - Actuate the tamping foot.
  - Adjust the compactor to the desired pressure.
  - Set the slides on the pressure regulator.
  - Repeat for the next pressure.

Once these preparations have been completed and the prescribed preheating time has elapsed, the mixes are ready for compaction.

**IMPORTANT:** Although the above preparations are presumably sufficient to preheat the tools, equipment, and mixes, it is necessary to ensure that preheating has actually occurred before compaction begins. Therefore, monitor the temperature of everything to be sure that the compaction temperature has been reached.

### A.4.2 Compaction

When the tools, equipment, and mixtures have reached the compaction temperature of 120°C (248°F), compaction proceeds as follows:

1. Set up the mold frame by placing the three shims equidistant around the mold base.

2. Remove a compaction mold from the oven and place it in the mold frame. Tighten the retaining knob against the mold and **place a paper disk in the mold**, making sure it rests flat against the mold base.

3. Remove the mold funnel from the oven and place it over the mold.

4. Remove the trough and scraper from the oven. Place the trough on a level stand so that the bottom of the trough is slightly above the top of the mold funnel.

5. Remove one mix from the oven and dump it into the trough, taking care not to lose material out of either end of the trough. Using the scraper, scrape all material from the cake pan into the trough.
6. Divide the mix in half and push one half into the mold using the scraper.

**IMPORTANT:** Push the material in such a way that it does not roll or tumble. The idea here is to “dump” the material into the mold without causing segregation.

7. Remove one of the tamping rods from the oven and rod the mix first 20 times on the outside (around the perimeter of the mold), and then 20 times in the middle of the mix.

8. Push the remaining material from the trough into the mold. Measure and record the temperature of the mix. Repeat step 7.

9. Place the trough, scraper, and tamping rod back into the oven. Remove the funnel from the mold and place it back into the oven.

10. Transfer the mold frame, with the mold and mixture, to the compactor. “Lock” the frame in place on the platform.

11. Reset the counter on the compactor. Begin primary compaction, making sure that the correct initial pressure is being applied.

12. After 15 tamps, begin removing the shims. Loosen the retaining knob so that the distance between the retaining knob’s screw and the mold is approximately the same as the distance between the two springs on the mold frame.

13. After 20 tamps, adjust the pressure to correspond to the second-stage compaction pressure. Compact the sample for an additional 150 tamps.

14. After secondary compaction has been completed, remove the mold frame with the mold and sample, from the compactor and place it on the work bench.

15. Remove a Marshall mold base from the oven and place the mold on the base.

16. Transfer the mold, with the sample and mold base, to a 60°C (140°F) oven and allow it to cure for 1.5 h at 60°C (140°F).

17. Repeat steps 1 through 16 until all of the mixes have undergone primary and secondary compaction.
18. While waiting for the 60°C (140°F) cure period to elapse, set up the Tinius-Olson static compression machine for tertiary compaction as follows:

- Make sure that both the load and unload valves are fully closed clockwise. **Do not close these valves too tightly!**
- Turn on the hydraulics. Apply load by opening the load valve until the load platen is floating. Reclose the valve.
- Set the load indicator dial to "Zero."
- Turn on the pacer. Set it to 100 percent.

19. After the 1.5 h cure, remove the first compacted sample and place it on a plunger. Place a paper disk on top of the sample, and place a second plunger on top of this paper disk.

20. Place and center the mold, specimen, and plunger arrangement on the load platen.

21. Begin applying load (slowly!) by opening the load valve. As load is applied to the specimen, track the applied loading rate by following the pacer. Regulate the loading rate to follow the pacer by continuously adjusting the load valve.

22. Load the specimen to a maximum load of 12,600 lb, close the load valve (**not too tightly!**), and open the unload valve. Unload the specimen at approximately the same rate as that used to load the specimen.

23. Remove the plungers. Transfer the mold to a counter and lay it on its side.

24. Repeat steps 20 through 24 until all specimens have undergone tertiary compaction.

25. Clean up all tools and equipment used during compaction!

**A.5 Extrusion**

After the specimens have cooled to room temperature, the final step in the sample preparation procedure is the extrusion of the specimen from the compaction mold. This is accomplished as follows:

1. Place the mold and specimen on a plunger. The specimen should be oriented so that the minimum distance through which the specimen must be pushed faces upward.

2. Place the extrusion collar on top of the mold and center the arrangement in the extrusion device.
3. Turn on the extrusion device and throw the control lever to the “Load” position. Continue to load the arrangement until the specimen is pushed out of the mold and into the extrusion collar.

4. Throw the control lever to the “Unload” position and continue to unload until there is enough room for the next mold-plunger-collar arrangement.

5. Disassemble the arrangement, remove and label the specimen, and repeat steps 1 through 5 until all of the specimens have been extruded.

6. Clean up the area and turn off the extrusion device.
Appendix B

Test Procedures

B.1 Bulk Specific Gravity Determination

A procedure developed by the Chevron Research Company was the protocol for determining the bulk specific gravity in specimens. This new procedure ensures consistency in the bulk specific gravity ($G_{mb}$) determinations by various laboratories involved in the A-003A project. An elastomeric film (Parafilm “M” manufactured by the American Can Company) is coated around the specimen, an approach similar to that used by ASTM D1188, which uses parafilm wax. Unlike the parafilm wax film, the elastomeric film does not contaminate the specimen and is easily removed. A brief description of the procedure follows

1. Weigh the sample in air (A).
2. Cut three pieces of the parafilm, two that are 10.2 × 10.2 cm (4 × 4 in.) and one that is 10.2 × 20.3 cm (4 × 8 in.).
3. Stretch the two square films to 15.3 × 15.3 cm (6 × 6 in.) and place them over both ends of the sample. Stretch the 10.2 × 20.3 cm (4 × 8 in.) piece to 10.2 × 40.6 cm (4 × 16 in.) and use it to cover the cylindrical side of the sample.
4. Weigh the coated specimen in air (D).
5. Submerge the coated specimen and record its weight in water at 25°C (77°F) (E).
6. Determine the specific gravity of the parafilm at 25°C (77°F); otherwise, assume a value of 0.9 (F).
7. **CALCULATIONS:**
   \[
   G_{mb} = \frac{A}{(D-E)-(D-A)/F}
   \]
B.2 Rice Specific Gravity

The theoretical maximum specific gravity (Rice specific gravity, \( G_{mm} \)) of bituminous mixtures, ASTM D 2041-78, provided guidance in the procedures for our research project. The following procedure is a brief description of the process used in the \( G_{mm} \) calculations.

1. Weigh the sample and record (A).
2. Determine the mass of the container filled with water at 25°C (77°F) (D). (For our project, \( D = 6215 \) grams.)
3. With the mixture placed in the container, add enough distilled water to entirely cover the sample. Apply a partial vacuum to remove the entrapped air. Shake the contents vigorously at 2-min intervals for 10 min.
4. Remove the vacuum and fill the container with distilled water. Place the cover slowly to remove existing air bubbles. Weigh the entire contents (container, mixture, and water) (E).
5. CALCULATIONS:

\[
G_{mm} = \left[ \frac{A}{(A+D-E)} \right]
\]

B.3 Permeability of Bituminous Mixtures

B.3.1 Scope

This test method is a laboratory measurement of the permeability of compacted bituminous mixtures. It measures the rate at which air can be drawn through bituminous mixtures.

This procedure takes advantage of previous experiences, providing accuracy and simplicity and eliminating the possibility of asphalt contamination, specimen deformation, and other deficiencies often found with other methods. In this procedure, the middle one-third of the specimen’s circumference is coated with silicone, enveloped with a cylindrical rubber membrane 3.8 cm (1.5 in.) high to provide a smooth surface, and cured overnight. A pressure differential is applied across the specimen by connecting the specimen setup to a vacuum pump. The rate of air flow through the specimen is recorded for different vacuum readings. Permeability is determined by calculating the rate of air flow and pressure differential.
B.3.2 Referenced Document

ASTM Standard

D 3637 Permeability of Bituminous Mixtures

B.3.3 Definition

Permeability as defined by Wyoff: Permeability (K) is the volume of fluid (Q) of unit viscosity (μ) passing in unit time (Δt) through a unit cross section (A) of a porous medium of length (L) under the influence of a unit-pressure gradient (Δp).

\[ K = \frac{Q \mu K}{A \Delta p \Delta t} \]

B.3.4 Summary of Method

From the permeability definition, air permeability can be measured by creating a known pressure differential through the specimen and then measuring the rate of air flow rate for a known period of time.

In order for the air flow to pass through the specimen only, the specimen wall must be sealed. Goode and Lufsey (1966) used paraffin for sealing in order to prevent leakage between the specimen wall and the membrane. However, this method contaminates the asphalt, making the specimen useless for further tests.

Another method is to place the specimen in a cylindrical rubber membrane fastened to a hollow metal cylinder with hose clamps. However, this method does not totally prevent leakage between the specimen wall and the membrane, especially with coarse mixtures. Another disadvantage of this method is that air pressure within the membrane may cause the specimen to deform.

Kumar and Goetz (1977) developed a different technique to prevent leakage. The specimen is placed between two collars and coated with silicone rubber seals around all of the specimen and part of both collars in order to bind the collars to the specimen. This method prevents leakage through the specimen wall, but it is rather involved and time-consuming.

The Oregon State University procedure is simple and eliminates these problems while preventing leakage. This procedure is outlined in the following sections.
B.3.5 Significance and Use

5.1 This method can be used only for laboratory tests for mix design purposes.

5.2 The following ideal test conditions are prerequisites for the laminar flow of air through a porous medium under constant-head conditions:

5.2.1 Continuity of flow with no volume change during a test.
5.2.2 Flow with the voids fully saturated with air.
5.2.3 Steady-state flow with no changes in pressure gradient.
5.2.4 Direct proportionality of flow velocity with the pressure gradient below the value at which turbulent flow starts.

B.3.6 Apparatus

6.1 The apparatus is capable of accommodating a range of specimen sizes.

B.3.7 Test Specimens

7.1 Since this test is part of the Moisture Induced Damage Study, the dry subset of AASHTO T 283 will be tested for permeability.

B.3.8 Procedure

8.1 Place the specimen on the specimen holder and seal the middle specimen wall with silicone for a width of 3.8 cm (1.5 in.). Immediately cover the seal with a cylindrical rubber membrane of the same width, 3.8 cm (1.5 in.). Cure the specimen overnight.

8.2 Place the specimen in the triaxial apparatus and envelop the specimen with a cylindrical rubber membrane, long enough to envelop the sample base, upper and lower porous teflon, and sample cap. Tie the assembly, using rubber bands at each end.

8.3 Attach the vacuum outlet to the manometer, vacuum pump, and flowmeter inlet.

8.4 To check for leaks, open valve (a) and close the line to the flowmeter until the manometer reads more than 250 mm Hg. Adjust the vacuum level with the vacuum regulator.

8.5 Close valve (a) and watch the manometer reading. If the reading does not decrease, the system is airtight and ready for testing.
8.6 Open the flow meter and valve (a) and apply the desired pressure difference by adjusting the vacuum regulator. Measure the air flow rate through the specimen using the air flow meter. Repeat for several different pressures. Calculate the pressure differential.

B.3.9 Calculation

9.1 The permeability of a porous medium (Goode and Lufsey 1966), defined in fundamental units, is

\[ K = \frac{\mu Q L}{A(P_1 - P_2)} \]

where:

- \( K \) = permeability, in centimeters per second,
- \( \mu \) = fluid viscosity, in poises,
- \( Q \) = volume flowrate, in cubic centimeters per second,
- \( L \) = sample height, in centimeters,
- \( A \) = sample area, in square centimeters, and
- \( P_1 - P_2 \) = pressure difference, in dynes per square centimeter.

9.2 The above formula was modified by Kumar and Goetz (1977) as shown below. For a specimen 10.2 cm (4 in.) in diameter, a test temperature of 20°C (68°F) and a value of \( \mu \) at 20°C (68°F) of \( 1.813 \times 10^{-4} \) poises, the formula in Section 9.1 reduces to:

\[ K = \frac{(3.812 \times 10^{-11} RH)}{\Delta P} \]

where:

- \( R \) = air flow rate, in ml/min,
- \( H \) = specimen height, in centimeters, and
- \( \Delta P \) = pressure differential, in centimeters of water.
Using the slope \((S)\) of the straight-line portion of the curve obtained from the plot of air flow rate \((R)\) (y axis) versus pressure differential \((\Delta P)\) (x axis), this formula reduces to:

\[
K = 3.812 \times 10^{-11} SH
\]

9.3 Since the pressure difference is measured in millimeters of mercury (mm Hg) and the air flow rate in \(\text{ft}^3/\text{h}\), the following conversion factors are used:

\[
\begin{align*}
1.0 \text{ ft}^3/\text{h} &= 471.9 \text{ cm}^3/\text{min} \\
1.0 \text{ mm Hg} &= 1.868 \text{ in. of water}
\end{align*}
\]

### B.4 Resilient Modulus

The Standard Test Method for Indirect Tension Test for Resilient Modulus of Bituminous Mixtures, ASTM D4123-82 (1987), provided the guidance for our laboratory procedure. Because this testing is nondestructive, the resilient modulus will be calculated for all specimens before and after aging. The general trend as noted by other researchers is that as the aging duration increases, the modulus tends to increase. A brief description of the process follows:

1. The specimen is set up in the MTS machine as shown in Figure B.1.
2. Compressive loads are applied to the specimen through a haversine waveform. The load is applied vertically to the specimen in the diametral plane. The corresponding deformation is recorded on the chart.
3. The specimen is rotated 90° and step 2 is repeated.
4. **CALCULATIONS:** With the load and deformation reading given on the chart, the resilient modulus, \(Mr\), is calculated as follows:

\[
Mr = [0.6183P]/(d*t)
\]

where:

\[
\begin{align*}
P &= \text{applied load, in lb,} \\
d &= \text{deformation, in inches, and} \\
t &= \text{thickness, in inches.}
\end{align*}
\]

Poisson’s ratio was assumed to be 0.35.
Figure B.1. Diametral resilient modulus test
B.5 Use of the MTS Testing Machine for Diametral Resilient Modulus Testing

B.5.1 Set up the MTS for resilient modulus testing as follows:

1.1 442 CONTROLLER (front panel):
   - Select LOAD CONTROL mode
   - Set STABILITY = 7.0 and GAIN = 5.8
   - Set SPAN 1 = 0.0 initially

1.2 442 CONTROLLER (inside):
   - On LIMIT DETECTOR (LOAD):
     • LOWER = -5.0 (set to INLK)
     • UPPER = (doesn’t matter)
   - On DC CONDITIONER (LOAD):
     • RANGE = 4 (10% should show on the front panel)

1.3 410 DIGITAL FUNCTION GENERATOR
   - CONTROL MODE = LOCAL
   - RATE 1 = 1.0 \times 10^6
   - RATE 2 = 0.0 \times 10^6
   - Select HAVERSINE and INVERT with the buttons
   - BREAK POINT = REVERSE LOCAL, 0.0 PERCENT
   - Not selected (buttons out):
     • DUAL SLOPE
     • HOLD AT BRKPT
     • RAMP THRU ZERO
     • MANUAL BRKPT

B.5.2 Set up the test specimen as follows:

2.1 Calibrate the load cell and linearly variable differential transducers (LVDTs) as described in the appendix.

2.2 Place the recorder between the actuator and the MTS control unit. Attach the LVDTs and load cell.

2.3 Set the following settings on the BRIDGE AMP (load amplifier):
   - Sensitivity = 0.2
• Upper toggle switch = CAL
• Lower toggle switch = OFF

2.4 Set the following settings on the CARRIER PREAMP (displacement amplifier):

• SENSITIVITY = OFF
• FILTER = 200 Hz
• OPR/BAL = OPR
• ZERO SUPPRESSION:
  - Switch set in OFF position
  - Upper dial set to 0.0 (unlocked)
  - Lower dial set to 10.0 (locked)

2.5 Place the specimen in the diametral yoke, centering it so that the LVDT gauge heads are centered on the sample. Clamp the sample firmly (not excessively!) using the clamp screws. Make sure that the screws are equally tight.

2.6 Make sure that neither LVDT gauge head is touching the sample. Start the chart paper running at 1 mm/sec (press the second button from the top of the CHART SPEED button panel).

2.7 With the SENSITIVITY for the displacement channel (the right-hand channel) in the OFF position, move the displacement pen (the right-hand pen) to the right-most grid line using the PEN POSITION knob for CHANNEL 2.

2.8 Turn the SENSITIVITY dial to 50. The displacement pen should move about 45 mm to the left (the actual distance depends on the pair of LVDTs but should be constant for a particular pair). If it does not, check that the gauge heads are not touching the sample and that the ZERO SUPPRESSION switch is in the OFF position.

2.9 Turn one LVDT thumbscrew until the LVDT gauge head touches the sample, and then continue turning until half the distance that the pen initially moved is recovered. Repeat this for the other LVDT thumbscrew so that the remaining distance is recovered. For example, if the pen moved 45 mm to the left when the SENSITIVITY was set to 50, turn the thumbscrew of the first LVDT until the pen moves 22.5 mm back to the right. Next, turn the thumbscrew of the other LVDT until the pen moves the remaining distance (22.5 mm) back to the right-most grid line.

IMPORTANT: This step approximately “zeroes” the LVDTs and is absolutely essential; otherwise, there is a high probability that the LVDTs will overload at the
more sensitive settings, resulting in erroneous data. Also, be careful to adjust each LVDT so that the pen moves exactly halfway back for each LVDT.

2.10 Turn the SENSITIVITY knob to 20 and adjust one LVDT, if necessary, so that the pen moves back (right or left) to the right-most gridline. This step fine tunes the location of the LVDT zero point.

2.11 Turn the SENSITIVITY knob to 0.5 and again adjust one LVDT, if necessary, so that the pen moves back to the right-most gridline. Finally, set the SENSITIVITY to 0.2.

**NOTE:** Trying to zero the LVDTs using the thumbscrews is very difficult and unwarranted for SENSITIVITY settings of 0.2 and 0.1.

**B.5.3 Start up the MTS hydraulics as follows:**

3.1 Make sure the appropriate settings have been established as described in Section B.5.1.

3.2 Make sure the **DC ERROR** indicator is centered at zero. If it is not, adjust the **SET POINT** dial until the **DC ERROR** indicator reads zero.

3.3 If necessary, reset the 442 CONTROLLER by pressing the black **RESET** button on the 442 CONTROLLER.

3.4 Reset the 413 MASTER CONTROL PANEL by pressing the blue **RESET** button.

3.5 Start the hydraulic pressure by pressing the **LOW/HIGH HYDRAULIC PRESSURE** button **twice** so that **HIGH** is lit. (You will note a low “whine” followed by the high whine of the pump.)

3.6 Place the specimen, with its diametral yoke but without the yoke chair, on the load strip attached to the load cell. Adjust the sample so that it rests firmly on the load strip and so that the LVDTs are horizontal.

3.7 Place the top load strip on the sample so that the load strip’s indentation (“hole”) faces upward and so that the load strip rests firmly and vertically above the bottom load strip.

**IMPORTANT:** Follow steps 3.6 and 3.7 very carefully; otherwise, inconsistent results are probable.
3.8 Start the strip chart paper at a rate of 1 mm/sec, and apply a small static load (10 to 20 lb) as follows:

- Note the pen position of the load channel (adjust to a convenient position if necessary using the CHANNEL 1 PEN POSITION knob).
- Slowly turn the SET POINT dial on the MTS counterclockwise.
- When the top load strip nears the MTS load ram make any necessary adjustments by moving the load cell base along the sample and yoke, so that the indentation in the top load strip properly aligns with the MTS load ram.
- Continue applying load until the pen on the load (left) channel registers that 10 to 20 lb has been applied. At this point, stop increasing the static load.

3.9 Begin loading the sample dynamically as follows:

- With SPAN 1 set to zero, press the START push button on the 410 DIGITAL FUNCTION GENERATOR (the ZERO OUTPUT light should turn off).
- Slowly increase the dynamic load by turning the SPAN 1 dial clockwise until a load of approximately 50 lb registers on the strip chart recorder.

3.10 If the displacement channel pen is off the grid ("pegged"), flip the ZERO SUPPRESSION switch to + (plus) if the pen is pegged to the right or to − (minus) if the pen is pegged to the left. Turn the ZERO SUPPRESSION top dial clockwise to bring the pen back on the grid.

3.11 With both pens registering movement on the grid, allow 20 to 30 repetitions of the 50-lb load to occur before increasing the load further.

3.12 Slowly increase the dynamic load so that the desired load or deformation is attained.

- If a specific load is desired, set the load and allow enough repetitions to occur so that the deformation does not change appreciably with additional dynamic load applications (i.e., load the specimen until the deformation remains relatively constant). Then "take the modulus."
- If a specific deformation is desired, increase the load until the desired deformation is attained. Allow several load repetitions to occur while
slightly adjusting the load to maintain the desired deformation. When the
desired deformation remains relatively constant at a given load, take the
modulus.

NOTE: Because of the viscoelastic nature of asphalt concrete, permanent
deformation will occur, causing the pen for the deformation
channel to drift off the grid. When this occurs, use the ZERO
SUPPRESSION top dial and the ± switch to keep the pen on the
grid. Also, if the OVERLOAD indicator on the CARRIER AMP
(displacement amplifier) lights while the pen is on the grid,
discontinue loading and re-zero the LVDTs (start over at step 2.4).
To discontinue loading, see Section 3.13.

3.13 Once the desired conditions are met and the modulus has been taken,
discontinue loading by turning SPAN 1 to 0.0, pressing the RETURN TO
ZERO button on the 410 DIGITAL FUNCTION GENERATOR, and turning
the SET POINT dial clockwise until the top load ram is no longer in contact
with MTS load ram.

3.14 Remove the sample and yoke from the load cell and place them in the yoke
chain.

3.15 Turn both LVDTs’ thumbscrews until the gauge heads no longer contact the
specimen.

3.16 Reset the recorder to the conditions described in step 2.4 and rotate the
sample (90°).

3.17 Repeat steps 2.8 through 3.15 for the second axis of the sample.

NOTE: Getting the modulus on the second axis of the sample will
typically take considerably more time because of the plastic
deformation that occurred during the first test. Also, the modulus
of the second axis will typically be lower.

3.18 Report the following for each specimen tested:

• Modulus and strain of each axis.
• Average modulus and strain of both axes.
• Specimen temperature.

It is also recommended that the sensitivity settings on the strip chart paper be
recorded.
B.6 Standard Test Procedure to Determine the Triaxial Resilient Modulus

B.6.1 Test Specimen Preparation

This procedure assumes that a test specimen has already been fabricated in the laboratory or cored from a roadway pavement. Described here is the preparation of the test specimen (surface treatment and instrumentation) for the purpose of obtaining the strain induced from an applied load during a triaxial resilient modulus test using the Automated Testing Software (ATS) package.

B.6.2 Buffing the Test Specimen

Each test specimen is to be buffed (sanded) on diametrically opposite sides using a coarse sanding disk or stone. A No. 80 grit Carborundum sanding disk attached to a wheel clamped in the chuck of a power drill has been found to be satisfactory for this purpose. The detailed procedure for buffing the specimen follows:

1. Assemble the sanding wheel arrangement with a No. 80 grit sanding disk or comparable stone.
2. Select two regions on the side of the specimen that are uniform (without excessive surface voids) and diametrically opposite.
3. Sand (buff) the two regions so that the asphalt film on the aggregate is removed. The sanded area should be at least 1.27 cm (0.5 in.) wide and should extend the thickness (height) of the specimen. The depth of the sanded region should be no deeper than that required to remove the asphalt film on the outer surface of the aggregate.
4. Blow spray the sanded regions with compressed air to remove any fine dust that may be present on the two surfaces. Again, use eye protection!

B.6.3 Instrumenting the Test Specimen

Each test specimen is to be instrumented with strain gauges. Gauges with a 3 cm (1.2 in.) gauge length have been found to be adequate for test specimens with thicknesses (heights) of approximately 6.35 cm (2.5 in.). For specimens of greater thickness (height), gauges...
having gauge lengths of 2.4 in or greater can be used. The detailed procedure for instrumenting the test specimens follows

1. Buff the test specimens as described in the previous section.

2. Obtain strain gauges (two for each specimen), cyanoacrylate ester (Super Glue adhesive), and small paper strips (preferably waxed) that have dimensions slightly larger than the strain gauges (e.g., 0.5 x 3 in. for 1.2 in. strain gauges).

3. Place the test specimen on its side so that the buffed region faces upward.

4. Apply cyanoacrylate along the back of the strain gauge (two to three drops should be sufficient) and spread the adhesive uniformly over the gauge surface.

5. Place the gauge on the buffed surface of the test specimen, quickly cover the gauge with a strip of paper, and apply a light uniform pressure for at least 15 sec. When placing the gauge on the surface, make sure that the gauge is as close as possible to being at a right angle (90°) to the top and bottom surfaces of the test specimen.

6. Repeat steps 4 and 5 for the diametrically opposite buffed surface of the test specimen, making sure that the leads from both strain gauges are aimed in the same direction (i.e., the leads from the gauges should both be directed toward either the top or the bottom of the specimen).

B.6.4 Temperature Conditioning the Test Specimen

Once the test specimens have been instrumented as described above, they need to be temperature conditioned before the resilient modulus test can be conducted. This is a very important aspect of the test procedure that should never be overlooked; otherwise, comparisons of the results from different samples that have been tested at different temperatures are unwarranted. Thus, each specimen needs to be conditioned at the test temperature to ensure that each specimen is at a constant temperature and that all specimens are at the same temperature prior to testing.

To achieve a constant temperature among the specimens, place them in a temperature-controlled environment for at least 24 h. Alternatively, you can monitor the internal temperature of a dummy specimen of the same mixture as the test specimens (e.g., with a thermistor placed in a hole bored to the center of the dummy specimen). However accomplished, it is important that all test specimens be at the same temperature (the test temperature) before and during the resilient modulus test.

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Test Procedure

Assumptions

The following test procedure assumes that the test specimen has been instrumented and temperature conditioned as described above and that all necessary files (i.e., channel, specimen, schedule, and test files) have been created previously — Appendix A describes how to create and edit these files. It is also assumed that the technician is familiar with the ATS software package (e.g., using the mouse, opening, manipulating, and closing windows, etc). Therefore, the following step-by-step procedure omits unnecessary detail — for additional detail, see Appendix A.

Procedure The procedure for running a triaxial resilient modulus test using the ATS software package (specifically, ATS.EXE) is as follows:

1. Execute Microsoft WINDOWS and Digital Control Systems ATS. At the DOS prompt type “WIN ATS” and press the Enter key as shown below:

   C:\>WIN ATS [←→]
2. Select the appropriate channel file. That is, under Setup click on Channels... and then open the appropriate channel (.SET) file.
3. Verify that the appropriate channel file is active. That is, under Setup click on Status... and make sure that the appropriate channel file name is displayed.
4. Select the appropriate test to be run. Under Test, click on Edit... and then open the appropriate test (.TST) file.

5. Make sure that all settings in the test file are appropriate. Edit these settings if necessary, Save or Save as... if desired, and click on Run.
6. Enter a name for the file that will contain the test results (.ATS file) and click on **Save**. Note that the default file name can also be accepted or edited.

7. If the name given to the test results file already exists, a warning is displayed. Answering **Yes** replaces the existing contents of the file with new data, while answering **No** returns control to the previous window.
8. Select (open) the appropriate specimen (.SPC) file.

9. Select the record channels for which data are to be saved to the test results (.ATS) file. Xs in the boxes next to the channel names indicate which channels will be saved (recorded) to disk. Click on OK when satisfied.
10. The *Control Panel* window is displayed next. Before starting the test, proceed to the next step.

11. Activate the monitor window and set the display to units. That is, under *Display* select *Units.*
12. Activate the scope window and set its display so that Channel A is load and Channel B is strn 4. That is, under Display in ATS, select Scope and set the Display and Channel B options in the scope window.

![Scope Window Display](image1)

13. Place the test specimen on the load cell platen using a Teflon disk between the specimen and the platen. Place the top-load platen on the specimen using a teflon disk between it and the specimen. Activate the manual control window for the servo valve (sv0), and set a small static load on the test specimen. The magnitude of the load is displayed in the monitor window and should be less than or equal to the command low load specified in the test (.TST) file.

![Manual Control Window](image2)
14. Reactivate the Control Panel, click on the selected test, click on Test, then Start.

15. Reactivate the scope to ensure that the load and strains are being properly administered and recorded.
16. Minimize (or close) the ATS window and execute ATS Report. Under ATS Report click on Analyze... and open the file (.ATS file) that contains the test data.

17. Make sure that the appropriate settings are shown in the template window before clicking the OK button. Note that template (.TPL) files can be read from the disk by clicking on Template and then on Read.
18. Rename or edit the name of the file (.DAT file) that will contain the results of the test data, and then click the OK button. Alternatively, you can accept the default name by merely clicking on the OK button.

19. The results are then displayed as shown below. This file needs to be imported into a program such as Microsoft EXCEL for further analysis and manipulation (e.g., printing, graphing, etc).
B.7 Split Tension Test

The Split Tension Test followed the procedure provided by Oregon State University. The following is a brief outline of the procedure

1. Set up the MTS machine for the split tension testing procedure. Note the difference between the dial settings for the resilient modulus procedure and the settings for this procedure.

2. Place the specimen in the MTS machine and set up the computer appropriately (the ATS software package is used to record the data).

3. Slowly and carefully monitor the load’s effect on the specimen. After the specimen fails, quickly remove the loading.

4. Reset the ATS software to record another test.

5. Place the broken specimen in a sealable plastic bag, identify it appropriately, and store it in the refrigerator.

6. **CALCULATIONS:** With the given chart readings and calibrations, the tensile strength is calculated as follows:

\[
S = \frac{2P}{\pi dt}
\]

where:

- \( S \) = tensile strength, in psi,
- \( P \) = load applied to failure, in lbs,
- \( d \) = diameter, in inches, and
- \( t \) = thickness, in inches.
Appendix C

Aging Procedures

C.1 Triaxial Aging (Low-Pressure Oxidation)

*Standard Practice for*

Long-Term Aging of Asphalt-Concrete Mixtures
Using Low-Pressure Oxidation Cell

**C.1.1 Scope**

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

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

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

**C.1.2 Referenced Documents**

2.1 *AASHTO Documents:*

   M P1 Performance-Graded Asphalt Binder
R 11 Practice for Indicating Which Places of Figures are to be Considered Significant in Specifying Limiting Values
T 27 Method for Sieve Analysis of Fine and Coarse Aggregates
T 164 Method for Quantitative Extraction of Bitumen from Paving Mixtures
T 168 Method of Sampling Bituminous Paving Mixtures
T 201 Method for Kinematic Viscosity of Asphalts
T 269 Method for Percent Air Voids in Compacted Dense and Open Bituminous Paving Mixtures
M 007 Short- and Long-Term Aging of Bituminous Mixtures
M 002 Preparation of Compacted Specimens of Modified and Unmodified Hot Mix Asphalt by Means of the SHRP Gyratory Compactor
M 008 Preparation of Test Specimens of Bituminous Mixtures by Means of Rolling Wheel Compactor

2.2 ASTM Documents:

D 8 Standard Definitions of Terms Relating to Materials for Roads and Pavements
D3549 Method for Thickness of Height of Compacted Bituminous Paving Mixture Specimens
E 1 Specification for Thermometers

C.1.3 Terminology

3.1 Desired Mixing Temperature — The target temperature for compacting asphalt-concrete mixtures in the laboratory. The desired mixing temperature should be equivalent to the anticipated field compaction temperature. If field compaction temperatures are unknown, select a compaction temperature that corresponds to a kinematic viscosity of 665 ± 80 cS for the asphalt binder used.

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

3.2.1 Standard Definitions D 8
3.2.2 Performance-Graded Asphalt Binder M P1
3.2.3 Short- and Long-Term Aging of Bituminous Mixtures M 007
3.2.4 Kinematic Viscosity of Asphalts T 201

C.1.4 Summary of Practice

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

C.1.5 Significance and Use

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

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

C.1.6 Apparatus

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

6.1.1 Load Frame Assembly — A load-frame assembly shall possess equipment capable of measuring the confining pressure within the cell and providing and measuring oxygen flow to the test specimen.

6.1.2 Perforated Teflon Disks

Table C.1. Minimum aging test system requirements

<table>
<thead>
<tr>
<th></th>
<th>Range, °C</th>
<th>Resolution, °C</th>
<th>Accuracy, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oxygen Flow Control</td>
<td>1 – 10 scfh</td>
<td>&lt; 0.5 scfh</td>
<td>± 0.5 scfh</td>
</tr>
<tr>
<td>Oxygen Flow Measurement</td>
<td>1 – 10 scfh</td>
<td>&lt; 0.5 scfh</td>
<td>± 0.25 scfh</td>
</tr>
<tr>
<td>Oxygen Pressure Measurement</td>
<td>0 – 600 psi</td>
<td>20 psi</td>
<td>10 psi</td>
</tr>
<tr>
<td>Confining Pressure Control</td>
<td>0 – 100 psi</td>
<td>2 psi</td>
<td>± 1 psi</td>
</tr>
<tr>
<td>Confining Pressure Measurement</td>
<td>0 – 100 psi</td>
<td>2 psi</td>
<td>± 1 psi</td>
</tr>
</tbody>
</table>

Note: 1 psi = 6.9 kPa; scfh is standard ft³/h = 0.0283 m³/h.

6.2 Oven — Any oven that is thermostatically controlled and capable of being set to maintain any desired temperature, from room temperature to 260°C (500°F). The oven shall be used for heating aggregates, asphalt binders, or laboratory equipment.
6.3 **Water Bath** — A water bath that is at least 457 mm (18 in.) deep and is thermostatically controlled so as to maintain the bath at 85°C ± 1°C (185°F ± 1.8°F). The tank requires a perforated false bottom or a shelf for supporting specimens 51 mm (2 in.) above the bottom of the bath. The water bath should also be equipped such that a constant flow of water is available to replenish any water loss from evaporation to maintain a constant water level.

6.4 **Miscellaneous Apparatus:**

6.4.1 One shallow metal oven pan/sheet about 305 by 457 mm (12 by 18 in.) for heating uncompacted asphalt-concrete mixtures.

6.4.2 Thermometers having a range of 50°C to 260°C (122°F to 500°F) and conforming to the requirements as prescribed in ASTM E-1.0, volume 14.03.

6.4.3 Waterproof marking sticks for identifying specimens.

6.4.4 Paper labeling tags.

6.4.5 Metal spatula or spoon.

6.4.6 Oven gloves.

6.4.7 36 cm (14 in.) long by 38 mm (1.5 in.) wide strip of butcher paper.

6.4.8 38 mm (1.5 in.) of 102 mm (4 in.) diameter rubber membrane.

6.4.9 152 mm (6 in.) of 102 mm (4 in.) diameter rubber membrane.

6.4.10 One specimen holder.

6.4.11 Two 102 mm (4 in.) by 1.8 in. (46 mm) thick O-rings.

### C.1.7 Materials

The following materials are required:

7.1 Oxygen for Aging Test System.

7.2 Clear rubber silicone.

### C.1.8 Hazards

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

### C.1.9 Sampling

9.1 The asphalt-concrete mixtures shall be sampled in accordance with T 168, or shall consist of specimens that have sampled and short-term aged in accordance with M 007.
9.2 Compacted roadway samples shall have a cut test specimen size of 102 ± 6 mm (4 ± 0.25 in.) in diameter by 152 ± 6 mm (6 ± 0.25 in.) in height.

C.1.10 Specimen Preparation

10.1 Uncompacted Laboratory Mixture Samples

10.1.1 Heat the asphalt concrete to the desired compaction temperature.

10.1.2 Compact an amount of asphalt-concrete mixture sufficient to give the desired specimen size in accordance with M 002.

NOTE 1: Compact a sufficient amount of material to ensure that the final test specimen size after step 9.1.4 is 102 ± 6 mm in diameter by 152 ± 6 mm in height.

10.1.3 Cool the compacted test specimen to 60°C ± 1°C (140°F ± 1.8°F).

10.1.4 After cooling the test specimen to 60°C (140°F), level the specimen ends by applying a static load to the specimen at a rate of 7260 ± 5 kg/min (16,005 ± 11 lb/min). Release the load at the same rate when the specimen ends are level or when the load applied reaches a maximum of 5715 kg (12,600 lb).

10.1.5 After cooling the test specimen at room temperature overnight, extrude the specimen from the compaction mold.

10.2 Sealing Compacted Laboratory and Roadway Specimens

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

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

NOTE 2: Covering the exposed portions of the specimen is extremely important, as large air voids or sharp edges may cause the rubber membrane to rupture under confining pressures at high temperatures. If the rubber membrane ruptures during testing, the specimen should be discarded.
C.1.11 Procedure

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

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

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

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

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

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

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

11.8 Place the entire cell in a 85°C ± 1°C (185°F ± 1.8°F) bath for 5 days ± 0.5 h. Periodically monitor the oxygen flow to ensure that there is a continuous supply.

11.9 After 5 days, turn off the oxygen flow and release the confining pressure. Remove the cell from the water bath and allow the entire assembly to cool to 25°C (77°F).

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

C.1.12 Report

12.1 Report the following information:

12.1.1 Asphalt Binder Grade

12.1.2 Asphalt Binder Content — in percent to the nearest 0.1 percent

12.1.3 Aggregate Type and Gradation
12.1.4 Short-Term Aging Conditions. The following information is applicable:

12.1.4.1 Plant Mixing Temperature — in °C to the nearest 1°C
12.1.4.2 Laboratory Mixing Temperature — in °C to the nearest 1°C
12.1.4.3 Short-Term Aging Temperature in Laboratory — in °C to the nearest 1°C
12.1.4.4 Short-Term Aging Duration in Laboratory — in min to the nearest 1 min

12.1.5 Compaction Temperature — in °C to the nearest 1°C
12.1.6 Compacted Specimen Height — in mm to the nearest 1 mm
12.1.7 Compacted Specimen Diameter — in mm to the nearest 1 mm
12.1.8 Compacted Specimen Density — in kg/m² to the nearest 1 kg/m²
12.1.9 Compacted Specimen Air Voids — in percent to the nearest 0.1 percent
12.1.10 Long-Term Aging Oxygen Flow — in cm³/s to the nearest 236 cm³/s
12.1.11 Long-Term Aging Oxygen Pressure — in kPa to the nearest 69 kPa (10 psi)
12.1.12 Long-Term Aging Confining Pressure — in kPa to the nearest 6.9 kPa (1 psi)
12.1.13 Long-Term Aging Duration — in min to the nearest 1 min
12.1.14 Long-Term Aging Bath Temperature — in °C to the nearest 1°C

C.1.13 Key Words

Aging, asphalt concrete, asphalt-concrete aging, bituminous mixtures, bituminous paving mixtures, long-term aging, low-pressure oxidation cell.
C.2 Pressure-Oxidation Aging

C.2.1 Increase the water bath temperature to the desired testing temperature. Because this process may take several hours, it is essential to start well before testing begins.

C.2.2 When the desired temperature is reached, place the asphalt mixtures in the pressure oxygen vessels (POVs). Cover the mixtures and tighten them with 30 ft-lb of torque.

C.2.3 Apply a vacuum to the vessels for 20 min. After 20 min, close the needle valve to prevent air from entering the vessel. Connect the oxygen or compressed air line to the vessel. Add the gas to the vessel at a rate of 1 psi/min until the desired pressure is reached (100 psi or 300 psi).

C.2.4 After closing the valve, check the POV for leaks.

C.2.5 Disconnect the oxygen or compressed air line from the vessel and carefully place the POV in the water bath.

C.2.6 Monitor the temperature during the two- or seven-day aging period. Adjust the temperature if necessary [25° or 60°C (77° or 140°F)].

C.2.7 At the end of the aging period, remove the POV from the water bath. Open the valve and release the pressure very slowly. Allow the POV to cool to room temperature.

C.2.8 Remove the top cover. Gently, remove the aged samples and place them inside the 25°C (77°F) environmental cabinet.

C.2.9 Prepare the aged samples for the bulk specific gravity test. Proceed with the permeability and resilient modulus tests and, finally, conduct the tensile strength test. Record the data from the tests. Save the samples in resealable plastic bags and store them in the refrigerator for future tests.

C.2.10 Clean all components of the POV with trichloroethylene. Store the POV in the designated area.
C.3 Extended Mixing

The following is a guideline of the procedure used during the extended mixing investigation. A rolling thin film oven (RTFO) was used to continuously mix the samples for a specified time duration. Because of the available equipment and the nature of this process, only one sample designated for a specific aging time period can be produced.

C.3.1 Prior to mixing, make sure that the RTFO is at the designated temperature of either 135° or 163°C (275° or 325°F).

C.3.2 Prepare an asphalt-aggregate mix as specified in the mixing protocol.

C.3.3 Place the sample in the RTFO's tumbler and allow the mixture to mix for 10 min, 120 min (2 h), or 360 min (6 h).

C.3.4 After the extended mixing process has been completed, place the sample in a flat pan. Place the mixture in the oven designated for compaction for about 10 min. This allows the mix to achieve the designated compaction temperature.

C.3.5 When compaction is completed for either low or high voids, place the specimen in the 60°C (140°F) oven and apply the static loading (12,600 lb).

C.3.6 Extrude the sample after allowing it to cool overnight.

C.3.7 Perform all physical tests and store the samples in the refrigerator when done.