Laboratory Aging of Asphalt Binders
Status Report on NCHRP 9-36

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Review - Project Objective

- Objective: To **select**, **refine**, and **validate** an improved method for the short-term laboratory aging of asphalt binders.
- Primary Use of Method: Conditioning procedure for use with asphalt binder purchase specifications.
- Requirements:
  - Replicate RTFOT aging for neat binders
  - Provide quantitative measure of volatility loss
  - Correlate with laboratory aging of mixtures
  - Extendable to long-term (in-service) aging
Review - Project focus

- Short-term aging with consideration of applicability to long-term aging
- Physical properties as opposed to chemical
- However....
  - Need to understand and be guided by chemistry of aging process
  - Tradeoff - Method must be as timely and simple to perform as possible but still properly reflect mechanisms operative during the aging process
Project tasks - Status

- Phase I
  - Literature search and research in progress
  - Identify promising procedure(s) for further study
  - Interim report
  - Selection study (SAFT vs. MGRF) and report

- Phase II
  - Refine test procedure - optimization study
  - Validate using short-term lab mixture aging
  - Final report and draft standard
Some Selection Criteria

- Applicable to purchase specification use
  - Simple, timely, easy to perform, repeatable
- Environmentally friendly
  - Minimal solvent usage
- Applicable to neat and modified binders
- Potentially applicable to long-term aging
  - Possible to obtain kinetic information
- Provide sufficient material for physical testing
- Includes procedure for collecting volatiles
  - Volatile loss not equal to mass change - different mechanisms

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Candidate methods

- Methods selected as meriting further study:
  - Modified German Rolling Flask (MGRF)
  - Stirred Air Flow Test (SAFT) after Glover at TA&M

- Methods rejected for further study:
  - Belgian Rolling Cylinder Aging Test (RCAT)
  - Microwave technology,
  - Thin films exposed to atmosphere,
  - Wiped film technology,
  - Incident light,
  - Catalytic acceleration, or
  - Refinements to existing standard methods
Phase II Studies

- Study 1: Study to choose between MGRF and SAFT
  - Criterion - acceptability for long-term aging
  - Completed, SAFT selected
- Study 2: Volatile Compounds Collection Study
  - Resulted in upgraded Volatile Collection System (VCS)
- Study 3: RTFOT Optimization Study
  - (A) Verify minimal aging during heat-up period
  - (B) Optimize SAFT operating parameters so that SAFT replicates RTFOT for neat binders
- Study 4: Verification Study
(1) Selection Study (MGRF vs. SAFT)

- Phase 1: Refine SAFT and MGRF procedures to better adapt them for long-term aging
- Phase 2: Conduct experiment to compare aging obtained with SAFT, MGRF, and PAV
  - Three binders - plain and modified
  - Visual assessment of mixing and polymer dispersal
  - Relative aging compared to PAV
  - Potential for implementation
Observations - Selection Study

- **MGRF Method**
  - Gravity not sufficient to produce comparable mixing for different grades
  - Mechanical aids to enhance mixing are not practical
  - Degree of aging is controlled by viscosity

- **SAFT Method**
  - Modifications to mixing system needed for long term simulation - remainder of apparatus unchanged
  - Approximately 40 hours will be required to replicate PAV aging

- **Conclusion:** SAFT more promising for long-term aging
Original SAFT Apparatus (Glover)

- Aging Conditions
  - 163°C
  - 30 minutes
  - Atmospheric air
  - 2 liters /minute
Commercial Version by Cox

- Built in response to Texas DOT request
- Operation more automated and user friendly
  - Flow of gas automated
  - Improved temperature control - PRT inside vessel
- Replaced heating mantle with oven
  - Vessel completely enclosed
- Aging significantly less in Cox model than in original
  - Attributed to excessive heating (skin temperatures) caused by heating mantle
  - Required recalibration of aging parameters
  - Led to optimization study - currently in progress
SAFT by COX

Sits on bench at convenient height

Vessel top fully enclosed
Aging Vessel

- Air/Nitrogen Inlet
- Exhaust
- Lid
- Impeller
- RTD
- Vessel
(2) Volatile Study - Initial Observations

- Based on work with original SAFT
- Observed change 1/10th of RTFOT
- Very little material collected on lid or in condenser
  - Could not explain discrepancy with RTFOT
  - However, Glover indicated that condenser temperature does not affect amount collected
- Required reconsideration of collection system
  - Charcoal filter and silica gel traps added
  - Water collected but gel turned brownish color
  - Charcoal filter collected significant material
- Revised system mass change more in line with RTFOT
Volatile Collection System - Initial

Activated Charcoal Hydrocarbon Trap

Silica Gel Moisture Trap

Dried Laboratory Air or N₂

SAFT VESSEL

Binder

Condenser 10 ft ¼” Cu Tubing

Silica Gel Moisture Trap

Activated Charcoal Hydrocarbon Trap
## Material Collected in Various Media

<table>
<thead>
<tr>
<th>Date</th>
<th>12/16/04</th>
<th>12/17/04</th>
<th>12/22/04</th>
<th>Avg</th>
<th>Percent of Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass of AC, g</td>
<td>250.2</td>
<td>250.2</td>
<td>250.3</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>Condenser, wt %</td>
<td>0.069</td>
<td>0.064</td>
<td>0.072</td>
<td>0.069</td>
<td>11</td>
</tr>
<tr>
<td>Water Filter, wt %</td>
<td>0.108</td>
<td>0.066</td>
<td>0.045</td>
<td>0.073</td>
<td>12</td>
</tr>
<tr>
<td>Hydrocarbon Filter, wt %</td>
<td>0.502</td>
<td>0.484</td>
<td>0.454</td>
<td>0.480</td>
<td>77</td>
</tr>
<tr>
<td>Total</td>
<td>0.678</td>
<td>0.615</td>
<td>0.571</td>
<td>0.622</td>
<td>--</td>
</tr>
</tbody>
</table>
Material Collected in Various Media

- Condenser
- Hydrocarbon Trap
- Moisture Trap
- RTFOT Mass Loss
- SAFT Mass Loss

Graph showing the quantity collected, wt %, under different absolute pressures, kPa, for Condenser, Hydrocarbon Trap, Moisture Trap, RTFOT Mass Loss, and SAFT Mass Loss.
Volatile Collection System - Current

- Removed condenser - ineffective
  - Caused by “blowthrough” – concentration not sufficient to cause condensation (dew point effect)
- Replaced condenser with plastic resin beads
  - Technique similar to Kreich et. al. in fume studies
  - Two types - high and low molecular weight compounds
- Added micro sieve to capture water
- Retained activated charcoal to capture gasses
- Planned analysis of collected material
  - Gravitimetric and GCF/FID after Kreich et. al.
- Do not fear - System will be simplified for routine use
Volatile Collection System Schematic

- Activated Charcoal Hydrocarbon Trap
- Silica Gel Moisture Trap
- Pre-dried Laboratory Air or N₂
- Resin Beads Supelco Tenax TX
- Resin Beads Supelco HaySeep Q
- Microsieve Supelco 5A
- Activated Charcoal

SAFT VESSEL

BINDER
(3) Optimization Study

- **Purpose:**
  1. Verify minimal aging during heat-up
  2. Select optimal operating parameters for aging

- **Operational parameters considered:**
  - Binders - 3 sources
  - Flow rate (2 and 4 l/min)
  - Stirring speed (700 and 1400 rpm)
  - Aging time (45 and 60 min)

- **Experiment Design**
  - Partial factorial
  - Response variables include
Aging During Heat-up

- Conclusion: Heat-up cycle causes minimal aging

<table>
<thead>
<tr>
<th>Binder</th>
<th>Average G*/sinδ, kPa</th>
<th>Tank</th>
<th>After Heat-up</th>
</tr>
</thead>
<tbody>
<tr>
<td>PG 58-28</td>
<td>1.24</td>
<td>1.25</td>
<td></td>
</tr>
<tr>
<td>PG 76-22</td>
<td>1.48</td>
<td>1.39</td>
<td></td>
</tr>
<tr>
<td>PG 82-22</td>
<td>1.71</td>
<td>1.71</td>
<td></td>
</tr>
</tbody>
</table>
## Binders for Optimization Study

<table>
<thead>
<tr>
<th>Binder/Source</th>
<th>PG Grade</th>
<th>RTFOT Mass Change (%)</th>
<th>Aging Index, (135°C Vis)</th>
</tr>
</thead>
<tbody>
<tr>
<td>California Coastal (AAD-2)</td>
<td>58-28</td>
<td>-0.71</td>
<td>2.86</td>
</tr>
<tr>
<td>West Texas Intermediate (AAM-1)</td>
<td>64-16</td>
<td>+0.05</td>
<td>1.98</td>
</tr>
<tr>
<td>California Valley (AAG-2/ABM-2)</td>
<td>58-22</td>
<td>-0.25</td>
<td>1.62</td>
</tr>
</tbody>
</table>
Complications - Heat-up Time

- Time required to bring binder to aging temperature
- Want heat-up time to be consistent for all operating conditions - BUT
  - Air flow rate and stirring speed affect heat-up rate
  - Requires trial and error to set PID’s so that heat-up time is same for all combinations of flow rate and stirring speed
  - Fortunately binder type does not affect heat-up rate
- Unique settings for each flow rate and stirring speed now available
- Optimization study to proceed as soon as revised volatilization system is on line
(4) Verification Study

- Mixtures with 12 binders
  - Mixtures aged in laboratory
  - Physical properties
- Binders aged with SAFT and RTFOT
  - Physical properties
  - Volatile collection and analysis
- Binder and mixture aging to be compared
  - Comparison using Hirsch model
- Status
  - Mixture work underway
  - Binder aging to follow optimization study
Scoping Study: Effect of Air Flow Rate

Air Flow, L/min

$G^*/\sin\delta$, kPa

- AAD-2
- AAM-1
- ABM-2
Scoping Study: Effect of Stirring Speed

- **G*/sinδ, kPa**
- **Impeller Speed, rpm**

Graph showing the effect of stirring speed on **G*/sinδ, kPa**.

- **AAD-2**
- **AAM-1**
- **ABM-2**
Scoping Study: Effect of Stirring Speed

- AAD-2
- AAM-1
- ABM-2

Duration, min

G*/sinδ, kPa
## Binders for Verification Study

<table>
<thead>
<tr>
<th>Binder</th>
<th>Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>AAC-1 Neat</td>
<td></td>
</tr>
<tr>
<td>AAD-2 Neat</td>
<td></td>
</tr>
<tr>
<td>AAF-1 Neat</td>
<td></td>
</tr>
<tr>
<td>ABM-2 Neat</td>
<td></td>
</tr>
<tr>
<td>ABL-1 Neat</td>
<td></td>
</tr>
<tr>
<td>AAM-1 Neat</td>
<td></td>
</tr>
<tr>
<td>Citgoflex</td>
<td>PG 82-22 SBS Modified</td>
</tr>
<tr>
<td>ALF 64-40</td>
<td>PG 70-34 SBS Modified</td>
</tr>
<tr>
<td>Air Blown</td>
<td>PG 76-16</td>
</tr>
<tr>
<td>Evaloy</td>
<td>PG 76-22 RET Modified</td>
</tr>
<tr>
<td>EVA</td>
<td>PG 76-22 EVA Modified</td>
</tr>
<tr>
<td>Novophalt</td>
<td>PG 76-22 LDPE Modified</td>
</tr>
</tbody>
</table>

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*“Engineering Services for the Asphalt Industry”*
Thanks for your attention

Questions?
  Questions?
    Questions?
      Questions?
        Questions?
          Questions?

Answers?
  Answers?
    Answers?
      Answers?
        Answers?
          Answers?

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