Solvent Extraction-Recovery Procedures and their Effect on Recovered Asphalt Properties

Ontario Case Study

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ONTARIO ASPHALT PAVEMENT COUNCIL

CENTRE FOR PAVEMENT AND TRANSPORTATION TECHNOLOGY

Research Objective

Compare physical properties and testing variability of original (tank) asphalt to recovered asphalt

Methodology

- Five industry labs across Ontario and one lab in the US participated in the mini interlaboratory study (ILS).
- Each lab received 7 sets of liquid asphalt cement (tank asphalt) and an asphalt mix produced with that asphalt.
- When the asphalt mix contained RAP, RAP material was sampled separately at the hot mix plant during mix production for testing
- Each lab received instructions outlining which procedures to follow
- Labs were instructed to use reagent grade trichloroethylene (TCE) for solvent extraction
- The specification at the time they study was conducted allowed the use of both Abson and Rotavapor recovery method. Only one lab used the Abson method.
- Recovered asphalt samples were treated as RTFO-aged since they had already gone through plant production.

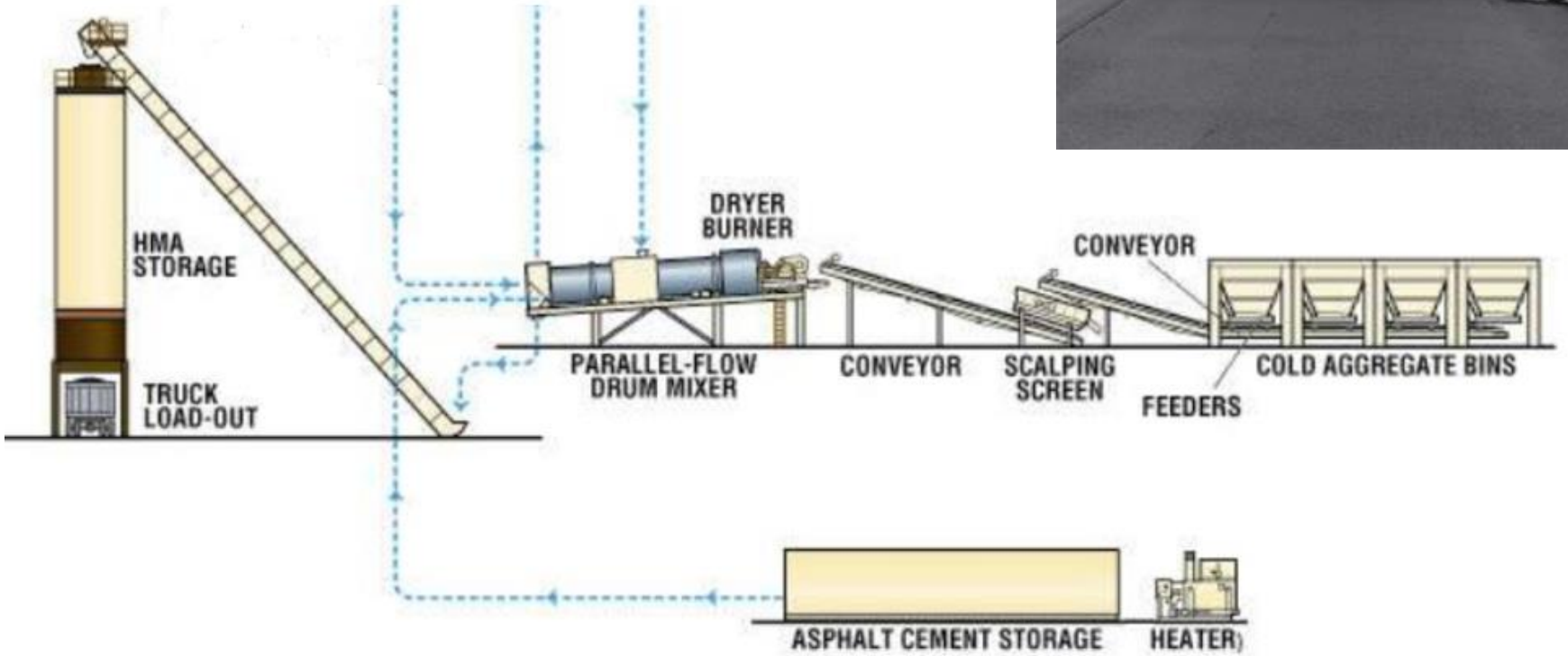
Test Methods

- MTO LS-227: Ash content
- AASHTO R29: PGAC continuous/true grading
- AASHTO T 350: Multiple Stress Creep Recovery (MSCR) Test
  - Test samples at 58^{o}C
- MTO LS-299: Double Edge Notch Tension (DENT) Test
- MTO LS-308: Extended Bending Beam Rheometer (ExBBR) Test
- MTO LS-228: Accelerated Aging of Asphalt Cement Using Modified Pressure Aging Vessel Protocols – Method C

Sampling Locations

Sample D – Job Site (Recovered)

Sample C – Plant Mix (Performance Testing)

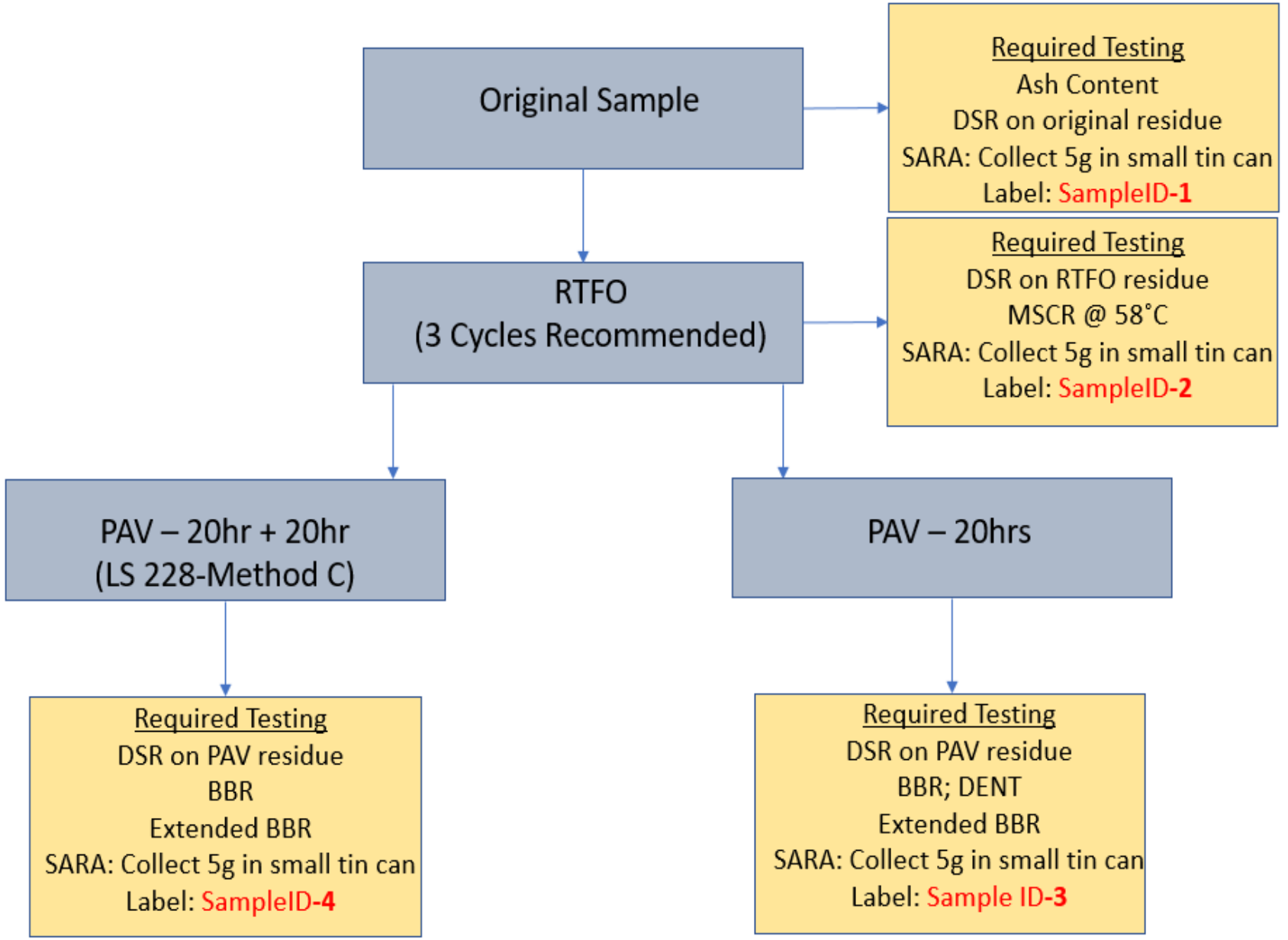


Sample A - Terminal

Sample B – Tank Asphalt

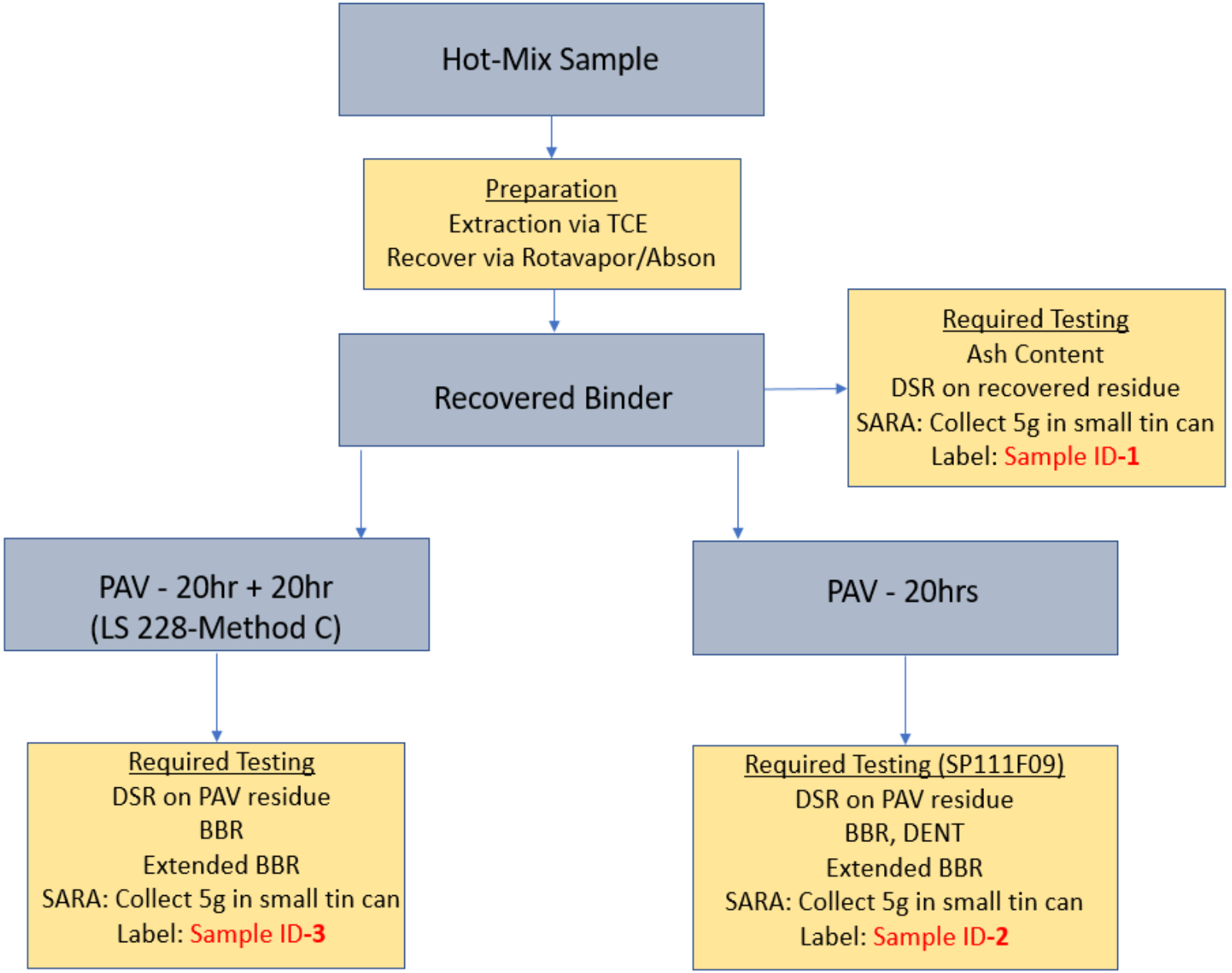
Testing Flow Chart: Sample B Tank Asphalt

Not all labs performed all test procedures. This is reflected in “Sample Size” later in summary of findings.



Testing Flow Chart: Sample D Recovered Asphalt

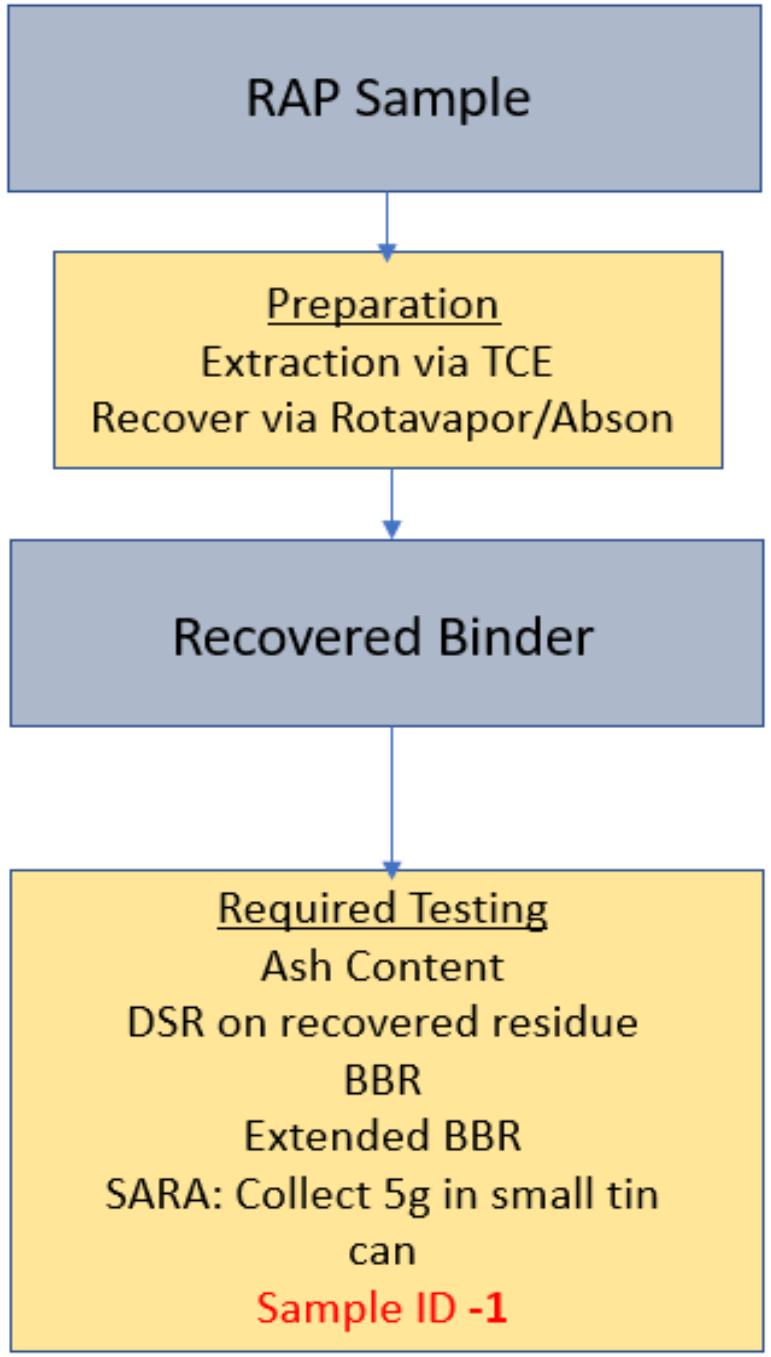
Not all labs performed all test procedures. This is reflected in “Sample Size” later in summary of findings.



Testing Flow Chart:

Recovered RAP Binder

Not all labs performed all test procedures. This is reflected in “Sample Size” later in summary of findings.



Asphalt Materials Collected

HMA Mix Class	PG Grade	RAP Content
HL1	70-28	0
12.5FC2	70-28	15
12.5	58-34	15
12.5	58-34	0
12.5	58-28	0
12.5FC2	64-28	0
12.5FC1	58-34	0

Summary of Findings: Part I

Tables show average values and standard deviations of the measure parameters for the various PG grades sampled as an evaluation of the ILS.

Asphalt mixes that contained RAP material are segregated to the tank asphalt to asphalt recovered from a virgin mix and a RAP mix.

A final summary table shows the %change (increase/decrease) in standard deviations or testing variability for each PG grade included in the ILS.

Charts show the individual measured results and are compared to the current Ontario Provincial Specification, OPSS.PROV 1101 (November 2014) and SSP 111F09 (August 2018)

PG 58-28

Tank	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa-1)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
Average	**0.1**	**59.8**	**-34.3**	**2.2**	**2.7**	**-30.2**	**13.8**
Min	0.05	58.9	-35.4	2.0	2.2	-31.2	9.7
Max	0.11	60.6	-33.0	2.4	4.0	-28.9	17.9
StDev	**0.0**	**1.2**	**1.2**	**0.1**	**0.9**	**1.0**	**3.5**
COV	0.0	2.0	3.5	4.5	33.3	3.2	25.4
Sample Size	4	2	3	4	4	4	4

Recovered Virgin Mix	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa-1)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
Average	**2.6**	**58.2**	**-35.5**	**4.8**	**5.3**	**-29.1**	**8.6**
Min	1.32	51.5	-37.8	1.3	3.5	-30.0	1.5
Max	4.11	64.8	-34.1	8.7	8.2	-27.5	14.7
StDev	**1.2**	**9.4**	**2.0**	**3.7**	**2.1**	**1.4**	**6.6**
COV	44.7	16.2	5.6	77.5	40.6	4.9	76.8
Sample Size	4	2	3	4	4	3	4

PG 58-28

Summary

	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa-1)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
StDev (Tank)	0	1.2	1.2	0.1	0.9	1.0	3.5
StDev (Rec - 0R)	1.2	9.4	2	3.7	2.1	1.4	6.6
% Change StDev	**100**	**87**	**40**	**97**	**57**	**29**	**47**

Asphalt mix did not contain RAP.

PG 64-28

Summary

	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa-1)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
StDev (Tank)	0	0.7	1.9	0.1	0.4	0.9	6.1
StDev (Rec - 0R)	2.7	9	0.5	0.3	2.8	3.4	1.9
% Change StDev	**100**	**92**	**-280**	**67**	**86**	**74**	**-221**

Asphalt mix did not contain RAP.

PG 64-28

Summary

	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa-1)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
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PG 64-28

Tank	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa-1)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
Average	**0.1**	**65.4**	**-35.3**	**0.3**	**3.5**	**-30.4**	14.0
Min	0.04	64.9	**-37.1**	0.2	3.1	-31.4	6.4
Max	0.1	65.9	**-33.3**	0.4	3.9	-29.2	21.2
StDev	0.0	0.7	**1.9**	0.1	0.4	0.9	6.1
COV	45.2	1.1	5.4	25.7	11.1	3.0	43.8
Sample Size	4	2	3	4	4	4	4

Recovered Virgin Mix	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa-1)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
Average	**5.0**	**76.3**	**-31.6**	**0.4**	**5.5**	**-25.1**	6.7
Min	1.68	69.9	**-32.2**	0.1	3.9	-28.7	4.9
Max	7.8	82.6	**-31.2**	0.8	9.7	-20.5	8.9
StDev	2.7	9.0	**0.5**	0.3	2.8	3.4	1.9
COV	54.7	11.8	1.6	80.9	50.5	13.6	28.0
Sample Size	4	2	3	4	4	4	4

PG 64-28

Tank	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa-1)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
Average	**0.1**	**65.4**	**-35.3**	**0.3**	**3.5**	**-30.4**	**14.0**
Min	0.04	64.9	-37.1	0.2	3.1	-31.4	**6.4**
Max	0.1	65.9	-33.3	0.4	3.9	-29.2	**21.2**
StDev	0.0	0.7	1.9	0.1	0.4	0.9	**6.1**
COV	45.2	1.1	5.4	25.7	11.1	3.0	43.8
Sample Size	4	2	3	4	4	4	4

Recovered Virgin Mix	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa-1)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
Average	**5.0**	**76.3**	**-31.6**	**0.4**	**5.5**	**-25.1**	**6.7**
Min	1.68	69.9	-32.2	0.1	3.9	-28.7	**4.9**
Max	7.8	82.6	-31.2	0.8	9.7	-20.5	**8.9**
StDev	2.7	9.0	0.5	0.3	2.8	3.4	**1.9**
COV	54.7	11.8	1.6	80.9	50.5	13.6	28.0
Sample Size	4	2	3	4	4	4	4

PG 58-34

Summary

	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa-1)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
StDev (Tank)	0.2	1.1	1.6	0.2	1.1	2.5	6.7
StDev (Rec - 0R)	1.8	0.8	1.3	0.6	1.2	2.3	4.2
StDev (Rec - 15R)	0.6	3.5	3.5	0.5	1.4	4.4	3.7
% Change StDev (0R)	**89**	**-38**	**-23**	**67**	**8**	**-9**	**-60**
% Change StDev (15R)	**67**	**69**	**54**	**60**	**21**	**43**	**-81**

PG 58-34 was sampled with a 0% RAP mix and a 15% RAP mix for comparison.

Standard deviation of test results for PG 58-34 both increased and decreased depending on the parameter.

Standard deviation of test results were mostly higher for mixes containing 15% RAP.

PG 58-34

Tank	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa-1)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
Average	**0.2**	**62.8**	**-37.4**	**0.6**	**3.3**	**-33.1**	**25.5**
Min	0.07	61.6	-39.9	0.4	1.5	-36.8	15.8
Max	0.65	65.0	-35.4	1.0	4.7	-27.8	37.4
StDev	**0.2**	**1.1**	**1.6**	**0.2**	**1.1**	**2.5**	**6.7**
COV	86.8	1.8	4.2	30.4	31.7	7.7	26.3
Sample Size	12	8	10	12	12	12	12

Recovered Virgin Mix	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa-1)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
Average	**3.0**	**67.4**	**-37.6**	**0.7**	**4.9**	**-32.2**	**12.1**
Min	1.10	66.9	-39.1	0.2	2.7	-34.9	7.0
Max	6.70	68.5	-36.0	1.9	6.4	-28.1	20.8
StDev	**1.8**	**0.8**	**1.3**	**0.6**	**1.2**	**2.3**	**4.2**
COV	59.6	1.1	3.4	82.9	24.3	7.1	35.0
Sample Size	8	4	6	7	8	8	8

PG 58-34

Tank	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa-1)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
Average	**0.2**	**62.8**	**-37.4**	0.6	3.3	**-33.1**	**25.5**
Min	0.07	61.6	-39.9	0.4	1.5	-36.8	15.8
Max	0.65	65.0	-35.4	1.0	4.7	-27.8	37.4
StDev	0.2	1.1	1.6	0.2	1.1	2.5	6.7
COV	86.8	1.8	4.2	30.4	31.7	7.7	26.3
Sample Size	12	8	10	12	12	12	12

Recovered RAP Mix	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa-1)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
Average	**2.4**	**70.9**	**-33.5**	0.6	6.6	**-23.4**	**4.5**
Min	1.68	68.0	-36.4	0.2	4.6	-28.1	-0.1
Max	3.0	75.5	-29.7	1.3	8.0	-18.0	8.2
StDev	**0.6**	**3.5**	**3.5**	**0.5**	**1.4**	**4.4**	**3.7**
COV	23.4	4.9	10.3	77.7	21.7	18.7	83.1
Sample Size	4	4	3	4	4	4	4

PG 70-28

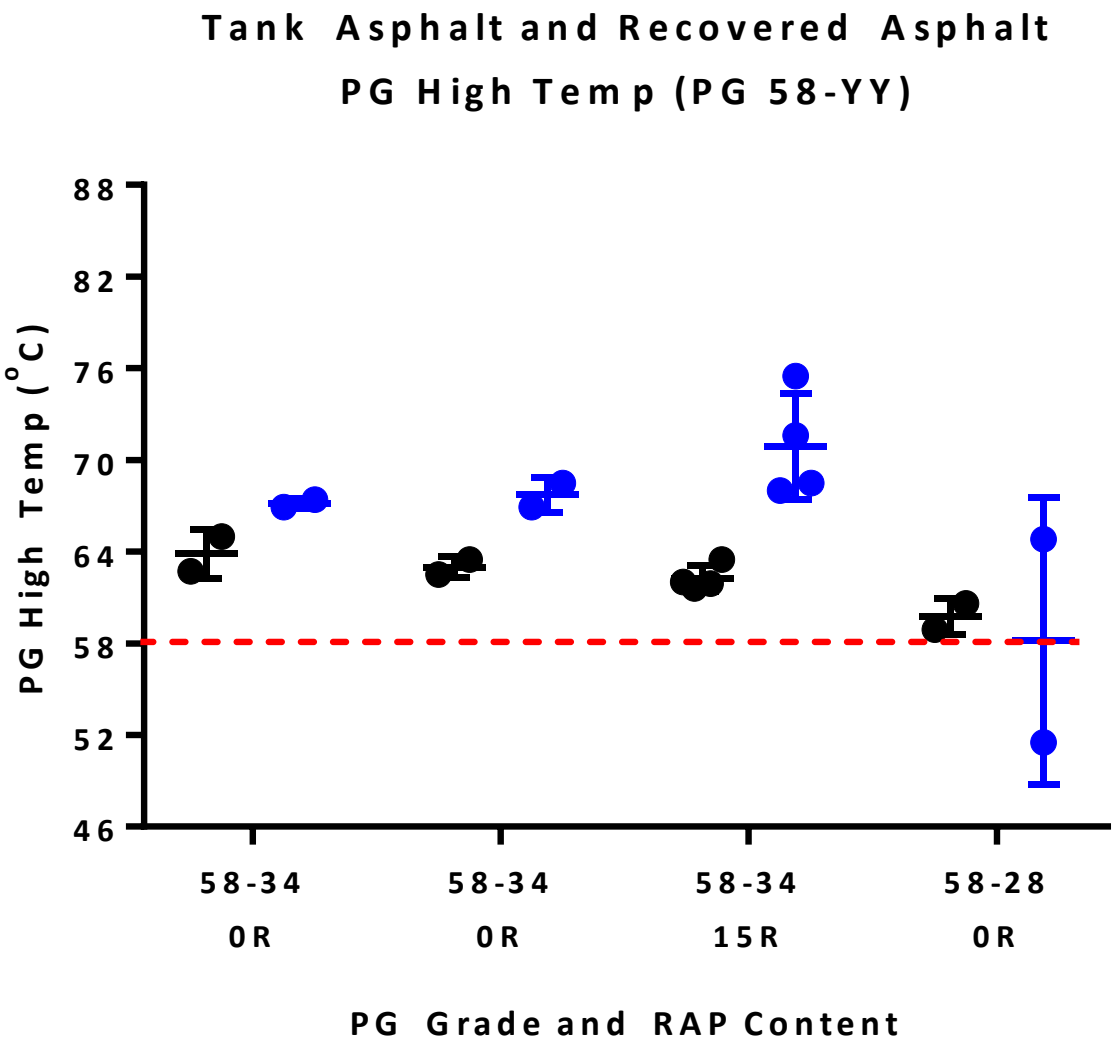
Summary

	Ash (%)	PG High (°C)	PG Low (°C)	MSCR Jnr (3.2kPa-1)	Grade Loss (°C)	LTLG (°C)	CTOD (15°C, mm)
StDev (Tank)	0.0	1.4	0.6	0.0	1.2	1.5	6.6
StDev (Rec - 0R)	0.7	2.3	1.3	0	0.6	3.7	1.6
StDev (Rec - 15R)	0.9	6.3	10.6	0	3.8	4.8	1.9
% Change StDev (0R)	**100**	**39**	**54**	**-**	**-100**	**59**	**-313**
% Change StDev (15R)	**100**	**78**	**94**	**-**	**68**	**69**	**-247**

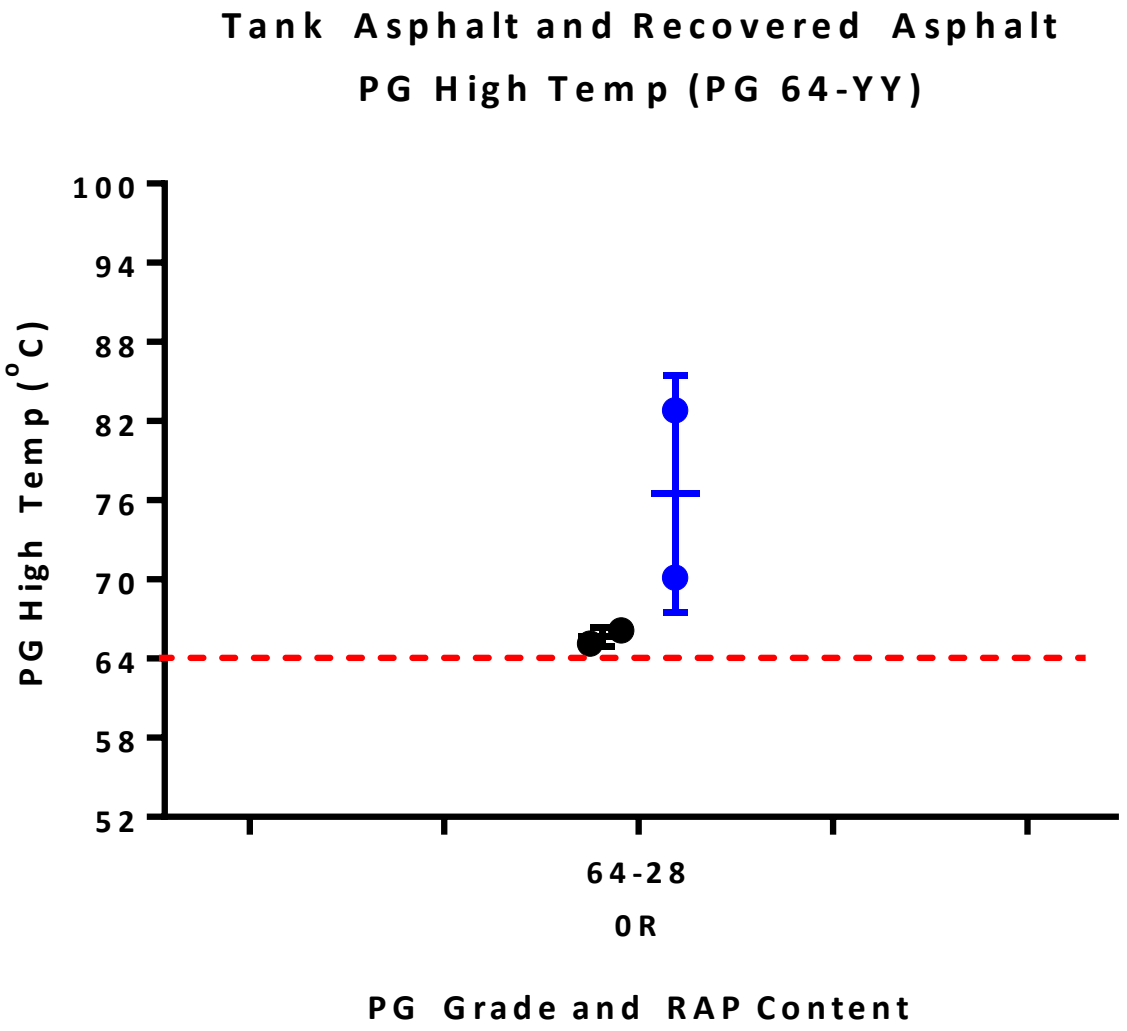
PG 70-28 was sampled with a 0% RAP mix and a 15% RAP mix for comparison.

Standard deviation of test results for PG 70-28 both increased and decreased depending on the measured parameter.

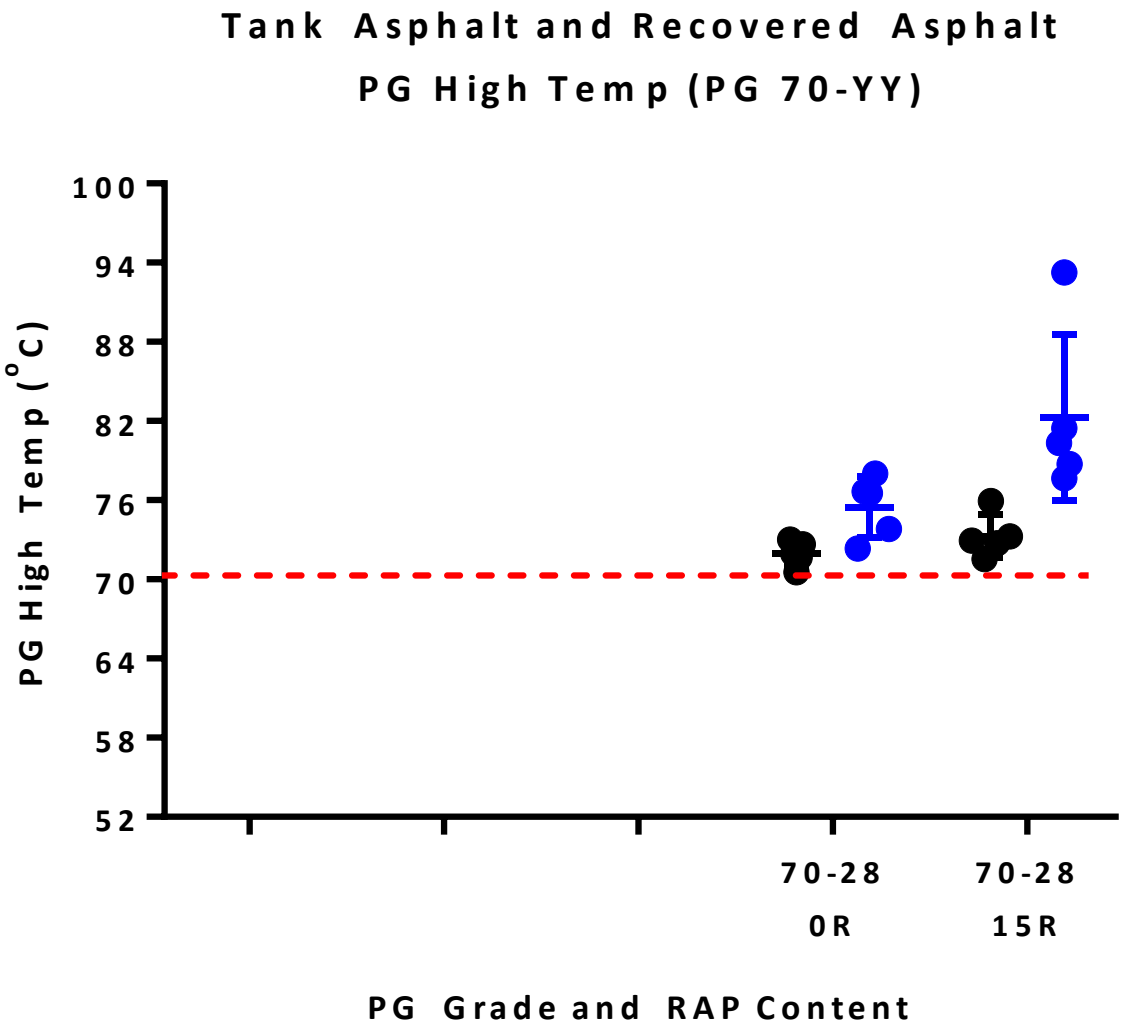
Standard deviation of test results were mostly higher for mixes containing 15% RAP.



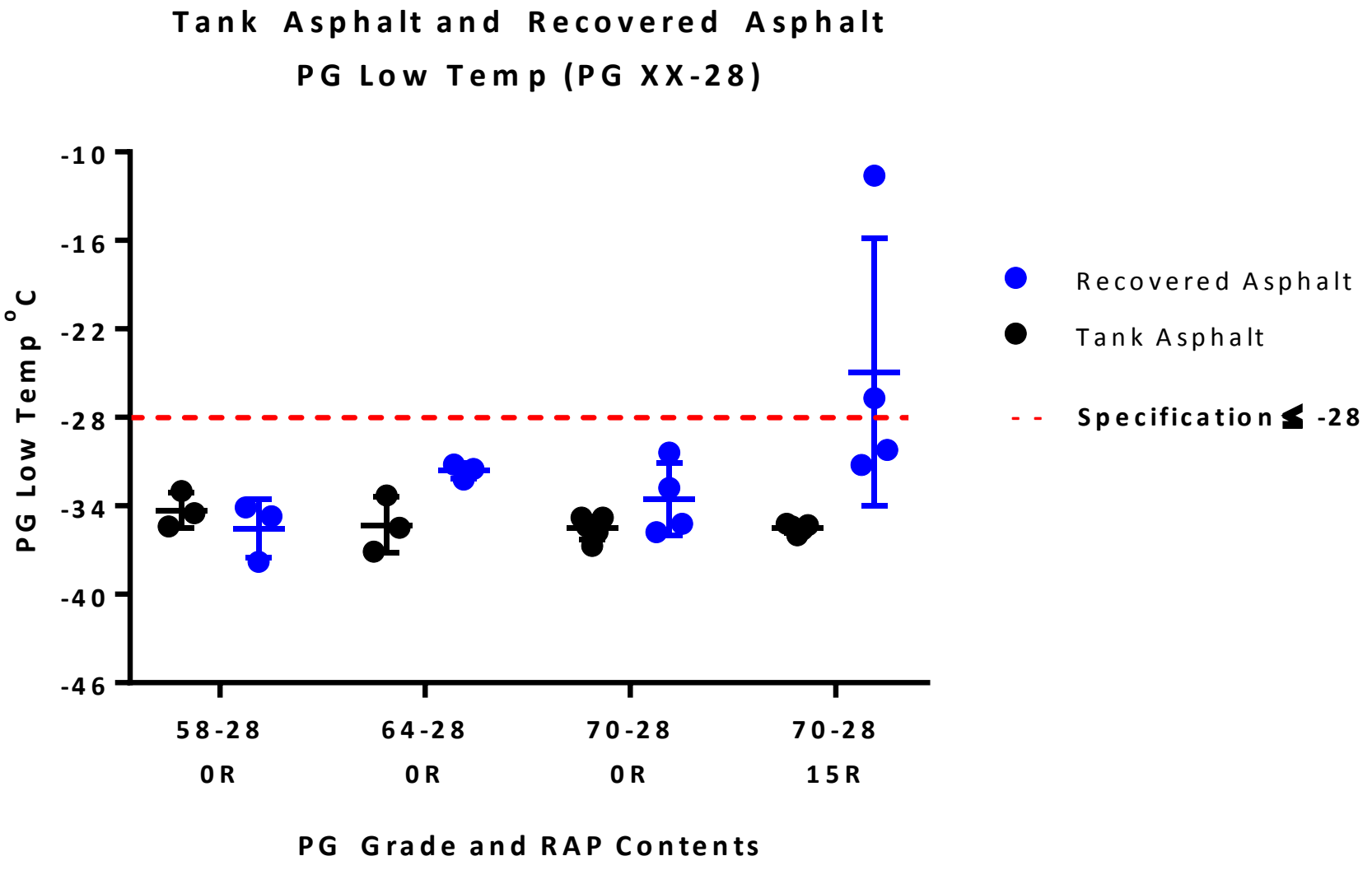
0% of tank samples failed
10% recovered asphalt failed



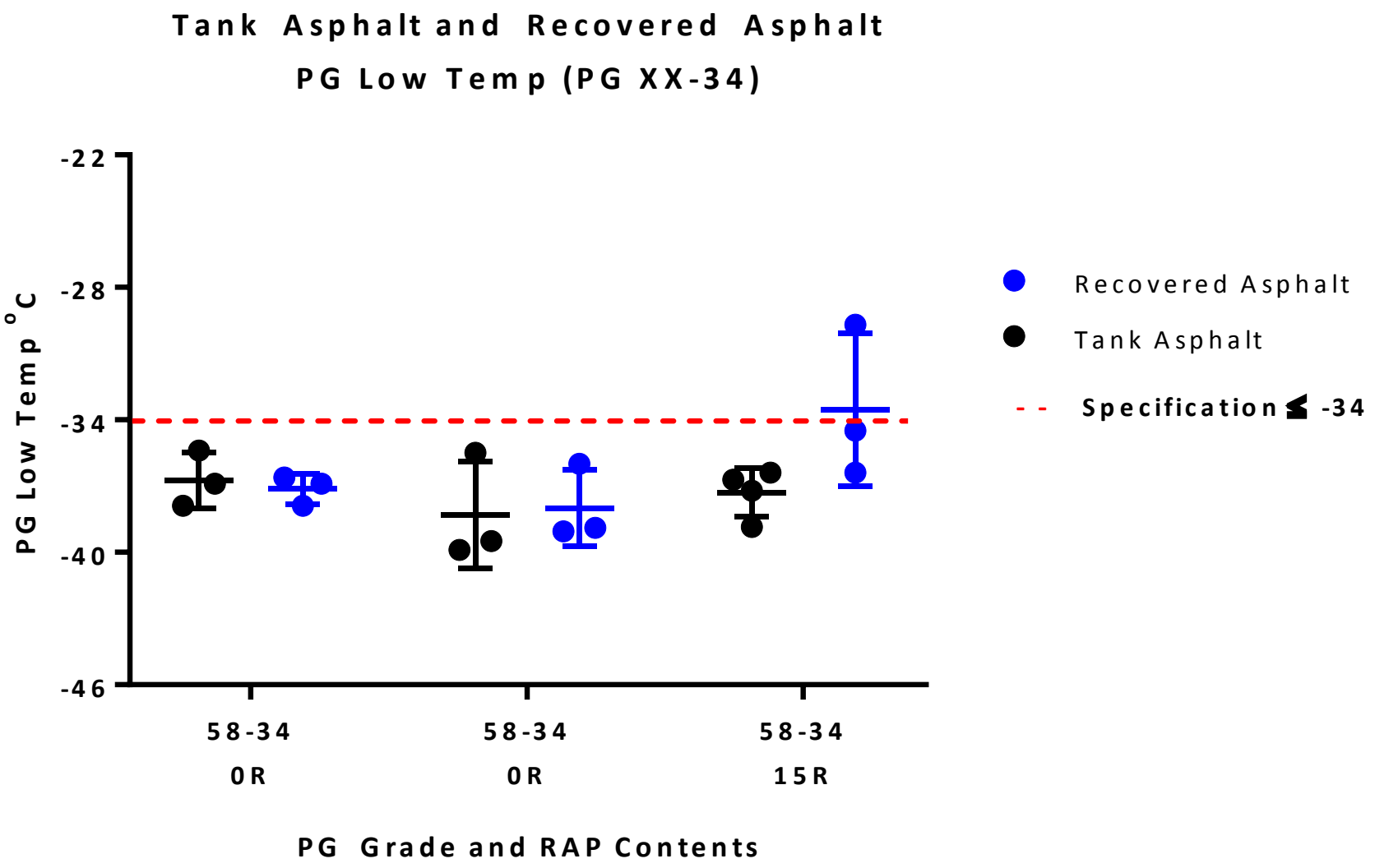
0% of tank samples failed
0% recovered asphalt failed



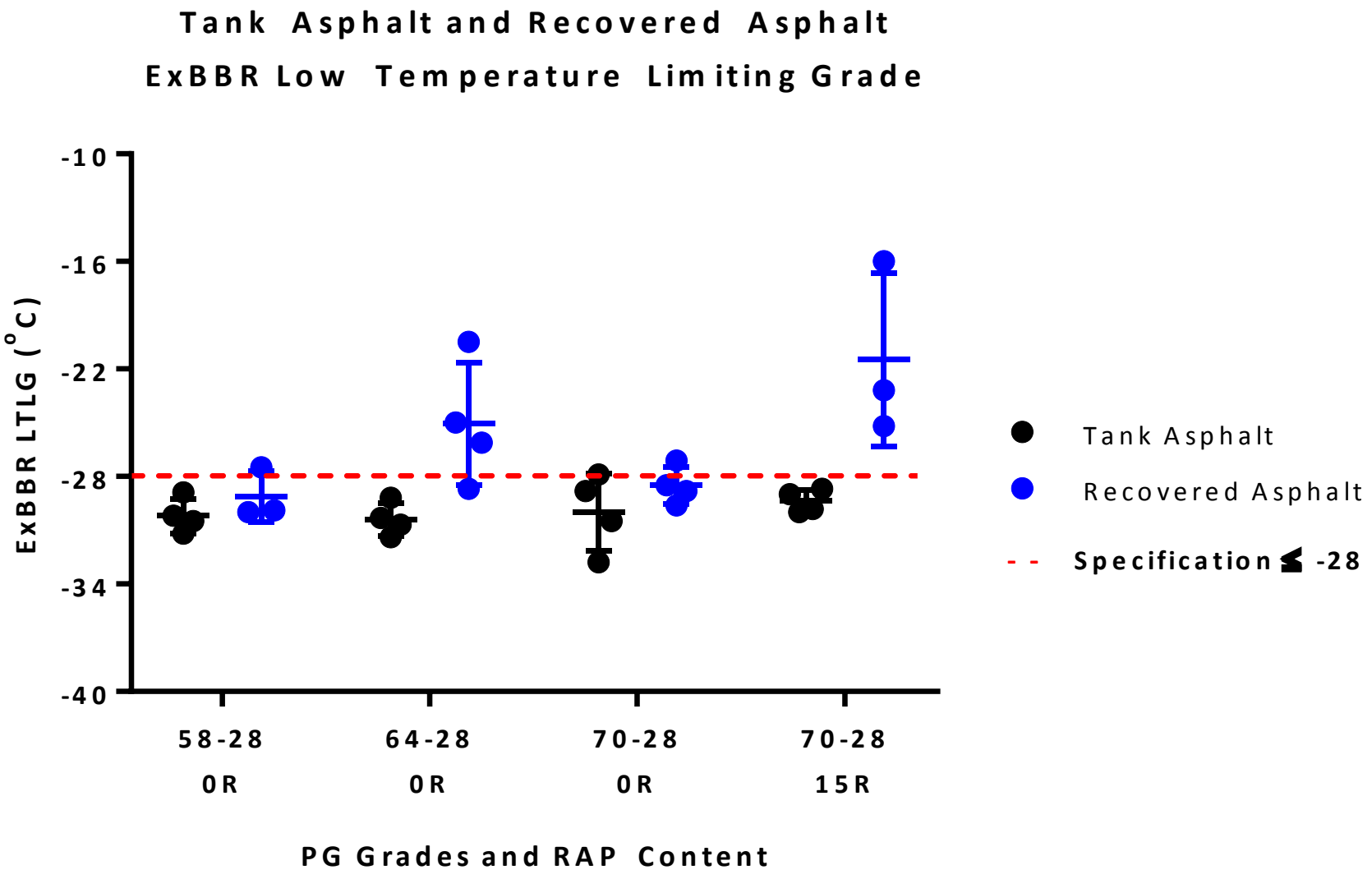
0% of tank samples failed
0% recovered asphalt failed



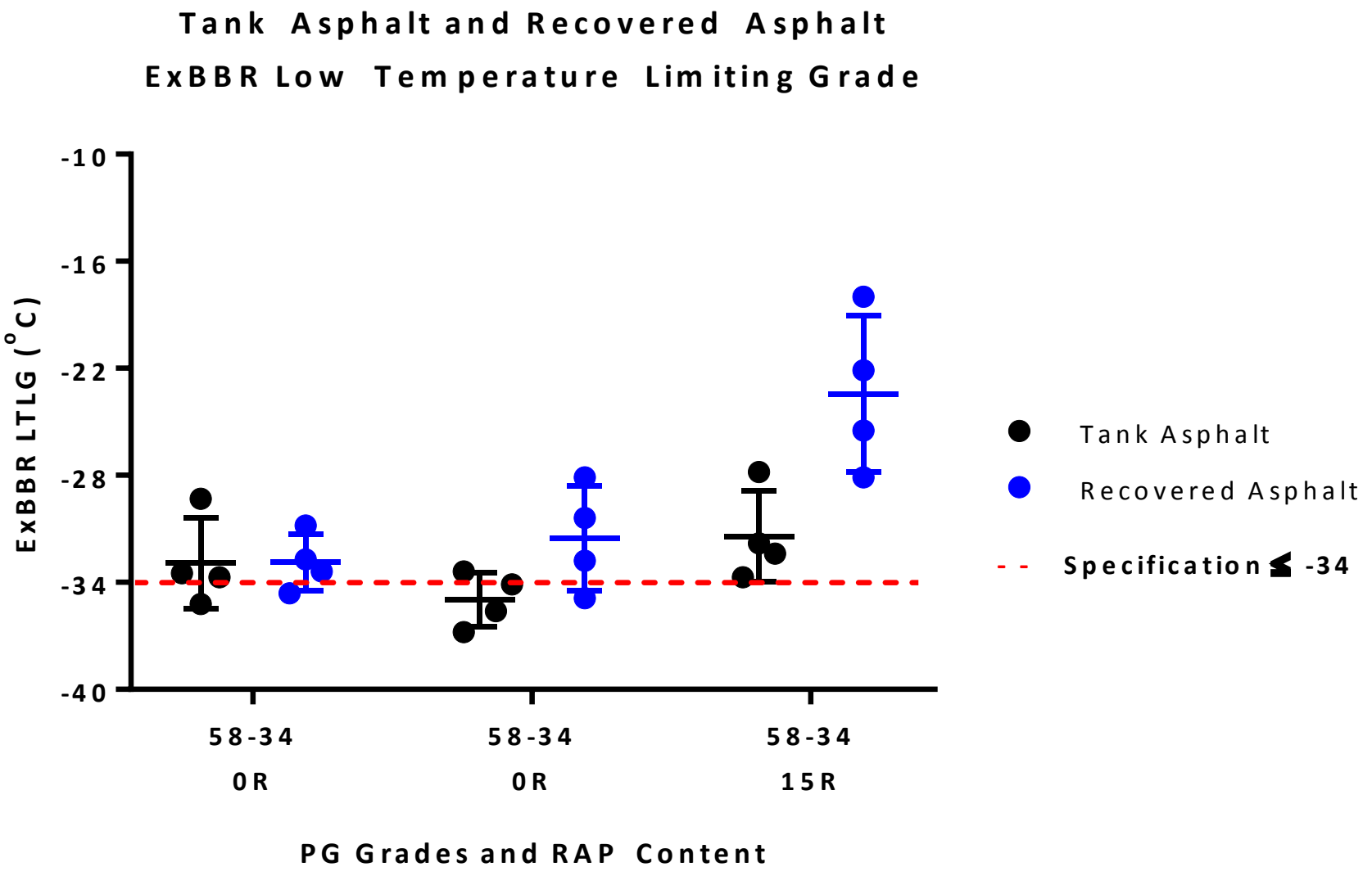
0% of tank samples failed
14 % recovered asphalt failed



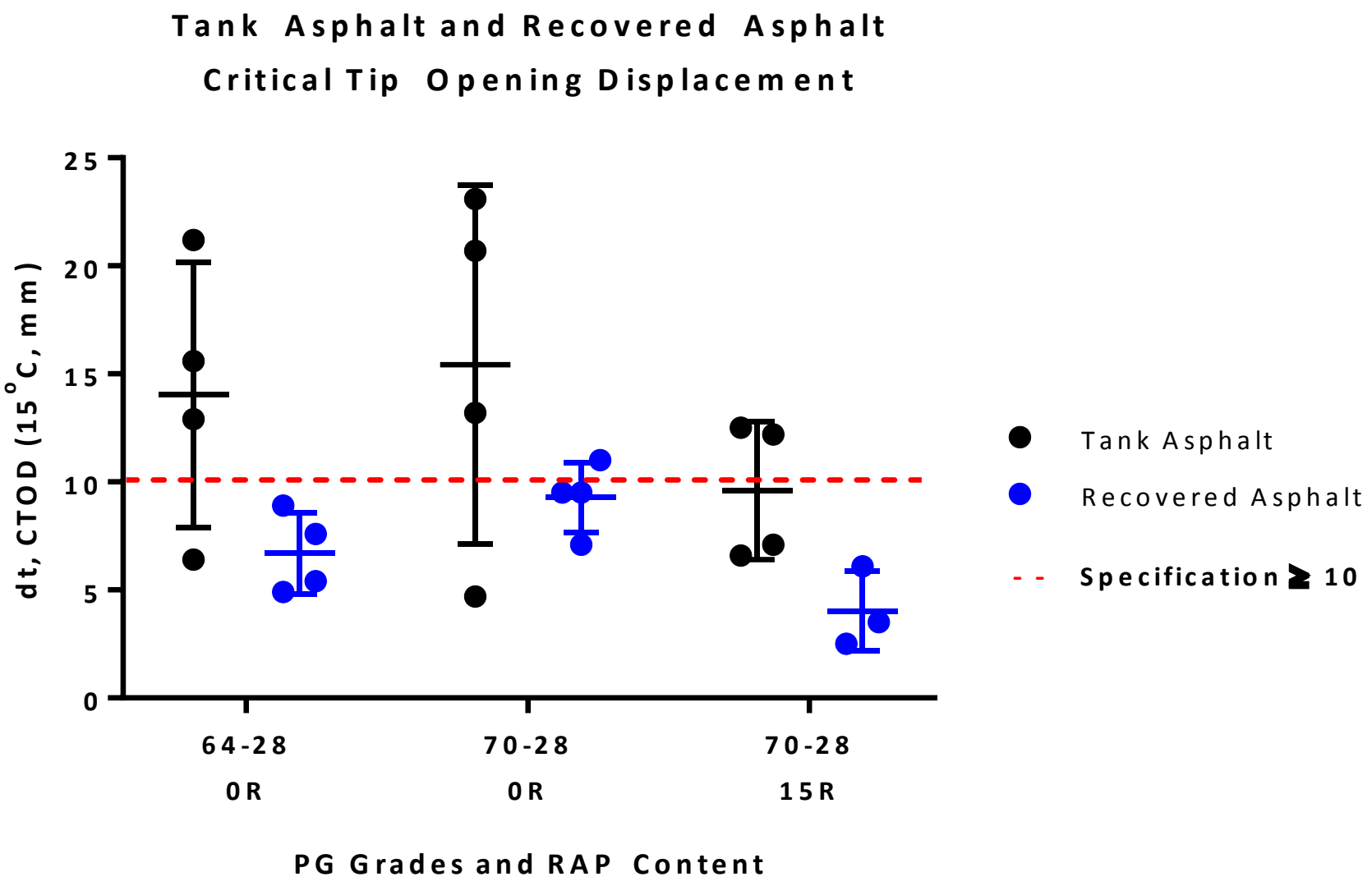
0% of tank samples failed
11% recovered asphalt failed



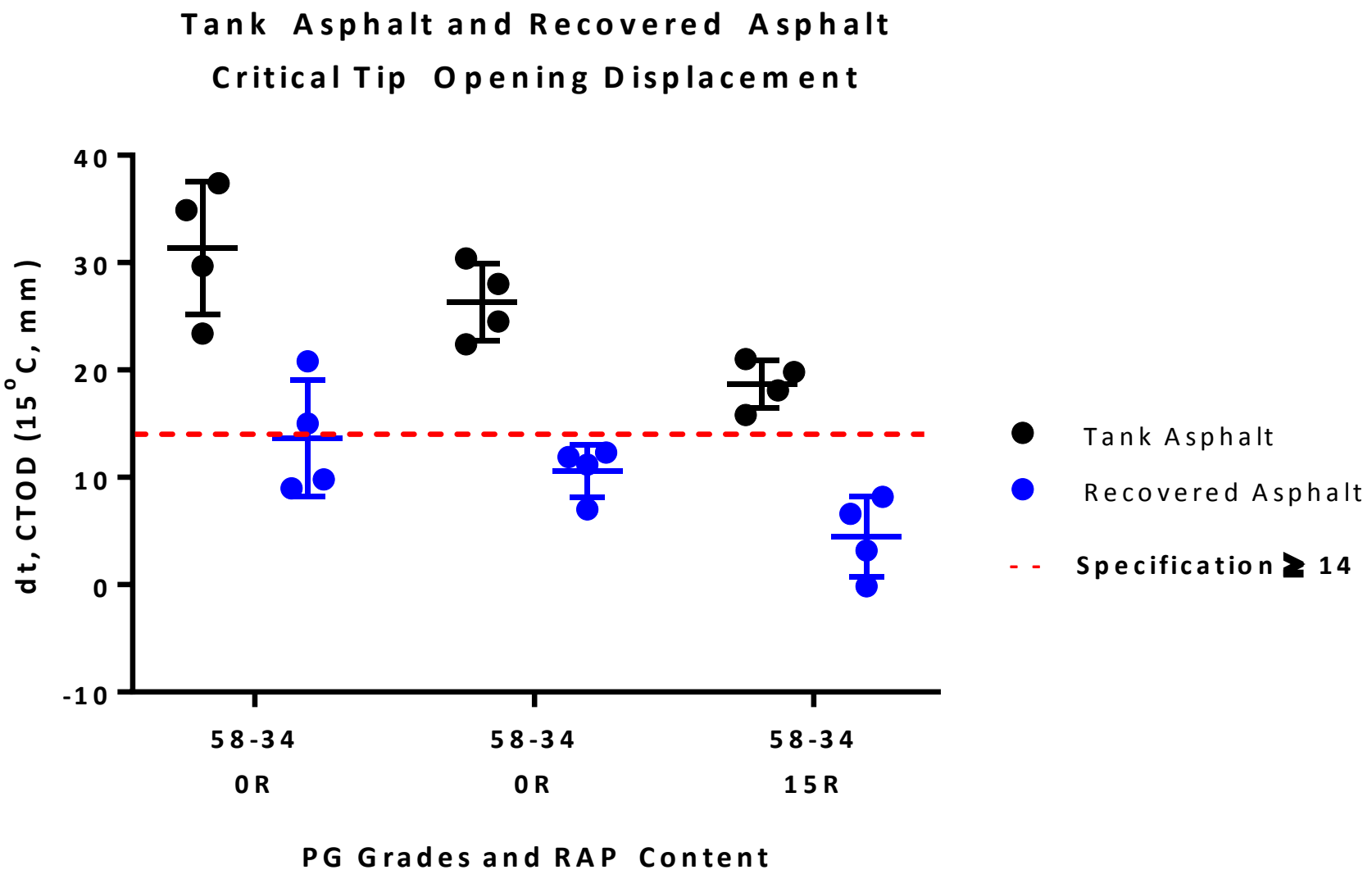
6% of tank samples failed
57% recovered samples failed



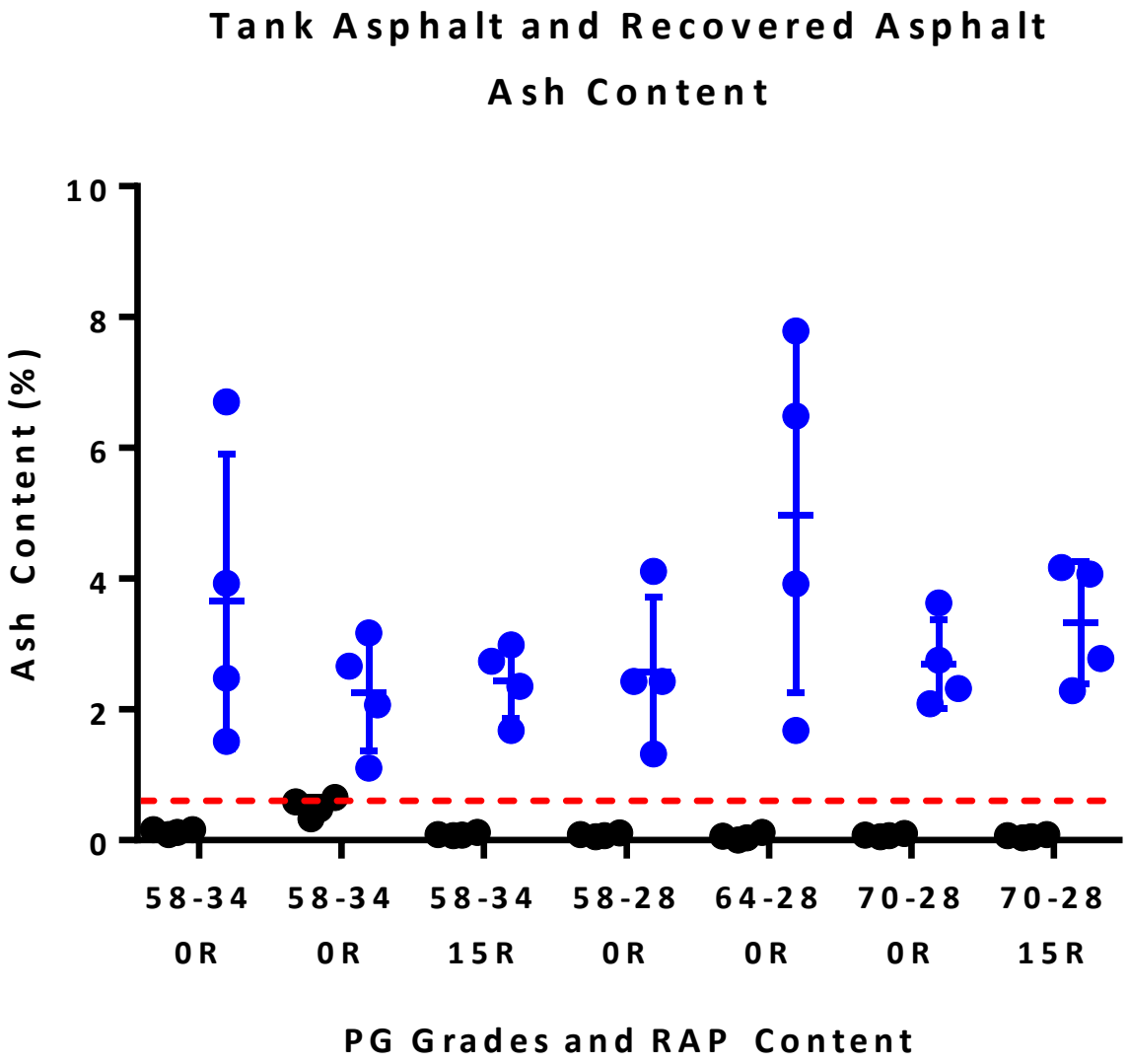
67% of tank samples failed
83% recovered samples failed



33% of tank samples failed
90% recovered samples failed



0% of tank samples failed
83% recovered samples failed

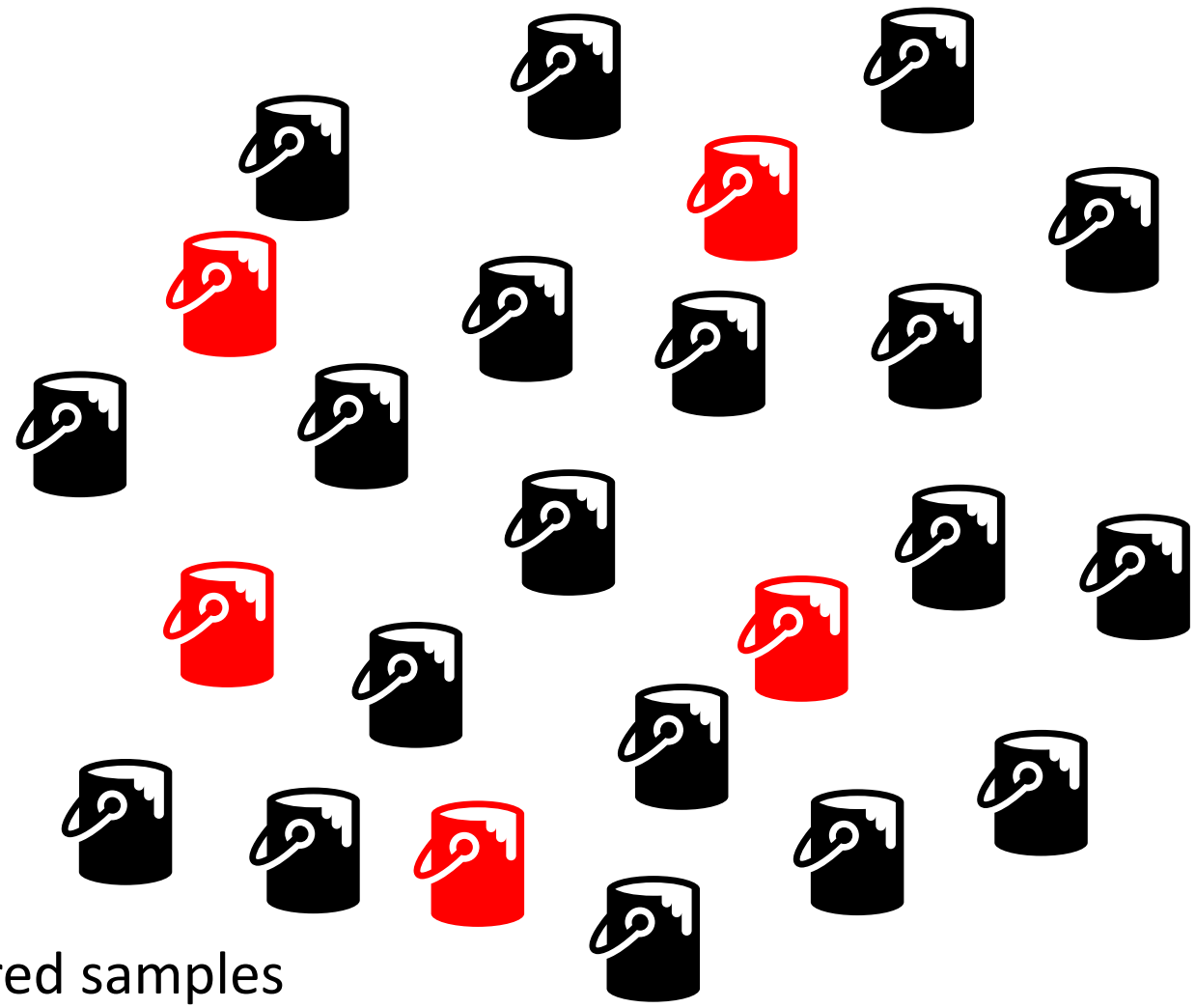


0% of tank samples failed
100% recovered asphalt failed

Summary of Findings: Part II
Gas Chromatography

Gas Chromatography is used to detect presence of solvent in recovered asphalt.

Removal of solvent is critical for obtaining valid test results.



No solvent detected – 90% of recovered samples

Presence of solvent – 10% of recovered samples

Summary of Findings: Part III Aging Index

Asphalt is composed of extremely large number of organic molecules grouped into:

- Saturates, Aromatics, Resins, Asphaltenes

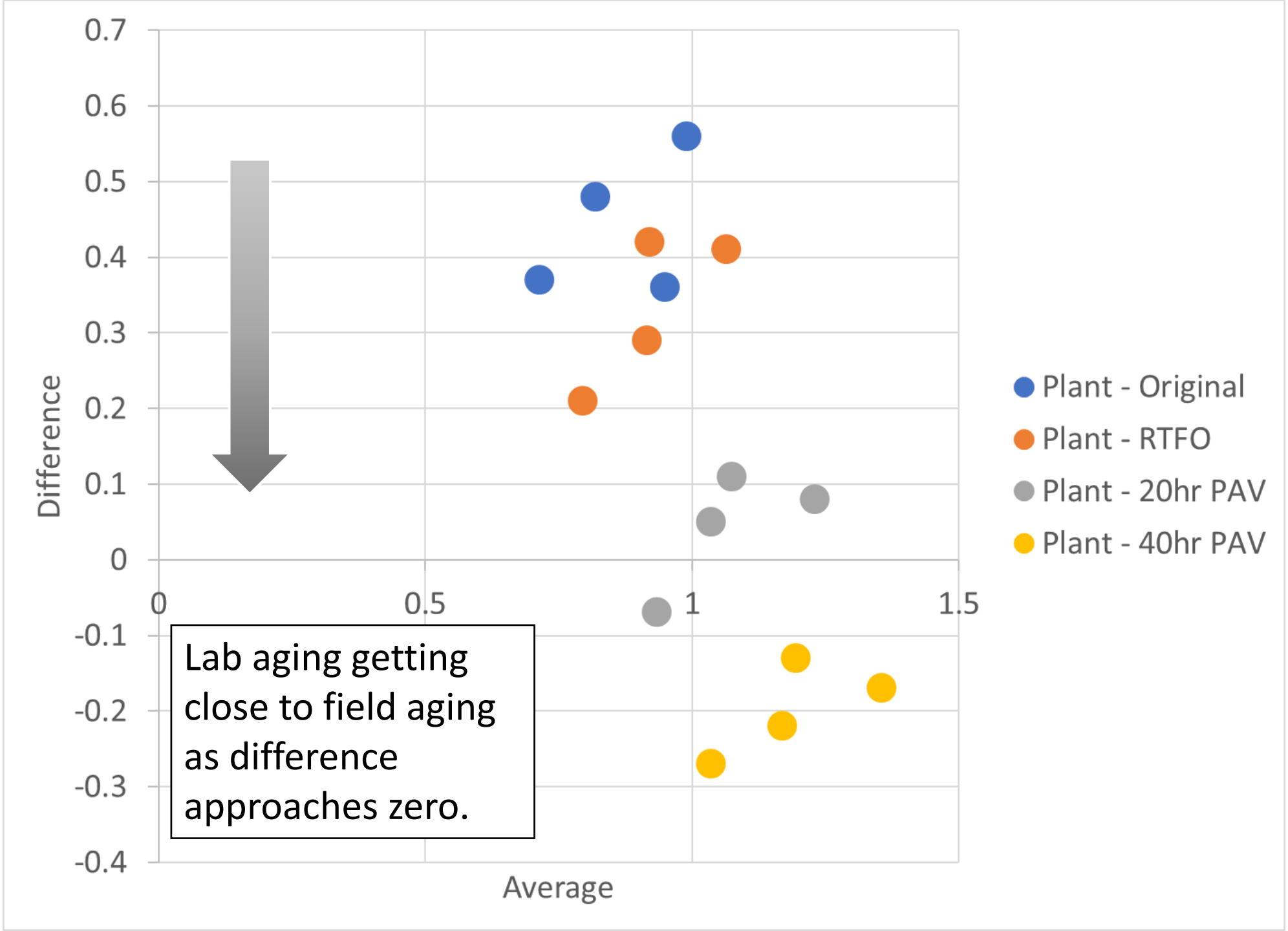
- SARA analysis uses liquid chromatography separating the material into different classes based on level of polarity

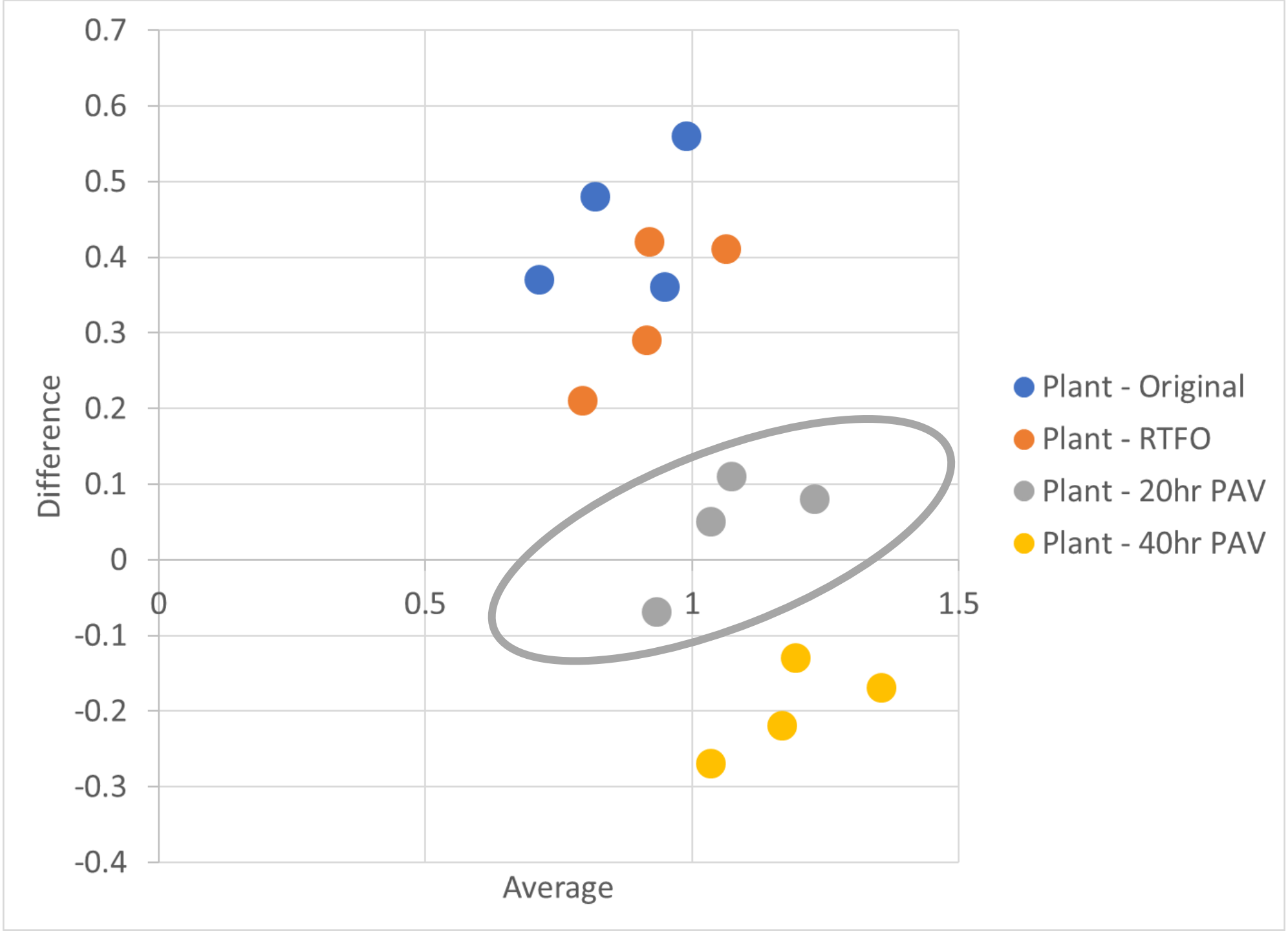
Summary of Findings: Part III
Aging Index (AI)

$$\text{Absorbance AI} = \frac{\text{Toluene Soluble Asphaltenes}}{\text{Resins}}$$

Aging index calculated using data from SAR-AD® on asphalt binders collected after various stages of aging in lab and field:

- RTFO **lab** aged binder
- 20hr PAV **lab** aged binder
- 40hr PAV **lab** aged binder
- Plant Mix (Recovered binder from mix that is **field** short term aged)


0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
-0.1
-0.2
-0.3
-0.4
Difference
0
0.5
1
1.5
Average
Lab aging getting close to field aging as difference approaches zero.
Plant - Original
Plant - RTFO
Plant - 20hr PAV
Plant - 40hr PAV


0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
-0.1
-0.2
-0.3
-0.4
Difference
0
0.5
1
1.5
Average
Plant - Original
Plant - RTFO
Plant - 20hr PAV
Plant - 40hr PAV

Summary

- Standard deviation on recovered asphalt binder are greater in general than the same values measured on tank asphalt binder.
- During recovery aggregate fines remains in the recovered asphalt that will affect the physical properties
- During recovery solvent left in the recovered asphalt as detected in the gas chromatography affects the physical properties
- Asphalt aging in the field is more severe than current lab aging protocols.