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16. Abstract

Two procedures to simulate HMAC plant aging are currently used. These are the rolling thin film oven test (RTFOT) and the thin film oven test (TFOT). When used for unmodified asphalts these methods are essentially identical in simulating asphalt short-term aging. However, when applied to modified binders, practitioners have encountered a number of problems with both RTFOT and TFOT procedures. Modified asphalts tend to form surface films that reduce oxygen diffusion and cause uneven aging. Many modified materials are too viscous to form good films in RTFOT bottles and are also difficult to remove after the test is completed.

The objective of this research is to develop an improved test procedure and a new apparatus to address the shortcomings of the existing aging techniques. The procedure should meet the following requirements: non-prohibitive cost, similar testing time and aging effect to the RTFOT, capable of producing up to 200 g of aged material per test, no prolonged handling of hot equipment or materials, and simplified cleanup.

Preliminary results show that an air blowing technique in an agitated vessel allows duplication of the RTFOT aging effect and therefore can serve as a procedure to simulate hot-mix asphalt aging. Constant agitation of material by the mixer prevents film formation and enhances air diffusion through the bulk of the asphalt. The time required to achieve the same aging is the same or even shorter than specified by the RTFOT. In addition to these advantages, the ability to control sample temperature, to collect volatile compounds and to use a single, easy to clean reusable container, as well as potentially lower equipment costs and decreased handling of hot equipment should be noted.

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# IMPROVED HMAC PLANT BINDER AGING SIMULATION REPORT OF PRELIMINARY FINDINGS AND INTENDED PROJECT DIRECTION

Project No 0-1742

## **PROBLEM STATEMENT AND RESEARCH OBJECTIVES**

Two procedures to simulate HMAC Plant Aging are currently used. These are the rolling thin film oven test (RTFOT) (ASTM D2872, AASHTO T240) and the thin film oven test (TFOT) (ASTM D1754, AASHTO T179). When used for unmodified asphalts these methods are essentially identical in simulating asphalt short-term aging. However, when applied to modified binders, practitioners have encountered a number of problems with both RTFOT and TFOT procedures. Modified asphalts tend to form surface films that reduce oxygen diffusion and cause uneven aging. Many modified materials are too viscous to form good films in RTFOT bottles and are also difficult to remove after the test is completed.

The objective of this research is to develop an improved test procedure and a new apparatus to address the shortcomings of the existing aging techniques. The procedure should meet the following requirements:

- non-prohibitive cost,
- similar testing time and aging effect to RTFOT,
- capable of producing up to 200 g (0.44 lbm) of aged material per test,
- no prolonged handling of hot equipment or materials, and
- simplified cleanup.

# LITERATURE REVIEW

#### **Review of Hot-mix Aging Simulation**

The history of accelerated asphalt aging dates back to1897 when Dow proposed two tests to analyze this material (Welborn, 1984; Lewis and Welborn, 1940). One of the tests involved heating 20 grams of asphalt at 204 °C (400 °F) for 30 hours, then weighing the residue to determine mass loss. The other test required mixing asphalt and sand at 149 °C (300 °F) and

sampling before and after heating for 30 minutes. The residue was extracted and recovered and the penetration determined. These tests served as the basis for currently used asphalt aging tests.

An ASTM Committee on Road and Paving Materials was formed to design appropriate test methods for asphalts (Welborn, 1984). By 1911 the test developed by the Office of Public Roads and Rural Engineering of the Department of Agriculture had been adopted. This test required that 20 g of asphalt be placed in a shallow tin and heated at 163 °C (325 °F) for 5 hours. The amount of volatilization and retained penetration were checked. A 1916 revision of this test included a larger sample of 50 grams.

Shattuck (1940) developed a test that simulated the actual conditions in the batch plant: 1880 grams of sand were heated to 204 °C (400 °F) and mixed with 20 grams of asphalt heated to 149 °C (300 °F). The mixing took place in a small pugmill-type laboratory mixer for one minute. The mixture was then placed in a pan and heated to 177 °C (350 °F) for 30 minutes. Extraction and recovery were performed after cooling the sample to room temperature. This test resulted in slightly more hardening than was present in actual hot-mix plants but provided a good indication of asphalt behavior under these severe conditions.

Lewis and Welborn (1940) designed a test consisting of shallow pans with asphalt 0.125 inch (3.2 mm) thick. These pans were placed on a rotating shelf in an oven maintained at 163 °C (325 °F) for 5 hours. Tests were also performed at thicknesses of 0.0625 inch (1.6 mm) and 0.0313 inch (0.8 mm) and at times of 5 and 7 hours. The penetration, ductility and softening point resulting from the hot-mix conditions were most closely duplicated by the test conducted at 163 °C (325 °F) for 5 hours and the 0.125 inch (3.2 mm) films. This test is still widely used and referred to as the Thin Film Oven Test.

The recognition that thinner films may be desired to simulate hot-mix aging led to the development of microfilm tests (Fink, 1958; Traxler, 1967). Thinner films correspond better to actual pavement mixtures, harden faster, and lessen any diffusion effects that may be present in thicker films. The Microfilm Durability Test involves placing a 5-micron film on glass slides before subjecting the sample to aging in an oven (Griffin et al., 1955). This test was designed to simulate road aging, but has been correlated with hot-mix as well (Heithaus and Johnson, 1958; Simpson et al., 1959). Only small amounts of asphalt can be aged with this method, and special devices for measuring viscosity are required. Other literature sources (Lewis and Welborn, 1940)

indicate that 1 mm films are thin enough to eliminate the diffusion effects with modified and unmodified asphalts.

Hveem et al. (1963) designed the RTFOT to age large amounts of asphalt uniformly in thin films. This test uses 35 grams of asphalt in special rotating glass bottles in an oven maintained at 163 °C (325 °F). Rotation of these bottles produces constantly renewed films of 5 to 10 micron thickness. Air is blown into the bottles to remove volatile compounds and promote oxidation. The RTFOT, run for 75 minutes after reaching 163 °C (325 °F) (85 minutes including warm-up), simulates the physical properties of hot-mix samples.

Numerous variations have been made on the TFOT and RTFOT methods with many intended to simulate road aging as well. Vallerga et al. (1957) proposed tilting the oven used for the TFOT and provided an effect similar to that of the RTFOT. Schmidt and Santucci (1969) developed a rolling microfilm test in which benzene-dissolved asphalt was cast in bottles and aged in a manner similar to that used in the RTFOT. The modification of adding a circulating fan as suggested by Schmidt (1973) has been maintained. Schmidt (1973) has also used several modifications to the rolling microfilm test at various temperatures and rates of air circulation, as well as in the presence of aggregate, in an effort to simulate hot-mix and road aging.

Drum mixers may require slightly different laboratory simulations than batch mixers. The presence of a fair amount of moisture in some plants initiated the theory that something similar to steam distillation may be occurring in drum plants. A Small Steam Distillation (SSD) technique involving steam bubbling through an asphalt sample was found not to represent drum hot-mix plants (Chollar et al., 1989). Two other methods, Forced Air Distillation (FAD) and Revolving Forced Air Distillation (RFAD), were found to closely resemble drum mixers. These methods involve blowing air over asphalt samples and collecting the volatile matter removed. The FAD and RFAD are similar in operation to the TFOT and RTFOT methods, respectively, but the FAD method is performed at a temperature of 328 °C (622 °F). The FAD and RFAD tests are reportedly better than TFOT and RTFOT in representing the aging that takes place in drum mixers (Chollar et al., 1989).

The TFOT results have been well correlated with extracted hot-mix samples based mostly on viscosity and penetration data (Chipperfield et al., 1970; Page at al., 1985; Chollar et al., 1989; Epps and Kari, 1983; Lewis and Welborn, 1940; Button et al., 1983; Lee, 1973; Hveem et al., 1959; Bright and Reynolds, 1962; Sisko and Brunstrum, 1968; Adam, 1988). The RTFOT results have also been shown to match extracted hot-mix samples (Chipperfield et al., 1967; Hveem et al., 1963; Chollar et al., 1989; Epps and Kari, 1983; Button et al., 1983; Kim et al., 1987; Thenoux et al., 1988) as well as TFOT results (Schmidt, 1973a; Schmidt, 1973b). The RTFOT is considered to be the better of the two methods. The RTFOT requires a shorter aging time, is easier to perform, and gives more precise results than the TFOT (Schmidt, 1973b; discussion by Schmidt in Skog, 1967). This added precision is attributed to the fact that the RTFOT ages more uniformly than the TFOT (Chipperfield et al., 1970). Both methods are considered interchangeable, however, to predict hot-mix aging.

Several properties and tools can verify these tests. The most common method of evaluation is the comparison of the physical properties of the test samples and the extracted asphalt. These properties include the penetration and viscosity values at various temperatures, ductility, softening point, and viscoelastic properties. The chemical composition as indicated by either Corbett or Rostler fractions has also been used (Chipperfield et al., 1970; Thenoux et al., 1988; Brule et al., 1986). Chipperfield et al. (1970) conclude that the chemical changes that occur in the hot mix are greater than those produced in the TFOT or RTFOT tests. Recently, more sophisticated tools have been used to evaluate the validity of hot-mix simulations. These include the use of Gel Permeation Chromatography (GPC) to provide a molecular size distribution of the asphalt molecules before and after the hot-mix and test procedures (Chollar et al., 1986; Edler et al., 1985; Sisko and Brunstrum, 1968; Jennings et al., 1982; Brule et al., 1986; Glover et al., 1989). Chemical functional group analysis using infrared spectroscopy has also been used effectively for this purpose (Chollar et al., 1989; Glover et al., 1989; Dickinson, 1980). These various methods do not necessarily support one another, however. For example, different infrared absorbances do not necessarily indicate different physical properties (Dickinson, 1980; discussion by Plancher in Dickinson, 1980). An ideal test would duplicate the changes that occur in the hot-mix plant as detected by all of these techniques.

Our laboratory has done extensive comparison of TFOT, RTFOT and the aging of asphalts extracted from hot mix (Jemison et al., 1991). Hot-mix samples were extracted and the properties were compared to the same asphalts following TFOT and RTFOT aging. The results showed TFOT and RTFOT to be essentially identical but the extracted hot-mix materials were

usually more aged as indicated by a variety of physical and chemical tests: complex viscosity at 60 °C (140 °F) and 135 °C (275 °F), penetration at 25 °C (77 °F), infrared analyses of carbonyl formation and the percent large molecular size as indicated by GPC analyses.

TFOT and RTFOT were in good agreement for all of these tests and the GPC chromatographs of the aged materials were identical. Others have reported that RTFOT is generally more severe than TFOT and may change the relative rankings (Zupanick, 1994; Bishara and McReynolds, 1995; Phromsorn and Kennedy, 1995; Huang et al., 1996). Agreement was not so good between the oven-aged materials and the extracted hot-mix material. There was considerable scatter among several asphalts, but every test showed the majority of the extracted asphalts to be more aged than the oven-aged materials. The best agreement was obtained for the 135 °C (275 °F) viscosity. Percent Large Molecular Size (LMS) showed all extracted hot-mix samples to be higher than the oven-aged materials. Additionally, the shape of the chromatograph was different for the extracted asphalt. These differences likely result from the different mechanisms of oxidation that occur in the hot-mix plant where oxidation is very rapid as the asphalt is exposed to the large surface area of hot aggregate.

A number of comparative studies have been conducted to evaluate the compatibility of different aging methods. Shiau et al. (1992) compared TFOT and RTFOT at different temperatures. They report that the rolling thin film oven (RTFO) method is more severe than the thin film oven (TFO) method at 141 °C (285 °F) and 163 °C (325°F) but at 185 °C (365 °F) the aging effect is approximately the same. Kandhal and Chakraborty (1996) report results from an aging procedure identified as SHRP N-1025 in which loose mix is aged in a forced draft oven for 4 hours at 135 °C (275 °F). The materials were extracted by the Abson method for analysis. The asphalt to aggregate ratio was varied to study the effect of film thickness and was not directly related to hot-mix aging. As expected, aging was greater at low film thickness.

Bishara and McReynolds (1995, 1996) compared aging by microwave to TFOT and RTFOT aging on the basis of Superpave specifications, finding in general a fairly good agreement with 18 asphalts. Dunning and Meeks (1968) compared the results of asphalt air blowing to those obtained by RTFOT. Comparisons were made only on the basis of softening point but the agreement was very good. According to this study the oxidation mechanism of a thin film of asphalt at elevated temperatures is essentially the same for RTFOT and air blowing.

#### **Modified Asphalts**

Sirin et al. (1998) have developed a rotovapor procedure to simulate short-term aging of modified and unmodified asphalts, replacing the usual vacuum connection by airflow. They compared results with TFOT and RTFOT for an AC-30 and an AC-30 with 10 percent 80 mesh crumb rubber. Fifty grams of sample were aged for 85 minutes at 163 °C (325 °F) and 185 °C (365 °F). The aging effect at 163 °C (325 °F) was between the ones caused by TFOT and RTFOT. The rate of aging decreased with increasing sample size.

Later the procedure was modified using a Morton flask (Sirin et al., 2000). This achieved lower temperature variation in the oil bath and better film agitation in the flask. At 163 °C (325 °F) and sample weight 200 g it takes 160 minutes to achieve the aging effect closest to that of TFOT and 210 minutes to compare with that of RTFOT.

Another approach to address the problems encountered with modified viscous materials was suggested by Bahia et al. (1998) who placed 127 mm by 6.4 mm diameter steel rods within the RTFOT bottles. This allowed the creation of shearing forces to improve the spreading of thin films that in turn accelerated aging of modified binders. No negative effect was attributed to the rods observed at the same time the spillage problems were eliminated.

# DISCUSSION

It is logical to assume that a realistic simulation of hot-mix aging would require the use of some solid phase with a large area comparable to the aggregate. While technically this may be true, there are several reasons for rejecting it. In the first place, it would considerably increase the time required to run the test, but a greater problem is the complexity introduced by the necessity of extracting the asphalt from the solid phase for the subsequent analysis. The fact that the principal objective of this project is to develop a method that can be used with modified asphalt rules out such a technique. It is nearly impossible to extract a complex composition from a solid phase without changing its molecular microstructure. For many additives it is nearly impossible to recover the entire modifier with the asphalt. Moreover, the extraction process changes the dispersion of the modifier and thus the properties of the recovered material.

Even with unmodified asphalts, extraction and recovery can introduce as much error as exists in procedures not requiring the solid phase. Accumulated experience with extraction and recovery of asphalts shows it to be much more complex and subject to more problems than is generally realized (Burr et al., 1990; Burr et al., 1991; Cipione et al., 1991; Burr et al., 1993; Burr et al., 1994).

Judging by the greater aging that typically occurs in the hot-mix plant than in the RTFOT, it might be desirable to make the test somewhat more severe. This severity could likely be accomplished by a small increase in temperature and the change could be justified by the fact that the rapid aging that occurs in the hot-mix plant might actually be better simulated by a higher temperature without aggregate. On the other hand, as long as Superpave specifications are based on RTFOT results, any new test must be able to duplicate the RTFOT aging.

The question remains whether the RTFOT can be made to work with modified asphalts. As noted above, it is claimed that putting steel rods in the bottles solves the problem. Even if this is generally true, which is questioned, the RTFOT has other problems. It is hard to clean and hard to remove the asphalt from the bottles, a difficulty complicated by rods. The problems of handling hot equipment as well as high cost of equipment are also important issues. It would be desirable to have equipment that is easier and safer to use as well as less expensive to build and maintain.

The rotovapor design has positive features. In particular, conducting the aging in a rotating flask prevents spillage and film formation problems. This method also allows a variety of operating conditions, such as airflow rate, speed of rotation, and sample weight. However, these possible advantages can pose additional problems in terms of selecting optimal values of multiple variables and ensuring test reproducibility. For instance, the airflow rate, sample size, even the angle of rotating flask and the depth of its submergence into the heating bath can potentially affect the test results. Moreover, heat transfer is not uniform in the system that creates varying temperature distribution and makes the sample temperature difficult to control.

The decreasing of film thickness to avoid the diffusion limitation might seem like a reasonable solution. The test time will also decrease. However, to age the necessary amount of material a large sample area will be required.

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The microwave idea is appealing in that it is rather simple and fast to carry out. However, the mechanism of hardening might be different and non-oxidative reactions might be occurring, which actually can be verified by infrared (IR) and GPC measurements. We are skeptical about applying microwave radiation to simulate a process that does not involve any source of microwaves as such.

The air blowing procedure appears to be particularly interesting. The equipment is easy to assemble, operate and clean. The entire sample is placed in a single, easily emptied container that could handle a sample of any desired mass. Some previous studies of asphalt air blowing have been carried out at our lab, particularly at lower temperatures (below 232 °C [450 °F]). The results indicate that good material can be produced without undergoing any harmful or undesirable changes. Therefore, the effect identical to RTFOT aging can be achieved by asphalt air blowing under conditions similar to those of the oven test. On the other hand, air blowing could be subject to the effects of viscosity and excessive volatile loss. It can be sensitive also to the air flow and distribution, so these aspects have to be addressed.

# **Modified Asphalts**

Several serious problems need to be recognized in the simulation of either hot-mix or road aging when modifiers are present. In the first place both are difficult to verify due to complexity of asphalt recovery from the hot-mix without serious changes in its composition and properties.

Secondly, oxidation of modified asphalt is much more complicated if the modifier reacts with asphalt or aggregate. Reaction rates of modifier and asphalt may, and probably will, respond differently to changing conditions of time and temperature. The presence of modifiers introduces very time dependent diffusion effects, which may be quite different in the rapid oxidation of a hot-mix plant and the slower oxidation of a test situation.

# PRELIMINARY RESULTS

Five asphalts were air blown using the prototype laboratory unit depicted in Figure 1. Three hundred grams of heated asphalt were placed in a one-quart can and air blown with constant agitation by a mixer attached to a drill press. The can was heated by an insulated heating tape and maintained at 163 °C (325 °F) by a thermocouple and temperature controller. The time of air blowing and airflow rate varied slightly (Table 1). TFOT and RTFOT aging procedures were performed on the same array of asphalts and a number of parameters were obtained to characterize the aging effect. These parameters include limiting complex viscosity, phase angle, loss, storage and complex moduli as determined by the Carri-Med dynamic shear rheometer, penetration at 25 °C (77 °F) (ASTM D 5-97), softening point (ASTM D 36-86), infrared spectroscopy measurements and size exclusion chromatography (SEC), also known as gel permeation chromatography (GPC). One modified asphalt, AMI's multigrade asphalt, was used in this preliminary testing phase to test the applicability of this technique to highly viscous materials.. Although it requires more attention and more elaborate testing procedure, the method proved to eliminate the shortcomings typical for the standard methods.

Aging	Aging Conditions						
Method							
	SHRP	SHRP	SHRP	SHRP	Exxon AC-10		
	AAA-1	AAD-1	AAF-1	AAM-1			
RTFOT	163 °C, 85 min	163 °C, 85 min	163 °C, 85 min	163 °C, 85 min	163 °C, 85 min		
TFOT	163 °C, 5 h	163 °C, 5 h	163 °C, 5 h	163 °C, 5 h	163 °C, 5 h		
Air	163 °C, 65 min,	163 °C, 80 min,	163 °C, 70 min,	163 °C, 65 min,	163 °C, 70 min,		
blowing	air flow – 2788	air flow - 2788	air flow - 2788	air flow - 2788	air flow – 2788		
	ml/min	ml/min	ml/min	ml/min	ml/min		

Table 1. Materials and procedures used in the preliminary experiments

IR spectra were obtained by a Mattson 5000 Galaxy Series FTIR spectrometer and compared on the basis of carbonyl area growth determined by calculating the area of the peak in the range of wavenumber between 1820 to 1650 cm<sup>-1</sup> in arbitrary units. The spectra obtained for Exxon AC-10 are shown in Figure 2. No significant amount of chemical changes occurs during short-term oxidation as indicated by the IR spectra. Only a slight difference was observed in the carbonyl and sulfoxide regions. The enlarged picture of carbonyl region of the IR spectra (Figure 3) shows that the carbonyl area of aged materials is generally bigger than the one of unaged asphalt, though the difference is not very significant. Therefore, IR measurement is not a very sensitive test of short-term aging. The comparison of carbonyl areas determined for different asphalts is given in Figure 4 and it shows generally good agreement between applied aging procedures.

Figures 5 through 8 summarize the rheological properties, penetration, and  $T_{R\&B}$  results. They also demonstrate good agreement of these properties among various aging techniques. Apparently, the softening point is not very sensitive to demonstrate the difference in aging effect, although rheological data show some difference among the procedures. The biggest difference was observed for SHRP AAF-1 asphalt. However, it should be noted that the viscosity of the virgin asphalt is almost twice as great as the viscosity of other materials that leads to higher viscosity values and higher sensitivity to aging conditions. The difference in aging effect might also be partly attributed to slightly varying aging conditions that will have to be optimized and specified for subsequent testing.

Figure 9 shows the SEC chromatogram obtained for aged and unaged Exxon AC-10. The air blown, TFOT, and RTFOT aged asphalts appear to be identical in terms of molecular weight distribution. They have greater concentration of higher weight molecules expressed by a larger peak of refractive index (RI) response recorded at 23 minutes of retention time. The chromatograms obtained for other asphalts demonstrated similar results.

#### CONCLUSIONS AND RECOMMENDATIONS

The main conclusion that can be deduced from the analysis of preliminary results is that the air blowing technique allows duplication of the RTFOT aging effect and therefore can serve as a procedure to simulate hot-mix asphalt aging. Constant agitation of material by the mixer prevents film formation and enhances air diffusion through the bulk of asphalt. Time required to achieve the same aging is the same or even shorter than specified by the RTFOT. In addition to these advantages, the ability to control sample temperature, to collect volatile compounds and to use a single, easy to clean reusable container, as well as potentially lower equipment costs and decreased handling of hot equipment should be noted. After conducting the preliminary studies, we will consider several tests and tasks as highlighted below.

#### **An Air Blowing Test**

This test is the primary direction of the further work. In the next stage of this project the prototype of testing equipment will be built according to the design in Figure 10. The apparatus will include a vessel holding the entire sample of asphalt with a place for air introduction and

dispersion. It is proposed to use a 500 mL can for the next stage of experiments. The can will be covered with a lid with a thermocouple and condenser attached. It will be possible to collect the volatile matter, determine the weight loss and analyze its chemical composition. The sample mass can be chosen from 150 to 250 grams. The mixer will definitely be introduced into the vessel since this will speed both the oxygen diffusion and asphalt oxidation rates and make the test less sensitive to asphalt viscosity.

Instead of heating tape and insulation, a heating mantle will be used to keep an airblowing vessel at a specified temperature. Temperature and airflow rate should be selected to optimally represent standard short-term aging procedure as well as to simulate hot-mix aging. Temperature and flow controlling devices will be used. Experiments with various materials will be conducted in order to determine the optimal testing conditions (time, airflow rate, temperature control, sample mass and RPM of mixer) and to make possible design corrections. The properties of aged asphalts will be evaluated by available analytical techniques such as rheological measurements, GPC, IR, penetration, softening point, and other physical and chemical tests.

#### A Very Thin Film Oven Test

This test will be an alternative procedure. The experimental settings will resemble the TFOT procedure with the exception that the depth of the trays will be 1 mm with correspondingly larger areas. As shown in Figure 11 the trays containing asphalt samples are placed into the oven without air circulation. The duration of the test has not been determined yet, but it will definitely be less than the 5 hours specified for the TFOT procedure. Most likely it will be between two and three hours. First, the conditions necessary to duplicate the standard tests will be determined and then the test will be run on various modified asphalts to evaluate its applicability.

#### A Microwave Oven Test

This test will probably not be studied at the next stage of the project due to the possibly different mechanism of hardening. However, this technique can potentially result in a fast and simple procedure making studies of microwave aging of asphalt and their correlation with conventional aging techniques an interesting and useful research direction. Some initial studies could be run in addition to the primary approach using a kitchen microwave unit. The purpose of these studies will be to determine if there is real potential for or whether there is too much difference between this and conventional oven tests. One test can be run under nitrogen and any aging effect can indicate non-oxidative reactions. If the test is favorable, then details of power, time, sample size, etc., can be studied.

### **Parallel Tasks**

As discussed in the literature review section, the oven aging, while doing a reasonable job of duplicating hot-mix viscosity, appears to produce a different material. Another task to be pursued throughout the course of the project is to assess whether, and how, the aging test might be a more faithful representative of the hot-mix process. In addition to physical properties, chemical analysis (e.g., GPC and IR) will be of particular interest since it allows making a conclusion about the degree of aging regardless of changes in a material's microstructure that occur due to extraction, as discussed above. Another problem is that the modifier and the asphalt have different mechanism of oxidation. It is nearly impossible to extract the entire modifier as well. The test procedure for evaluating the extracted modified asphalt to improve simulation is the second parallel task to be carried out during the next stages of this project.

A possible procedure may include the following steps:

- Extract the hot-mix sample as thoroughly as possible.
- Centrifuge or filter to remove all but dissolved or colloidal material.
- Take an aged sample of the same modified asphalt from the hot-mix simulation test.
- Put it in an equal amount of solvent and centrifuge or filter this sample.

It is very likely that the material not recovered by centrifuging or filtration will be similar, so the IR and GPC on this material should indicate the similarity of this phase. The solid material can also be analyzed by IR or for total oxygen content to see if it is also in a similar state of oxidation. Similarity of the phases will at least be a good indication that hot-mix and aging simulations are producing similar results.

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Figure 1. Air Blowing Unit Used for Preliminary Experiment.



Figure 2. IR Spectra of Unaged and Aged Exxon AC-10.



Figure 3. Carbonyl Area of Unaged and Aged Exxon AC-10.



Figure 4. Carbonyl Area of Unaged and Aged Asphalts.



Figure 5. Limiting Complex Viscosity at 60 °C and 0.1 rad/s of Aged and Unaged Asphalts.



Figure 6. Loss Modulus G" at 60 °C and 10 rad/s of Aged and Unaged Asphalts.



Figure 7. Penetration at 25 °C of Aged and Unaged Asphalts.



Figure 8. Softening Point  $(T_{R\&B})$  of Aged and Unaged Asphalts.



Figure 9. GPC Spectra of Unaged and Aged Exxon AC-10.



Figure 10. Proposed Experimental Setup.



Figure 11. Very Thin Film Oven Test.