ASPHALT INSTITUTE

NCHRP 09-63 Update: Interlaboratory Study

ETF Meeting
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PRESENTED BY

Wes Cooper

Laboratory Manager &
Sr. Research Scientist

Phase III – Key Laboratory Tasks

- **Task 10**: Further develop and refine the asphalt emulsion residue recovery procedure(s)
- **Task 11**: Train asphalt industry professionals on the asphalt emulsion residue recovery/test procedures used to determine grading in the EAPG draft specification.
- **Task 12**: Conduct one or more interlaboratory studies (ILS) to assess the variability resulting from the residue recovery procedure and the associated residue tests

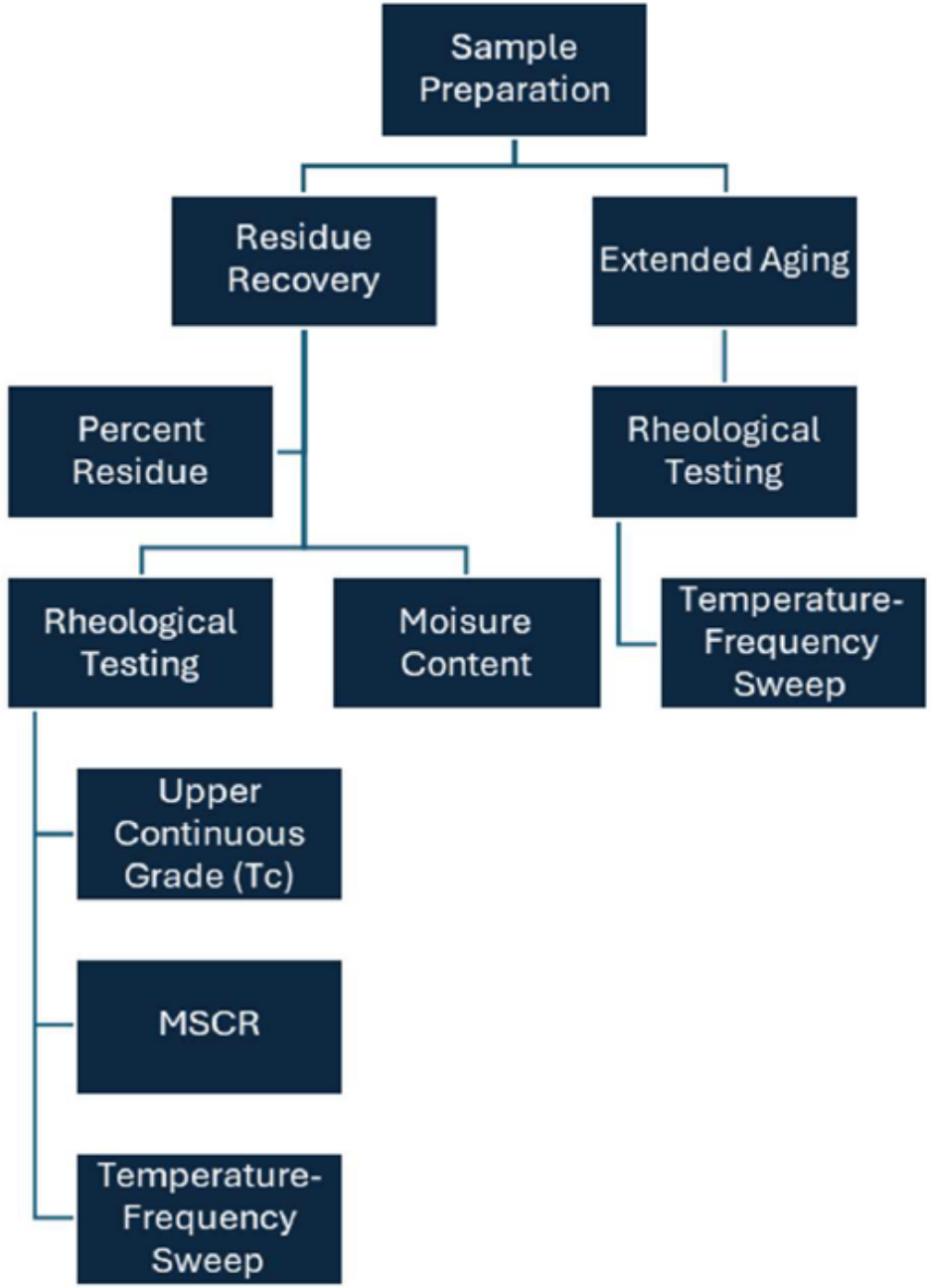
Interlaboratory Study

Interlaboratory Study (ILS) – Overview

- Eight (8) volunteer labs
  - Three (3) from DOTs
  - Two (2) from private industry
  - Two (2) from industry research institutions
  - One (1) from academia
- Dataset was reduced to 6 labs due to equipment issues at 2 volunteer labs

Interlaboratory Study (ILS) – Test Plan

- Test plan included:
  - Residue recovery and aging
  - Determination of % residue and moisture content of binder
  - Rheological testing of recovered residues
  - Rheological testing of aged residues



Interlaboratory Study (ILS) – Overview

- Primary goals of ILS
  - Evaluation of a modified version of AASHTO R 78 Procedure B (i.e. Procedure C Iteration #2) as a viable means of moisture removal and residue recovery for emulsified asphalts
  - Examination of statistical variance in high and low temperature rheological testing of recovered emulsified asphalt residues before and after extended oven aging
  - Assessment of test procedures in a production environment through participant feedback

Interlaboratory Study (ILS) – Overview

- Each lab was supplied:
  - Emulsified asphalts
  - Watt meter for oven verification
  - Test kit
  - Instructions
  - Spreadsheet for data reporting

Material	Description
CMS-2	Unmodified medium-setting cationic emulsified asphalt
CMS-2L	SBR-modified medium-setting cationic emulsified asphalt[1]
CMS-2P	SBS-modified medium-setting cationic emulsified asphalt[2]

[1]SBR: styrene-butadiene rubber; [2]SBS: styrene-butadiene-styrene

Procedure C – Iteration #2

- Use readily available silicone muffin trays
  - Diameter of ~3”
- Application method is similar to 1st iteration
  - i.e. use of silicone spatula to achieve film coverage

Laboratory Aging Process – Procedure C + 20hrs

Procedure C (6 hours, 60°C

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After 6 hours remove material for HT/MC testing

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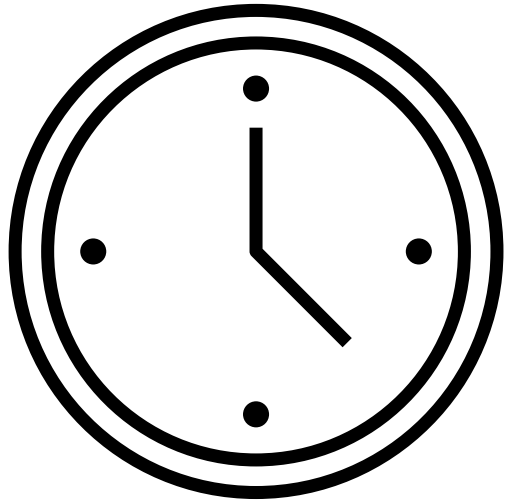
Return to 60°C oven for 20 additional hours



Laboratory Aging Process – Procedure C + 20hrs

Procedure C (6 hours, 60°C → After 6 hours remove material for HT/MC testing → Return to 60°C oven for 20 additional hours → Remove aged residue for IT testing

Interlaboratory Study (ILS) – Key Findings

- Low-temperature recovery – Procedure C
  - Low moisture content values of residue indicate that the procedure is viable in other laboratories besides Asphalt Institute

Lab ID	Moisture Content of Recovered Residue, %		
	CMS-2	CMS-2L	CMS-2P
1	32.56	32.86	32.76
2	1.37	1.29	1.18
4	0.66	0.54	0.72
5	0.58	0.57	0.29
6	0.37	0.23	0.19
7	0.13	0.01	0.10
Avg.	0.62	0.53	0.50

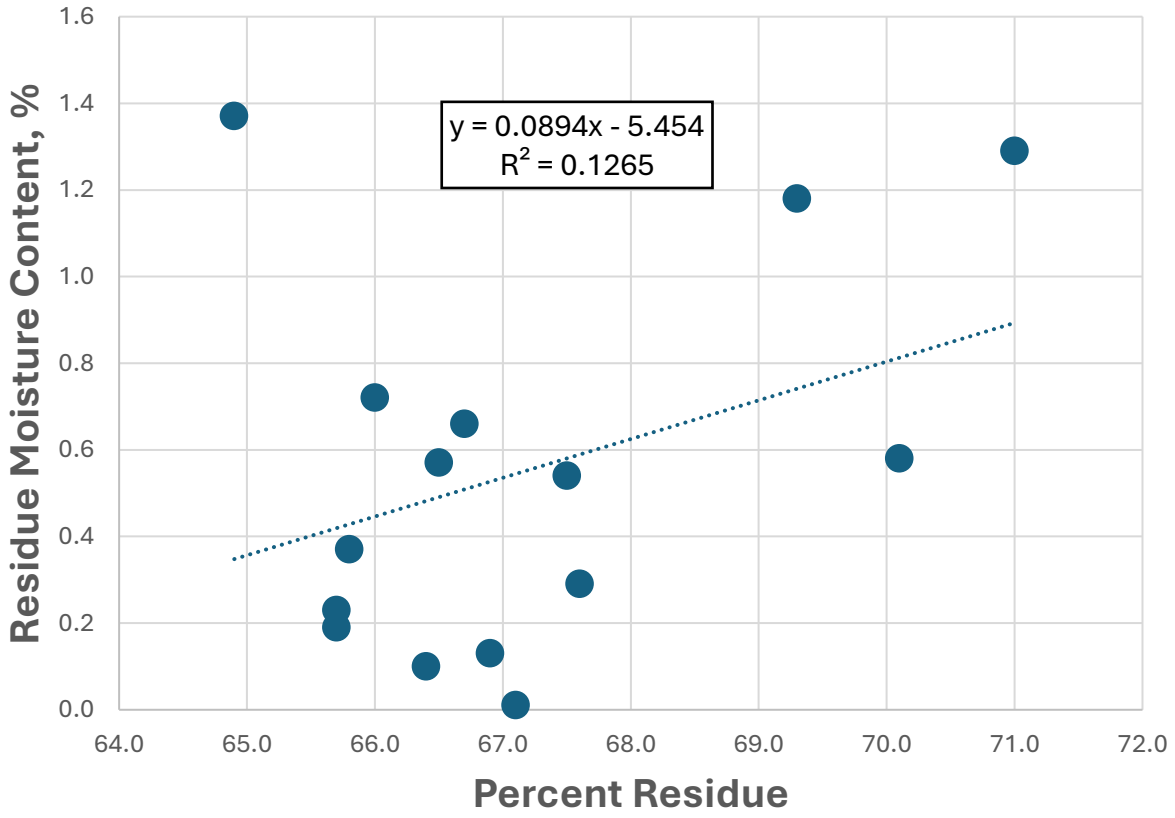
Interlaboratory Study (ILS) – Key Findings

- Low-temperature recovery – Procedure C
  - Low moisture content values of residue indicate that the procedure is viable in other laboratories besides Asphalt Institute
  - Percent residue variability illustrates need for continued training and oven verification/ruggedness testing

Statistical Measure	Percent Residue, %		
	CMS-2	CMS-2L	CMS-2P
Count	6	6	6
Mean	66.6	66.3	67.0
1s	1.9	3.7	1.3
3s	5.6	11.0	4.0
1s%	2.8	5.5	2.0
d2s	5.3	10.3	3.7
d2s%	8.0	15.6	5.6
Max.	70.1	71.0	69.3
Min.	64.9	59.8	65.7

Interlaboratory Study (ILS) – Key Findings

- Low-temperature recovery – Procedure C
  - Some error may be attributed to simple measures such as tray transport, mass determination, etc.



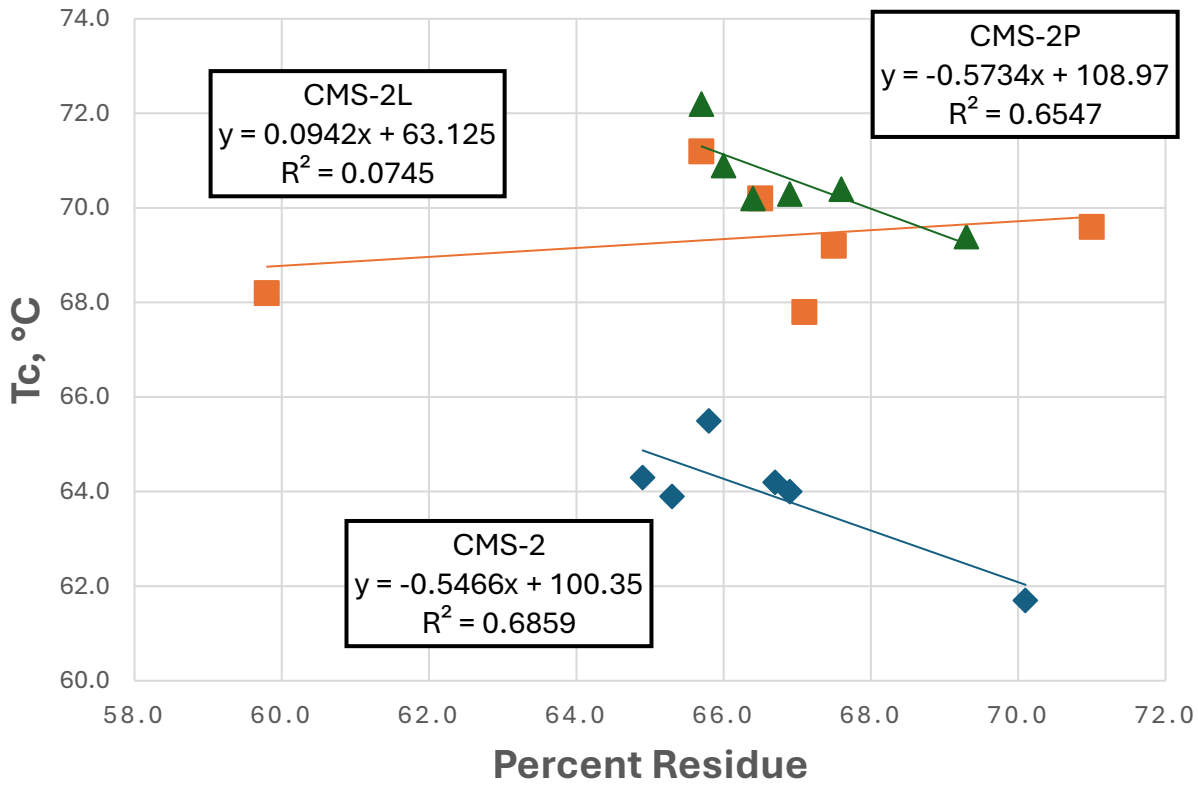
Interlaboratory Study (ILS) – Key Findings

- High temperature rheology
  - Testing within linear viscoelastic range showed good reproducibility, but the same can not be said for non-LVE test

Statistical Measure	Tc, °C			$Jnr_{3.2}$, kPa^{-1}			$R_{3.2}$, %		
	CMS-2	CMS-2L	CMS-2P	CMS-2	CMS-2L	CMS-2P	CMS-2	CMS-2L	CMS-2P
Count	6	6	6	6	6	5	-	6	5
Mean	63.9	69.4	70.6	4.725	2.717	1.396	-	8.5	44.1
1s	1.2	1.3	0.9	0.789	0.765	0.141	-	4.5	6.6
3s	3.7	3.8	2.8	2.367	2.296	0.423	-	13.4	19.8
1s%	1.9	1.8	1.3	16.7	28.2	10.1	-	52.6	15.0
d2s	3.5	3.6	2.6	2.233	2.166	0.399	-	12.6	18.7
d2s%	5.5	5.1	3.8	47.3	79.7	28.6	-	148.8	42.4
Max.	65.5	71.2	72.2	5.631	3.692	1.592	-	15.5	50.4
Min.	61.7	67.8	69.4	3.371	1.572	1.274	-	3.4	34.2

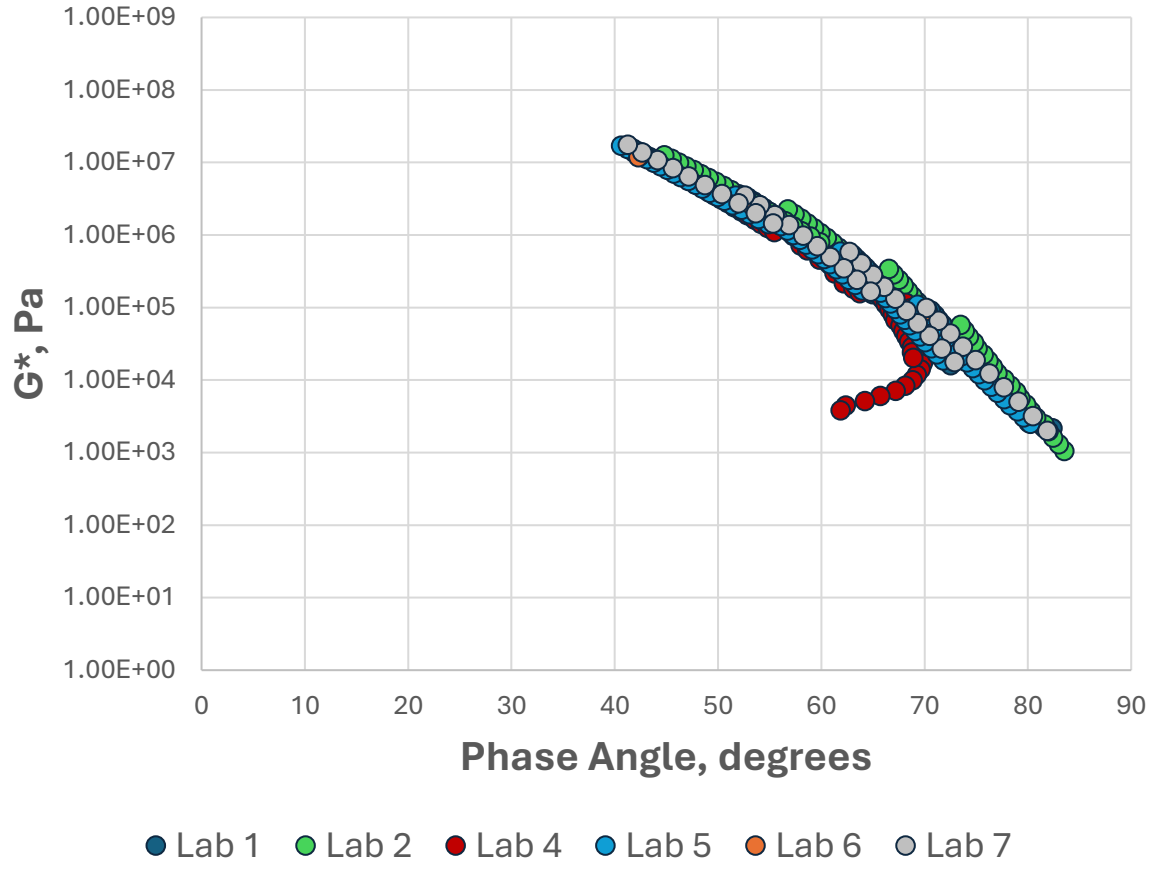
Interlaboratory Study (ILS) – Key Findings

- High temperature rheology
  - General correlation was seen between T_c and percent residue for CMS and CMS-2P, but CMS-2L showed no trend
  - May speak to higher variability in recovery and/or material sensitivity to temperature and handling



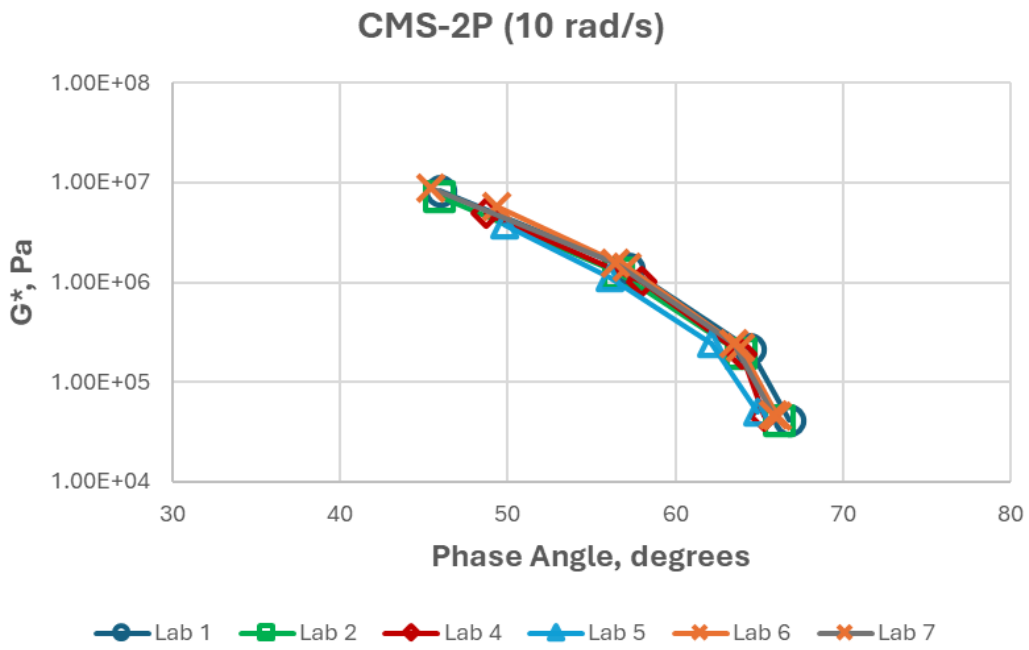
Interlaboratory Study (ILS) – Key Findings

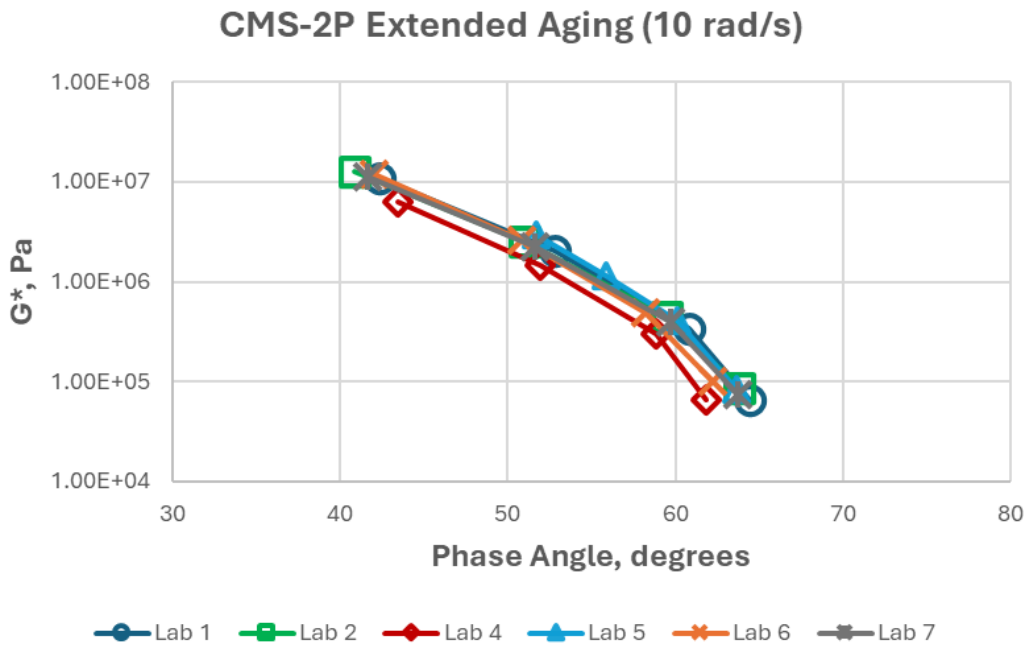
- Intermediate temperature rheology
  - Lab 4 had some testing issues



Interlaboratory Study (ILS) – Key Findings

- Intermediate temperature rheology
  - 10 rad/s isochrones distinguish material variability in aging





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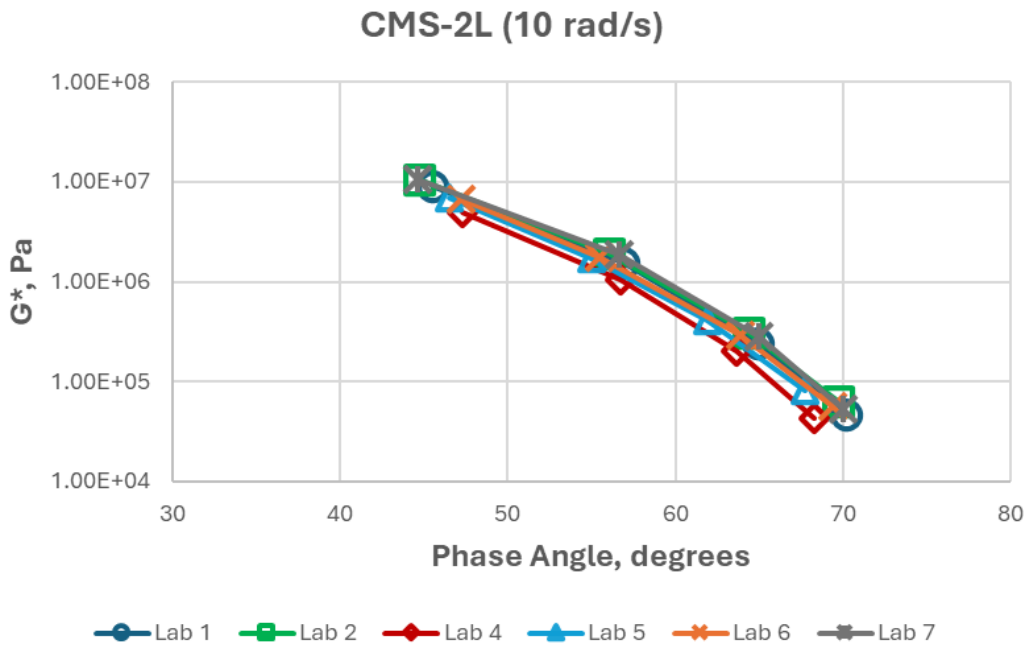
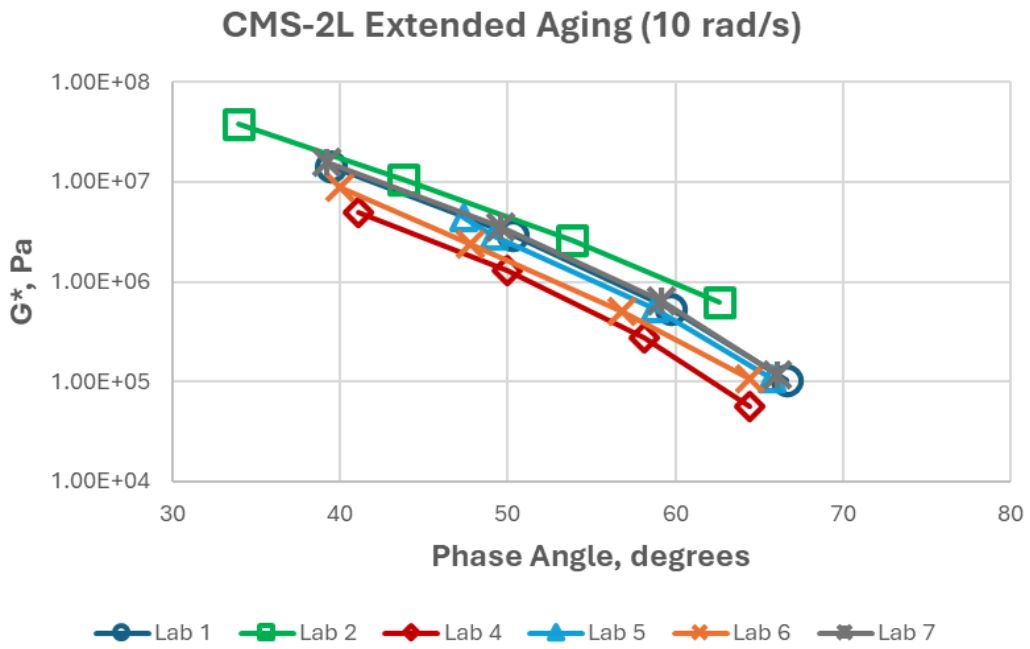
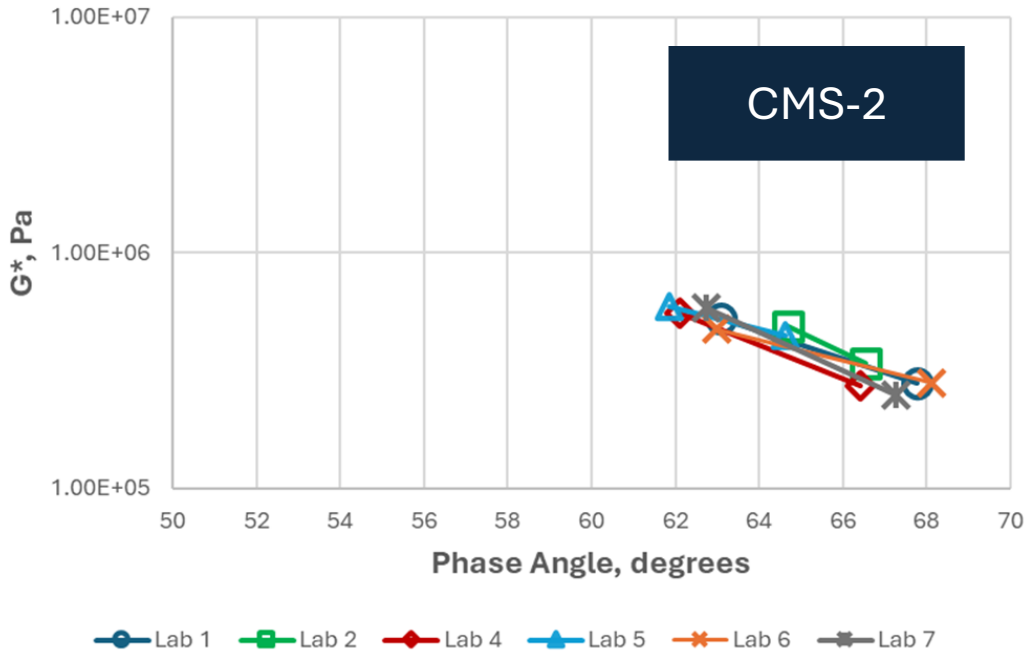
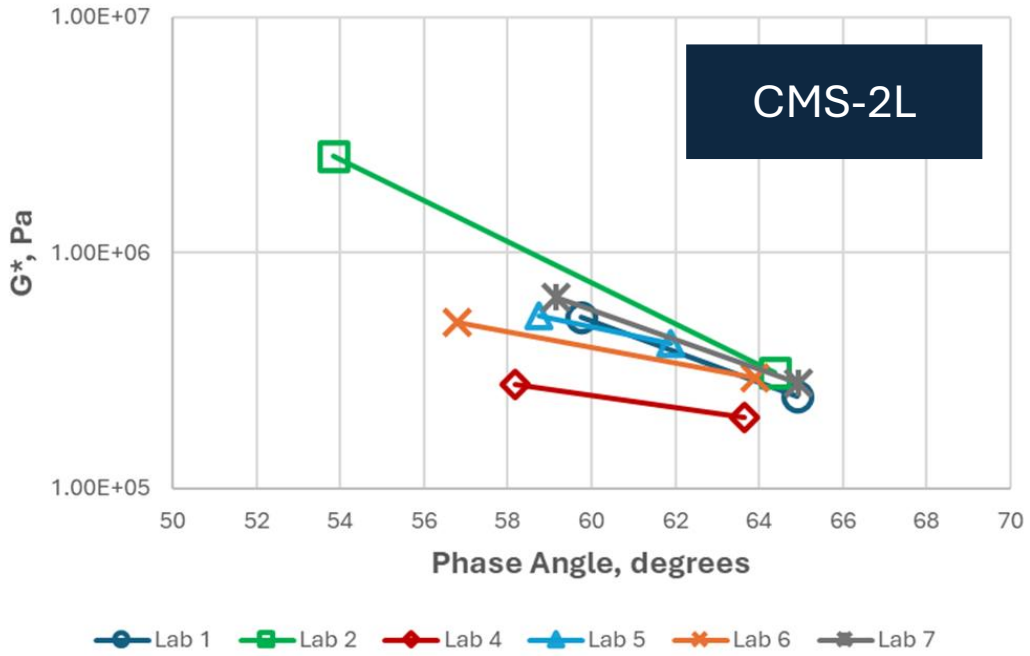




Interlaboratory Study (ILS) – Key Findings

- Intermediate temperature rheology
  - Aging profiles highlight challenges with SBR testing and aging





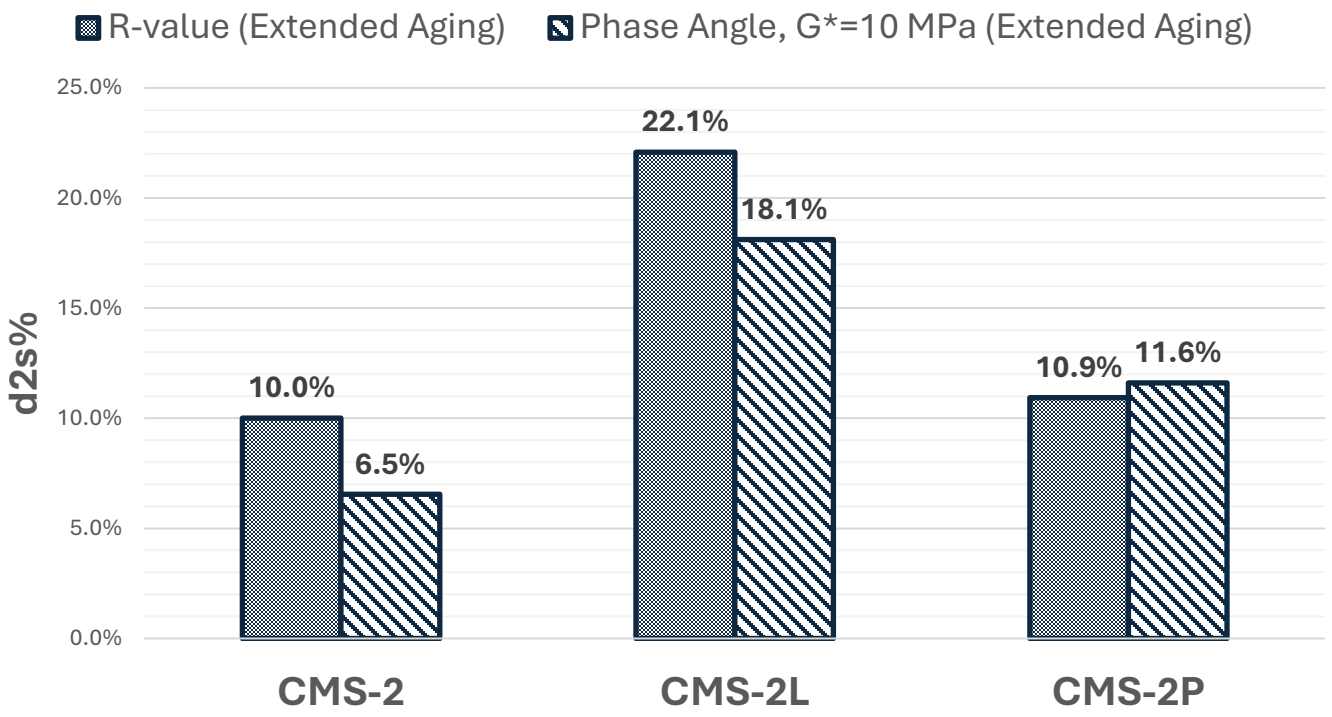
Interlaboratory Study (ILS) – Key Findings

- Intermediate temperature rheology
  - GRP variability is higher than standard fatigue parameter, which was expected

Parameter	Condition	Stated or measured d2s%			
		CMS-2	CMS-2L	CMS-2P	Other
G*sin(δ), AASTHO T315	PAV[1]	-	-	-	27.4
GRP, 35°C, 10 rad/s	Mod. AASHTO R78 Proc. B + Extended Aging	51.6	63.4	58.6	-
GRP, 25°C, 10 rad/s		43.5	66.1	61.5	-
GRP, 15°C, 10 rad/s		60.1	80.8	103.6	-
GRP, 5°C, 10 rad/s		76.7	119.2	76.6	-

Interlaboratory Study (ILS) – Key Findings

- Intermediate temperature rheology
  - Shape parameters followed material trends



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Final Thoughts and Next Steps

What's next?

- **Moving toward a specification**
  - Framework is there but need to continue to refine through benchmarking
- **Gain further clarity on extended aging**
  - Test older residues to add to dataset
- **ILS Follow-up**
  - Collect feedback
  - Use ILS as launch point for education and training of industry professionals

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Thank You!

QUESTIONS?