Investigation of Short-Term Laboratory Aging of Neat and Modified Asphalt Binders

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AUTHORS

Ramon Bonaquist; David A Anderson; Transportation Research Board

NATIONAL COOPERATIVE HIGHWAY RESEARCH PROGRAM

NCHRP REPORT 709

Investigation of Short-Term Laboratory Aging of Neat and Modified Asphalt Binders

David A. Anderson
State College, PA

Ramon Bonaquist
Advanced Asphalt Technologies, LLC
Sterling, VA

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FOREWORD

By Edward T. Harrigan
Staff Officer
Transportation Research Board

This report provides a proposed method of test for short-term laboratory aging of neat and modified asphalt binders using the Modified German Rotating Flask (MGRF) as an alternative to the Rolling Thin Film Oven Test (RTFOT, AASHTO T 240). Thus, the report will be of immediate interest to staff of state highway agencies, materials suppliers, and paving contractors with responsibility for specification and testing of asphalt binders.

NCHRP Project 9-36, "Improved Procedure for Laboratory Aging of Asphalt Binders in Pavements," was awarded to Advanced Asphalt Technologies, LLC, Sterling, Virginia, with major participation of consultant Dr. David A. Anderson, State College, Pennsylvania.

The objective of this research was to develop and validate a proposed procedure for the short-term laboratory aging of asphalt binders usable in a purchase specification such as AASHTO M 320 that (1) applied equally to neat and modified materials, (2) mimicked the physical changes that occur in asphalt mixes conditioned in accordance with AASHTO R 30, (3) quantified binder volatility, and (4) was extendable to long-term binder aging.

The new procedure was envisioned as a replacement for the RTFOT selected as the method for short-term asphalt binder aging during the Strategic Highway Research Program on the basis of previous experience. It was hoped that the replacement method would also provide a means to measure binder volatility (mass loss) and be suitable for adaptation to replace the Pressure Aging Vessel test (PAV, AASHTO R 28) for simulating long-term aging of asphalt binders.

In the research, existing short-term binder aging procedures, as well as procedures under development, were reviewed to select candidates for improvement and validation in NCHRP Project 9-36. Laboratory studies were then conducted to improve selected aspects of the candidate procedures. Finally, a laboratory validation study was carried out to compare rheological properties of neat and modified binders aged in the candidate procedures with binders aged in the RTFOT and mixtures aged in accordance with the conditioning procedure for performance testing in AASHTO R 30.

The research concluded that the MGRF is an acceptable alternative to the RTFOT for both neat and modified asphalt binders. Moreover, there is a reasonable relationship between rankings of aging susceptibility measured by the RTFOT and the MGRF and that measured by AASHTO R 30. Finally—and unexpectedly—for the binders tested in the research, the average aging of the neat binders was approximately the same as that for the modified binders for AASHTO R 30, RTFOT, and MGRF conditioning. The key advantage of the MGRF over the otherwise equivalent RTFOT is that the MGRF can produce aged binders in substantially larger quantities per run. However, the research was not successful in adapting the MGRF to provide the long-term aging of asphalt binders presently achieved with the PAV, and the research concluded that binder volatility is best measured using a test independent of the short-term aging procedure.

The report fully documents the research leading to the proposed MGRF method and provides the method in AASHTO format in an appendix. In addition, five appendixes are available for download from the NCHRP Project 9-36 webpage at http://apps.trb.org/cmsfeed/TRBNetProjectDisplay.asp?ProjectID=970:

- APPENDIX A: Binder Aging Bibliography
- APPENDIX B: Selection Study Report
- APPENDIX C: Volatile Collection System Study Report
- APPENDIX D: SAFT Optimization Study Report
- APPENDIX E: Verification Study Report

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Note: Many of the photographs, figures, and tables in this report have been converted from color to grayscale for printing. The electronic version of the report (posted on the Web at www.trb.org) retains the color versions.

SUMMARY

Investigation of Short-Term Laboratory Aging of Neat and Modified Asphalt Binders

The objective of NCHRP Project 9-36 was to select, refine, and validate an improved procedure for the short-term laboratory aging of asphalt binders for use in a purchase specification such as AASHTO M320, *Standard Specification for Performance-Graded Asphalt Binder.* The selected procedure would be considered to replace the Rolling Thin Film Oven Test (RTFOT, AASHTO T240) that was selected during the Strategic Highway Research Program (SHRP) based on previous experience. The following were major considerations in the selection of the improved procedure:

1. The short-term conditioning procedure should be equally applicable to neat and modified materials.
2. It should mimic the physical changes that occur in hot mix asphalt (HMA) mixes conditioned in accordance with AASHTO R30.
3. It should include a method to quantify binder volatility.
4. If possible, it should be extendible to long-term aging.

The general approach adopted for NCHRP Project 9-36 was to improve existing technologies rather than develop a completely new aging procedure. The project started with a review of existing binder aging procedures to identify viable candidate methods for possible improvement. Two viable methods were identified: the Stirred Air Flow Test (SAFT) and the Modified German Rotating Flask (MGRF). From the review it was determined that both the SAFT and the MGRF are relatively inexpensive, easy to perform, applicable to both neat and modified binders, and—based on available literature—can reasonably reproduce the level of aging that occurs in the RTFOT. However, it was not clear from the review if either test could be extended to long-term aging. Therefore, a selection study was conducted to choose one of these methods for further development. The selection study investigated whether at a temperature of 100°C either test can adequately mix air with stiff binders to produce a level of aging similar to that obtained in the Pressure Aging Vessel (PAV, AASHTO R28). From this study, the SAFT was selected for further development. The additional development for the SAFT included (1) a volatile collection system (VCS) study to design an improved system for quantifying the volatility of binders tested in the SAFT and (2) a SAFT optimization study to determine operating parameters for the commercial version of SAFT so that it would reproduce the level of aging obtained with the RTFOT for neat binders. The commercial version of the SAFT used a different heating system and control than the prototype SAFT reported in the literature.

The final study conducted in NCHRP Project 9-36 was a verification study. In this study, the properties of binders aged in both the SAFT and the MGRF were compared to properties of binders aged in the RTFOT and the properties of binders from mixtures that were

short-term oven aged in accordance with the performance testing procedure in AASHTO R30. Initially, only the SAFT was included in the study, but the study was expanded to include the MGRF. The results of the verification study were the basis for the final recommendations for short-term aging that are the primary product of NCHRP Project 9-36. The key findings from these studies are summarized below.

Selection Study

It was not feasible to develop a long-term aging version of the MGRF or SAFT. At 100°C, the maximum temperature considered viable for a long-term aging test, the MGRF does not generate a moving film and a number of attempts to modify the apparatus by adding scrapers and balls or rollers was not successful. Adequate mixing of air was also a serious problem at 100°C for the SAFT and a number of impeller designs were evaluated in order to improve the mixing efficiency and, consequently, the degree of aging. These designs improved the mixing and the rate of aging so that aging consistent with the PAV could be obtained after 40 hours, twice the duration of the current PAV test.

Volatile Collection System (VCS) Study

The SAFT included a VCS to collect volatiles from the binder during short-term aging. The VCS was considered an improvement over the current mass change procedure used in the RTFOT, but the reported low mass of volatiles collected with the VCS, one tenth of the mass loss in the RTFOT procedure, prompted a review of the VCS developed for the SAFT. This review confirmed that the original design of the VCS was inadequate and that volatiles were passing through the VCS. After considerable trial and error, a VCS based upon absorbents commonly used for chromatographic studies was found to be effective for collecting the volatiles produced during the SAFT procedure. This system includes hydrocarbon and moisture traps on the inlet side of the SAFT vessel and a 100-mm-long resin bed and molecular sieve filters on the outlet to collect hydrocarbons and water, respectively. It also was found that the majority of the volatiles collected are water, not hydrocarbons.

SAFT Optimization Study

An unexpected finding from early work with the commercial version of the SAFT was that the degree of aging in the commercial SAFT was significantly less than that obtained with the prototype SAFT. The difference was attributed to rapid aging in the prototype at the vessel wall that was in direct contact with the heating mantle. The commercial SAFT uses an oven to heat the vessel and limits the oven temperature to 176°C. This finding led to an extensive optimization study to establish operating parameters appropriate for the commercial SAFT. Operating parameters for impeller speed, airflow rate, and aging time were developed to provide a residue that best approximated the rheological properties of the RTFOT residue for PG 58-XX binders.

Verification Study

The study to verify the equivalency of the SAFT and MGRF relative to the RTFOT was conducted in the following two parts:

1. RTFOT verification experiment where rheological properties of binders aged in the SAFT and MGRF were compared to rheological properties of binders aged in the RTFOT and

2. Oven-aged mixture experiment where rheological properties of short-term aged binders were compared to properties back-calculated from oven-aged mixtures.

The RTFOT Verification Experiment included comparisons of high-temperature continuous grades, master curve parameters, and aging indices for SAFT and MGRF residues to those for RTFOT residues. These comparisons showed that the MGRF and RTFOT provided similar aging. The aging for the SAFT relative to the RTFOT was binder dependent, becoming significantly less as the binder stiffness increased.

In the oven-aged mixture experiment, properties of the short-term aged binders back-calculated from dynamic modulus tests on oven-aged mixtures were used to compare the degree of aging that occurs in the RTFOT, SAFT, and MGRF to that occurring in AASHTO R30. This experiment showed that the binder aging that occurs when a mixture is short-term conditioned in a forced draft oven for 4 hours at 135°C per AASHTO R30 generally exceeds the aging that occurs in the short-term binder aging procedures. However, there was a reasonable relationship between rankings of aging susceptibility measured by the RTFOT and the MGRF and that measured by AASHTO R30. The ranking of binders aged in the SAFT did not correlate well with the AASHTO R30 rankings.

An interesting and unexpected finding from both of the experiments in the verification study was that for the binders tested, the average aging of the neat binders was approximately the same as that for the modified binders for AASHTO R30, RTFOT, and MGRF conditioning. This finding is in contrast with other studies that have reported less aging in the RTFOT for modified binders. For the SAFT, the average aging of the neat binders was greater than that of the modified binders.

Based on the findings summarized above, the MGRF was considered an acceptable replacement for the RTFOT. For neat binders, MGRF and RTFOT conditioning produced similar rheological properties. MGRF and RTFOT conditioning also produced similar rheological properties for typical polymer-modified binders. The ranking of the aging susceptibility of binders for both the MGRF and the RTFOT correlated well with the ranking of aging susceptibility from mixtures that were short-term oven aged for 4 hours at 135°C in accordance with AASHTO R30. Although rheological properties were the same, mass change in the MGRF is less than in the RTFOT, averaging approximately 40 percent of the RTFOT mass change for the binders tested in this study. A modification to the AASHTO M320 mass change criteria would be needed if the MGRF is to be used in its current form.

The SAFT, on the other hand, is not an acceptable replacement for the RTFOT for a wide range of binders. There is a significant difference in the rheological properties of SAFT-conditioned and RTFOT-conditioned neat binders, and the difference is more apparent for higher stiffness binders. Additionally, there is poor correlation in the ranking of the aging susceptibility of binders as measured by the SAFT and as measured by oven-aged mixtures.

For the binders used in this study, AASHTO R30, the RTFOT, and the MGRF treated the neat and modified binders similarly. There was no difference in average aging indices between neat and modified binders in any of the three tests. For the specific aggregate used, AASHTO R30 aged the binders more than the RTFOT and the MGRF. The ranking of binder aging was similar for AASHTO R30, the RTFOT, and the MGRF. Future research was proposed to calibrate short-term aging procedures for binders and mixtures. The research completed in NCHRP Project 9-36 showed a difference between the short-term binder and mixture aging procedures, with the mixture procedure providing somewhat greater levels of aging. It should be possible to calibrate the binder and mixture procedures to provide similar levels of aging. This proposed research should include evaluations of plant-produced mixtures to ensure that the procedures produce representative levels of actual construction aging.

A consideration in selecting a replacement for the RTFOT was that the test, or at least the associated equipment, should show promise for future development as a replacement for the PAV. Unfortunately, it does not appear that short-term binder aging procedures can be adapted to long-term aging because it is very difficult to provide sufficient mixing of air with the binder at temperatures considered reasonable for simulating long-term aging. Attempts to modify the MGRF to improve mixing by adding scrapers and balls or rollers were not successful. Adaptation of the SAFT to long-term aging was more successful. Through changes in the design of the SAFT impeller, it was demonstrated that equivalent PAV aging could be accomplished in approximately 40 hours, twice the time required for the current PAV. As a result, it was recommended that different conditioning procedures are needed for short- and long-term aging. In general, procedures designed to expose binder to air at plant-mixing temperatures are not capable of mixing air with the binder at the lower temperatures representative of aging during the service life of the pavement.

Future research beyond NCHRP Project 9-36 was proposed to adequately address long-term aging. This research should include work with the PAV and other alternatives that may be identified in the future. It should generally be directed at establishing operating conditions for simulated laboratory aging tests that reproduce the degree of aging that occurs in field pavements for typical binders. Mirza and Witczak's Global Aging Model, while highly empirical, provides an estimate of site-specific aging based on an analysis of historical data. Work in NCHRP Project 9-23 that was reviewed during NCHRP Project 9-36 shows that the current PAV produces aged binders with viscosities that are in reasonable agreement with estimates from this model for a time of 10 years and moderate mean annual air temperature conditions. Based on this finding, the potential for the development of a long-term aging procedure that represents a reasonable period of service in the field is encouraging.

Another consideration in NCHRP Project 9-36 was an alternate to the current RTFOT mass change procedure for quantifying binder volatility. The SAFT included a VCS that used an air-cooled condenser to collected vapors produced during aging. With appropriate glassware, the MGRF also could be modified to use a similar VCS. Based on the VCS study, it was concluded that the air-cooled condenser was inadequate because it only collected a small amount of the volatile compounds generated during the test. An improved VCS that incorporates a resin bead filter and a molecular sieve that are commonly used in chromatographic studies was developed. This system can be adapted to the MGRF, but not the RTFOT. Although the redesigned VCS represents an improvement over the mass change measurement currently used in the RTFOT and the MGRF, its implementation was not proposed. Instead, consideration should be given to separating the measurement of binder volatility from the short-term aging procedure. This study clearly showed that only a small mass of hydrocarbon volatiles are collected during short-term aging. A simple mass change test performed under heat and vacuum could be developed to quantify binder volatility. The test could be developed to adapt equipment (scale, pans, and vacuum oven) already available in binder testing laboratories. It is proposed that this test be pursued for use with both the RTFOT and the MGRF.

In addition to investigating the vacuum volatility test described, further development of the MGRF was proposed. Consideration should be given to an investigation of modifying the MGRF test to allow aging of different volumes of binder. One of the advantages of the RTFOT is that it can be used to condition small quantities of binder. This has practical application in binder quality control testing and in designing mixtures with high percentages of reclaimed asphalt pavement. Consideration also should be given to conducting a formal ruggedness test for the current MGRF procedure to identify appropriate tolerance for the testing conditions. Factors that should be considered include bath temperature, rotational speed, flask submersion depth, airflow rate, flask angle, binder quantity, duration, and the need for a bath cover to better control temperature.

CHAPTER 1

Introduction

1.1 Background

The Standard Specification for Performance-Graded Asphalt Binder, AASHTO M320, includes short- and long-term laboratory conditioning procedures to address the aging that occurs in asphalt binders during construction and over the service life of the pavement. The conditioning procedures used in AASHTO M320 are (1) the Rolling Thin Film Oven Test (RTFOT, AASHTO T240) for short-term aging and (2) the Pressure Aging Vessel (PAV, AASHTO R28) for long-term aging. These procedures were selected during the Strategic Highway Research Program (SHRP) based on previous experience and limited validation studies using neat asphalt binders recovered from in-service pavements. The resources available to the SHRP asphalt research program did not allow an extensive validation of either short- or long-term binder laboratory conditioning procedures.

Experience with AASHTO M320 has shown that the RTFOT, although satisfactory for neat binders, may not be an appropriate aging procedure for modified binders. During the procedure, some users have reported that modified asphalt binder films often do not flow within the rotating bottle, violating the basic premise of the test method (i.e., that the binder is exposed to heated air in a continuously moving thin film). Users also have reported that the binder can climb out of the bottle and recovery of stiff modified binders is difficult. These problems were evaluated in NCHRP Project 9-10, Superpave Protocols for Modified Asphalt Binders. Research completed in Project 9-10 concluded that the present RTFOT does not adequately simulate the aging of modified asphalt binders that occurs during construction. Although NCHRP Project 9-10 proposed modifications to the RTFOT in an effort to improve it, the general consensus in the industry is that the test should be replaced with one that is equally applicable to neat and modified binders.

The PAV test was refined during the SHRP asphalt research program and adopted as the accelerated laboratory procedure for simulating the aging that occurs during the service life of a pavement. Elevated temperature and pressure and a single conditioning period are used in the PAV to accelerate aging. This methodology has been questioned by several researchers. It is well known that the kinetics of the binder aging process are binder specific. Thus, the degree of aging that occurs in the PAV may simulate 5 years of life in service for one binder and 10 or more years for a different binder.

To improve AASHTO M320, new procedures to simulate short-term and long-term aging that are suitable for routine specification use should be developed. In these procedures, a single apparatus, but with different operating conditions, should be considered for the replacement of both the RTFOT and PAV procedures. NCHRP Project 9-36, Improved Procedure for Laboratory Aging of Asphalt Binders in Pavements, is the first in an anticipated series of projects to address the development and validation of new procedures for short-term and long-term aging that can be included in future revisions of AASHTO M320.

1.2 Objective and Scope

The objective of NCHRP Project 9-36 was to select, refine, and validate an improved procedure for the short-term laboratory aging of asphalt binders for use in a purchase specification such as AASHTO M320. The following were considered throughout NCHRP Project 9-36:

1. The short-term conditioning procedure should be equally applicable to neat and modified materials.
2. It should mimic the physical changes that occur in hot mix asphalt (HMA) mixes conditioned in accordance with AASHTO R30.
3. It should include a method to quantify binder volatility.
4. If possible, it should be extendible to long-term aging.

This report documents the research completed in NCHRP Project 9-36.

CHAPTER 2

Research Approach

2.1 Overview

Figure 2-1 presents a flowchart for the project. The general approach adopted for NCHRP Project 9-36 was to improve existing technologies rather than develop a completely new aging procedure. The project started with a review of existing binder aging procedures to identify viable candidate methods for possible improvement. Two viable methods were identified: the Stirred Air Flow Test (SAFT) and the Modified German Rotating Flask (MGRF) (*1, 2*). Figures 2-2 and 2-3 are schematics of these two devices. The SAFT uses air blowing to simulate short-term aging of the binder. Air from a nozzle submerged in the binder is dispersed in the binder by an impeller mounted to an external motor. The SAFT also includes a condenser to collect volatile compounds that are released during the aging process. The MGRF uses a rotary evaporator similar to that specified in ASTM D5404 and AASHTO T319 for recovery of binders after solvent extraction. In the MGRF, air is introduced into the rotating flask to age the binder. The exhaust gases from the MGRF are not collected; mass change measurements are used to quantify binder volatility. From the review, it was determined that both the SAFT and the MGRF are relatively inexpensive, easy to perform, applicable to both neat and modified binders, and—based on available literature—can reasonably reproduce the level of aging that occurs in the RTFOT. However, it was not clear from the review if either test could be extended to long-term aging. The selection study was, therefore, conducted to choose one of these methods for further development. The selection study investigated whether, at a temperature of 100°C, either test can adequately mix air with stiff modified binders to produce a level of aging similar to that obtained in the PAV. From this study, the SAFT was selected for further development. The additional development for the SAFT included (1) a volatile collection system (VCS) study to design an improved system for quantifying the volatility of binders tested in the SAFT and (2) a SAFT optimization study to determine operating parameters for the SAFT so that for neat binders, it would reproduce the level of aging obtained with the RTFOT. The last study that was conducted in NCHRP Project 9-36 was the verification study. In this study, the properties of binders aged in both the SAFT and the MGRF were compared to properties of binders aged in the RTFOT and the properties of binders from mixtures that were short-term oven-aged in accordance with the performance-testing procedure in AASHTO R30. Initially, only the SAFT was included in the study, but the study was expanded to include the MGRF. The verification study served as the basis for the final proposals for short-term aging that are the primary product of NCHRP Project 9-36. Each of the studies shown in Figure 2-1 is described in greater detail in the sections that follow.

2.2 Identify Viable Candidate Methods

2.2.1 Ideal Aging Procedure for Specification Testing

A critical review of existing binder aging procedures was conducted to identify viable candidates for improvement in NCHRP Project 9-36. To guide this critical review, the following requirements for ideal short- and long-term aging procedures for specification testing in the United States were developed.

1. **Both long-term and short-term aging must be simulated.** The aging of asphalt binders, whether in the field or during accelerated laboratory aging, is a very complex process that has received considerable attention from researchers for many years. It is generally agreed that the aging process occurs in two distinct steps: (1) during construction (plant mixing, placement, and compaction) and (2) during the service life of the pavement. During construction, the aging occurs at an elevated temperature, and there is opportunity for the asphalt binder to both oxidize and to lose volatile

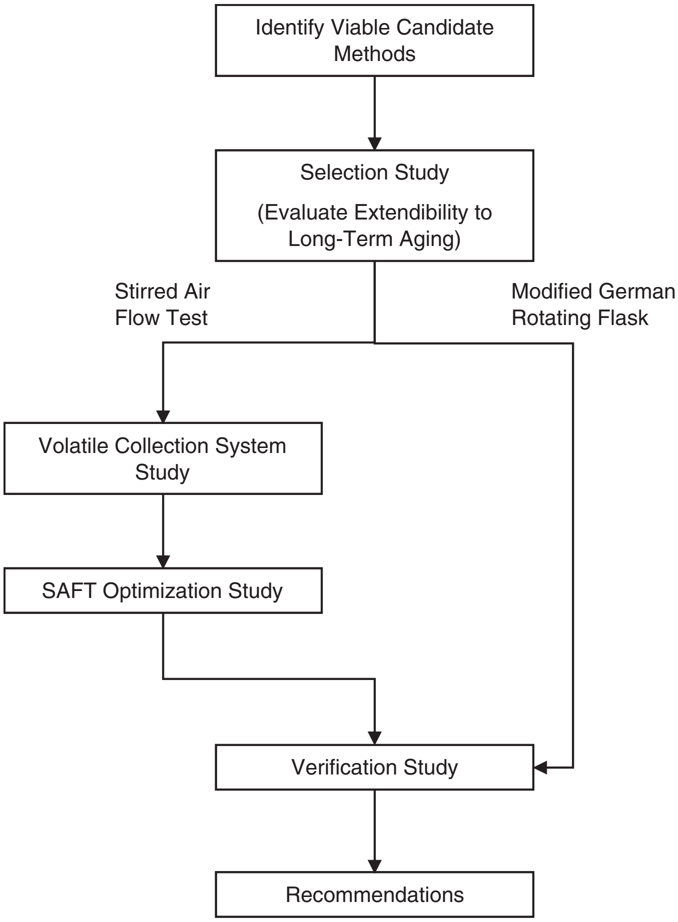


Figure 2-1. Flowchart for NCHRP Project 9-36.

compounds. In contrast, aging during the service life of a pavement occurs at a much lower temperature where oxidation is the primary aging mechanism. There is relatively little volatile compound loss during the service life of a pavement. Therefore, the ideal binder aging procedure must accommodate both the short-term aging that occurs during the construction process as well as the long-term aging that occurs during the service life of a pavement.

2. **Simulated laboratory aging must be conducted at two temperatures, one representing conditions at the hot mix plant and the other as close as possible to pavement service temperatures.** Short-term aging is simulated with the RTFOT. In this test the asphalt binder is exposed to a stream of air at 163°C, which is representative of mixing and compaction temperatures. For a pavement, maximum service temperatures range from 58° to 70°C. Research conducted during the SHRP asphalt research program clearly demonstrated that the aging mechanisms that occur in the laboratory during simulated aging change significantly when the aging temperature rises above approximately 110°C. This finding limits the extent to which temperature can be used to accelerate the simulation of long-term aging. Consequently, the short- and long-term aging must be conducted at different temperatures. Previous studies also indicate that the long-term aging mechanism, and its associated kinetics, is more reliably simulated when the accelerated aging is conducted as close as possible to the service temperature.

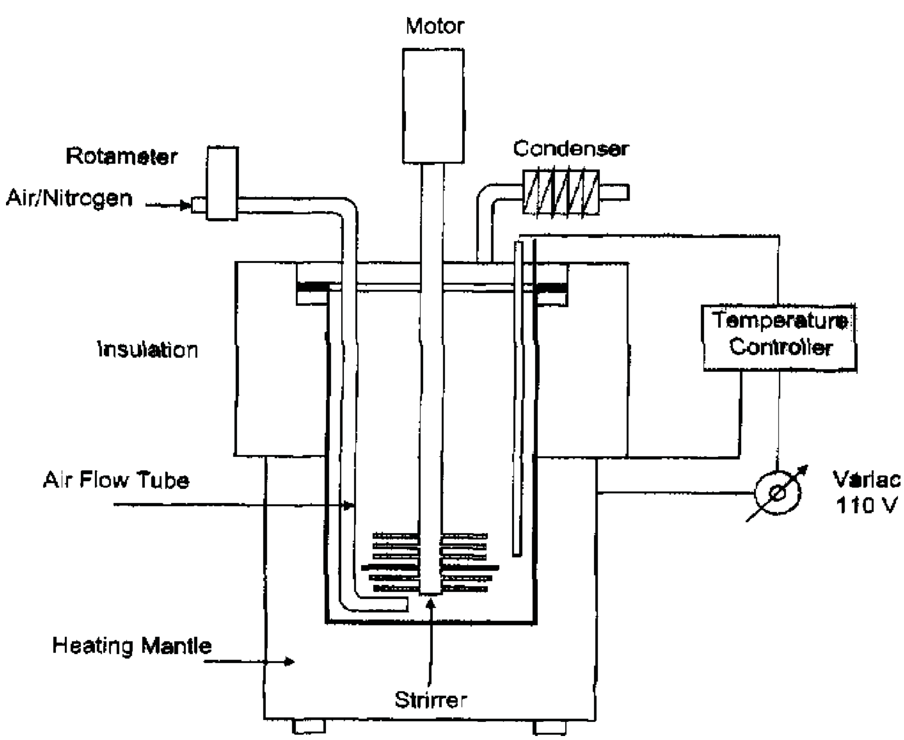


Figure 2-2. Schematic of the prototype Stirred Air Flow Test (1).

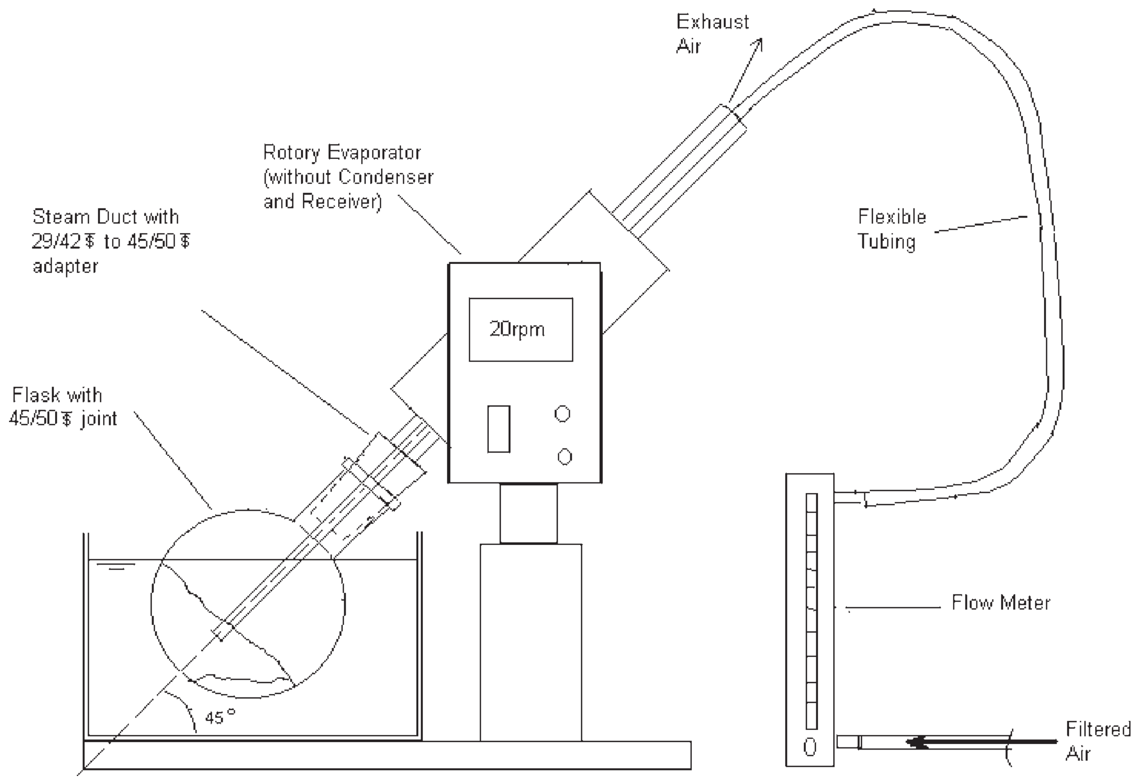


Figure 2-3. Schematic of the Modified German Rotating Flask (2).

3. **The short-term aging process must include a procedure for capturing and measuring volatile loss.** During the mixing and compaction process, some of the material of lighter molecular weight can vaporize and escape into the atmosphere. This is, in part, essentially an extension of the distillation process. As well, components—primarily oils used in the preparation of modified asphalt binders—also may vaporize and escape into the atmosphere. The RTFOT provides a means of measuring mass change, which includes the loss of mass resulting from volatilization as well as mass gain that can result from oxidation. Volatile loss is more significant to the paving industry than mass change. Volatile loss should be more directly related to "blue smoke" than mass change, wherein excessive vapors are lost to the atmosphere, causing an environmental problem. Further, volatile loss provides better control than mass change over certain undesirable refining practices (for example, when a hard base stock is "cut" with oils). Therefore, the ideal binder aging procedure should capture the volatile compounds that are lost during the simulation of short-term aging. For the purposes of a specification test, it is not necessary to characterize these materials, but simply to collect and weigh them. Including such a procedure in the short-term aging process also should be of benefit to research studies where it may be desirable to characterize the nature of the volatile compounds.
4. **Sufficient material must be available for characterizing the physical properties of the asphalt binder after short- and long-term aging.** The ideal aging procedure, as envisioned within the context of this project, will be used for specification purposes. The specifications for asphalt binders are based upon physical properties. Consequently, the ideal aging procedure must yield sufficient material to characterize the physical properties of the asphalt binder. Adequate material must be available after the short- and long-term aging steps.
5. **The long-term aging must be completed within 46 hours.** Whereas the short-term aging can be conducted at temperatures representative of the hot mix plant and at realistic times (less than 4 hours), the long-term aging must be accelerated by some means in order to meet the needs of both the producers and users of the asphalt binder. Currently, both pressure and temperature are used to accelerate the aging process in the PAV. This results in a 20-hour test. During the SHRP asphalt research program, a 144-hour test conducted at 60°C was proposed. Industry strongly objected to this protocol and, consequently, the aging temperature was increased to 100°C. It is clear that a specification test that can be used for quality control and acceptance purposes, and that is applicable to U.S. practice, cannot be of 1-week duration. Recognizing that there must be a compromise between test reliability and

test length, the long-term aging test must be completed within approximately 46 hours (2-day cycle time for equipment). As discussed with the NCHRP project panel, limiting the long-term aging procedure to a 1-day test may be too restrictive in terms of developing a reliable long-term aging test. On the other hand, a test that requires more than 2 days to complete will be unacceptable to the industry.

6. **The short- and long-term aging procedure must be applicable to both neat and modified asphalt binders.** One of the primary reasons for NCHRP Project 9-36 is that the RTFOT is not applicable to some modified binders. Some modified asphalt binders do not roll uniformly within the bottle during the test, thereby negating a basic assumption of the test (i.e., that during the test the asphalt binder is exposed, in the form of a thin film, to the stream of air). A second problem with the RTFOT is that some modified binders tend to "crawl" out of the bottle during the test. These problems were addressed during NCHRP 9-10 by placing steel rods in the RTFOT bottles. Subsequent work by FHWA showed that the steel rods were not effective. During the long-term aging process, the asphalt binder must form a film, or be stirred in a manner that is equally applicable to neat or modified binders. This is especially of concern during the simulation of long-term aging where oxidation is the aging mechanism. In order for the long-term test to be equally applicable to neat and modified binders, the availability of oxygen must be independent of the type of binder.
7. **The long-term aging test must accommodate binder- and modifier-specific aging kinetics.** The rate of hardening, whether in the field or in the laboratory under accelerated conditions, is binder-specific. Different binders are affected differently by changes in temperature or length of exposure during accelerated long-term aging. In other words, aging kinetics are binder-specific. The current PAV test may simulate the aging that occurs in 5 years for one binder and 10 years for another binder. The fact that the PAV test does not treat all binders equally was well known to researchers during the SHRP asphalt research program. To further complicate the matter, aging kinetics are also modifier-specific and differ from the aging kinetics of asphalt binders. To characterize aging kinetics, physical property measurements must be made at multiple times or temperatures, or a combination thereof. Although it is unrealistic to expect that a complete characterization of aging kinetics will be part of a future specification, the ideal long-term aging test should, as a minimum, provide for sampling at multiple aging times. During the SHRP asphalt research program, researchers noted that the aging process caused a shift in the rheological master curve as opposed to a change in the shape of the rheological master curve. This was demonstrated with both laboratory- and field-aged materials. Thus, aging kinetics can be captured by using a very limited rheological testing protocol. The ideal aging procedure will allow sampling at multiple aging times so that aging kinetics can be included in a future specification.
8. **The aging test procedure must not expose the operators to unsafe or hazardous conditions.** Safety is a key issue that must be considered in any laboratory procedure. Of primary concern with respect to an ideal aging test is the aging atmosphere during the long-term test. Some researchers have suggested the use of pure oxygen as the aging environment, but there are concerns over the safety of the use of pure oxygen in commercial laboratories. Further, the use of high pressure, regardless of atmosphere, is to be avoided if possible.
9. **Equipment for the aging procedure must have a reasonable cost, be reliable, easy to operate and clean, and be configurable for both the short-and long-term aging procedures.** There are a number of other features that an ideal procedure must embody. Without elaborating, the equipment must be reasonably priced, reliable, easy to operate, and easy to clean (especially with respect to the use of solvents). Although the operating conditions for short-term aging and long-term aging must be different, this does not necessarily militate against the use of the same equipment for both short-term aging and long-term aging. Various scenarios are possible, and the same equipment might be used for both long-term and short-term aging, but the temperature may be changed. On the other hand, the equipment may be the same but operated under different conditions as, for example, a container switched between replicate devices operated at different temperatures.

A summary listing of the key features of the ideal aging procedure for specification testing is presented as follows. This list was used to guide the review of existing tests and the selection of viable procedures. The ideal aging procedure should

- Simulate both long-term and short-term aging;
- Be performed at two temperatures, one representing conditions at the hot mix plant and the other representing, as close as possible, service temperatures;
- Include a procedure for capturing and measuring volatile loss during short-term aging;
- Provide sufficient material for characterizing the physical properties of the binder after short- and long-term aging;
- Complete long-term aging within 46 hours;
- Be applicable to both neat and modified asphalt binders;
- Accommodate binder- and modifier-specific aging kinetics in the long-term aging;
- Be safe and technician-friendly; and

- Use reasonably priced equipment that can be shared between the short- and long-term procedures.

2.2.2 Review and Selection Process

A three-step process was used to select promising procedures for further evaluation in NCHRP Project 9-36. The first step included two activities that were conducted simultaneously at the beginning of the project. These were the development of the requirements of the ideal aging procedure for specification testing as discussed previously in Section 2.2.1, and a review of literature and research in progress associated with binder aging methods. Excellent literature reviews on the aging of asphalt binders and mixtures were conducted during the SHRP asphalt research program (*3–5*). These formed the basis for the expanded literature review performed during this study. Approximately 90 references that post-date the SHRP asphalt research were identified and reviewed. Much of this information was published in the European literature, especially that of Eurobitume and RILEM. Several ongoing studies associated with the development of improved binder aging methods were identified, and contact was made with the principals conducting these studies. The work in progress that was reviewed included

- Efforts at the Western Research Institute under FHWA Contract DTFH61-99-C-00022, "Fundamentals of Asphalts and Modified Asphalts II," associated with the chemistry of binder aging;
- FHWA studies to evaluate the Modified Rolling Thin Film Oven Test, MGRF, and SAFT;
- Texas Department of Transportation studies associated with the development and implementation of the SAFT;
- Activities in the European Committee for Standardization (CEN) Task Group on Binder Aging (CEN TC336 SG1 TG3); and
- Shell Global Solutions' research associated with the development of the Long-Term Rotating Flask Test.

Appendix A (available on the TRB website) presents a comprehensive bibliography related to binder aging that was assembled by adding the references identified in this project to the unpublished literature review prepared during the SHRP asphalt research (*3*). Relevant findings from the literature review and the review of research in progress are presented in Chapter 3 of this report.

The second step in the evaluation and selection process was the selection of candidate procedures from those identified through the literature review and the review of research in progress. Procedures based on (1) microwave technology, (2) thin films, and (3) air blowing were identified in the review of the literature and research in progress. Consideration of the fundamental aging process in these three approaches and the requirements of the ideal aging procedure for specification testing were used to select approaches and candidate procedures for further consideration. This step eliminated microwave technology.

The third step in the evaluation and selection process consisted of a detailed comparison of candidate procedures based on the two viable approaches with the requirements of the ideal aging procedure. This step also included the consideration of a hybrid procedure combining meritorious elements of the various procedures reviewed. From this step, two existing procedures, the MGRF and the SAFT, were identified as promising methods for further consideration.

2.3 Selection Study

The objective of the selection study was to evaluate whether the two promising short-term aging tests, the MGRF and the SAFT, can be extended to long-term aging. The extension of either of these to a low-temperature, long-term aging test using air at atmospheric pressure depends on the mixing efficiency of the device. The selection study evaluated the mixing efficiency of the devices at conditions representative of those that should be used in a long-term aging test. In this study, the level of aging obtained for long-term conditions was compared to that obtained in the PAV. Table 2-1 presents the conditions selected by the research team for developing prototype versions of long-term aging tests based on the MGRF and the SAFT.

The selection study was conducted in two parts. The first part of the study was an assessment of various modifications that could be made easily to the MGRF and the SAFT to produce prototype long-term versions of these tests. The goal in this effort was to obtain approximately the same degree of aging that occurs in the PAV subject to the constraints on temperature, atmosphere, and time given in Table 2-1. The binders used in this part of the study were a neat PG 58-28, a styrene-butadiene-styrene (SBS) modified PG 82-22, and a low-density polyethylene (LDPE) modified PG 76-22. Table 2-2 presents the test conditions that were varied during this first part of the selection study. The suitability of each of the various configurations was assessed based on

- The degree of aging obtained relative to the PAV for the PG 58-28 and the PG 82-22,

Table 2-1. Summary of conditions for long-term aging used in the selection study.

Condition	Value
Temperature	100°C max
Atmosphere	Air at atmospheric pressure
Duration	< 48 hours
Quantity	Per current short-term testing protocol
Degree of Aging	Approximate PAV aging

Table 2-2. Summary of conditions investigated during the selection study.

Test	Conditions Investigated
Modified German Rotating Flask	Morton versus smooth flask Rotational speed Mixing enhancers Scrapers
Stirred Air Flow Test	Impeller type Position of air supply Rotational speed of impeller

- Visual assessment of the degree of mixing during the test,
- Visual assessment of separation for the two polymer modified binders, and
- Potential for implementation as a specification test.

The second part of the selection study was a formal experiment designed to address whether the degree of aging in the prototype long-term versions of the tests was affected by the large differences in viscosities for neat and modified binders at the selected aging temperature of 100°C. To emphasize the significance of the "viscosity effect," in the PAV condition, the unmodified binder has the consistency of light cream whereas the modified binder has the consistency of molasses. Versions of the long-term tests judged successful based on the first part of the selection study were subjected to this formal experiment. In this experiment, the PG 58-28 and the SBS-modified PG 82-22 were aged in the prototype long-term version of the test and in the PAV. Rheological measurements at high, intermediate, and low pavement temperatures were used to compare the level of aging to that produced by the PAV. Replication was included in this experiment to permit statistical analysis of the differences in aging that were observed.

Based on the results of the selection study, the SAFT was chosen for further development as an improved procedure for short-term aging of binders. Relevant findings from the selection study are presented in Chapter 3 of this report. The selection study is documented in detail in Appendix B (available on the TRB website).

2.4 Volatile Collection System Study

The prototype SAFT included a volatile collection system (VCS), which consisted of a copper coil condenser operated at ambient temperature. Data published for several binders during the development of the SAFT showed that the mass of volatiles collected was a factor of 10 lower than the mass change in the RTFOT (*1*). Since mass change during the RTFOT includes mass loss due to volatilization and mass gain due to oxidation, the RTFOT mass change was expected to be less than the mass of the volatiles collected with the SAFT. Several possible causes for this discrepancy were identified including

1. Condensation of volatile compounds on the lid of the SAFT before they entered the air-cooled condenser,
2. Inefficiency of the air-cooled condenser allowing volatiles to pass completely through the VCS,
3. Production of fewer volatile compounds in the SAFT compared to the RTFOT due to the shorter duration of the SAFT conditioning procedure and the lower airflow rate used in the SAFT,
4. Rapid saturation of the small air bubbles produced in the SAFT with volatiles so that they are not able to absorb additional volatiles as they move upward through the binder, and
5. Suppression of volatilization caused by the build up of air pressure in the SAFT.

The VCS study consisted of a series of small experiments to evaluate these potential causes and to design a more effective VCS for the SAFT. The product of the VCS study was an improved VCS system employing reusable adsorbents that are commonly employed for chromatographic analyses. Findings from the VCS study are presented in Chapter 3. The VCS study is documented in Appendix C (available on the TRB website).

2.5 SAFT Optimization Study

During NCHRP Project 9-36, the Texas Department of Transportation contracted with James Cox and Sons, Inc., to produce a commercial version of the SAFT. Figure 2-4 shows photographs of the prototype and commercial versions of the SAFT. The major difference between the prototype and commercial versions is how the binder in the aging vessel is heated. In the prototype device, the aging vessel was heated by direct contact with a heating mantle, while the commercial version used an oven to heat the aging vessel. Because the aging vessel was in direct contact with the heating mantle in the prototype, the aging vessel and the binder in contact with it were exposed to very high temperatures during the heat-up portion of the test. In the commercial version, the oven was limited to a temperature approximately 13°C above the test temperature, so the temperature of the aging vessel and the binder in contact with it were much lower. This resulted in less aging in the commercial version compared to the prototype for the same operating parameters. The difference in aging between the prototype and commercial versions of the SAFT necessitated a study to optimize the operating parameters of the commercial version of the SAFT to reproduce the aging from the RTFOT for neat binders. This study was called the SAFT optimization study.

The SAFT optimization study included two parts. The first part was a series of tests to verify that the heat-up phase of the test does not result in significant aging of the binder. During the heat-up phase, the temperature of the binder is increased from approximately 100°C to the testing temperature of 163°C

Figure 2-4. Prototype SAFT (left) and commercial SAFT (right).

while nitrogen flows through the SAFT vessel. Since the starting temperature (temperature of the binder after charging the SAFT vessel) cannot be accurately controlled, it is critical that the heat-up phase not contribute significantly to the degree of aging that occurs in the test. The second component of the SAFT optimization study was an experiment to determine the effects of impeller speed, airflow rate, and test duration on the degree of aging measured by the high pavement temperature rheology of the binder. From this component of the SAFT optimization study, operating parameters for the commercial SAFT were selected. These operating parameters were used in the verification study described in the next section. Findings from the SAFT optimization study are presented in Chapter 3. The SAFT optimization study is documented in Appendix D (available on the TRB website).

2.6 Verification Study

The objective of the verification study was to (1) verify that the commercial version of the SAFT and the MGRF reproduce the degree of aging obtained in the RTFOT for a wide range of neat binders and (2) compare the aging from the SAFT, MGRF, and RTFOT with that from mixture samples aged in a forced draft oven in accordance with the performance testing protocol contained in AASHTO R30. Initially, the verification study only included the SAFT, but it was expanded at the request of the NCHRP project panel to include the MGRF.

The verification study consisted of two parts: (1) the RTFOT verification experiment and (2) the oven-aged mixtures experiment. The RTFOT verification experiment, which included dynamic shear rheometer (DSR) and bending beam rheometer (BBR) measurements, was designed to provide master curves for the binders in the tank condition and after SAFT, MGRF, and RTFOT conditioning. These binder master curves served two purposes: (1) to allow a comparison of the rheological properties of material conditioned in the SAFT, MGRF, and RTFOT and (2) to allow a comparison of the master curves measured for the binders and the master curves back-calculated from mixture properties. In the oven-aged mixtures experiment, hot mix asphalt was prepared with the binders from the RTFOT verification experiment and aged in accordance with the performance testing protocol in AASHTO R30. Dynamic modulus master curve tests were performed on the mixture samples. From the mixture modulus master curves, the binder stiffness was estimated using the Hirsch Model (*6*) and compared to the measured stiffnesses obtained from the SAFT,

Table 2-3. AASHTO M320 properties for the neat binders used in NCHRP 9-36.

Condition	Test	Method	CITGO 58-28	AAC-1	AAD-2	AAF-1	AAM-1	ABL-1	ABM-2
Unaged	Viscosity at 135°C, Pa·s	AASHTO T316	0.23	0.18	0.26	0.34	0.55	0.42	0.19
	Temperature for G*/sinδ of 1.00 kPa at 10 rad/s, °C	AASHTO T315	59.7	56.5	57.0	65.4	67.7	68.5	62.7
RTFO Aged Residue	Mass Change, %	AASHTO T240	-0.358	-0.058	-1.058	-0.008	+0.122	-0.654	-0.348
	Temperature for G*/sinδ of 2.20 kPa at 10 rad/s, °C	AASHTO T315	59.8	56.3	65.6	67.0	68.0	69.5	60.7
PAV Aged Residue	Temperature for G*sinδ of 5000 kPa at 10 rad/s, °C	AASHTO T315	13.0	17.7	11.9	27.2	19.0	17.5	25.2
	Temperature for Creep Stiffness of 300 MPa, at 60 s, °C	AASHTO T313	-20.1	-18.1	-23.7	-12.2	-16.6	-19.6	-6.0
	Temperature for m-value of 0.300 at 60 s, °C	AASHTO T313	-23.1	-16.7	-24.6	-9.8	-11.3	-19.9	-10.2
M320 Grade			58-28	52-22	52-28	64-16	64-16	64-28	58-16

MGRF, and RTFOT. The verification study was the last study conducted in NCHRP Project 9-36 and provided the basis for making the final recommendation with respect to a replacement for the RTFOT. Findings from the verification study are presented in Chapter 3. The verification study is documented in Appendix E (available on the TRB website).

2.7 Materials

2.7.1 Binders

NCHRP Project 9-36 used 13 different asphalt binders: seven neat binders and six modified binders. The binders were selected to provide a wide range of physical and chemical properties and modification processes. Tables 2-3 and 2-4 present AASHTO M320 properties for the binders used in Project 9-36.

The neat binders included a PG 58-28 from the Paulsboro, New Jersey, refinery of the Citgo Asphalt Refining Company (Citgo) and six binders (AAC-1, AAD-2, AAF-1, AAM-1, ABL-1, and ABM-2) from the FHWA Materials Reference Library (MRL). Detailed information on the chemical composition of the MRL binders is contained elsewhere (*7*).

The modified binders were selected to represent a range of binder grades, modifiers, and modification processes. These included an airblown binder, two binders modified with SBS, one binder modified with Elvaloy, one binder modified with LDPE, and one binder modified with ethyl vinyl acetate (EVA). All of the modified binders except the EVA binder were obtained from commercial sources. A commercial source supplying EVA-modified binders for paving applications could not be identified; therefore, the EVA binder was produced in the laboratory of Advanced Asphalt Technologies, LLC, by modifying a PG 64-22 binder from the Paulsboro, New Jersey, refinery of Citgo with 7 percent by weight Repsol Quimica PA-420. The Repsol Quimica PA-420 was obtained from Momentum Technologies, Inc. The airblown binder graded as a PG 76-16. It was obtained from Western Refining's El Paso, Texas, refinery. Citgoflex is an SBS-modified binder produced in several grades by Citgo. The Citgoflex binder used in NCHRP Project 9-36 was a PG 82-22 grade and was obtained from Citgo's Paulsboro, New Jersey, refinery. The second SBS-modified binder was that included in the FHWA Pooled Fund Study TPF-5(019), *Full Scale Accelerated Performance Testing for Superpave and Structural Validation.*

Table 2-4. AASHTO M320 properties for the modified binders used in NCHRP 9-36.

Condition	Test	Method	Airblown	Citgoflex	ALF	Elvaloy	Novophalt	EVA
Modifier			Airblown	SBS	SBS	Elvaloy	LDPE	EVA
Unaged	Viscosity at 135°C, Pa·s	AASHTO T316	0.79	2.94	1.20	0.65	1.72	2.07
	Temperature for G*/sinδ of 1.00 kPa at 10 rad/s, °C	AASHTO T315	77.3	88.5	73.6	67.4	77.9	82.9
RTFO Aged Residue	Mass Change, %	AASHTO T240	+0.031	-0.196	-0.207	-0.173	-0.132	-0.132
	Temperature for G*/sinδ of 2.20 kPa at 10 rad/s, °C	AASHTO T315	80.3	85.2	75.4	69.9	78.9	81.0
PAV Aged Residue	Temperature for G*sinδ of 5000 kPa at 10 rad/s, °C	AASHTO T315	22.2	23.6	5.8	16.0	22.0	16.5
	Temperature for Creep Stiffness of 300 MPa, at 60 s, °C	AASHTO T313	-17.2	-14.5	-29.8	-19.1	-14.2	-15.4
	Temperature for m-value of 0.300 at 60 s, °C	AASHTO T313	-8.1	-14.1	-27.1	-18.6	-11.1	-12.9
M320 Grade			76-16	82-22	70-34	64-28	76-16	76 –22

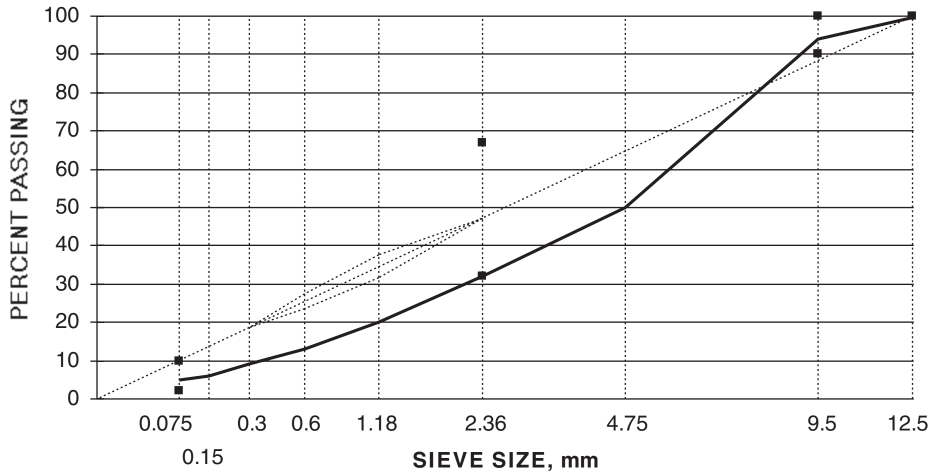


Figure 2-5. Gradation of the 9.5-mm limestone mixture.

This binder graded as a PG 70-34. The Elvaloy binder was provided by Mathy Construction. It graded as a PG 64-28. Finally, the Novophalt binder was provided by Advanced Asphalt Technologies, LLC. This binder contains recycled LDPE and graded as a PG 76-16.

The Citgo PG 58-28 was used in the selection study, the VCS study, and the SAFT optimization study. MRL binders AAD, AAM, and ABL2 were used in the VCS study and the SAFT optimization study. The Citgoflex and Novophalt binders also were used in the selection study. All of the MRL binders and all of the modified binders were used in the verification study.

2.7.2 Aggregate

Because research completed during SHRP showed a relatively minor effect of aggregate type on short-term mixture aging (*4*), only one aggregate and one gradation were used in the oven-aged mixture experiment of the verification study. The aggregate was a limestone from Frazier Quarry, Incorporated's North Quarry in Harrisonburg, Virginia. This aggregate was used extensively in various mixture volumetric studies completed in NCHRP Projects 9-25 and 9-31 (*8*). Figure 2-5 shows the gradation of the coarse graded 9.5-mm nominal maximum aggregate size mixture that was used. The optimum binder content for this mixture was selected using a PG 64-22 binder. Pertinent volumetric properties at the optimum binder content are summarized in Table 2-5. For the oven-aged mixture experiment, mixtures were produced using the six MRL binders and the six modified binders from Tables 2-3 and 2-4. The same binder content of 5.5 percent was used in each of the 12 mixtures produced.

Table 2-5. Volumetric properties for the 9.5-mm limestone mixture.

Property	Sieve Size, mm	Value
Gradation	12.5	100
	9.5	94
	4.75	50
	2.36	32
	1.18	20
	0.600	13
	0.300	9
	0.150	6
	0.075	5.1
Asphalt Content, %		5.5
N_{design}		100
VMA, %		15.8
VFA, %		72.6
Dust/Binder Ratio (Weight Basis)		1.0
Fine Aggregate Angularity, %		46.0
Coarse Aggregate Angularity, %		100/100
Flat and Elongated Particles, %		0.8
Sand Equivalent		75
Surface Area, m^2/kg		4.40

CHAPTER 3

Findings

3.1 Introduction

This chapter presents key findings from the five major studies conducted in NCHRP Project 9-36 that were described in Chapter 2: (1) identify candidate methods, (2) selection study, (3) VCS study, (4) SAFT optimization study, and (5) verification study. Each of these studies is documented in detail in an appendix to this report (see TRB website). Conclusions and proposals based on these findings are presented in Chapter 4.

3.2 Identify Candidate Methods

3.2.1 Post-SHRP Approaches Used for Laboratory Aging of Asphalt Binders

The review of the literature and research in progress identified three approaches for laboratory aging of binders that have been pursued by various researchers in the United States and abroad since the completion of the SHRP asphalt research program. The three approaches are

- Microwave technology, which increases the energy level of the binder molecules and the bulk temperature of the binder, thereby accelerating the oxidation process;
- Various techniques for producing thin films of binder and thereby increasing the availability of oxygen and the opportunity for volatilization; and
- Air blowing, which increases the availability of oxygen and the opportunity for volatilization.

This section discusses the viability of using each of these approaches in an improved aging procedure considering the state of knowledge of the chemistry of binder aging and the requirements of an ideal aging test developed in this project.

3.2.1.1 Microwave Technology

A significant amount of work has been completed by Bishara and his colleagues on developing short- and long-term aging procedures based on microwave technology (*9–13*). Early work used a standard household microwave oven to age a 10-g sample of binder. Based on the performance grading system high temperature parameter, G*/sin δ, 33 minutes of microwave treatment without pressure was found to be equivalent to Thin Film Oven Test (TFOT) aging, but 63 minutes was required to reproduce RTFOT aging. A total of 158 minutes of microwave treatment without pressure was found to be approximately equivalent to the combination of TFOT plus PAV aging based on the performance grading system intermediate temperature parameter G*sinδ. In later work, a MARS5 scientific microwave produced by CEM Corporation with temperature and pressure control was used for the long-term version of the test. In the latest protocol for long-term aging, 66 g of binder are aged at 135°C in a scientific microwave unit under 3,200 kPa (460 psi) of air pressure for 190 minutes. Comparison of rheological properties between long-term microwave aging and RTFOT plus PAV aging has shown good agreement.

For Project 9-36, the principal concern with basing improved binder aging methods on microwave technology is a lack of understanding of the mechanism whereby microwave energy accelerates the aging process. Bishara and McReynolds (*9*) describe how microwave energy is dissipated as heat in an asphalt sample, but its effect on volatile loss and oxidation reactions has not been studied. For long-term aging, high pressures are needed to accelerate the aging process, which is one of the major criticisms of the PAV. Approximate cost of the CEM microwave unit is $20,000. Throughput and the inability to measure volatile loss also are significant concerns for the microwave approach. Because the microwave aging mechanism is not well understood and microwave technology must rely on high pressures to simulate long-term aging, it was not considered a viable approach for further development in Project 9-36.

3.2.1.2 Thin Films

Laboratory aging of binders in thin films at elevated temperature has been the method of choice of asphalt technologists for over 60 years. An asphalt binder's exposure to air during mixing and in asphalt concrete mixtures is in the form of a thin film. The current RTFOT was developed in California in response to the need for a short-term aging test (*14*). In an attempt to simulate long-term aging, Griffin and his colleagues (*15*) performed extensive studies with very thin films and developed a special viscometer to characterize the viscosity of the binder (*16–17*). More recently, Petersen (*18*) reported on the Thin Film Aging Test in which thin films of asphalt binder are exposed to the atmosphere at service temperatures. The disadvantage of these tests, which may be considered as ultra-thin film tests, is that they yield a very small quantity of material, making them impractical for specification use. Thin film aging is the approach used in the Thin Film Oven Test, (AASHTO T170), the Rolling Thin Film Oven Test (AASHTO T240), the Pressure Aging Vessel (AASHTO R28), and two European methods (the German Rotating Flask and the Rolling Cylinder Aging Test). Welborn (*19*) presented an excellent summary of the development of the various U.S. laboratory aging tests based on thin films. The primary difference in all of these methods is the thickness of the film and the method used to obtain the thin film.

Methods based on thin films have been used extensively to simulate both short- and long-term aging. Our current knowledge of the chemistry of binder aging is founded in the interpretation of large amounts of data obtained using this approach (*20*). Clearly, improved methods based on thin films should be considered as viable approaches for NCHRP Project 9-36.

3.2.1.3 Air Blowing

Air blowing was one of the first approaches recommended as a laboratory aging test for asphalt binders. In 1937, Nicholson (*21*), and Rashig and Doyle (*22*) proposed short duration, high temperature, high airflow tests for accelerated aging of asphalt binders. In these tests, a 250-g sample of asphalt was aged at 218°C for 15 minutes with an airflow rate of 9 L/min. Later, Skidmore (*23*) proposed a longer, lower temperature air blowing test that aged a 100-g sample at 177°C for 2 hours using an airflow rate of 1 L/min. Although these tests reasonably reproduced the ductility and penetration of asphalts recovered from recently constructed pavements, an air blowing test for simulating short-term aging was never standardized. Little work was done using this approach after 1940 until the development of the Stirred Air Flow Test (SAFT) by Glover et al. (*1*) in 2001.

Laboratory aging using thin films and air blowing are mechanically similar, as long as the air bubbles remain small and well dispersed. Smaller air bubbles increase the ratio of surface area to volume of the asphalt, and the reaction becomes less oxygen-diffusion limited and behaves more like a thin film reaction. The primary concern with air blowing is whether air bubbles can be dispersed in highly viscous modified binders at temperatures considered reasonable for a long-term aging test. There also is concern regarding the partial pressure of the volatiles within the bubbles and whether the thermodynamics of volatile transfer to the atmosphere is duplicated within the closed bubbling system. However, since air blowing produces oxidation reactions that are similar to those of thin films, air blowing was considered a viable approach for Project 9-36.

In summary, the review of the literature and research in progress identified significant post-SHRP research on binder aging methods using the three approaches of microwave technology, thin films, and air blowing. Of these three, microwave technology was eliminated because the mechanism whereby microwave energy accelerates the aging process is not well understood, and high pressures are needed for simulation of long-term aging. The use of thin films for laboratory aging is well established and accepted by asphalt technologists. Air blowing is similar to thin film reactions, provided the air bubbles remain small. Candidate methods for an improved short-term binder aging test based on thin films and air blowing are discussed in the following section.

3.2.2 Methods Based on Viable Approaches

3.2.2.1 German Rotating Flask

The German Rotating Flask (GRF) is the common name given to German Standard DIN 52 016, *Testing the Thermal Stability of Bitumen in a Rotating Flask*. This test was developed in Germany as an inexpensive alternative to the RTFOT. It uses a rotary evaporator similar to that used in ASTM D5404 and AASHTO T319 for recovery of binders after solvent extraction. Figure 2-3 showed a schematic of the GRF. In the test, air is introduced into the rotating flask to age the binder. A 100-g sample is aged in the rotating flask at 165°C for 160 min, 10 min without airflow, followed by an additional 150 min with air at ambient temperature flowing at the rate of 500 mL/min.

Three studies to evaluate the GRF as an alternative short-term aging procedure have been conducted in the United States. Sirin et al. (*24*) and Tia et al. (*25*) evaluated the test for the Florida Department of Transportation for use with modified asphalts that could not be properly aged in the RTFOT. They concluded that the GRF could be used to simulate short-term aging in hot mix plants and that various degrees of aging could be obtained by varying the temperature, airflow, duration, and sample weight used in the test. They recommended a cover for the oil bath and the use of a Morton flask to better control temperature and provide uniform mixing of modified binders. They also recommended testing conditions to approximately reproduce the aging that occurred in the TFOT (AASHTO T170) and the RTFOT. Reported mass changes for the modified test incorporating the cover

Table 3-1. Properties used in the MGRF optimization.

Property	Conditions	Value
G*/sinδ	Short-term aged	Temperature at 2.2 kPa
G*sin δ	PAV aged after short-term aging	Temperature at 5,000 kPa
S	PAV aged after short-term aging	Temperature at 200 MPa
m-value	PAV aged after short-term aging	Temperature at 0.300
Mass Loss	Short-term aged	Report

and Morton flask, and using an airflow rate of 4 L/min, were greater than those for the RTFOT.

At the same time as the Florida studies, the Western Research Institute under FHWA's contract, "Fundamentals of Asphalts and Modified Asphalts," initiated a study to modify the German Standard DIN 52 016 to simulate RTFOT aging (*2*). The study included four phases and ultimately led to the development of a draft AASHTO standard test method. In each of these phases the properties listed in Table 3-1 were compared for binders short-term aged in various modifications to the GRF and the RTFOT. In the first phase, the method was altered to test 200 grams of binder using a 2-L round-bottom flask at 165°C with a flow rate of 500 mL/min. Eight Materials Reference Library (MRL) asphalts (AAA-1, AAB-1, AAC-1, AAD-1, AAF-1, ABM-1, AAK-1, and AAM-1) were aged in the Modified German Rolling Flask (MGRF) with split samples aged in the RTFOT. Comparisons of rheological properties and mass change revealed that the MGRF consistently aged binders less than the RTFOT. In the second and third phases the airflow was increased to 1 L/min, and 2 L/min, respectively. The results were better, but the degree of aging was still less than in the RTFOT. Based on these results, the round-bottom flask was replaced with a 2-L Morton flask to increase the surface area of the binder during the aging process. All eight MRL asphalts plus three modified binders (Styrelf, Ultrapave, and Novophalt) were aged using the Morton flask at 2 L/min airflow during the fourth phase of testing. The resulting rheological property data are shown in Figure 3-1 for the neat binders. The agreement for the two methods is excellent over a very wide range of temperatures. Mass change also was considered in the study. Figure 3-2 compares mass change data from the MGRF and RTFOT. The MGRF produced less mass loss than the RTFOT, particularly for binders with high mass losses. Table 3-2 summarizes the operating conditions for the MGRF.

Ramaiah and D'Angelo have conducted a study for FHWA to further evaluate the MGRF (*26*). This project included studies to assess the effect of variations in the operating parameters to establish initial tolerances, and a study to compare short-term aging from the MGRF with the RTFOT for five polymer modified binders, one air blown binder, and three neat binders. The tolerances established from this study are included in

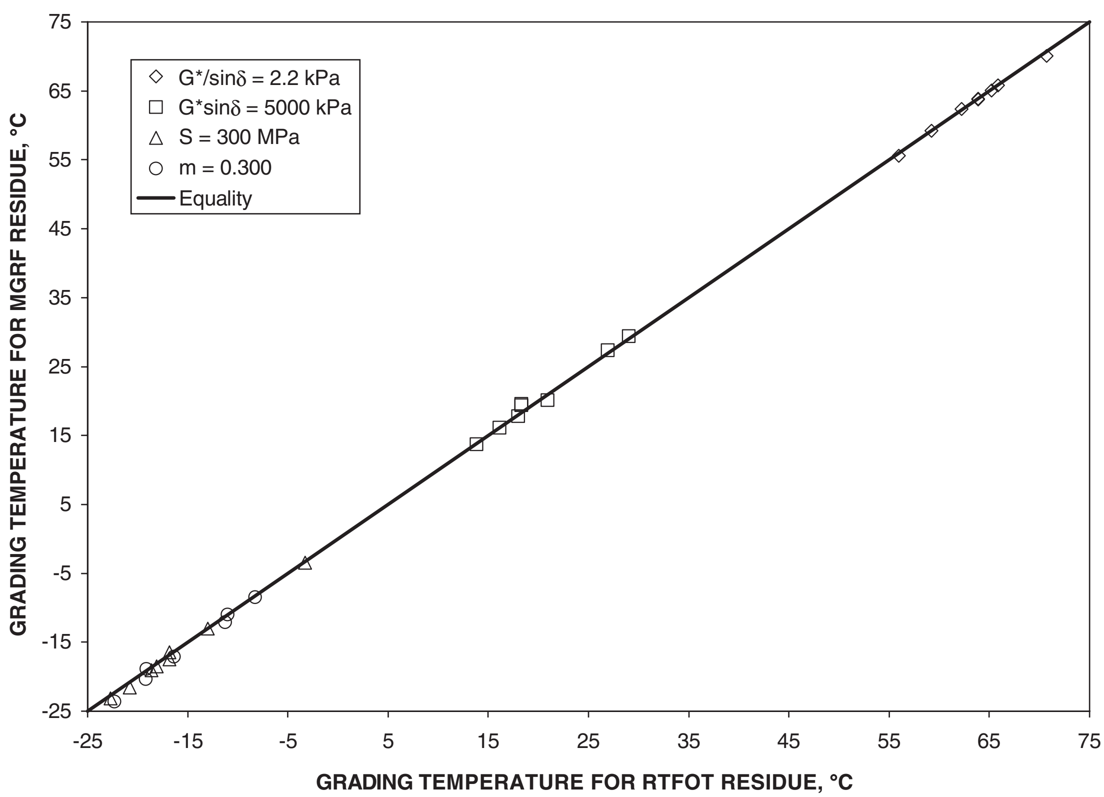


Figure 3-1. Comparison of continuous grade temperatures from the MGRF and RTFOT for eight MRL asphalts.

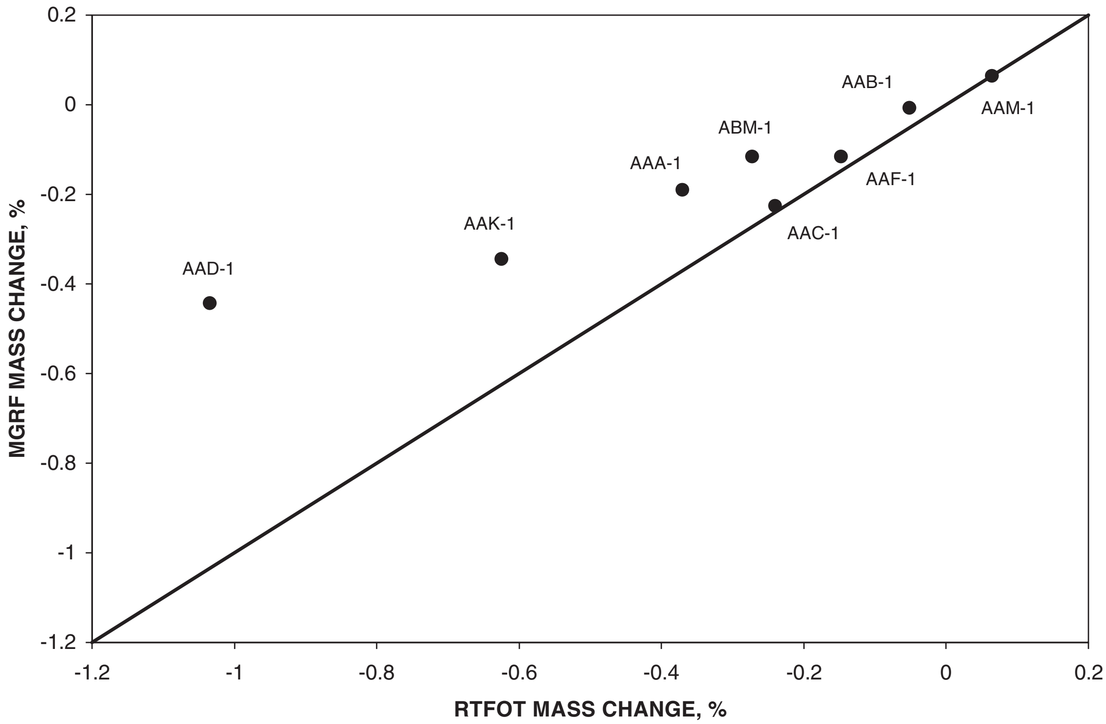


Figure 3-2. Comparison of mass change data from the MGRF and RTFOT.

Table 3-2. Figures 3-3 and 3-4 present rheological and mass change data for the binders used in the FHWA study. The rheological data in Figure 3-3 shows excellent agreement with the RTFOT similar to that from the Western Research Institute study. The mass change data, on the other hand, is significantly different than that from the Western Research Institute study. The conclusion from the FHWA study is that the mass changes in the MGRF are greater than those in the RTFOT. This conclusion is in general agreement with that from the Florida studies reported by Sirin et al. (*24*) and Tia et al. (*25*).

Research was conducted in Germany to develop a long-term aging procedure based on the GRF (*27*). This test, referred to as the Long-Term Rotating Flask (LTRF), uses the same equipment as the GRF to long-term-age binders in an oxygen atmosphere at lower temperatures. Three steel balls are introduced into the round-bottom flask from German Standard DIN 52 016 to promote mixing of binders at the lower aging temperatures. The long-term aging is performed at 95°C or 103°C depending on the grade of the binder. During the aging, oxygen is introduced into the flask at the rate of 7 L/h, and the duration of the test is 47 hours. Table 3-3 summarizes operating conditions for the LTRF.

Table 3-2. Recommended operating parameters for the MGRF.

Parameter	Condition (*2*)	Tolerance (*26*)
Sample Size	200 g	± 1 g
Temperature	165°C	± 1.5°C
Airflow	2 L/min	± 0.04 L/min
Rotational Speed	20 rpm	± 5 rpm
Heat-Up Time	10 min with no air flow	± 1 min
Aging Time	200 min under air flow	± 1 min

The degree of aging obtained with the LTRF was compared to that obtained with the Rotating Cylinder Aging Test (described in the next section of this report) using nine unmodified and nine modified binders. Sörensen (*27*) reported only moderate correlation for various properties measured on binders aged in the two devices. Linear correlation coefficients ranged from 0.75 to 0.97. Properties considered included softening point, penetration, penetration index, Frass breaking point, ductility, complex shear modulus, creep stiffness, and m-value.

3.2.2.2 Rotating Cylinder Aging Test

The Rotating Cylinder Aging Test (RCAT) was developed in Belgium to accurately simulate long-term aging (*28*). The device is shown in Figure 3-5. The main components are an oven for temperature control, and a cylindrical vessel and rotating mechanism for aging the binder. The vessel includes a unique rotating shaft mechanism to keep the binder evenly dispersed in the vessel during the test. Although the device was developed to simulate long-term aging, it can be used for short-term aging using temperature and airflow rates that are the same as those for the RTFOT.

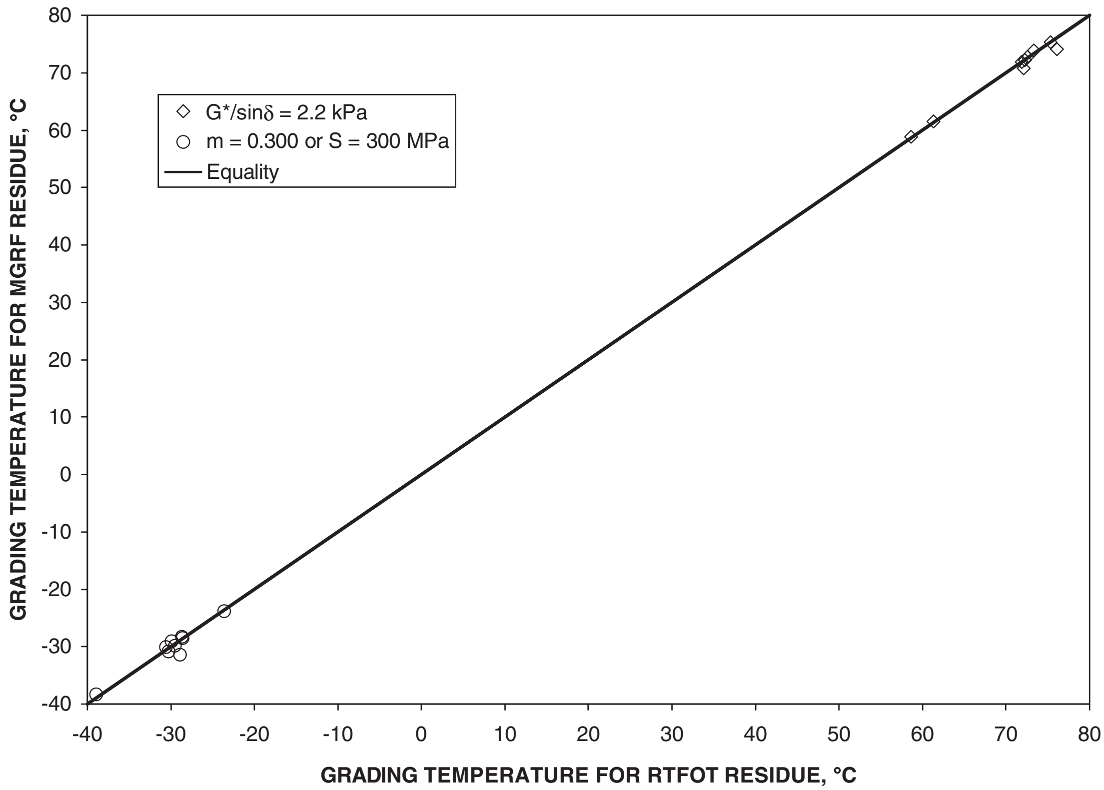


Figure 3-3. Comparison of continuous grade temperatures from the MGRF and RTFOT from the FHWA study.

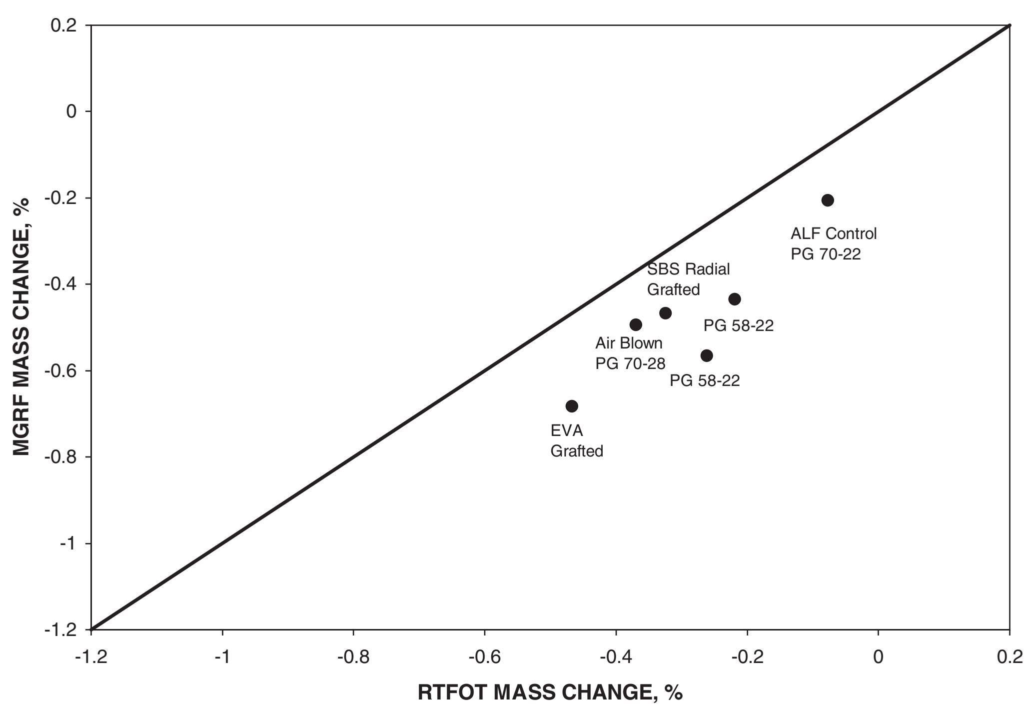


Figure 3-4. Comparison of mass change data from the MGRF and RTFOT from the FHWA study.

Table 3-3. Operating parameters for the LTRF.

Parameter	Condition
Sample Size	100 g
Mixing	3, 30-mm diameter steel balls
Temperature	95 or 103°C depending on binder grade
Rotational Speed	4 rpm
Atmosphere	Oxygen at 7 L/h
Aging Time	47 h

Table 3-4. Operating parameters for the RCAT.

Test	Parameter	Condition
	Sample Size	500 g
Short Term	Temperature	163°C
	Speed	1 rpm
	Airflow	Air at 4 L/min
	Aging Time	235 min
Long Term	Temperature	90°C
	Speed	1 rpm
	Airflow	Oxygen at 4.5 L/hr
	Aging Time	140 hr

Table 3-4 summarizes the operating conditions for the RCAT. Using the commercially produced version of the device, both short- and long-term aging can be performed with the same equipment. First, a 500-g sample of binder is short-term aged. Upon completion of the short-term aging, a portion of the sample is removed for physical property measurements. The remainder of the sample is then long-term aged. A unique concept included in the long-term aging procedure is the removal of samples at various times to allow characterization of aging kinetics. Using the operating conditions listed in Table 3-4, the RCAT reasonably reproduces the short-term aging that occurs in the RTFOT and the long-term aging that occurs in the PAV (*29, 30*).

3.2.2.3 Stirred Air Flow Test

Glover, et al. (*1*) proposed an air blowing test called the Stirred Air Flow Test (SAFT) for short-term aging of asphalt binders. The test was shown schematically in Figure 2-2. In

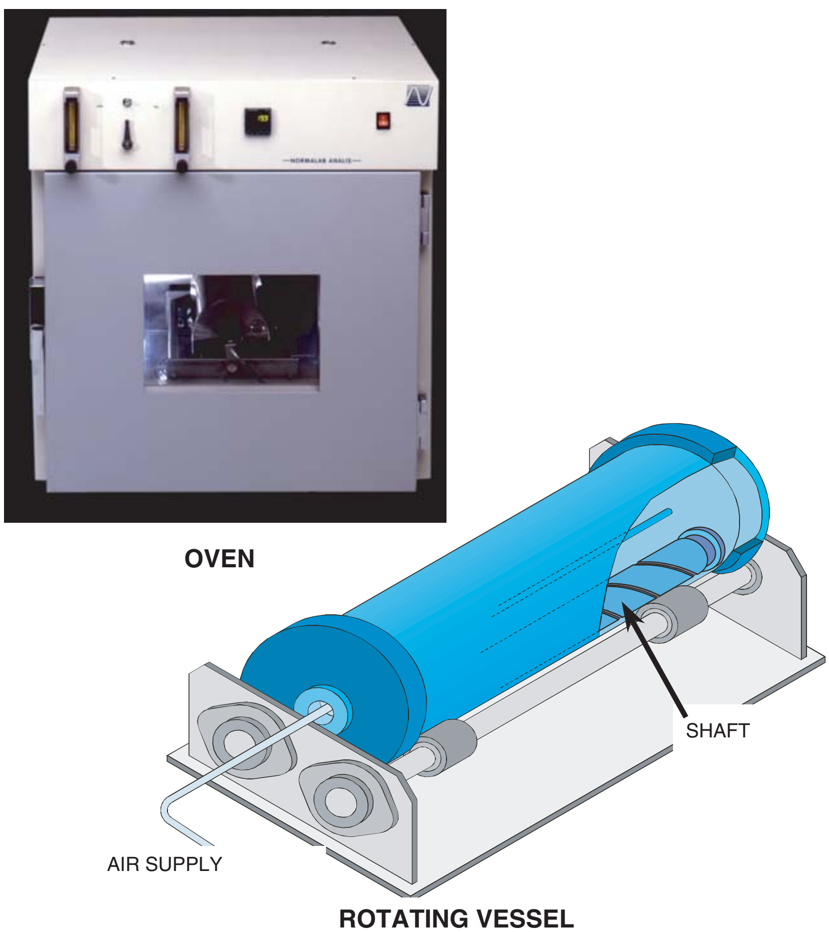


Figure 3-5. Rotating cylinder aging test.

the SAFT, air from a nozzle submerged in the binder is dispersed in the binder by an impeller mounted to an external motor. Through trial and error, operating parameters for the device were determined that approximate the level of aging that occurs in the RTFOT. This work included consideration of the effects of sample size, impeller type, nozzle type, airflow rate, and impeller speed on 60°C viscosity measurements and carbonyl growth. Impeller speed had the greatest effect on the viscosity and carbonyl growth measurements. Increasing the speed of the impeller significantly increased aging as measured by viscosity and carbonyl growth. The other operating conditions considered had only a minor effect on the aging process. After appropriate operating parameters were selected, changes in 60°C complex viscosity and carbonyl growth were used to compare the device and the RTFOT for several unmodified and modified binders. Figure 3-6 presents a typical comparison reported for the SAFT. The proposed operating parameters are summarized in Table 3-5. A draft ASTM standard test method for the SAFT was developed.

The SAFT also includes a volatile compound collection system that consists of a simple air-cooled condenser through which the exhaust air from the vessel passes before exiting to the atmosphere. Volatile compounds produced during the short-term aging process become trapped in the condenser. They are removed by washing the condenser with solvent, and then they are weighed after the solvent evaporates. Figure 3-7 presents a comparison of the mass change from the RTFOT and the mass of volatile compounds collected with the SAFT as reported by Glover, et al. (*1*). As shown in Figure 3-7, the mass of volatile compounds collected with the SAFT is approximately an order of magnitude less than the RTFOT mass loss. It is less sensitive to binder source than in the RTFOT mass change and the order of ranking varies. Glover, et al. (*1*) also reported high variability for the condensed volatile compounds measurements. Coefficients of variation for multiple tests on the same asphalt were high, averaging approximately 35 percent. Glover, et al. (*1*) dismissed the poor agreement between the RTFOT mass change and the mass of condensed volatile compounds collected in the SAFT as being the result of errors in the RTFOT mass change measurements. They hypothesized that the high variability in the SAFT measurements may be the result of condensation of volatile compounds on the lid caused by temperature gradients over the height of the vessel. They recommended using a heating mantel that heats the device over its entire height in an effort to reduce the potential for volatile compounds to condense on the lid.

Since its development, additional evaluation of the SAFT has been completed by the Texas Department of Transportation and FHWA. The Texas effort was directed at an interlaboratory study to develop precision statistics for the SAFT and to compare them to the RTFOT (*31*). Six laboratories and six materials were used in the study. Precision statistics

Table 3-5. Proposed operating parameters for the SAFT.

Parameter	Condition
Sample Size	250 g
Temperature	163°C
Airflow	2000 mL/Min
Stirrer Speed	700 rpm
Heat-Up Time	15 Min under Nitrogen
Aging Time	30 Min under Air

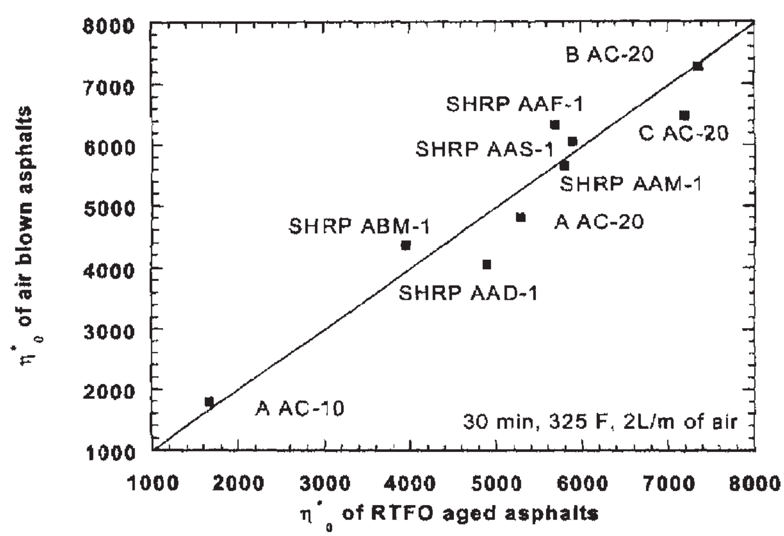


Figure 3-6. Comparison of viscosity for asphalts aged in the SAFT and RTFOT (1).

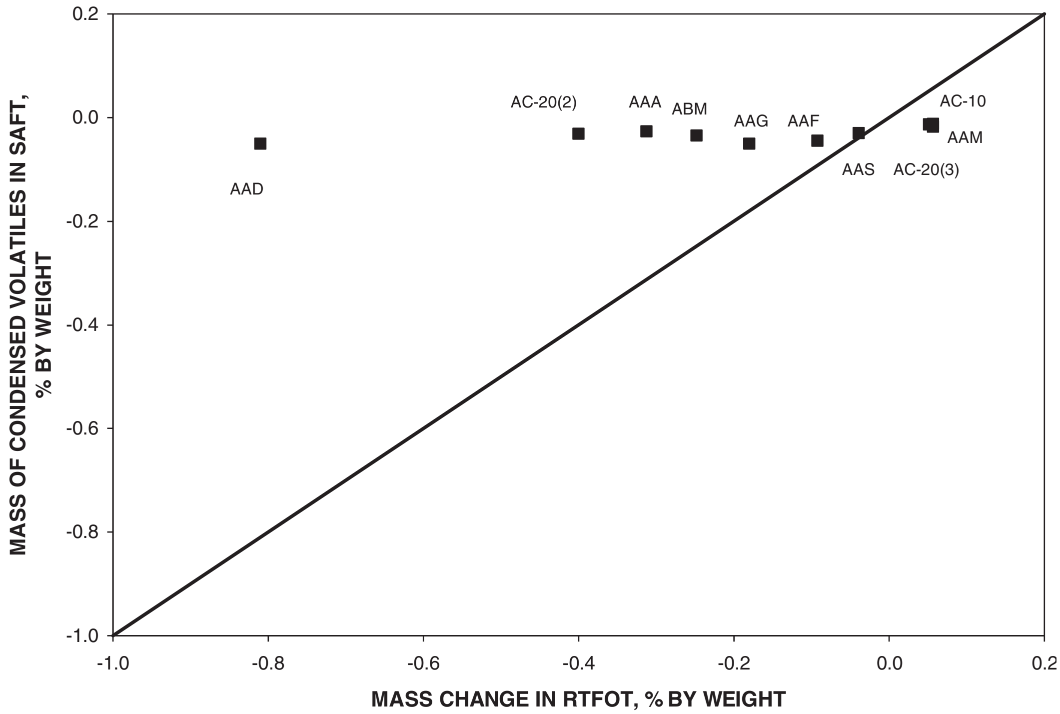


Figure 3-7. Comparison of mass of condensed volatile compounds collected in the SAFT and the mass change from the RTFOT.

were developed for G*/sinδ measured at the specified high-temperature grade of the material tested, and the mass of volatile compounds collected. For the degree of aging as measured by G*/sinδ, the authors concluded that

1. The degree of aging in the SAFT at the current operating conditions is somewhat less than that in the RTFOT. The aging time should be increased from 30 to 35 min to provide better agreement between the two methods.
2. The precision statistics for the SAFT were slightly poorer than those for the RTFOT, but should improve as operators become more familiar with the device.

The authors also concluded that the volatile loss procedure in the SAFT worked very well and provided a significant improvement of the RTFOT mass change determinations. This conclusion for the volatile loss procedure is suspect because three of the laboratories could not provide valid volatile loss data, and the corresponding data for the RTFOT were not collected.

The FHWA evaluation of the SAFT included a comparison of high- and low-temperature rheological property measurements for four binders aged in the SAFT and the RTFOT (*26*). The aging time in the SAFT test was 35 min as recommended by Glover, et al. (*31*). Figure 3-8 compares the continuous performance grade temperature for binders that are short-term aged in the two tests. As shown, rheological properties of short-term-aged binders from the SAFT show a similar degree of agreement with the RTFOT as short-term-aged binders from the MGRF.

3.2.3 Candidate Short-Term Binder Aging Procedures

Based on the review of the literature and research in progress, three candidate procedures were identified for further consideration in Project 9-36. They are the (1) MGRF, (2) RCAT, and (3) SAFT. This section discusses the advantages of adapting positive elements of the procedures in a hybrid procedure, and presents the procedures that were recommended for further consideration in NCHRP Project 9-36.

3.2.3.1 Comparison of Candidate Tests

Table 3-6 compares pertinent details of the three candidate tests. This table is arranged to quickly compare the three tests. The upper section presents general information about the test, such as the cost and complexity of the equipment, the quantity of material that can be aged, how easy it is to use and clean, and whether NCHRP Project 9-36 could take advantage of further development that was ongoing. The second section presents specific information relative to application of the test to short-term aging, and the last section presents specific information relative to long-term aging.

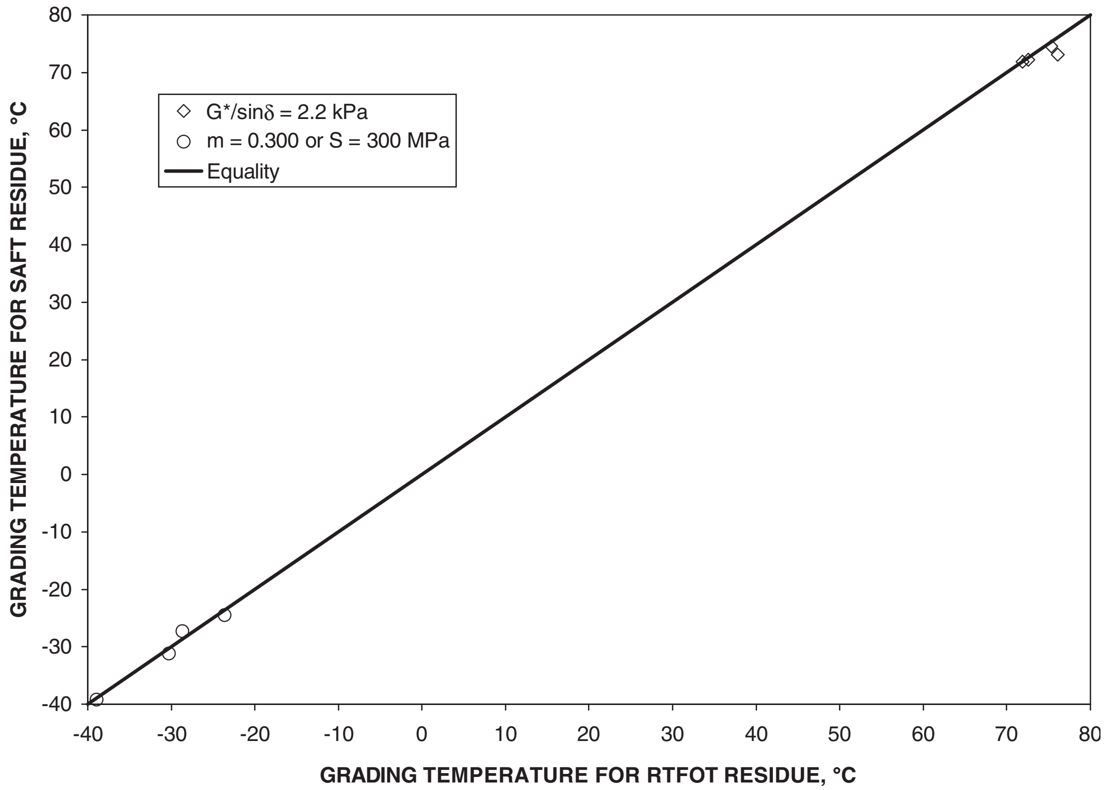


Figure 3-8. Comparison of continuous performance grade temperatures for binders aged in the SAFT and RTFOT from the FHWA study.

Table 3-6. Comparison of candidate methods.

Considerations		Rotating Cylinder Aging Test	Modified German Rotating Flask	Stirred Air Flow Test
General	Neat and Modified Binders	Yes	Yes	Yes
	Amount of Material	500 g	200 g	250 g
	Equipment Cost	$18,000	$3,500	$5,000 estimated
	Equipment Complexity	Moderately complex	Simple	Moderately complex
	Availability	Low	High	Low
	Test Complexity	Reasonable	Simple	Reasonable
	Binder Recovery	Easy	Easy	Easy
	Clean Up	Solvent	Ignition Oven	Solvent
	Active Development	Yes	Yes	Yes
Short-Term Aging	Published Procedure	Yes	Yes	Yes
	Further Refinement Needed	No	No	Yes
	Temperature	163°C	165°C	163°C
	Duration	235 min	210 min	45 min
	Atmosphere	Air at 4 L/min	Air at 2 L/min	Air at 2 L/min
	Measure Volatility	None—configuration of vessel makes volatile recovery difficult	Mass change—adaptable to volatile recovery	Volatile recovery system
	Mimics Aged Binder Chemistry	Yes	Yes	Yes
	Repeatability/Reproducibility	Limited data	Limited data	Some data
Long-Term Aging	Adaptable to Long Term	Yes—work completed	Yes—steel balls to enhance mixing	Probably with new impeller
	Published Procedure	Yes	Yes	No
	Temperature	90°C	95°C neat 103°C modified	NA
	Duration	140 hr	47 hr	
	Atmosphere	Oxygen at 4.5 L/hr	Oxygen at 7 L/hr	
	Aging Kinetics Possible	Yes	Yes	Yes
	Mimics Aged Binder Chemistry	Yes	Yes	Likely
	Repeatability/Reproducibility	Limited	Limited	None

As shown, all three tests generally meet the requirements established for an improved short-term binder aging test. Each has been shown to be relatively simple, applicable to both neat and modified binders, and to reasonably reproduce the level of aging that occurs in the RTFOT. Procedures for extending the RCAT and the MGRF to long-term aging at reasonable temperatures and without pressure have been developed, but these will require modification and further development to adapt them to specification testing in the United States. Although a long-term aging procedure has not been developed for the SAFT, it appears that the device is extendable to long-term aging, provided sufficient dispersion of air can be accomplished in stiff modified binders at temperatures around 90°C to 100°C. This may require redesign of the stirring mechanism to adequately disperse air in highly viscous media. The flexibility to design this portion of the device is a distinct advantage for extending the test to long-term aging.

3.2.3.2 Promising Tests

The RCAT and MGRF are conceptually similar and also very similar to the RTFOT. Both involve exposing a relatively thin film of binder to air or oxygen. The binder film is renewed by rotating the vessel containing the binder. The RCAT vessel and the MGRF both include elements to mix the binder using gravity. The RCAT includes an internal shaft mechanism that keeps the binder evenly dispersed in the vessel. In the MGRF, the indentations in the Morton flask mix the binder during the short-term test. In the long-term test proposed in Germany, steel balls are added to a smooth flask to provide mixing. The MGRF is, by far, the more appealing of these two devices from the perspective of cost, equipment availability, and testing time for long-term aging. The test can be assembled from off-the-shelf components for a reasonable cost. The RCAT is more expensive (approximately $18,000) and available from only a single supplier. Because the long-term RCAT requires 1 week to complete, and the equipment for the MGRF is more readily available at a lower cost, the RCAT was not considered for further development in NCHRP Project 9-36.

The SAFT uses a different approach in which air is dispersed in the binder using a stirring mechanism. It is essentially a small, laboratory-scale air blowing still. A thin film is not produced in this device. Instead, air from a nozzle submerged in the binder is dispersed in the binder by an impeller attached to an external motor. It appears that for short-term aging, this approach is much more efficient at mixing air with the binder because the duration of the test is approximately one-half to one-quarter of that for the tests involving thin films with similar temperatures and airflow rates. It is not known whether the more rapid aging observed in the SAFT is the result of more rapid volatile removal or more rapid oxidation. Of concern is the possibility that more vigorous oxidation in the SAFT may be overwhelming volatile loss, especially given the low amounts of volatiles that have been collected by previous researchers using this test. Whether adequate mixing of air and binder will occur at the lower temperatures needed for extending the test to long-term aging is an issue that was addressed experimentally in NCHRP Project 9-36.

Another important consideration for an improved short-term aging procedure is the ability to quantify the amount of volatile material lost from the binder. Only the SAFT offers a method for directly measuring the volatile compounds lost during the aging process. It includes a condenser for collecting the volatile compounds from the exhaust air from the vessel. The volatile compounds trapped by the condenser can be weighed to determine the amount of material lost and can be analyzed to determine their composition. Although this approach requires additional development to reduce the level of variability reported in the initial development studies, it is better than determining mass change by weighing the filled vessels before and after aging as is done with the RTFOT and MGRF. The change in mass after short-term aging is the net result of a loss in mass due to the loss of volatile compounds and an increase in mass due to oxidation. It appeared that the MGRF could be modified to collect volatile compounds using an approach similar to that used in the SAFT.

3.3 Selection Study

3.3.1 Introduction

The primary finding from the review of the literature and research in progress was that the MGRF and SAFT are both promising approaches for an improved short-term aging procedure to be used in the United States with AASHTO M320. The equipment required for both tests is relatively inexpensive, and they are easy to perform, applicable to both neat and modified binders, and reasonably reproduce the level of aging that occurs in the RTFOT. The major issue unresolved through the literature review and review of research in progress was which of these two tests was best suited for extension to long-term aging. Only limited data was available on a long-term version of the MGRF, and no long-term aging data was available for the SAFT. The selection study was designed to investigate the feasibility of extending these tests to long-term aging. Since cost, complexity, and ability to simulate the RTFOT were judged to be similar for the two tests, the extendibility to long-term aging became an important factor in selecting the short-term test method for further development in Project 9-36.

The selection study was conducted in two parts. The first part of the study was an assessment of various modifications that could easily be made to the MGRF and SAFT to produce prototype long-term versions of these tests. The goal in this effort was to obtain approximately the same degree of aging that occurs in the PAV. The second part of the selection study was a formal experiment designed to address whether the degree of aging in the prototype long-term versions of the tests is affected by the large differences in viscosities for neat and modified binders at the selected long-term aging temperature of 100°C. This section presents key findings from the selection study. The complete selection study report is included as Appendix B.

3.3.2 Development of Initial Prototype Long-Term Aging Tests

3.3.2.1 MGRF

Attempts to develop a prototype long-term version of the MGRF that approximates the aging produced in the PAV focused on methods to enhance mixing and to create a film that is continuously renewed within the flask. This was accomplished by adding various mixers and scrapers and varying the rotational speed of the flask. For all of this testing, a temperature of 100°C, an airflow rate of 36 L/h, and an aging time of 48 hours were used. Table 3-7 summarizes the chronological order of the various configurations that were attempted. Figure 3-9 compares the degree of aging achieved with each configuration relative to the aging obtained with the PAV. Schematic diagrams of selected configurations are shown in Figures 3-10 through 3-13. The measure of the degree of aging shown in Figure 3-9 is defined by Equation 1. The relative aging according to this equation is simply the change in viscosity above RTFOT aging caused by the prototype long-term test divided by the increase in viscosity that occurs during PAV aging. For all equipment configurations and binders, relative aging is reported based on the dynamic viscosity measured at 60°C and 0.1 rad/s.

$$RA = \left(\frac{|\eta^*|_i - |\eta^*|_{RTFOT}}{|\eta^*|_{PAV} - |\eta^*|_{RTFOT}} \right) \times 100\% \qquad (1)$$

where

RA = relative long-term aging

$|\eta*|_i$ = dynamic viscosity for configuration i, measured at 60°C, 0.1 rad/s

$|\eta*|_{RTFOT}$ = dynamic viscosity for RTFOT aged, measured at 60°C, 0.1 rad/s

$|\eta*|_{PAV}$ = dynamic viscosity for PAV aged, measured at 60°C, 0.1 rad/s

The investigation of various alternatives for a long-term version of the MGRF procedure started with the MGRF, which uses a 2,000-mL Morton flask. The alternatives that were investigated included a 2,000-mL round flask with the addition of steel balls and rollers to enhance mixing and formation of a film and the use of scrapers to create and renew the film. As shown in Figure 3-9, the Morton flask was marginally successful for long-term aging for the PG 58-28 binder, but was not successful for aging the PG 82-22 binder. The use of a round flask with steel balls, as used in Germany, increased the aging of the PG 82-22 slightly while the use of rollers that conformed to the shape of the flask did not. The simple scrapers designed to fit in a round flask appear to remove much of the viscosity effect, resulting in similar aging of the PG 58-28 and PG 82-22 binders, but the degree of aging after 48 hours is only one-third of that obtained in the PAV.

3.3.2.2 SAFT

Attempts to develop a prototype long-term version of the SAFT that approximates the aging produced in the PAV

Table 3-7. Summary of long-term rotating flask configurations tested.

Number	Flask	Mixer	Speed	Figure	Observations
1	Morton	None	4 rpm	Not shown	1. Adequate film for PG 58-22 binder. 2. Does not produce a moving film for PG 82-22 binder. 3. Low relative aging for both binders.
2	Morton	3 Steel Balls	1 rpm	Figure 3-10	1. Not used with PG 58-28 binder. 2. Does not produce a moving film for PG 82-22 binder. 3. Low relative aging for PG 82-22 binder
3	Round	1 Football-Shaped Roller	1 rpm	Not shown	1. Not used with PG 58-28 binder. 2. Does not produce a moving film for PG 82-22 binder. 3. Low relative aging for PG 82-22
4	Round	2 Football-Shaped Rollers	1 rpm	Figure 3-11	1. Not used with PG 58-28 binder. 2. Does not produce a moving film for PG 82-22 binder. 3. Low relative aging for PG 82-22.
5	Round	Single Scraper	1 rpm	Figure 3-12	1. Does not produce a film for PG 58-28 binder. 2. Generated a renewed film for PG 82-22, but film thickness increased with aging time. 3. Low relative aging for both binders.
6	Round	Double Scraper	1 rpm	Figure 3-13	1. Not used with PG 58-28 binder. 2. Generated a renewed film for PG 82-22. Film thickness relatively constant with aging time. 3. Low relative aging for PG 82-22 binders.

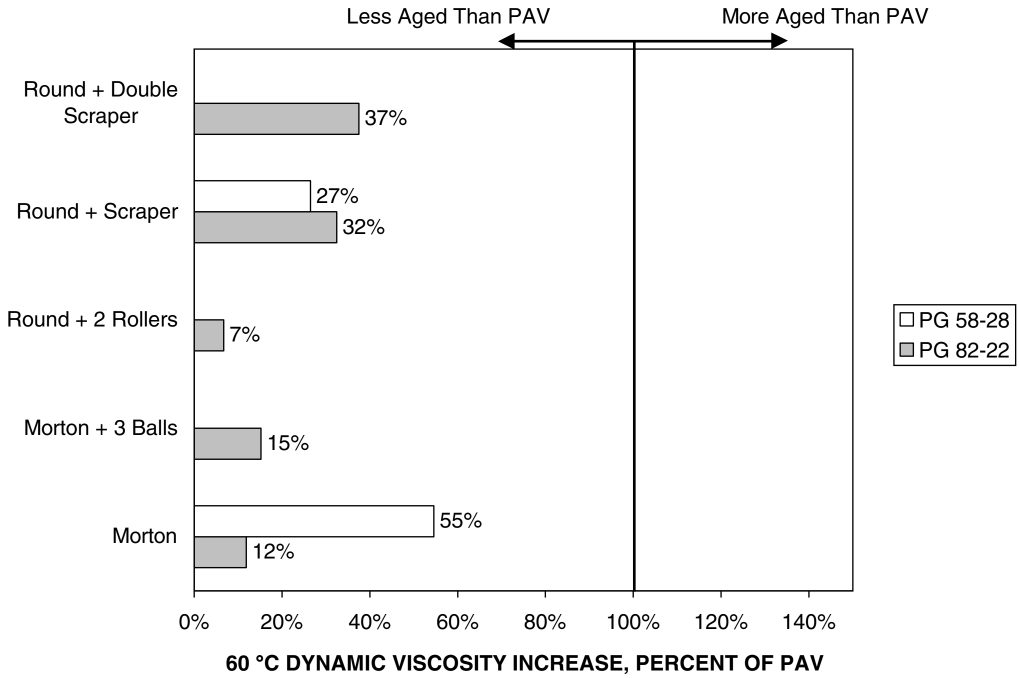


Figure 3-9. Relative aging from Equation 1 for various long-term rotating flask configurations.

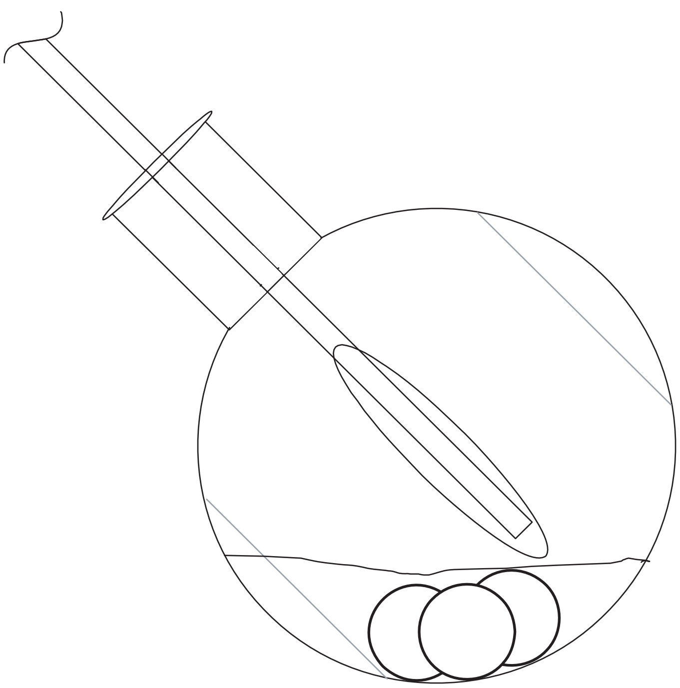


Figure 3-10. Schematic of 2,000-mL Morton flask with three steel balls.

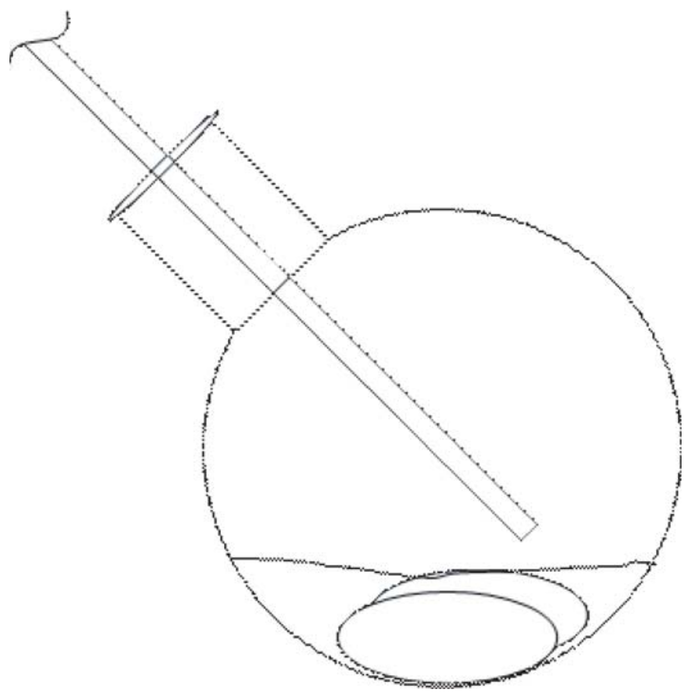


Figure 3-11. Schematic of 2,000-mL round flask with two football-shaped rollers.

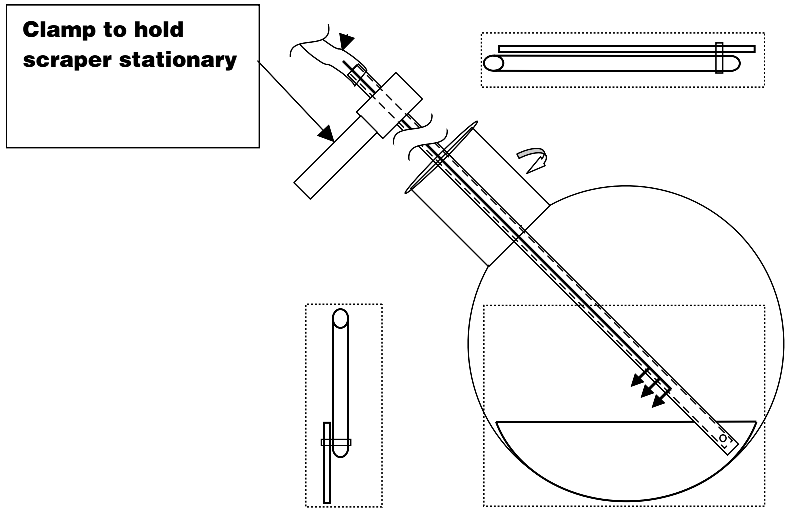


Figure 3-12. Schematic of 2,000-mL round flask with single scraper.

focused on the design of an impeller that could efficiently mix air with the binders over a wide range of viscosities. The design proceeded from the impeller used in the short-term version of the test, which is very efficient at mixing air with low viscosity binders, to a helix impeller which is efficient at mixing highly viscous fluids, and finally to a helix/turbine impeller which combines the benefits of both. For all of this testing, a temperature of 100°C and an airflow rate of 36 L/h were used. Table 3-8 summarizes the chronological order of the various configurations that were attempted. Figure 3-14 compares the degree of aging achieved with each configuration relative to the aging achieved with the PAV. Schematic diagrams of selected configurations are shown in Figures 3-15 through 3-17. The measure of the degree of aging shown in Figure 3-14 is the same used in Figure 3-9 and defined in Equation 1. The relative aging according to this equation is simply the change in viscosity above RTFOT aging caused by the prototype long-term test divided by the increase in viscosity that occurs during PAV aging. The dynamic viscosity was measured at 60°C, 0.1 rad/s.

The original impeller worked well with the PG 58-28 binder, but it did not provide adequate mixing of the PG 82-22 binder. When this impeller is used with extremely viscous materials, the entire mass of material spins with the impeller. The helix impeller, which is frequently used to mix very viscous and particulate-filled fluids, worked well with the PG 82-22 binder, but apparently did not disperse air as efficiently in the less viscous PG 58-28 binder. The helix/turbine impeller, which includes a helix to move the binder vertically in the

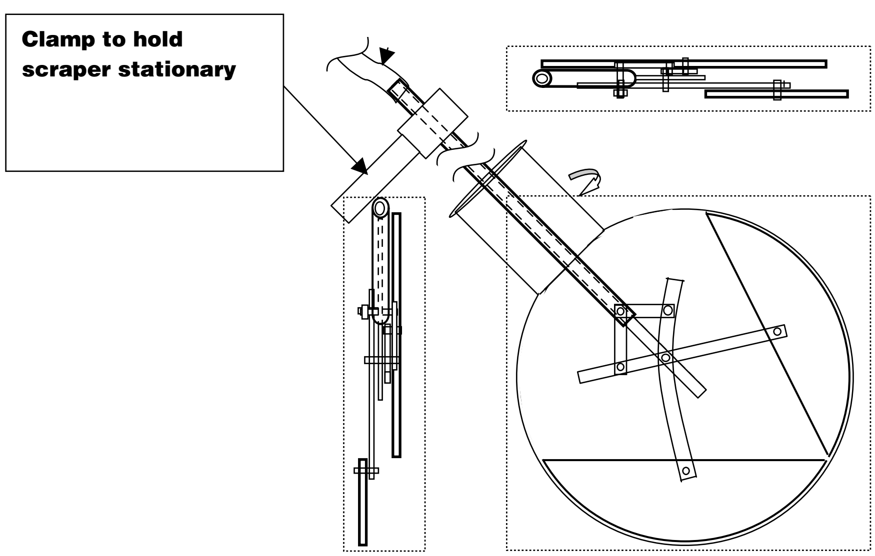


Figure 3-13. Schematic of 2,000-mL round flask with double scraper.

Table 3-8. Summary of long-term SAFT configurations tested.

Number	Impeller	Speed	Schematic	Observations
1	Original	700 rpm	Figure 3-15	1. Good mixing of PG 58-28 binder. 2. Could not stir PG 82-22 binder. 3. High relative aging for PG 58-28.
2	Helix	220 rpm	Figure 3-16	1. Good mixing of both PG 58-28 and PG 82-22 binders. 2. Better mixing of PG 82-22 binder occurs at lower speeds. 3. High relative aging of PG 82-22 binder. 4. Moderate relative aging of PG 58-28 binder.
3	Helix/ 4-Bladed Turbine	350 rpm	Figure 3-17	1. Good mixing of PG 58-28, PG 82-22, and PG 76-22 binders. 2. Aging at 48 hrs exceeds PAV conditions for the PG 58-28 and PG 82-22. 3. Less difference in aging between PG 58-28 and PG 82-22 than observed with helix.
4	Helix/ 8-Bladed Turbine	350 rpm	Not shown	1. Good mixing of PG 58-28, PG 82-22, and PG 76-22 binders. 2. PAV aging obtained at approximately 40 hours.

vessel, and a turbine to mix air with the binder, resulted in the best performance over the range of binders investigated. At 48 hours, the degree of aging obtained in the PG 58-28 and PG 82-22 binders exceeded that obtained in the PAV.

The last iteration of the impeller design was a helix/turbine impeller with eight turbine blades. With this impeller, PAV aging conditions were reached after approximately 40 hours. Figure 3-18 shows the degree of aging obtained with this configuration for the three binders included in the selection study. The measure of the degree of aging shown in Figure 3-18 is the same used in Figures 3-9 and 3-14 and defined in Equation 1. The relative aging according to this equation is simply the change in viscosity above RTFOT aging caused by the prototype long-term test divided by the increase in viscosity that occurs during PAV aging. The dynamic viscosity was measured at 60°C, 0.1 rad/s. The degree of aging appears to increase with increasing binder stiffness, which is counterintuitive.

The testing described earlier found that it is possible to extend the SAFT to a long-term aging test. The following section discusses the experiment on viscosity effects that was conducted to quantify the significance of the differences between the aging of the PG 58-28 and the PG 82-22 binder shown in Figure 3-18.

3.3.3 Viscosity Effects Experiment

Only the final iteration (a helix/turbine impeller with eight turbine blades) of the long-term version of the SAFT was subjected to the experiment on viscosity effects. Table 3-9 summarizes the testing conditions for the long-term SAFT.

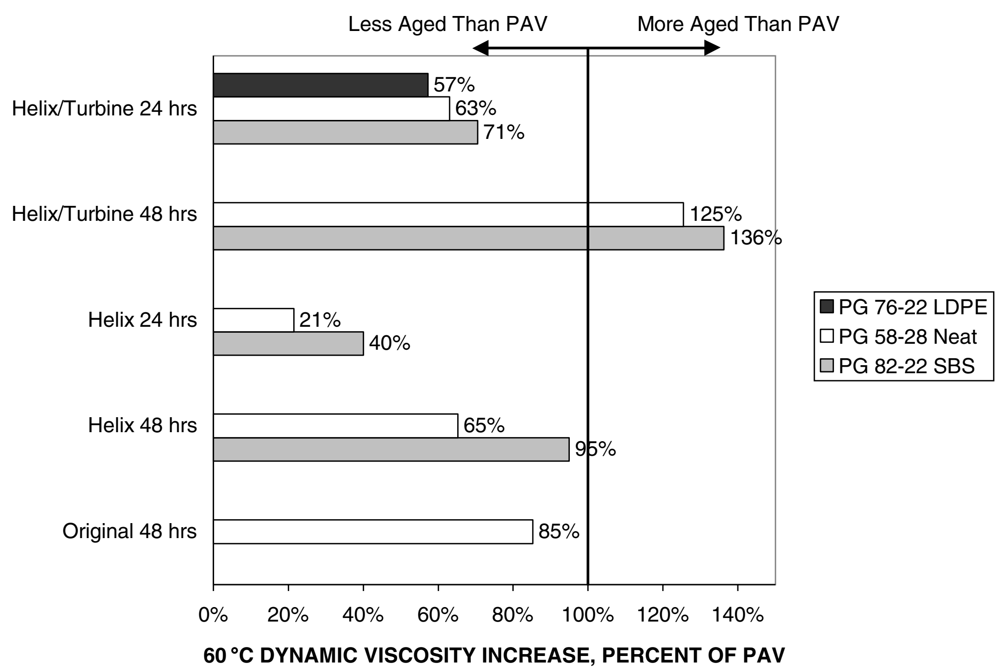


Figure 3-14. Relative aging from Equation 1 for various long-term SAFT configurations.

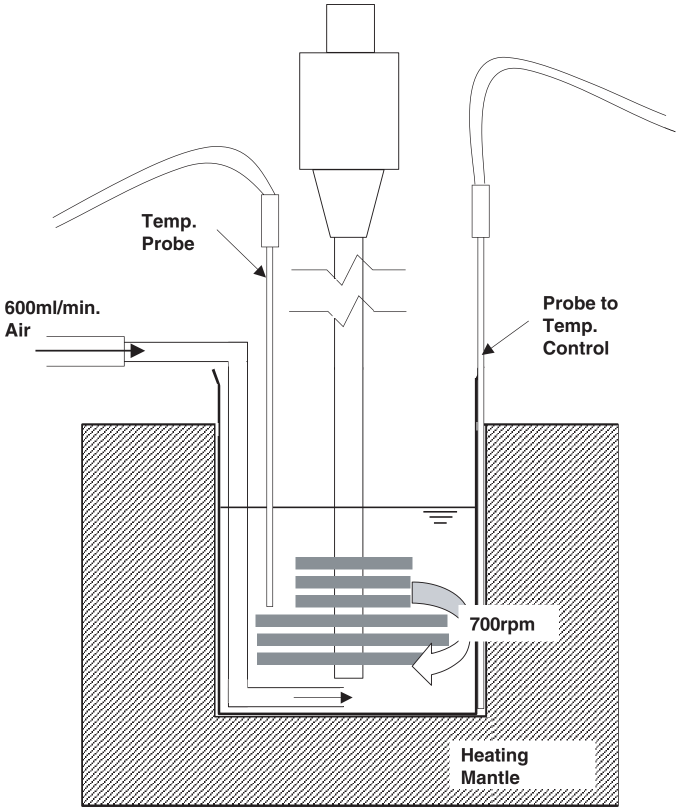


Figure 3-15. Schematic of long-term SAFT with original impeller.

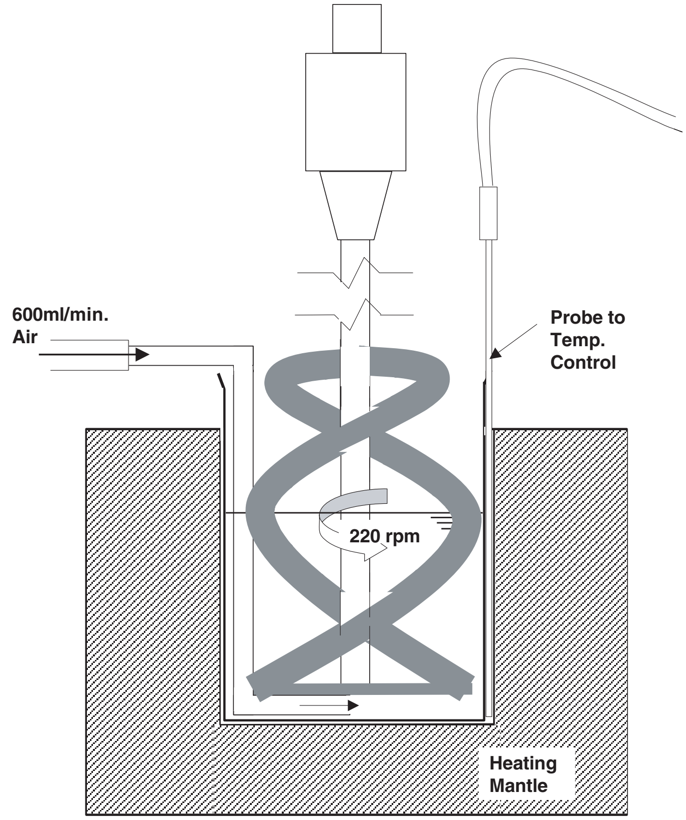


Figure 3-16. Schematic of long-term SAFT with helix impeller.

The viscosity effects experiment was designed to investigate the effect of viscosity on the degree of aging that occurs in the long-term SAFT. This was accomplished by aging split samples of RTFOT-aged PG 58-28 and PG 82-22 binders in the PAV and the long-term SAFT, and comparing rheological properties at high, intermediate, and low pavement temperatures. The following properties were measured for the RTFOT, PAV, and long-term SAFT:

- Shear modulus and phase angle from a DSR frequency sweep at 60°C using frequencies from 0.1 to 100.0 Hz.
- Shear modulus and phase angle from a DSR frequency sweep at 25°C using frequencies from 0.1 to 100.0 Hz.
- Creep stiffness and m-value at 60 sec from BBR tests conducted at −12°C.

Three independent samples were aged in the long-term SAFT and PAV and tested as outlined above. Regression analysis was used to compare the rheological properties between the PAV and the long-term SAFT. Details of the statistical analysis are presented in Appendix B on the project webpage. The statistical analysis found that there was a statistically significant difference in aging between the PAV and the long-term SAFT and that the difference was binder dependent. Table 3-10 summarizes the key differences and compares them to the single operator coefficient of variation for the DSR and BBR

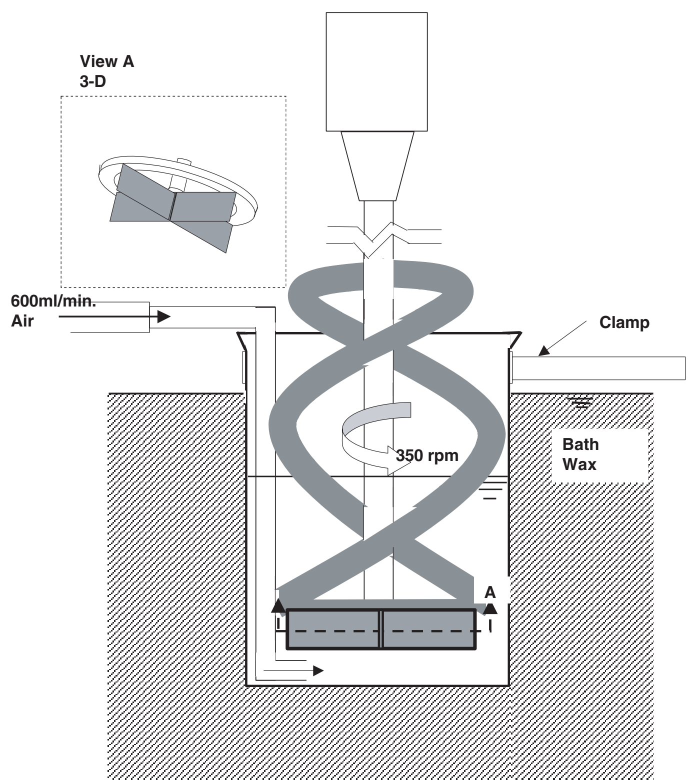


Figure 3-17. Schematic of long-term SAFT with helix/turbine impeller.

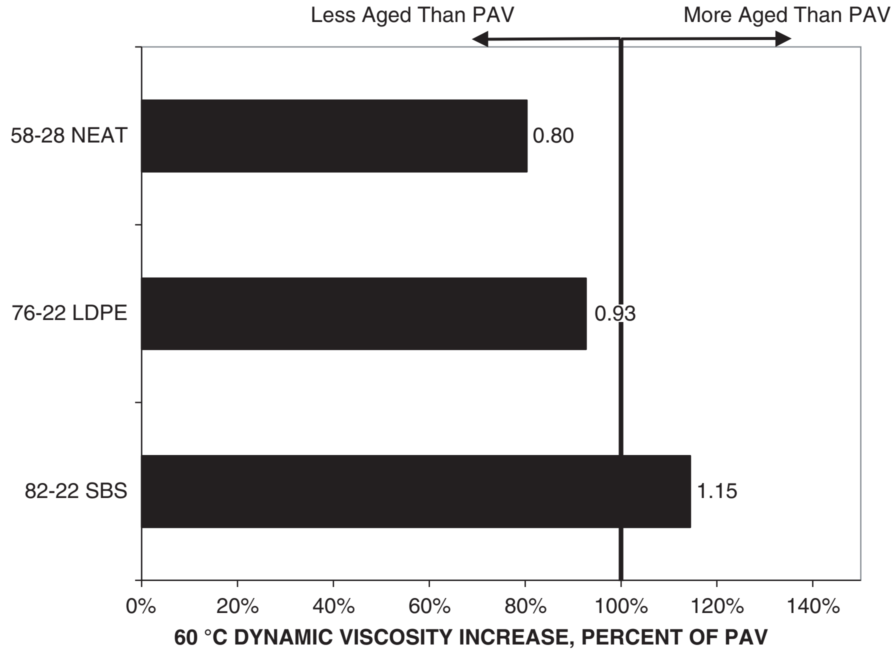


Figure 3-18. Relative aging from Equation 1 for final iteration of the long-term SAFT (helix/eight-bladed turbine, 200 rpm, 36 L/h airflow, 100°C, 40 hours).

tests. The bias of the long-term SAFT relative to the PAV is approximately twice the AASHTO single-operator precision; therefore, these biases have engineering significance as well statistical significance. Probably more important than the finding that the aging was different between the PAV and the long-term SAFT was the fact that the two binders aged differently. The PG 82-22 binder aged more in the long-term SAFT than in the PAV, while the PG 58-28 binder aged more in the PAV than in the long-term SAFT. This difference was most evident in the high pavement temperature tests, but also occurred in the intermediate and low pavement temperature tests that are used in AASHTO M320. Differences between the aging produced by the long-term SAFT and the PAV appear to be temperature dependent. The differences were greater at the upper grading temperature than at the lower grading temperature. This implies that the two aging procedures produce materials that are different rheologically.

There are two possible explanations for the binder effect. First, the helix/turbine impeller and its rotational speed may not be properly optimized for lower viscosity binders. Second, the air dispersion mechanism in the long-term SAFT may age polymers more than, or in a different way than, the high-pressure aging occurring in the PAV. Additional testing of neat and modified binders, both having a wide range of consistency, is needed to determine the cause of this effect and to further improve the long-term SAFT. This additional testing was beyond the scope of NCHRP Project 9-36.

Table 3-9. Testing conditions for the long-term SAFT.

Condition	Value
Sample Size	250 g
Aging Temperature	100°C
Impeller Type	Helix + 8-Bladed Turbine
Impeller Speed	350 rpm
Airflow Rate	36 L/h
Aging Time	40 hours

Table 3-10. Comparison of long-term SAFT bias with DSR and BBR precision.

Property	Long-Term SAFT Bias Relative to PAV, %		AASHTO Single Operator Coefficient of Variation, %
	PG 58-28	PG 82-22	
G* at 60 °C	-10	+14	7.9
G* at 25 °C	-6	+13	7.9
S at -12 °C	+3	+7	3.2
m at -12 °C	-3	+3	1.4

Based on the findings of the selection study, the SAFT was selected for further development in NCHRP Project 9-36. The SAFT was selected because the selection study found that with additional development it may be possible to extend this test to long-term aging. The MGRF, on the other hand, could not be extended to long-term aging because of inadequate mixing of air and binder in this test at the lower aging temperatures that should be used to simulate in-service conditions.

3.4 Volatile Collection System Study

The objective of the volatile collection system (VCS) study was to design and evaluate an improved VCS for the SAFT. The prototype SAFT included a VCS, which consisted of a copper coil condenser operated at ambient temperature. As shown previously in Figure 3-4, the mass of volatiles collected using this system was a factor of 10 lower than the mass change in the RTFOT and showed little difference between binders. The VCS study, which is described in detail in Appendix C (see the project webpage on the TRB website), included an evaluation of the air-cooled condenser used in the prototype SAFT, the design of an improved VCS employing reusable adsorbents that are commonly used for chromatographic analyses, and evaluation of the improved VCS. This section presents key findings of the VCS study.

3.4.1 Evaluation of Prototype SAFT VCS

A mass change experiment was conducted with the SAFT to identify the cause of the unexpectedly low volatile mass collected in the prototype VCS. The neat PG 58-28 binder that was included in the selection study was aged under the following conditions:

- Condition 1—RTFOT using standard conditions;
- Condition 2—SAFT with the prototype VCS in place, airflow rate 2,000 mL/min;
- Condition 3—SAFT without the lid or prototype VCS, airflow rate 2,000 mL/min; and
- Condition 4—SAFT without the lid or prototype VCS, airflow rate 4,000 mL/min.

The mass of the volatiles was determined for Condition 2 by flushing the volatiles from the VCS with solvent and evaporating the solvent. In Conditions 2, 3, and 4 mass change was determined by weighing the vessel and its components before and after conditioning. Table 3-11 summarizes the results of the testing.

This experiment provided important insight into the behavior of the prototype VCS. First, the SAFT without the lid and prototype VCS produced mass losses that are less than the RTFOT, but a factor of 10 higher than the mass of the volatiles collected in the prototype VCS. The mass of the volatiles collected in this experiment by the prototype VCS is of the same size as those reported by the original developers of the SAFT, which ranged from 0.013 to 0.051 percent by weight (*1*). Thus, it appeared that only a small percentage of the volatiles that are produced by the SAFT was being collected by the VCS. At a flow rate of 2,000 mL/min, the mass of the volatiles trapped in the collection system was only 0.013 percent by mass compared to a mass loss of 0.11 percent when the lid and collection system were removed. Based on this finding, the design of an improved VCS that would capture a greater amount of volatiles was initiated.

3.4.2 Improved VCS Design

The design of an improved VCS was an incremental process. In the first iteration, called VCS-I, silica gel and activated carbon filters were added after the condenser from the prototype VCS to collect moisture and hydrocarbon material passing through the condenser. Silica gel and activated carbon filters also were added before the SAFT vessel to remove moisture and hydrocarbon material from the incoming air. Figure 3-19 is a schematic of VCS-I. This version was used in a study to investigate the effect of pressure inside the SAFT on the amount of volatiles produced. All testing was performed with

Table 3-11. Summary of SAFT mass change measurements.

Test and Test Parameters	Airflow, mL/Min	Mass Change, wt %[a]	Average Mass Change, wt %	Mass of Collected Volatiles, wt %	Average Mass of Collected Volatiles, wt %
RTFOT	4,000	-0.357 -0.349	-0.35	NA	NA
SAFT with lid and collection system	2,000	NA	NA	0.016 0.011	0.014
SAFT without lid and collection system	2,000	-0.108 -0.112	-0.11	NA	NA
SAFT without lid and collection system	4,000	-0.184 -0.176	-0.18	NA	NA

Note: [a] Negative values in this table indicate a loss in mass.

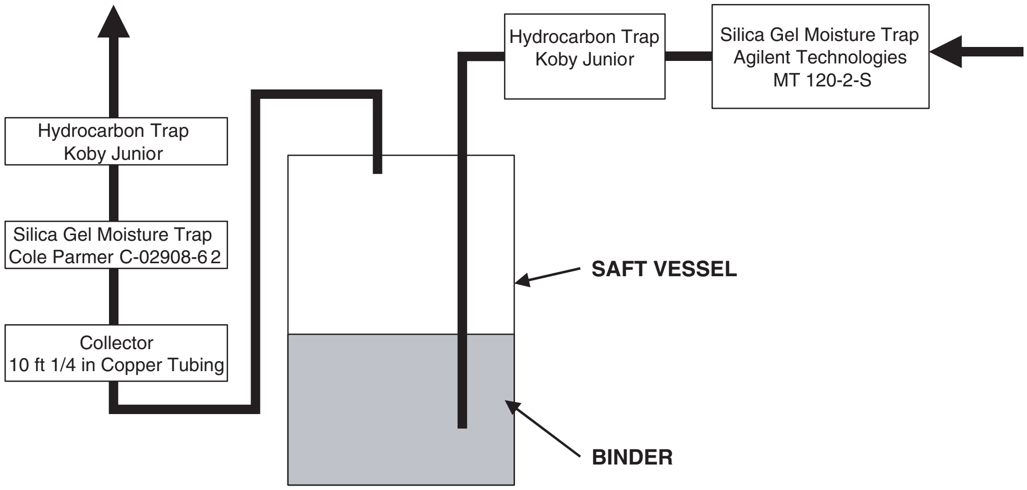


Figure 3-19. Schematic of VCS-I.

a PG 58-28 binder with an average RTFOT mass change of –0.343 percent. Triplicate 250-g samples were aged for 45 min at 163°C using an airflow rate of 4,000 mL/min. Three different conditions were used to produce the flow: slight positive pressure, vacuum with 90-kPa absolute pressure, and vacuum with 70-kPa absolute pressure. The subatmospheric pressures (70 kPa and 90 kPa absolute) were produced by applying a vacuum downstream, essentially sucking the air through the SAFT vessel.

Figure 3-20 presents the results of this testing. As shown, a significant mass is collected in each component of the VCS. Approximately 73 percent of the total is collected in the hydrocarbon trap, 17 percent in the moisture trap, and 10 percent in the condenser. The amount collected in the hydrocarbon trap exceeds the RTFOT mass change for this binder, which is reasonable considering the mass change measurement includes mass loss due to volatilization and mass gain due to oxidation.

VCS-I also was used to collect volatiles from successive runs of the same binder. In this study, dedicated condensers, silica gel filters, and activated hydrocarbon filters were used, and the components of the VCS were not cleaned or purged

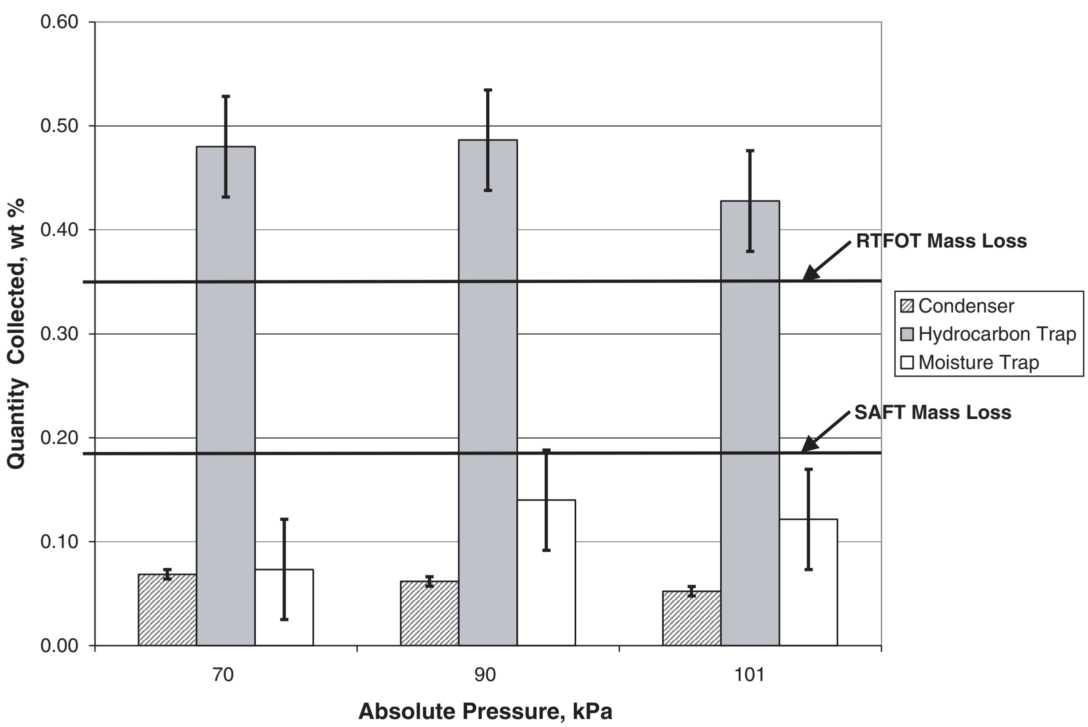


Figure 3-20. Mass of material collected in the improved VCS, VCS-I.

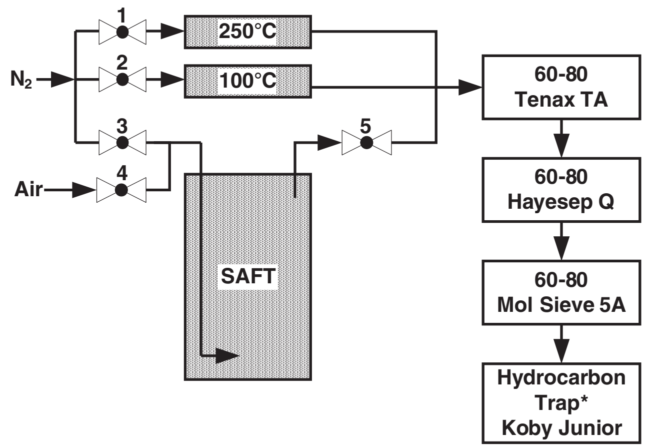
between successive runs. This allowed the volatiles from multiple runs to be combined to collect sufficient materials for analysis. The mass collected in each component for each run is shown in Figure 3-21. The mass collected in the silica gel decreases significantly from the first to the third run. Based on this observation and the observation that the silica gel acquired a brownish color, it appeared that the silica gel adsorbs hydrocarbons as well as water. Further, the results show that the efficiency of the silica gel decreases with successive runs, most likely caused by interference from absorbed polar compounds. These observations prompted the need for a revised VCS design.

The second iteration of the improved VCS, called VCS-II, was designed to use filters that are commonly used in chromatographic analyses. The resulting system is shown in Figure 3-22. Although not shown in Figure 3-22, the inlet silica gel and activated carbon filters from the VCS-I were retained to condition the incoming nitrogen and air.

VCS-II used two absorbent polymer resin filters, Tenax TA and HayeSep Q to remove, respectively, the larger molecular weight polar materials (aromatics) and the remaining hydrocarbons, while the molecular sieve was selected to remove water. The activated carbon filter was included only to ensure the efficiency of the resin beads and molecular sieve. Nothing should be collected in the activated carbon filter if the system is properly designed. The system shown in Figure 3-22 was not intended for routine use but instead was selected to provide an understanding of the nature of the volatiles that are being released and to form a basis for selecting a simplified system.

Each of the absorbents was contained in a 7-in. (175-mm) long, 0.5-in (12.5-mm) diameter stainless steel tube and were connected with rubber tubing. A photograph of an assembled filter is shown in Figure 3-23. The filter beds can be reused by purging them of collected volatiles by passing high-temperature

Process Step	Flow Path				
	1	2	3	4	5
1. Purge filter system	O	C	C	C	C
2. Preheat binder	C	C	O	C	O
3. Age binder	C	C	C	O	O
4. Flush H_2O at end of run	O	C	C	C	C



*Hydrocarbon trap may be redundant for this system.

Note: Flow diagram for descriptive purposes only and not to be used to lay out system. Air is pre-dried with a commercial drier and further cleaned by passing the air through a silica gel and charcoal filter before it is introduced into the SAFT.

Figure 3-22. VCS based on chromatographic filters, VCS-II.

nitrogen through them. Estimated life of the absorbents, as predicted by the manufacturer's technical support staff, is in excess of 50 runs.

Data on the magnitude and repeatability of the mass changes for each of the collectors were obtained for duplicate runs of binders: Citgo PG 58-28, ABM-2, AAM-1, and AAD-2. The

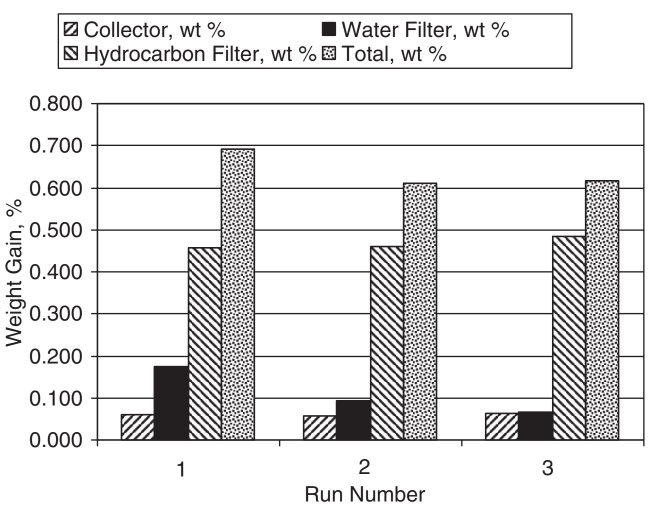


Figure 3-21. Average amount of volatiles captured in successive runs, VCS-I.

mass of the volatiles collected during replicate runs for the four binders is shown in Table 3-12 for the SAFT with the VCS-II and the RTFOT. Surprisingly, the majority of the volatiles collected in the VCS-II were collected in the molecular sieve and not on the Tenax TA and HayeSep Q filter beds. For example, for the 58-28 binder, 80 percent of the volatiles were collected on the molecular sieve while only 20 percent were collected on the Tenax TA and HayeSep Q, with similar results for the other three binders. When the Tenax TA was challenged (purged) at the end of the run, considerable smoking was observed as the volatiles were released. The odor was acrid in nature, unlike the smell of asphalt binder. When the HayeSep Q was purged, no smoke was observed but an asphalt-like odor was observed. The volatiles driven from the molecular sieve have a hydrocarbon odor somewhat similar to asphalt cement.

Material collected during the last run for binder AAD-2 was sent to Heritage Research Group to determine the composition of the volatiles collected in each filter. The procedure used by Heritage was to first sequentially elute each of the three tubes with hexane, methylene chloride, and methanol. Each eluent was analyzed by gas chromatography with flame ionization detection (GC/FID) for quantification of the organic chromatographic material. Use of the three different solvents allowed the removal of compounds of varying polarities. Water present in each of the eluents was determined by Karl Fisher testing using a Mettler Toledo DL-38 unit. This analysis showed that essentially all of the water was collected in the molecular sieve. The Tenax TA filter collected 91.2 percent of the organic material, the HayeSep Q filter collected 8.2 percent, and the molecular sieve collected the remaining 0.6 percent. The mass of organic material collected on each of the filters varied somewhat from the mass changes presented in Table 3-12; however, given the tendency to lose highly volatile components during handling and the nature of the GC measurements, the results were considered compatible. The chromatograms were, as expected, showing compounds similar to those obtained previously in asphalt fume studies conducted by Heritage Research Group.

The results of the mass change and chemical analyses indicated that further refinements of the VCS were warranted. The compositional analysis of the material collected in each filter indicated that some organic material was collected on the molecular sieve. This indicated that the length of the HayeSep Q was too short. As part of the VCS-III design study, an experiment was conducted to establish the length of HayeSep Q collector required to minimize breakthrough. The length of the HayeSep Q collector was varied from 1.5 in. (37 mm) to 4.5 in. (111) mm by adding one or two 1.5-in. (37-mm) lengths to the original 1.5-in. (37-mm) length and the mass collected in each length was then weighed. This experiment found very little difference in the total amount captured when the HayeSep Q bed varies from 1.5 in. (37 mm) to 4.5 in. (111 mm). A conservative HayeSep Q filter bed length of 3.9 in. (100 mm) was selected for the VCS-III based on these results.

Internal discussions as well as input from others outside the study suggested that the Tenax TA filter could be removed from the system. Removing the Tenax TA filter bed would simplify and reduce the cost of the VCS. Replicate SAFT runs were conducted with various lengths of HayeSep Q with and without the Tenax TA. This experiment found very little difference in the weights captured on the HayeSep Q plus Tenax TA filters and on the HayeSep Q filter only.

The final configuration for the VCS-III is shown schematically in Figure 3-24. It consists of silica gel and activated carbon filters in the inlet gas stream. The outlet stream passes through a (3.9-in.) 100-mm long HayeSep Q collector to collect hydrocarbons and a 5-angstrom molecular sieve to collect water. The HayeSep Q collector and the molecular sieve are challenged prior to testing by passing nitrogen gas at 2 L/min and 250°C inlet temperature for 15 minutes.

3.4.3 Evaluation of Improved VCS

The commercial SAFT fitted with the VCS-III was used to condition the 12 binders in the validation study. Three replicate samples of each binder were conditioned in the commercial SAFT and the RTFOT. The amount of material collected on the HayeSep Q and the molecular sieve for each of the asphalt binders is shown in Table 3-13 and Figure 3-25. Also shown in Table 3-13 is the negative value of the RTFOT mass

Table 3-12. Mass change for RTFOT and SAFT with VCS-II.

Aging Device	Measured Value	Filter Media	CITGO 58-28	ABM-2	AAM-1	AAD-2
SAFT	Mass Change, Percent of Initial Binder Mass	Tenax TA	0.025	0.020	0.007	0.034
		HayeSep	0.026	0.012	0.008	0.011
		Mol. Sieve	0.204	0.179	0.128	0.166
		Tenax + HayeSep	0.051	0.032	0.015	0.045
		Total	0.255	0.211	0.143	0.211
	Mass Change, Percent of Total Volatiles Collected	Tenax + HayeSep	20	15	10	21
		Mol. Sieve	80	85	90	79
RTFOT	Mass Change	None	0.345	0.348	-0.122	1.058

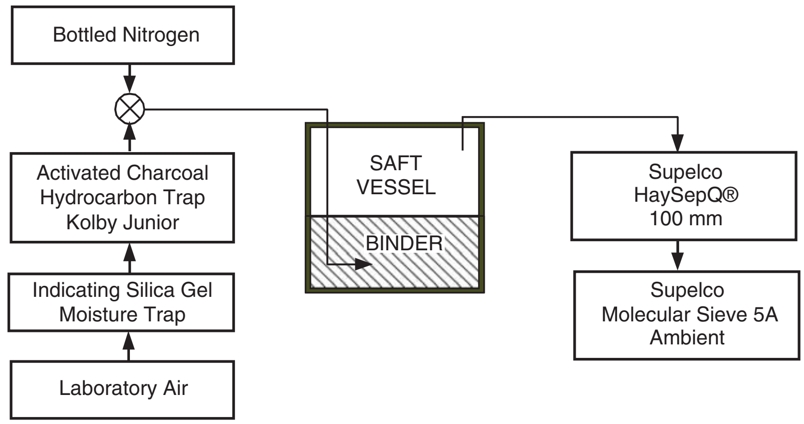


Figure 3-24. Final version of VCS-III.

change and the combined mass of the material collected on the HayeSep Q and the molecular sieve. The negative value of the RTFOT mass change is used so that a positive value for the absorbents and the RTFOT indicate material lost during the conditioning procedure. From Figure 3-25 it appears that the mass of hydrocarbon volatiles collected on the HayeSep Q filter is similar for all of the binders tested ranging from 0.00 to 0.07 percent of the original mass of binder. This range is similar to the range reported for the SAFT prototype VCS. The mass of water collected on the molecular sieve is much lower for the five polymer-modified binders compared to the neat binders and the air blown binder.

There is no apparent correlation between the mass lost during RTFOT conditioning and the mass collected on the individual or combined absorbents. This is illustrated graphically in Figure 3-26 where the mass collected on the absorbents is plotted versus the mass lost during the RTFOT conditioning. This is not surprising given that the RTFOT represents the balance between the oxygen consumed by oxidation, loss of hydrocarbon volatiles, and water resulting from oxidation or other chemical reactions.

Table 3-13. Mass collected on VCS-III filters and RTFOT mass change.

Binder	Mass Collected on Filters, % of Original Mass			Negative of RTFOT Mass Loss, % of Original Mass
	HayeSep Q	Molecular Sieve	Combined HayeSep Q and Molecular Sieve	
AAC-1	0.072	0.141	0.214	0.058
AAD-2	0.041	0.134	0.175	1.058
AAF-1	0.043	0.114	0.157	0.008
ABM-2	0.024	0.113	0.137	0.349
ABL-1	0.049	0.129	0.178	0.654
AAM-1	0.014	0.085	0.100	-0.122
Citgoflex	0.020	0.052	0.072	0.196
ALF 64-40	0.046	0.007	0.053	0.207
Airblown	0.020	0.061	0.081	-0.031
Elvaloy	0.000	0.003	0.003	0.173
EVA	0.020	0.005	0.026	0.132
Novophalt	0.025	0.006	0.031	0.132

3.5 SAFT Optimization Study

The objective of the SAFT optimization study was to enhance the efficiency of the operating parameters for the commercial SAFT so that it more nearly reproduced the degree of aging that occurs in the RTFOT for neat binders. During the VCS study, it was observed that the commercial version of the SAFT, when operated using the parameters recommended for the prototype, produced significantly less aging than the RTFOT. Recall, the developers of the SAFT documented good agreement between the prototype SAFT and the RTFOT. The likely cause of the lower aging in the commercial SAFT is the improved temperature control provided by this device. In the prototype, temperature control was provided by a heating mantle that was in direct contact with the SAFT vessel. In the commercial SAFT, the vessel is heated in an oven. The process controller limits the maximum temperature in the oven to 176°C. The heating mantle, on the other hand, can reach very high temperatures, and because it is in direct contact with the steel SAFT vessel, the binder at the wall of the vessel can also reach temperatures well above the test temperature of 163°C. More rapid aging of the binder occurs in areas of the SAFT vessel with the highest temperature.

The SAFT optimization study included two experiments. The first was an experiment to verify that the heat-up phase of the test does not result in significant aging of the binder. During the heat-up phase, the temperature of the binder is increased from approximately 100°C to the testing temperature of 163°C while nitrogen flows through the SAFT vessel. Since the starting temperature (temperature of the binder after charging the SAFT vessel) cannot be accurately controlled, it is critical that the heat-up phase not contribute significantly

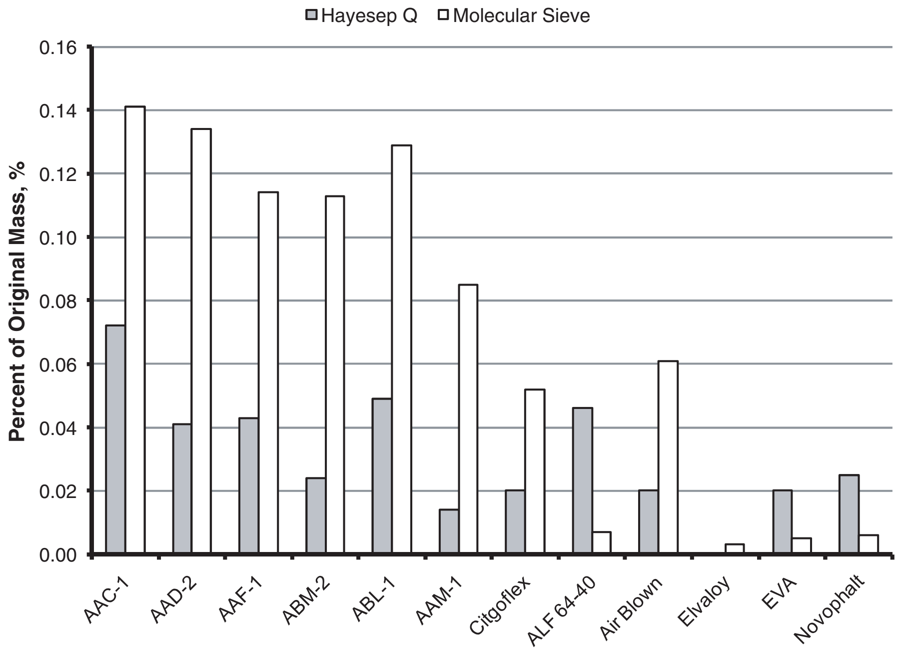


Figure 3-25. Mass collected on filters.

to the degree of aging that occurs in the test. The second experiment was an experiment to determine the effects of impeller speed, airflow rate, and test duration on the degree of aging measured by the rheology of the binder at high in-service pavement temperatures. Operating parameters for the commercial SAFT were selected from this experiment.

The SAFT optimization study is described in detail in Appendix D (see the project webpage on the TRB website). Its key findings are discussed in the following sections.

3.5.1 Heat-Up Effects Experiment

During the heat-up portion of the SAFT test, the temperature of the binder is increased from approximately 100°C to the testing temperature of 163°C while nitrogen flows through the SAFT vessel. Since the starting temperature (temperature of the binder after charging the SAFT vessel) cannot be accurately controlled, it is critical that the heat-up phase not contribute significantly to the degree of aging that occurs in the

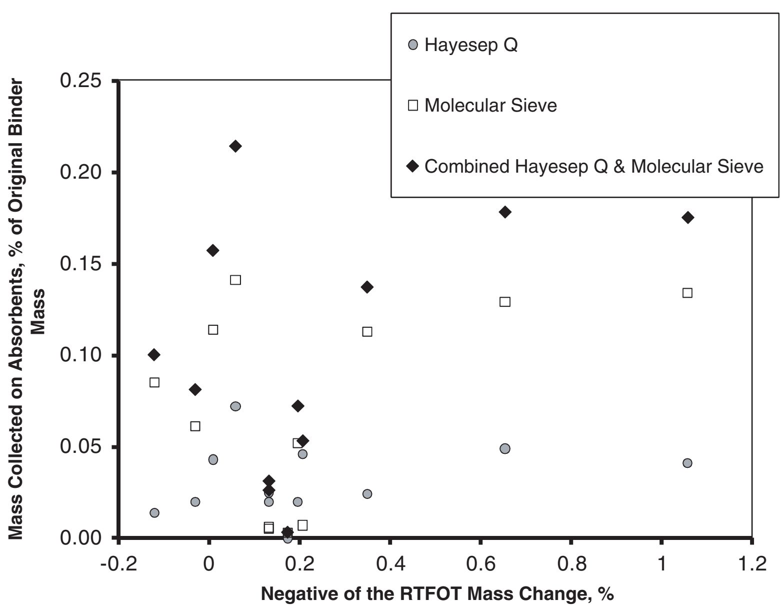


Figure 3-26. Material collected on molecular sieve and HayeSep Q versus RTFOT mass change.

Table 3-14. High-temperature DSR data from heat-up effects experiment.

Binder	Temp, °C	Rep	Tank G*, kPa	Tank δ, deg	Tank G*/sinδ, kPa	Tank Average G*/sinδ, kPa	After Heat Up G*, kPa	After Heat Up δ, deg	After Heat Up G*/sinδ, kPa	After Heat Up Average G*/sinδ, kPa
PG 58-28	58	1	1.18	87.1	1.18	1.24	1.20	87.0	1.20	1.25
		2	1.30	86.9	1.30		1.29	87.0	1.29	
PG 76-22	76	1	1.45	83.6	1.46	1.48	1.44	84.1	1.45	1.39
		2	1.50	83.3	1.51		1.33	83.9	1.34	
PG 82-22	82	1	1.54	66.3	1.68	1.71	1.54	65.9	1.69	1.71
		2	1.59	66.1	1.74		1.58	65.6	1.73	

test. To assess the change in the properties of the asphalt binder during the heat-up period DSR, measurements at 10 rad/s were made at the high pavement temperature for three binders (PG 58-28, PG 76-22, and PG 82-22). DSR measurements were made for each binder in the tank condition and on material removed from the SAFT after completion of the heat-up phase. Initial testing showed that the SAFT trapped air bubbles in the PG 82-22 binder; therefore, for this experiment and the operating parameters experiment discussed in the next section, both the tank and the materials sampled at the end of the heat up-up period were exposed to the PAV vacuum degassing procedure to remove the trapped air before they were tested in the DSR.

The DSR data from this experiment are summarized in Table 3-14. For a given binder, all of the DSR data are within the AASHTO T315 single-operator precision of 9.5 percent for testing of original binder. Since the complex modulus before and after the heat-up phase varies by less than the single-operator precision, this experiment found that the binder does not significantly stiffen during the SAFT heat-up phase.

3.5.2 Operating Parameters Experiment

The purpose of this experiment was to determine the effect of three operating parameters—impeller speed, airflow rate, and conditioning time—on the properties of asphalt binder aged in the commercial SAFT. Three different unmodified binders, listed in Table 3-15, were chosen for this experiment. The binders, which have been studied extensively elsewhere, represent a range in chemical composition, mass change, and sensitivity to short-term aging. No modified binders were included because the purpose of the experiment was to determine the SAFT operating parameters that best mimic the aging that occurs in the RTFOT for neat binders.

The experiment design for the operating parameters experiment is presented in Table 3-16. It employed a Plackett-Burman design to simultaneously assess the effects of changes in impeller speed, airflow rate, and conditioning time on the aging that occurs in the commercial SAFT. Plackett-Burman designs are often used in ruggedness testing to assess the effect of changes in multiple test parameters. These are extremely efficient designs that allow the main effects to be determined with a limited amount of testing. For the three variables included in this experiment only four test results are needed to assess the main effects. ASTM E1169-02 (*32*) presents detailed information on the design and analysis of the type of experiment used.

The rheological data from the operating parameters experiment are summarized in Table 3-17. Two statistical analyses were performed on the DSR data shown in Table 3-17. The first analysis was performed to determine if the PAV degassing procedure affects the DSR data obtained after short-term aging of neat binders in the SAFT. Recall that previous work with modified binders during the heat-up effects experiment showed that degassing was a necessary step for modified

Table 3-15. Binders for the optimization study.

Binder/Source	Comments	PG Grade	RTFOT Mass Change, %	Aging Index, 135°C Viscosity
California Coastal (AAD-2)	SHRP core binder except AAD-1 used during SHRP	52-28	-1.058	2.86
West Texas Intermediate (AAM-1)	SHRP core asphalt	64-16	+0.122	1.98
California Valley (ABM-2)	Replacement for SHRP core binder AAG-2, except AAG-1 used during SHRP	58-16	-0.348	1.62

Table 3-16. Operating parameters experiment design.

Binder Source	Impeller Speed (rpm)	Air Flow (L/Min)	Aging Time (Min)	Testing Plan
AAD-2	700	2	45	Yes
			60	-- --
		4	45	-- --
			60	Yes
	1400	2	45	-- --
			60	Yes
		4	45	Yes
			60	-- --
AAM-1	700	2	45	Yes
			60	-- --
		4	45	-- --
			60	Yes
	1400	2	45	-- --
			60	Yes
		4	45	Yes
			60	-- --
ABM-2	700	2	45	Yes
			60	-- --
		4	45	-- --
			60	Yes
	1400	2	45	-- --
			60	Yes
		4	45	Yes
			60	-- --

binders aged in the SAFT. For simplicity, the study team used the vacuum degassing procedure currently specified in AASHTO R28-06, *Standard Practice for Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel (PAV)*. This procedure exposes the binder to 170°C for 40±2 minutes. For the last 30±1 minutes, the binder is exposed to vacuum with a residual pressure of 15±2.5 kPa absolute. The effect of the degassing process was evaluated by performing DSR tests on three split samples of the SAFT-aged material. One sample was immediately cooled at ambient temperature, a second sample was exposed to 170°C for 40 minutes, and the third sample was exposed to the PAV degassing procedure. A paired difference analysis was used to assess the effect of degassing. Details of the analysis are presented in Appendix D (see the project webpage on the TRB website). The following three differences were considered:

1. Difference between degassed and air-cooled binder,
2. Difference between air-cooled binder and binder heated per the degassing procedure but without the application of vacuum (oven treatment), and
3. Difference between degassed binder (degassed) and binder heated per the degassing procedure but without the application of vacuum (oven treatment).

The results of this analysis are summarized in Table 3-18 and include the mean difference ($\bar{d}$), its standard deviation (S_d), the calculated *t* statistic (T), and its percentage value (p). Low p values (bold and underlined in Table 3-18) indicate significant differences. This analysis found that the degassing procedure significantly affects the rheology of neat SAFT-aged binders, and that it is probably the additional exposure to high temperature that causes this additional aging. Since degassing is needed to remove entrapped air from stiff modified binders, this experiment shows that it must be included as part of the procedure for all binders, and that the degassing must be standardized.

The second analysis that was performed investigated the effects of impeller speed, airflow rate, and conditioning time on the degree of aging that occurs in the SAFT to determine the sensitivity of the SAFT aging to these parameters. Temperature was not included in the experiment because the

Table 3-17. DSR data from the operating parameters experiment.

Binder	Order	Operating Parameters			DSR at 58°C											
					Degassed			Oven Treatment			Air Cooled			RTFOT, AVG		
		Speed rpm	Flow L/Min	Time Min	G*, kPa	δ, deg	G*/sinδ, kPa	G*, kPa	δ, Deg	G*/sinδ, kPa	G*, kPa	δ, Deg	G*/sinδ, kPa	G*, kPa	δ, deg	G*/sinδ, kPa
AAD-2	1	700	4	60	1.72	81.5	1.74	1.69	81.6	1.71	1.68	81.5	1.70	2.60	79.0	2.65
AAD-2	2	1,400	4	45	4.16	74.6	4.31	4.20	74.6	4.36	4.03	75.0	4.17			
AAD-2	3	1,400	2	60	4.73	73.2	4.94	4.71	73.4	4.91	4.37	73.6	4.56			
AAD-2	4	700	2	45	1.30	83.4	1.31	1.36	83	1.37	1.32	83.0	1.33			
AAM-1	1	700	4	60	5.43	82.4	5.48	5.31	82.3	5.36	5.04	82.5	5.08	5.94	81.9	6.00
AAM-1	2	1,400	2	60	8.99	78.7	9.17	9.22	78.6	9.41	8.52	78.7	8.69			
AAM-1	3	1,400	4	45	7.37	79.9	7.49	7.57	79.9	7.69	7.03	80.0	7.14			
AAM-1	4	700	2	45	4.13	83.8	4.15	4.37	83.5	4.40	3.77	83.2	3.80			
ABM-2	1	1,400	4	45	4.39	88.8	4.39	4.27	88.8	4.27	4.44	88.8	4.44	3.14	89.5	3.14
ABM-2	2	1,400	2	60	5.24	88.5	5.24	5.04	88.5	5.04	5.39	88.5	5.39			
ABM-2	3	700	2	45	2.45	89.4	2.45	2.50	89.5	2.50	2.54	89.5	2.54			
ABM-2	4	700	4	60	2.85	89.4	2.85	2.83	89.3	2.83	2.97	89.3	2.97			

Table 3-18. Summary of degassing effects.

Parameter	Paired Differences		
	Degassed—Air Cooled	Oven Treatment—Air Cooled	Degassed—Oven Treatment
$\bar{d}$	0.14	0.17	-0.03
S_d	0.23	0.34	0.14
T	2.11	1.75	-0.64
p	**0.03**	**0.05**	0.74

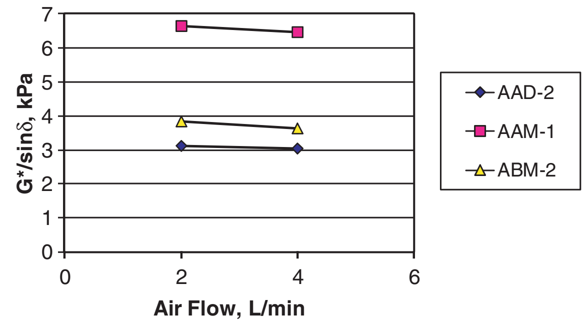


Figure 3-28. Effect of airflow on G/sinδ for SAFT-aged material.*

review of the literature and research in progress indicated a strong desire to perform the short-term aging test at 163°C, which reasonably simulates hot mix plant operating temperatures. Figures 3-27 through 3-29 show the effect of the three operational parameters on G*/sinδ measured after SAFT aging and degassing.

From these figures it is clear that the aging in the SAFT is affected by impeller speed and conditioning time, but not by airflow rate over the ranges studied. Using the slopes in these figures, the RTFOT G*/sinδ values measured for three binders, and assuming linear relationships, the impeller speed required to reproduce RTFOT aging at the average conditioning time of 52.5 minutes and the conditioning time required to reproduce RTFOT aging at the average impeller speed of 1,050 rpm can be estimated. These are summarized for the three binders in Table 3-19.

Based on Table 3-19, it appeared that using an impeller speed of 1,000 rpm, an airflow rate of 2,000 mL/min, and a conditioning time of 45 minutes would result in aging in the commercial SAFT that is approximately equivalent to that which occurs in the RTFOT.

The analysis presented in this section assumes linear effects. Developmental work on the VCS-II provided an opportunity to assess the tentative operating parameters. Independent rheological data (average of duplicate runs) collected during the VCS-II testing are presented in Table 3-20. These data show that the operating conditions listed above for the SAFT underestimates the aging that occurs with the RTFOT in three of the four cases. Either the conditioning time or the impeller speed can be increased to increase the degree of aging in the SAFT. The magnitude of these changes was estimated to be a 100-rpm increase in impeller speed or a 5-minute increase in conditioning time to increase G*/sinδ by 7 percent. After careful consideration, the longer conditioning time was selected because of concern that further increases in impeller speed could result in significant quantities of binder splashing onto the lid of the SAFT with the potential for asphalt droplets to exit into the VCS. Visual observation from completed tests indicates that at 1,000 rpm no asphalt splashes on the lid, but at 1,400 rpm a significant amount of asphalt splashes onto the lid.

Table 3-21 presents rheological data for the Citgo PG 58-28 binder where the conditioning time was increased from 45 to 50 minutes. As shown, this change increased G*/sinδ by approximately 6.5 percent. With the new operating parameters, the SAFT ages the Citgo PG 58-28 binder slightly more than the RTFOT, but based on Table 3-20, this increased aging should provide better agreement when a wide range of binders is considered.

The final operating parameters selected from the operating parameters experiment for the commercial SAFT for use in the verification study were

- 163°C aging temperature,
- 2,000 mL/min airflow,

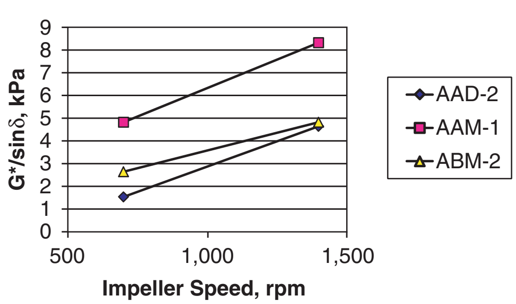


Figure 3-27. Effect of impeller speed on G/sinδ for SAFT-aged material.*

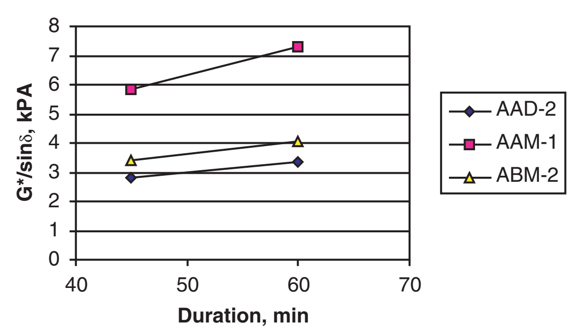


Figure 3-29. Effect of conditioning time on G/sinδ for SAFT-aged material.*

Table 3-19. Estimated SAFT operational parameters to reproduce RTFOT aging.

Binder	RTFOT G*/sinδ, kPa	Estimated Impeller Speed, for 52.5-Min Conditioning, rpm	Estimated Conditioning Time for 1,050 rpm Impeller Speed, Min
AAD-2	2.65	962	40
AAM-1	6.00	935	47
ABM-2	3.14	857	38
Average		918	42

- 1,000 rpm impeller speed,
- 50-minute aging time,
- 250-g sample mass, and
- Vacuum degassing per AASHTO R28 after short-term aging in the SAFT.

Figure 3-30 illustrates the sequence of operations for the commercial SAFT determined from the SAFT optimization study. This sequence was used in the verification study.

3.6 Verification Study

The objectives of the verification study were to (1) confirm that the commercial versions of the SAFT and MGRF reproduce the degree of aging obtained in the RTFOT for a wide range of neat binders and (2) compare the aging from the SAFT, MGRF, and RTFOT with that from mixture samples aged in a forced-draft oven in accordance with the performance testing protocol contained in AASHTO R30. Initially, the verification study only included the SAFT, but it was expanded at the request of the project panel to include the MGRF.

The verification study consisted of two main components—the RTFOT verification experiment and the oven-aged mixtures experiment. The RTFOT verification experiment, which included DSR and BBR measurements, was designed to provide master curves for the binders in the tank condition and after SAFT, MGRF, and RTFOT conditioning. These binder master curves served the following two purposes:

1. To enable a comparison of the rheological properties of material conditioned in the SAFT, MGRF, and RTFOT, and
2. To allow a comparison of the master curves measured for the binders with master curves back-calculated from mixture properties.

In the oven-aged mixtures experiment, hot mix asphalt using the binders from the RTFOT verification experiment were prepared and aged in accordance with AASHTO R30. Dynamic modulus master curve tests were performed on the mixture samples. From the mixture modulus master curves, the binder stiffness was estimated using the Hirsch Model (*6*) and compared to the measured stiffnesses obtained from the SAFT, MGRF and RTFOT.

The verification study used six neat MRL binders and six polymer-modified binders in both experiments. A single limestone mixture was used in the oven-aged mixture experiment. Detailed information for the binders and the mixture was presented earlier in Chapter 2.

The sections that follow present key findings from the verification study. Full details of the study are included in the verification study report in Appendix E (see the project webpage on the TRB website). The verification study was the last study in NCHRP 9-36 and provided the basis for making the final recommendation with respect to a replacement for the RTFOT.

3.6.1 RTFOT Verification Experiment

The RTFOT verification experiment included comparisons of the short-term conditioned specification parameter, G*/sinδ, mass change, master curves, and aging indices for binders conditioned in the RTFOT, SAFT, and MGRF. It was not possible to obtain good quality data for the EVA modified

Table 3-20. Rheological properties from VCS-II development study.

Property	Aging Condition	Citgo 58-28	ABM-2	AAM-1	AAD-2
G*, kPa	Original	1.18	1.89	2.95	0.88
	After SAFT	2.44	3.17	4.94	2.19
	After RTFOT	2.48	3.15	5.41	2.60
Phase Angle, deg	Original	87.3	89.7	85.6	85.8
	After SAFT	83.7	89.2	82.7	79.7
	After RTFOT	84.1	89.5	80.3	79.0
G*/sinδ	Original	1.18	1.89	2.96	0.88
	After SAFT	2.45	3.17	4.98	2.23
	After RTFOT	2.49	3.15	5.49	2.65

Table 3-21. Rheological properties for the Citgo PG 58-28 for SAFT aging times of 45 and 50 minutes compared to RTFOT.

Property	RTFOT	SAFT 45-Min Aging	SAFT 50-Min Aging
G*, kPa	2.48	2.44	2.65
Phase Angle, deg	84.1	83.7	83.9
G*/sinδ, kPa	2.49	2.45	2.66

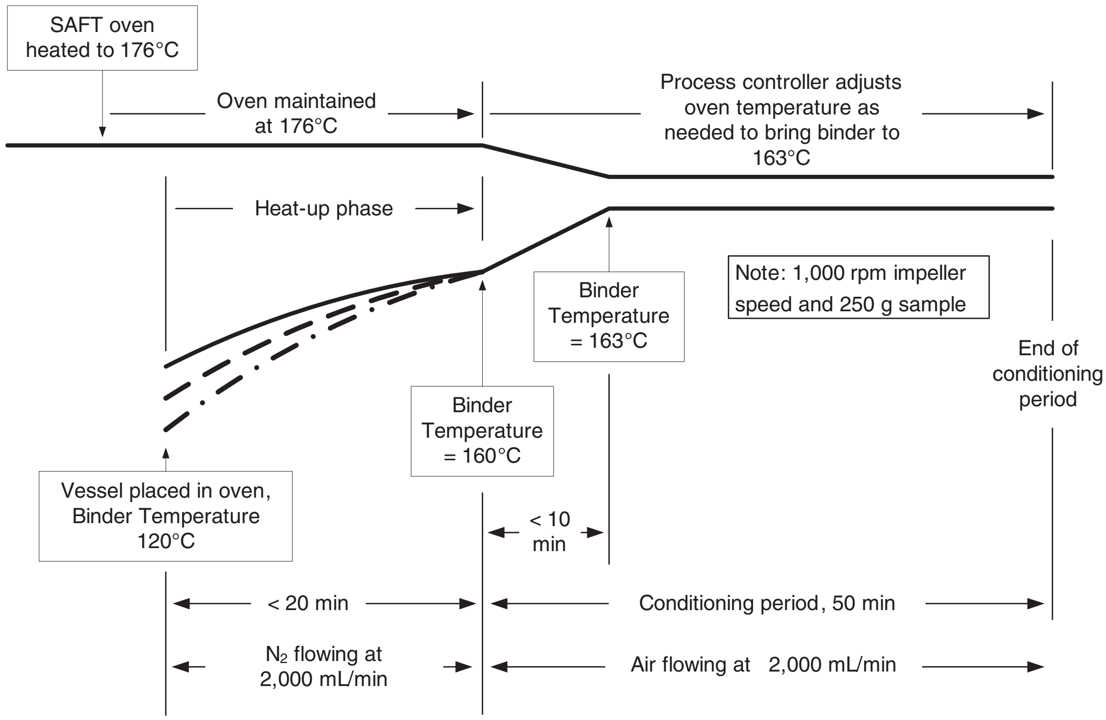


Figure 3-30. Sequence of operations used in final SAFT configuration.

binder because the polymer tended to separate during testing; therefore, this binder was not included in the comparisons described below.

3.6.1.1 Specification Parameter G/sinδ*

The continuous-grading temperatures for RTFOT-, SAFT-, and MGRF-conditioned binders were calculated by interpolating between the logarithm of two measurements, one obtained above the specification criterion of 2.20 kPa and the other below the specification criterion. The continuous-grading temperatures are summarized in Table 3-22. This table includes the average and standard deviation from two separate runs for each device.

Figure 3-31 compares the average continuous-grading temperatures from SAFT and MGRF conditioning to RTFOT conditioning. Figure 3-31 includes trend lines for the SAFT and MGRF data. This plot clearly shows the better agreement for the MGRF compared to the SAFT. The trend line for the MGRF data coincides with the line of equality, while that for the SAFT

Table 3-22. Continuous-grade temperatures for RTFOT, SAFT, and MGRF conditioning.

Binder	Continuous-Grade Temperature, °C					
	RTFOT		MGRF		SAFT	
	Average	Standard Deviation	Average	Standard Deviation	Average	Standard Deviation
AAC-1	56.3	0.21	58.1	0.35	57.2	0.07
AAD-2	59.7	0.49	59.5	0.14	58.0	0.00
ABM-2	60.7	0.35	61.1	0.21	60.5	0.35
AAF-1	67.0	0.44	68.4	2.90	65.5	0.28
AAM-1	68.0	0.35	68.6	1.27	65.0	0.28
ABL-1	69.5	0.35	68.0	0.21	65.3	0.57
Elvaloy	69.9	1.13	68.9	0.14	61.6	0.35
ALF	75.4	0.00	75.2	0.49	69.2	0.21
Novophalt	78.9	0.57	80.0	0.28	73.9	0.14
Airblown	80.2	0.92	79.9	2.05	76.5	0.57
Citgoflex	85.3	0.21	85.9	0.14	82.2	0.21

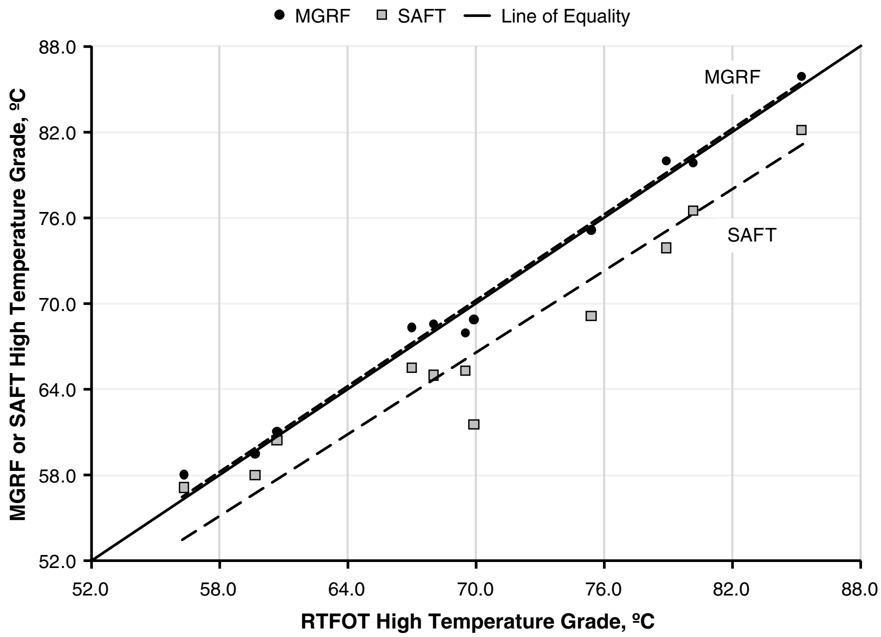


Figure 3-31. Comparison of SAFT and MGRF continuous-grading temperatures to RTFOT continuous-grading temperature.

indicates less stiffening, particularly for high-stiffness binders. Figure 3-32 shows the difference between the continuous-grading temperatures for the SAFT- and MGRF-conditioned binders compared to the RTFOT-conditioned binders. The difference for the SAFT appears to depend on the stiffness of the binder, and for stiffer binders the difference can reach as much as a full grade. The difference for the MGRF is within ±1.8°C and does not appear to depend on the stiffness of the binder.

Paired difference t-testing was used to assess the significance of the differences shown in Figures 3-31 and 3-32. The analysis was conducted for three groups: (1) neat binders, (2) modified binders, and (3) all binders. The results of this analysis are summarized in Tables 3-23, 3-24, and 3-25 for

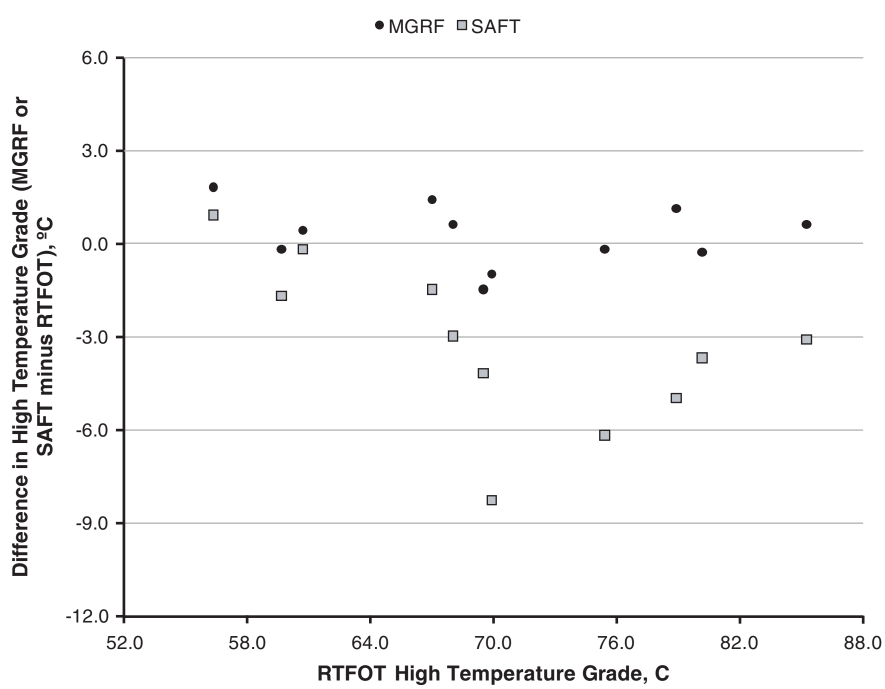


Figure 3-32. Difference in continuous-grading temperature for SAFT and MGRF residue compared to RTFOT residue.

Table 3-23. Summary of paired t-test for neat binders.

Binder	Continuous-Grading Temperature, °C			Differences (MGRF or SAFT minus RTFOT), °C	
	RTFOT	MGRF	SAFT	MGRF	SAFT
AAC-1	56.3	58.1	57.2	1.8	0.9
AAD-2	59.7	59.5	58.0	-0.2	-1.7
ABM-2	60.7	61.1	60.5	0.4	-0.2
AAF-1	67.0	68.4	65.5	1.4	-1.5
AAM-1	68.0	68.6	65.0	0.6	-3.0
ABL-1	69.5	68.0	65.3	-1.5	-4.2
Average Difference, °C				0.42	-1.62
Standard Deviation of Differences, °C				1.18	1.84
Calculated t				1.034	-2.151
$t_{critical}$ (0.05, 5 degrees of freedom)				2.015	2.015
Conclusion				No Difference	SAFT Lower

the three groups. Details of the analysis are presented in Appendix E. This analysis shows that the continuous grade is the same for RTFOT and MGRF conditioning. The high-temperature grade is lower for SAFT conditioning. This finding holds for both neat and modified binders.

Since the testing included replicate samples of each binder conditioned in the three short-term conditioning devices, an initial evaluation of the variability of the SAFT and MGRF relative to the RTFOT was made. This evaluation was done using an F-test on the pooled variance computed from the variances of the duplicate tests for the 11 binders. This analysis is summarized in Table 3-26 and shows that the test variability is somewhat greater for the MGRF compared the RTFOT. Variability for the SAFT is similar to the RTFOT. Details of this analysis are presented in Appendix E.

The MGRF variability was high for 2 of the 11 binders tested: AAF-1 and Airblown. The variability for the other binders was similar to that from the RTFOT. It is expected

Table 3-24. Summary of paired t-test for modified binders.

Binder	Continuous-Grading Temperature, °C			Differences (MGRF or SAFT minus RTFOT), °C	
	RTFOT	MGRF	SAFT	MGRF	SAFT
AAC-1	56.3	58.1	57.2	1.8	0.9
AAD-2	59.7	59.5	58.0	-0.2	-1.7
ABM-2	60.7	61.1	60.5	0.4	-0.2
AAF-1	67.0	68.4	65.5	1.4	-1.5
AAM-1	68.0	68.6	65.0	0.6	-3.0
ABL-1	69.5	68.0	65.3	-1.5	-4.2
Average Difference, °C				0.42	-1.62
Standard Deviation of Differences, °C				1.18	1.84
Calculated t				1.034	-2.151
$t_{critical}$ (0.05, 5 degrees of freedom)				2.015	2.015
Conclusion				No Difference	SAFT Lower

Table 3-25. Summary of paired t-test for all binders.

Binder	Continuous-Grading Temperature, °C			Differences (MGRF or SAFT minus RTFOT), °C	
	RTFOT	MGRF	SAFT	MGRF	SAFT
AAC-1	56.3	58.1	57.2	1.8	0.9
AAD-2	59.7	59.5	58.0	-0.2	-1.7
ABM-2	60.7	61.1	60.5	0.4	-0.2
AAF-1	67.0	68.4	65.5	1.4	-1.5
AAM-1	68.0	68.6	65.0	0.6	-3.0
ABL-1	69.5	68.0	65.3	-1.5	-4.2
Elvaloy	69.9	68.9	61.6	-1.0	-8.3
ALF	75.4	75.2	69.2	-0.2	-6.2
Novophalt	78.9	80.0	73.9	1.1	-5.0
Airblown	80.2	79.9	76.5	-0.3	-3.7
Citgoflex	85.3	85.9	82.2	0.6	-3.1
Average Difference, °C				0.27	-3.24
Standard Deviation of Differences, °C				1.00	2.65
Calculated t				0.882	-4.054
$t_{critical}$ (0.05, 10 degrees of freedom)				1.812	1.812
Conclusion				No Difference	SAFT Lower

that as technicians gain more experience with the MGRF, its variability should become similar to that for the RTFOT.

3.6.1.2 Mass Change

The MGRF test includes a mass change measurement that is determined in the same manner as the RTFOT: the change in mass is calculated from the mass of the flask measured before and after aging. As discussed in Section 3.4, the SAFT uses a VCS to collect and then weigh the mass of volatiles exiting the vessel during aging. The performance of the VCS was discussed earlier in Section 3.4.3, so only the comparison of the mass change in the MGRF and RTFOT will be presented here.

Mass change data from the MGRF and RTFOT are summarized in Table 3-27 and compared in Figure 3-33. Figure 3-33 shows that there is a good relationship between the mass change measured in the MGRF and that measured in the RTFOT. The MGRF values are approximately 40 percent of the RTFOT values. This is in agreement with the data collected in the Western Research Institute study of the MGRF (*2*) that was discussed earlier in Section 3.2.2.1.

3.6.1.3 Christensen-Anderson Master Curve Parameters

Binder master curves were developed from the combined DSR and BBR data using the Christensen-Anderson Model (*33*). The Christensen-Anderson Model was used because the parameters in the model are useful in interpreting changes in rheology that occur during laboratory conditioning or in-service aging. Equation 2 presents the Christensen-Anderson

Table 3-26. Analysis of variability of MGRF and SAFT relative to RTFOT.

Binder	Standard Deviation, °C			Variance, $(°C)^2$		
	RTFOT	MGRF	SAFT	RTFOT	MGRF	SAFT
AAC-1	0.21	0.35	0.07	0.043	0.125	0.005
AAD-2	0.49	0.14	0.00	0.243	0.020	0.000
ABM-2	0.35	0.21	0.35	0.120	0.045	0.125
AAF-1	0.44	2.90	0.28	0.190	8.405	0.080
AAM-1	0.35	1.27	0.28	0.123	1.620	0.080
ABL-1	0.35	0.21	0.57	0.120	0.045	0.320
Elvaloy	1.13	0.14	0.35	1.280	0.020	0.125
ALF	0.00	0.49	0.21	0.000	0.245	0.045
Novophalt	0.57	0.28	0.14	0.320	0.080	0.020
Airblown	0.92	2.05	0.57	0.845	4.205	0.320
Citgoflex	0.21	0.14	0.21	0.045	0.020	0.045
Number of Replicates				2	2	2
Pooled Variance				0.3027	1.3482	0.1059
Computed F				NA	4.45	2.86
Critical F (0.05, 10, 10)				NA	2.98	2.98
Conclusion				NA	MGRF Higher	No Difference

Model for the frequency dependency of the binder shear modulus.

$$G^*(\omega) = G_g\left[1+\left(\frac{\omega_c}{\omega_r}\right)^{\frac{\log 2}{R}}\right]^{\frac{-R}{\log 2}} \qquad (2)$$

where

$G^*(\omega)$ = complex shear modulus

G_g = glass modulus, approximately equal to 1GPa

ω_r = reduced frequency at the reference temperature, rad/s

ω_c = cross over frequency at the reference temperature, rad/s

R = rheological index

The shift factors relative to the defining temperature are given by Equations 3 and 4 for temperatures above and below the defining temperature, respectively (*33*).

$$\log a(T) = \frac{-19(T-T_d)}{92+T-T_d} \qquad (3)$$

$$\log a(T) = 13016.07\left(\frac{1}{T}-\frac{1}{T_d}\right) \qquad (4)$$

where

a(T) = shift factor

T = temperature, °K

T_d = defining temperature, °K

Above T_d the Williams-Landel-Ferry (WLF) equation is valid, but below T_d it is no longer valid and the Arrhenius equation must be used. This is because the asphalt binder is not in an equilibrium condition as a result of physical hardening. The four unknown parameters: G_g, ω_c, R, and T_d, were obtained through non-linear least squares fitting of Equations 2, 3, and 4 using the data from the testing program. The parameter, ω_c, changes with temperature and therefore is always given at the reference temperature, selected as 22°C for direct comparison with the data back-calculated from the mixture master curves. To construct the complete master curve, the bending beam rheometer creep stiffness was converted to shear modulus using the following approximate interconversions.

$$G^*(\omega) \approx \frac{S(t)}{3} \qquad (5)$$

$$(\omega) \approx \frac{1}{t} \qquad (6)$$

where

$G^*(\omega)$ = shear modulus

S(t) = creep stiffness

Table 3-27. Mass change data for RTFOT and MGRF.

Binder	Mass Change, %	
	RTFOT	MGRF
AAC-1	-0.058	-0.232
ABL-1	-0.654	-0.345
AAM-1	0.122	0.113
Citgoflex	-0.196	-0.103
Airblown	0.031	0.033
Novophalt	-0.132	-0.045
AAF-1	-0.008	-0.063
ABM-2	-0.349	-0.100
AAD-2	-1.058	-0.362
Elvaloy	-0.173	-0.060
ALF 64-40	-0.207	-0.073

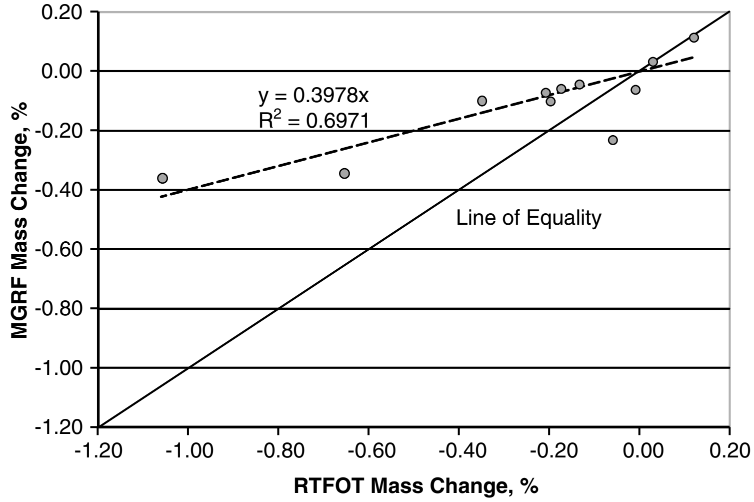


Figure 3-33. Comparison of mass change for MGRF and RTFOT.

ω = frequency in rad/s
t = time in seconds

Figure 3-34 presents an example of the fitted master curve and the nomenclature used with the Christensen-Anderson Model. A major advantage of the Christensen-Anderson Model is that the model parameters have physical significance. The glassy modulus is the limiting maximum modulus and is approximately equal to 1 GPa, reflective of the stiffness of carbon–carbon bonds that predominate in asphalt binders. The viscous asymptote is the 45° line that the master curve approaches at low frequencies and is an indicator of the steady-state viscosity of the binder. The crossover frequency is the frequency where the phase angle is 45 degrees and is typically close to the point where the viscous asymptote intersects the glassy modulus. The crossover frequency, ω_c, is an indicator of the hardness of the binder. Finally, the rheological index, R, is the difference between the log of the glassy modulus and the log of the dynamic modulus at the crossover frequency. It is an indicator of the rheological type.

Table 3-28 summarizes the Christensen-Anderson Model parameters for tank, RTFOT, SAFT, and MGRF conditioning. The effect of conditioning on the rheology of the binder is best represented by changes in the model parameters from tank condition to short-term aged condition. Figures 3-35 through 3-37 compare changes for SAFT and MGRF condi-

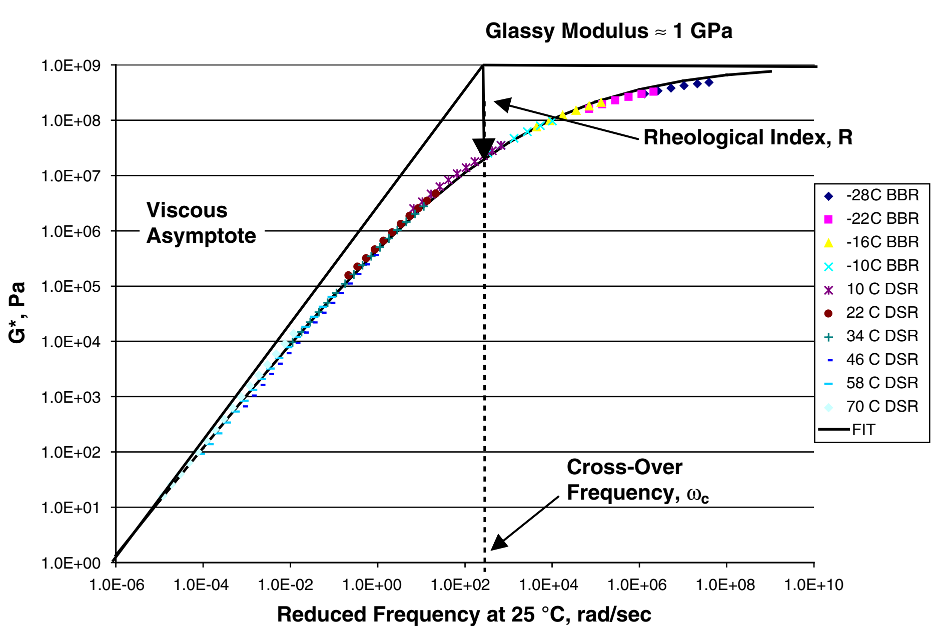


Figure 3-34. Christensen-Anderson Model master curve.

Table 3-28. Christensen-Anderson Model parameters.

Binder Source	Log_{10} Glassy Modulus, Pa	Rheological Parameter, R, log_{10} Pa				log_{10} Crossover Frequency, ω, rad/s				Defining Temperature, T_d, °C			
		Tank	SAFT	RTFOT	MGRF	Tank	SAFT	RTFOT	MGRF	Tank	SAFT	RTFOT	MGRF
AAC-1	8.6	1.21	1.62	1.50	1.55	3.42	2.41	2.68	2.42	-9.8	-0.7	-3.6	-1.5
AAD-2	9.2	1.92	2.17	2.16	2.14	4.11	3.18	3.28	3.31	-18.1	-15.6	-15.1	-14.7
AAF-1	8.6	1.39	1.63	1.65	1.85	2.39	1.56	1.41	1.24	-1.3	3.7	3.8	4.0
AAM-1	8.6	1.49	1.67	1.86	1.91	2.39	1.92	1.41	1.40	-3.1	0.3	2.7	3.7
ABL-1	8.7	1.55	1.65	1.77	1.75	2.97	2.62	2.15	2.27	-14.9	-13.1	-10.7	-11.0
ABM-2	8.7	0.90	0.91	0.90	1.02	2.97	2.74	2.69	2.59	-4.1	-4.1	-3.1	-2.9
Airblown	8.8	2.31	2.42	2.41	2.29	0.88	0.35	0.12	0.38	3.7	5.0	4.2	3.8
ALF	10.9	4.69	5.13	5.36	5.47	2.71	1.48	0.81	0.70	-15.2	-12.6	-12.8	-10.4
Citgoflex	9.8	2.95	3.06	3.33	3.18	1.82	1.35	0.63	0.92	-7.5	-6.2	-2.6	-2.1
Elvaloy	9.2	2.06	2.18	2.44	2.41	3.29	2.77	1.99	2.32	-13.4	-11.1	-9.5	-10.8
Novophalt	8.8	1.60	1.63	1.83	1.87	2.20	1.80	1.14	1.26	-9.7	-9.4	-5.1	-4.2

tioning to RTFOT conditioning for R, ω_c, and T_d, respectively. The general trends shown in these figures are reasonable. The rheological index increases with short-term conditioning, indicating that the master curve is becoming flatter. The crossover frequency decreases, indicating that the binder is becoming harder with short-term conditioning. Finally, the defining temperature increases on short-term aging, indicating greater temperature dependency.

If the changes in model parameters are the same for SAFT- and MGRF-conditioned binders compared to RTFOT-conditioned binders, the data should plot along the line of equality. As shown, there is significant scatter in the data because the master curve parameters are somewhat interrelated and depend on the quality of the data. Trend lines are shown for the SAFT and the MGRF data in Figures 3-35 and 3-36 to make it easier to interpret these plots. The trend lines for the MGRF data are much closer to the line of equality than those for the SAFT data.

The results of regression analyses for the change in the Christensen-Anderson Model parameters are summarized in Table 3-29. Details of this statistical analysis are presented in Appendix E (see the project webpage on the TRB website).

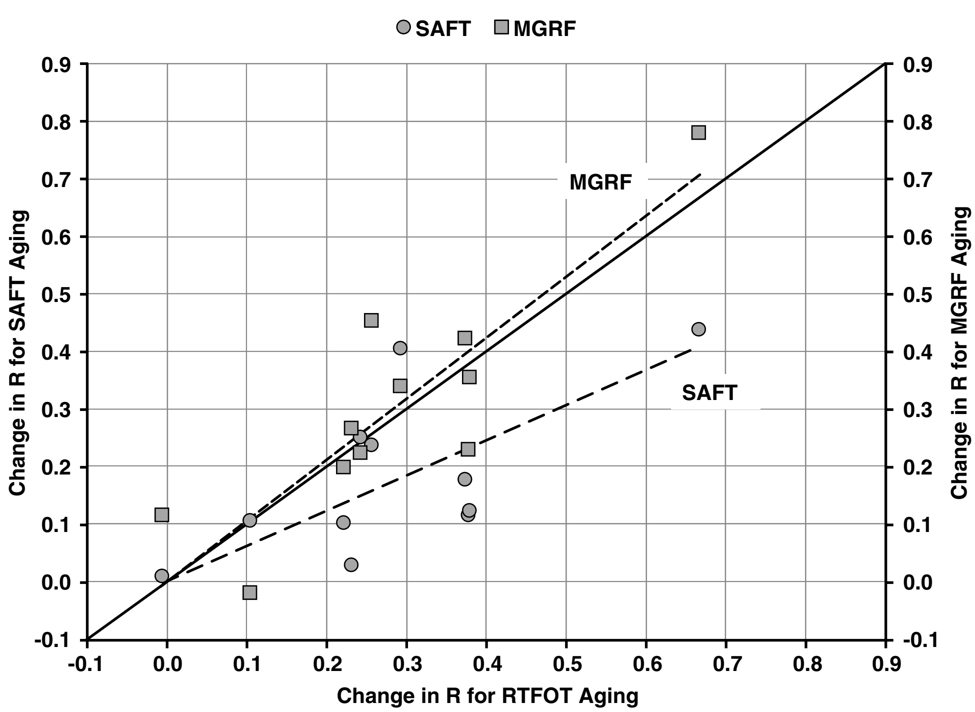


Figure 3-35. Comparison of change in rheological index for RTFOT, SAFT, and MGRF aging.

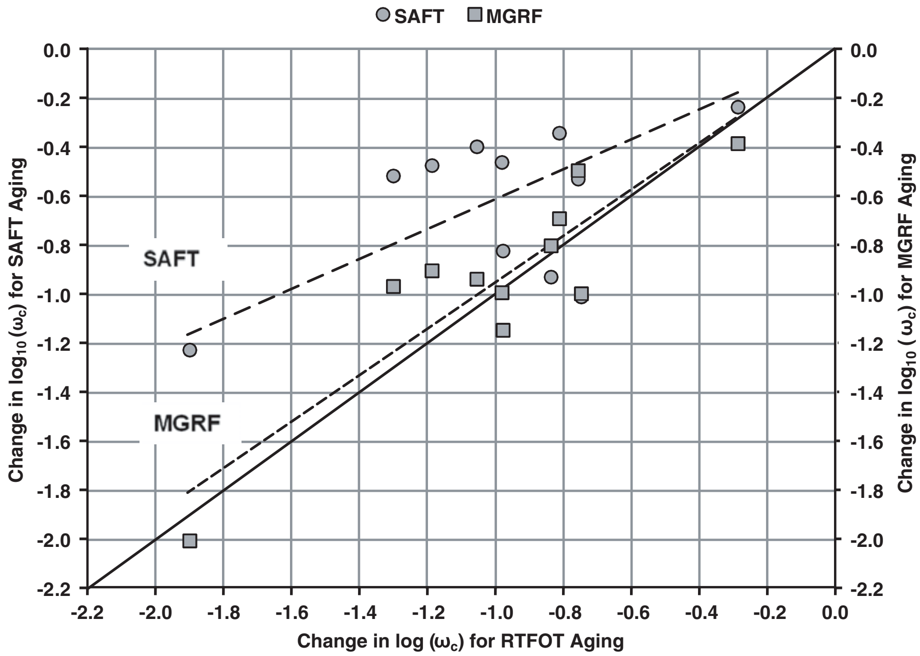


Figure 3-36. Comparison of change in crossover frequency for RTFOT, SAFT, and MGRF aging.

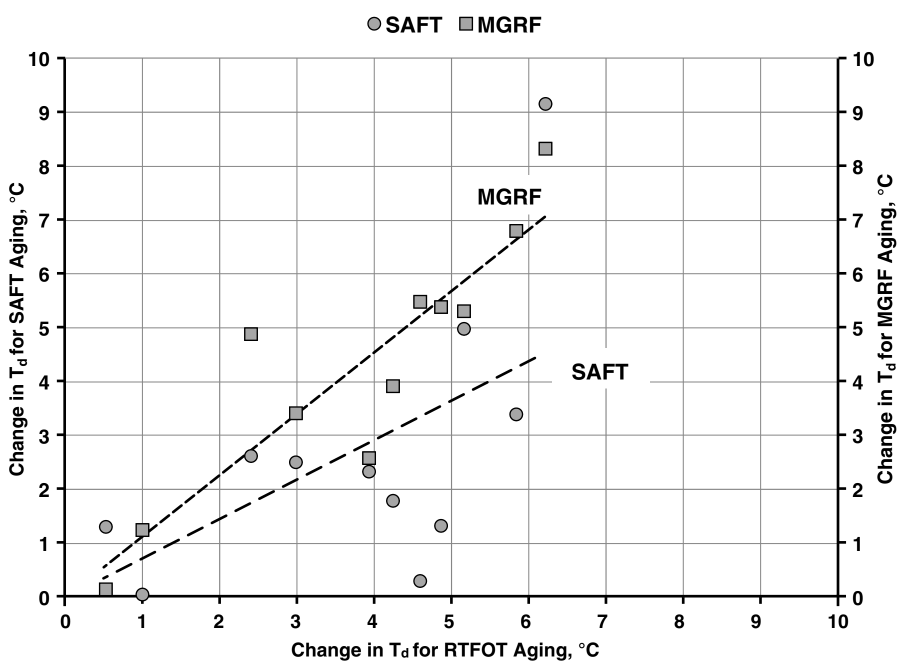


Figure 3-37. Comparison of change in defining temperature for RTFOT, SAFT, and MGRF aging.

Table 3-29. Regression analysis of change in Christensen-Anderson Model parameters.

Method	Neat		Modified		Hypothesis Test of Equality of Average Aging Index for Neat and Modified Binders			
	Average	Standard Deviation	Average	Standard Deviation	Pooled s	t	$t_{critical}$	Conclusion
R30	3.30	1.08	3.05	1.32	1.19	0.43	2.26	No difference
RTFOT	2.44	0.46	2.26	0.58	0.52	0.55	2.26	No difference
MGRF	2.44	0.47	2.30	0.35	0.42	0.54	2.26	No difference
SAFT	1.97	0.48	1.34	0.27	0.40	2.60	2.26	Neat > Modified

This analysis shows that there is a significant relationship in the change in the model parameters between the SAFT and RTFOT and the MGRF and RTFOT. The strength of the relationships, as indicated by the R^2 values, is better for the MGRF compared to the SAFT. Additionally, the slopes for the MGRF data are close to 1, and the 95 percent confidence intervals for the slope capture 1 for all three parameters. The slopes for the SAFT data range from 0.6 to 0.7, and only the 95 percent confidence interval for the slope of the change in T_d relationship captures 1. This analysis shows that MGRF aging produces changes in the Christensen-Anderson master curve parameters that are not significantly different from those for the RTFOT, while the SAFT aging produces different changes in the master curve parameters.

3.6.1.4 Aging Indices

Another technique for judging the conditioning that occurs in the SAFT and MGRF relative to the RTFOT is to calculate and compare aging indices for the procedures. This was done by calculating aging indices at a common temperature for each aging condition (but different for each binder) and at a series of moduli. This approach parallels the aging that occurs in an actual pavement—the change that occurs in stiffness at temperatures corresponding to different unaged moduli. The temperatures where G* for the unaged binders at 10 rad/s is equal to 1, 10, 100, 1,000, 10,000, and 100,000 kPa were calculated using the fitted Christensen-Anderson Model for each binder. These temperatures are shown in Table 3-30. The fitted Christensen-Anderson Model was then used to calculate the complex moduli for the binders for the three aging conditions at these temperatures and 10 rad/s. The moduli for the aged binders were then divided by the moduli for the unaged binders. The resulting aging indices are shown in Table 3-30.

Figure 3-38 compares aging indices from the SAFT and MGRF with those from the RTFOT for the binders used in the study. If the aging indices for the SAFT and MGRF are the same as the RTFOT, the data should plot along the line of equality. As shown, there is significant scatter in the data. Trend lines are shown for the SAFT and MGRF data in Figure 3-38. The trend line for the MGRF data falls nearly on the line of equality while the trend line for the SAFT data is much lower. The results of regression analyses for the aging indices are summarized in Table 3-31. Details of this statistical analysis are presented in Appendix E. This analysis shows that there are significant relationships between the aging indices from the SAFT and RTFOT, and the MGRF and RTFOT. The strength of the relationship, as indicated by the R^2 value, is approximately the same for the MGRF compared to the SAFT. However, the slope for the MGRF data is close to 1, and the 95 percent confidence interval for the slope captures 1. The slope for the SAFT data is 0.74 and the 95 percent confidence interval for the slope does not capture 1. The conclusion from this analysis is that the MGRF produces aging indices that are approximately the same as the RTFOT. Aging indices from the SAFT are less than those from the RTFOT.

3.6.2 Oven-Aged Mixture Experiment

In the oven-aged mixture experiment, binder properties back-calculated from mixture dynamic modulus test data were used to assess how well the binder aging procedures simulate the aging that occurs in mixtures during short-term oven conditioning. Dynamic modulus master curve tests were performed on samples prepared from unconditioned mixture and from mixture conditioned for 4 hours at 135°C as specified in AASHTO R30. From the mixture dynamic modulus master curves, the binder shear modulus master curves were estimated using the Hirsh Model (*6*). The back-calculated binder modulus data were then analyzed to assess how well the binder aging procedures simulate the aging that occurs in mixtures during short-term oven conditioning. The EVA-modified binder was not included in the comparisons due to the difficulties in testing this binder that were discussed earlier. The sections that follow describe the major findings from the oven-aged mixture experiment. The complete analysis is included in the verification study report in Appendix E (see the project webpage on the TRB website).

3.6.2.1 Back-Calculated Binder Properties

Binder properties for the mixtures were obtained by preparing mixtures, developing a dynamic modulus master curve for each mixture, and then back-calculating binder properties from the mixture modulus data. The dynamic modulus master curve testing was performed in accordance with AASHTO TP79, "Determining the Dynamic Modulus and Flow Number for Hot Mix Asphalt (HMA) Using the Asphalt Mixture Performance Tester (AMPT)," and PP61, "Developing Dynamic Modulus Master Curves for Hot Mix Asphalt (HMA) using the Asphalt Mixture Performance Tester (AMPT)," which were developed in NCHRP Project 9-29 (*34*). The testing was conducted at three temperatures and four frequencies as summarized in Table 3-32. The low and middle temperatures were set at 4°C and 20°C, respectively. The upper temperature was selected based on the binder grade: 34°C for the softer binders (AAC-1, AAD-2, ABL-1, ABM-2, ALF, and Elvaloy) and 40°C for AAF-1,

Table 3-30. Complex moduli and temperatures used to calculate aging indices.

Binder	G*, kPa	T, °C	Aging Index			Binder	G*, kPa	T, °C	Aging Index		
			SAFT	RTFOT	MGRF				SAFT	RTFOT	MGRF
AAC-1	1	56.9	2.35	2.11	2.70	Airblown	1	77.1	1.86	3.17	2.56
	10	43.0	2.56	2.21	2.92		10	59.5	1.76	2.81	2.40
	100	30.8	2.59	2.19	2.94		100	44.0	1.64	2.42	2.20
	1,000	19.7	2.35	2.00	2.66		1,000	29.8	1.49	2.00	1.95
	10,000	8.6	1.86	1.63	2.06		10,000	15.5	1.31	1.59	1.65
	100,000	-5.2	1.16	1.16	1.27		100,000	-2.5	1.09	1.20	1.30
AAD-2	1	57.1	2.51	3.08	2.87	ALF 64-40	1	70.1	1.52	2.51	2.58
	10	40.6	2.32	2.85	2.73		10	46.6	1.41	2.12	2.25
	100	26.2	2.08	2.55	2.51		100	27.2	1.32	1.78	1.96
	1,000	13.1	1.80	2.18	2.20		1,000	10.7	1.23	1.49	1.72
	10,000	0.5	1.49	1.77	1.82		10,000	-4.0	1.15	1.26	1.51
	100,000	-13.2	1.20	1.36	1.42		100,000	-17.6	1.03	1.03	1.18
AAF-1	1	64.5	2.29	2.99	3.18	Citgoflex	1	87.1	1.05	1.43	1.68
	10	50.4	2.32	2.97	2.90		10	64.4	1.04	1.39	1.73
	100	38.0	2.24	2.78	2.46		100	45.3	1.03	1.35	1.76
	1,000	26.5	2.01	2.39	1.93		1,000	28.6	1.02	1.30	1.76
	10,000	14.9	1.65	1.85	1.40		10,000	13.2	1.01	1.24	1.71
	100,000	0.3	1.14	1.19	0.92		100,000	-2.0	1.00	1.17	1.59
AAM-1	1	65.6	1.42	2.29	1.92	Elvaloy	1	66.0	1.21	3.10	3.05
	10	50.8	1.44	2.23	1.90		10	48.3	1.22	2.78	2.67
	100	37.7	1.42	2.05	1.79		100	32.9	1.22	2.40	2.25
	1,000	25.6	1.35	1.77	1.58		1,000	19.1	1.20	1.98	1.82
	10,000	13.5	1.22	1.41	1.30		10,000	5.7	1.16	1.57	1.43
	100,000	-1.9	1.03	0.98	0.92		100,000	-8.7	1.10	1.22	1.11
ABL-1	1	67.7	1.55	2.71	2.27	Novophalt	1	78.0	1.32	2.23	2.49
	10	50.3	1.53	2.63	2.23		10	59.6	1.30	2.20	2.46
	100	35.2	1.48	2.43	2.10		100	43.7	1.26	2.09	2.33
	1,000	21.4	1.39	2.12	1.88		1,000	29.3	1.22	1.88	2.07
	10,000	7.8	1.26	1.70	1.57		10,000	15.2	1.15	1.59	1.71
	100,000	-8.5	1.10	1.25	1.21		100,000	-1.1	1.06	1.24	1.29
ABM-2	1	61.5	1.70	1.69	1.96						
	10	48.3	1.68	1.73	1.96						
	100	36.8	1.65	1.76	1.89						
	1,000	26.6	1.58	1.74	1.74						
	10,000	16.8	1.44	1.64	1.48						
	100,000	5.4	1.21	1.36	1.14						

AAM-1, Air Blown, Citgoflex, Novophalt, and EVA. This testing protocol produces 10 dynamic modulus and phase angle measurements for each specimen. Three replicate specimens were tested in this project.

Dynamic modulus master curves were fitted to the measured data using the procedure in AASHTO PP61. Equations 7 and 8 present the form of the master curve that was fitted to the data. Equation 7 is the form of the dynamic modulus master curve recommended in the Mechanistic-Empirical Pavement Design Guide (MEPDG) for use in pavement structural design (*35*). This sigmoidal function describes the frequency dependency of the modulus at the reference temperature,

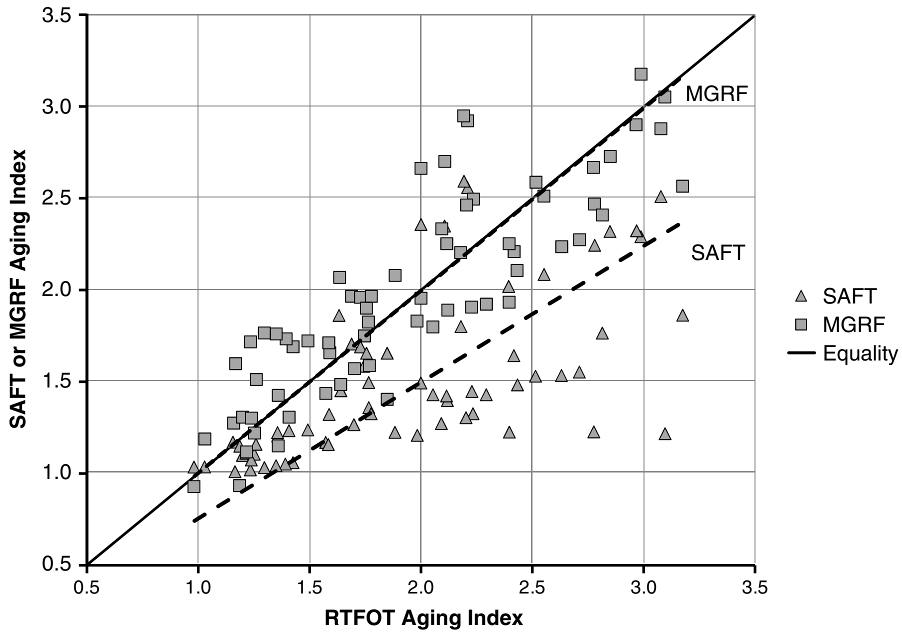


Figure 3-38. Comparison of aging indices for RTFOT, SAFT, and MGRF.

which for this testing was selected to be 22°C to coincide with that used in the binder testing. The shift factors describe the temperature dependency of the modulus. Equation 8 provides the form of the Arrhenius equation used for the shift factors.

$$\log(E^*) = \log E^*_{\min} + \frac{(\log E^*_{\max} - \log E^*_{\min})}{1 + e^{\beta + \gamma \omega_r}} \tag{7}$$

where

E^* = dynamic modulus
E^*_{max} = limiting maximum modulus
E^*_{min} = limiting minimum modulus
ω_r = frequency of loading at the reference temperature
β, γ = parameters describing the shape of the sigmoidal function

$$\log a(T) = \frac{\Delta E_a}{19.14714}\left(\frac{1}{T} - \frac{1}{T_r}\right) \tag{8}$$

where

$a(T)$ = shift factor as a function of temperature
T = temperature, Kelvin
T_r = reference temperature, Kelvin
ΔE_a = activation energy

The final form of the dynamic modulus master curve equation is obtained by substituting Equation 8 into Equation 7.

$$\log|E^*| = \log E^*_{\min} + \frac{(\log E^*_{\max} - \log E^*_{\min})}{1 + e^{\beta + \gamma\left\{\log\omega + \frac{\Delta E_a}{19.14714}\left[\left(\frac{1}{T}\right) - \left(\frac{1}{T_r}\right)\right]\right\}}} \tag{9}$$

The limiting maximum modulus of the mixture E^*_{min} was estimated using the Hirsch Model (*6*) and a limiting maximum binder shear modulus of 1 GPa (145,000 psi) (*32*). Equation 10 presents the Hirsch Model. As shown, the limiting maximum modulus of the mixture is a function of VMA and VFA. It ranges from about 3,100 to 3,800 ksi for typical ranges of VMA and VFA.

$$|E^*|_{mix} = P_c\left[4{,}200{,}000\left(1 - \frac{VMA}{100}\right) + 3|G^*|_{binder}\left(\frac{VFA\,xVMA}{10{,}000}\right)\right] + \frac{1 - P_c}{\left[\frac{\left(1 - \frac{VMA}{100}\right)}{4{,}200{,}000} + \frac{VMA}{3VFA|G^*|_{binder}}\right]} \tag{10}$$

Table 3-31. Regression analysis of aging indices.

Measure	SAFT	MGRF
Slope	0.64	1.00
Lower 95% CI	0.70	0.96
Upper 95% CI	0.79	1.03
R^2	0.93	0.96

Table 3-32. Temperatures and frequencies used in the dynamic modulus master curve testing.

Temperature, °C	Frequency, Hz
4.0	10, 1, and 0.1
20.0	10, 1, and 0.1
34.0 or 40.0	10, 1, 0.1, and 0.01

where

$$P_c = \frac{\left(20 + \frac{VFAx3|G^*|_{binder}}{VMA}\right)^{0.58}}{650 + \left(\frac{VFAx3|G^*|_{binder}}{VMA}\right)^{0.58}}$$

$|E^*|_{mix}$ = mixture dynamic modulus, psi
VMA = voids in mineral aggregates, %
VFA = voids filled with asphalt, %
$|G^*|_{binder}$ = shear complex modulus of binder, psi

Table 3-33 summarizes the dynamic modulus master curve parameters obtained by numerical optimization of Equation 9 after substitution of the limiting maximum modulus obtained from Equation 10. Figure 3-39 shows a typical comparison of unconditioned and conditioned mixture master curves and temperature shift factors as well as the measured data for ABL-1. Similar plots for all of the 12 binders are included in Appendix E. The error bars shown are 95 percent confidence

Table 3-33. Mixture modulus master curve parameters.

Parameter	Binder	Unconditioned	Conditioned	Binder	Unconditioned	Conditioned
Air Voids, %	AAC-1	4.6	4.4	Airblown	4.3	4.0
VMA		16.4	16.0		16.4	15.9
VFA		72.3	72.7		73.6	74.6
E^*_{max}, ksi		3342.2	3366.2		3349.1	3381.6
E^*_{min}, ksi		7.5	4.1		3.6	2.2
β		-0.1910	-0.5708		-1.1127	-1.3366
γ		0.6503	0.4924		0.4485	0.3721
E_a, kJ/mole		213344	211294		217602	208852
Air Voids, %	AAD-2	4.2	4.2	ALF	4.3	4.3
VMA		16.0	15.5		16.4	15.7
VFA		73.7	73.1		73.6	72.7
E^*_{max}, ksi		3371.5	3396.0		3363.9	3382.8
E^*_{min}, ksi		5.1	5.3		21.7	21.1
β		-0.0012	-0.3389		0.5936	0.2370
γ		0.5674	0.4767		0.5903	0.5194
E_a, kJ/mole		180577	182085		169894	173512
Air Voids, %	AAF-1	3.9	4.0	Citgoflex	3.7	3.9
VMA		15.6	15.6		15.5	15.5
VFA		75.2	74.2		76.1	76.1
E^*_{max}, ksi		3401.2	3396.1		3411.3	3417.9
E^*_{min}, ksi		5.3	2.6		18.0	40.0822
β		-0.8889	-1.3679		-0.6852	-0.8515
γ		0.5905	0.5199		0.5239	0.6811
E_a, kJ/mole		212784	211229		187462	194955
Air Voids, %	AAM-1	4.4	4.2	Elvaloy	4.2	4.0
VMA		16.4	16.1		15.9	15.5
VFA		73.1	74.1		73.6	74.0
E^*_{max}, ksi		3346.5	3368.1		3376.5	3400.7
E^*_{min}, ksi		3.6	2.4		12.5	7.6
β		-0.6677	-0.9797		-0.1281	-0.6076
γ		0.5018	0.4312		0.5684	0.4448
E_a, kJ/mole		225645	234911		188958	193486
Air Voids, %	ABL-1	4.2	4.0	EVA	4.0	4.0
VMA		15.8	15.4		15.9	15.6
VFA		73.3	74.2		75.0	74.1
E^*_{max}, ksi		3380.4	3407.3		3383.7	3395.6
E^*_{min}, ksi		6.2	8.1		29.0	7.1
β		-0.4518	-0.6041		-0.7557	-1.1755
γ		0.5407	0.5080		0.5999	0.4138
E_a, kJ/mole		187271	188768		190346	191384
Air Voids, %	ABM-2	4.1	4.2	Novophalt	3.8	3.8
VMA		15.9	15.8		15.3	15.5
VFA		73.9	73.0		75.2	75.4
E^*_{max}, ksi		3378.0	3378.8		3385.7	3409.8
E^*_{min}, ksi		7.4	8.7		9.0	13.8
β		-0.7397	-0.9640		-0.9371	-1.1633
γ		0.8694	0.8167		0.5481	0.5569
E_a, kJ/mole		195125	200619		183832	190785

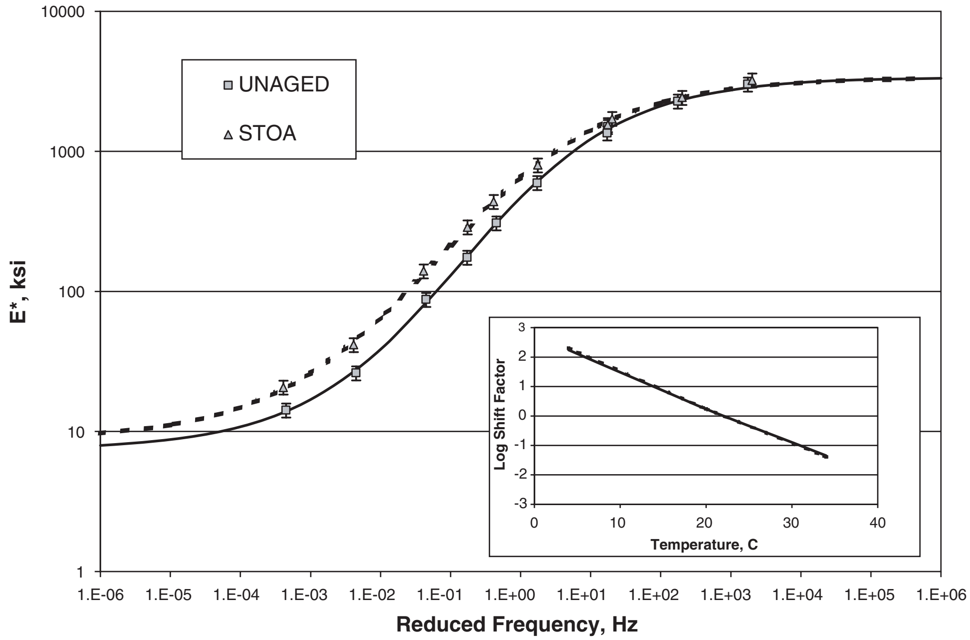


Figure 3-39. Mixture dynamic modulus master curves for ABL-1.

intervals based on the pooled standard deviation of the dynamic modulus data for all binders. As shown in this figure, short-term oven conditioning in accordance with AASHTO R30 results in significant stiffening of the mixture.

The mixture dynamic modulus is highly sensitive to the stiffness of the binder in the mixture. The shear modulus of the binder can be estimated from the mixture dynamic modulus using the Hirsch Model (*6*) previously presented as Equation 10. Knowing the dynamic modulus of the mixture and the VMA and VFA of the test specimens, Equation 10 can be solved for the binder shear modulus. This is best done by trial and error. Figure 3-40 presents an example of the back-calculated binder shear moduli for the testing conditions used in the dynamic modulus testing. Back-calculated binder shear modulus data for all of the binders is included in Appendix E. Figure 3-40 presents data for both the unconditioned mixture and the mixture conditioned for 4 hours at 135°C in accordance with AASHTO R30. The effect of the short-term oven conditioning is clearly evident. It results in significant stiffening, particularly at the higher temperatures.

Master curves were developed for the back-calculated binder modulus data by fitting the Christensen-Anderson Model to the data as described previously for the binder test data. The glassy modulus from the binder testing was used in fitting the back-

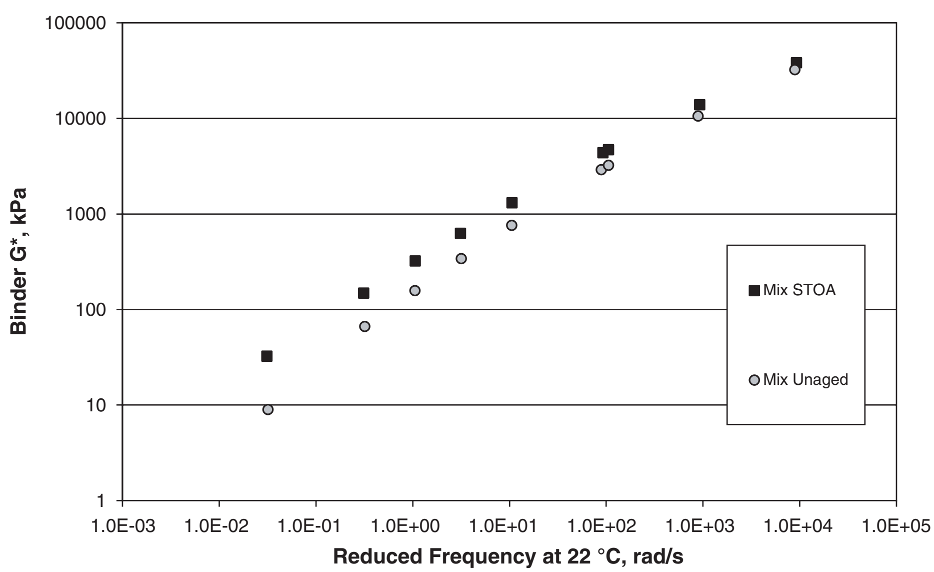


Figure 3-40. Back-calculated binder modulus master curves for ABL-1.

Table 3-34. Christensen-Anderson Model parameters for back-calculated binder moduli.

Binder Source	Log_{10} Glassy Modulus, Pa	Rheological Parameter, R, Log_{10} Pa		Log_{10} Crossover Frequency, ω, rad/s		Defining Temperature, T_d, °C	
		Unaged	R30	Unaged	R30	Unaged	R30
AAC-1	8.6	1.16	1.85	3.69	2.57	-6.4	-5.5
AAD-2	9.2	1.90	2.47	4.33	2.98	-14.4	-15.6
AAF-1	8.6	1.32	1.46	2.64	1.85	-3.5	-4.2
AAM-1	8.6	1.83	2.07	2.51	1.66	0.4	2.3
ABL-1	8.7	1.72	2.01	2.92	2.07	-13.3	-12.4
ABM-2	8.7	0.59	0.68	3.43	3.09	-9.5	-7.3
Airblown	8.8	2.27	2.94	1.14	-0.57	-2.2	-3.1
ALF	10.9	4.49	5.18	3.18	1.32	-18.4	-15.8
Citgoflex	9.8	3.82	3.33	0.15	0.29	-4.2	2.0
Elvaloy	9.2	2.32	3.03	3.02	1.23	-12.1	-10.4
Novophalt	8.8	1.73	2.10	2.03	0.73	-13.5	-4.5

calculated master curves. The resulting Christensen-Anderson master curve parameters are summarized in Table 3-34 for the 11 binders included in the binder evaluation.

3.6.2.2 Comparison of Master Curve Parameters

The purpose of the oven-aged mixture experiment was to compare the degree of aging from the short-term binder aging procedures with that from mixtures conditioned at 135°C for 4 hours in accordance with AASHTO R30. The first approach for comparing the short-term binder and mixture aging procedures was to compare the Christensen-Anderson master curve parameters obtained from the short-term binder aging procedures with those obtained from the back-calculated binder moduli from the mixture dynamic modulus testing. The parameters include the rheological index, R, the crossover frequency, ω_c, and the defining temperature, T_d. Recall, the physical significance of the Christensen-Anderson master curve parameters are as follows:

- The rheological index, R, is the difference between the log of the glassy modulus and the log of the dynamic modulus at the crossover frequency. It is an indicator of the rheological type.
- The crossover frequency, ω_c, is the frequency where the phase angle is close to 45 degrees and is an indicator of the hardness of the binder.
- The defining temperature, T_d, is an indicator of the glass transition of the binder.

Figures 3-41 through 3-43 compare the Christensen-Anderson master curve parameters back-calculated from the unaged mixture dynamic modulus tests with those measured for the tank binder. These figures show that there is reasonable agreement in the master curve parameters for unaged conditions. Table 3-35 summarizes the results of regression analyses for each of the parameters. Details of this statistical analysis are presented in Appendix E (see the project webpage on the TRB website). The 95 percent confidence intervals for the slope of the best-fit regression line for each of the three parameters captures 1, the line of equality, indicating that the back-calculated master curves vary in a similar manner as the tank master curves for the range of binders tested.

Figures 3-45 through 3-46 compare changes in the master curve parameters for AASHTO R30 mixture aging with RTFOT and SAFT aging for the binders. Comparisons were not made for MGRF aging because analysis of the binder aging data showed RTFOT and MGRF aging were essentially the same. Figures 3-44 and 3-46 show that there is no relationship for the change in the rheological index and the change in the defining temperature between short-term mixture and binder aging. The trend lines in Figure 3-45 show that there is a weak relationship for the crossover frequency between the short-term mixture and binder aging, with the mixture aging producing somewhat harder binders.

3.6.2.3 Aging Indices

The second approach for comparing the short-term mixture and binder aging procedures was to compare aging indices computed from the fitted binder master curves. Aging indices were computed from the back-calculated binder modulus data using the method previously described for the binder testing. Table 3-36 presents aging indices computed in this manner for each of the binders for various unaged binder modulus values ranging from 1 to 100,000 kPa. The 1 kPa values are based on unaged binder modulus values that were below the range measured in the dynamic modulus test and

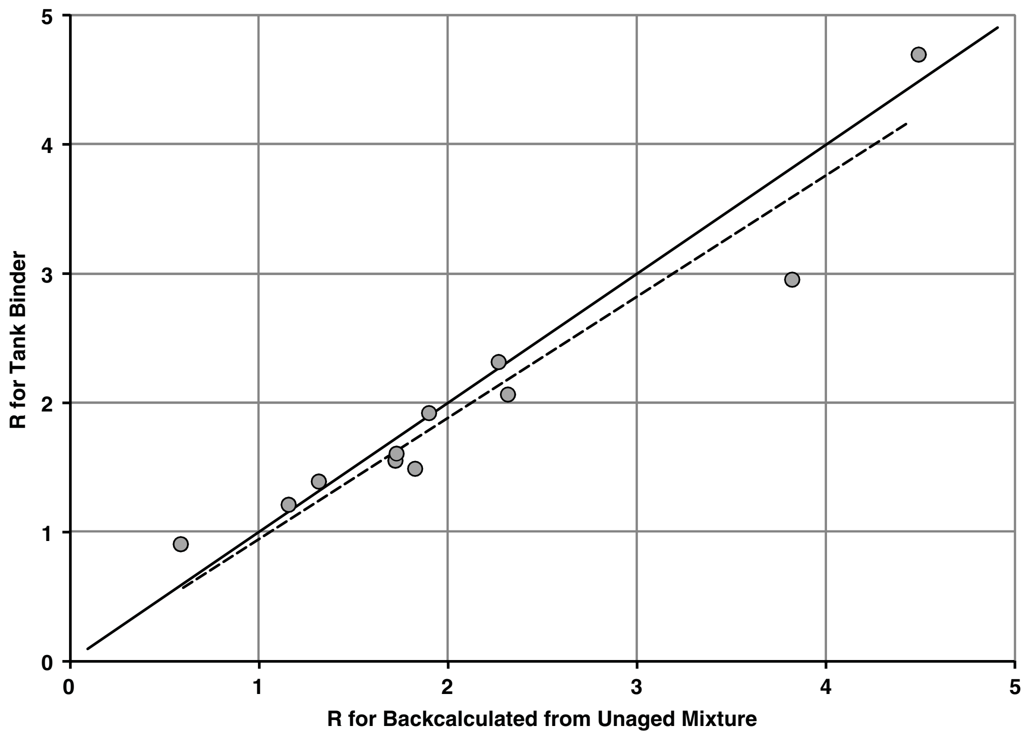


Figure 3-41. Comparison of rheological index from unaged mixture and tank binder tests.

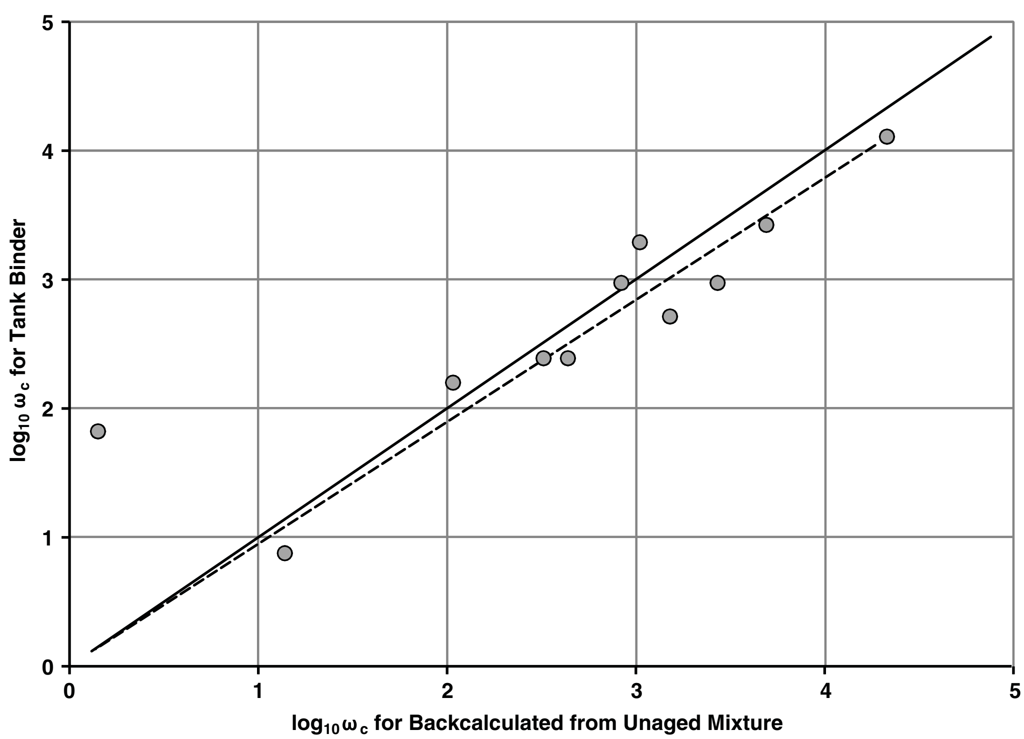


Figure 3-42. Comparison of crossover frequency from unaged mixture and tank binder tests.

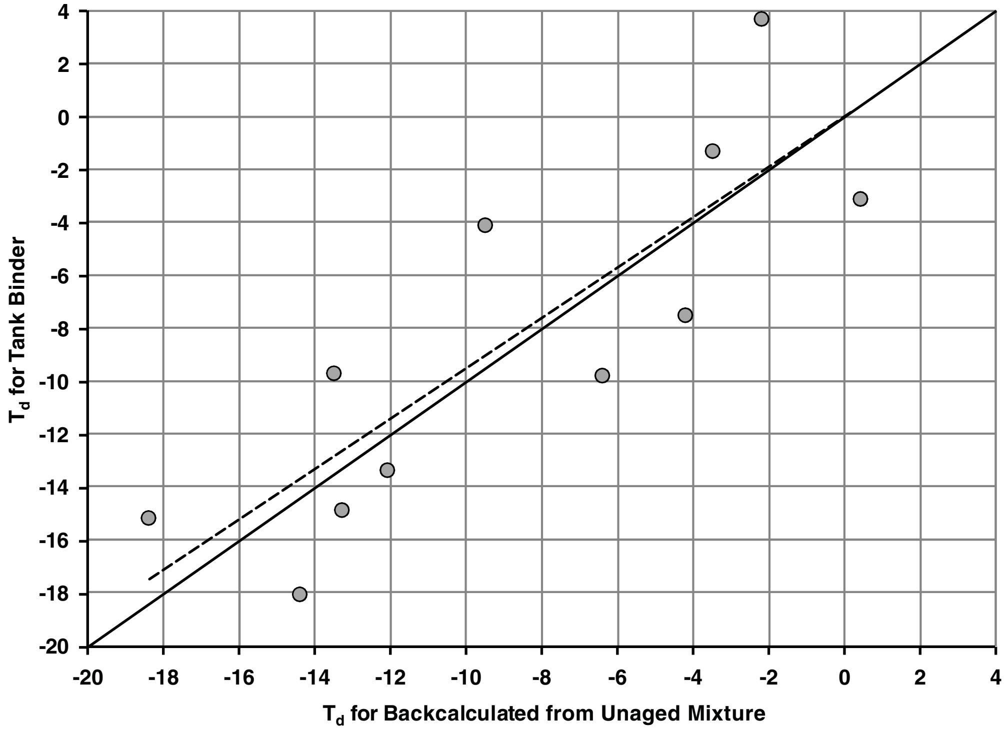


Figure 3-43. Comparison of defining temperature from mixture and binder tests.

the 100,000 kPa values were above the range measured in the dynamic modulus test. The remaining values were within the range of the measured data.

Figure 3-47 compares aging indices from the mixture testing with those from the RTFOT, SAFT, and MGRF for an unaged binder modulus of 10 kPa, the lowest stiffness included in the mixture testing conditions. Figure 3-47 shows a reasonable correlation between the AASHTO R30 mixture aging and the RTFOT and MGRF. The correlation for the SAFT is much poorer. The regression analysis is summarized in Table 3-37. Details of this statistical analysis are presented in Appendix E (see the project webpage on the TRB website). The slope and intercept from the RTFOT and the MGRF regression models are both significant at the 99 percent level, while those for the SAFT are not significant at the 95 percent level. The slopes of the relationships indicate that the aging index from the short-term binder aging tests is less than that obtained from short-term aging of mixtures in accordance with AASHTO R30.

Rankings for the aging indices can be used to compare the short-term binder aging procedures to AASHTO R30. Table 3-38 summarizes the rankings for each test, with the rank of 1 given to the binder with the highest aging index. Table 3-38 also presents the Spearman's rank correlation coefficient and its significance level (p-value) for the three short-term binder aging procedures. Higher values of the Spearman's rank correlation coefficient indicate greater similarity in the rankings. The p-values for the Spearman's rank correlation coefficient indicate the level of statistical significance for the rankings. The ranking analysis in Table 3-38 shows that the RTFOT provides the closest rankings compared to AASHTO R30 with a Spearman's rank correlation coefficient of 0.91 and a significance level exceeding 99.9 percent. The MGRF also provides similar rankings to AASHTO R30 with a Spearman's rank correlation coefficient of 0.80 and a significance level of 99.7 percent. The SAFT provides rankings having the greatest difference compared to AASHTO R30.

Table 3-35. Regression analysis of unaged mixture and tank, Christensen-Anderson Model parameters.

Parameter	Measure	Value
R	Slope	0.94
	Lower 95 % CI	0.85
	Upper 95 % CI	1.02
	R^2	0.88
ω_c	Slope	0.95
	Lower 95 % CI	0.81
	Upper 95 % CI	1.08
	R^2	0.86
T_d	Slope	0.95
	Lower 95 % CI	0.71
	Upper 95 % CI	1.19
	R^2	0.78

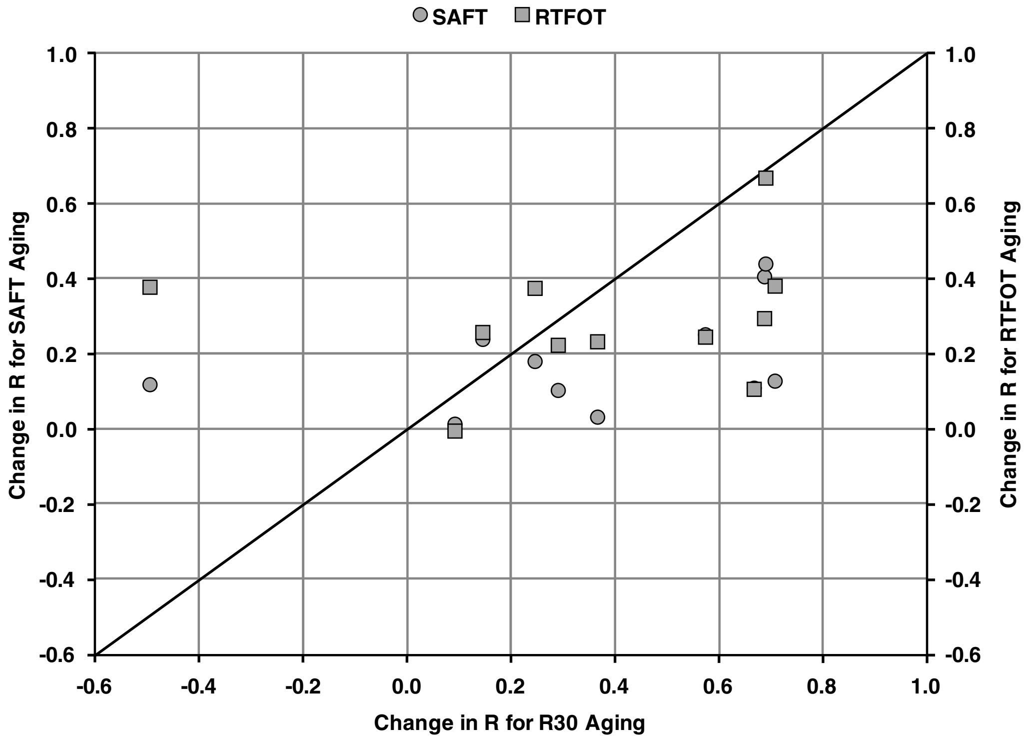


Figure 3-44. Comparison of change in rheological index for short-term mixture and binder aging.

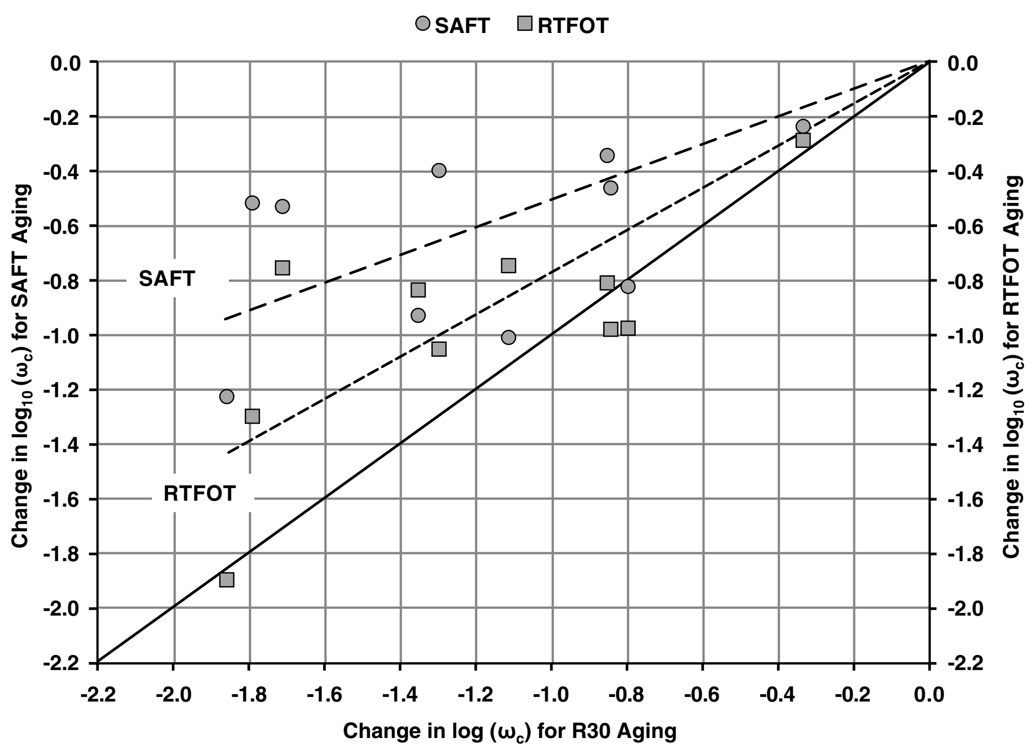


Figure 3-45. Comparison of change in crossover frequency for short-term mixture and binder aging.

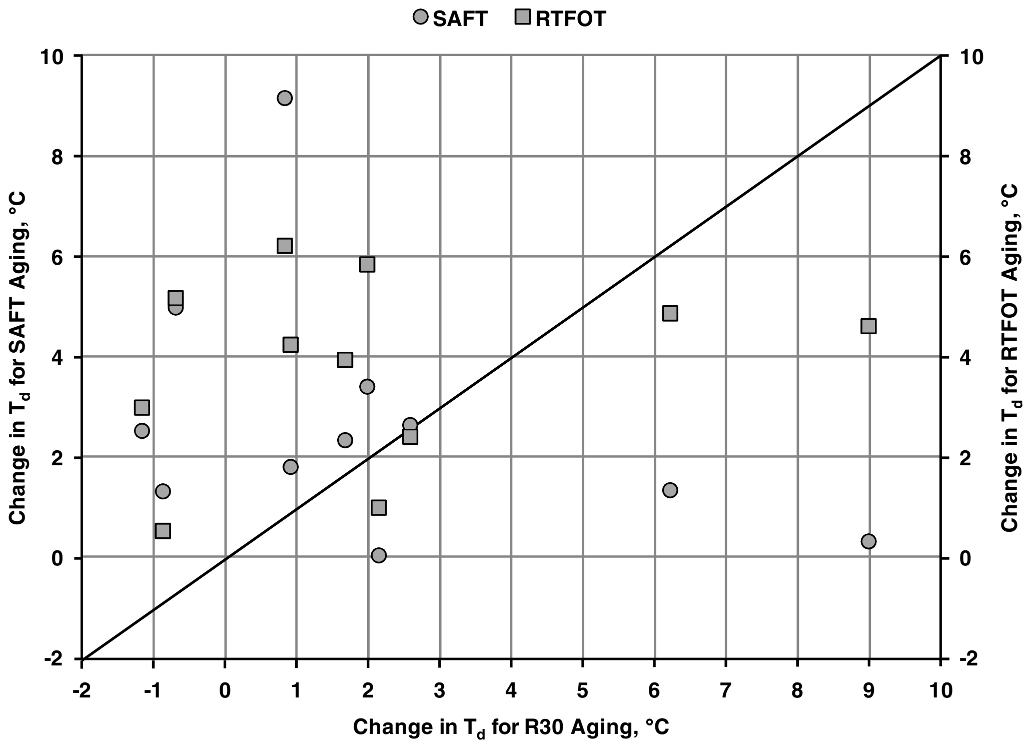


Figure 3-46. Comparison of change in defining temperature for short-term mixture and binder aging.

The Spearman's rank correlation coefficient for the SAFT rankings is only 0.60 and the correlation is not significant at the 95 percent level.

3.7 Short-Term Aging of Modified Binders

One of the purported issues with the RTFOT is that the aging of modified binders is less because the higher viscosity modified binders do not maintain a thin film during the test. Table 3-39 summarizes average aging indices for the neat and modified binders for 10 kPa unaged binder stiffness. Data are presented for four aging procedures (AASHTO R30 mixture aging and binder aging using the RTFOT, MGRF, and SAFT). In computing the averages and standard deviations given in Table 3-38, the data for the EVA binder were eliminated due to the testing difficulties caused by phase separation that were discussed previously.

The average aging indices for neat and modified binders are compared in Figure 3-48 for the four short-term aging procedures. The error bars shown in Figure 3-48 are 95 percent confidence intervals for the average. These comparisons show that there is little difference in the average aging index for the neat and modified binders included in NCHRP Project 9-36.

Table 3-39 also summarizes the results of hypothesis testing for the average of the mean 10-kPa aging index between neat and modified binders for the four short-term aging procedures. This testing shows that the average aging index is the same for neat and modified binders for short-term mixture aging in accordance with AASHTO R30 and short-term binder aging in the RTFOT and the MGRF. For the SAFT, the hypothesis testing shows that the average aging index for the neat binder is greater than that for the modified binder.

Table 3-36. Aging indices for AASHTO R30 conditioning based on back-calculated binder modulus values.

	Aging Indices from AASHTO R30 for Various Unaged Binder Modulus Values					
Binder	1 kPa	10 kPa	100 kPa	1,000 kPa	10,000 kPa	100,000 kPa
AAC-1	4.78	3.50	2.34	1.43	0.84	0.55
AAD-2	6.46	4.53	3.01	1.91	1.19	0.77
AAF-1	5.26	4.49	3.61	2.68	1.83	1.17
AAM-1	2.95	2.62	2.23	1.80	1.40	1.00
ABL-1	3.43	2.89	2.32	1.77	1.30	0.97
ABM-2	1.70	1.79	1.87	1.89	1.73	1.32
Airblown	6.66	4.34	2.71	1.65	1.02	0.72
ALF	3.33	2.74	2.24	1.82	1.48	1.19
Citgoflex	0.85	1.20	1.72	2.40	3.09	2.79
Elvaloy	6.13	4.34	2.94	1.93	1.26	0.86
Novophalt	2.61	2.66	2.58	2.34	1.94	1.43

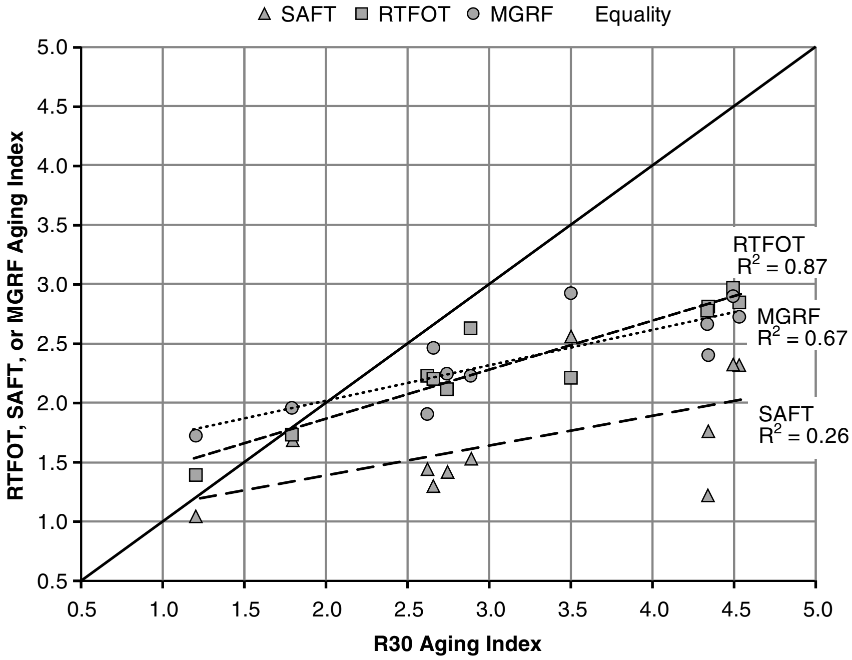


Figure 3-47. Comparison of 10 kPa aging indices for mixture and binder testing.

Table 3-37. Regression analysis of aging indices from AASHTO R30, RTFOT, SAFT, and MGRF.

Model	Measure	Value
RTFOT vs R30	R^2	0.87
	Slope	0.41
	p-value for slope	0.0002
	Intercept	1.04
	p-value for intercept	>0.0001
SAFT vs R30	R^2	0.26
	Slope	0.25
	p-value for slope	0.0639
	Intercept	0.88
	p-value for intercept	0.0563
MGRF vs R30	R^2	0.67
	Slope	0.30
	p-value for slope	0.0012
	Intercept	1.42
	p-value for intercept	0.0010

Table 3-38. Comparison of ranking of binder aging indices.

Binder	Rank			
	R30	RTFOT	SAFT	MGRF
AAC-1	5	7	1	1
AAD-2	1	2	3	3
AAF-1	2	1	2	2
AAM-1	9	6	7	10
ABL-1	6	5	6	8
ABM-2	10	10	5	9
Airblown	3	3	4	6
ALF	7	9	8	7
Citgoflex	11	11	11	11
Elvaloy	4	4	10	4
Novophalt	8	8	9	5
Spearman Rank Correlation Coefficient	ρ	0.91	0.60	0.80
	p-value	0.0001	0.0510	0.0031

Table 3-39. Average 10 kPa aging indices for neat and modified binders.

Method	Neat		Modified		Hypothesis Test of Equality of Average Aging Index for Neat and Modified Binders			
	Average	Standard Deviation	Average	Standard Deviation	Pooled s	t	$t_{critical}$	Conclusion
R30	3.30	1.08	3.05	1.32	1.19	0.43	2.26	No difference
RTFOT	2.44	0.46	2.26	0.58	0.52	0.55	2.26	No difference
MGRF	2.44	0.47	2.30	0.35	0.42	0.54	2.26	No difference
SAFT	1.97	0.48	1.34	0.27	0.40	2.60	2.26	Neat > Modified

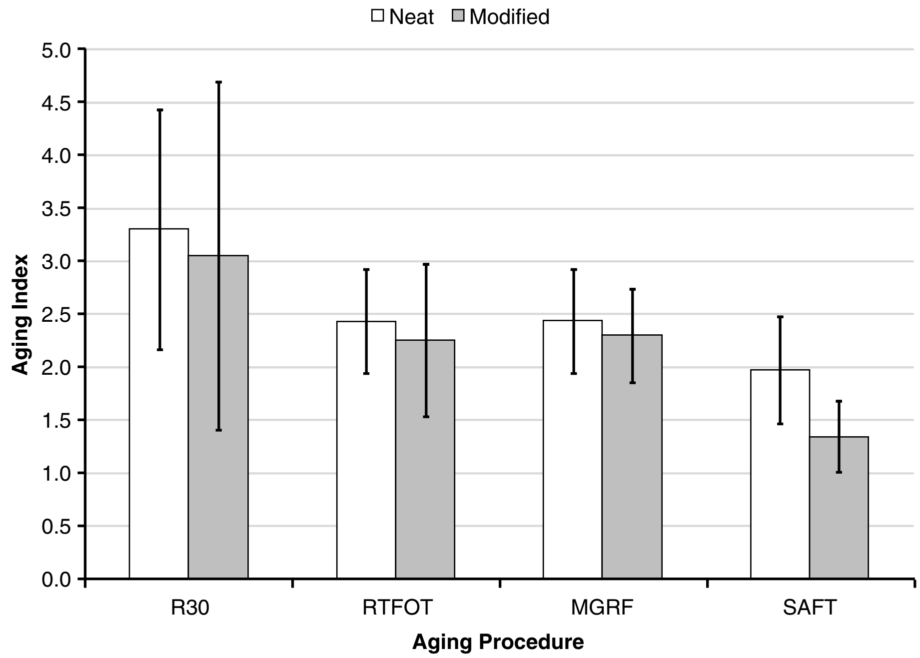


Figure 3-48. Comparison of average 10-kPa aging indices for neat and modified binders.

CHAPTER 4

Conclusions and Recommendations

4.1 Summary of Findings

The general approach adopted for NCHRP Project 9-36 was to improve existing binder aging technologies rather than develop a completely new procedure. The project started with a review of existing binder aging procedures to identify viable candidate methods for possible improvement. Two viable methods were identified—the SAFT and MGRF. From the review, the study team determined that both the SAFT and MGRF are relatively inexpensive, easy to perform, applicable to both neat and modified binders, and—based on available literature—can reasonably reproduce the level of aging that occurs in the RTFOT. However, it was not clear from the review if either test could be extended to long-term aging. Therefore, a selection study was conducted to choose one of these methods for further development. The selection study investigated whether at a temperature of 100°C either test can adequately mix air with stiff binders to produce a level of aging similar to that obtained in the PAV. From this study, the SAFT was selected for further development. The additional development for the SAFT included a VCS study to design an improved system for quantifying the volatility of binders tested in the SAFT and an optimization study to determine operating parameters for the SAFT so that it would reproduce the level of aging obtained for neat binders with the RTFOT. The last study conducted in NCHRP 9-36 was a verification study. In this study, the properties of binders aged in both the SAFT and MGRF were compared to properties of binders aged in the RTFOT and the properties of binders from mixtures that were short-term oven-aged in accordance with the performance testing procedure in AASHTO R30. The verification study served as the basis for the final recommendations for short-term aging that are the primary product of NCHRP Project 9-36. The major findings from the various studies conducted in NCHRP Project 9-36 are summarized in the following sections.

4.1.1 Selection Study

At 100°C, the maximum temperature considered viable for a long-term aging test, the MGRF does not generate a moving film and a number of attempts to modify the apparatus by adding scrapers and balls or rollers were not successful. A long-term aging test in the MGRF would require considerably in excess of 2 days to complete in order to simulate the aging that occurs in the PAV.

Adequate mixing of air was also a serious problem at 100°C for the SAFT, and a number of impeller designs were evaluated in order to improve the mixing efficiency and, consequently, the degree of aging. These designs improved the mixing and the rate of aging so that aging consistent with the PAV could be obtained after 40 hours. However, the degree of aging relative to the PAV was found to be dependent on the binder. Unexpectedly, the stiffer polymer-modified binders aged more relative to the PAV than did the neat asphalt binders.

4.1.2 VCS Study

The SAFT included a volatile collection system (VCS) to collect volatiles from the binder during short-term aging. The reported small mass of volatiles collected with the original VCS, one-tenth of the mass lost with the RTFOT procedure, prompted a review of the VCS supplied with the prototype version of the SAFT. This review confirmed that only a small amount of volatiles was collected by the air-cooled condenser. Additionally, the study team found that condensation of volatiles on the inside of the lid of the SAFT vessel, saturation of the air passing through the SAFT with volatiles, and suppression of volatilization caused by air pressure within the SAFT could not explain the relatively small amount of volatiles collected in the original prototype VCS. It was shown that the design of the original VCS was inadequate, and that volatiles were passing through the VCS. After considerable trial and error, a VCS based upon adsorbents commonly used for chromatographic studies was found to be effective for collecting the volatiles produced during the SAFT procedure. This system includes hydrocarbon and moisture traps on the inlet side of the SAFT vessel and a 3.9-in.-long (100-mm-long) resin bed and molecular sieve filters to collect hydrocarbons and water, respectively, on the outlet side. It also was found that the majority of the volatiles collected are water, not hydrocarbons.

4.1.3 SAFT Optimization Study

An unexpected finding from early work with the commercial version of the SAFT was that the degree of aging in the commercial SAFT was significantly less than that obtained with the prototype SAFT. The difference was attributed to rapid aging in the prototype at the vessel wall that was in direct contact with the heating mantle. The commercial SAFT uses an oven to heat the vessel and limits the oven temperature to 176°C. This finding led to an extensive optimization study to establish operating parameters appropriate for the commercial SAFT. Based upon a Plackett-Burman statistical experiment design that included impeller speed, airflow rate, and aging time as variables, the study team found that the following operating parameters provided a residue that best approximated the rheological properties of the RTFOT residue for PG 58-XX binders:

- 163°C aging temperature,
- 2,000 mL/min airflow,
- 1,000 rpm impeller speed,
- 50-minute aging time,
- 250-g sample mass, and
- Vacuum degassing per AASHTO R28 after short-term aging in the SAFT.

The degassing step was added because the researchers found that the bubbles entrapped in the SAFT residue significantly affect its measured rheological properties. Although the degassing significantly affected the properties of the binder, the study team deemed it necessary because of the error otherwise caused by the presence of air bubbles.

4.1.4 Verification Study

The study to verify the equivalency of the SAFT and MGRF relative to the RTFOT was conducted in the following two parts:

- RTFOT verification experiment where rheological properties of binders aged in the SAFT and MGRF were compared to rheological properties of binders aged in the RTFOT.
- Oven-aged mixtures experiment where rheological properties of short-term aged binders were compared to properties back-calculated from oven-aged mixtures.

4.1.4.1 RTFOT Verification Experiment

The RTFOT verification experiment included comparisons of high-temperature, continuous-grade Christensen-Anderson master curve parameters and aging indices for SAFT and MGRF residue to those for RTFOT residue. The comparisons of the continuous high-temperature grade after short-term aging showed the MGRF results in the same high-temperature grade as the RTFOT over the range of binders tested. The maximum difference in the short-term-aged, high-temperature continuous grade between MGRF- and RTFOT-aged binders was 1.8°C, and the difference was not a function of the high-temperature grade of the binder over the range from 56°C to 86°C. The short-term aged, high-temperature continuous grade for SAFT-conditioned binders, on the other hand, was similar to that for RTFOT-aged binders between 58°C and 60°C, but was as much as 6°C (one grade level) lower for higher stiffness binders. Variability of the short-term-aged, high-temperature continuous grade for the MGRF was somewhat higher than for the RTFOT and SAFT.

The continuous high-temperature grade analysis only investigated the effect of the aging procedures on high-temperature rheology. The master curve analysis compared the rheology of the binders over their entire stiffness range. This analysis showed the MGRF aging produced similar changes in the Christensen-Anderson master curve parameters as RTFOT aging, while the changes for SAFT aging were different. This indicates that the MGRF aging produces similar changes in the structure of the binder as RTFOT aging, but the SAFT results in different structural changes.

The aging index, the ratio of short-term aged G^* to unaged G^* for a particular test temperature and frequency of loading, is another way to evaluate the aging procedures over a range of binder stiffnesses. This analysis showed the aging indices to be similar for the MGRF and RTFOT, but those for the SAFT were lower.

4.1.4.2 Oven-Aged Mixture Experiment

In the oven-aged mixture experiment, properties of the short-term-aged binders back-calculated from dynamic modulus tests on oven-aged mixtures were used to compare the degree of aging that occurs in the RTFOT, SAFT, and MGRF to that occurring in AASHTO R30. The comparisons were based on changes in Christensen-Anderson master curve model parameters and aging indices. This experiment produced the following findings:

1. The binder aging that occurs when a mixture is short-term conditioned in a forced-draft oven for 4 hours at 135°C per AASHTO R30 generally exceeds the aging that occurs in the short-term binder aging procedures. Hardening measured by the change in the Christensen-Anderson crossover frequency was greater for AASHTO R30 compared to the RTFOT, SAFT, and MGRF. Binder aging indices also were greater for AASHTO R30 compared to the three short-term binder aging procedures.
2. Based on an analysis of aging index rankings, short-term binder aging in the RTFOT and MGRF provided similar rankings as short-term mixture aging in accordance with AASHTO R30. The ranking of binders aged in the SAFT did not correlate well with the AASHTO R30 rankings.

An interesting and unexpected finding from both of the experiments in the verification study was that for the binders tested, the average aging of the neat binders was approximately the same as that for the modified binders for AASHTO R30, RTFOT, and MGRF conditioning. This finding is in contrast with other studies that have reported less aging in the RTFOT for modified binders. For the SAFT, the average aging of the neat binders was greater than that of the modified binders.

4.2 Conclusions

The primary objective of NCHRP 9-36 was to identify and verify a test procedure that can be used as a replacement for the RTFOT. Based on the findings summarized above, the MGRF is considered an acceptable replacement for the RTFOT. For neat binders, MGRF and RTFOT conditioning produced similar rheological properties. MGRF and RTFOT conditioning also produced similar rheological properties for typical polymer-modified binders. The ranking of the aging susceptibility of binders for both the MGRF and RTFOT correlated well with the ranking of aging susceptibility from mixtures that were short-term oven-aged for 4 hours at 135°C in accordance with AASHTO R30. Although rheological properties were the same, mass change in the MGRF is less than in the RTFOT, averaging approximately 40 percent of the RTFOT mass change for the binders tested in this study.

The SAFT, on the other hand, is not an acceptable replacement for the RTFOT for a wide range of binders. There is a significant difference in the rheological properties of SAFT-conditioned and RTFOT-conditioned neat binders, and the difference is more apparent for higher stiffness binders. Additionally, there is poor correlation in the ranking of the aging susceptibility of binders as measured by the SAFT and as measured by oven-aged mixtures.

A consideration in selecting a replacement for the RTFOT was that the test, or at least the associated equipment, should show promise for future development as a replacement for the PAV. Unfortunately, it does not appear that short-term binder aging procedures can be adapted to long-term aging because it is very difficult to provide sufficient mixing of air with the binder at temperatures considered reasonable for simulating long-term aging. Attempts to modify the MGRF to improve mixing by adding scrapers and balls or rollers were not successful. Adaptation of the SAFT to long-term aging was more successful. Through changes in the design of the SAFT impeller, researchers demonstrated that equivalent PAV aging could be accomplished in approximately 40 hours, twice the time required for the current PAV.

Another consideration in NCHRP Project 9-36 was an alternate to the current RTFOT mass change procedure for quantifying binder volatility. The SAFT included a VCS that used an air-cooled condenser to collect vapors produced during aging. With appropriate glassware, the MGRF also could be modified to use a VCS. Based on the VCS study, it was concluded that the air-cooled condenser was inadequate because it only collected a small amount of the volatile compounds generated during the test. An improved VCS that incorporates a resin bead filter and a molecular sieve that are commonly used in chromatographic studies was developed. This system can be adapted to the MGRF, but not the RTFOT.

For the binders used in this study, AASHTO R30, the RTFOT, and the MGRF treated the neat and modified binders similarly. There was no difference in average aging indices between neat and modified binders in any of the three tests. For the specific aggregate used, AASHTO R30 aged the binders more than the RTFOT and MGRF. The ranking of binder aging was similar for AASHTO R30, the RTFOT, and the MGRF.

4.3 Proposals for Future Action

Based on the experiments and analyses completed in NCHRP Project 9-36, the following proposals for future action are made.

1. The MGRF is a viable alternative to the RTFOT for short-term binder aging. For a wide range of neat and modified binders, the researchers demonstrated that rheological properties for MGRF and RTFOT residue are the same. Additionally, the MGRF and RTFOT provide ranking of short-term aging susceptibility that is similar to that for mixtures aged in a forced-draft oven in accordance with AASHTO R30.
2. A modification to the mass change criterion in AASHTO M320 is needed if the MGRF is used in its current form. The mass change in the MGRF is approximately 40 percent of the mass change in the RTFOT; therefore, the mass change criterion for MGRF residue should be ± 0.40 percent to be equivalent to ± 1.0 percent for the RTFOT.

3. Different conditioning procedures are needed for short- and long-term aging. In general, procedures designed to expose binder to air at plant mixing temperatures are not capable of mixing air with the binder at the lower temperatures representative of aging during the service life of the pavement.
4. Consideration should be given to separating the measurement of binder volatility from the short-term aging procedure. This study clearly showed that only a small mass of hydrocarbon volatiles is collected during short-term aging. A simple mass-change test performed under heat and vacuum could be developed to quantify binder volatility. The test could be developed to adapt equipment (scale, pans, and vacuum oven) already available in binder testing laboratories. It is recommended that this test be pursued for use with both the RTFOT and MGRF.
5. The following additional development for the MGRF should be considered:
   - Replace the mass-change measurements in the current MGRF procedure with the vacuum volatility test outlined above.
   - Investigate modifying the MGRF test to allow aging of different volumes of binder. One of the advantages of the RTFOT is that it can be used to condition small quantities of binder. This has practical application in binder quality control testing and in designing mixtures with high percentages of recycled asphalt pavement.
   - Conduct a formal ruggedness test for the current procedure to identify appropriate tolerances for the testing conditions. Factors that should be considered include bath temperature, rotational speed, flask submersion depth, airflow rate, flask angle, binder quantity, duration, and the need for a bath cover to better control temperature.
6. Additional research to calibrate short-term aging procedures for binders and mixtures should be considered. The research completed in NCHRP Project 9-36 showed a difference between the short-term binder and mixture aging procedures, with the mixture procedure providing somewhat greater amounts of aging. It should be possible to calibrate the binder and mixture procedures to provide similar levels of aging. This research should include evaluations of plant-produced mixtures to ensure that the procedures produce representative levels of actual construction aging.
7. Additional research should be considered to adequately address long-term binder aging. Future research into long-term aging should include work with the PAV and other alternatives that may be identified in the future. This work should generally be directed at establishing operating conditions for simulated laboratory aging tests that reproduce the degree of aging that occurs in field pavements for typical binders. Mirza and Witczak's Global Aging Model, while highly empirical, provides an estimate of site-specific aging based on an analysis of historical data. Work in NCHRP Project 9-23 that was reviewed during NCHRP Project 9-36 shows that the PAV operated at 100°C for 20 hours under 304.6 psi (2.1 MPa) air pressure produces aged binders with viscosities that are in reasonable agreement with this model for a time of 10 years and moderate mean annual air temperature conditions. Based on this finding, the potential for the development of a long-term aging procedure that represents a reasonable period of service in the field is encouraging.

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APPENDIXES A THROUGH E

Appendixes A through E are not published herein but are available on the NCHRP Project 9-36 project webpage on the TRB website.

APPENDIX F

Draft AASHTO Standard for the MGRF

Standard Method of Test for Modified German Rotating Flask Equivalent of Rolling Thin Film Oven Test for Conventional Asphalts

1. Scope

1.1 This standard serves to simulate the changes in rheological properties in a conventional asphalt sample due to exposure to heat and a simultaneous supply of air. It is a large-scale version of the Rolling Thin Film Oven Test (AASHTO T-240).

1.2 This method is applicable to conventional asphalts.

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

2. Reference Documents

2.1 AASHTO and DIN Standards

T-240,	Effects of Heat and Air on a Moving Thin Film of Asphalt [Rolling Thin Film Oven Test].
T-179,	Effects of Heat and Air on Asphalt Materials [Thin Film Oven Test].
DIN 52016,	Testing the Thermal Stability of Bitumen in a Rotating Flask.

3. Summary

3.1 A 200-gram conventional asphalt sample is placed in a Morton flask attached to a rotary evaporator with a spring clip for 210 minutes at 165°C, with 2,000 mL/min air being supplied continuously. The flask is continuously rotated at 20 rpms to prevent a hard skin forming on the asphalt sample's surface.

3.2 The mass loss is determined and the conventional asphalt sample is then tested to determine the effects of heat and air.

4. Apparatus

The following apparatus shall be used:

(a) Rotary evaporator (without condenser and receiver) fitted with an adapter for a 45/50 $ joint, capable of being adjusted to a speed of (20 ± 5) rpm;

(b) 2,000 mL Morton flask with a 45/50 $ joint;

(c) Glass air supply tube, minimum of 550 mm long with an inside diameter of 7 mm, to be inserted into the flask;

(d) Compressor equipped with filter and air drier or bottled compressed air of industrial or breathing grade quality;

(e) Gas flow control device, capable of adjusting the flow rate to 2,000 ± 40 mL/min corrected to standard barometric pressure;

(f) Gas flow meter with a limit of error of ± 40 mL/min at a flow rate of 2,000 mL/min;

(g) Thermostatically controlled bath capable of being maintained at a temperature of 165 ± 1.5°C while immersing a 2,000 mL Morton flask to a depth where the sample, while rotating, is below the level of the bath fluid (see Figure 1);

(h) Bath wax heat transfer liquid with a flash point of greater than 300°C, capable of being safely maintained at operating temperatures;

(i) ASTM Loss on Heat Thermometer 13C, immersed in the bath up to the 163°C mark in a vertical position;

(j) Balance with a capacity of 1,000 grams and accurate to 0.01 g;

(k) Oven capable of being adjusted to 165 ± 1.5°C.

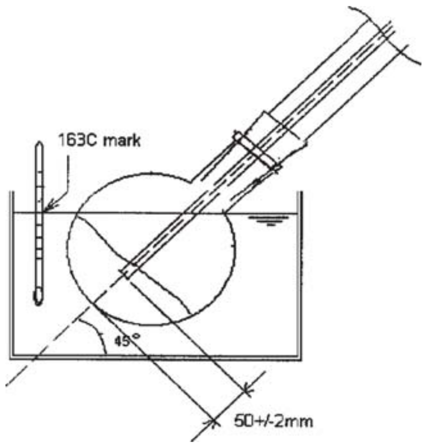

Figure 1. Test arrangement.

5. Procedure

5.1 Exposure of Asphalt to Heat Including Determination of Change in Mass

Weigh 200 ± 1 g of the sample (i.e., the specimen) into the tared flask and, after the specimen has cooled to ambient temperature (18 to 28°C), determine the initial mass (m_e) and report it to the nearest 0.01 g.

Place the Morton flask containing the specimen in the bath, with its axis at 45° to the horizontal and its bulbous part immersed so that the sample, while rotating, is below the level of the bath fluid maintained at a temperature of 165 ± 1.5°C (see Figure 1). The 13C thermometer should be immersed in the bath up to the 163°C mark in a vertical position. *Use caution during this step to avoid splashing of the hot bath wax.*

Fix the air supply tube so that its axis coincides with that of the flask, with a clearance of 50 ± 2 mm between tube and flask bottom.

Heat the specimen for 10 ± 1 minutes while rotating the flask at 20 ± 5 rpm. Then, introduce air at ambient temperature, at a rate of 2,000 ± 40 mL/min. During the test, maintain the bath temperature at 165 ± 1.5°C and rotate the flask at 20 ± 5 rpm.

After 210 ± 1 minutes, measured from the time when flask was first immersed, stop both the air flow and the flask rotation, and immediately remove the flask from the bath. When the flask has cooled slightly, carefully remove all bath wax residue adhering to its surface for proper mass determination. *Caution: The surface may still be hot.*

Cool the flask to ambient temperature (18 to 28°C), determine the final mass of the flask (m_a) and report it to the nearest 0.01g.

Heat the contents of the flask in an inverted position with an 8-oz ointment tin placed under the mouth of the flask in an oven at 160 ± 5°C for approximately 15 to 20 minutes, or until the sample has stopped dripping out of the flask. Following that, transfer the specimen to the vessels or molds required for subsequent testing.

5.2 Exposure of Bitumen to Heat without Determining the Change in Mass (TB)

Proceed as specified in Subclause 5.1 using a bitumen specimen of 200 ± 1 g, determination of the initial mass (m_e) to an accuracy of 0.01 g not being required here.

Following exposure to heat, carefully remove all bath wax residue adhering to its surface. *Caution: The surface may still be hot.* Place the flask in an inverted position with an 8-oz. ointment tin placed under the mouth of the flask in an oven at 160 ± 5°C for approximately 15 to 20 minutes, or until the sample has stopped dripping out of the flask. Following that, transfer the specimen to the vessels or molds required for subsequent testing.

6. Evaluation and Expression of Results

The change in mass, Δm, shall be calculated as a percentage by mass, expressed to the nearest 0.01%, using the following equation:

$$\Delta m\% = \frac{m_a - m_e}{m_e} \times 100$$

where:

m_e is the specimen mass prior to heat exposure;
m_a is the specimen mass after heat exposure.

7. Precision and Bias

Precision—The research required to develop precision estimates for this test method has not been conducted.

Bias—The research required to develop precision estimates for this test method has not been conducted.

Abbreviations and acronyms used without definitions in TRB publications:

AAAE	American Association of Airport Executives
AASHO	American Association of State Highway Officials
AASHTO	American Association of State Highway and Transportation Officials
ACI–NA	Airports Council International–North America
ACRP	Airport Cooperative Research Program
ADA	Americans with Disabilities Act
APTA	American Public Transportation Association
ASCE	American Society of Civil Engineers
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
ATA	Air Transport Association
ATA	American Trucking Associations
CTAA	Community Transportation Association of America
CTBSSP	Commercial Truck and Bus Safety Synthesis Program
DHS	Department of Homeland Security
DOE	Department of Energy
EPA	Environmental Protection Agency
FAA	Federal Aviation Administration
FHWA	Federal Highway Administration
FMCSA	Federal Motor Carrier Safety Administration
FRA	Federal Railroad Administration
FTA	Federal Transit Administration
HMCRP	Hazardous Materials Cooperative Research Program
IEEE	Institute of Electrical and Electronics Engineers
ISTEA	Intermodal Surface Transportation Efficiency Act of 1991
ITE	Institute of Transportation Engineers
NASA	National Aeronautics and Space Administration
NASAO	National Association of State Aviation Officials
NCFRP	National Cooperative Freight Research Program
NCHRP	National Cooperative Highway Research Program
NHTSA	National Highway Traffic Safety Administration
NTSB	National Transportation Safety Board
PHMSA	Pipeline and Hazardous Materials Safety Administration
RITA	Research and Innovative Technology Administration
SAE	Society of Automotive Engineers
SAFETEA-LU	Safe, Accountable, Flexible, Efficient Transportation Equity Act: A Legacy for Users (2005)
TCRP	Transit Cooperative Research Program
TEA-21	Transportation Equity Act for the 21st Century (1998)
TRB	Transportation Research Board
TSA	Transportation Security Administration
U.S.DOT	United States Department of Transportation