#### Formulation of Precision and Bias Statements for Performance Grading of Crumb Rubber Modified Asphalt Binders

PCCAS Round Robin Committee Sallie Houston Shauna Teclemariam

Independent data analysis by: Shakir Shatnawi, Ph.D., P.E. Shatec Engineering Consultants

# 16 Participating Labs

- Albina Asphalt
- Asphalt Institute
- Asphalt Pavement & Recycling Technologies
- California DOT Translab
- Chico State University California Pavement Preservation Center
- Federal Highway Administration (FHWA)
- FHWA Turner Fairbank Highway Research Center
- FHWA/WFLHD
- Nevada DOT
- Texas DOT-Asphalt Lab
- UC Pavement Research Center (UCPRC)
- University of Nevada, Reno (UNR)
- US Oil & Refining
- Valero Energy
- VSS Emultech
- WSDOT

#### Round Robin Phase 2

- Three field blended samples
- 3 Qt. cans of each
- Each test was done in triplicate
- Random selection of the sample for testing
- Full performance grading plus elastic recovery
- Tested as
  - PG 88-16 for Sample A
  - PG 82-22 for Sample B
  - PG 88-22 for Sample C

#### Round Robin Phase 2

- Original binder simulating *no aging* (per ASTM D 7175)
- Aged using the Rolling Thin Film Oven (RTFO) (per ASTM D 2872) simulating short-term aging of the binder, and
- Aged using Pressure Aging Vessel (PAV) (per ASTM D 6521) simulating *long-term aging* of the binder.

|   | SAMPLE A (PG 88-16) |                | SAMPLE B (PG 8      | 82-22) | SAMPLE C (PG 88-22) |       |
|---|---------------------|----------------|---------------------|--------|---------------------|-------|
| Asphaltª  | PG 70-10            | 76             | PG 64-16            | 77     | PG 64-22            | 80    |
| Extender Oil <sup>b</sup>   | Supplier A          | 2              | Supplier A          | 2      | Supplier A          | 2     |
| CRM Scrap Tire <sup>c</sup>   | Supplier A          | 17             | Supplier A          | 15.75  | Supplier B          | 13.5  |
| CRM High Nat <sup>c</sup>   | Supplier A          | 5              | Supplier A          | 5.25   | Supplier B          | 4.4   |
| Binder/Rubber <sup>d</sup>  | Ratio               | 78/22          | Ratio               | 70/21  | Ratio               | 82/18 |
|   |                     |                |                     |        |                     |       |
| a asphalt from 3 different suppliers  |                     |                |                     |        |                     |       |
| <sup>b</sup> Extender oil from same supplier for the three samples                        |                     |                |                     |        |                     |       |
| <sup>c</sup> CRM source from two s  | uppliers            | -              |                     |        |                     |       |
| <sup>d</sup> Each sample (A,B,C)fiel  | d produced by a o   | different supp | olier in California |        |                     |       |
|   |                     |                |                     |        |                     |       |
| The three materials were field produced by different suppliers, all according to Caltrans |                     |                |                     |        |                     |       |
| Section 39–3.02 for Asphalt Rubber Binder for Rubber Hot Mix Asphalt.                     |                     |                |                     |        |                     |       |

#### Precision Analysis

- ASTM C 802 "Standard Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods for Construction Materials"
- ASTM C670 "Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials"
- ASTM E177 "Practice for Use of the Terms Precision and Bias in ASTM Test Methods"

## Precision Analysis ASTM C 802

- single-operator precision (repeatability)
  - estimates of the inherent variability
  - expected difference between replicate measurements
  - same material
  - same laboratory
  - same operator
  - same apparatus

# Precision Analysis ASTM C 802

- Multilaboratory precision (reproducibility)
  - estimates of the inherent variability between labs
  - expected difference between measurements between labs
  - same material
  - different laboratory
  - different operator
  - similar apparatus

#### Bias Analysis ASTM C 670

- systematic error inherent in the test method
- contributes to the difference between the mean of the test results and an accepted reference or true value
- Bias cannot be measured in this study- No known standard

#### Interlaboratory Study ILS

- multiple laboratories
- replicate test results
- multiple materials
- Develop precision statements
  - identify and quantify the sources of variation in a process
  - May indicate further improvement is needed in the test method

#### Statistical Analysis Procedure

- Step 1. Plot the data to look for potential inconsistency
- Step 2. Perform single-operator and between-laboratory analysis
- Step 3. Check for data inconsistency
- Step 4. Check for interactions
- Step 5. Calculate single-operator and multilaboratory variances
- Step 6. Determine the forms of and write precision statements

# Step 1. Plot the data to look for potential inconsistency

- A scatter plot points out inconsistent data
- visually or statistically
- This study used outlier detection method by Hoaglin et al.
  - used in the NCHRP 29-6 study
  - Data that positively proven to be outliers must be removed before proceeding with the analysis.
- Hoaglin, D. C., Iglewicz, B., Tukey, J. W., "Performance of Some Resistant Rules for Outlier Labeling", Journal of the American Statistical Association, Vol. 81, No. 396 (Dec., 1986), pp. 991-999.
- Holsinger, R., Fisher, A., Spellerberg, P. "Precision Estimates for AASHTO Test Method T308 and the Test Methods for Performance-Graded Asphalt Binder in AASHTO Specification M320", February 2005, 139 p.

# Step 1. Plot the data to look for potential inconsistency



. Example scatter plot of test results (G\*/sinδ) of the three samples (3 replicates per test)

# Step 2. Perform single-operator and betweenlaboratory analysis

- (1) the pooled single-operator variance
- (2) the overall average
- (3) the variance of laboratory averages
- (4) the between-laboratory component of variance

# Step 2. Perform single-operator and betweenlaboratory analysis

• Determine single operator variance first



single test determination g by laboratory i for material j

$$\bar{X}_{ij} = \frac{\sum x_{gij}}{n}$$
 (1) = average of *n* replicate test determinations for laboratory *i* on material

$$s_{rij}^{2} = \frac{\Sigma \left( x_{gij} - \bar{X}_{ij} \right)^{2}}{n-1}$$
(2)

single-operator variance of replicate
 determinations for laboratory i on material j

# Step 2. Perform single-operator and betweenlaboratory analysis

 $s_{Lj}^2 = s_{\bar{X}_1}^2 - \frac{s_{fj}^2}{n}$ 

• Determine between-laboratory variance

$$s_{rj}^2 = \frac{\sum s_{rij}^2}{p}$$
 (3) = pooled single-operator variance for material *j* (Note 4)

$$\bar{X}_j = \frac{\Sigma \bar{X}_{ij}}{p}$$
 (4) = overall average for all labora-  
tories for material  $j$ 

$$s_{\bar{x}_j}^2 = \frac{\Sigma (\bar{X}_{ij} - \bar{X}_j)^2}{p-1} \qquad (5) = \begin{array}{c} \text{variance of laboratory aver-}\\ \text{ages for material } j \end{array}$$

(6) = between-laboratory component of variance for material
 *j*. If the calculated values is negative, the between-laboratory component of variance is taken as zero.

- Bartlett's test (recommended ASTM C 802)
- Check each lab for consistency in terms of the average and the dispersion of the results
- Inconsistent data may inflate the calculated precision values and thereby encourage laboratories to tolerate less careful testing
- **h-value** check whether the average value for a lab is consistent with the overall average of the other labs for a given material
- k-value check the consistency of the single-operator variability for a given material
- Compared against a critical value (found in ASTM C 802)
  - Determine if data or lab needs elimination

 $h_{ij} = \frac{\bar{X}_{ij} - \bar{X}_j}{\bar{X}_{ij}}$ (7)where: average of results for laboratory *i* and material *j* (Eq 1),
overall average of results for material *j* (Eq 4), and
standard deviation of laboratory averages for material  $\bar{X}_{ij}$  $\bar{X}_j$ *j*, which is the square root of Eq 5.  $k_{ij} = \frac{S_{rij}}{S_{ri}}$ (8)where:  $s_{rii}$  = single-operator standard deviation of replicate determinations for laboratory i and material j, which is the square root of Eq 2, and = pooled single-operator standard deviation for material Sri *j*, which is the square root of Eq 3.

Figure 3-4. Equations for the calculation of the consistency k-value and h-value.





# Step 4. Check for interactions

- Interactions in the data mean that the pattern of the results obtained on the material by one laboratory differs from the pattern obtained by the other laboratories (ASTM C 802)
- plot of the averages by each laboratory.
- similar patterns = absence of interactions
- If one or two laboratories show noticeably different pattern they should be considered for elimination
- If multiple labs show different patterns the test method needs to be investigated and the causes of the interactions identified and eliminated

### Step 4. Check for interactions



Figure 3-6. A plot of the average  $(G^*/\sin \delta)$  for the three original (unaged) binders as tested by all laboratories in the current ILS. The plot is used to detect interactions.

# Step 5. Calculate single-operator and multilaboratory variances

- Recalculate after removing outliers and inconsistent
- The single-operator variance (repeatability variance)
  - Recalculate after removing outliers

 $s_{rj}^2 = \frac{\sum s_{rij}^2}{p}$  (3) = pooled single-operator variance for material *j* (Note 4)

- The multilaboratory variance (reproducibility variance)
  - Sum of single operator variance and between lab variance

 $s_{RJ}^2 = s_{rJ}^2 + s_{LJ}^2$  (9) = multilaboratory variance for material j

• Standard deviataion is Sq root of variance. If  $S_{Rj} < S_{rj}$  then  $S_{Rj}$  set to equal  $S_{rj}$ 

# Step 6. Determine the forms of and write precision statements

- single-operator (repeatability) and multilaboratory (reproducibility)
- standard deviations and coefficients of variation are plotted against the average value of the property
  - Look for trends
- Standard Deviation precision limits d2s = 2.8 x std dev
- Coefficient of variation precision limits d2s% = 2.8 x cv (std dev/mean)
- With 95% confidence

# Step 6. Determine the forms of and write precision statements



- Plot std dev and CV against avg
- Is it constant of approx. constant
- Run Bartlett or other test to determine if from same population
- Set precision statement with limits

# Data Quality Testing

- Invalid and Outlier Data
  - Review of data
  - Determine if run in triplicate
    - Lab 10 eliminated from study
    - Less BBR data 8 labs
  - Invalid data- Hoaglin Method (NCHRP 9-26)
    - Upper and lower limits
    - Remove invalid data and analyze for outliers via Hoaglin
- Testing for Homogeneity of Variance
  - Determine if all variations are equal (null hypothesis)

#### Analyzed Properties

G\* Original Phase Angle  $\delta$  Original G\*/sinδ Original G\* RTFO Phase Angle  $\delta$  RTFO  $G^*/sin\delta RTFO$ Elastic Recovery % RTFO G\* RTFO/PAV Phase Angle  $\delta$ G\*sinδ RTFO/PAV m-value BBR Measured Stiffness BBR

#### Analyzed Properties

- 1. Plots of variation of property replicates for each lab and utilizing all the data.
- 2. Plots of the h-value and k-value for all the data.
- 3. Plots of variation of the average property with sample number (trend or pattern plot)
- 4. Standard deviation and coefficient of variation tables after data processing by the validity, outlier and consistency filters.
- 5. Plots of variation of the standard deviation and coefficient of variation of both the singleoperator (within-laboratory or repeatability) and multilaboratory (or reproducibility) against property average values.
- 6. Calculated single-operator and multilaboratory standard deviation and/or coefficient of variation and corresponding precision limits required for formulating precision statements.
- 7. Proposed repeatability and reproducibility precision statements for the property.

### Original Binder – Unaged G\*



Figure 6-1. variation of the complex modulus G\* for the three replicates tested by the 15 laboratories for the three samples A, B, and C.

# Original Binder – Unaged G\*



Figure 6-2. The consistency h-value and k-value plots for G\*original plotted by laboratory.



Figure 6-3. The consistency h-value and k-value plots for G\*original plotted by material type.





Figure 6-4. Variation of average G\* per material type for the 12 labs used in subsequent precision analysis.



Figure 6-5. Variation of the single-operator and multilaboratory standard deviation and coefficient of variation with average G\*-Original.

| BARTLETT TEST FOR HOMOGENEITY OF MULTI-VARIANCES                             | BARTLETT TEST FOR HOMOGENEITY OF MULTI-VARIANCES                              |  |  |  |
|--|---|--|--|--|
| SINGLE-OPERATOR VARINCES   | MULTILABORATORY VARIANCES   |  |  |  |
| k=number o 3   | k=number o 3  |  |  |  |
| group name A B C   | group name A B C  |  |  |  |
| Ni=number 12 12 12   | Ni=number 12 12 12  |  |  |  |
| N=total nun 36   | N=total nun36   |  |  |  |
| σ <sup>-</sup> i=variance 0.0193 0.0318 0.0126                               | σ <sup>2</sup> i=variance 0.0294 0.0420 0.0135                                |  |  |  |
| (Ni-1)σ <sup>2</sup> i 0.212487 0.34958 0.138768                             | (Ni-1) $\sigma^2$ i 0.322856 0.461976 0.148762                                |  |  |  |
| σ <sup>2</sup> p = 0.021237 This is the pooled variance                      | $\sigma^2 p = 0.028291$ This is the pooled variance                           |  |  |  |
| (N-k)ln(σ²p) -127.116  | $(N-k)\ln(\sigma^2 n) = 117.652$  |  |  |  |
| (Ni-1)ln(σ²i) -43.4145 -37.9381 -48.1013                                     | $(N_i = 1) \ln(\sigma^2 i) = 22.923 + 24.9715 + 47.2264$                      |  |  |  |
| Σ(Ni-1)ln(σ <sup>2</sup> -129.454  | (N-1)(0, 0) = -50.0125 = -54.0715 = -47.5504                                  |  |  |  |
| 1/3(k-1) 0.166667  | $2(N-1)\ln(0 - 121.021)$<br>1/2(k-1) = 0.166667                               |  |  |  |
| 1/(Ni-1) 0.090909 0.090909 0.090909  | 1/S(k-1) = 0.100007<br>1/(Ni-1) = 0.090909 = 0.090909 = 0.090909              |  |  |  |
| Σ1/(Ni-1) 0.272727   | Σ1/(Ni-1) 0.272727  |  |  |  |
| 1/N-k 0.030303   | 1/N-k 0.030303  |  |  |  |
| numerator 2.338194   | numerator 3.368446  |  |  |  |
|  | denominatc 1.040404   |  |  |  |
| χ <sup>2</sup> Calculate 2.247391 This is the Bartlett test statistic        | χ <sup>2</sup> Calculate: <b>3.237632</b> This is the Bartlett test statistic |  |  |  |
| sig.level (a) 0.05 This is significance level                                | sig.level (α) 0.05 This is significance level                                 |  |  |  |
| 1-α 0.95   | 1-α 0.95  |  |  |  |
| d.f.(=k-1) 2   | d.f.(=k-1) 2  |  |  |  |
| χ <sup>2</sup> <sub>1-α,k-1</sub> <b>5.991465</b> This is the critical value | χ <sup>2</sup> <sub>1-α,k-1</sub> 5.991465 This is the critical value         |  |  |  |
| Critical regic Reject Ho if Bartlett statistic>Critical value                | Critical regicReject Ho if Bartlett statistic>Critical value                  |  |  |  |
| Ho (null hypothesis): All k population variances are equal.                  | Ho (null hypothesis): All k population variances are equal.                   |  |  |  |
| Conclusion: Accept Ho that all k population variances are equal.             | Conclusion: Accept Ho that all k population variances are equal.              |  |  |  |

Figure 6-6. Bartlett's test for homogeneity of single-operator and multilaboratory variances of G\*-Original.



|                           |           | Comp. of Variance |           | Variand              | es        |  |
|---------------------------|-----------|-------------------|-----------|----------------------|-----------|--|
| (1)                       | (2)       | (3)               | (4)       | (5)                  | (6)       |  |
| Material                  | Average   | Single Op         | b/n lab   | Single Op            | b/n lab   |  |
| Α                         | 2.01      | 0.0193            | 0.0100    | 0.0193               | 0.0294    |  |
| В                         | 1.37      | 0.0318            | 0.0102    | 0.0318               | 0.0420    |  |
| С                         | 1.43      | 0.0126            | 0.0009    | 0.0126               | 0.0135    |  |
| Pooled Va                 | riance    | 0.021             | 0.028     |                      |           |  |
| Pooled Standard Deviation |           |                   |           | 0.146                | 0.168     |  |
|                           |           | Standard De       | viations  | Coef. Of Variation % |           |  |
| (1)                       | (2)       | (3)               | (4)       | (5)                  | (6)       |  |
| Material                  | Average   | Single Op         | Multi-Lab | Single Op            | Multi-Lab |  |
| A                         | 2.01      | 0.139             | 0.171     | 6.91                 | 8.51      |  |
| В                         | 1.37      | 0.178             | 0.205     | 13.05                | 15.01     |  |
| С                         | 1.43      | 0.112             | 0.116     | 7.88                 | 8.16      |  |
| AVG. COEF                 | F. OF VAR | IATION (%)        |           | 9.28                 | 10.56     |  |

Table 6-1. Single-operator and multilaboratory variances, standard deviations and coefficient of variations for G\*-Original. Note that G\* has units of kPa.



- Single-Operator precision (repeatability): The pooled standard deviation was found to be 0.146 kPa and the results of two properly conducted tests by the same operator on the same material are not expected to differ by more than 0.408 kPa.
- Multilaboratory precision (reproducibility): The pooled standard deviation was found to be 0.168 kPa and the results of two properly conducted tests by two different laboratories on the same material are not expected to differ by more than 0.471 kPa.

- Precision estimates established within-laboratory (repeatability) and between laboratories (reproducibility) for the individual properties have been obtained and precision statements have also been formulated
- The precision estimates are primarily based on standard deviations and the acceptable range (difference) between two test measurements (precision limits).
- Precision limits based on repeatability and reproducibility coefficients of variation (CoV) were also developed for five properties that were found to have small CoVs. The maximum CoV, rather than the average CoV, was used to estimate the precision limit.

| Table 9-1. Summary of precision estimate of all the tested properties. |                                    |                                      |                                    |                                      |                                    |                                      |                                    |                                      |
|--|------------------------------------|--------------------------------------|------------------------------------|--------------------------------------|------------------------------------|--------------------------------------|------------------------------------|--------------------------------------|
|  | Pooled Standard Deviation          |                                      | Acceptable Difference (d2s)*       |                                      | Average Coef. of Variation (CoV)   |                                      | Acceptable Difference (d2s%)**     |                                      |
| Binder property & (Unit)   | Single-Operator<br>(repeatability) | Multilaboratory<br>(reproducibility) | Single-Operator<br>(repeatability) | Multilaboratory<br>(reproducibility) | Single-Operator<br>(repeatability) | Multilaboratory<br>(reproducibility) | Single-Operator<br>(repeatability) | Multilaboratory<br>(reproducibility) |
| ORIGINAL (UNAGED) CRM BINDER   |                                    | •                                    |                                    |                                      |                                    |                                      |                                    |                                      |
| G* (kPa) <sup>a</sup>  | 0.15                               | 0.17                                 | 0.41                               | 0.47                                 | 9.3%                               | 10.6%                                | _                                  | _                                    |
| δ (deg) <sup>b</sup>   | 1.7                                | 3.0                                  | 4.8                                | 8.4                                  | 2.3%                               | 4.1%                                 | 9.2% <sup>(1)</sup>                | 15.7% <sup>(2)</sup>                 |
| (G*/sinδ) (kPa) <sup>c</sup>   | 0.19                               | 0.23                                 | 0.54                               | 0.64                                 | 11.6%                              | 13.8%                                | _                                  | _                                    |
| RTFO-AGED CRM BINDER   |                                    |                                      |                                    |                                      |                                    |                                      |                                    |                                      |
| G* (kPa) <sup>d</sup>  | 0.34                               | 0.50                                 | 0.94                               | 1.39                                 | 11.5                               | 16.6                                 | _                                  | _                                    |
| δ (deg) <sup>e</sup>   | 1.6                                | 2.2                                  | 4.4                                | 6.1                                  | 2.4                                | 3.4                                  | 9.0% <sup>(3)</sup>                | <b>10.4%</b> <sup>(4)</sup>          |
| (G*/sinδ) (kPa) <sup>f</sup>   | 0.37                               | 0.61                                 | 1.04                               | 1.70                                 | 11.6                               | 18.0                                 |                                    |                                      |
| Elastic Recovery, ER (%) <sup>g</sup>                                  | 1.7%                               | 2.4%                                 | 4.6%                               | 6.8%                                 | 1.9%                               | 2.8%                                 | <b>7.6%</b> <sup>(5)</sup>         | 11.5% <sup>(6)</sup>                 |
| RTFO/PAV-AGED BINDER   |                                    |                                      |                                    |                                      |                                    |                                      |                                    |                                      |
| G* (kPa) <sup>h</sup>  | 111.9                              | 173.7                                | 313.4                              | 486.4                                | 17.3%                              | 26.8%                                | _                                  | _                                    |
| δ (deg) <sup>i</sup>   | 1.5                                | 1.7                                  | 4.2                                | 4.7                                  | 3.0%                               | 3.5%                                 | 9.0% <sup>(7)</sup>                | 10.4% <sup>(8)</sup>                 |
| (G*.sinδ) (kPa) <sup>j</sup>   | 80.5                               | 122.9                                | 225.2                              | 344.2                                | 16.7%                              | 25.7%                                | -                                  | -                                    |
| BBR m-value <sup>k</sup>   | 0.010                              | 0.013                                | 0.028                              | 0.036                                | 2.8%                               | 3.7%                                 | 9.2% <sup>(9)</sup>                | 11.2% <sup>(10)</sup>                |
| BBR measured Stiffness, S <sub>m</sub> (MPa) <sup>1</sup>              | 6.2                                | 11.7                                 | 17.5                               | 32.8                                 | 7.8%                               | 15.7%                                | _                                  | -                                    |

\* Acceptable difference between two test results. \*\* Acceptable difference between two test results expressed as percent of their mean.

<sup>a</sup> Labs 2,5 and 9 removed. <sup>b</sup> Lab 9 removed, single-operator H<sub>o</sub> marginally accepted. <sup>c</sup> Labs 2 and 9 removed. <sup>d</sup> No labs removed. <sup>e</sup> Labs 5 and 9 removed. <sup>f</sup> Labs 4 and 9 removed. <sup>g</sup> No labs removed but only 10 labs conducted the test. <sup>h</sup> Labs 1, 6, and 9 removed. <sup>l</sup> Lab 9 removed. <sup>j</sup> Labs 1, 6 and 9 removed. <sup>k</sup> Lab 5 removed, but only 8 labs conducted the test. <sup>l</sup> Lab 5 removed, but only 8 labs conducted the test.

<sup>(1)</sup> Based on maximum CoV of 3.3%. <sup>(2)</sup> Based on maximum CoV of 5.6%. <sup>(3)</sup> Based on maximum CoV of 3.2%. <sup>(4)</sup> Based on maximum CoV of 3.7%. <sup>(5)</sup> Based on maximum CoV of 2.7%. <sup>(6)</sup> Based on maximum CoV of 4.1%. <sup>(7)</sup> Based on maximum CoV of 3.2%. <sup>(8)</sup> Based on maximum CoV of 3.2%. <sup>(9)</sup> Based on maximum CoV of 3.3%. <sup>(10)</sup> Based on maximum CoV of 4.0%.

|   | Pooled Standard Deviation          |                                      | Acceptable Difference (d2s)*       |                                      |  |
|---|------------------------------------|--------------------------------------|------------------------------------|--------------------------------------|--|
| Binder property & (Unit)                                  | Single-Operator<br>(repeatability) | Multilaboratory<br>(reproducibility) | Single-Operator<br>(repeatability) | Multilaboratory<br>(reproducibility) |  |
| ORIGINAL (UNAGED) CRM BINDER                              |                                    |                                      |                                    |                                      |  |
| G* (kPa) <sup>a</sup>                                     | 0.15                               | 0.17                                 | 0.41                               | 0.47                                 |  |
| δ (deg) <sup>b</sup>                                      | 1.7                                | 3.0                                  | 4.8                                | 8.4                                  |  |
| (G*/sinδ) (kPa) <sup>c</sup>                              | 0.19                               | 0.23                                 | 0.54                               | 0.64                                 |  |
| RTFO-AGED CRM BINDER                                      |                                    |                                      |                                    |                                      |  |
| G* (kPa) <sup>d</sup>                                     | 0.34                               | 0.50                                 | 0.94                               | 1.39                                 |  |
| δ (deg) <sup>e</sup>                                      | 1.6                                | 2.2                                  | 4.4                                | 6.1                                  |  |
| (G*/sinδ) (kPa) <sup>f</sup>                              | 0.37                               | 0.61                                 | 1.04                               | 1.70                                 |  |
| Elastic Recovery, ER (%) <sup>g</sup>                     | 1.7%                               | 2.4%                                 | 4.6%                               | 6.8%                                 |  |
| RTFO/PAV-AGED BINDER                                      | <u> </u>                           | <u> </u>                             |                                    |                                      |  |
| G* (kPa) <sup>h</sup>                                     | 111.9                              | 173.7                                | 313.4                              | 486.4                                |  |
| δ (deg) <sup>i</sup>                                      | 1.5                                | 1.7                                  | 4.2                                | 4.7                                  |  |
| (G*.sinδ) (kPa) <sup>j</sup>                              | 80.5                               | 122.9                                | 225.2                              | 344.2                                |  |
| BBR m-value <sup>k</sup>                                  | 0.010                              | 0.013                                | 0.028                              | 0.036                                |  |
| BBR measured Stiffness, S <sub>m</sub> (MPa) <sup>1</sup> | 6.2                                | 11.7                                 | 17.5                               | 32.8                                 |  |

- The variability of the complex modulus G\* of CRM binders; both within-laboratory or between laboratories, increased with binder aging. There was a greater increase in the variability of G\* measurement between-laboratories than within the same laboratory.
- The variability of the phase angle measurement with aging; both for the repeatability and reproducibility, was relatively small compared to G\*.
- The average coefficients of variation obtained for all the properties are somewhat reasonable enough for the products being tested and indicate a relatively repeatable and reproducible test procedures. The CoVs were higher for the properties of the PAV-aged residues compared to the unaged or the RTFO-aged.
- The repeatability and reproducibility CoVs for the m-value of the PAV-aged residues were reasonably small.
- The variability of the flexural creep stiffness was somewhat higher than that for the mvalue especially between laboratories. Note that fewer laboratories participated in the BBR testing; which may have affected the obtained precision estimates.

|   | Average Coef. of Variation (CoV) |                   | Acceptable Difference (d2s%)** |                       |
|---|----------------------------------|-------------------|--------------------------------|-----------------------|
|   | Single-Operator                  | Multilaboratory   | Single-Operator                | Multilaboratory       |
|   | (repeatability)                  | (reproducibility) | (repeatability)                | (reproducibility)     |
| ORIGINAL (UNAGED) CRM BINDER                              |                                  |                   |                                |                       |
| G* (kPa) *  | 9.3%                             | 10.6%             | -                              | -                     |
| δ (deg) <sup>b</sup>                                      | 2.3%                             | 4.1%              | 9.2% <sup>(1)</sup>            | 15.7% <sup>(2)</sup>  |
| (G*/sinð) (kPa) °   | 11.6%                            | 13.8%             | -                              | -                     |
| RTFO-AGED CRM BINDER                                      | L                                | I                 | I                              |                       |
| G* (kPa) d  | 11.5                             | 16.6              | -                              | -                     |
| δ (deg) °   | 2.4                              | 3.4               | 9.0% <sup>(3)</sup>            | 10.4% <sup>(4)</sup>  |
| (G*/ <u>sinð</u> ) ( <u>kPa</u> ) <sup>f</sup>            | 11.6                             | 18.0              |                                |                       |
| Elastic Recovery, ER (%) <sup>g</sup>                     | 1.9%                             | 2.8%              | 7.6% <sup>(5)</sup>            | 11.5% <sup>(6)</sup>  |
| RTFO/PAV-AGED BINDER                                      | <u> </u>                         |                   | 1                              |                       |
| G* (kPa) <sup>h</sup>                                     | 17.3%                            | 26.8%             | -                              | -                     |
| δ (deg)   | 3.0%                             | 3.5%              | 9.0% <sup>(7)</sup>            | 10.4% <sup>(8)</sup>  |
| (G*. <u>sinδ</u> ) (kPa) <sup>j</sup>                     | 16.7%                            | 25.7%             | -                              | -                     |
| BBR m-value <sup>k</sup>                                  | 2.8%                             | 3.7%              | 9.2% <sup>(9)</sup>            | 11.2% <sup>(10)</sup> |
| BBR measured Stiffness, S <sub>m</sub> (MPa) <sup>1</sup> | 7.8%                             | 15.7%             | -                              | -                     |

1. Elastic Recovery (ER): ASTM D 6084 provides precision estimates for unmodified and polymer modified binders. The estimates are shown below.

| Materials Index                | Standard<br>Deviation <sup>4</sup> | Acceptable<br>Range of<br>Two<br>Results <sup>A</sup> |
|--------------------------------|------------------------------------|---|
| 1 (unmodified)                 | 0.91                               | 2.6   |
| Multilaboratory precision:     | 0.50                               | 1.0   |
| 1 (unmodified)<br>2 (modified) | 2.32<br>1.71                       | 6.5<br>4.8  |

The single-operator and multilaboratory <u>standard deviation</u> were found as follows:

- i. Single-operator: 0.91 for unmodified and 0.56 for polymer modified binder. Compare these values to 1.7 for the CRM binder found in this study (Table 9-1).
- ii. Multilaboratory: 2.32 for unmodified and 1.71 for polymer modified binder. Compare these values to 2.4 for the CRM binder (Table 9-1).

1. BBR m-Value: ASTM D 6648 provides estimates of precision in terms of the coefficient of variation. The estimates are shown below. Note that ASTM D 6648 reported these values based on 300 tests on variety of binders, whereas the current study used data from a much smaller sample size.

| Condition             | Coefficient of<br>Variation (1 s %) <sup>4</sup> | Acceptable Range of<br>Two Test Results<br>(d2s %) <sup>A</sup> |
|-----------------------|--|---|
| Single-Operator       |  |   |
| Precision             |  |   |
| Creep Stiffness (MPa) | 3.2  | 9.1   |
| Slope                 | 1.4  | 4.0   |
| Multi-laboratory      |  |   |
| Precision             |  |   |
| Creep Stiffness (MPa) | 9.5  | 26.9  |
| Slope                 | 4.6  | 13.0  |

The estimates for the coefficient of variation (CoV) are as follows:

- i. Single-operator (m-value): 1.4%. Compare to 2.8% for the CRM binder testing (Table 9-1).
- ii. Multioperator (m-value): 4.6%. Compare to 3.7% for the CRM binder testing (Table 9-1).

- 1. **BBR Flexural Creep Stiffness:** ASTM D 6648 also provides estimates of precision for this property in terms of the coefficient of variation. The values are shown in the table above along with the m-value estimates. Note that the precision estimates for flexural stiffness could not be precisely compared because the ASTM method's values are based on CoV, while Table 9-1 values are based on standard deviation. However, if the <u>average</u> CoVs in Table 9-1 are used as the basis for precision estimates, then one should compare the following values:
  - i. Single-operator: 3.2% (ASTM method) versus 7.8% (Table 9-1)
  - ii. Multioperator: 9.5% (ASTM method) versus 15.7% (Table 9-1).

1. **Rutting Parameter (G\*/sin\delta):** ASTM D 7175 provides estimates of precision for the rutting parameter (G\*/sin $\delta$ ) of unaged binder and RTFO-aged residues. These are shown below in the table copied from the test method.

| Condition                       | Coefficient<br>of Variation<br>(1s%) <sup>A</sup> | Acceptable Range<br>of Two Test Results<br>(d2s%) <sup>A</sup> |
|---------------------------------|---|--|
| Single-Operator Precision:      |   |  |
| Original Binder: G*/sin δ (kPa) | 2.3   | 6.4  |
| RTFO/TFO Residue: G*/sin δ      | 3.2   | 9.0  |
| (kPa)                           |   |  |
| PAV Residue: G*sin δ (kPa)      | 4.9   | 13.8   |
| Multilaboratory Precision:      |   |  |
| Original Binder: G*/sin δ (kPa) | 6.0   | 17.0   |
| RTFO/TFO Residue: G*/sin δ      | 7.8   | 22.2   |
| (kPa)                           |   |  |
| PAV Residue: G*sin δ (kPa)      | 14.2  | 40.2   |

AS seen, the precision estimates given in the test method are reported on the basis of coefficient of variation, whereas those derived in the current study (summarized in Table 9-1) are based on standard deviations. Therefore, precise comparison between the reported values and those derived in the current study is not possible. However, if the <u>average</u> CoVs in Table 9-1 are used as the basis for precision estimates, then one should compare the following values:

i. Single-operator

- Original binder: 2.3% (test method) versus 11.6% (current study)
- RTFO-aged residue: 3.2% (test method) versus 11.6% (current study)

#### ii. Multioperator

- Original binder: 6.0% (test method) versus 13.8% (current study)
- RTFO-aged residue: 7.8% (test method) versus 18.0% (current study)

- Cracking Parameter (G\*.sinδ): ASTM D 7175 also provides estimates of precision for the fatigue cracking parameter (G\*.sinδ) of RTFO/PAV-aged residues. These are shown in the table above copied from the test method. As discussed previously, only comparison based on average CoVs reported in Table 9-1 could be made:
  - i. Single-operator: 4.9% (ASTM method) versus 16.7% (Table 9-1).
  - ii. Multioperator: 14.2% (ASTM method) versus 25.7% (Table 9-1).

#### Next Steps

- Publish Results
- Develop Specification
  - Caltrans
  - ASTM, RTFO procedure
- RTFO bottle creep
  - Small group, 4 labs
  - Different amounts of material
  - Other: smaller opening, insert
- BBR
  - Discussion on stiffness value