Statistical Approach to DSR-PAV Test Improvement

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ISAP 2016 Symposium, Jackson, WY
July 19, 2016
Select Correct Glasses for Observation

“The observation is only as good as the measurement method”

Poor Test Resolution $\Rightarrow$ Increased Cost

- Representation (Pass or Fail?)
- Feedstock management
- Production/quality control
- Logistics
Case for Action: DSR-PAV Is Too Variable

6163 kPa

2728 kPa

One Sample

SQC Data

Reproducibility, 40.2%

DSR-PAV, kPa

Gauge R&R

57%

60%

50%

40%

30%

20%

10%

0%

Unacceptable

Acceptable w/limits

Acceptable
Approach to DSR-PAV Variability Improvement

- Sample RTFO & PAV aging shown insignificant to DSR-PAV variability
- Study focused on DSR test improvement

Focus of this presentation

1. Standardize within T315
   - Sample preparation
     • Direct pour
     • Plates at 46 °C
   2. Trimming & gap setting
     • Plates at 46 °C
   3. Conditioning
     • Fixed cooling rate
     • Fixed wait time

   - Review setting in T315 for contributions to variability
   - Test variables in Statistical Design of Experiment
**Statistical Design of Experiment (DoE)**

- DoE = a powerful approach to maximize output at minimized effort
- A number of possibly interdependent factors or variables is studied
- The tests are strategically selected to represent each factor equally

Following variables were standardized prior to applying DoE:

- Large volume of QC sample PAV residue (PG 64-22, 25 °C test T.)
- Modern, Peltier cooled, base DSR instrument
- Sample aliquot, container size, oven preheat (temperature & time)
- Loading, trimming, gap temperature = 46 °C
- Trimming technique & tool
- Cooling rate to test temperature, isothermal time prior to test
DoE Factors and Levels

<table>
<thead>
<tr>
<th>Factor</th>
<th>+1</th>
<th>-1</th>
<th>Reason</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermal Geometry</td>
<td>Direct Transfer</td>
<td>Mold</td>
<td>Use of molds, 46 °C loading T</td>
</tr>
<tr>
<td>Geometry</td>
<td>8 mm PP*</td>
<td>25 mm PP*</td>
<td>Simple shear, trimming</td>
</tr>
<tr>
<td></td>
<td>2 mm gap</td>
<td>1 mm gap</td>
<td></td>
</tr>
<tr>
<td>Strain (%)</td>
<td>0.1</td>
<td>1</td>
<td>Linear viscoelasticity</td>
</tr>
<tr>
<td>Sample</td>
<td>Naphthenic</td>
<td>Waxy</td>
<td>Hardening tendency</td>
</tr>
<tr>
<td>Operator</td>
<td>New</td>
<td>Experienced</td>
<td>Experience</td>
</tr>
</tbody>
</table>

- 5 factors at 2 levels total $2^5$ or 32 individual test settings
- Test matrix was generated and randomized using Minitab® software
- Each setting was repeated four times to calculate standard deviation
- Half design (16 settings) found to be statistically significant in identifying contributors to test variability

*Parallel plates
Each point represents a mean of half of the 64 total experiments.

Two geometries provide different results.
Strain = Major Factor Affecting Variability
• 8PP: modulus increases with strain likely due to edge effect
• Strain below 0.1 % desirable
High Test Strain & 8 mm Plates = Artifact of 1990s DSR Capability

1993 instrument min. torque

2008, 2014 instruments min. torque
Conclusions

1. DSR-PAV test is not able to distinguish quality easily
2. High test variability is partly driven by a test method
3. Lower strain & higher plate diameter-to-gap ratio is desirable

Recommendation:

1. Adopt 0.1% (or lower) strain and 25 mm PP for DSR-PAV test
2. Increase specification limit (e.g. to 6000 kPa) to ensure DSR (Original/RTFO) & BBR (m or S) are PG limiting specifications

Output:

• Improved asphalt production without impact to performance
Question & Comments?

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Appendix
Lean Six Sigma

- Lean Six Sigma offers a powerful approach to continuous improvement

- DMAIC approach & numerous tools ranging from brainstorming & mind mapping to design of experiments & statistical analysis were utilized
1. Wait Time = silicon mold time standardized at 10 minutes
2. Gap Temperature = Sample load, gap setting, trimming done at 46 °C
3. Direct Transfer = molds discontinued, hot asphalt transferred to plates

Standardizing Sample Management

Gradual Improvement

Improvements Were Not Sustained
1. No significant difference among 3 instruments ($n > 30$ datapoints)
2. Minor increase (sample dependent) due to hardening
   • 10-25 min wait time increased modulus by $\sim 5\%$