

# Pacific Coast Conference on Asphalt Specifications Round robin committee

Interlaboratory Study (ILS)  
For  
Precision of Measurements  
With the  
Dynamic Shear Rheometer (DSR)  
March 26, 2007



# Topics

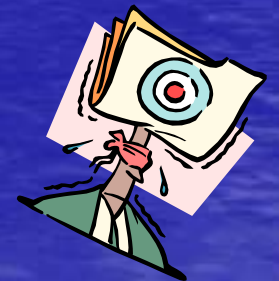
1. *Objectives of the Study*
2. *General Information*
3. *Precision Statements*
4. *Data Management*
5. *Outlier Analysis*



Cont'd. →

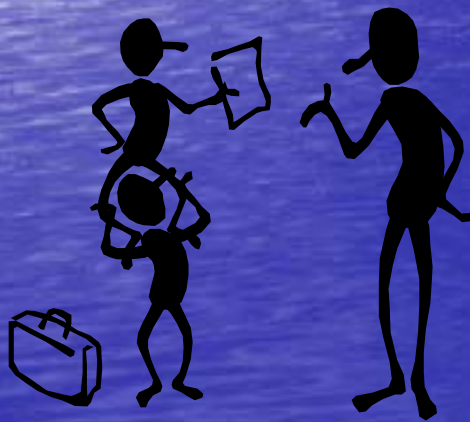
## Objectives

- *Reconsider Revised Procedures and Develop Precision Statements Based on These Procedures*
- *Determine Effect (If Any) of Modification*
- *Effect of PAV Temp on Precision Statements*
- *Examine Possibility of Using Phase Angle,  $\delta$  To Identify Polymer Modified Binders*





# General Information



## Task Group on DSR Precision



**Steve Davis, Washington DOT**

**Steve Landers, Washington DOT**

**Brad Neitzke, Western Federal Lands, FHWA**

**Julie Nodes, Arizona DOT**

**Ray Pavlovich, HRC Research Consultants**

**Don Powell, San Joaquin Refining**

**Shauna TecleMariam, U.S. Oil and Refining, Chair**

**Pat Turpen, McCall Oil**



# History

- Started the round robin study in 2000
- Three regional meetings were held to go over the procedures
- Completed one round robin where 84 items were identified as possible problems with the procedures. Approximately 15 items were significant.
- The report was published and given at ASTM and ETG meeting
- Revision of the procedures were started

# History

- Four regional meetings were held before the second round robin was started.
- Second round robin was started.

# Participating Laboratories

**Albina Asphalt**  
**Klamath Falls, OR 97603-5747**

**Albina Asphalt**  
**Vancouver, WA 98668**

**Arizona Department of Transportation**  
**Phoenix, AZ 85009-3740**

**Asphalt Paving & Recycling Technologies**  
**Shafter, CA 93263**

**CALTRANS**  
**Sacramento, CA 95819-0128**

**Ergon Asphalt Products, Inc.**  
**Las Vegas, NV 89139**

**FHWA, Western Federal Lands**  
**Vancouver, WA 98661**

**FHWA**  
**McLean, VA 22101**

**Idaho Asphalt Supply, Inc.**  
**Nampa, ID 83651**

**McCall Oil & Chemical**  
**Portland, OR 97210**



**Nevada Department of Transportation**  
**Las Vegas, NV 89101**

**Nevada Department of Transportation**  
**Carson City, NV 89712**

**Oregon Department of Transportation**  
**Salem, OR 97310**

**Paramount Petroleum**  
**Portland, OR 97210**

**Paramount Petroleum**  
**Phoenix, AZ 85009**

**Paramount Petroleum**  
**Paramount, CA**

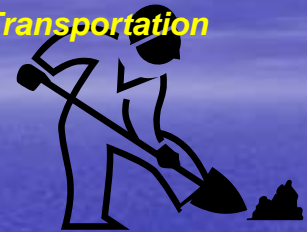
**San Joaquin Refining Company**  
**Bakersfield, CA 93308**

**U S Oil & Refining Co.**  
**Tacoma, WA**

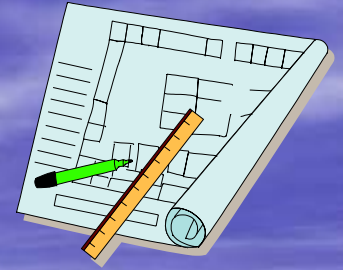
**Valero Energy**  
**Benicia, CA 94510**

**Washington Department of Transportation**  
**Tumwater, WA 98504**

**Wright Asphalt**  
**Channelview, TX 77530**

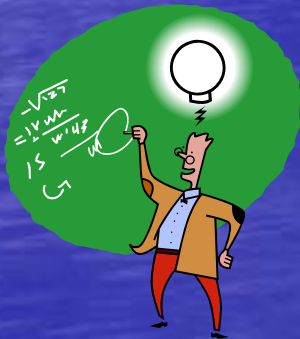


# Design of the Experiment



## Independent Variables

- **23 Materials**
- **21 Participating Laboratories**
- **3 Replicates**
- **Completely Randomized Test Sequence**



## Design of the Experiment, Cont'd.

### Dependent Variables

- **Unaged and RTFO Conditioning**
  - ↗ **Complex Shear Modulus,  $G^*$**
  - ↗ **Phase Angle,  $\delta$**
  - ↗ **Dynamic Shear,  $G^*/\sin \delta$**
- **Pressure Aging Vessel (PAV) Conditioning**
  - ↗ **Complex Shear Modulus,  $G^*$**
  - ↗ **Phase Angle,  $\delta$**
  - ↗ **Dynamic Shear,  $G^*\sin \delta$**
  - ↗ **Slope,  $m$ -value**
  - ↗ **Stiffness,  $S$**



## Some Facts and Figures



- **Set-Ups by Participating Labs:  
23 Matls x 3 Replicates = 69 Tests**
- **Samples = 23 Matls x 25 Labs x 3 Replicates  
= 1,725 Quarts**
- **Data Points = 23 Matls x 14 Properties (Including  $\sin \delta$ ) x 21 Labs x 3 Replicates = 20,286**
- **Database Including Temperatures and Binder  
Type = 28 Columns x 1,449 Lines = 40,572 Cells**

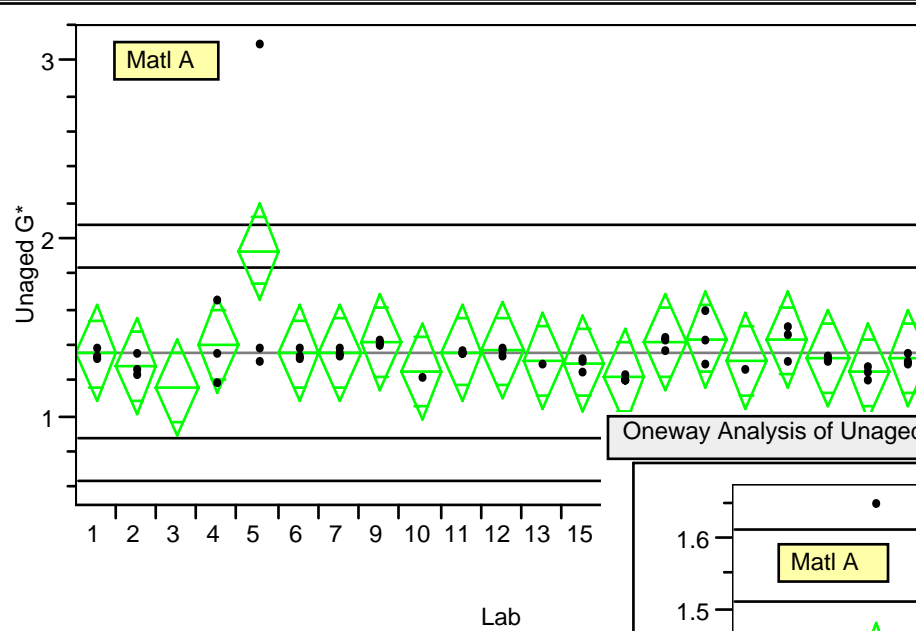


# Data After Sorting by Material

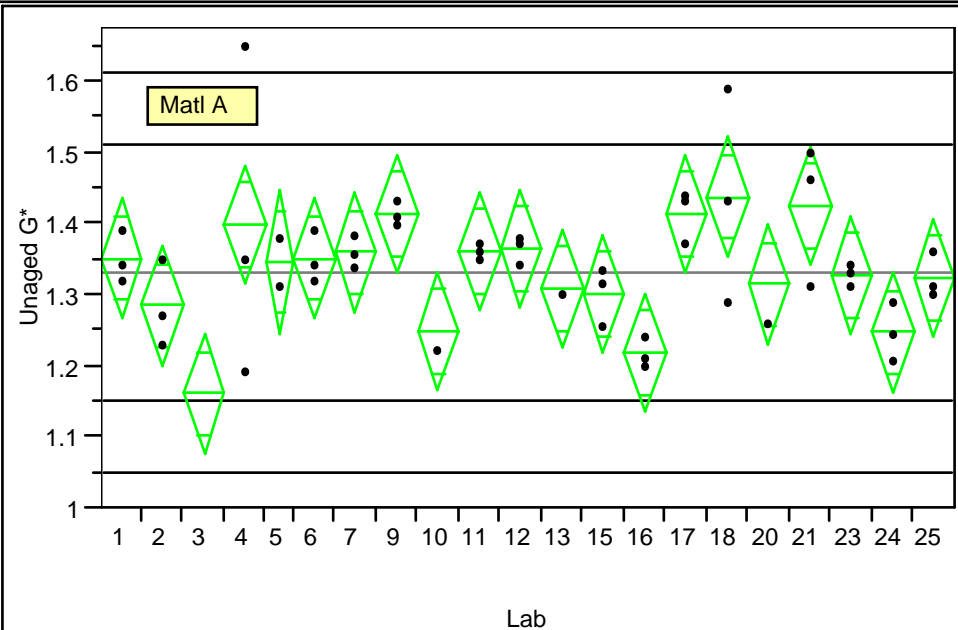
| Lab | Matl | Test | Unaged   |          |            | RTFO     |          |            | PAV      |          |          |        |         |
|-----|------|------|----------|----------|------------|----------|----------|------------|----------|----------|----------|--------|---------|
|     |      |      | G* (kPa) | $\delta$ | G* / sin d | G* (kPa) | $\delta$ | G* / sin d | G* (MPa) | $\delta$ | G* sin d | m      | S (MPa) |
| 1   | A    | 25   | 1.32     | 80.0     | 1.36       | 2.84     | 73.4     | 3.09       | 2,240    | 46.2     | 1,620    | 0.336  | 232     |
|     | A    | 64   | 1.39     | 80.0     | 1.44       | 3.11     | 72.3     | 3.42       | 1,720    | 46.7     | 1,250    | 0.329  | 216     |
|     | A    | 65   | 1.34     | 80.3     | 1.38       | 3.22     | 72.3     | 3.55       | 2,090    | 46.4     | 1,520    | 0.331  | 217     |
|     | Ave  |      | 1.350    | 80.10    | 1.393      | 3.057    | 72.67    | 3.353      | 2,017    | 46.43    | 1,463    | 0.3320 | 221.7   |
| 1   | B    | 2    | 1.46     | 82.9     | 1.48       | 3.80     | 76.5     | 4.02       | 2,190    | 44.9     | 1,550    | 0.309  | 191     |
|     | B    | 8    | 1.52     | 83.0     | 1.54       | 3.49     | 77.4     | 3.67       | 2,510    | 44.9     | 1,770    | 0.332  | 173     |
|     | B    | 55   | 1.43     | 83.3     | 1.45       | 3.23     | 77.8     | 3.38       | 1,850    | 45.7     | 1,320    | 0.325  | 165     |
|     | Ave  |      | 1.470    | 83.07    | 1.490      | 3.507    | 77.23    | 3.690      | 2,183    | 45.17    | 1,547    | 0.3220 | 176.3   |
| 1   | C    | 6    | 1.17     | 83.4     | 1.18       | 2.64     | 78.8     | 2.74       | 1,960    | 47.9     | 1,450    | 0.312  | 172     |
|     | C    | 16   | 1.40     | 83.1     | 1.42       | 3.06     | 77.8     | 3.21       | 1,450    | 47.7     | 1,070    | 0.327  | 195     |
|     | C    | 37   | 1.17     | 83.5     | 1.18       | 2.72     | 78.8     | 2.83       | 1,950    | 48.8     | 1,470    | 0.313  | 209     |
|     | Ave  |      | 1.247    | 83.33    | 1.260      | 2.807    | 78.47    | 2.927      | 1,787    | 48.13    | 1,330    | 0.3173 | 192.0   |
| 1   | D    | 10   | 2.46     | 85.0     | 2.48       | 5.06     | 81.5     | 5.17       | 2,120    | 48.5     | 1,580    | 0.370  | 115     |
|     | D    | 15   | 2.24     | 85.1     | 2.26       | 5.79     | 81.1     | 5.93       | 2,780    | 47.4     | 2,050    | 0.368  | 114     |
|     | D    | 23   | 2.37     | 85.0     | 2.39       | 2.87     | 67.0     | 3.38       | 351      | 55.0     | 288      | 0.000  | 0       |
|     | Ave  |      | 2.357    | 85.03    | 2.377      | 4.573    | 76.53    | 4.827      | 1,750    | 50.30    | 1,306    | 0.2460 | 76.3    |
| 1   | E    | 3    | 1.54     | 87.6     | 1.55       | 3.36     | 86.0     | 3.38       | 2,630    | 52.0     | 2,070    | 0.368  | 113     |
|     | E    | 59   | 1.71     | 87.6     | 1.72       | 3.13     | 86.2     | 3.14       | 0        | 0.0      | 0        | 0.000  | 0       |
|     | E    | 63   | 1.73     | 87.4     | 1.74       | 3.06     | 86.1     | 3.07       | 0        | 0.0      | 0        | 0.000  | 0       |
|     | Ave  |      | 1.660    | 87.53    | 1.670      | 3.183    | 86.10    | 3.197      | 877      | 17.33    | 690      | 0.1227 | 37.7    |
| 1   | F    | 33   | 1.15     | 54.8     | 1.72       | 2.01     | 54.1     | 3.06       | 452      | 48.8     | 340      | 0.404  | 29      |
|     | F    | 34   | 1.19     | 54.5     | 1.79       | 1.90     | 54.1     | 2.90       | 353      | 50.7     | 273      | 0.414  | 31      |
|     | F    | 48   | 1.14     | 54.9     | 1.70       | 1.89     | 54.3     | 2.86       | 0        | 0.0      | 0        | 0.000  | 0       |
|     | Ave  |      | 1.160    | 54.73    | 1.737      | 1.933    | 54.17    | 2.940      | 268      | 33.17    | 204      | 0.2727 | 20.0    |
| 1   | G    | 44   | 1.70     | 78.0     | 1.77       | 3.83     | 73.0     | 4.19       | 0        | 0.0      | 0        | 0.318  | 261     |
|     | G    | 61   | 1.82     | 78.0     | 1.74       | 3.60     | 74.0     | 3.89       | 0        | 0.0      | 0        | 0.000  | 0       |
|     | G    | 66   | 1.68     | 77.8     | 1.76       | 3.73     | 73.5     | 4.06       | 0        | 0.0      | 0        | 0.000  | 0       |
|     | Ave  |      | 1.733    | 77.93    | 1.757      | 3.720    | 73.50    | 4.047      | 0        | 0.00     | 0        | 0.1060 | 87.0    |

# Appendix B, Participant Review and Outlier Analysis

Oneway Analysis of Unaged G\* By Lab



Oneway Analysis of Unaged G\* By Lab



**Laboratory 5,  
Test 64 Removed. →  
Laboratory 4  
Possible Suspect**

## Laboratory Testing By Temperature Grouping



- *Some Laboratories Departed From Random Testing Schedule Provided by Task Group*
- *Concern Is Introduction of Laboratory Bias*

*i,e., Say Temperature is Off by a Constant Amount for This Particular Setting — This Would be Balanced Out if Temperatures Were Assigned According to Test Schedule*

- *Laboratories 1,3,4 and 5 — Non-Random Testing*

# Laboratory Testing By Temperature Grouping, Cont'd.



- *Conclude:*

*Non-Random Test Sequence Responsible  
for Disproportionate Number of Outliers*

*Result:*

- *Reject All Data From Laboratories That  
Did Not Follow Random Testing Schedule*



*Analyst Comments:  
"Please Follow Directions"*



# Action Items

- Need to check the data to make sure that all of the data transferred correctly
- A study is going on about where you place the pans in the PAV and if it effects the results
- Polymers are still a problem. Need group meetings to discuss this issue
- Report will hold until we have more issues resolved.