DSR-PAV Test Improvement
2Q17 Status Update

AI TAC TF members:
Pavel Kriz (Imperial Oil/ExxonMobil)
Gerry Reinke (Mathy)
Mike Anderson (Asphalt Institute)
Wes Cooper (Asphalt Institute)
Dave Anderson (Consultant)

Expert Task Group Meeting, Ames IA
May 3, 2017
Case for Action: DSR-PAV Is Too Variable

One Sample

Multiple Samples

SQC Data

Production Data

Gauge R&R

Reproducibility, 40.2%

2728 kPa

6163 kPa

DSR-PAV, kPa

Observation

Production Data

UCL=5507

LCL=3190

Observation

\( \bar{X} = 4349 \)

SQC Data

57%

Unacceptable

Acceptable

Acceptable w/limits
Connect from September ETG

- Initial study presented - indicated test strain & plate size as likely contributors to DSR-PAV variability
- TF formed within AI TAC
- Labs volunteered to participate in RR to collect data for the study

17 labs participating in round robin

<p>| | | |</p>
<table>
<thead>
<tr>
<th></th>
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</thead>
<tbody>
<tr>
<td>Imperial Oil/ExxonMobil</td>
<td>Asphalt Institute</td>
<td>MTO</td>
</tr>
<tr>
<td>Holly Frontier Corp.</td>
<td>PRI Asphalt Technologies</td>
<td>Delaware DOT</td>
</tr>
<tr>
<td>Flint Hills Resources (3 labs)</td>
<td>Road Science</td>
<td>Washington State DOT</td>
</tr>
<tr>
<td>Paragon Technical Services</td>
<td>Kraton Polymers</td>
<td></td>
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<td>Jebro</td>
<td>Pike Industries Inc.</td>
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<tr>
<td>MTE Services</td>
<td>Alon Asphalt</td>
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</table>

Thank you all for volunteering!!!
Development since the last ETG

1. TF expanded scope \(\rightarrow\) DSR conditioning time
   1. Stage 1 to determine appropriate conditioning time
   2. Stage 2 to test effect of strain & plate size on variability

2. Asphalt Institute developed & distributed 2 PAV asphalt samples (NC-B, NC-D)

3. Test protocol developed & shared
   – Included a diverse set of DSRs & T-control systems to ensure broad applicability
   – Standardized sample preparation & loading
   – Developed excel sheet to collect and analyze data for stage 1
   – 2 PAV aged asphalts & **Cannon standard** tested

### Instruments in round robin

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>DSR Type</th>
<th>Count</th>
</tr>
</thead>
<tbody>
<tr>
<td>Anton Paar</td>
<td>101, 102, SmartPave</td>
<td>4</td>
</tr>
<tr>
<td>Thermal Analysis</td>
<td>AR500</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>AR2000, AR2000ex</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>DHR-2</td>
<td>2</td>
</tr>
<tr>
<td></td>
<td>ARES (rheometrics)</td>
<td>1</td>
</tr>
<tr>
<td>Malvern (Bohlin)</td>
<td>DSR II</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>CVO-100</td>
<td>2</td>
</tr>
<tr>
<td></td>
<td>Kinexus</td>
<td>2</td>
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</tbody>
</table>
Test Protocol for Stage I

Test Protocol Highlights

- Samples aliquoted to small tins by AI & distributed
- Standardized approach on sample heating & loading
- Cooling from 46 °C to target temperature left to instrument control system
- Dynamic data collected in 30 s interval during cooling & isothermal portion for 30 mins in total

Test Setups Used in DoE

<table>
<thead>
<tr>
<th>Sample</th>
<th>Plate size, mm</th>
<th>Temperature, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cannon</td>
<td>8</td>
<td>13</td>
</tr>
<tr>
<td>Cannon</td>
<td>25</td>
<td>13</td>
</tr>
<tr>
<td>Cannon</td>
<td>8</td>
<td>25</td>
</tr>
<tr>
<td>Cannon</td>
<td>25</td>
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</tr>
<tr>
<td>NC-B</td>
<td>8</td>
<td>13</td>
</tr>
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<td>25</td>
<td>13</td>
</tr>
<tr>
<td>NC-B</td>
<td>8</td>
<td>19</td>
</tr>
<tr>
<td>NC-B</td>
<td>25</td>
<td>19</td>
</tr>
<tr>
<td>NC-D</td>
<td>8</td>
<td>19</td>
</tr>
<tr>
<td>NC-D</td>
<td>25</td>
<td>19</td>
</tr>
<tr>
<td>NC-D</td>
<td>8</td>
<td>25</td>
</tr>
<tr>
<td>NC-D</td>
<td>25</td>
<td>25</td>
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Stage I – Finding Conditioning Time

Objective: Verify that current 10 min time is appropriate across variety of systems

Analysis: Complex approach – **Dave Anderson** is going to explain later

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**Stage I – Finding Conditioning Time**

Objective: Verify that current 10 min time is appropriate across variety of systems

Analysis: Complex approach – **Dave Anderson** is going to explain later
Initial Stage 1
Data Analysis
(8* out of 17 labs)

*Labs 13 & 15 (same instrument type) excluded from analysis, >3 sigma.
Cooling to Temp. is Relatively Fast

<table>
<thead>
<tr>
<th>Time (from beginning of cooling), min</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 10 20 30 40 50 60</td>
</tr>
</tbody>
</table>

\[ |G^*| \sin \delta, \text{kPa} \]

- Manufacturer 1
- Manufacturer 2
- Manufacturer 3
DSRs Differ in time=0 Determination

Test at different thermal history

Manufacturer 2 tests about 6 minutes sooner

<table>
<thead>
<tr>
<th>G*</th>
<th>sin delta, kPa</th>
</tr>
</thead>
</table>

Manufacturer 1

Manufacturer 2

Manufacturer 3
NC-B Asphalt, Little Hardening

13 °C

19 °C

| Time After Reaching Temperature within 0.1°C, min |
|-----------------------------------------------|-----------------------------------------------|
| Manufacturer 1                             | Manufacturer 2                             |
| Manufacturer 3                             |                                               |

6 kPa/min

2.2 kPa/min

2.5 kPa/min

0.9 kPa/min

| Time After Reaching Temperature within 0.1°C, min |
|-----------------------------------------------|-----------------------------------------------|
| Manufacturer 1                             | Manufacturer 2                             |
| Manufacturer 3                             |                                               |

8 mm

25 mm
NC-D Asphalt, Some Hardening

19 °C

Time After Reaching Temperature within 0.1 °C, min

8 mm

|G*|sin delta, kPa

- Manufacturer 1
- Manufacturer 2
- Manufacturer 3
- 10 min

13 kPa/min

25 mm

19 °C

Time After Reaching Temperature within 0.1 °C, min

8 mm

|G*|sin delta, kPa

- Manufacturer 1
- Manufacturer 2
- Manufacturer 3
- 10 min

9.4 kPa/min

25 mm

|G*|sin delta, kPa

- Manufacturer 1
- Manufacturer 2
- Manufacturer 3
- 10 min

3.6 kPa/min

25 °C

Time After Reaching Temperature within 0.1 °C, min

8 mm

|G*|sin delta, kPa

- Manufacturer 1
- Manufacturer 2
- Manufacturer 3
- 10 min

5.2 kPa/min
Early Observation

1. Older instruments challenged with experiment
2. Instruments differ in approach to conditioning time
3. Conditioning time for 25mm plates shorter than for 8mm
4. Hardening/conditioning time is not a major factor in variability
5. Cannon standard data are much less variable than asphalt data
Current Status & Next Steps

• 10 out of 17 labs provided results for stage 1
• TF agreed that sufficient data provided for phase 1, ready for stage 2 → use 10 min

Next Steps

1. Communicate labs with conditioning times for stage 2
2. Execute stage 2 testing
3. Analyze data and propose updates to T315
4. Report at next ETG