
Digital Paddle Viscometer (DPV) and Moisture Analyzer Balance (MAB)

Delmar R Salomon
Pavement Preservation Systems LLC

Anchorage, Alaska
October 7-9, 2013
Viscometers

Rotational viscosity

Saybolt Furol

Viscometer

ASTM D7496

AASHTO T-59

Rotational Paddle

ASTM D 7226
Emulsified Asphalt new proposed viscosity method

- Background and previous work on rotational viscosity method at different shear rate (2001–2004)

- ASTM D 7226 viscosity by rotational Paddle viscometer (2006 to present)
Effect of constant shear at 50 RPM and 50°C on emulsion viscosity

- Microstructure breakdown
- Steady-state viscosity
- Breaking of emulsion

Viscosity (mPa s) vs. Time (minutes)
Effect of constant shear at 50 RPM and 70 °C on emulsion viscosity

- Microstructure breakdown
- Steady-state viscosity
- Breaking of emulsion
CRS-2 Equilibrium Viscosity at 50, 100, 200 RPM and 50 °C, Spindle #21

- 50-200-21: 75 mPa s
- 50-50-21: 129 mPa s
- 50-100-21: 128 mPa s
Saybolt Viscometer

AASHTO T59
ASTM D 7496
Temperature probe (thermistor)

Paddle (SS)

Sample cup (SS)

ASTM D7226
SCOPE:

“Determine Precision statements for D7496 (Saybolt viscometer) and D7226 (Paddle Viscometer);

Actual participant laboratories submitting data

--17 labs for D7496 (ILS 431) –closed oct, 2009
___15 labs for D7226 (ILS 424)-closed oct, 2009
-14 labs for ILS 604 & 605 (closed Nov, 2010)
ASTM ILS Samples and Labs

- **ILS 604 & 605**
  - Sample CSS-1 at 25C
  - 2. Sample SS- at 25C
  - 3. Sample CRS-2 at 50C
  - 4. Sample CRS-2P at 50C; (14 LABS)

- **Cannon STANDARDS**
  - N44 measured at 25C
  - N415 measured at 50C
  - N1400 measured at 50C

- **ILS 424 & 431**
  - 17 labs for D7496
  - _15 labs for D7226
  - CSS-1 at 25C
  - 2. CRS-2 at 50C
  - 3. CMS-2 at 50C
  - 4. CRS-2P at 50C
  - 5. CRS-2LM at 50C
  - 6. HFRS-2 at 50C
  - S-600 Standard @ 50C
Participant laboratories submitting data (36% users; 50% producers; 14% independents)

1. Flint Hills Resources, Rosemount, MN
2. GECAN, Calgary, Alberta -CANADA
3. IOWA DOT, Ames, IA -
4. Idaho Transportation Department, Boise, ID -
5. Mariani Asphalt, Tampa, FL
6. Martin Asphalt Company, South Houston, TX
7. Minnesota DOT, Maplewood, MN -
8. PA Department of Transportation, Harrisburg, PA -
9. Paragon Technical Services Inc., Richland, MS -
10. Pounder Emulsions, Saskatoon, SK, CANADA
11. SEACO, Inc., Columbia, SC
12. TXDOT, Austin, TX
14. US Oil & Refining Company, Tacoma, WA
### Single operator precision

<table>
<thead>
<tr>
<th>Test Temperature</th>
<th>Viscosity</th>
<th>Repeatability</th>
</tr>
</thead>
<tbody>
<tr>
<td>°C (°F)</td>
<td>s</td>
<td>% of the mean</td>
</tr>
<tr>
<td>25 (77)</td>
<td>20 to 100</td>
<td>6.7</td>
</tr>
<tr>
<td>50 (122)</td>
<td>75 to 400</td>
<td>10.8</td>
</tr>
</tbody>
</table>

### Multi laboratory Precision

<table>
<thead>
<tr>
<th>Test Temperature</th>
<th>Viscosity</th>
<th>Repeatability</th>
</tr>
</thead>
<tbody>
<tr>
<td>°C (°F)</td>
<td>s</td>
<td>% of the mean</td>
</tr>
<tr>
<td>25 (77)</td>
<td>20 to 100</td>
<td>22</td>
</tr>
<tr>
<td>50 (122)</td>
<td>75 to 400</td>
<td>88</td>
</tr>
</tbody>
</table>

### Single Operator Precision

<table>
<thead>
<tr>
<th>Test Temperature</th>
<th>Viscosity</th>
<th>Repeatability</th>
</tr>
</thead>
<tbody>
<tr>
<td>°C (°F)</td>
<td>mPa.s</td>
<td>% of the mean</td>
</tr>
<tr>
<td>25 (77)</td>
<td>25 to 200</td>
<td>8.2</td>
</tr>
<tr>
<td>50 (122)</td>
<td>100 to 1000</td>
<td>12.9</td>
</tr>
</tbody>
</table>

### Multi laboratory Precision

<table>
<thead>
<tr>
<th>Test Temperature</th>
<th>Viscosity</th>
<th>Repeatability</th>
</tr>
</thead>
<tbody>
<tr>
<td>°C (°F)</td>
<td>mPa.s</td>
<td>% of the mean</td>
</tr>
<tr>
<td>25 (77)</td>
<td>25 to 200</td>
<td>22</td>
</tr>
<tr>
<td>50 (122)</td>
<td>100 to 1000</td>
<td>64</td>
</tr>
</tbody>
</table>
Reproducibility: multilaboratory precision

Repeatability: single operator precision

- Saybolt
- DPV
Emulsified asphalt shall conform to the requirements prescribed in Table 1 or Table 2. If no table is specified, default is Table 1.

ASTM D977 - 2013 - Standard Specification for Emulsified Asphalt

ASTM D2397 –2012 - Standard Specification for Cationic Emulsified Asphalt (under Main Committee Ballot)
New Developments on Residue Recovery
Moisture Analyzer Balance = MAB

<table>
<thead>
<tr>
<th>Designation</th>
<th>Agency</th>
<th>Test Method Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>(A) D6997</td>
<td>ASTM</td>
<td>Distillation @ 260°C</td>
</tr>
<tr>
<td>(B) D7403</td>
<td>ASTM</td>
<td>Vacuum Distillation @ 135°C</td>
</tr>
<tr>
<td>(C) D6934-08</td>
<td>ASTM</td>
<td>Oven Evaporation</td>
</tr>
<tr>
<td>(D) Various</td>
<td>Various</td>
<td>Distillation @ 204°C (Usually modified)</td>
</tr>
<tr>
<td>(E) Various</td>
<td>Various</td>
<td>Distillation @ 177°C (Usually modified)</td>
</tr>
<tr>
<td>(F) ARIZ 504</td>
<td>Arizona DOT</td>
<td>Vacuum Recovery of Residue (115°C)</td>
</tr>
<tr>
<td>(G) CT 331</td>
<td>CalTrans</td>
<td>Oven Evaporation (Latex Modified AE)</td>
</tr>
<tr>
<td>(H) MDOT 904</td>
<td>Michigan DOT</td>
<td>Oven Evaporation @ 60°C, 24h, glass plate</td>
</tr>
<tr>
<td>(I) MDOT 904</td>
<td>Michigan DOT</td>
<td>Oven Evaporation @ ambient, 3 days, glass plate</td>
</tr>
</tbody>
</table>

The residue obtained from this test method may also be subjected to rheological characterizations.
Emulsified Asphalt new/proposed Tests for selected emulsified asphalt properties

- ASTM D 7497 practice residue by low temp evaporative technique
- DXXXX–YY Proposed practice developed at TXDOT (silicone mat with the emulsified asphalt into a 60ºC +/- 2ºC forced draft oven for 6 hours +/- 15 min)
$y = 1.0191x - 0.0162$

$R^2 = 0.96$
More direct recovery in a silicone mold (~22 mm diameter)

First experiments with silicone mold carried out at Idaho Transportation Department (2010)
Emulsion sample
In the silicone mold
Residue to DSR
Silicone mold

Emulsion
Sample placed into silicone mold

Recovery process on MAB

Recovery on MAB

Recovered sample
Moisture Analyzer Balance = MAB

2006

2010
MAB recent work (not using DSR mold directly)

2013

- Tests are run at 163° C for non polymer modified emulsions (100° C for polymer modified)

- A sample of 4g (+/-0.1 g) was used until change in weight of emulsion was less that 1 mg/ 140 seconds.
Emulsion recovery using Moisture Analyzer

- Immediately following recovery, the residue is transferred to a mold for testing with a DSR
Preliminary, high temperature, PG classification of the emulsified asphalt residue can be made using the combined MAB-DSR procedure.

Further details on the combined MAB-DSR performed at the University of Texas (Austin) with Dr. Amit Bhasin and Dr. Arash Motamed and to be presented at 2014 TRB annual meeting.

delmar@technopave.com
Publications and On-Going Research

- **NCHRP Project 09-50**: Performance-Related Specifications for Asphaltic Binders Used in Preservation Surface Treatments (from a Research Problem statement submitted by TRB AFK20 Committee: “Characteristics of Asphalt Materials”)

- **Manual for Emulsion-Based Chip Seals for Pavement Preservation- NCHRP #14-7 (end: 2-13-2009)**

- **MAB-DSR Procedure**: Assessment of the Performance of a Moisture Analyzer Balance (MAB) to Obtain the Residue of an Emulsified Asphalt using a Dynamic Shear Rheometer (DSR) Silicone Mold and Determining its Rheological Properties. World Congress on Emulsions, October, 2010

- **Asphalt Emulsion Technology, TRB Circular, EC102, August, 2006**

- **Asphalt Emulsion Technology, TRB Circular, EC-122 Review of Asphalt Emulsion Residue Procedures, October, 2007**

**Basic Asphalt Emulsion Manual, AEMA & Asphalt Institute** (see [www.aema.org](http://www.aema.org))

[www.pavementpreservationsystems.com](http://www.pavementpreservationsystems.com) for publications on emulsified asphalt