Rocky Mountain West Pavement Preservation Partnership Annual Meeting

New Test Method for Viscosity and Development of a Fast Residue Recovery Method for Emulsified Asphalts:

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

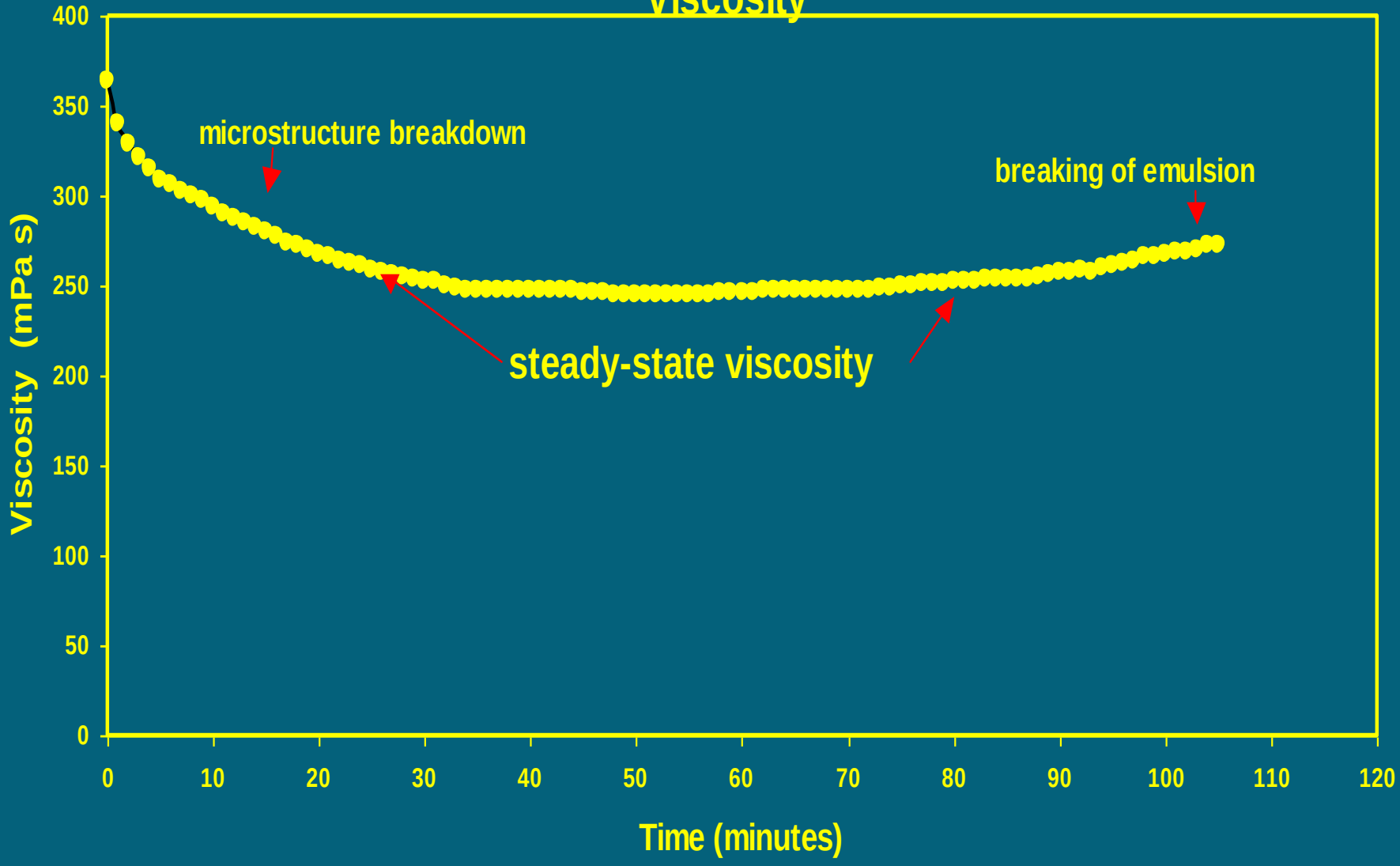
ASTM D 7226

Emulsified Asphalt new proposed viscosity method

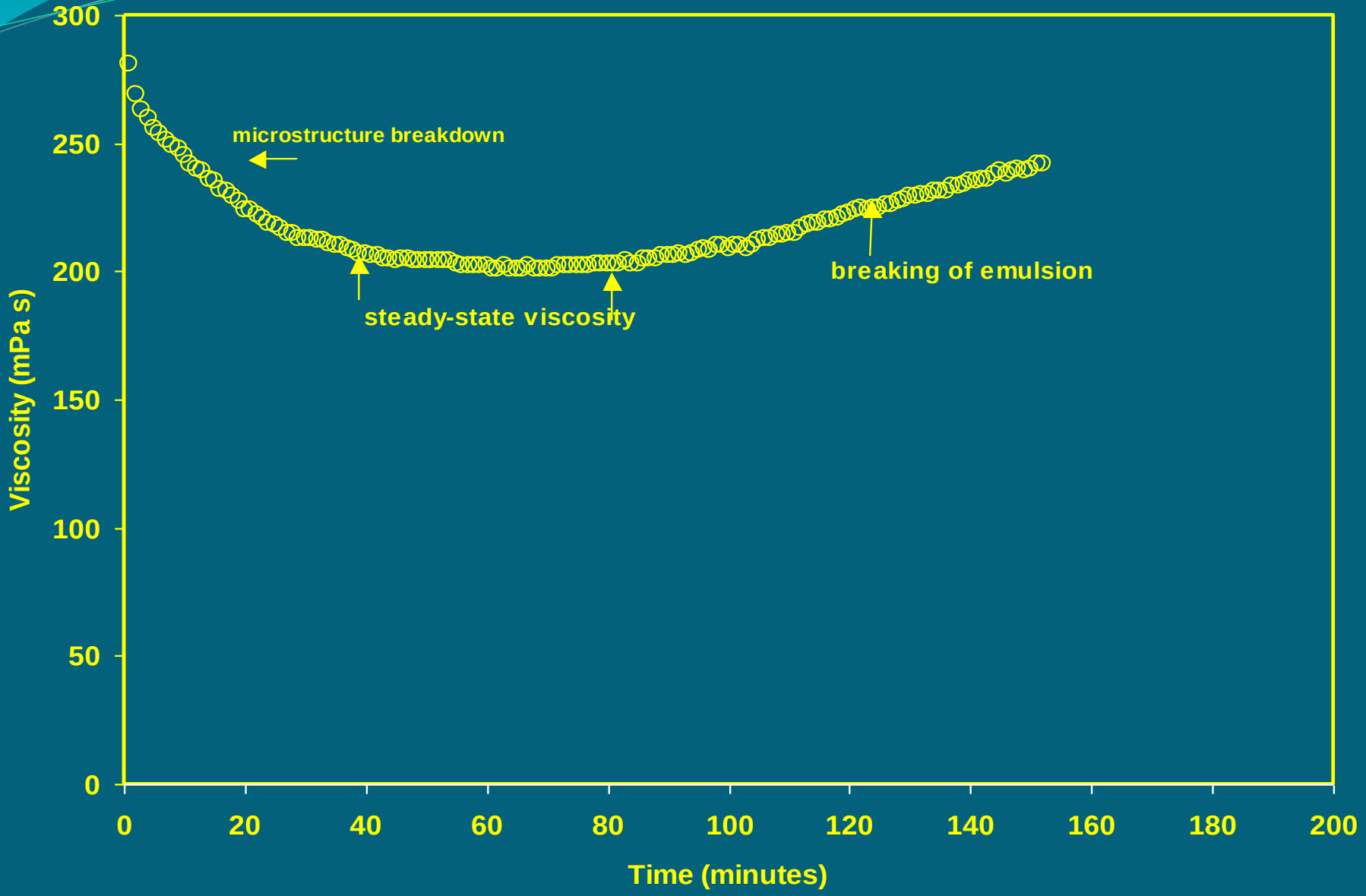
- Background and previous work on rotational viscosity method at different shear rate (2001-2004) Presented to ASTM/2002-2003

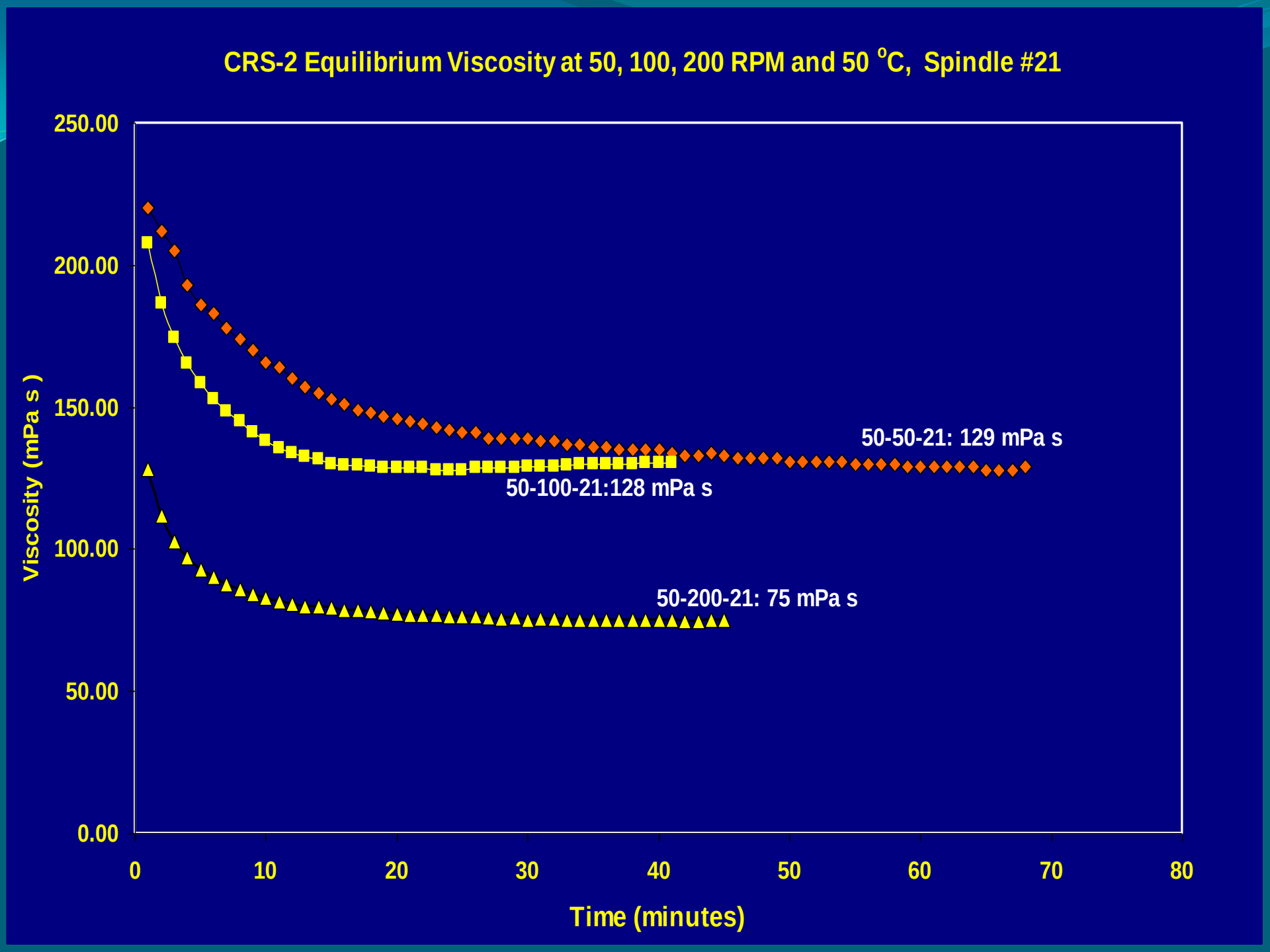
- ASTM D 7226 viscosity by rotational Paddle viscometer (2006 to present)

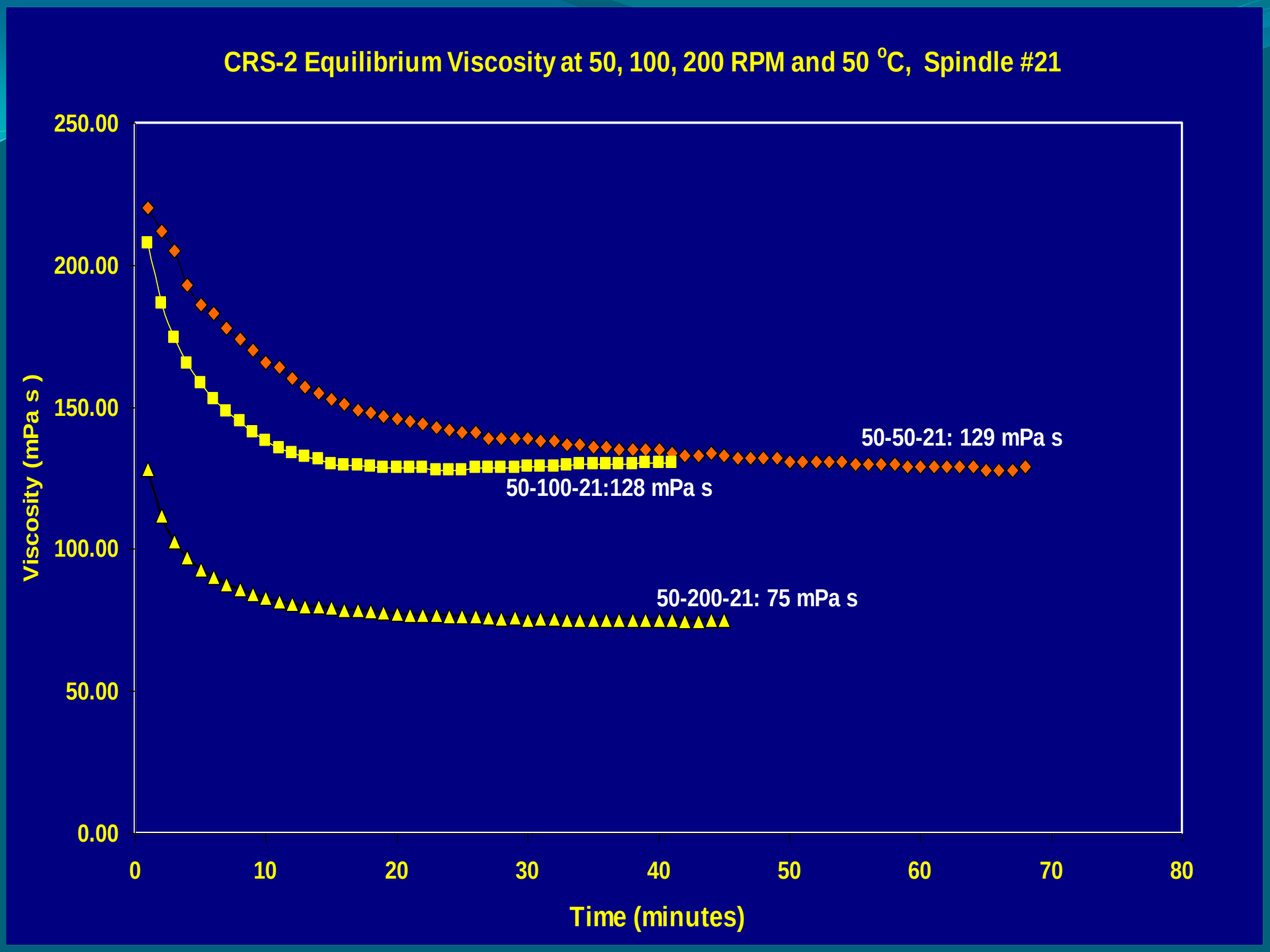
Effect of constant shear at 50 RPM and 50°C on emulsion viscosity



Effect of constant shear at 50 RPM and 70 °C on emulsion viscosity




CRS-2 Equilibrium Viscosity at 50, 100, 200 RPM and 50 °C, Spindle #21
250.00
200.00
150.00
100.00
50.00
0.00
Viscosity (mPa s)
0
10
20
30
40
50
60
70
80
Time (minutes)
50-50-21: 129 mPa s
50-100-21:128 mPa s
50-200-21: 75 mPa s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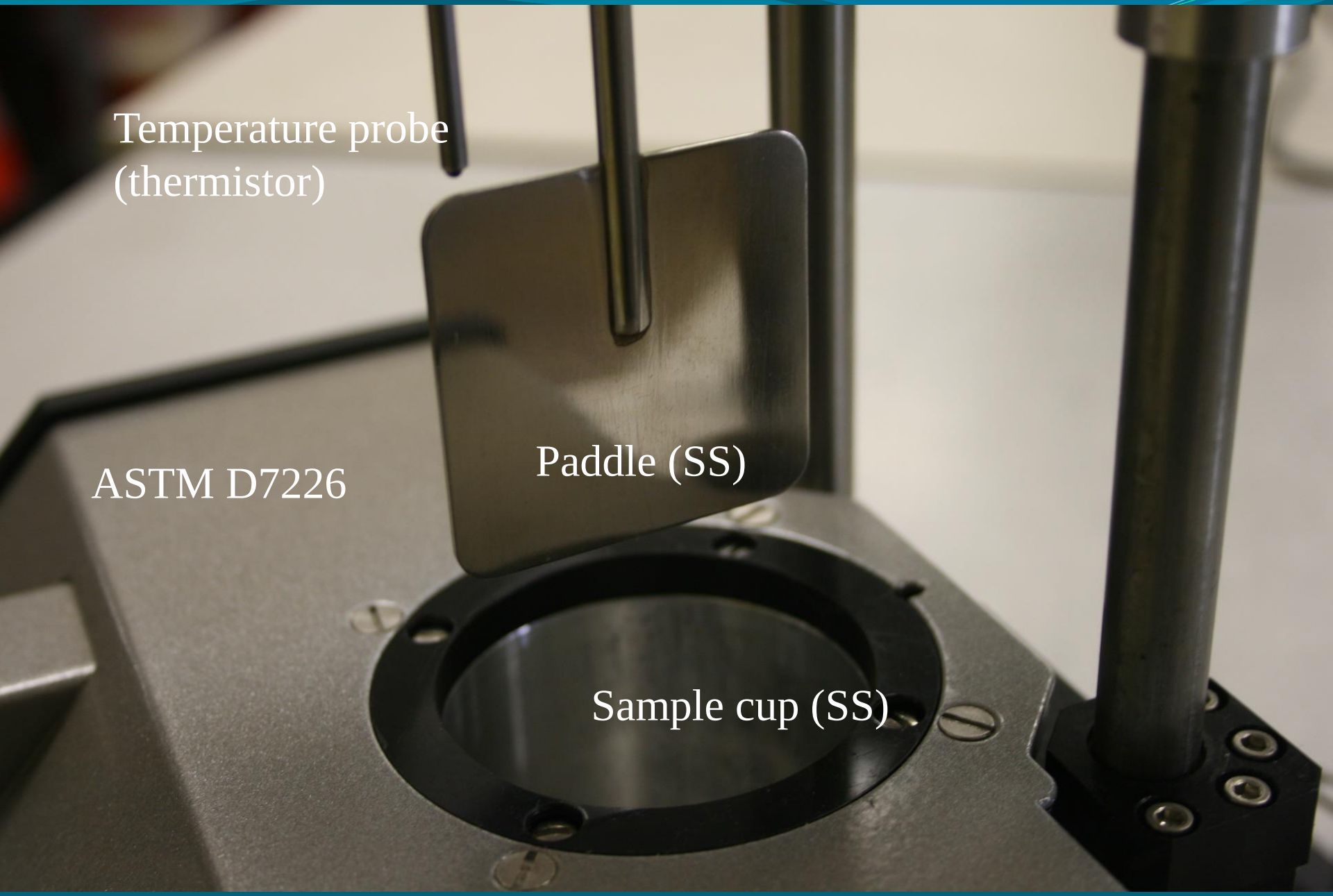
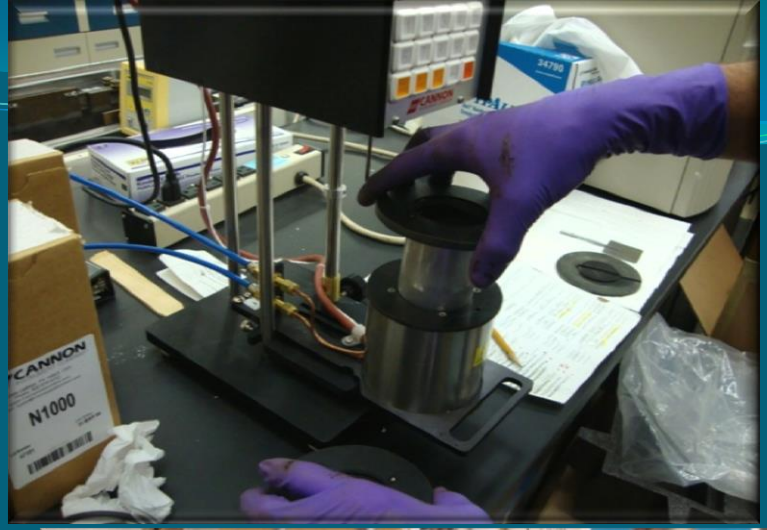
50-200-21: 75 mPa s


AASHTO T59
ASTM D 7496
Saybolt Viscometer
HI-INPUT
LO-INPUT


Temperature probe
(thermistor)
Paddle (SS)
ASTM D7226
Sample cup (SS)


DIGITAL PADDLE VISCOMETER
CANNON
N4000


CANNON
N1000


Status:Acquiring 2/3
Temp: 25.02°C 25°C
Time: 02:06 00:00
Visc: 78.72 cP
DIGITAL PADDLE VISCOMETER

Summary ASTM ILS 431,424 604, 605

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**
- 13. Terry Asphalt Materials, Inc., Hamilton, OH
- 14. US Oil & Refining Company, Tacoma, WA

ASTM D7496 - 11 Standard Test Method for Viscosity of Emulsified Asphalt by Saybolt Furol Viscometer

Single operator precision

Test Temperature °C (°F)	Viscosity s	Repeatability % of the mean
25 (77)	20 to 100	6.7
50 (122)	75 to 400	10.8

Multi laboratory Precision

Test Temperature °C (°F)	Viscosity s	Repeatability % of the mean
25 (77)	20 to 100	22
50 (122)	75 to 400	88

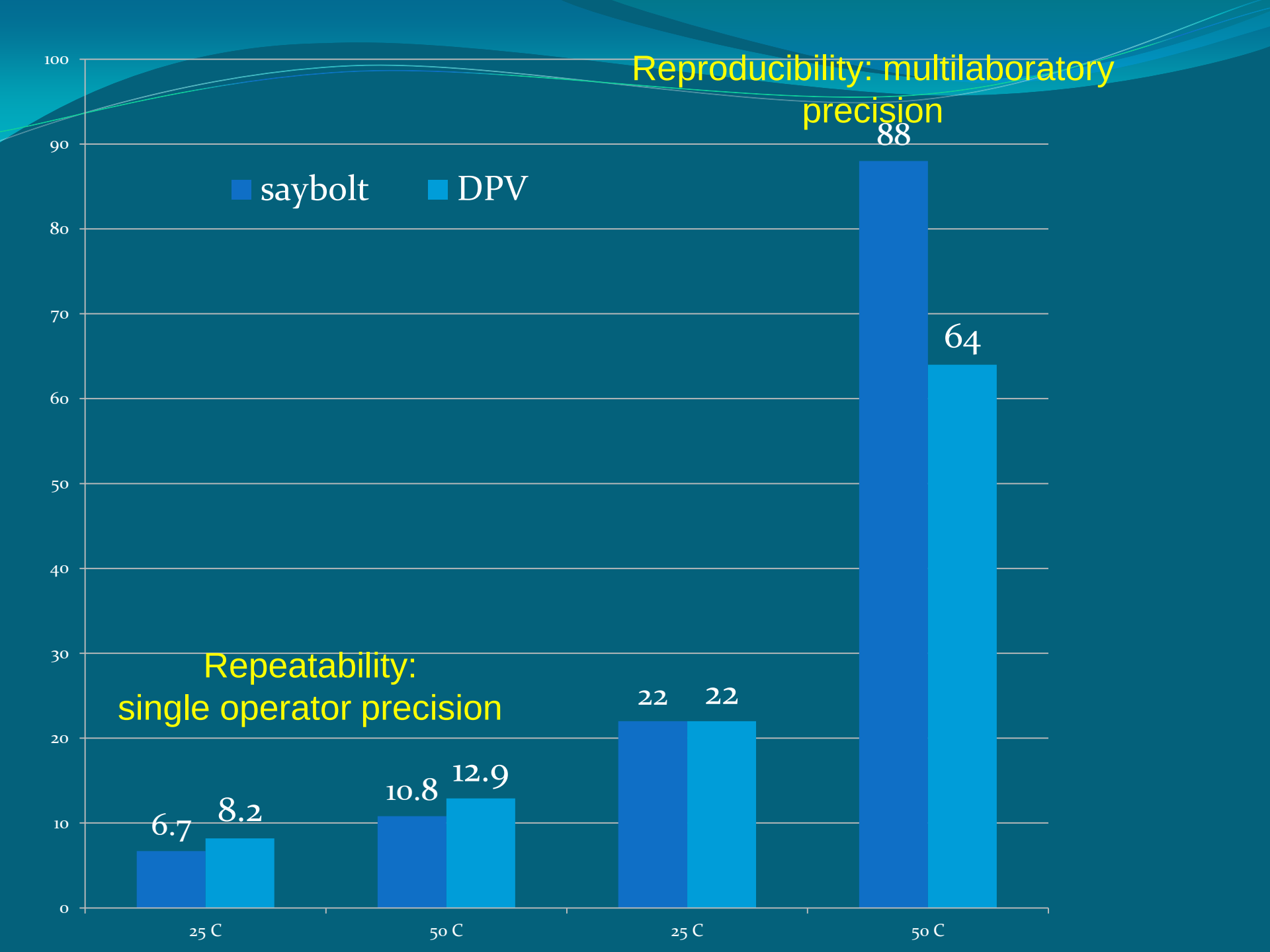
ASTM D7226 - 11 Standard Test Method for Determining the Viscosity of Emulsified Asphalts Using a Rotational Paddle Viscometer

Single Operator Precision

Test Temperature °C (°F)	Viscosity mPa.s	Repeatability % of the mean
25 (77)	25 to 200	8.2
50 (122)	100 to 1000	12.9

Multi laboratory Precision

Test Temperature °C (°F)	Viscosity mPa.s	Repeatability % of the mean
25 (77)	25 to 200	22
50 (122)	100 to 1000	64


Reproducibility: multilaboratory precision
saybolt
DPV
88
64
22
22
Repeatability:
single operator precision
6.7
8.2
10.8
12.9
100
90
80
70
60
50
40
30
20
10
0
25 C
50 C
25 C
50 C

Optional Table for viscosity Now in Standard

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 –20 12- Standard Specification for Cationic Emulsified Asphalt (under Main Committee Ballot)

New Developments on Residue Recovery

Moisture Analyzer Balance = MAB

ASTM D7404 - 07(2012) Standard Test Method for Determination of Emulsified Asphalt Residue by Moisture Analyzer

Recovery Methods

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

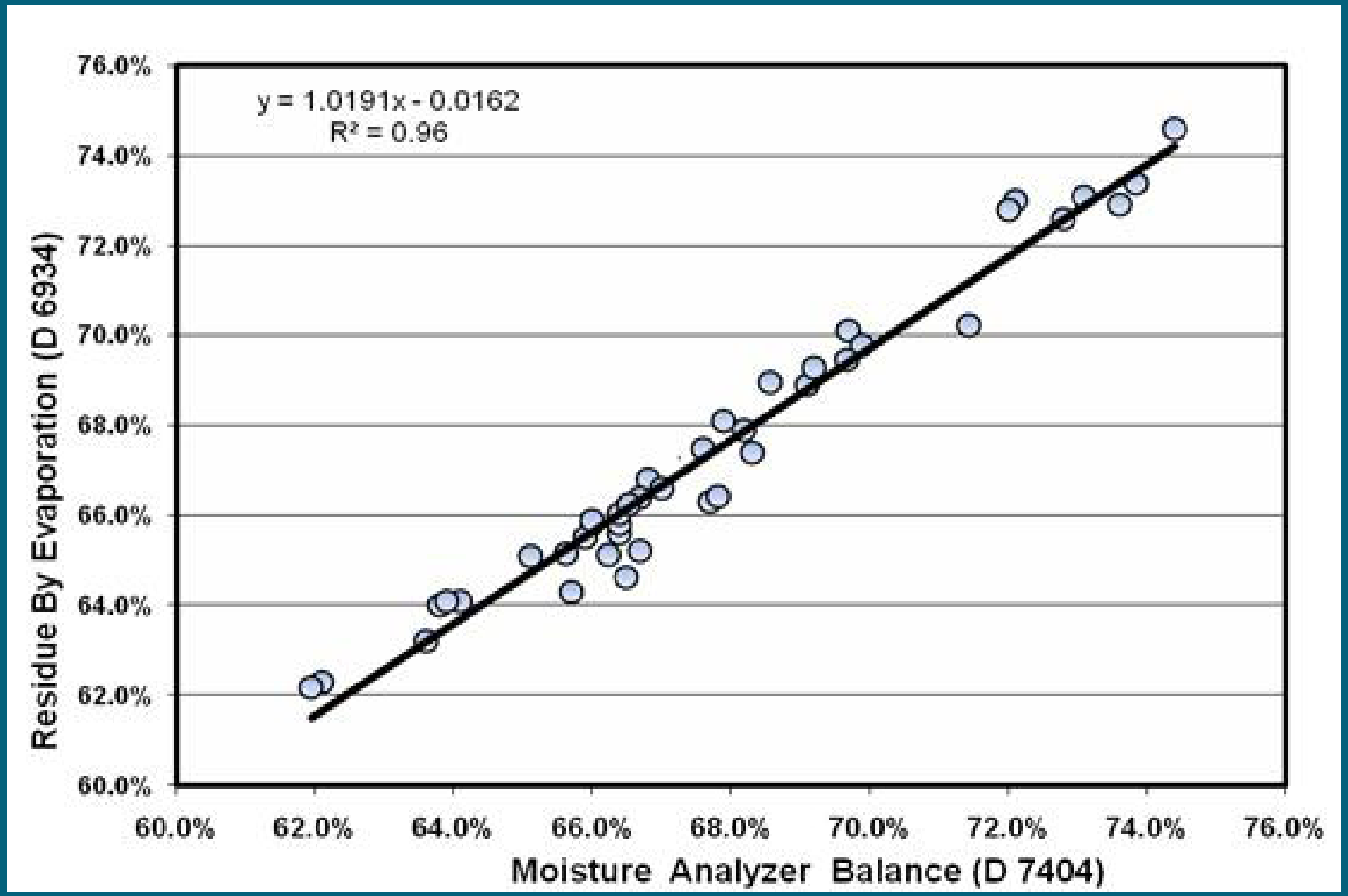
ASTM D7404 - 07(2012) Standard Test Method for Determination of Emulsified Asphalt Residue by Moisture Analyzer

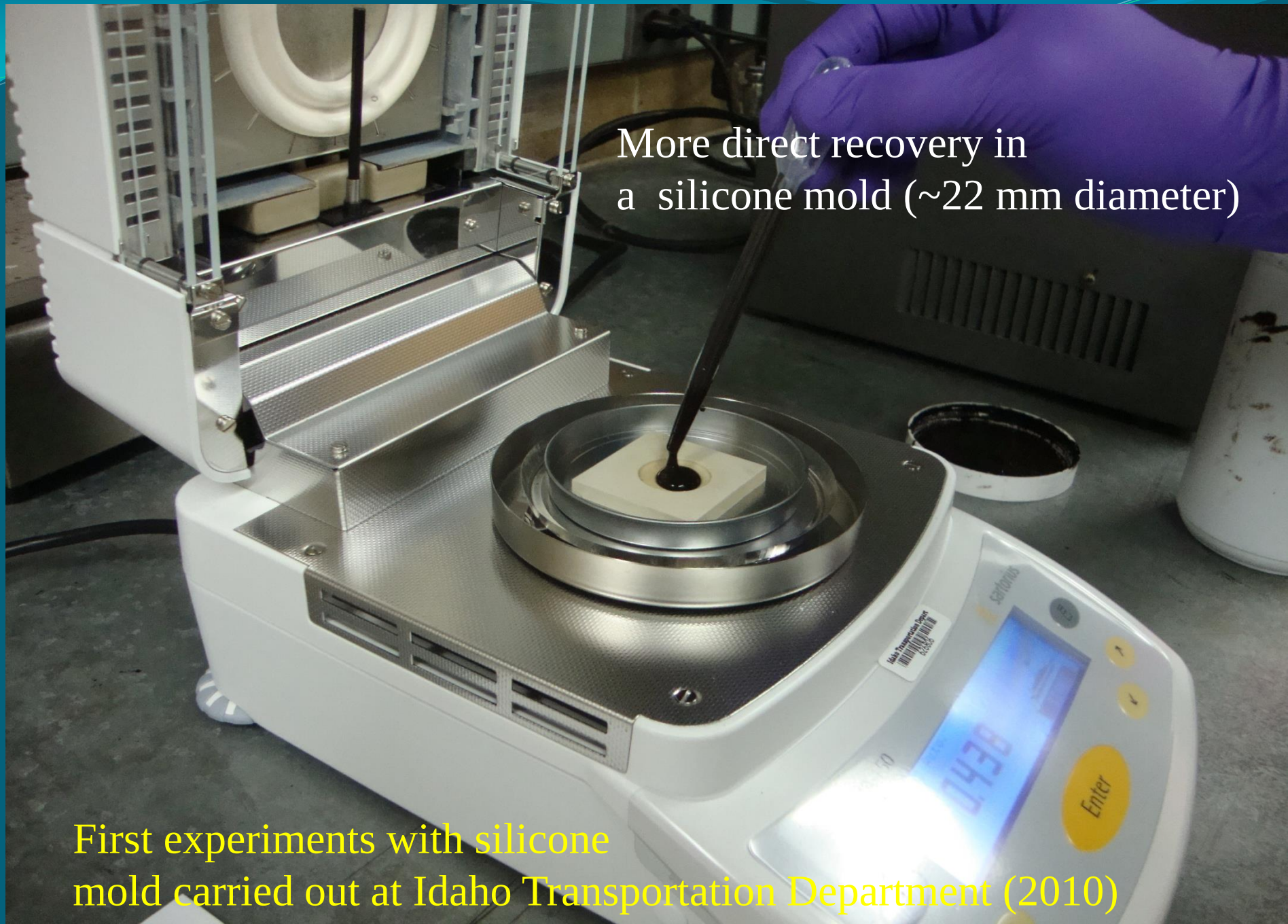
The residue obtained from this test method may also be subjected to rheological characterizations.

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)


FIRE


y = 1.0191x - 0.0162
R² = 0.96
76.0%
74.0%
72.0%
70.0%
68.0%
66.0%
64.0%
62.0%
60.0%
Residue By Evaporation (D 6934)
60.0%
62.0%
64.0%
66.0%
68.0%
70.0%
72.0%
74.0%
76.0%
Moisture Analyzer Balance (D 7404)


More direct recovery in
a silicone mold (~22 mm diameter)
Enter
First experiments with silicone
mold carried out at Idaho Transportation Department (2010)

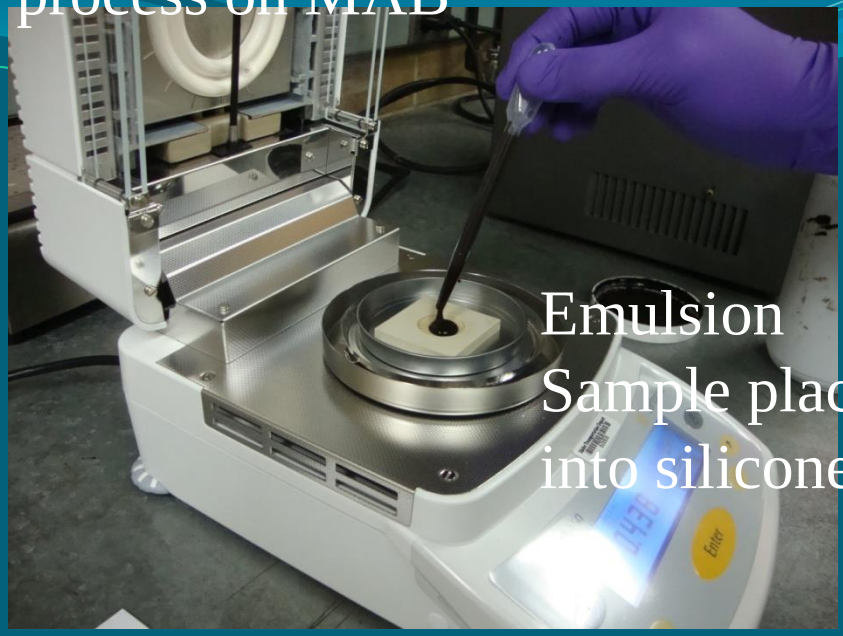

Emulsion sample
In the silicone mold
Idaho Transportation Depart
626806
sartorius
MA 150
P6 125°C Auto.
+ 1.230 g
START

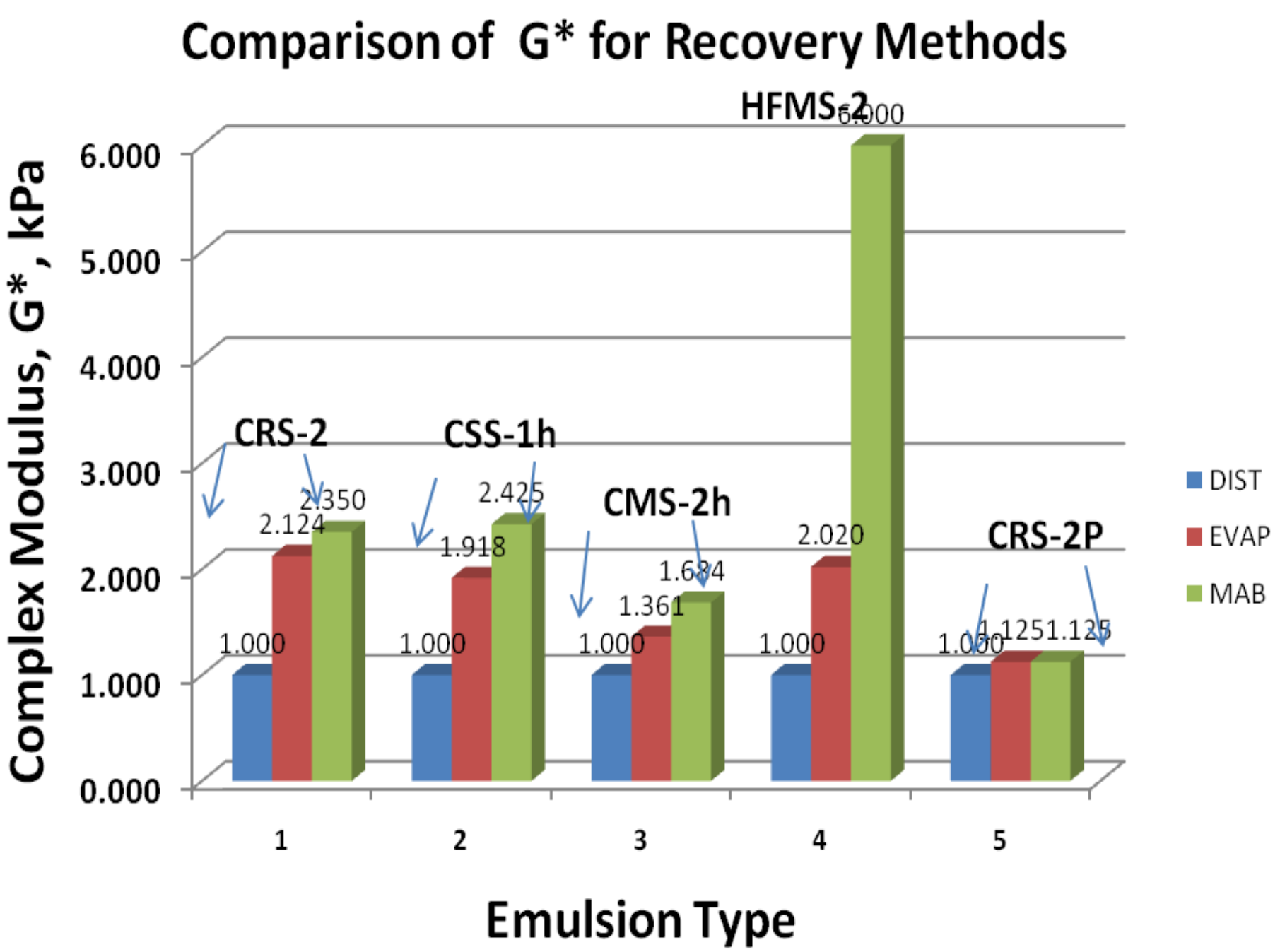

Idaho Transportation Depart
626806
MA 150
sartorius
P6 125°C Auto.
+ 0.8 12 g
SETUP
PROGRAM
TARE
I/O
CF
Enter


Recovered residue


Residue to DSR

Recovery process on MAB




Comparison of G* for Recovery Methods
Complex Modulus, G*, kPa
6.000
5.000
4.000
3.000
2.000
1.000
0.000
CRS-2
CSS-1h
CMS-2h
HFMS-2
CRS-2P
1.000
2.124
2.350
1.000
1.918
2.425
1.000
1.361
1.684
1.000
2.020
6.000
1.000
1.125
1.125
1
2
3
4
5
Emulsion Type
DIST
EVAP
MAB

Moisture Analyzer Balance = MAB

2006

2010

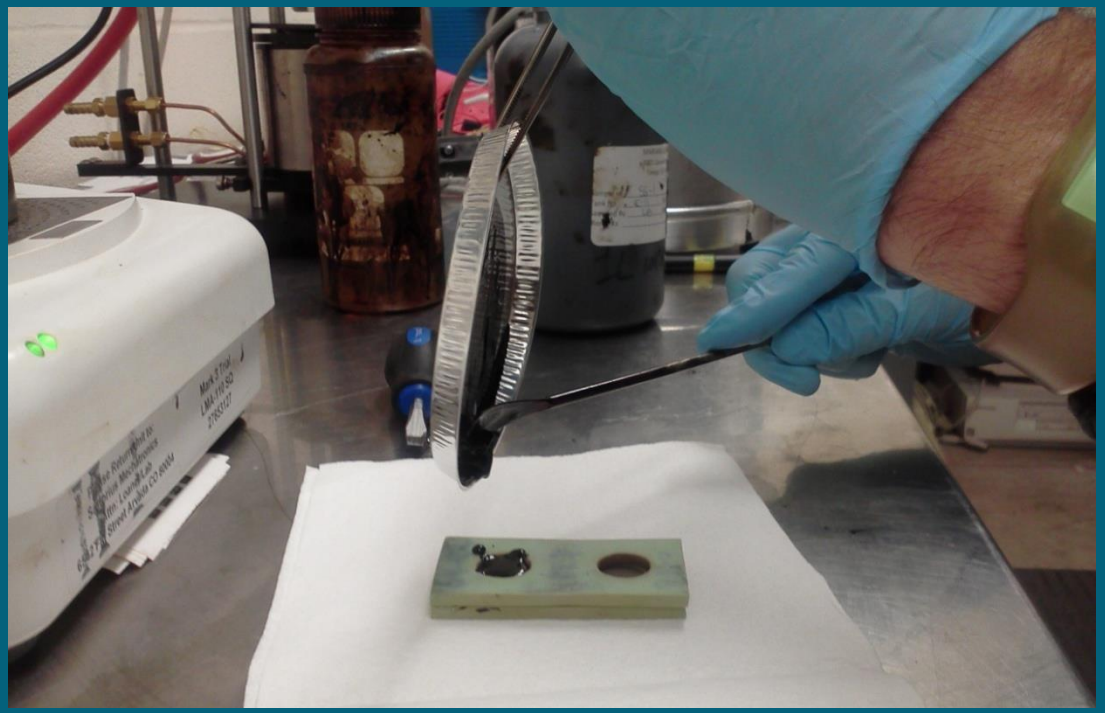
MAB recent work (not using DSR mold directly)

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)

www.pavementpreservationsystems.com for publications on emulsified asphalt