Common Misconceptions and Mistakes in Asphalt Binder Testing

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Acknowledgments

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  • Mike Beavin, Technical Training Coordinator
  • Jason Lamb, Senior Technician
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• Thanks to the Member Companies of the Asphalt Institute
Presentation Format

• Background
• Common Mistakes and Misconceptions
  • Tests
  • Specifications
  • Concepts/Principles
• Resources
Common Misconceptions and Mistakes in Asphalt Binder Testing:

Background
What is Asphalt?

- Viscous liquids or solids essentially consisting of hydrocarbons and their derivatives
  - soluble in carbon disulfide (CS₂)
- Substantially nonvolatile at ambient temperatures
- Soften gradually when heated
- Often referred to as viscoelastic materials
  - behave as elastic solids at low temperatures and viscous liquids at high temperatures.
Asphalt Types

• Natural Asphalts
  • Evaporation of volatile portions of natural asphalt deposits leaving the asphalt fractions
    • Also insoluble material

• Petroleum Asphalts
  • Distillation of crude petroleum to produce asphalt
    • Little, if any, insoluble material
Petroleum Asphalt

Asphalt Binder Properties Dependent on:
- Crude source
- Refinery operations
Crude Selection

• Percent of resid or asphalt cement varies according to the crude source
  • May range from 1% to more than 50%
• Some crudes contain a high percentage of wax
  • Wax contents in asphalt cement greater than ~5% are generally considered undesirable
  • Waxy asphalts typically exhibit poor performance
Asphalt binder is composed primarily of carbon and hydrogen. Hence the name “hydrocarbon.”

Sulfur and heavy metals play a significant role in determining physical properties. Molecular structuring in asphalt binder is largely due to the presence of sulfur and heavy metals.
Chemical Changes During Processing

- **Vacuum Distillation**
  - Selectively removes higher volatility, lower molecular weight hydrocarbons
    - results in a concentration of higher molecular weight (lower volatility) components in asphalt

- **Air-Blowing and Oxidation**
  - Cyclics converted to resins, which are in turn converted to asphaltenes.
  - As asphaltene content increases, stiffness increases
Chemical Model of Asphalt

• Petroleum Asphalt
  • Collection of polar and non-polar molecules
  • Polar molecules associate strongly to form organized structures through non-polar (continuous) phase
  • As temperature increases, least polar molecules dissociate and asphalt becomes less viscous
    • Reverse happens as temperature decreases
Chemical Separation of Asphalt

• Four Broad Classes of Compounds (ASTM D4124)
  • Asphaltenes
  • Saturates
  • Cyclics (Naphthene Aromatics)
  • Resins (Polar Aromatics)
Chemical Composition and Oxidation

• Oxidation
  • Asphalt molecules react with oxygen
    • New polar sites are formed
      • Increases the tendency to self-assemble
      • Thereby increases the stiffness of the asphalt
    • Can occur at the refinery, during mixing at an asphalt mixture production facility, and/or in service in the pavement
Oxygen reacts with asphalt cement
   - Molecular size increases
   - Polarity increases
   - The binder becomes stiffer
   - This reaction is not reversible

Rate of reaction is highly dependent upon temperature
   - Rule of thumb – reaction rate doubles for every 10°C increase in temperature
Oxidation

- Asphalt molecules react with oxygen
  - Asphalts come from different crude sources
    - Have different molecular distributions and different attractive strength connecting the molecules
  - The rate of stiffening can be different
Chemical Composition and Oxidation

TRB Circular 499

Size exclusion chromatogram of three asphalts
• Different molecular profiles
  • **AAM** (West Texas Intermediate)
    • High amount of large molecules
    • Continuous molecular size
  • **AAG** (California Valley)
    • Low amount of large molecules
    • High amount of small molecules
    • Not continuous (lacking intermediate sized molecules)
  • **AAK** (Boscan)
    • High amount of large molecules
    • Low amount of small molecules
    • Not continuous (lacking intermediate sized molecules)
Chemical Composition and Oxidation

• What happens during oxidation?
  • **AAM** (West Texas Intermediate)
    • Continuous molecular size allows for conversion of smaller molecules to larger (more polar) molecules
    • “normal” increase in stiffness during aging
  • **AAG** (California Valley)
    • High amount of smaller molecules
    • More polar molecules can develop without changing physical properties as significantly
    • Less increase in stiffness during aging
  • **AAK** (Boscan)
    • High amount of larger molecules
    • Already dominated by polar species; increased by oxidation
    • Greater increase in stiffness during aging
Chemical Composition and Oxidation

$G^*, \text{ Pa (60°C, 10 rad/s)}$

- AAG
- AAM
- AAK

Original, RTFO, PAV
Factors/Conditions Affecting Asphalt Binder Properties

• All Asphalt Binders
  • Oxidation
  • Volatilization
  • Steric Hardening
  • Physical Hardening

• Polymer Modified Asphalt Binders
  • Polymer Separation
  • Polymer Degradation
Minimizing Oxidation in the Lab

• Heat to the lowest possible temperature
• Heat for the shortest possible time
• Avoid hot plates, open flames, or other heat sources that cause hotspots
• Avoid heating near-empty cans of asphalt binder or in containers with low fill heights
  • Heat small containers very carefully to avoid overheating
• Avoid multiple heating of samples
• Split into sub-samples during first heating
What is Volatilization?
- Loss of lighter weight or more volatile molecules caused by heating
  - Causes the binder to become stiffer
  - This reaction is not reversible

Minimizing Volatilization in the Lab
- Minimized by following same precautions as listed above for oxidation
- Volatilization is probably of less concern than oxidation
Steric hardening is a reversible process that occurs at room temperature.

- Polar molecules become structured with time.
- This structuring increases binder stiffness.
- Steric hardening starts immediately upon cooling and continues at a reduced rate for an extended period of time (months? years?).

- The amount of steric hardening that occurs is binder-specific:
  - Some binders show relatively small amounts.
Steric hardening is destroyed by heating
  • Referred to as annealing in test methods

Always control the amount of time between sample pouring and testing
  • For example...limit the amount of time asphalt binder is held in silicone molds before testing
    • Holding a DSR test specimen in a silicone mold for several hours or more may be sufficient to pass/fail a binder that would otherwise fail/pass
• Physical hardening is a reversible process that occurs below room temperature
  • Mechanism that causes physical hardening is not well understood
  • Apparently a different mechanism than steric hardening
  • More prevalent in binders with high wax contents
• Reversed by heating to room temperature
Physical hardening occurs over an extended period of time but at a decreasing rate.

- Increase in stiffness from physical hardening after 24 hours may be greater than the aging that occurs in the PAV.

- Unless testing time is controlled physical hardening can affect test variability.
  - $60 \pm 5$ minute testing window with BBR gives “standard” condition.
  - Need repeatable time from one lab to another.
Physical Hardening is Binder-Specific

- AAD-1 at -25°C
- AAF-1 at -15°C
- AAD-1 at -10°C
- AAM-1 at -15°C

Graph showing stiffness in MPa at 60 seconds vs time in minutes. The graph compares different binders at various temperatures.

- AAF-1 (-15°C)
- AAD-1 (-25°C)
- AAM-1 (-15°C)
- AAD-1 (-10°C)
Polymer Separation

• Separation during storage
  • Tendency is binder (and modifier) specific
  • Some systems are more stable than others
  • Separation may occur during storage

• Polymer tends to float to top giving “scum”
  • If this scum persists with stirring test results may not be representative
  • Removing “scum” removes polymer and test results are no longer representative
• Some polymers may degrade when heated at high handling temperatures
  • Temperature is polymer-specific
  • Use manufacturer recommendations for handling and do not overheat
• Polymer degradation cannot be reversed
  • Degradation causes a softening of the asphalt binder
Common Misconceptions and Mistakes in Asphalt Binder Testing:
Tests, Specifications, and Concepts
Penetration

• Penetration
  • ASTM D5 (AASHTO T49)
    • One of oldest asphalt tests
  • Standard needle allowed to penetrate into sample under specified loading conditions
    • 25°C – 100 grams, 5 seconds
    • 0°C – 200 grams, 60 seconds
    • 46°C – 50 grams, 5 seconds
  • Depth of penetration is recorded in 0.1-mm units (dmm)
  • Three penetration readings per test
Penetration
Mistakes and Misconceptions

- Not strictly adhering to the conditioning times listed in the standard
# Steric Hardening Experiment – SHRP

<table>
<thead>
<tr>
<th>Asphalt ID</th>
<th>Penetration, 1/10 mm</th>
<th>3 hours (A)</th>
<th>24h hours (B)</th>
<th>14 Days (C)</th>
<th>Many years (D)</th>
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<tbody>
<tr>
<td></td>
<td>Value</td>
<td>Value</td>
<td>B / A</td>
<td>Value</td>
<td>C / A</td>
</tr>
<tr>
<td>AAA1</td>
<td>89.0</td>
<td>92.7</td>
<td>1.04</td>
<td>79.7</td>
<td>0.90</td>
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<td>AAD1</td>
<td>95.3</td>
<td>85.3</td>
<td>0.90</td>
<td>76.3</td>
<td>0.80</td>
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<td>AAB1</td>
<td>58.0</td>
<td>53.7</td>
<td>0.93</td>
<td>36.7</td>
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<td>AAK1</td>
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<td>0.65</td>
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<tr>
<td>AAG1</td>
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<td>42.3</td>
<td>0.95</td>
<td>33.7</td>
<td>0.76</td>
</tr>
<tr>
<td>AAM1</td>
<td>43.7</td>
<td>40.0</td>
<td>0.92</td>
<td>37.7</td>
<td>0.86</td>
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</table>

Sealed 3-oz tin. Sample not reheated before testing.
• **Mistakes and Misconceptions**
  • Dimpling the surface of the asphalt binder prior to the test
Penetration at 25°C:
Paving and Roofing Asphalt Binders

#### Z-Scores

<table>
<thead>
<tr>
<th>Date</th>
<th>Pen-25C A</th>
<th>Pen-25C B</th>
<th>AI Roofing PSP</th>
</tr>
</thead>
<tbody>
<tr>
<td>4/15</td>
<td>80</td>
<td>65</td>
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<td>4/16</td>
<td>86</td>
<td>70</td>
<td>12</td>
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<td>4/17</td>
<td>129</td>
<td>58</td>
<td>11</td>
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<tr>
<td>4/18</td>
<td>84</td>
<td></td>
<td>16.3</td>
</tr>
</tbody>
</table>

AASHTO re:source PSP

AI Roofing PSP
Cleveland Open Cup (COC) Flash Point Test

Filling marks
Reference for flame size
• **Mistakes and Misconceptions**
  
  • Keeping the flash cups clean is more important than many technicians appreciate.
• **Mistakes and Misconceptions**
  
  • Keeping the flash cups clean
    
    • If the fill mark is obscured by carbonized binder, accurately filling the cup becomes difficult
    
    • Under-filled or over-filled cups result in variability in test results
      
      • If the cup is under-filled, it may take longer for the volatiles to accumulate and flash
      
      • If the cup is over-filled, the opposite results may occur
        
        • Also a potential safety hazard due to sample overflow from thermal expansion
Mistakes and Misconceptions

Practical safety interpretation

- New requirement in ASTM D92 Section 11.1.13
  - “When the apparatus has cooled down to a safe handling temperature, less than 60°C (140°F), remove the test cup and clean the test cup and apparatus as recommended by the manufacturer.”

- As interpreted by our technicians, this requirement is impractical.
  - Technicians performed the test and recorded a flash point of 314°C and immediately turned off the apparatus.
  - It took the sample 55 minutes to cool to the “safe handling” temperature.
  - Technicians had to reheat the cup to a temperature considered “unsafe” in order to empty it.
Rolling Thin Film Oven Aging
Mistakes and Misconceptions

- Air nozzle is easy to knock out of alignment
  - Lessens air flow during test which leads to lower aging
• Mistakes and Misconceptions
  • Should use heated scraping tools for RTFO bottles
    • Not a mistake, per se, but a good practice
• **Mistakes and Misconceptions**
  - Tilting the RTFO is needed for highly-modified asphalt binders to prevent asphalt binder from creeping out
    - Had been a problem when PG system first started
    - Bottle lip should help
    - Now mostly only done for GTR-modified asphalt binders
      - May need modification to procedure
      - Attempt to stay within levelness requirement (within 1 degree of horizontal)
Mistakes and Misconceptions

RTFO mimics the aging seen in mixing plants

- Standard provides “…a means for conditioning asphalt binders to simulate the hardening that occurs during the mixing and compaction of hot-mix asphalt (HMA) {4.1}. The changes in PG grading properties that occur during this procedure approximate the changes that occur in a batch plant operating at approximately 150°C.”
- Not “exactly reproduces”, but “approximates”
- Dependent on plant type (drum, batch), operating temperature, mixing time, storage time
- WMA
Performance Graded (PG) Asphalt Binder

Specified Asphalt Binder Based on Climatic Conditions

PG 58-28

- Performance Grade
- Max pavement Design temp
- Min pavement Design temp
Mistakes and Misconceptions

- The high temperature grade is based on the 7-day maximum pavement temperature
  - That used to be correct, but now it is based on a degree day-damage concept
Most damage is at many hours of high temperatures, not the highest temperatures.
Same SHRP PG, but different temperature profile

Temperatures of Two Sites With Same SHRP PG

Ala Mohseni, 2003
**Mistakes and Misconceptions**

- If an asphalt binder grade is chosen that satisfies 98% reliability, there is only a 2% chance in any given year that it will fail.
  - “Reliability” refers to temperature reliability; it has nothing to do with actual performance of the asphalt binder.
  - Won’t necessarily fail because properties exceed minimum values.
  - Mixture design, thickness design, construction and traffic have lots to do with performance.
    - Thermal cracking may be exception.
• **Mistakes and Misconceptions**
  
  • Not a mistake, but a question...
  
  • What should you do if your rotational viscosity sample chamber cools too much to allow you to ‘Gently lower the spindle into the asphalt binder...’?
  
  • Is it permissible to reheat the binder in the viscometer during part of the 30 minute preheat period? If not, how do we handle this?

  • Storing the sample in an oven while waiting for a free RV would be much more damaging to the sample than reheating from a cold state.
• **Mistakes and Misconceptions**
  
  • Rotational viscosity measurements can be used with viscosity-temperature charts to provide mixing and compaction temperatures
    • Correct, but...
    • ...not for modified asphalt binders
      • Equiviscous procedure often results in temperatures that are too high for modified asphalt binders
    • ...not for use in the field
      • Laboratory temperatures to ensure that proper coating and asphalt absorption occurs
The Asphalt Institute recommends two options:

• follow the recommendation of the supplier, as many suppliers have determined mixing and compaction temperatures for their individual products that have proven to be appropriate; or

• conduct testing using one of the two procedures recommended by NCHRP Report 648, *Mixing and Compaction Temperatures of Asphalt Binders in Hot-Mix Asphalt*
  - DSR Phase Angle or DSR Steady Shear Flow procedures
• **Mistakes and Misconceptions**
  • Before you do a reference fluid check on the DSR, you need to verify temperature
    • There can be no confidence in a reference fluid result without confidence in temperature.
• **Mistakes and Misconceptions**
  
  • Trimming into the side of the 8-mm DSR because the trimming tool is not heated enough
    
    • Let the heat of the trimming tool do the work
• **Mistakes and Misconceptions**
  • You cannot test asphalt binders that have particulates greater than 250 microns
    • You can test the asphalt binder, but the larger particle size may interact with the thickness of the sample
      • affects the assumption of linear viscoelastic behavior
      • Increases testing variability
• **Mistakes and Misconceptions**
  
  • When filling the PAV pans, the asphalt binder should be heated enough so that it flows to fill the pan.
  
  • Be careful stacking pans after use before putting in parts washer. Can result in pans getting stuck and deformed.
    • Key is to create a uniform thickness of asphalt binder that is exposed to accelerated aging
    • 50 grams of asphalt binder will create an approximate uniform thickness of 3.1 mm
**Mistakes and Misconceptions**

- Degassing of PAV-aged residue is always necessary
  - Was not included in the original PG system
  - Added when the Direct Tension Test was being refined
    - More bubbles lead to possible earlier failure in DT test
  - Studies have shown little statistical difference in test results of samples that were degassed versus those that were not degassed
- But...
  - Repeatability may be improved by degassing
  - Like steric hardening, it might have an effect for some asphalt binders
• **Mistakes and Misconceptions**
  • PAV aging represents approximately 5-15 years of in-service aging
    • Original message during training in 1990s
    • Now consider that PAV aging may be much less than previously thought
      • Part of reasoning for research considering more severe PAV aging
• **Mistakes and Misconceptions**
  
  • There is no need to “calibrate” the BBR every time you use it.
  
  • It only needs to be done if the verification procedures fail (verifying loads, zero position, etc.)
• **Mistakes and Misconceptions**
  • BBR trimming tool not hot enough
    • Can cause divots in beam
  • Finding the point where the BBR shaft is just touching the beam can be tricky.
    • Not in contact can lead to errors in stiffness versus time plot.
  • Make sure beam is seated against back supports
Mistakes and Misconceptions

- Reported values for Stiffness and m-value are actually measured
  - Both are calculated from a polynomial regression
Common Misconceptions and Mistakes in Asphalt Binder Testing:
Resources (other than AASHTO re:source)
• Standards within United States
  • AASHTO
    • Standard Specifications for Transportation Materials and Methods of Sampling and Testing, and AASHTO Provisional Standards
  • ASTM
    • Volume 04.03 (Road and Paving Materials; Vehicle-Pavement Systems)
    • Volume 04.04 (Roofing and Waterproofing)
    • Volume 05.01 (Petroleum Products, Liquid Fuels, and Lubricants (I))
    • Volume 14.03 (Sensory Evaluation; Temperature Measurement; Language Services and Products)
Asphalt Institute References

Asphalt Binder Testing

The ASPHALT BINDER HANDBOOK

MS-26
Third Edition

1st Edition
• Specification Databases
  o US Paving Grade Asphalt Binder
  o US Asphalt Emulsions
  o Canadian Provinces

• asphaltinstitute.org
• Started in 2012
  • Means of providing some (hopefully) light reading for asphalt binder technicians

• Key Components
  • Feature Article
  • Tech Tip
  • Ask Mike
  • Technician Spotlight

• Mail Chimp Distribution
• Access to information and emerging technologies
• Technical community comprised of individuals from all parts of the asphalt industry
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