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**COMMITTEE D02 ON PETROLEUM PRODUCTS, LIQUID FUELS, AND LUBRICANTS**

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Unapproved Minutes of the November 15, 2017  
Sequence IV Surveillance Panel Meeting.

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The meeting was called to order by Chairman Buscher at 2:00 PM Central Time.

A list of attendees is not available.

A copy of the agenda is included as attachment 1.

Minutes from the October 3, 2017, October 12, 2017, and October 16, 2017 meetings were approved by voice vote.

## **Action Items from Previous Meeting**

A review of the status of action items from the previous meeting was undertaken. The action items and their disposition are included as attachment 2. Action items marked as completed will be removed from the list.

## **IVA Update**

There are currently 4 stands in three labs and there appears to be sufficient hardware for approximately two years. One lab indicated they may no longer run the IVA test after the current calibration period is up, and another lab indicated they may attempt to calibrate a stand. The laboratory which will drop IVA testing will advise the panel of hardware quantities they may be able to make available to the industry.

## **TMC Report**

The section(s) from the TMC D02.B01 semi-annual report pertaining to IVA were reviewed. The complete report can be found at:

<http://www.astmtmc.cmu.edu/ftp/docs/gas/B01SemiAnnualReports/semiannualreports/B01%20SemiAnnualReport%20-%20OCT%202017.pdf>

## **Fuel Supplier Report**

There is currently one batch of fuel being used for both the IVA and IVB and is being blended in 50,000 gallon batches at the IVB sulfur specification. All labs are currently using the same batch.

## **IVB Matrix Status and Timeline**

The panel reviewed tests completed to date and anticipated matrix completion, which is included as attachment 3. Considerable discussion took place regarding anticipated matrix completion and analysis of data with presentation to AOAP the week of February 8, 2018. Also discussed was what to do with matrix engines open completion. Jason Bowden reminded the panel that the engines were on “consignment” from OHT and were not to be used for any candidate work post matrix. Bill Buscher indicated he may run an additional test on reference oil 300.

## **IVB Operational Review**

The panel reviewed operational plots from nine matrix tests, included as attachment 4. There were several concerns expressed about operations in certain labs/test and are summarized as follows:

Blowby coolant out temperature and its possible impact on severity.

Blowby flow rate was considerably different on test 102-0-62, the lab will follow up on potential blowby flow meter issues during the test.

There was some variability in coolant out temperature in one lab that needs further investigation.

Differences in oil gallery temperature were noted and may be traced to tuning.

The impact of oil sump temperature needs to be better understood.

There were some oddities in both engine speed and coolant temperature in one lab that need further investigation.

## **D3525 Capillary Column**

Due to lack of time, this item will be addressed during a conference call on a later date. Labs are to review the presentation (attachment 5) with their personnel in anticipation of the upcoming call.

### **Alternate Suppliers Statement**

The panel heard a brief presentation from Pat Lang, TGC Chair, regarding proposed changes/implementation of an updated alternate supplier approval protocol. See attachment 6.

The meeting was adjourned at 5:11 PM.

Attachment F  
**Sequence IV Surveillance Panel**  
San Antonio, TX  
Southwest Research Institute  
November 15, 2017  
2:00 p.m. - 5:00 p.m.

A G E N D A

1. Chairman comments
2. Attendance sign-in sheet distribution
3. Membership changes
4. Motion and action recorder
5. Approval of minutes for October 3, 2017, October 12, 2017  
And October 16, 2017
6. Action item review
7. TMC report
8. Fuel supplier report – KA24E Green fuel
9. Sequence IVA update
10. Prove-out and precision matrix testing update
11. Precision matrix testing operational data analysis review
12. Sequence IVB timeline review
13. D3525 capillary column chromatography
14. TGC alternate supplier protocol
15. Review scope & objectives
16. Old business
17. New business
18. Motion and action item review



19. Next meeting
20. Adjourn

Sequence IV Surveillance Panel  
June – November, 2017  
Open Action Items

Open Action Items

As Recorded at the Meeting by Bill Buscher

1. Action Item – Revise Sequence IVB data dictionary and report forms accordingly for the change from Engine Coolant In Temperature to Engine Coolant Out Temperature control point. Also include other revisions that been on hold until after the original Sequence IVB precision matrix completed. Expedite revisions and implementation prior to the restart of the Sequence IVB precision matrix.  
**In process, and close to completion.**
2. Action Item – Revisit and amend the 10% volume acceptance with respect to mixing the new fuel batch. Strongly recommend that tanks be de-inventoried prior to receiving newly adjusted batch of KA24E Green fuel.  
**In process.**
3. Action Item – OHT to design and supply a clutch alignment tool.  
**In process. Design completed.**
4. Action Item – OHT to design and supply a timing chain wedge (timing chain holding tool).  
**In process.**
5. Action Item – OHT to design and supply an engine rotation locking tool.  
**In process.**
6. Action Item – SwRI and Intertek to draft Section 4 of the Sequence IVB Engine Assembly Manual (Engine disassembly, cleaning and reassembly after camshaft lobe failure), if needed.  
**In process.**
7. Action Item – Lubrizol to investigate oil sampling valve effect on aeration of the drawn oil sample.  
**Completed.**
8. Action Item – Once the design of the additional oil pan modifications has been finalized and approved by this surveillance panel, all labs are to return their OHT modified oil pans (p/n OHTIVB-022-1) to OHT for the additional modifications to be performed, free of charge.  
**Completed.**
9. Action Item – At conclusion of the prove-out testing the Sequence IV surveillance panel will set an oil consumption validity limit.  
**Pending the completion of the precision matrix.**

10.Action Item – OHT to evaluate the potential to add a thermal barrier ceramic coating to the exterior surfaces of the OHT modified front cover, OHT tensioner access plate and OHT modified oil pan.

**In process.**

11.Action Item – SwRI and Intertek to swap EOT oil samples from their ASTM REO 300 and 1012 prove-out tests and perform D5185 Metals, D6304 Karl Fischer H<sub>2</sub>O Content, D3525 Fuel Dilution, D664 Total Acid Number and D4739 Total Base Number analysis for a lab-to-lab oil analysis comparison.

**In process. Samples swapped, analysis complete, to compile date.**

12.Action Item – Perform a sectional Rockwell hardness test on a test intake camshaft.

**Completed.**

13.Action Item – Ask the supplier of IVB-LFO-1 if the directional performance between prove-out tests, of the intake lifter wear, can be shared with the surveillance panel.

**In process.**

14.Action Item – Surveillance panel chair to collect the individual Intake Lifter Average Volume Loss results from the two ASTM REO 1012 and the seven ASTM REO 300 prove-out tests and forward to the industry statisticians group for statistical analysis by individual lifter.

**In process.**

15.Action Item – Labs to compare intake lifter profile traces to volume loss results from their ASTM REO 1012 and ASTM REO 300 prove-out tests.

**In process.**

16.Action Item – SwRI and Intertek to share their PDI profile traces from their ASTM REO 1012 and ASTM REO 300 prove-out tests for comparison and review.

**In process.**

17.Action Item – Intertek to provide a procedure for adding a chilled water booster pump and a blower to the engine oil temperature control system.

**Completed.**

18.Action Item – Re-evaluate the engine oil gallery temperature Qi targets and limits using the operational data from the latest prove-out and precision matrix tests.

**Pending the completion of the precision matrix.**

19.Action item – Afton to provide operational data plots of their engine coolant flow and RAC coolant flow for comparison to the operational data plots from the other labs.

**In process.**

20.Action Item – Lab to provide 1 hour, test hour 101 to 102 (NOTE: if an unscheduled shutdown occurred between test hour 101 and 102, then obtain data from the next full hour of test time without any scheduled or unscheduled shutdowns), of operational data, using the updated operational data review Excel template, from any additional prove-out tests and the Row 1 precision matrix tests, to the TMC for posting, so that an additional operational data analysis can be performed by Rich Grundza and Kevin OMalley. A total of 3 additional prove-out tests and 5 precision matrix tests to be

included. Labs to have data uploaded by 11/1/17. A follow-up conference call to be scheduled for 11/7/17.

**Completed.**

- 21.Action Item – Lab to complete and submit test reports to the TMC for any previously or recently completed Sequence IVB reference oil tests.

**In process.**

- 22.Action Item – Utilize the final prove-out and precision matrix tests to identify which controlled parameters have a significant influence test severity and re-evaluate the Qi targets and windows for those parameters at the conclusion of the precision matrix.

**Pending the completion of the precision matrix.**

- 23.Action Item – Create a database of the temperature parameters that have a significant influence test severity, including the different temperatures and their corresponding test results, and have the industry statisticians group to perform a statistical analysis to identify correlations.

**Pending the completion of the precision matrix.**

- 24.Action Item – Histogram

**Chris Mileti to provide details.**

- 25.Action Item – Labs to work together to investigate, understand and possibly correct difference in CAN data.

**In process.**

- 26.Action Item – Analyze blowby coolant out temperature to water content and test results to see if there is any correlation.

**In process.**

- 27.Action item – Intertek to draft a blowby flow meter cleaning procedure to add to the Sequence IVB test procedure.

**In process.**

- 28.Action Item – Surveillance panel chair to update the Sequence IVB timeline based on the current status of the precision matrix labs

**Completed.**

- 29.Action Item – Intertek and SwRI to provide 1 hour, test hour 101 to 102 (NOTE: if an unscheduled shutdown occurred between test hour 101 and 102, then obtain data from the next full hour of test time without any scheduled or unscheduled shutdowns), of operational data, using the updated operational data review Excel template, from the Row 2 precision matrix tests, with the exception of SwRI Stand 1, to the TMC for posting, so that an additional operational data analysis can be performed by Rich Grundza and Kevin OMalley. A total of 4 precision matrix tests to be included. Labs to have data uploaded by end of business on 11/9/17.

**Completed.**

- 30.Action item – For review at the 11/15/17 face-to-face Sequence IV surveillance panel meeting, Rich Grundza and Kevin OMalley to perform an operational data analysis on

the 9 available precision matrix tests only. Do not include any prove-out tests in this analysis.

**Completed.**

# SEQUENCE IVB UPDATE

November 2017



# GF-6 TEST STATUS





Test	Test Development Complete	Precision Matrix Complete	Test Accepted By AOAP/PCEOCP	LTMS Established	Registration Started	GF-5 Equivalency Limits Established	BOI/VGRA Established
IIIH						IIIF - Proposed IIIG -	Waiting for BC 5 Hardware
IVB		Currently Running	Expected Feb-18	Expected Feb-18	Expected Feb-18	IVA - TBD	Expected Q1 2018
VH					Expected Nov-17	VG - Proposed	Expected Q1 2018
VIE						VID - Proposed	Expected Q1 2018
VIF						VID - Proposed	Expected Q1 2018
VIII	N/A	N/A	N/A				
IX						N/A	Expected Q1 2018
X			Expected Dec-17	Expected Dec-17	Expected Dec-17	N/A	Expected Q1 2018

# PROVE-OUT AND PRECISION MATRIX STATUS



PROVE-OUT TESTING						
Run Order	Required				Supplemental	
	IAR Stand 1	IAR Stand 2	SwRI Stand 1	SwRI Stand 2	Lubrizol	ExxonMobil
1	300	IVB-LFO-1	1012	IVB-LFO-1	300	300
2	1012		300	IVB-LFO-2	1012	1012
3		300	300	300		

 = Completed

 = Running

PRECISION MATRIX								
Run Order	Precision Matrix					Supplemental		
	IAR Stand 1	IAR Stand 2	IAR Stand 3	SwRI Stand 1	SwRI Stand 2	Lubrizol	ExxonMobil	Afton
1	1012	300	1011	300	1012	1012	300	1011
2	1011	1012	1012	1011	300	300	1011	1012
3	300	1011	300	1012	1011	1012	300	1011
4	1012	300	1011	300	1012			

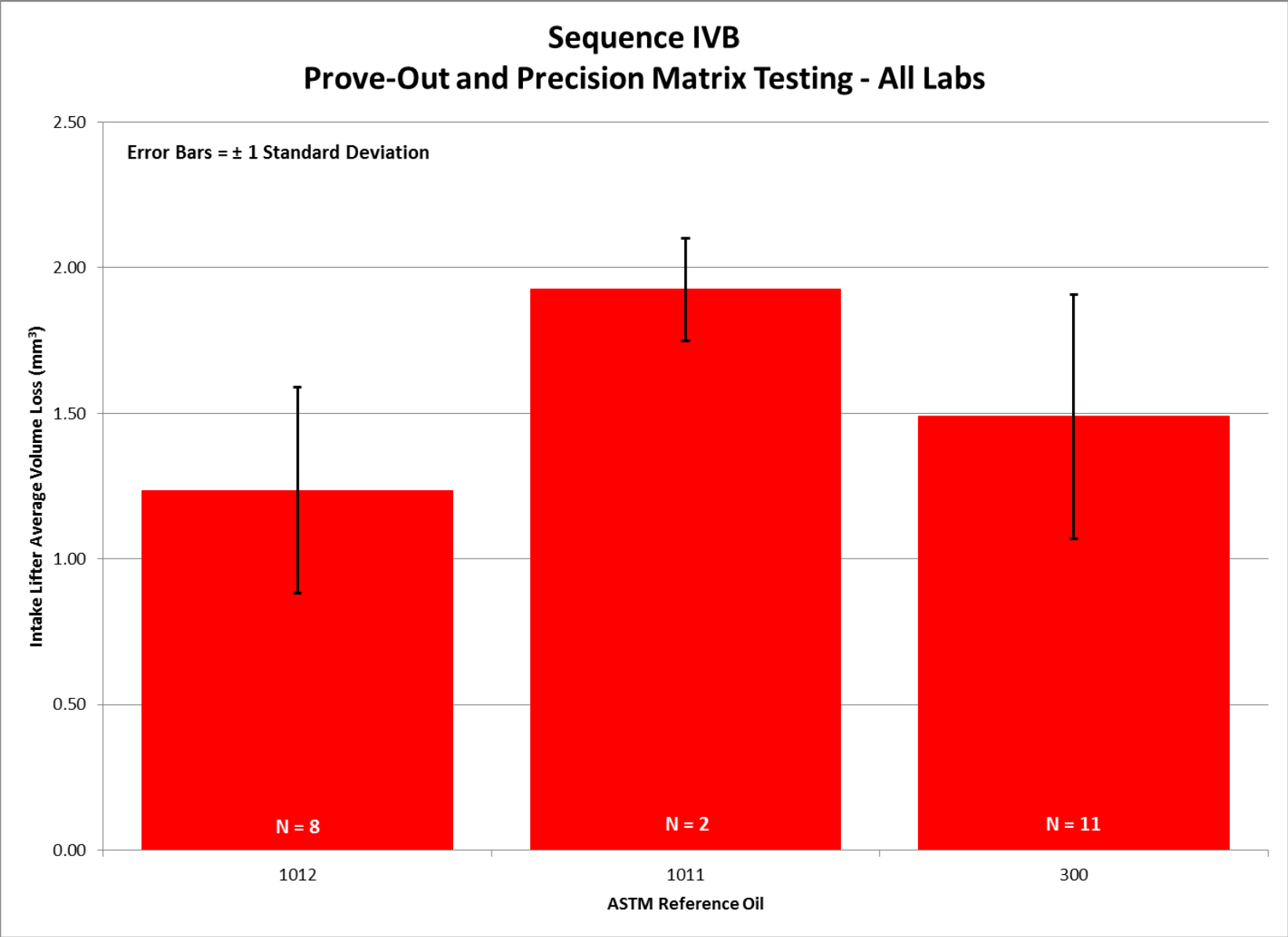


# PROVE-OUT AND PRECISION MATRIX TEST DATA

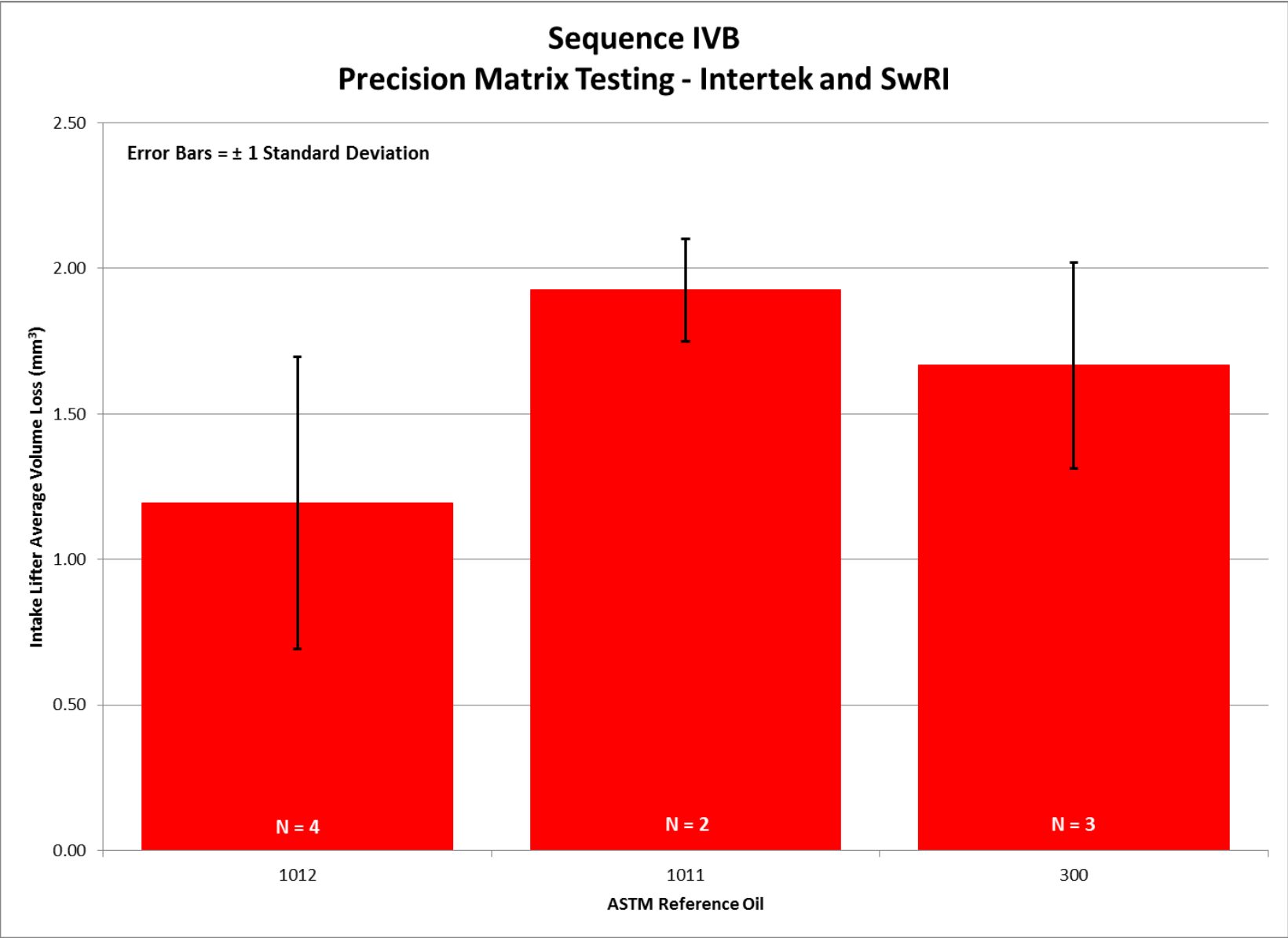


Run Order	Purpose	Lab	Stand	Oil	Intake Lifter Average Volume Loss mm <sup>3</sup>
1	prove-out test 1	Intertek	102	300	1.79
2	prove-out test 1	SwRI	18	1012	1.01
3	prove-out test 2	SwRI	18	300	1.16
4	prove-out test 2	Intertek	102	1012	1.32
5	prove-out test 2	Intertek	165	300	2.26
6	prove-out test 3	SwRI	18	300	1.15
7	prove-out test 3	SwRI	20	300	1.50
8	prove-out test 1	ExxonMobil	3109	300	1.53
9	prove-out test 1	Lubrizol	347	300	1.10
10	prove-out test 2	Lubrizol	347	1012	1.46
11	precision matrix test 1	Intertek	100	1012	1.55
12	precision matrix test 1	Intertek	101	300	1.78
13	precision matrix test 1	Intertek	102	1011	1.80
14	prove-out test 2	ExxonMobil	3109	1012	1.31
15	precision matrix test 1	SwRI	20	1012	0.71
16	precision matrix test 2	Intertek	100	1011	2.05
17	precision matrix test 2	Intertek	101	1012	1.70
18	precision matrix test 2	Intertek	102	1012	0.82
19	precision matrix test 1	SwRI	18	300	1.95
20	prove-out test 1	SwRI	19	300	0.89
21	precision matrix test 2	SwRI	20	300	1.27

# PROVE-OUT AND PRECISION MATRIX VOLUME LOSS PLOT



# PRECISION MATRIX VOLUME LOSS PLOT



# TIMELINE



Task	5-Stand Precision Matrix
Complete Test Fuel Blending	DONE
Complete Test Hardware Procurement and Preparation	DONE
Complete Preparation for Prove-out Testing	DONE
Complete Row 1 Prove-out Tests	DONE
Complete Row 2 Prove-out Tests	DONE
Complete Procedure Update	DONE
Complete Precision Matrix Lab Audits	DONE
Seq. IV Surveillance Panel Meeting	DONE
Complete 5 ASTM REO 300 Prove-out Tests	DONE
Complete Preparation for Precision Matrix	DONE
Seq. IV Surveillance Panel Conference Call - 10/12/17	DONE
Seq. IV Surveillance Panel Conference Call - 10/16/17	DONE
Restart Precision Matrix (Start Row 1 Tests)	DONE
Complete Row 1 Precision Matrix Tests	DONE
Complete Row 1 Precision Matrix Operational Review	DONE
Complete Precision Matrix	12/15/2017
Complete Final Precision Matrix Operational Review	12/22/2017
Start Statistical Analysis of Precision Matrix	1/2/2018
Complete Statistical Analysis of Precision Matrix	2/2/2018
Complete Development and Approve LTMS	
PCEOCP/AOAP Vote for Test Acceptance	2/8/2018
Stand Calibration Starts	2/9/2018

## **WILLIAM A BUSCHER III - IVB**



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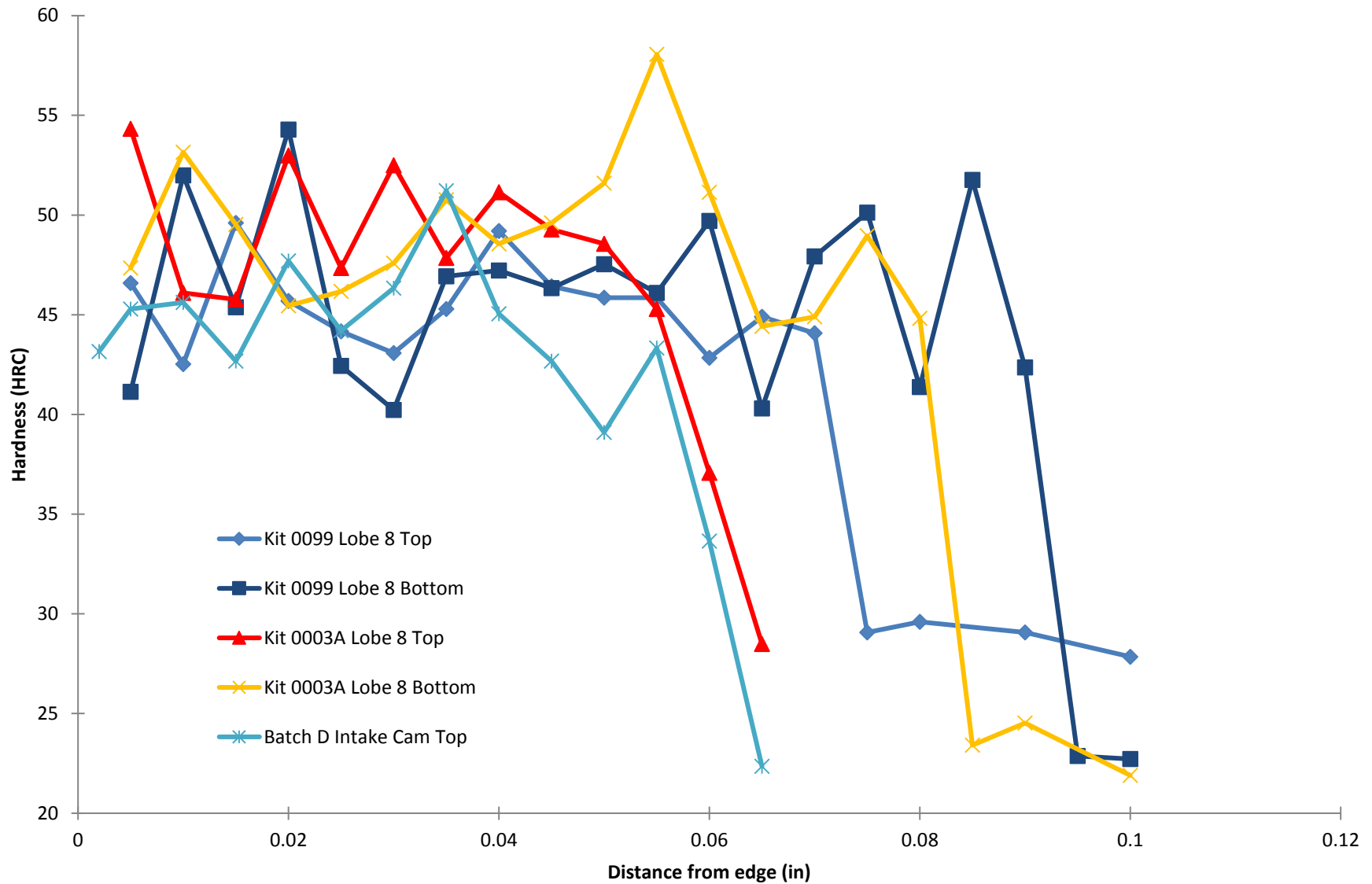
[intertek.com/automotive/lubricants-fuel-systems/](https://intertek.com/automotive/lubricants-fuel-systems/)



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## Measured Cam Lobe Hardness



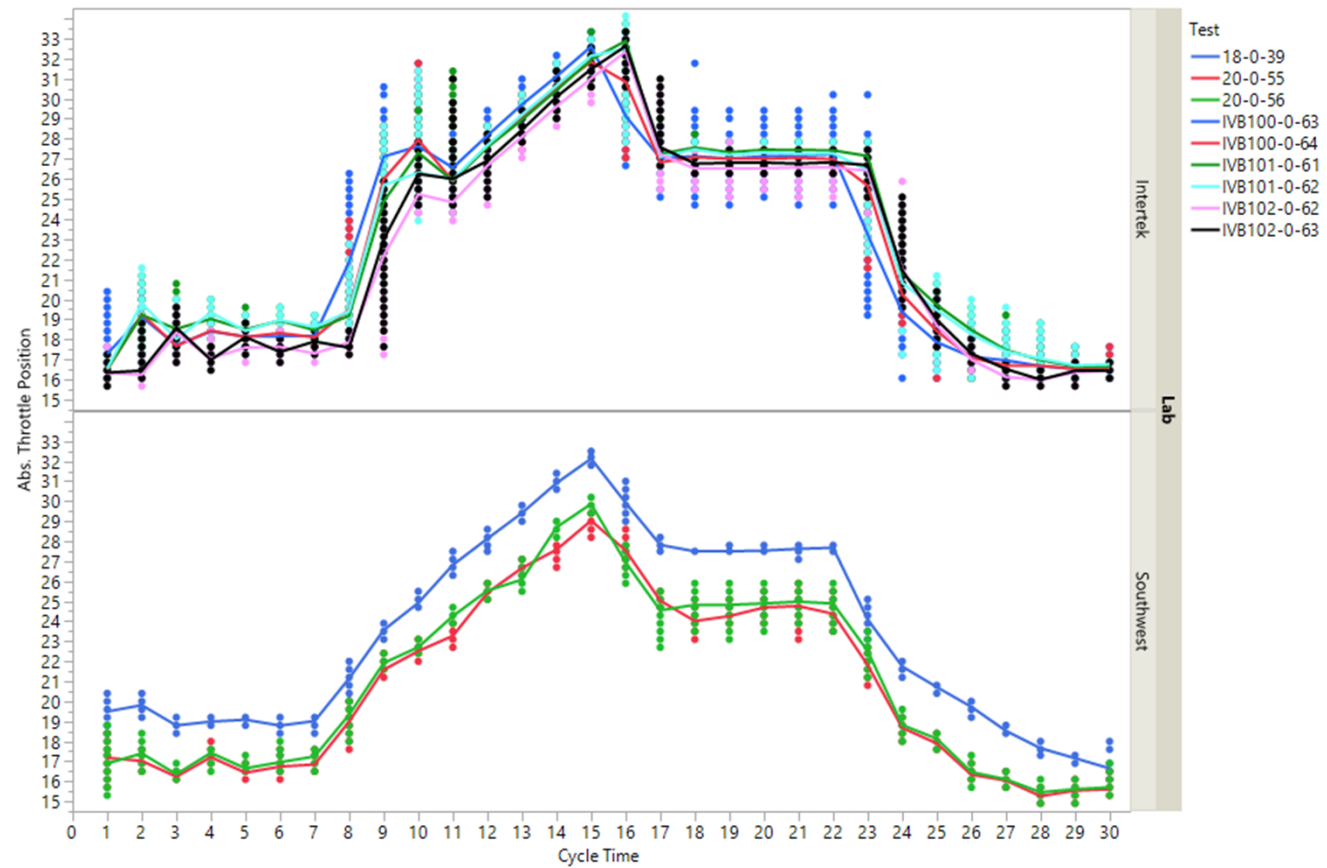
# IVB Operational Data Review 9 Matrix Tests

Prepared by: Kevin O'Malley

11-15-17

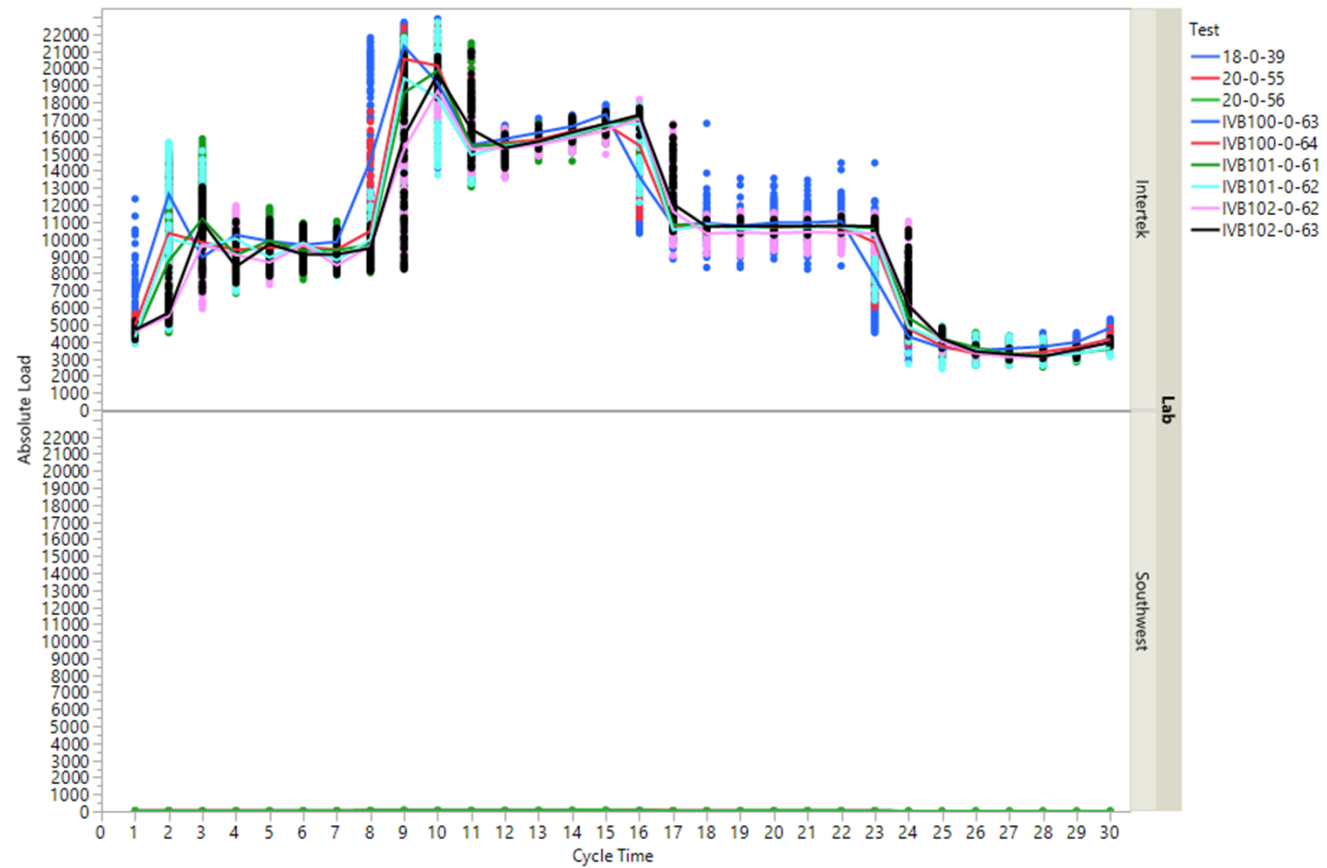


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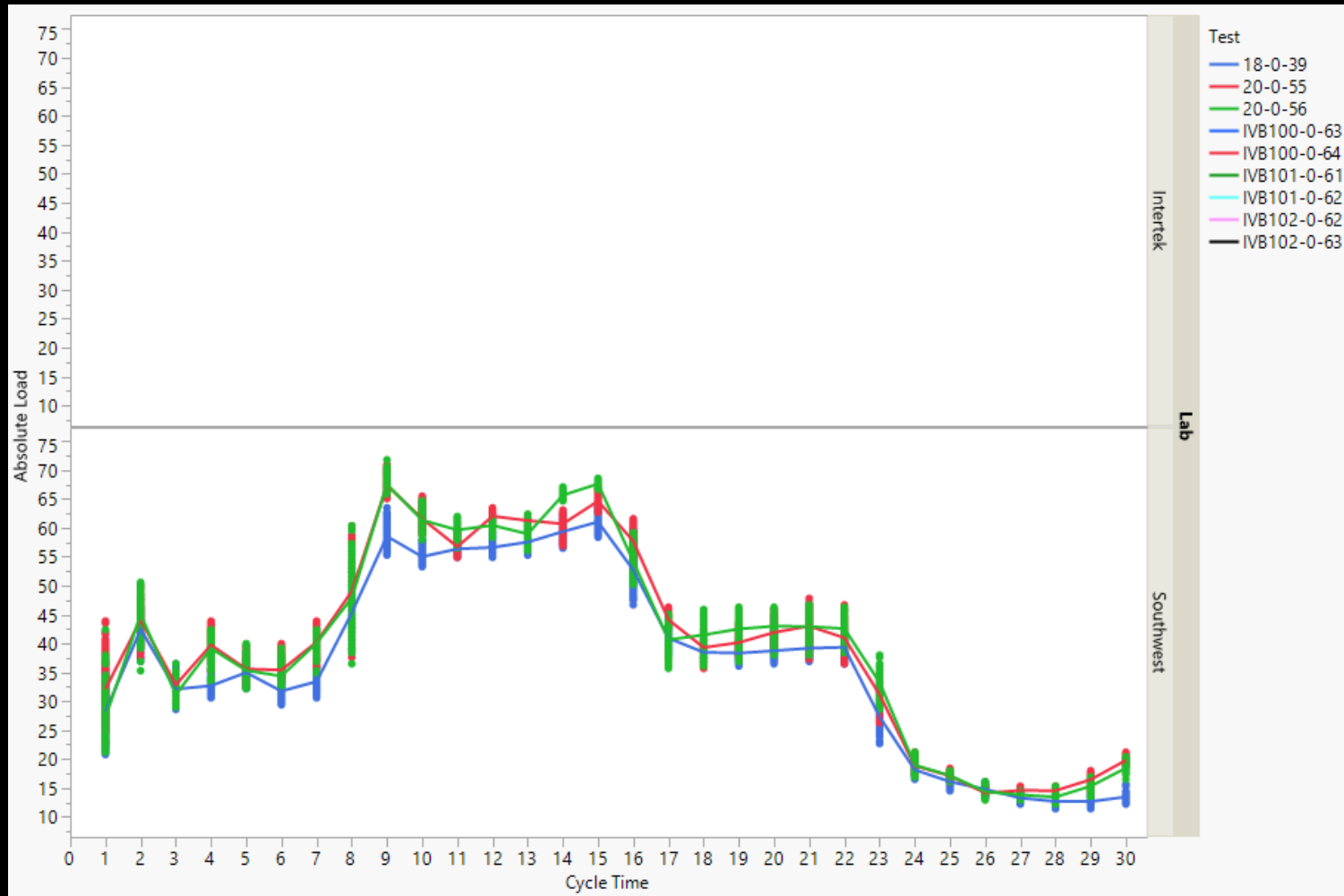


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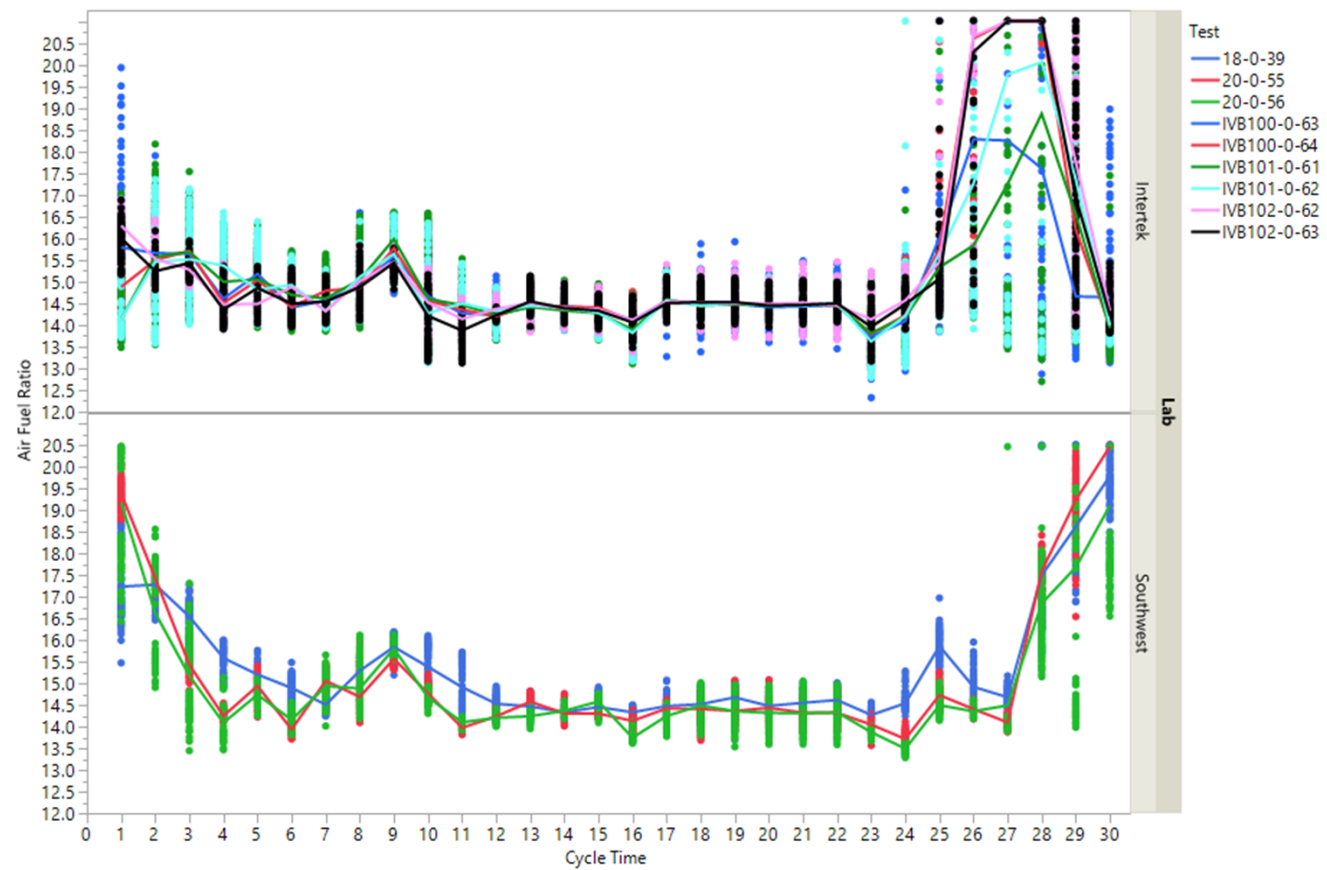
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# ZOOMED IN SwRI ONLY

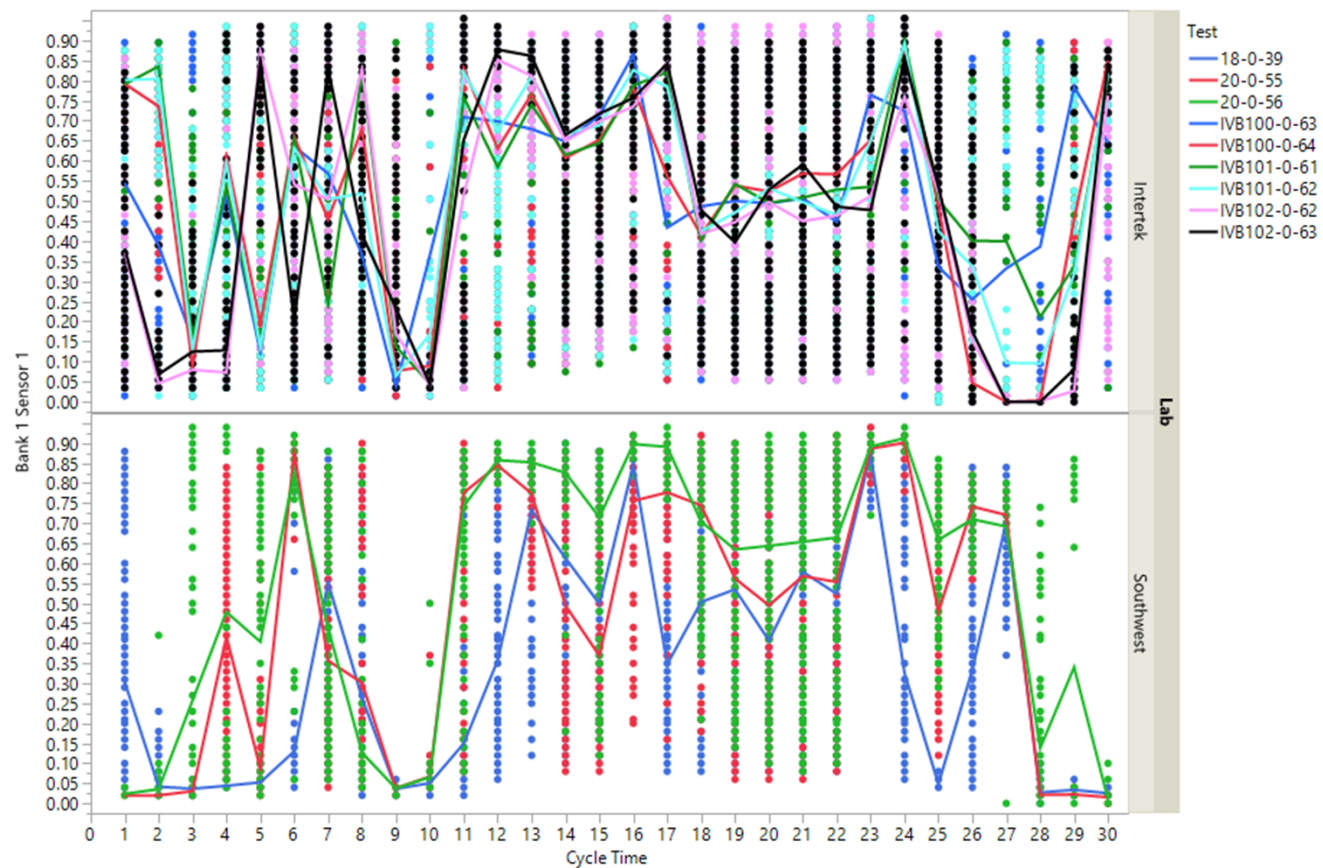


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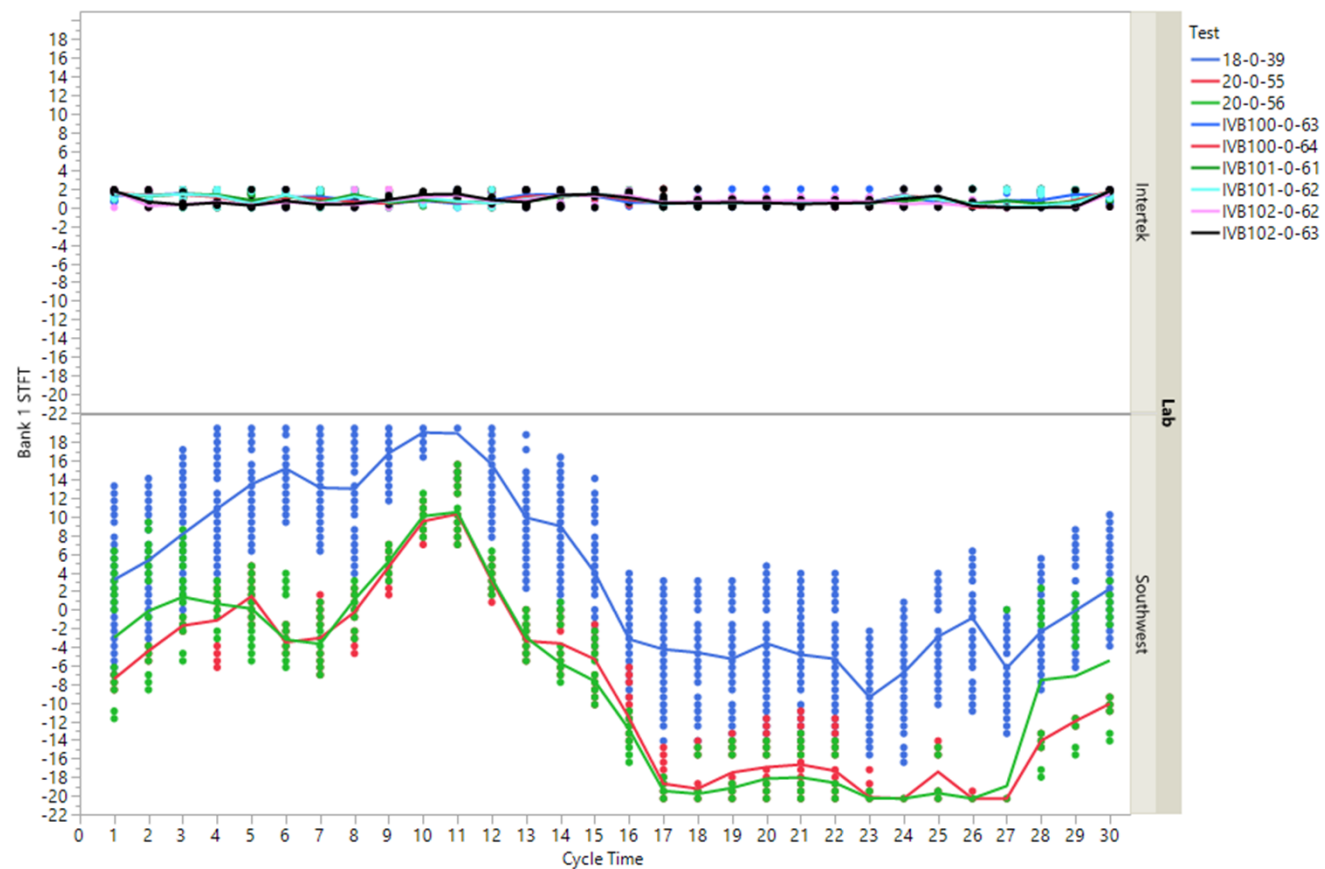


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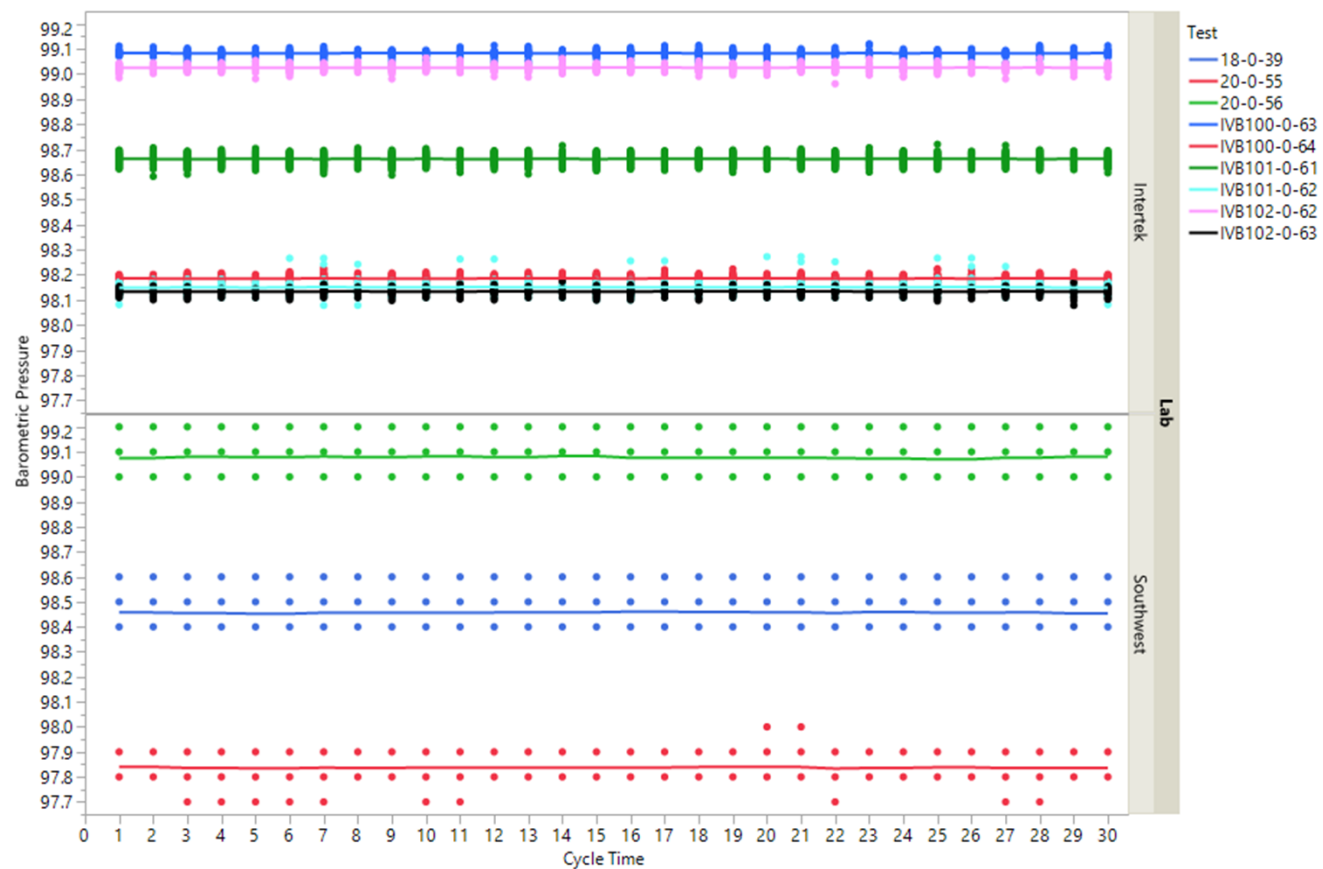
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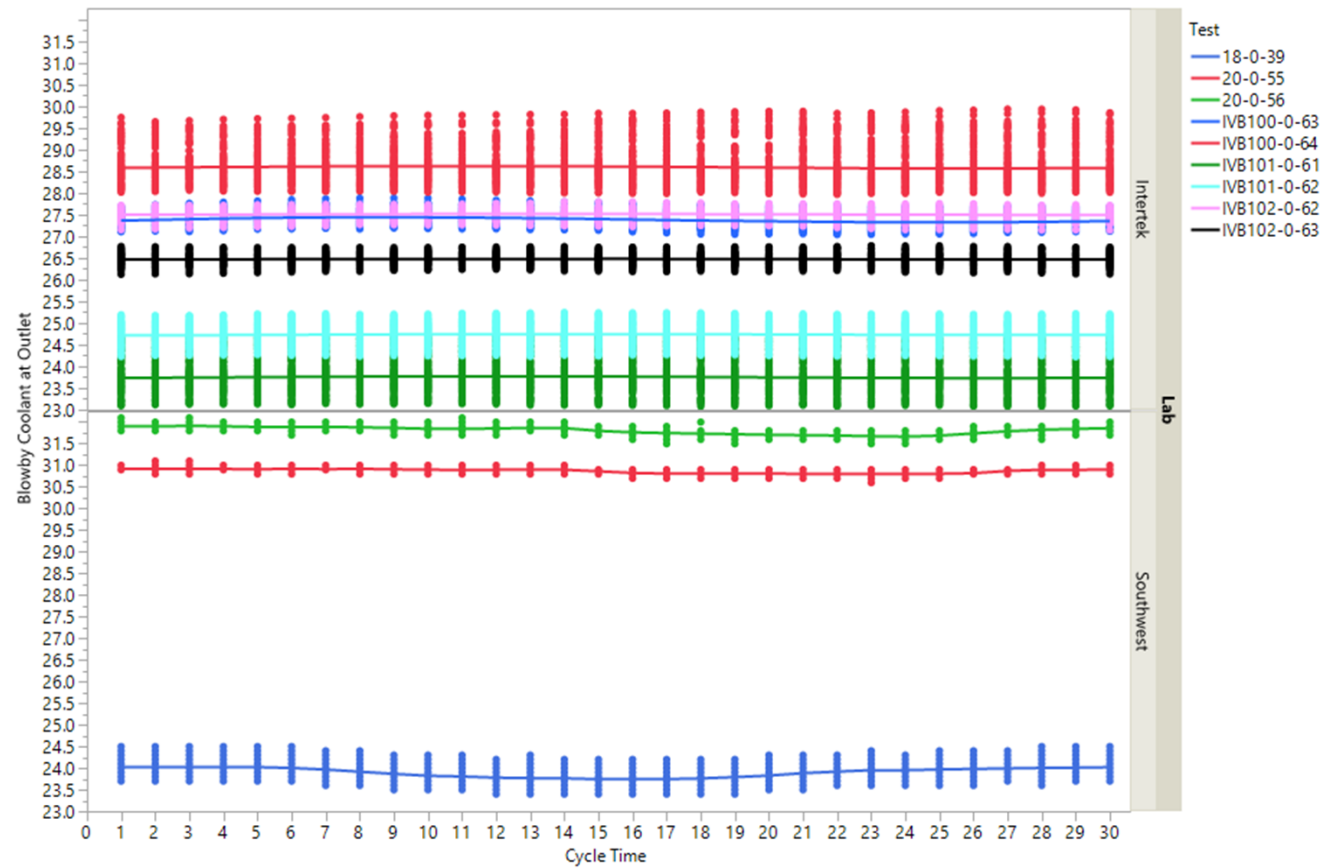
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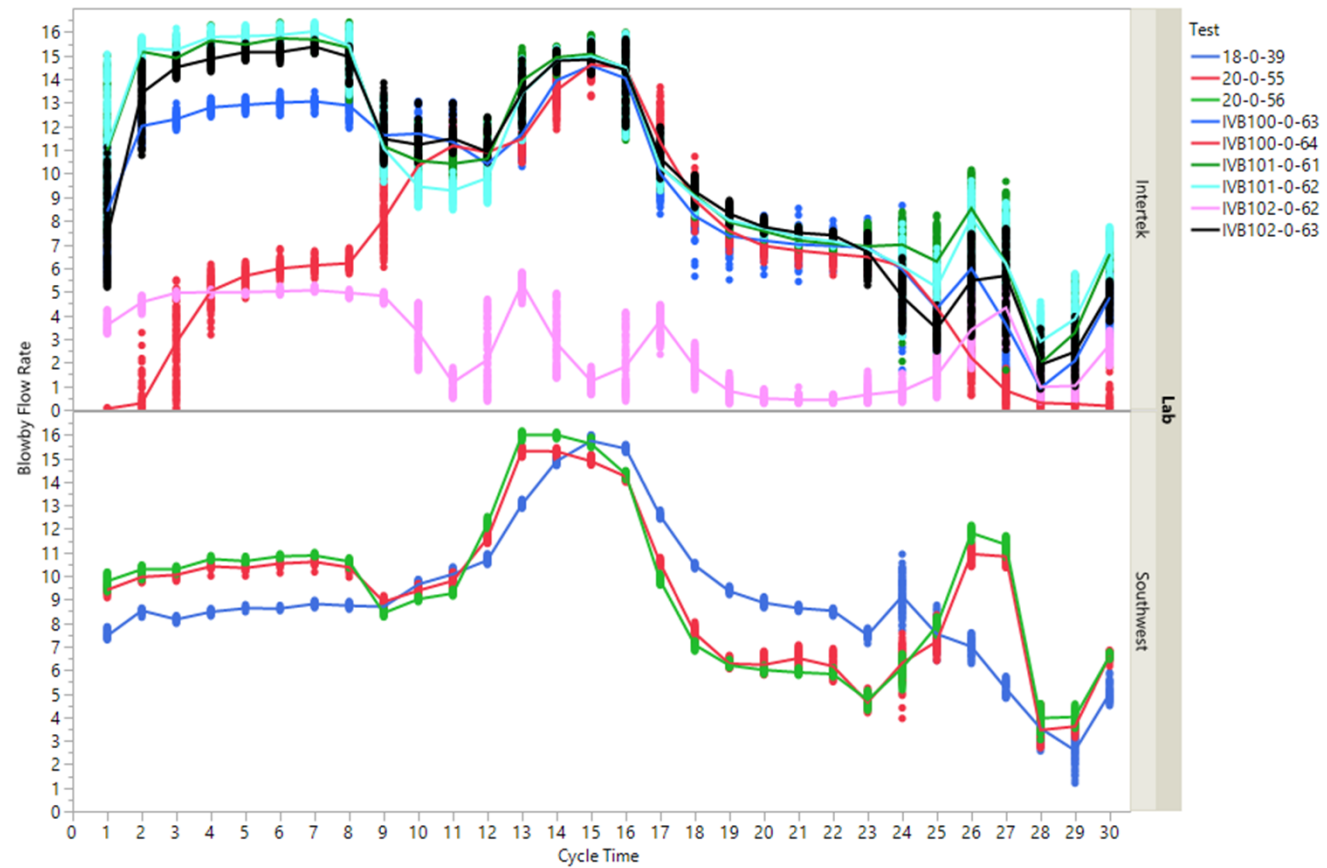


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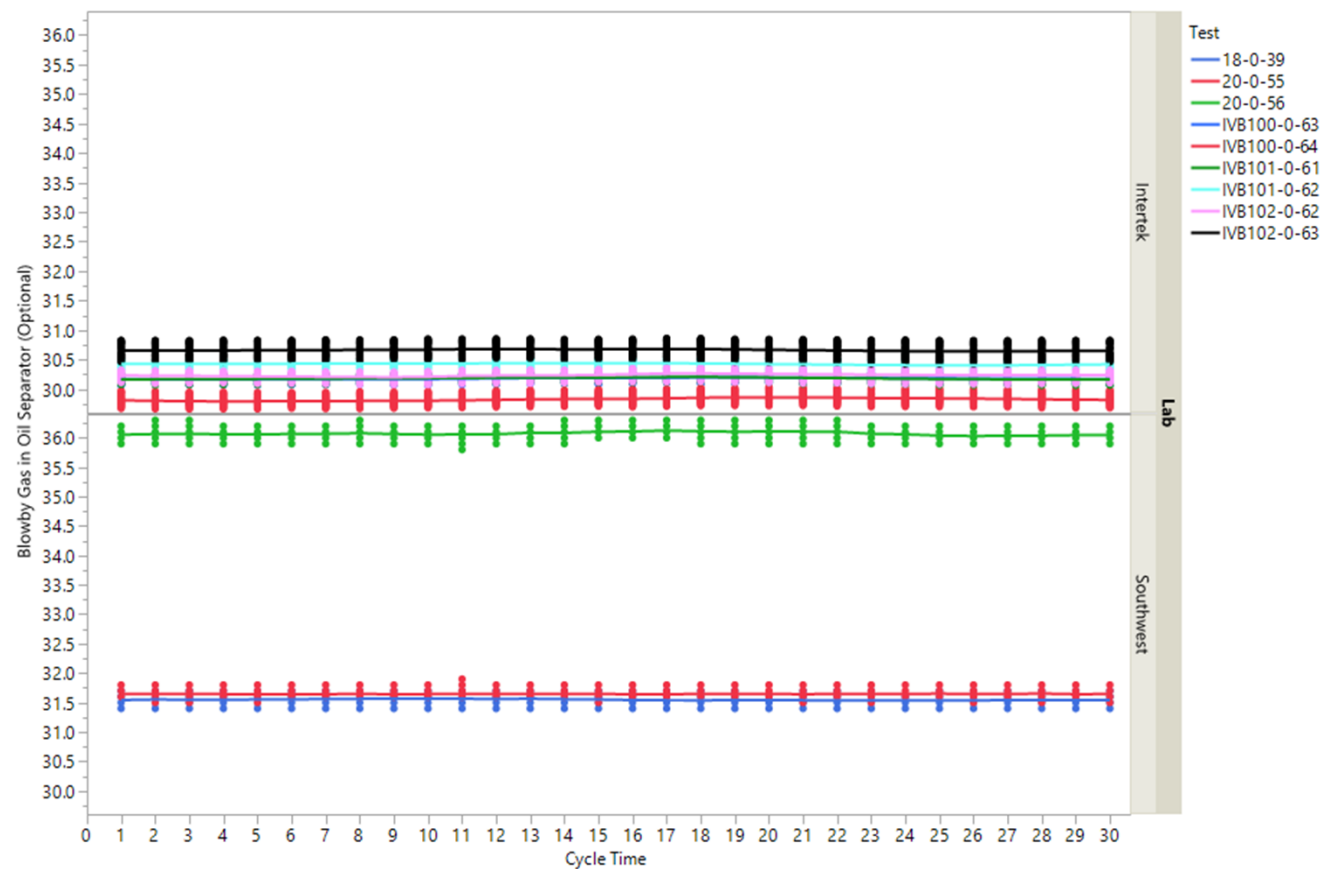




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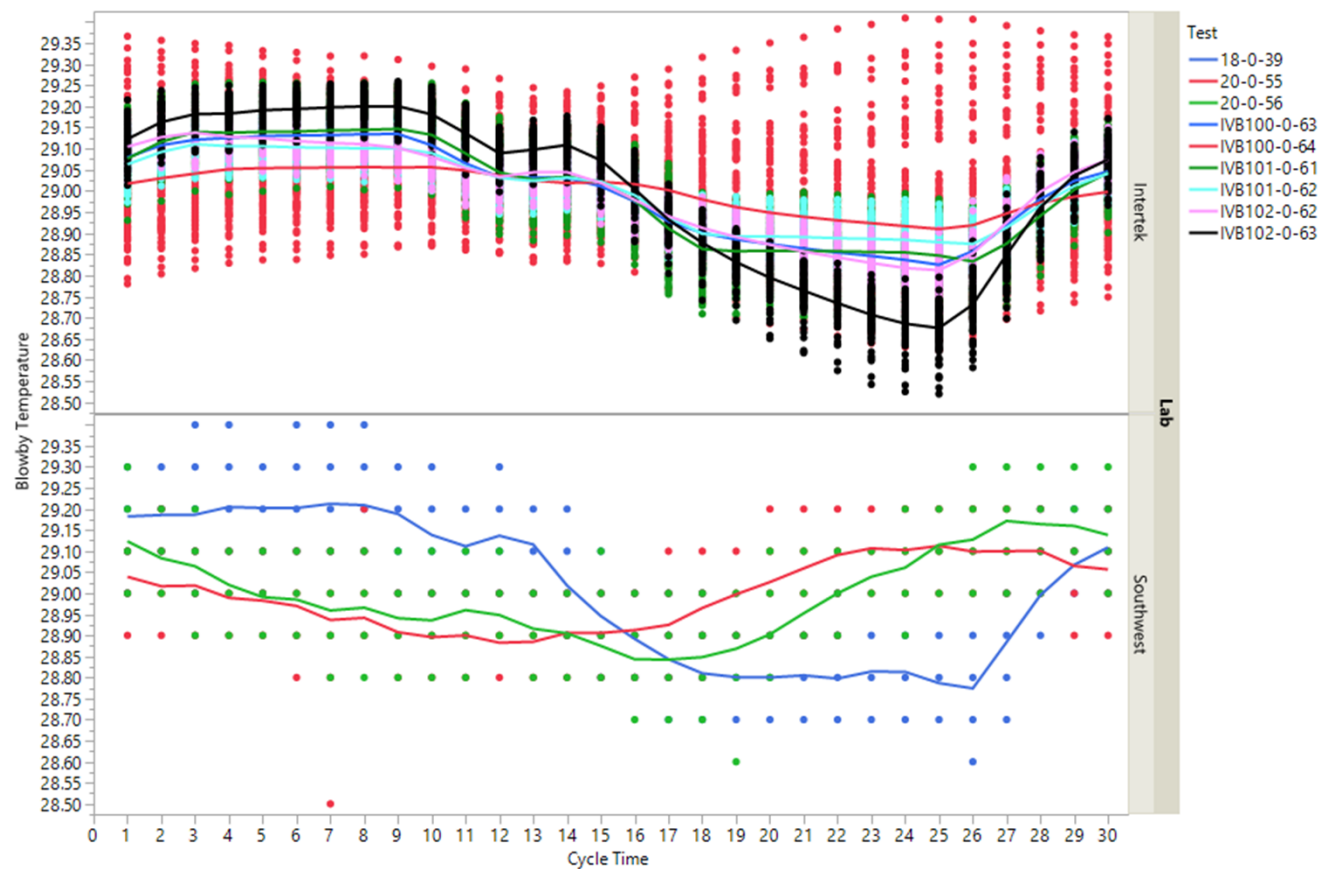


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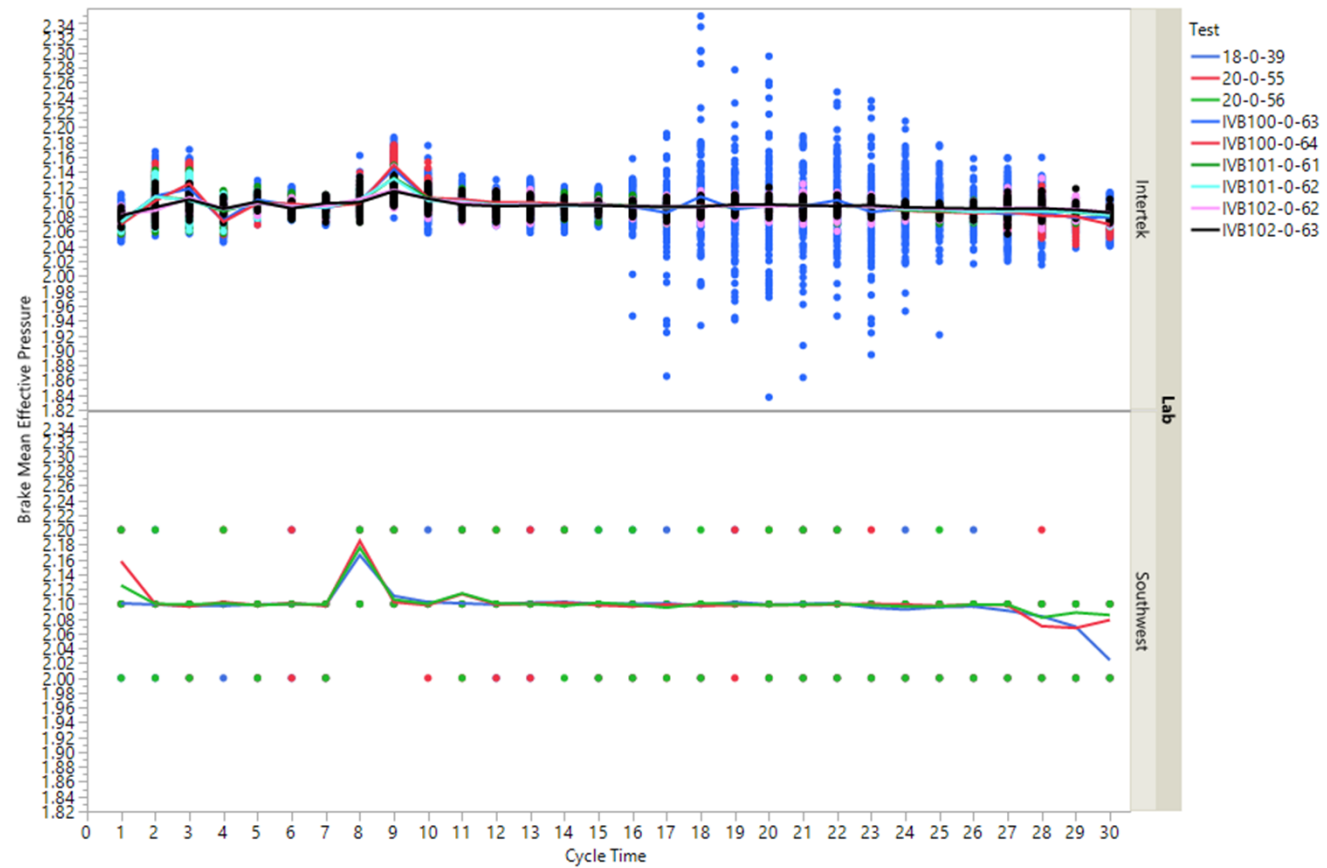


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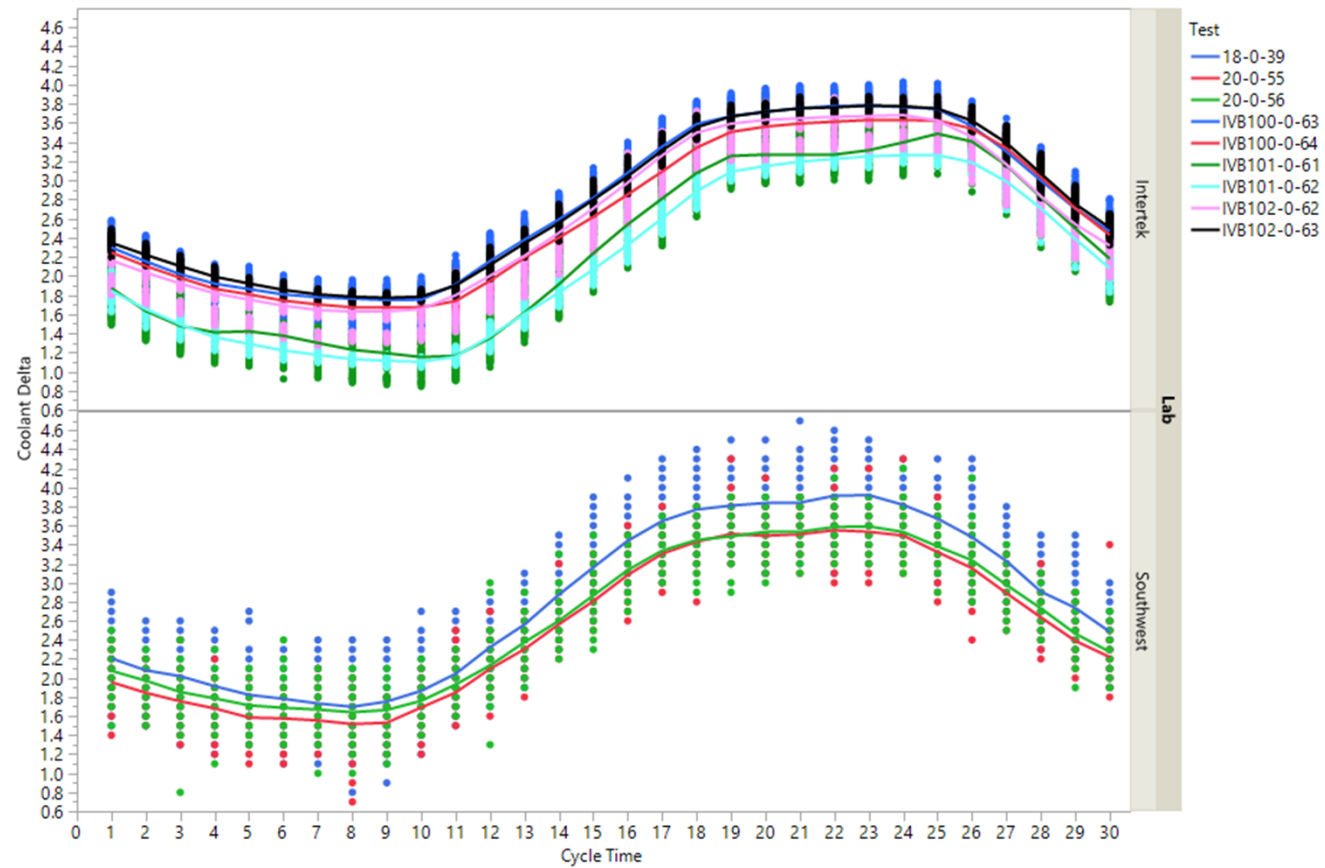
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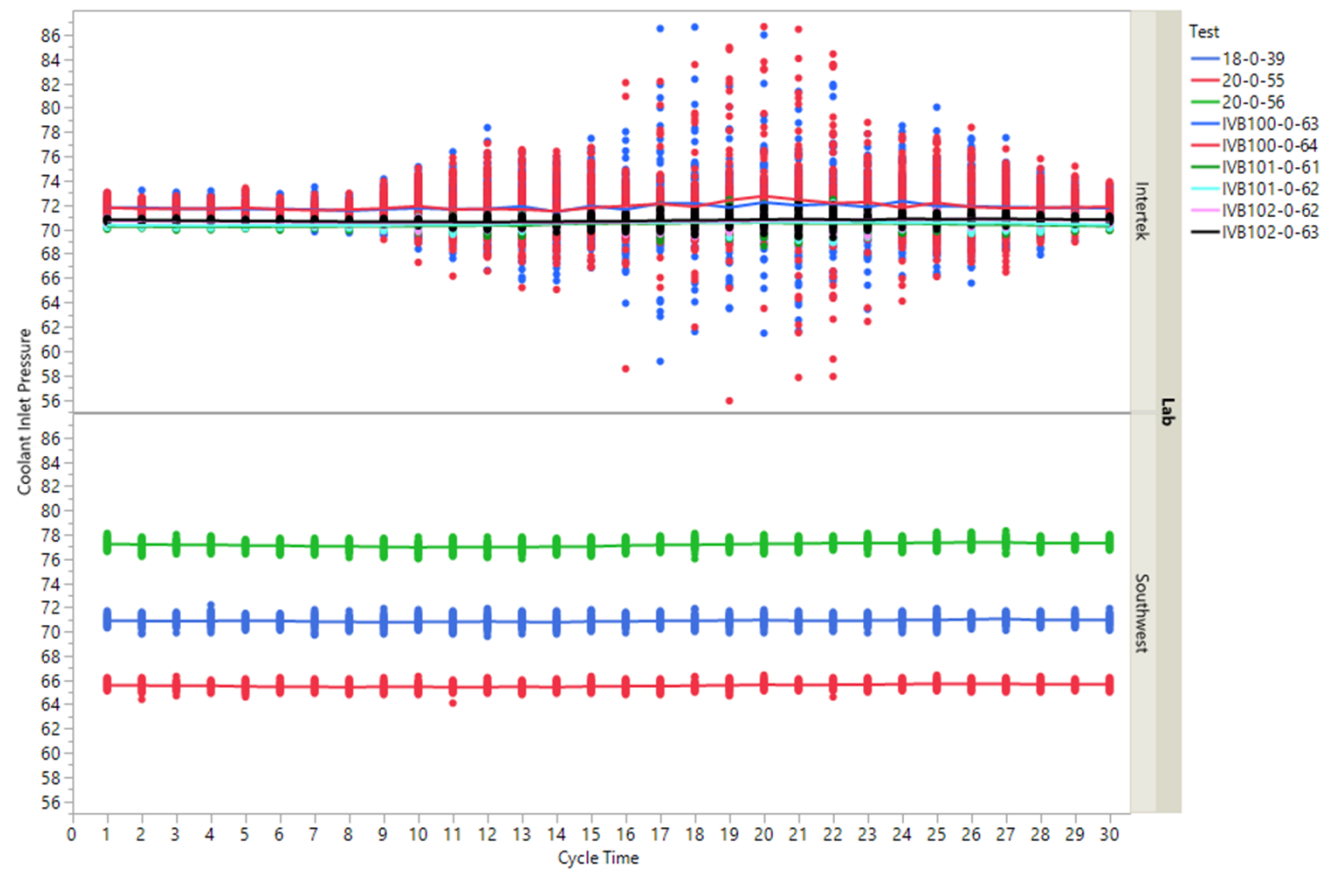
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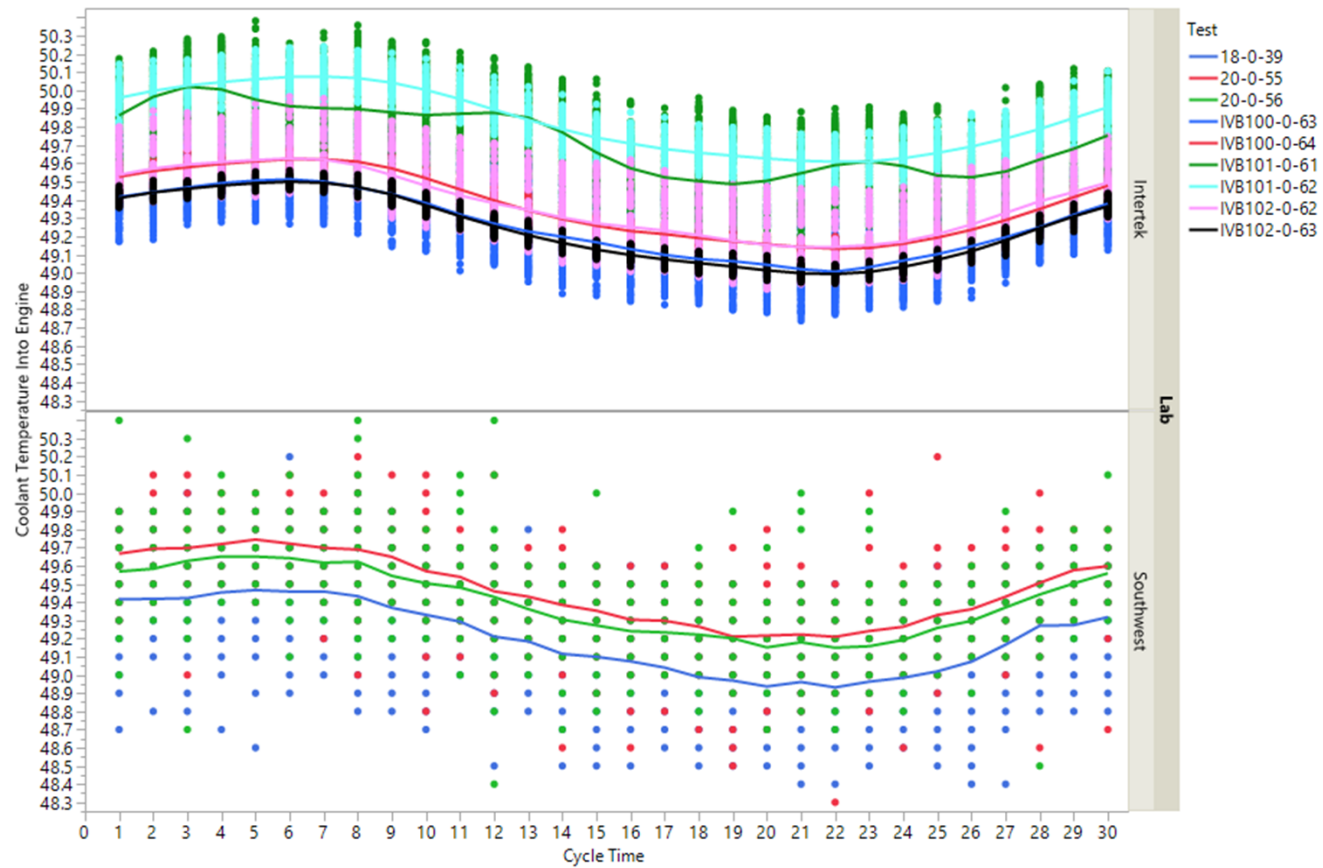
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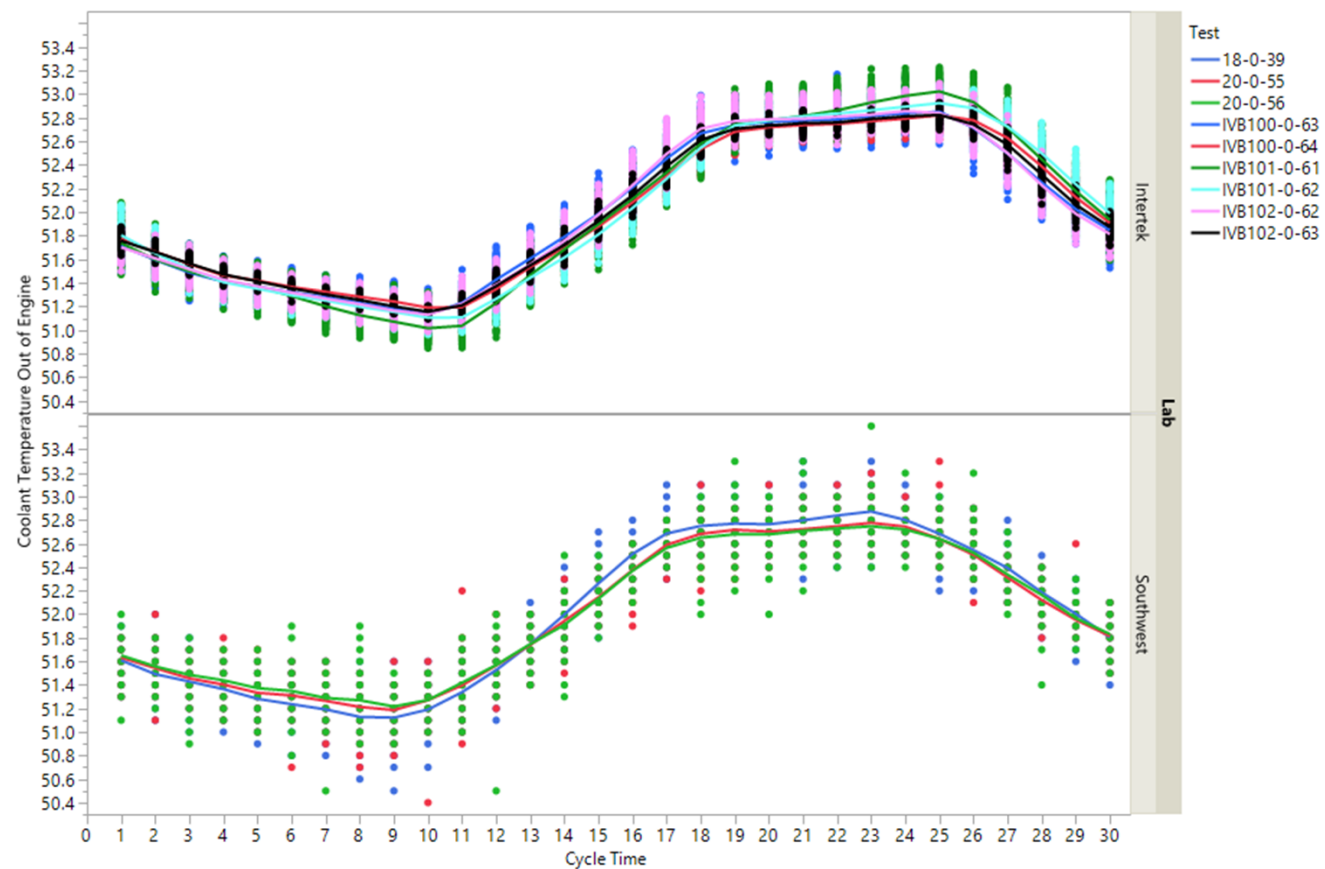


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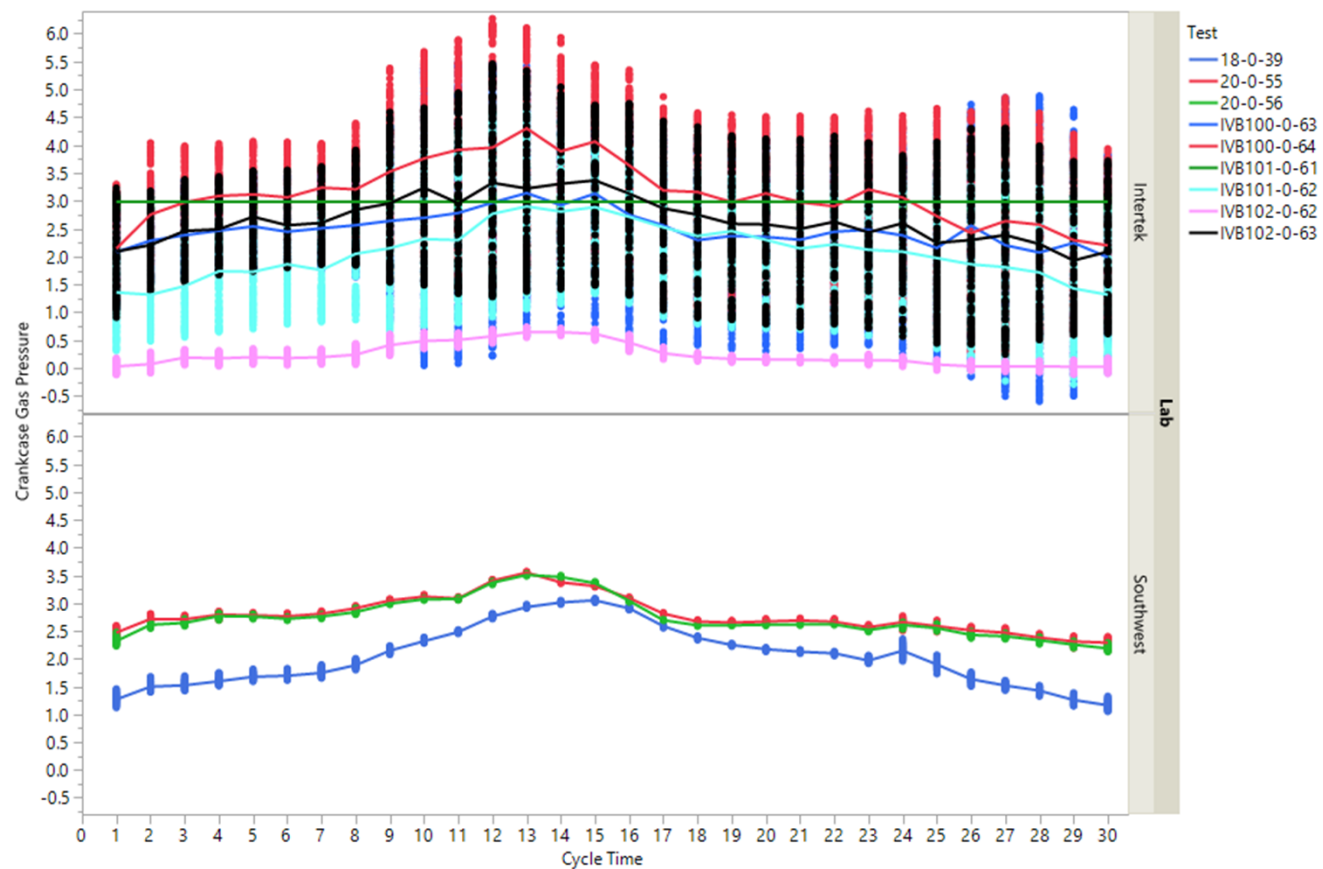
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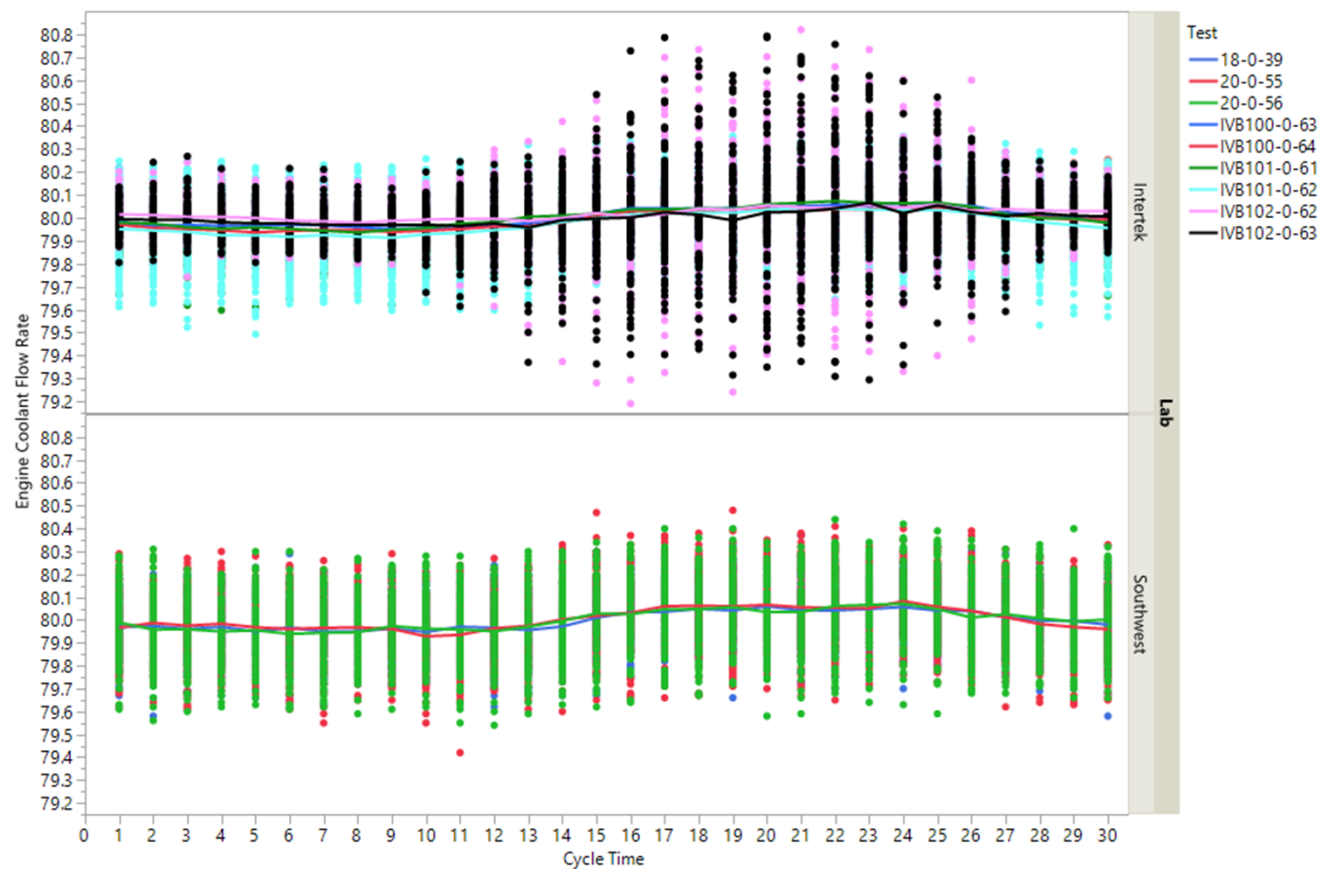


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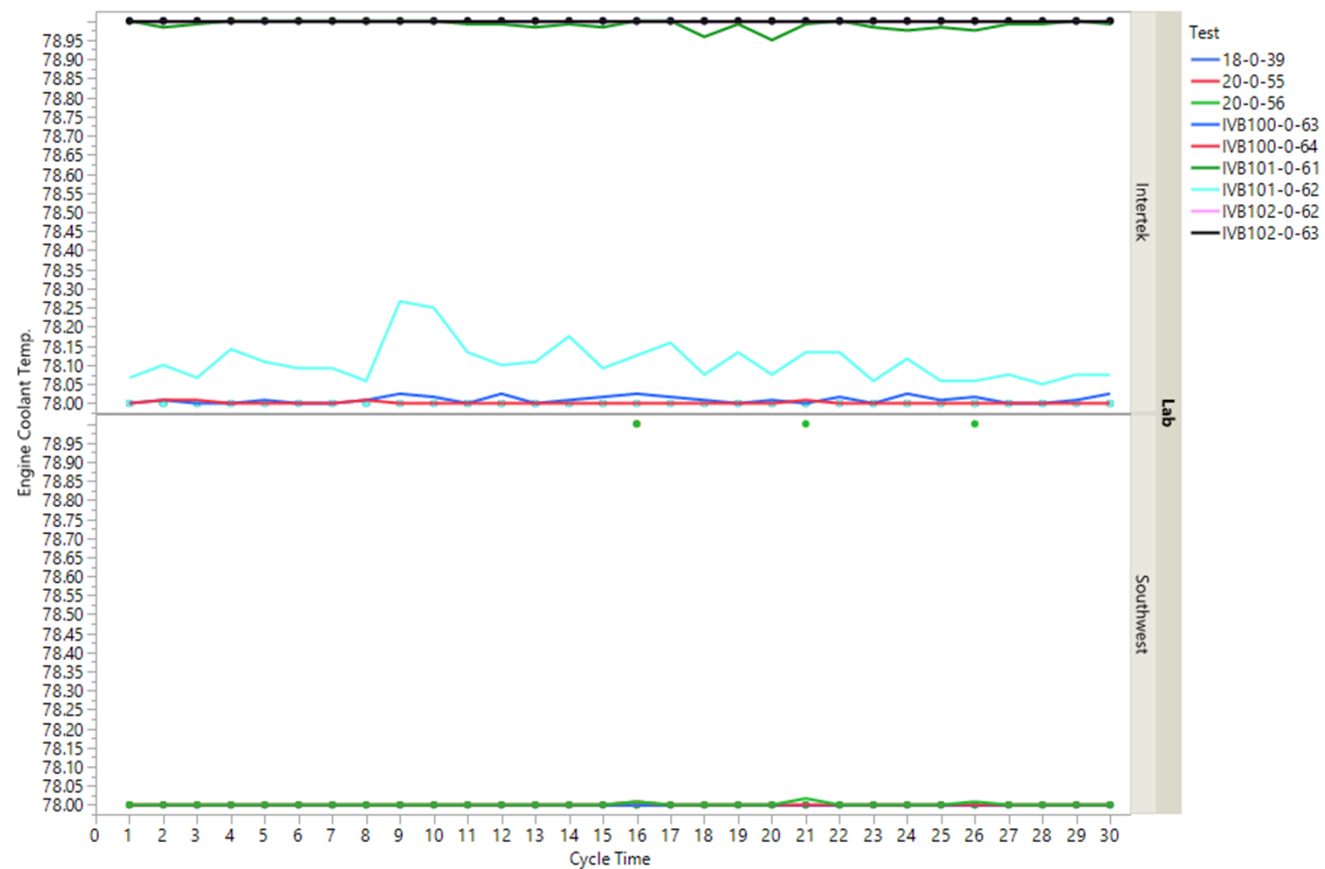


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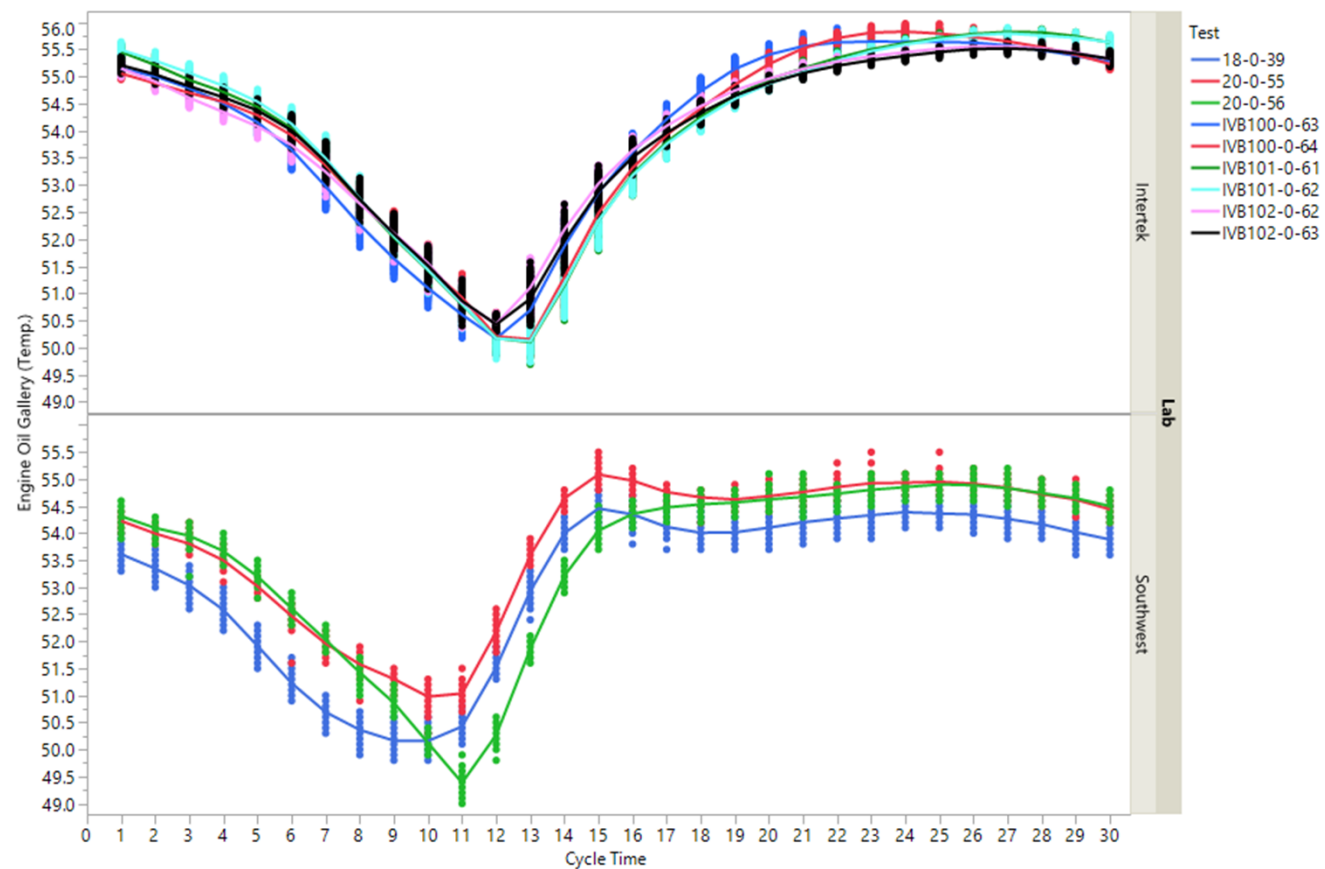


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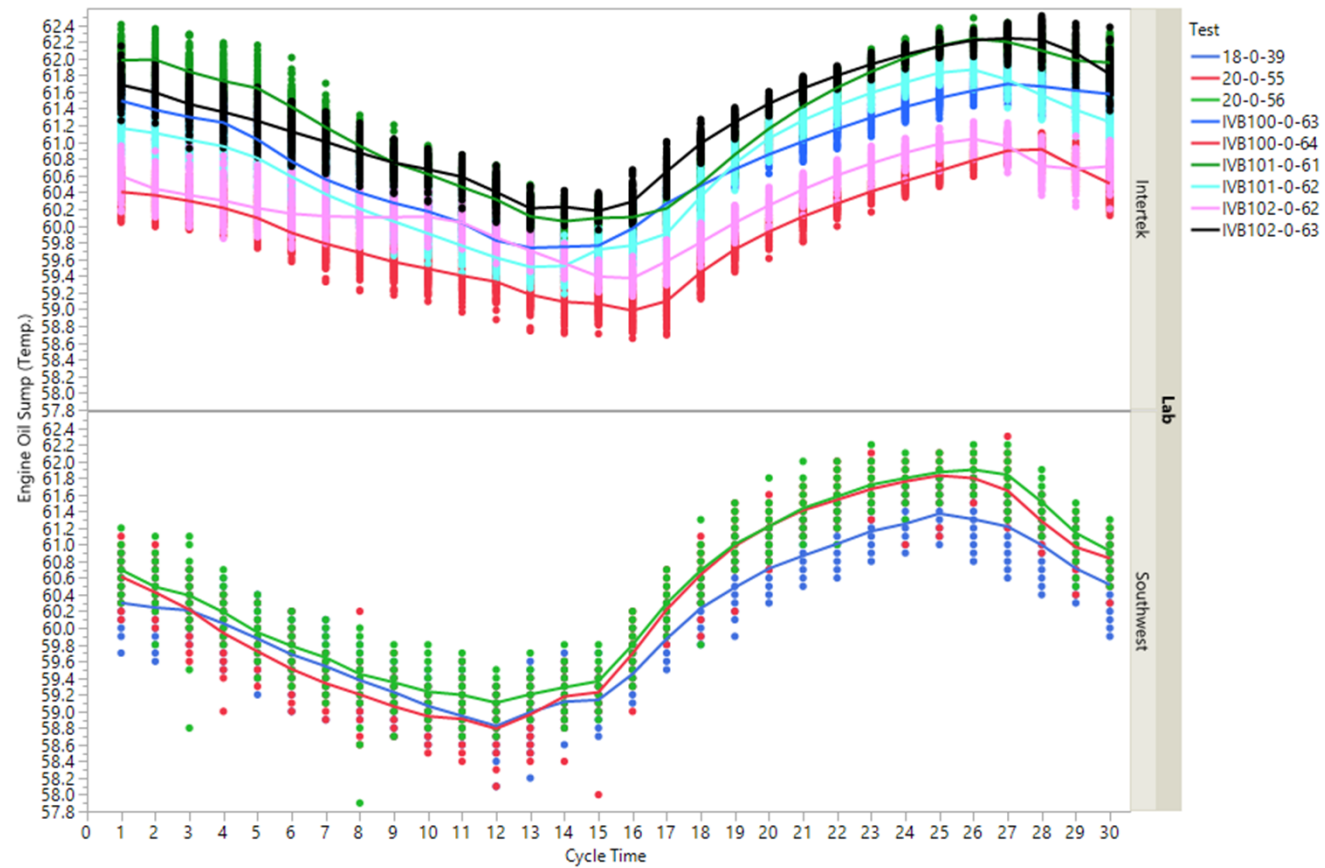


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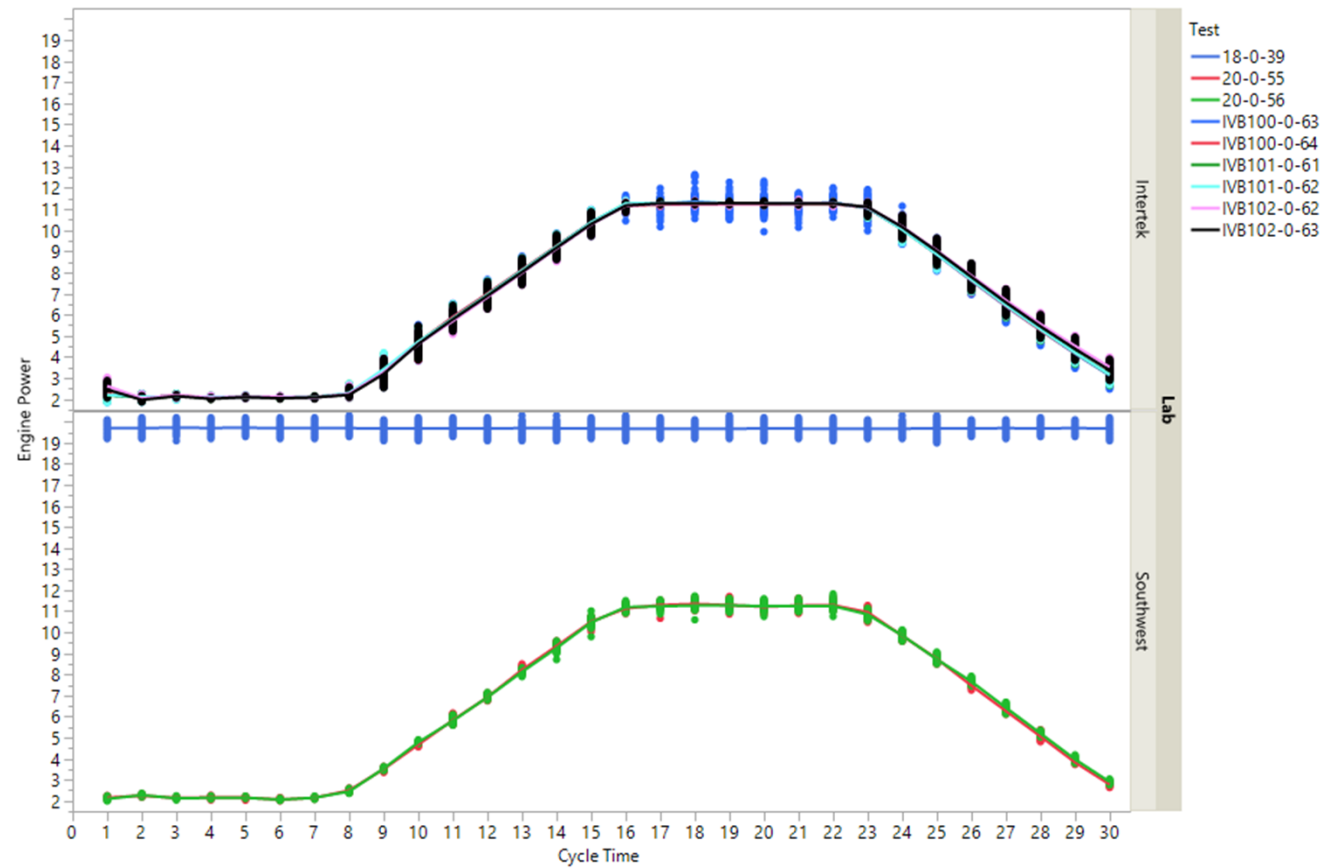


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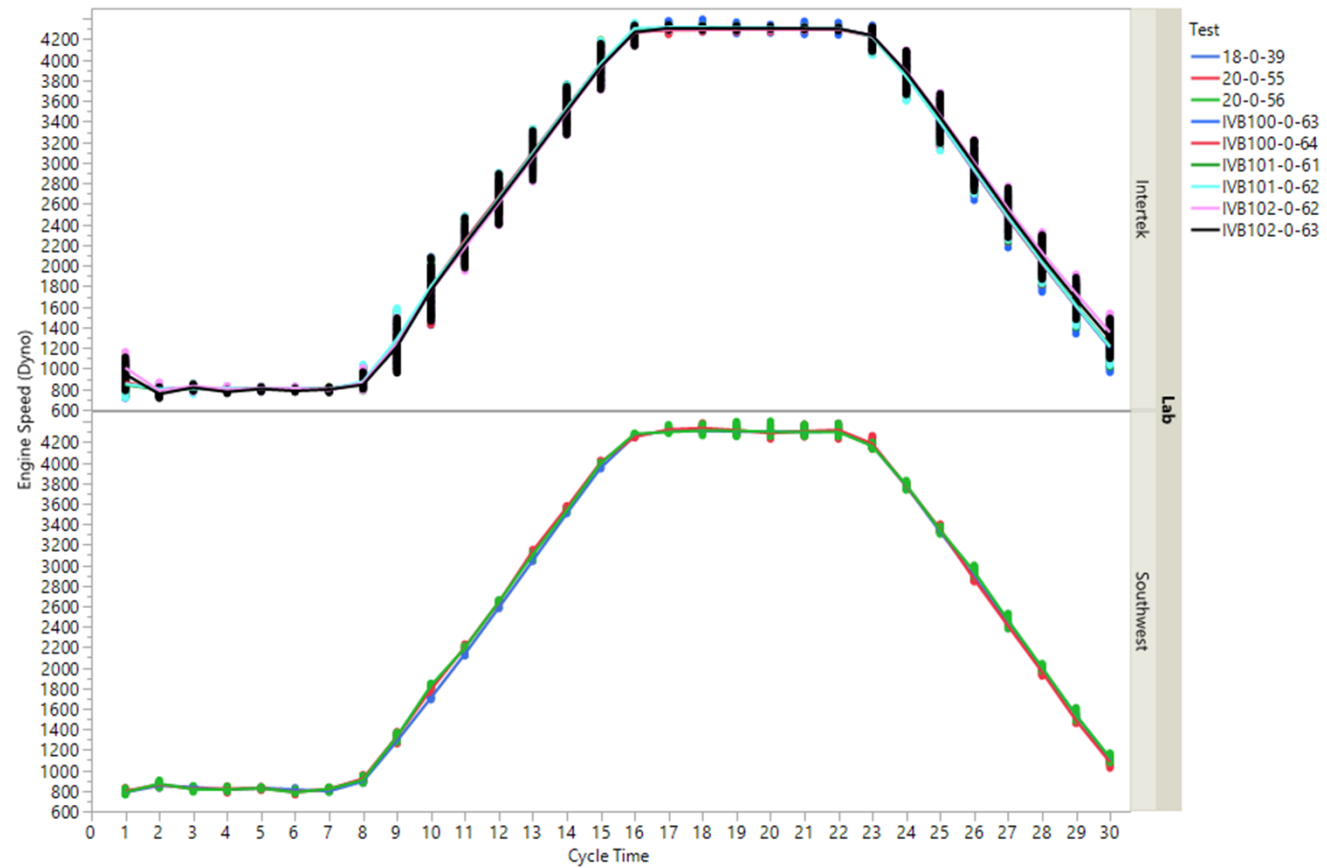
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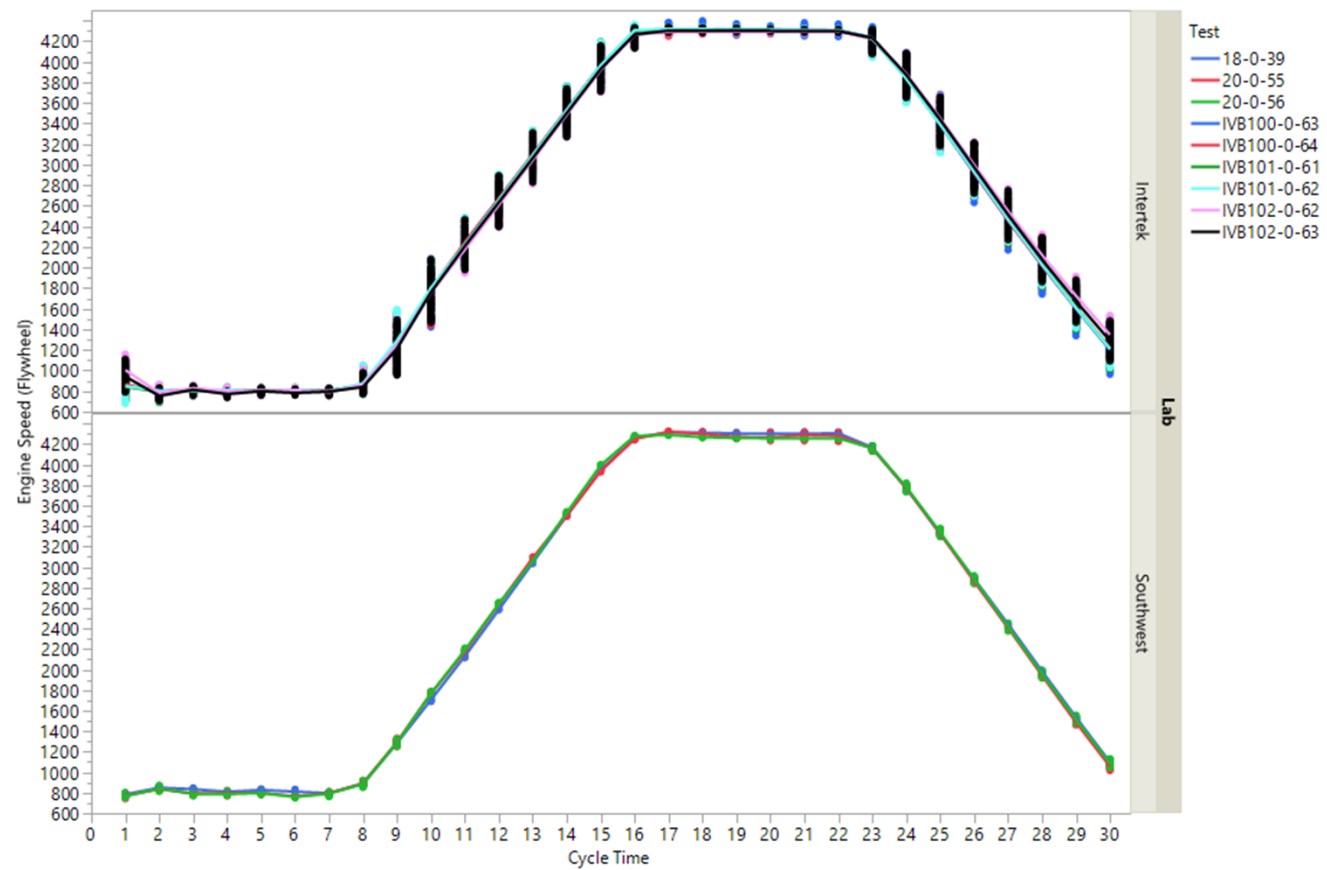
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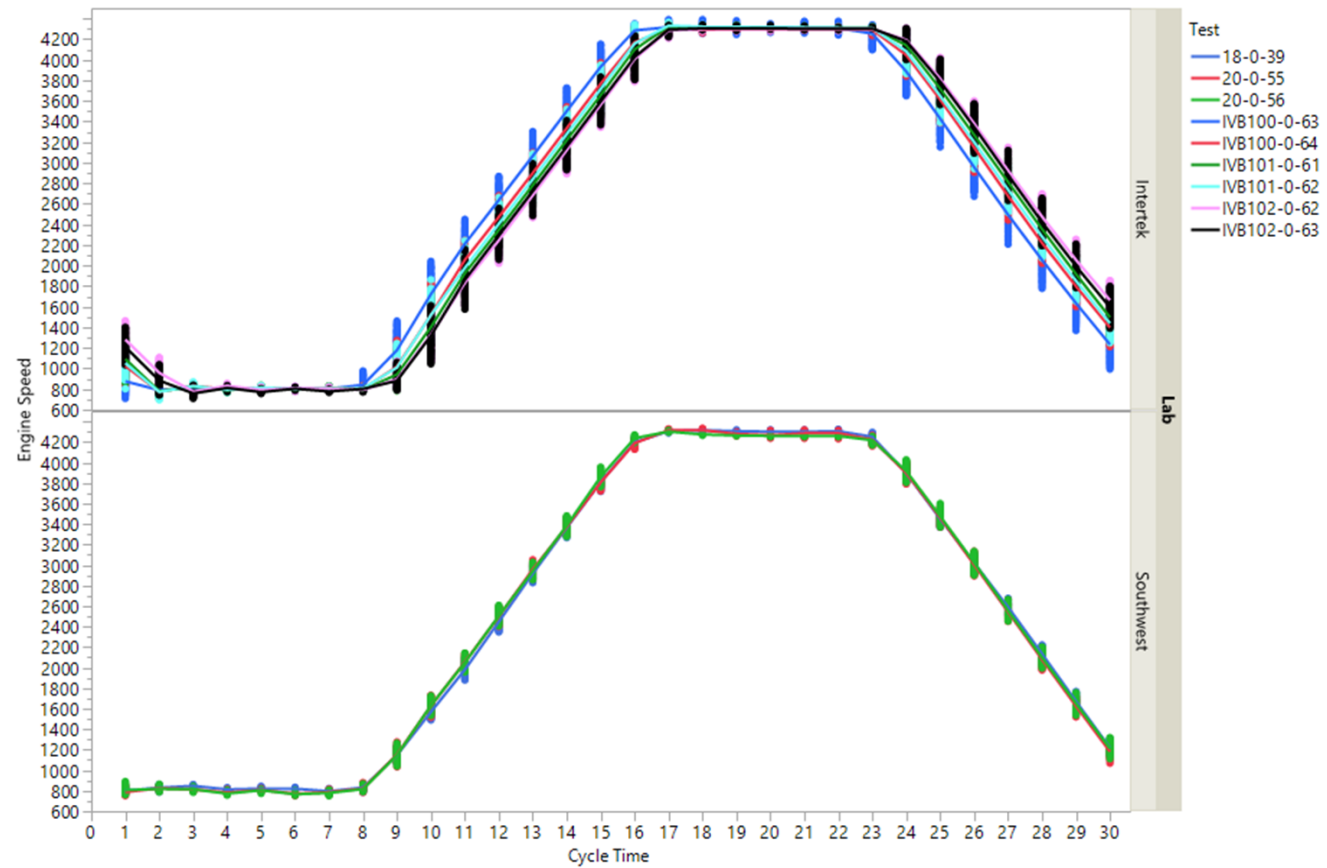
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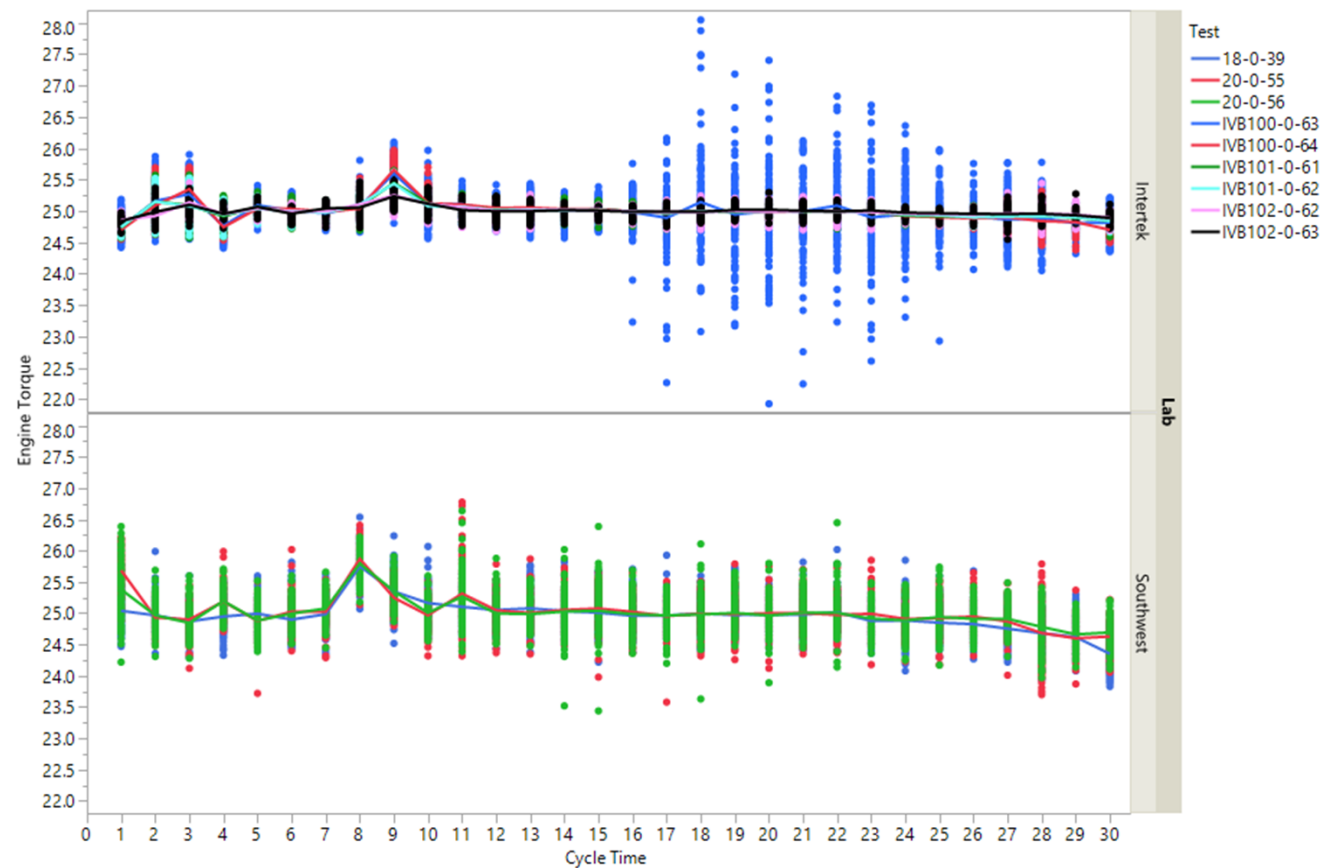




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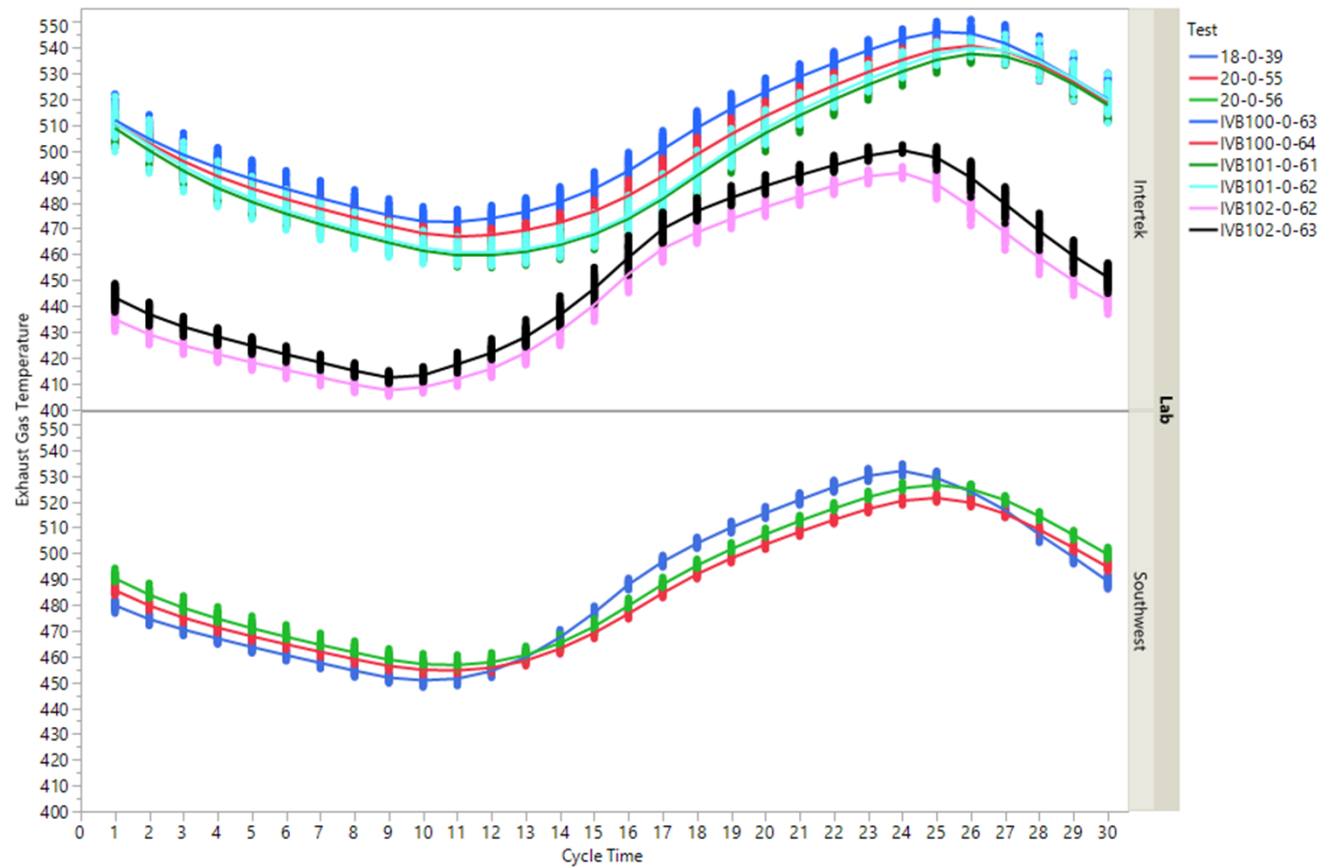


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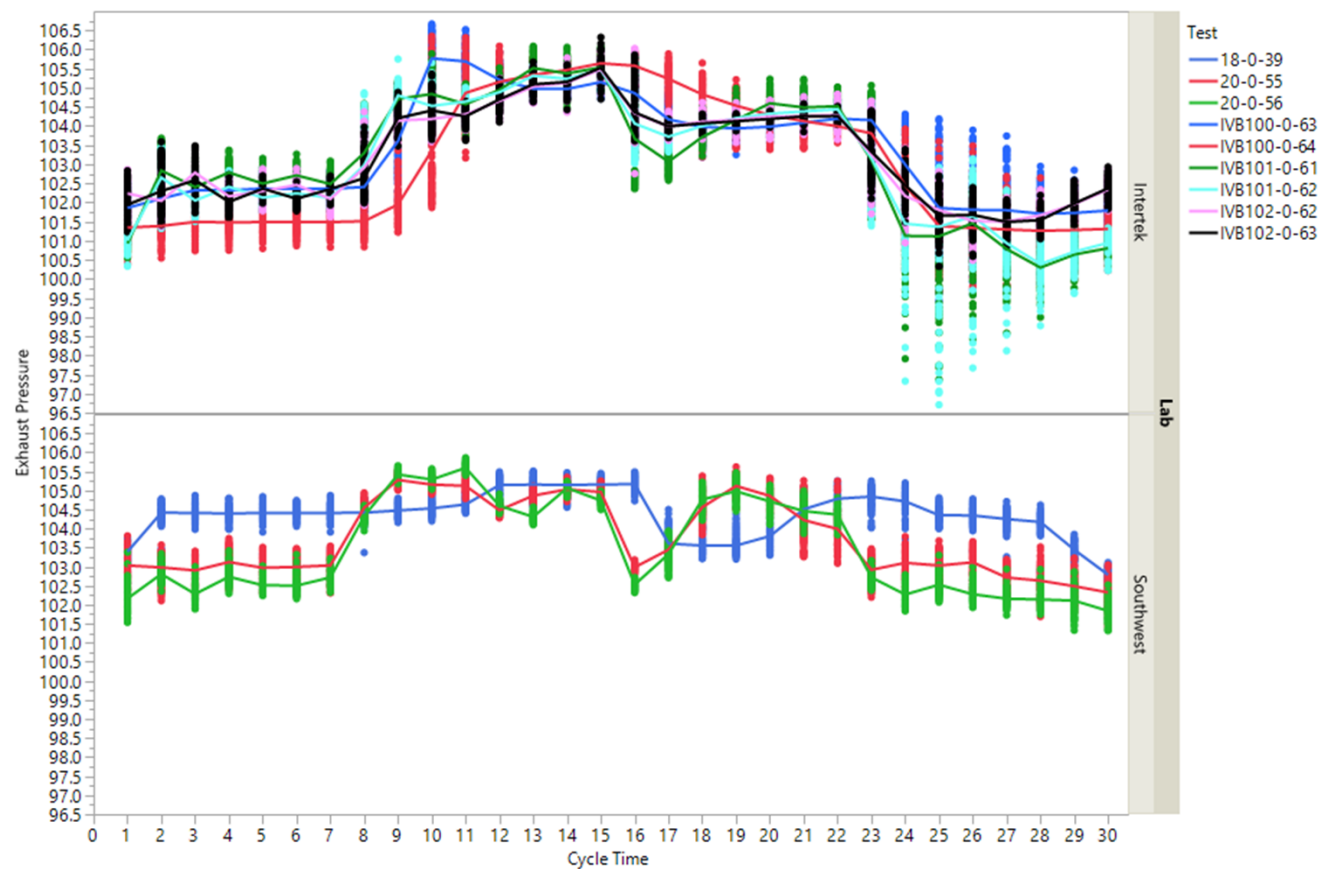
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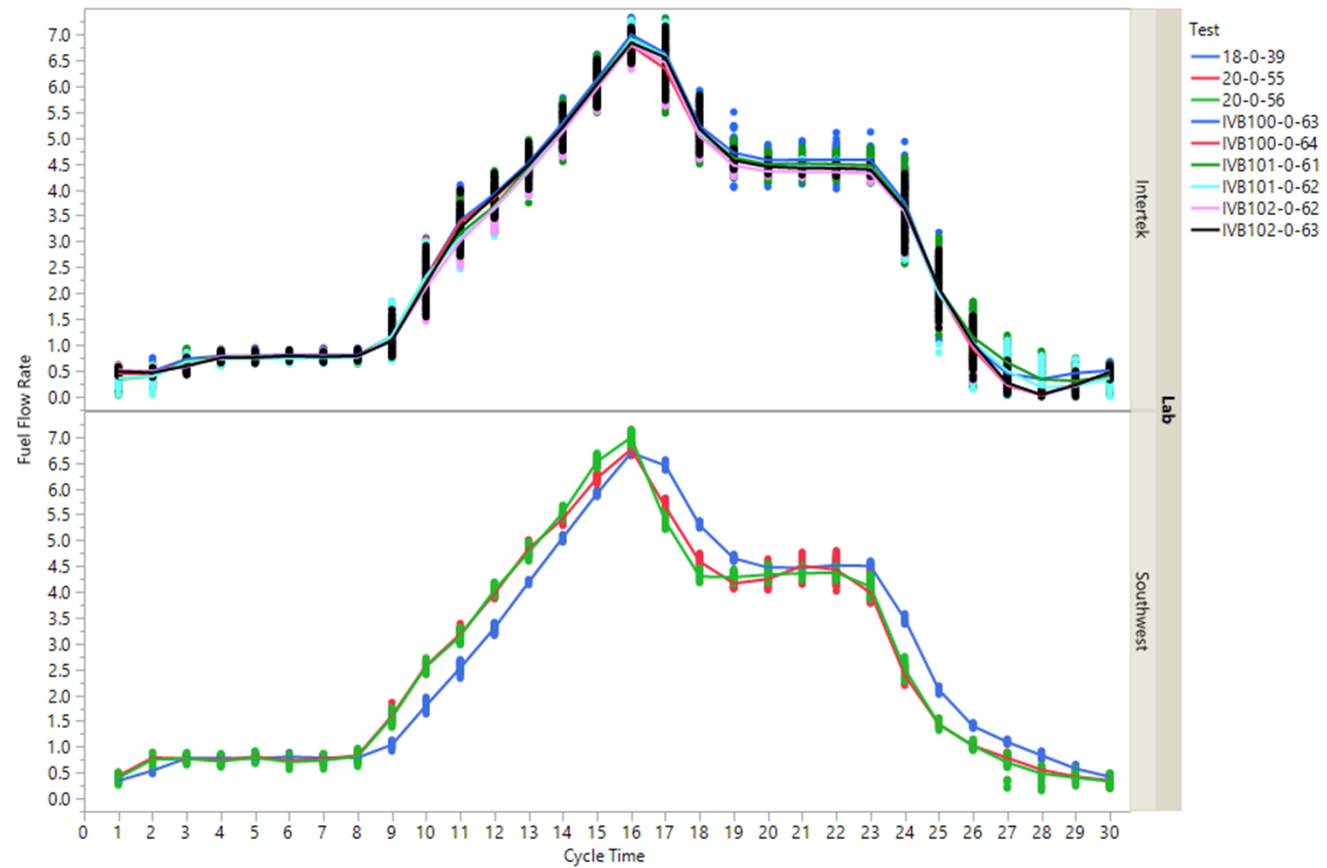
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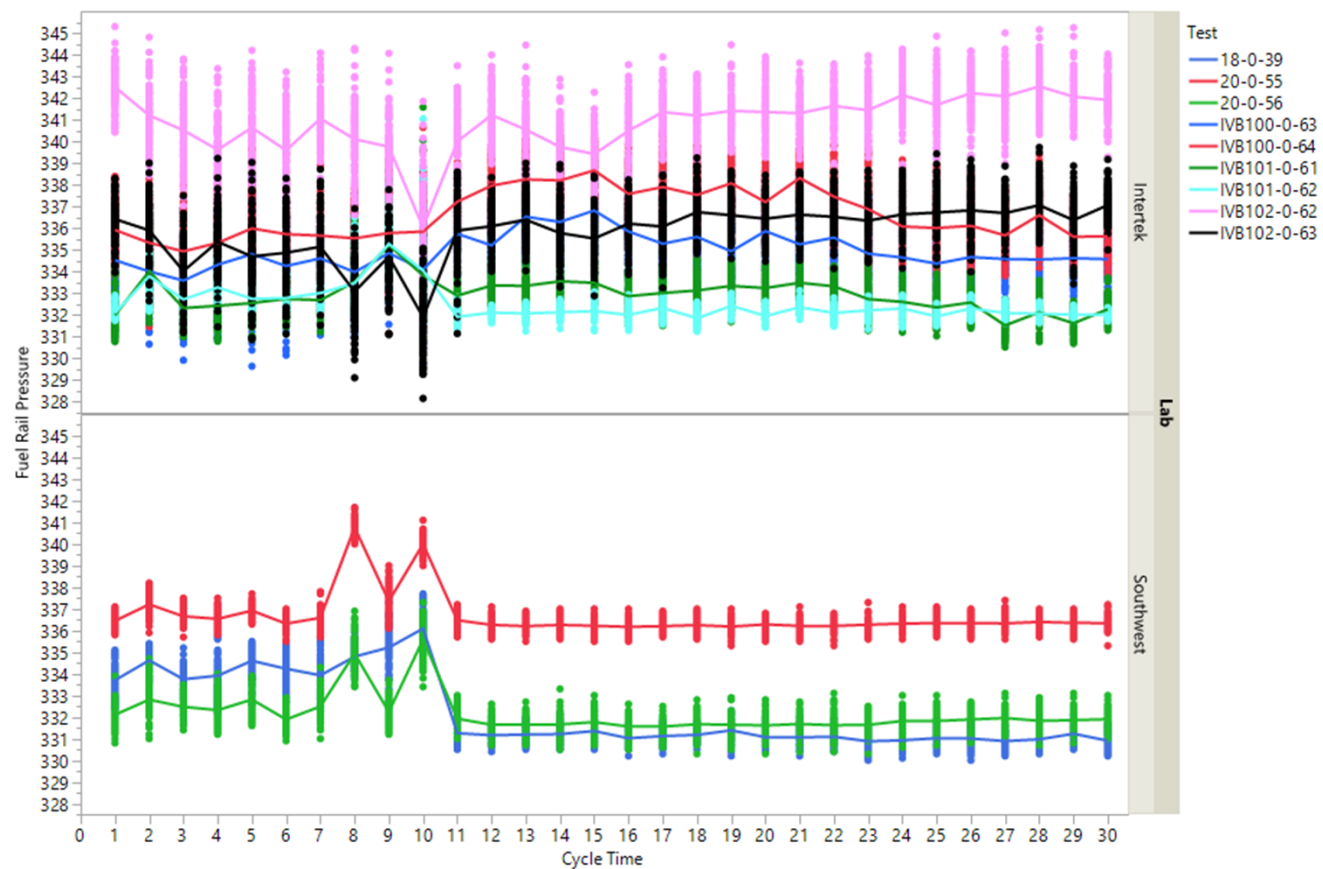
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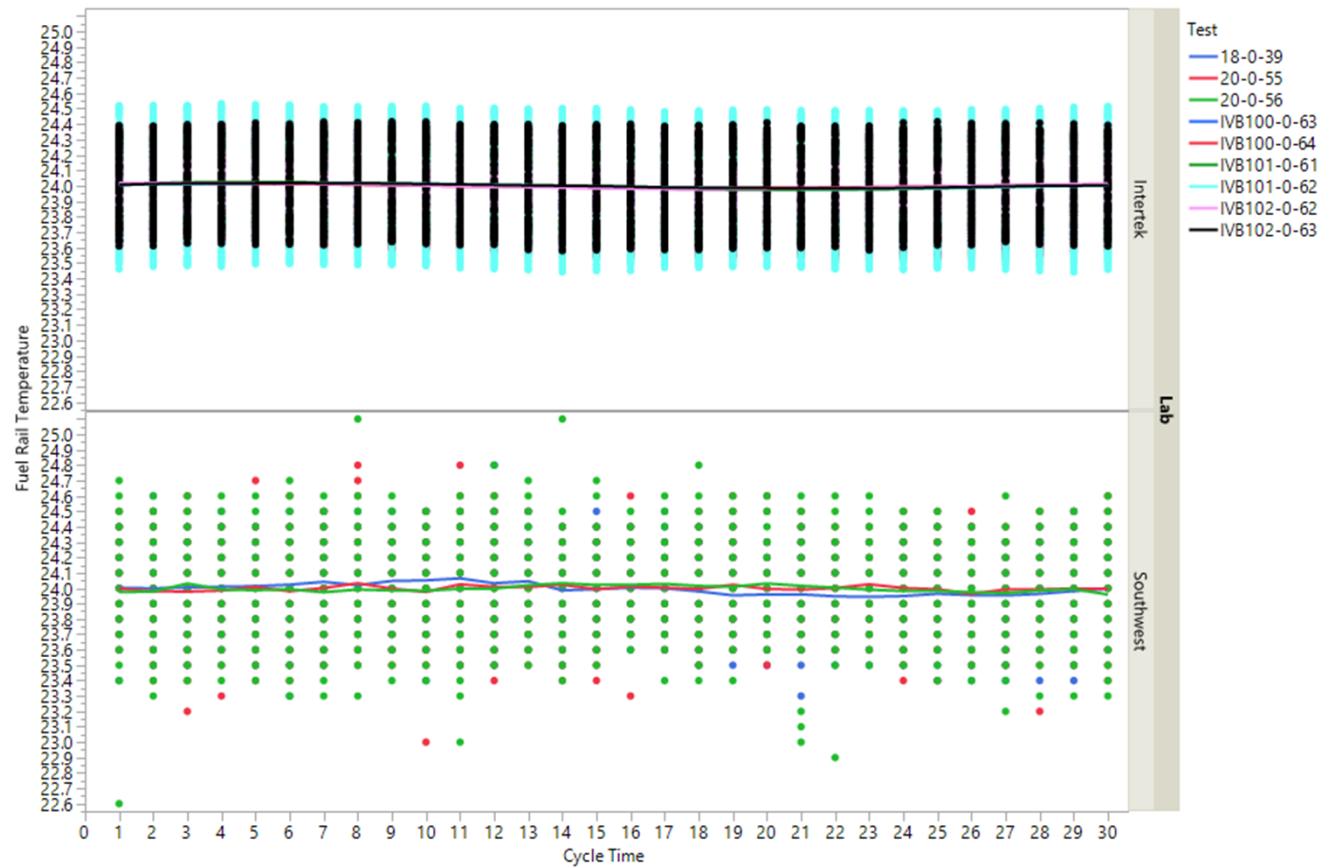


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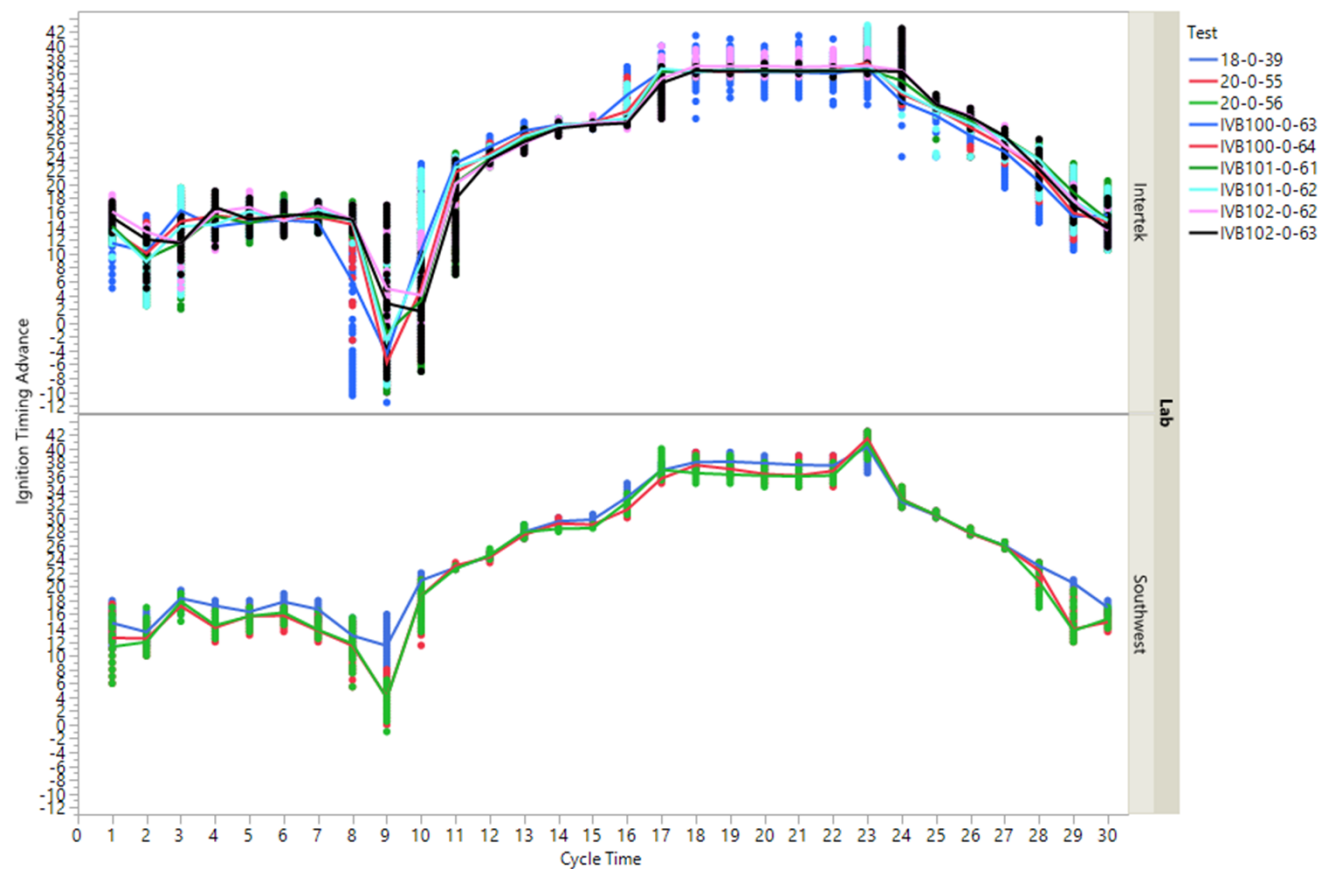
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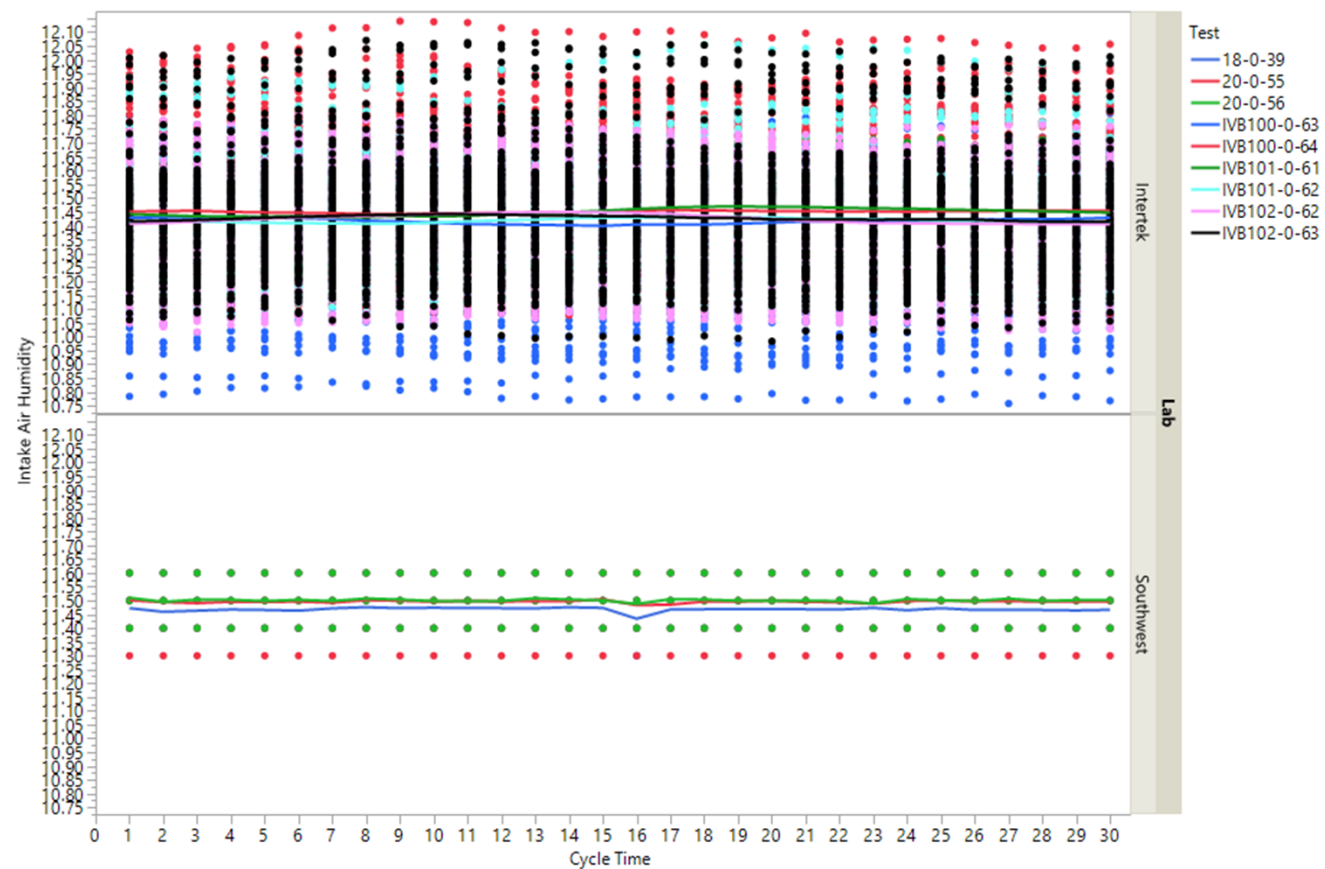


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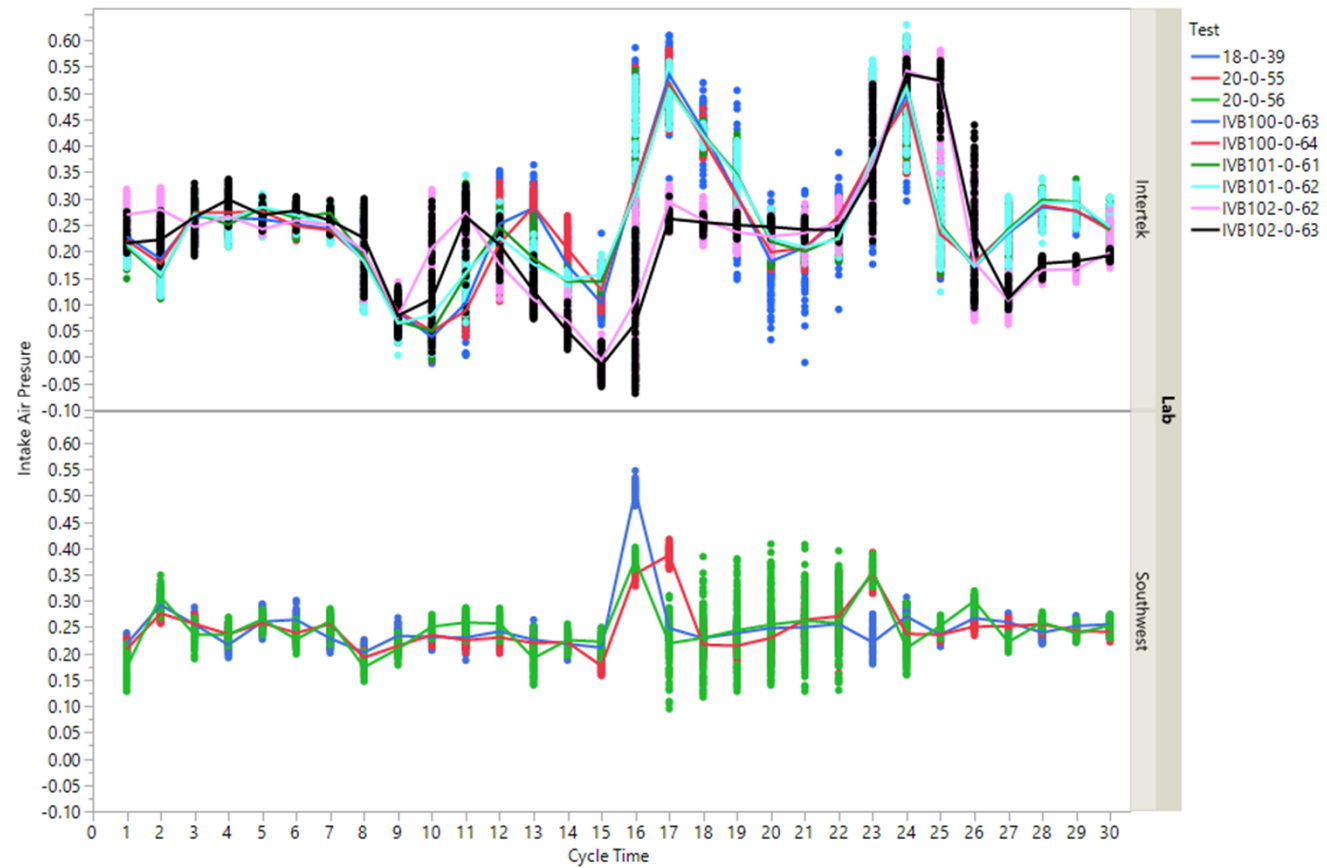




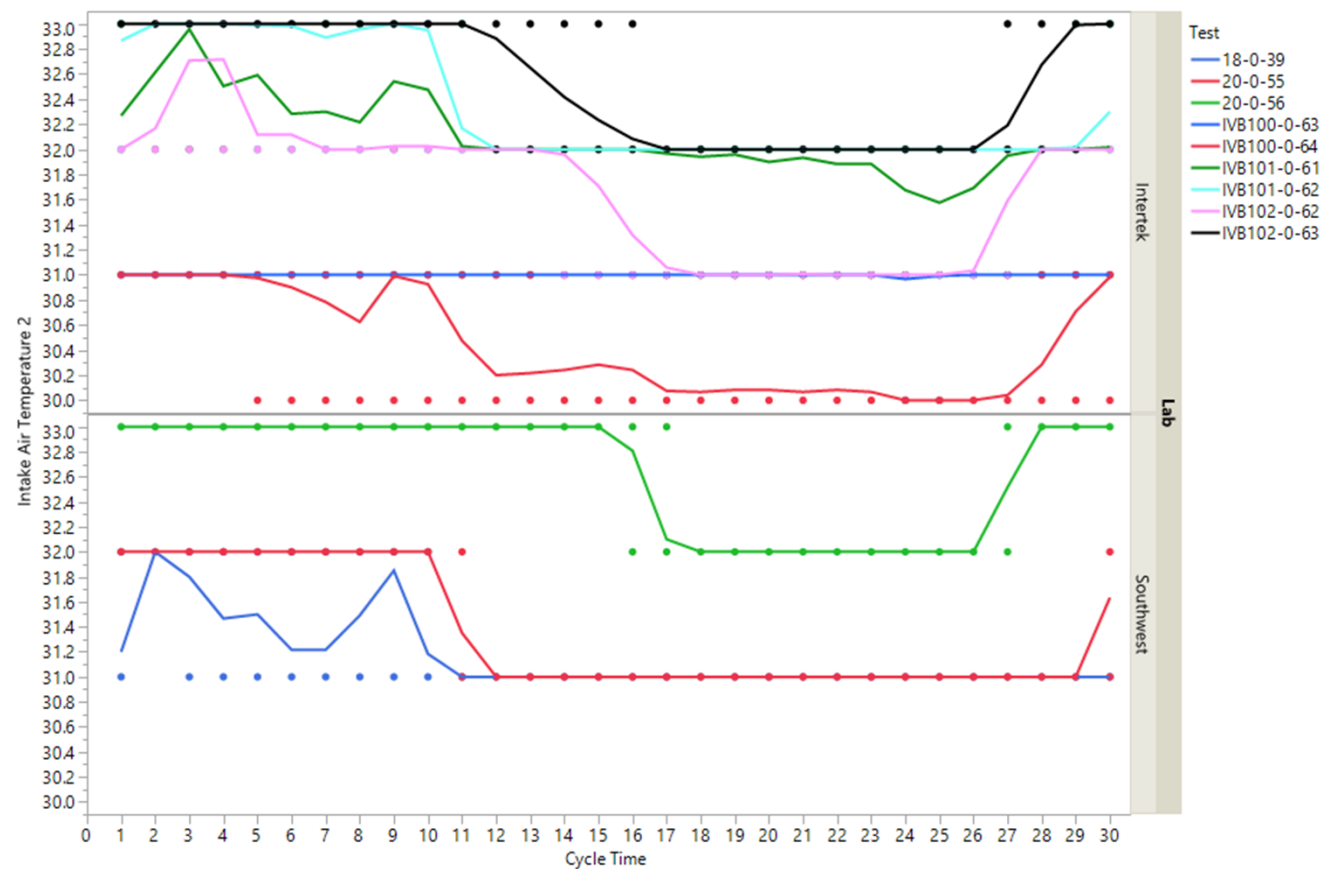
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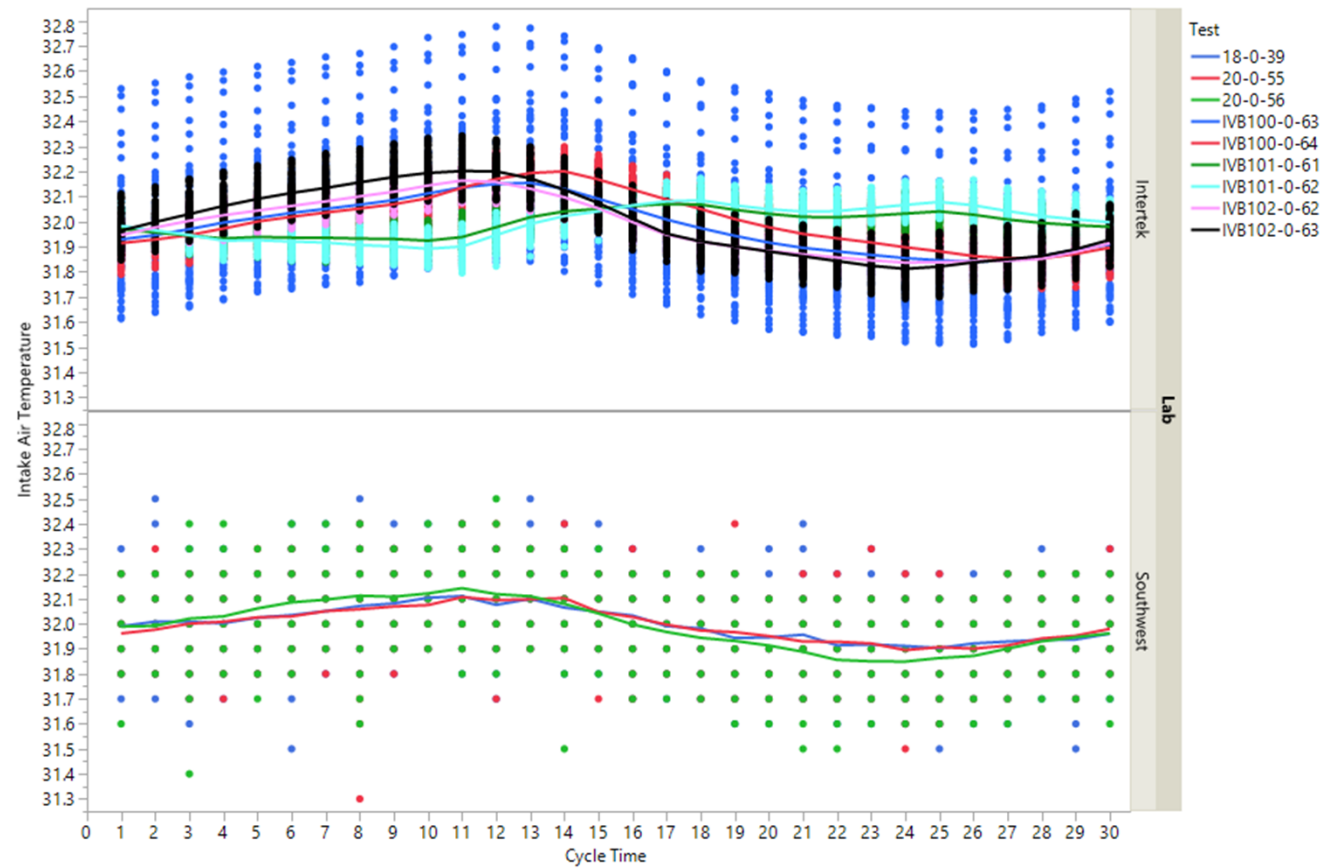
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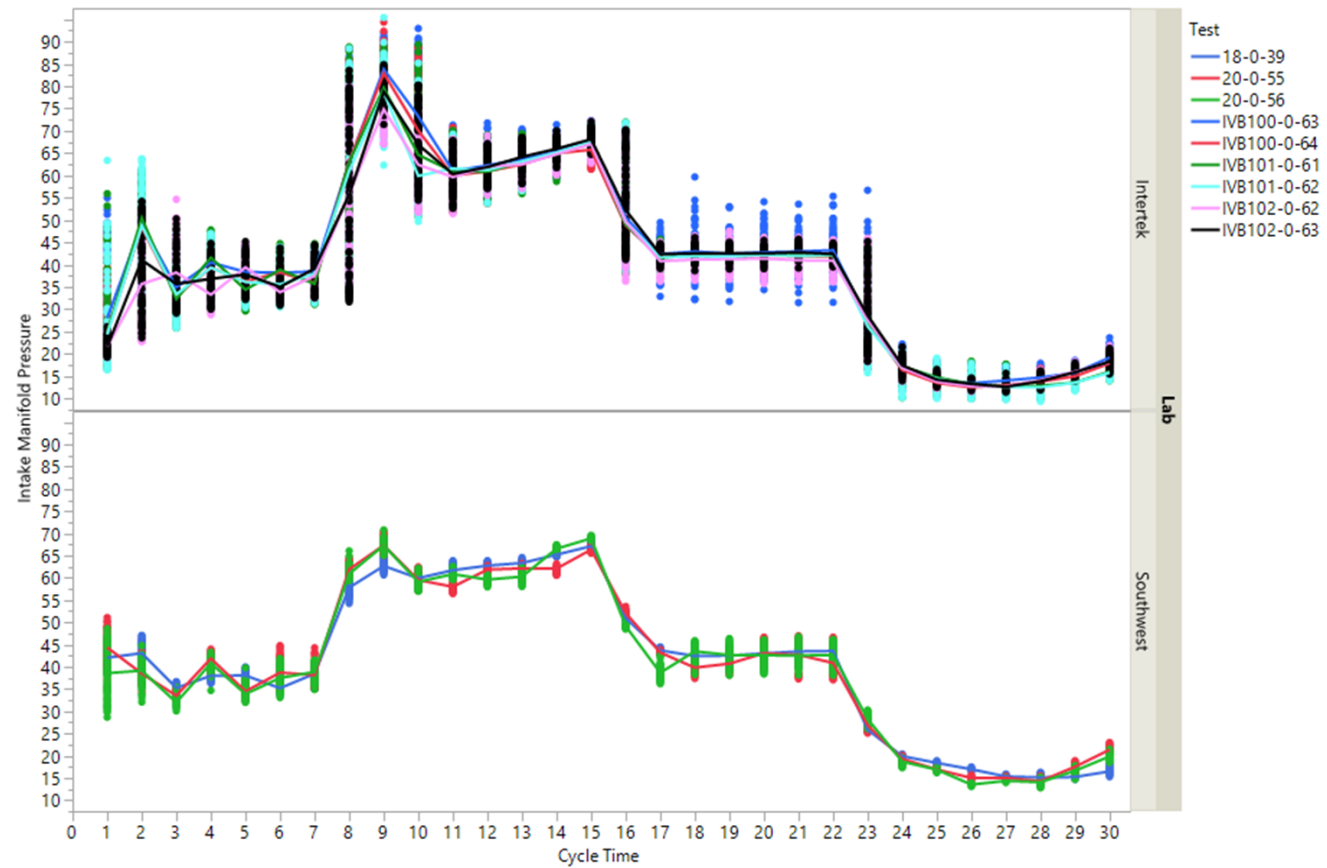


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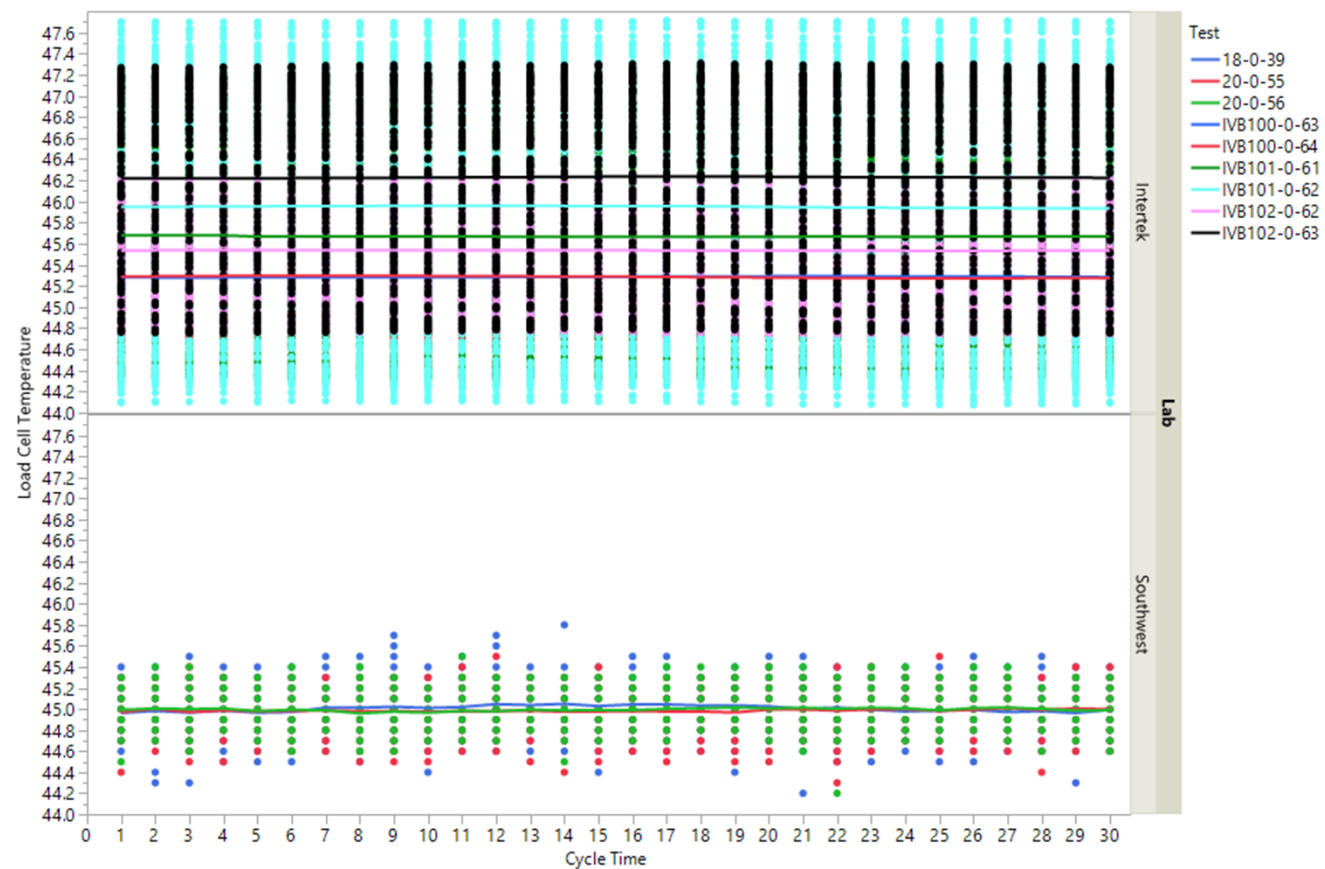
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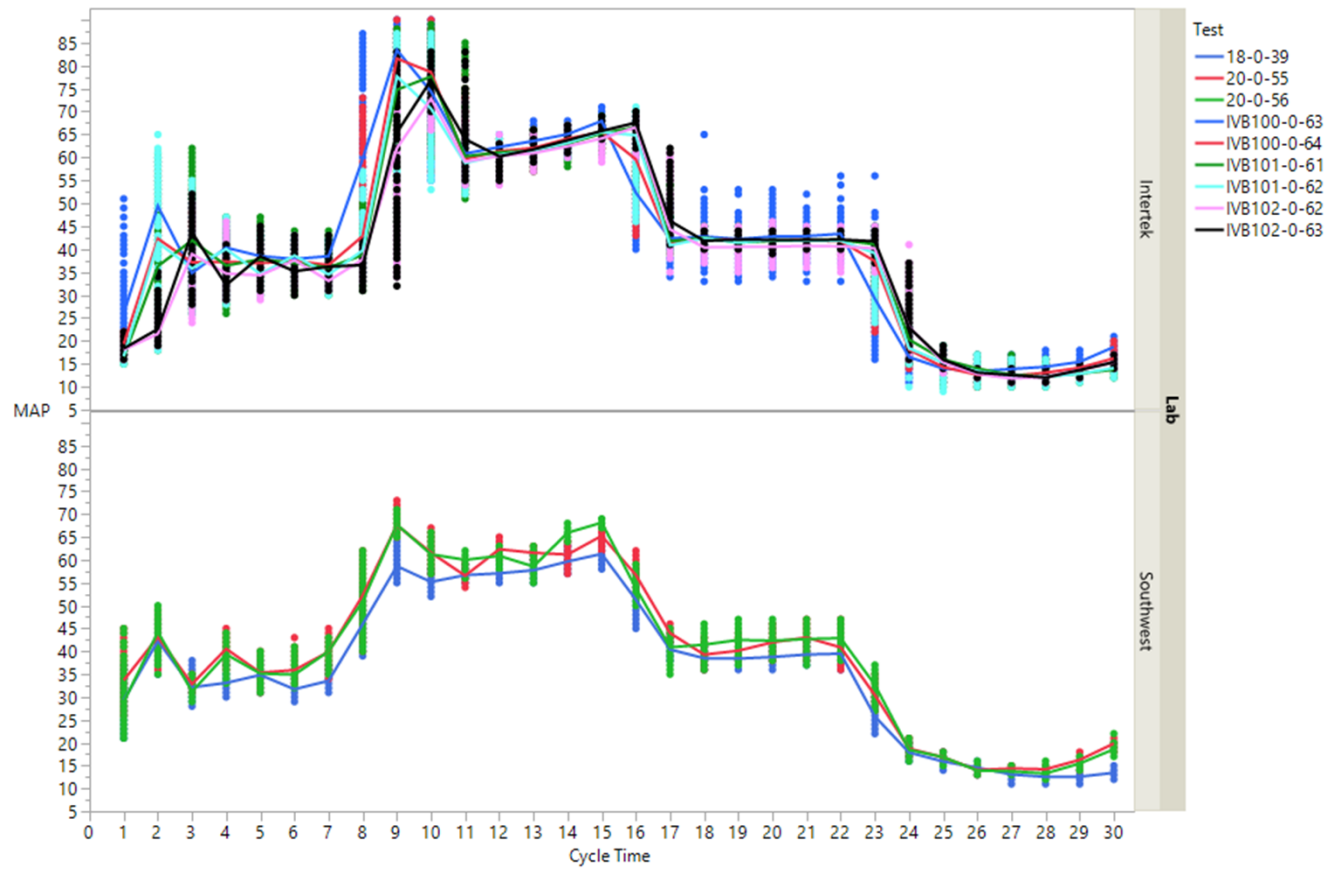


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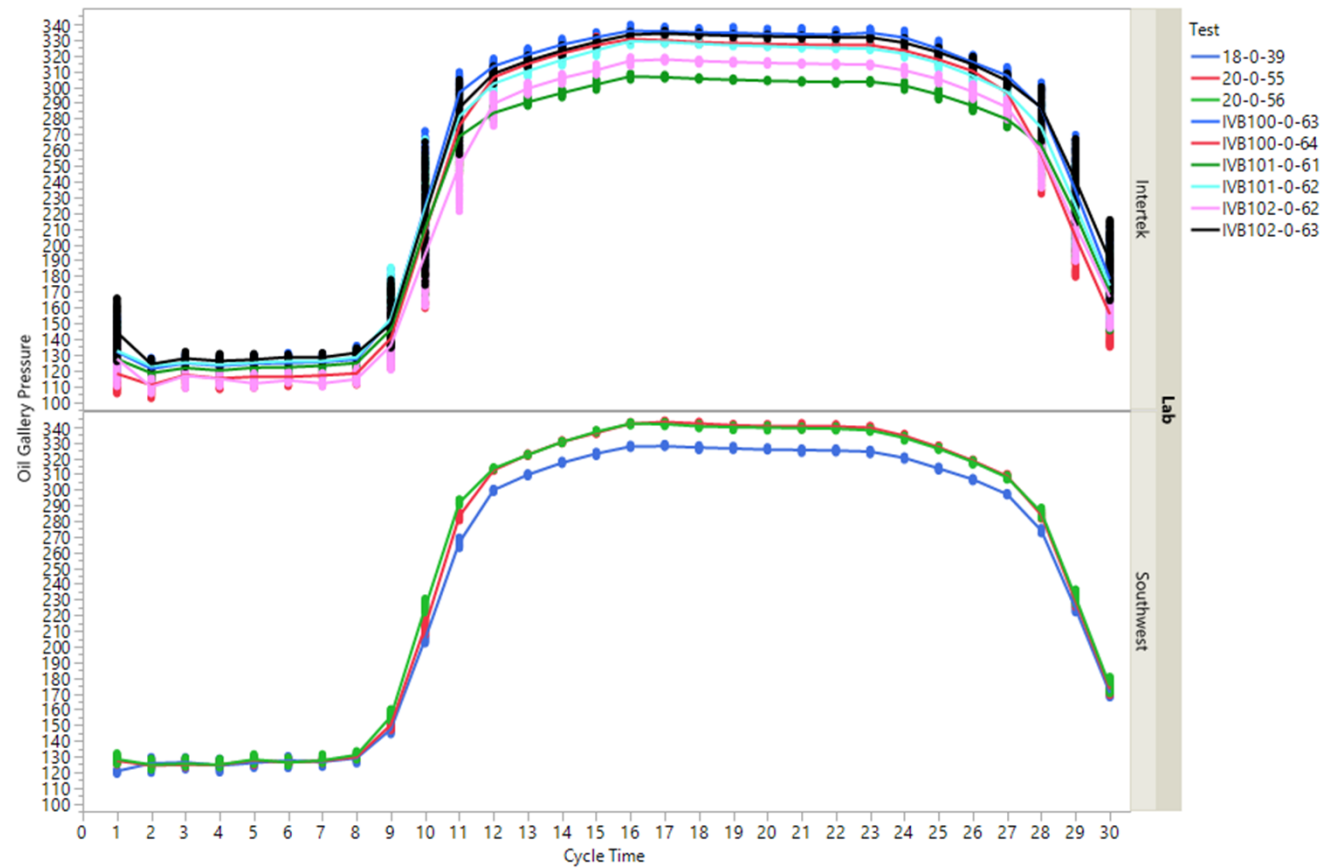
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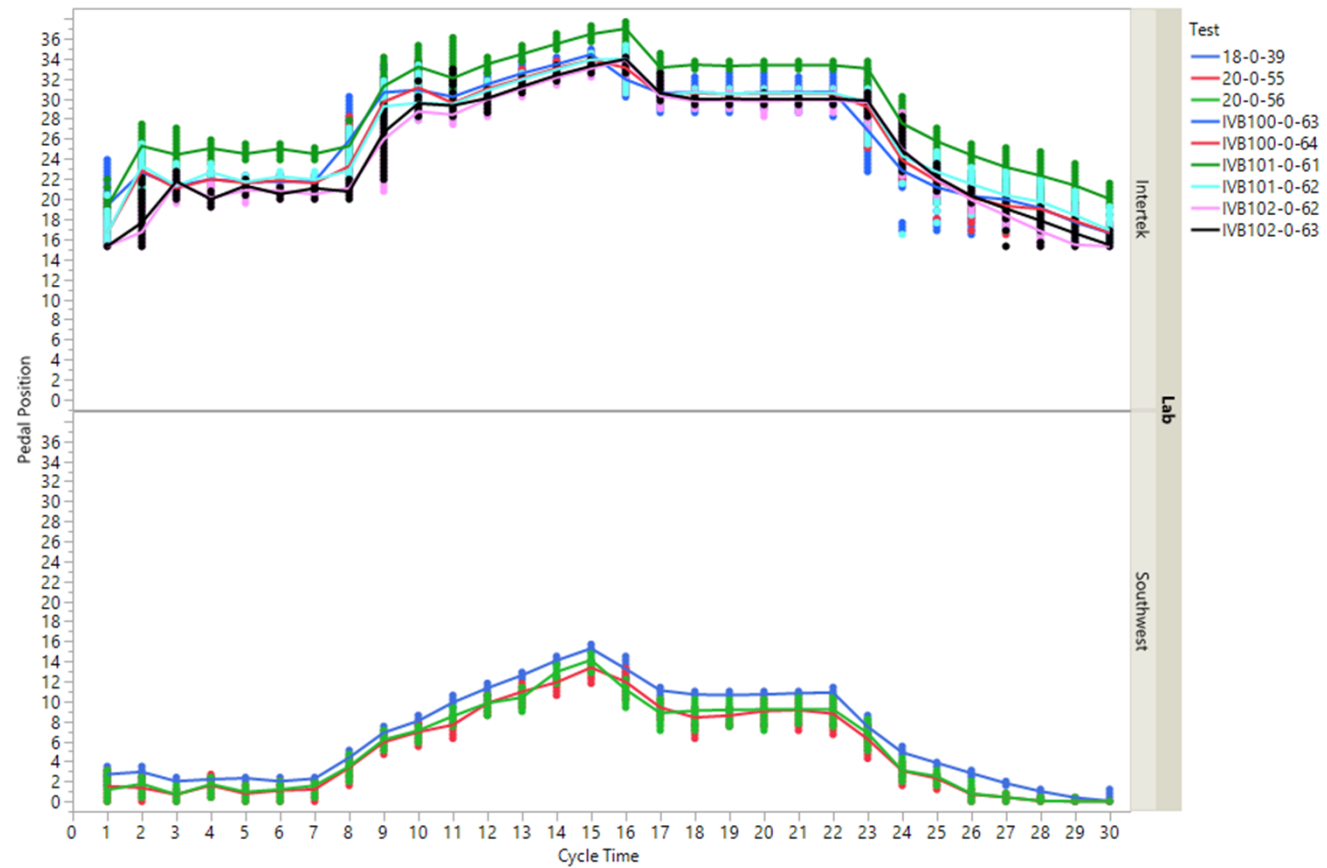


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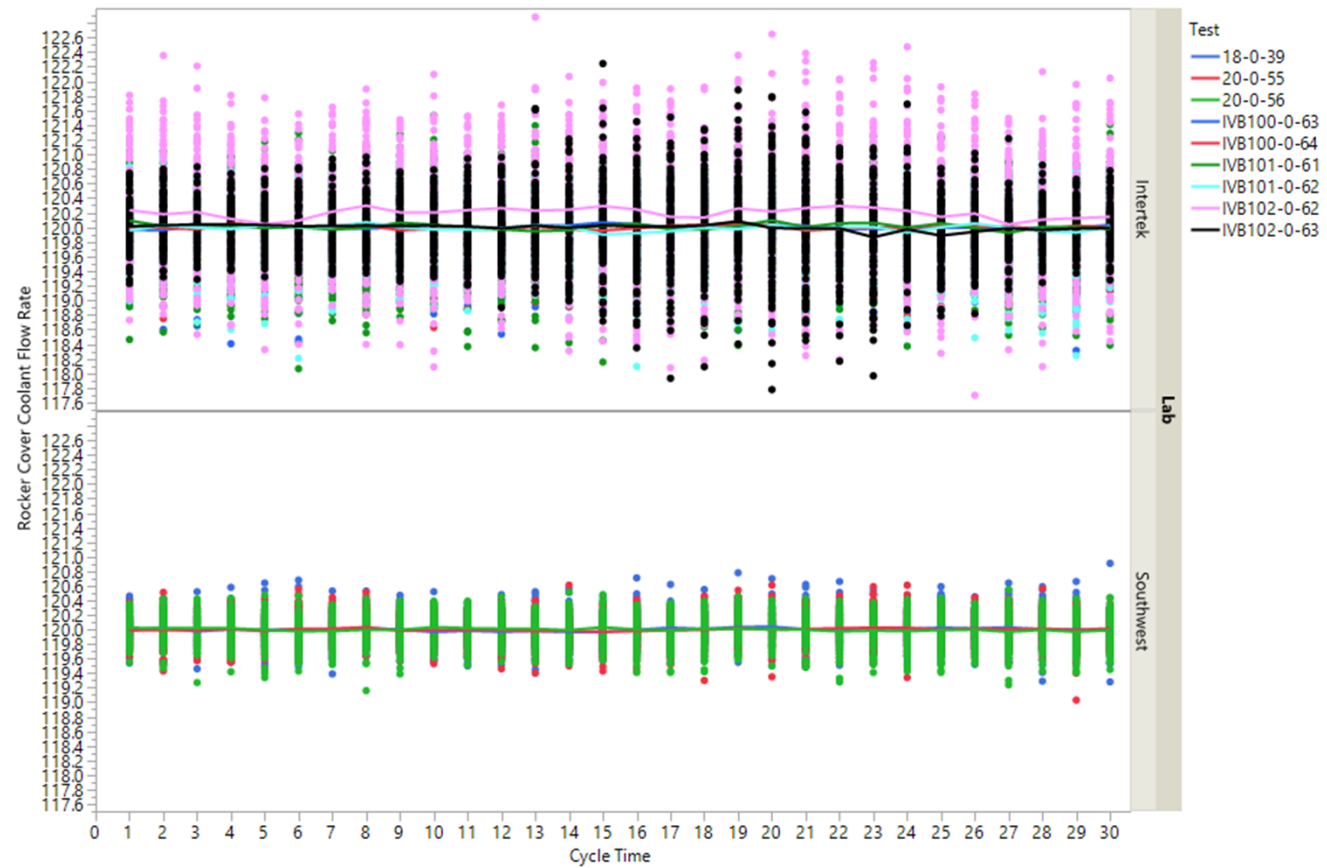




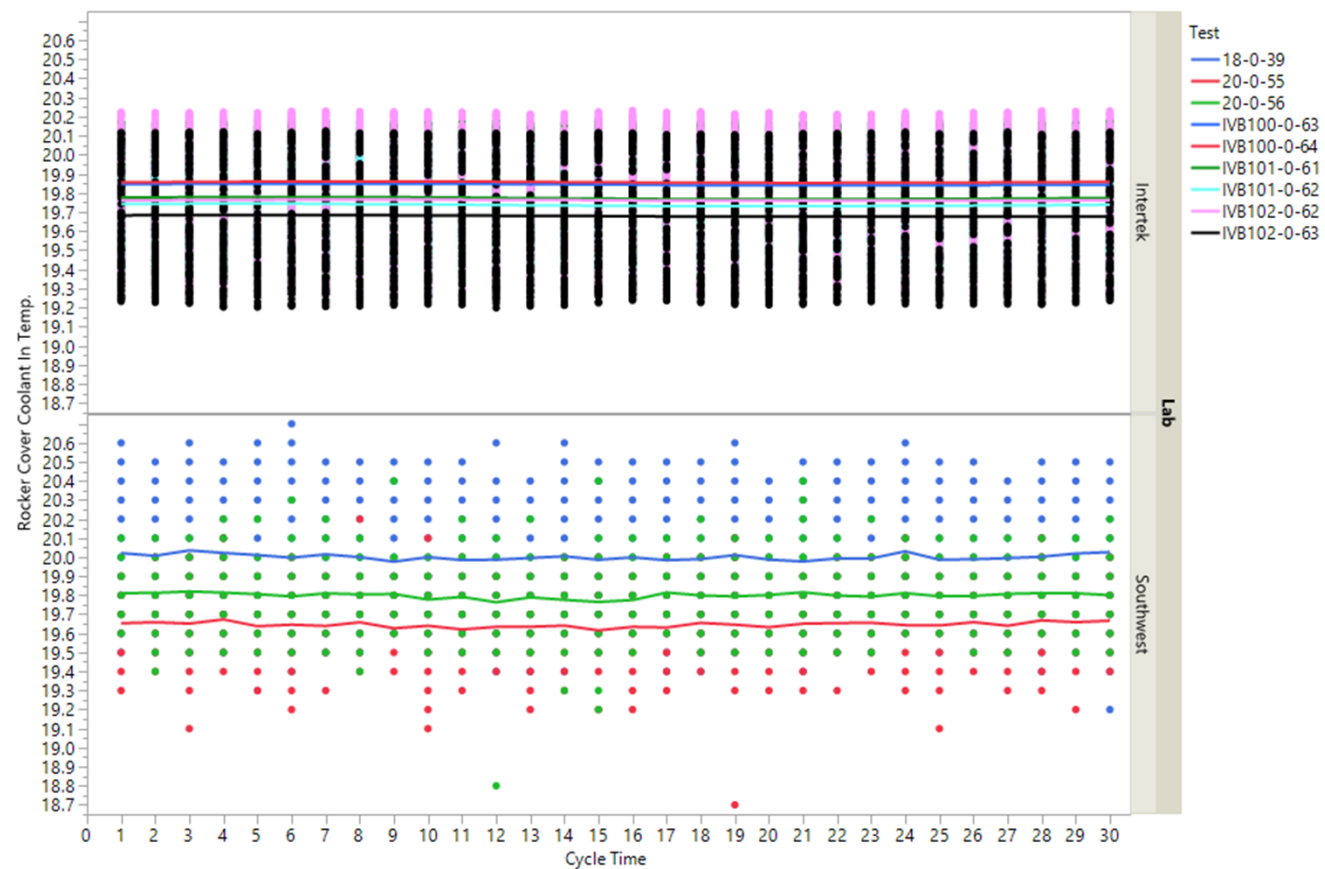
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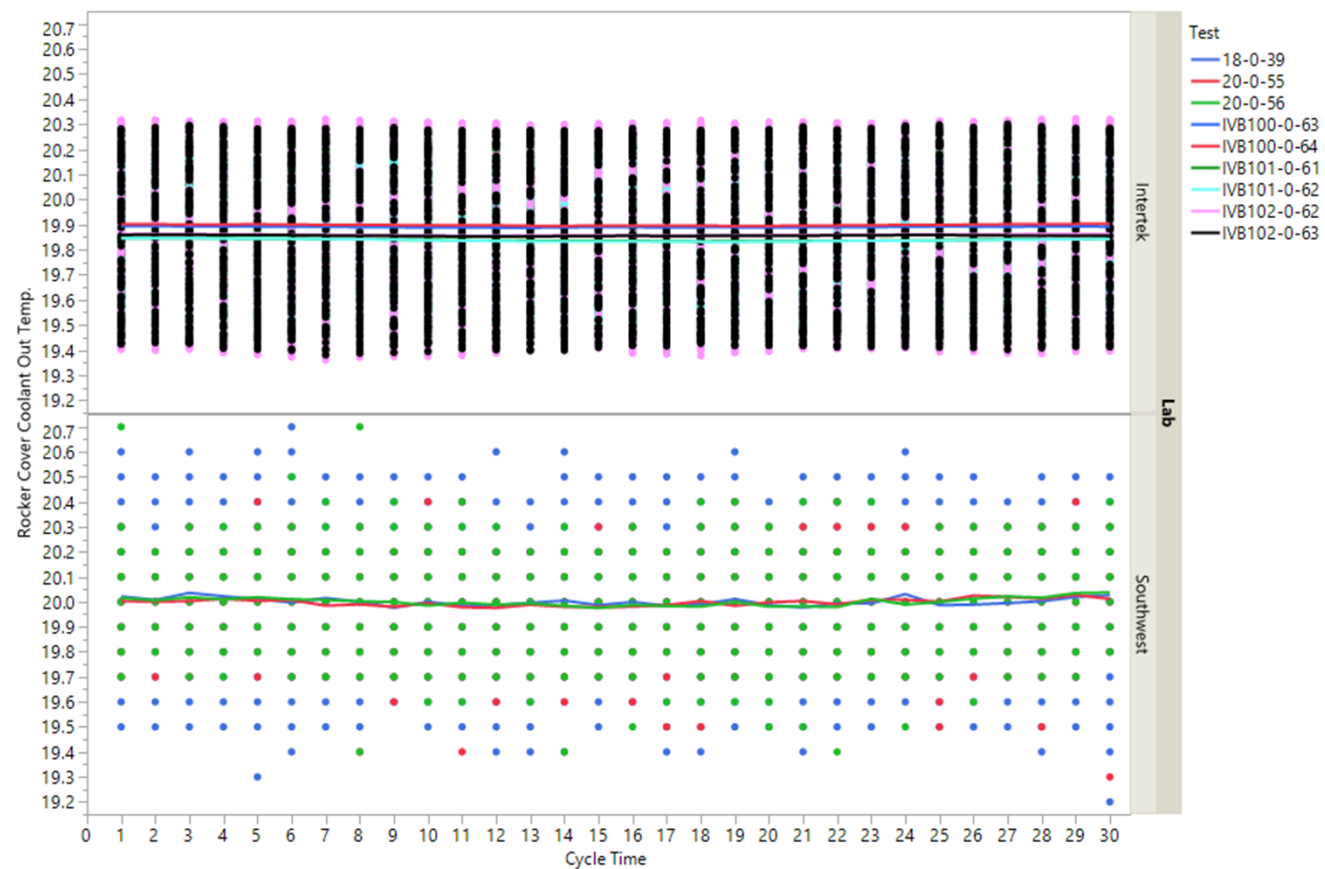
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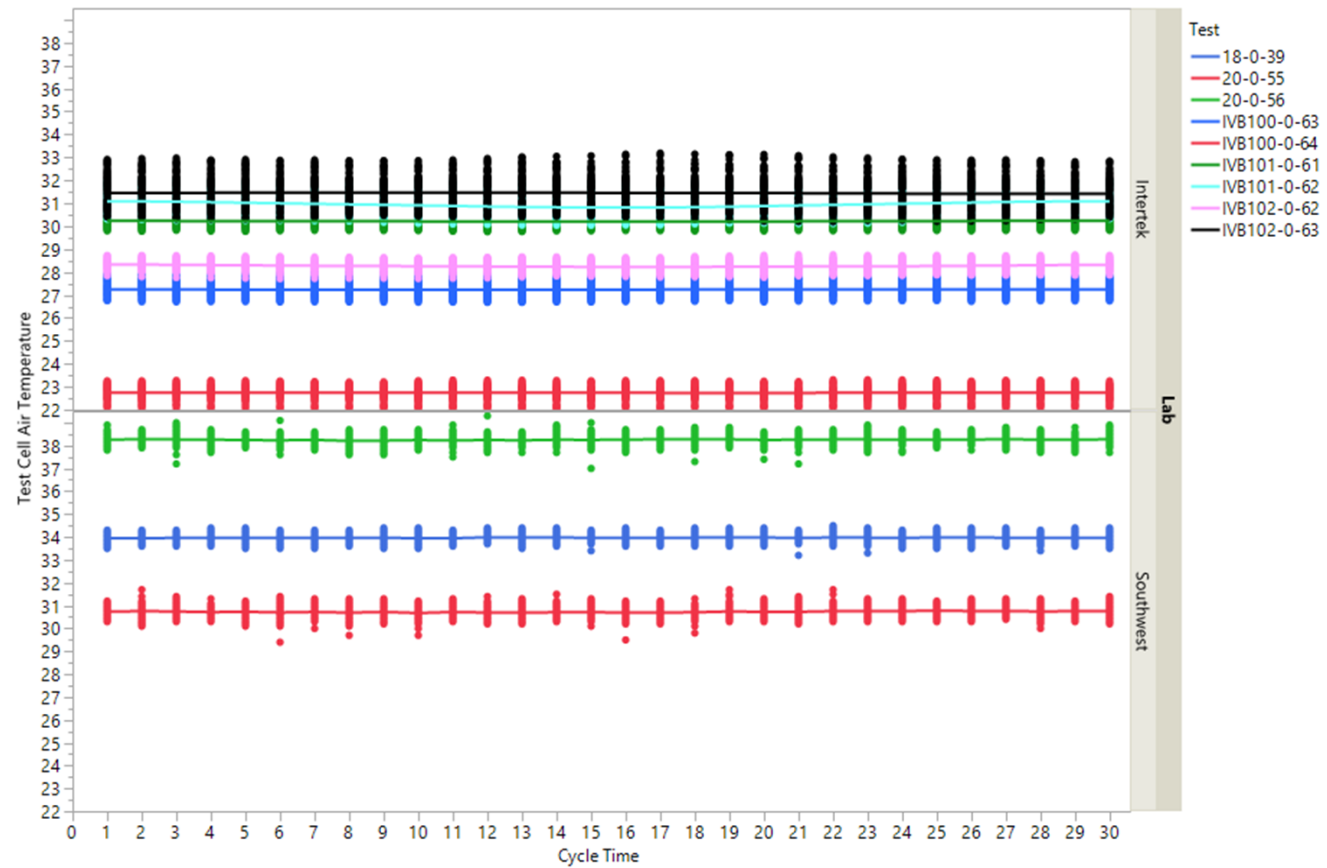
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# Graph Builder

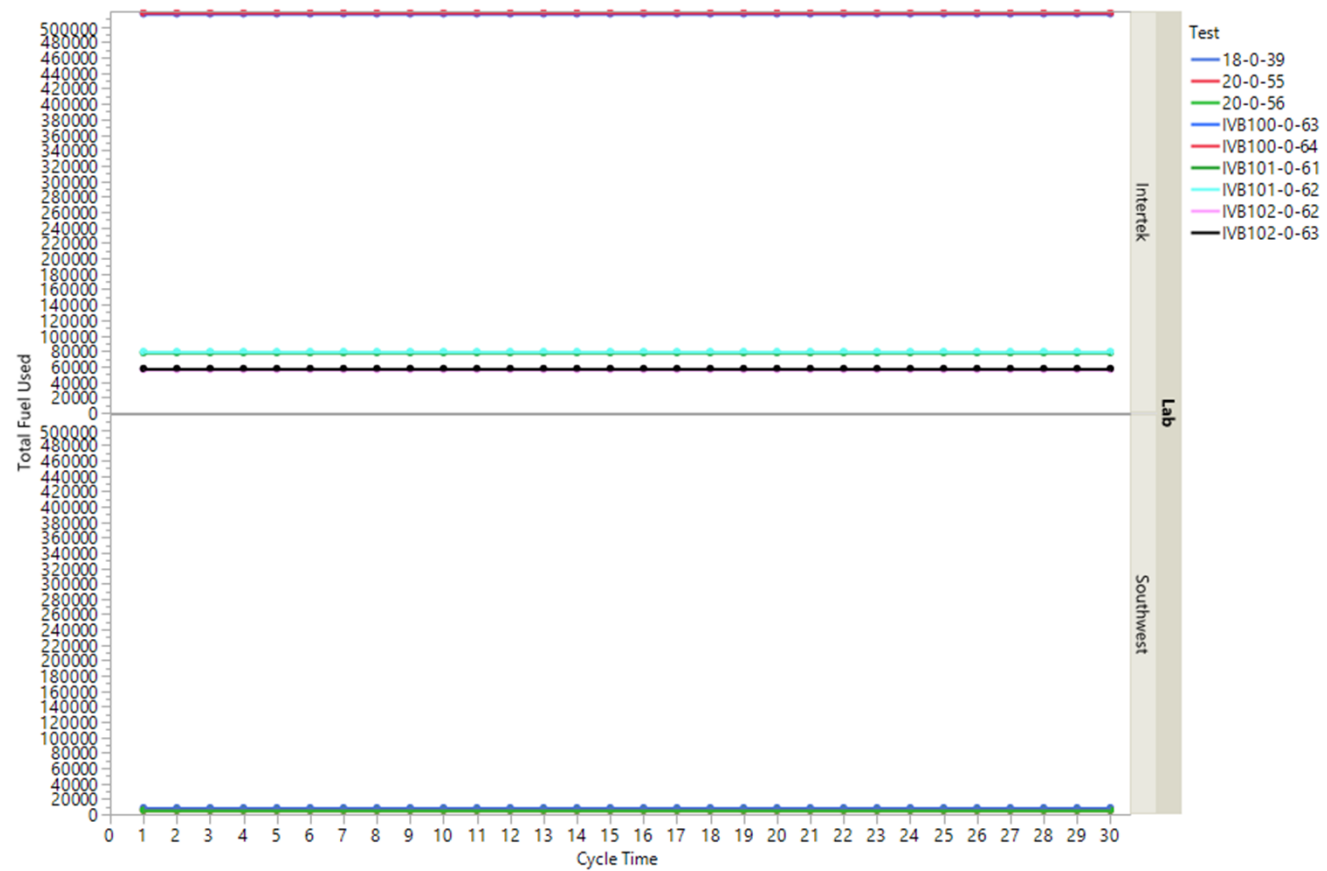


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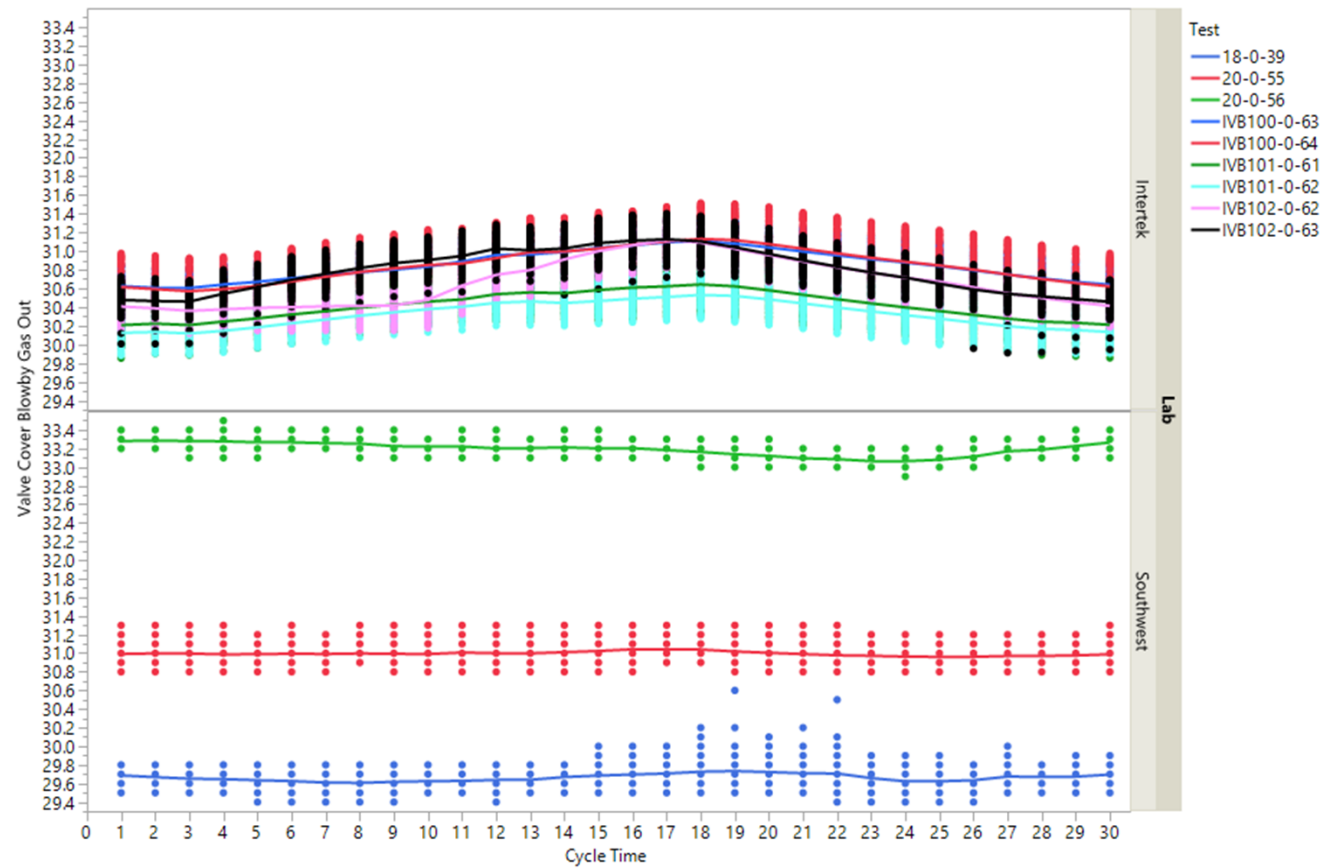
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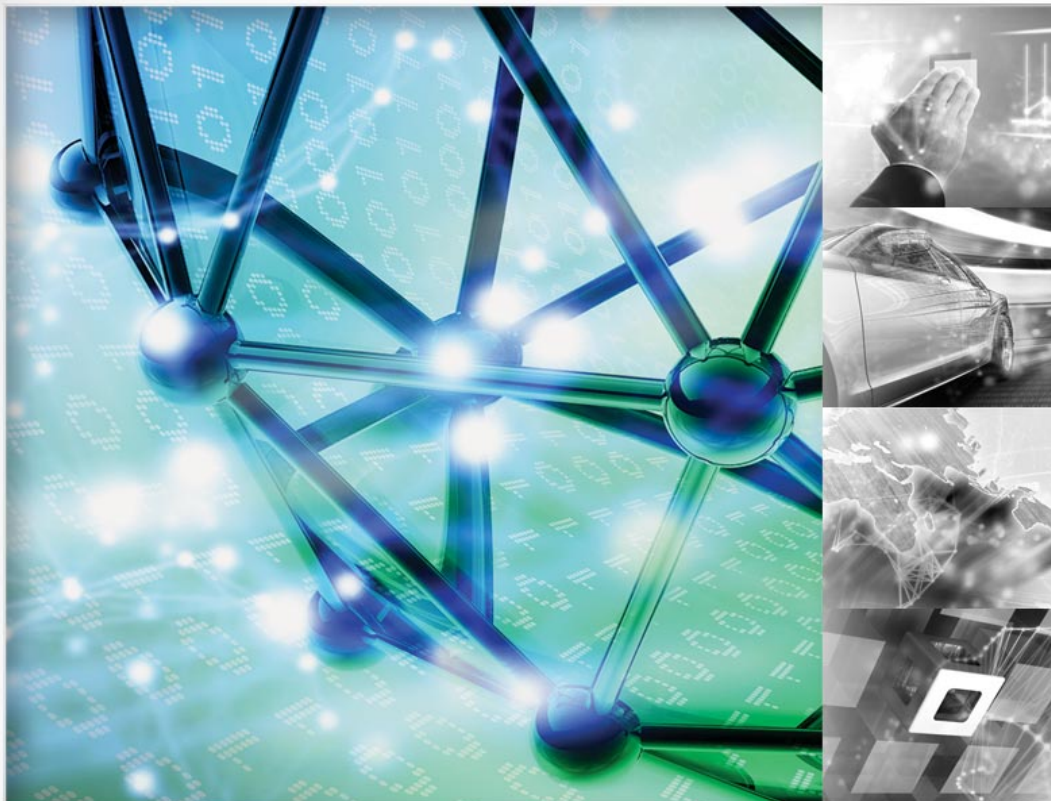
# Graph Builder



Where(50400 rows excluded)

# Graph Builder





# D3525 Capillary Column Chromatography

## A Better Approach

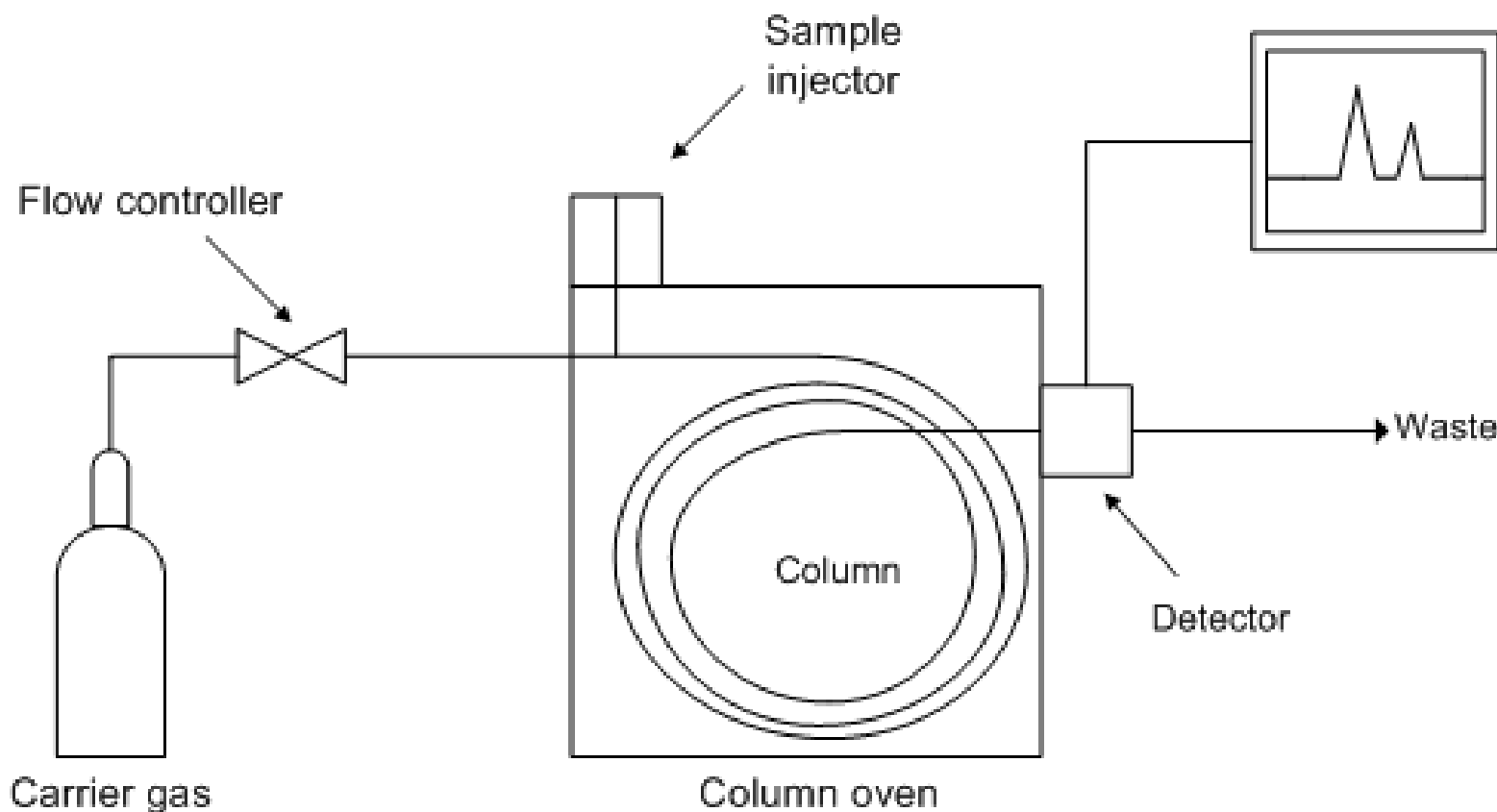
November 2017



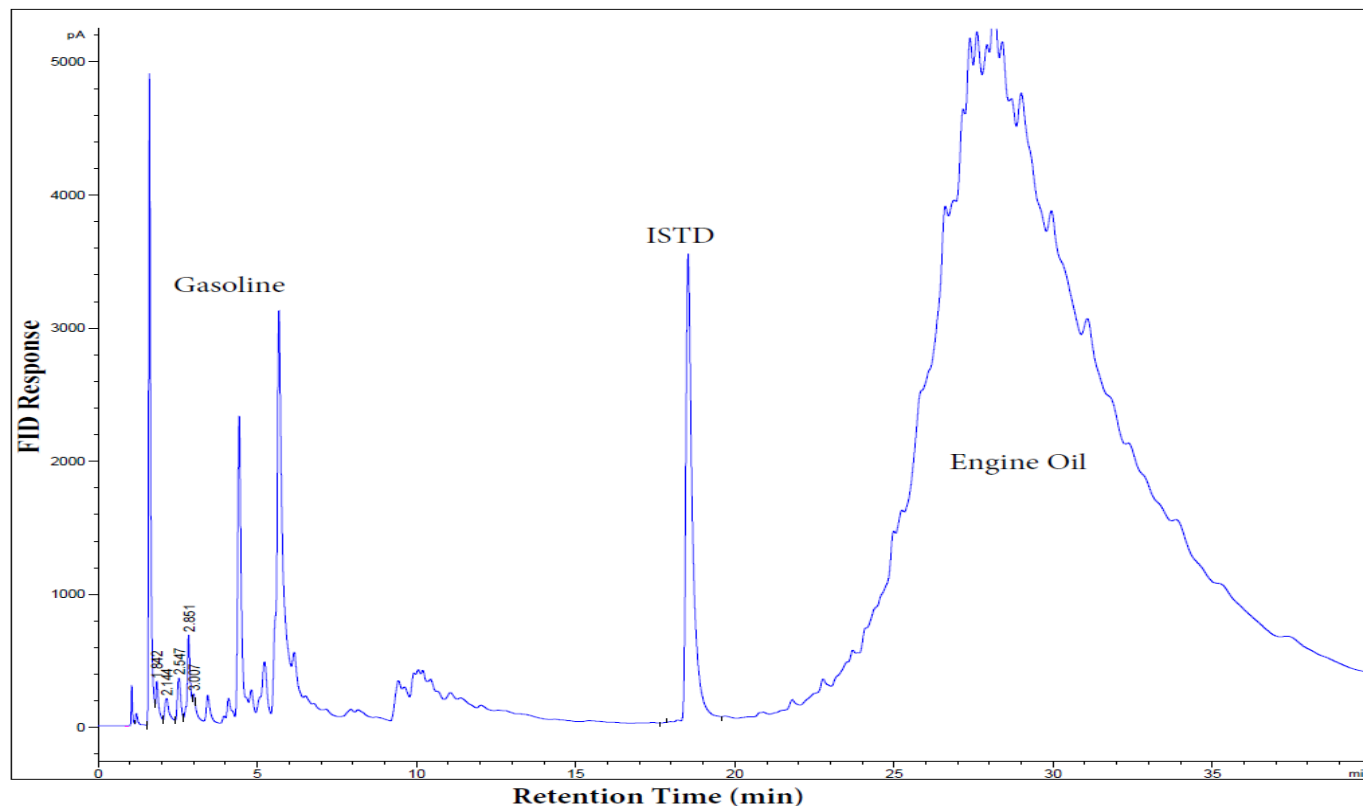
- D3525 Determines Gasoline Fuel Dilution in used engine oils by Gas Chromatography (GC).
- Fuel Dilution lowers the Viscosity of the Engine Oil.
- D3525 is a Child or Subtest of Gasoline Engine Testing Methods:
  - D6891 (Sequence IVA)
  - D6593 (Sequence VG)

# Gas Chromatography (GC) – Background

## Gas Chromatography Separates Components of a Mixture



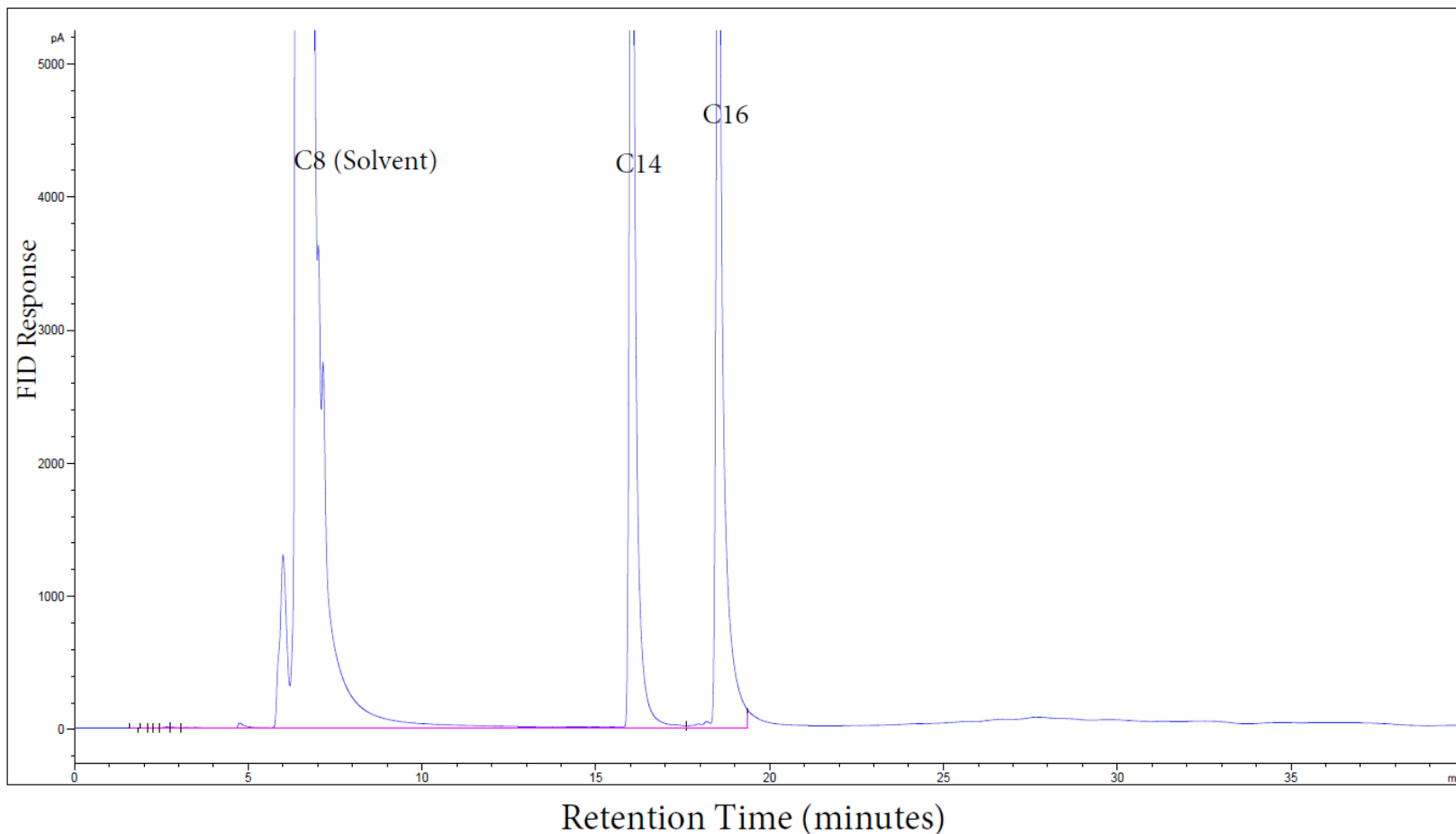
# D3525 Chromatogram – 8% Gasoline in Engine Oil



## D6593 & D6891 Specify a Different Internal Standard

- Parent Engine Tests specify **C16** n-Alkane
- D3525 Child Drain Test specifies **C14** n-Alkane
- This Internal Standard (ISTD) modification defines a *different* Chromatographic Window (different analyte):

# C14 vs. C16 n-alkane Internal Standard



**Different ISTD define Different chromatographic Analytes!**

- Efforts are currently underway to modify D3525 to specify the C16 n-Alkane Internal Standard
  - Work Item WK59475
  - Task Group being formed
  - ILS being organized

## **D6593 & D6891 Specify Packed Columns**

- Parent Engine Tests limit the subtest to Packed Columns.
- D3525 allows both Packed and Capillary Columns.
- What's the difference?

# See It – D3525 Packed vs. Capillary GC Columns

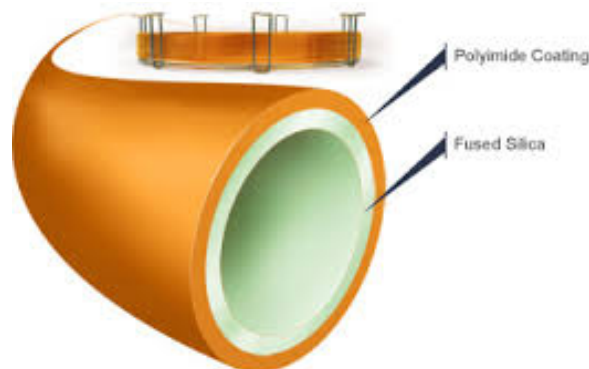
## Packed Columns

- 1/4" (3.2 mm) SS Tubes
- ~ 6 - 10 Feet long
- Packed with coated beads (Stationary Phase)



## Capillary Columns

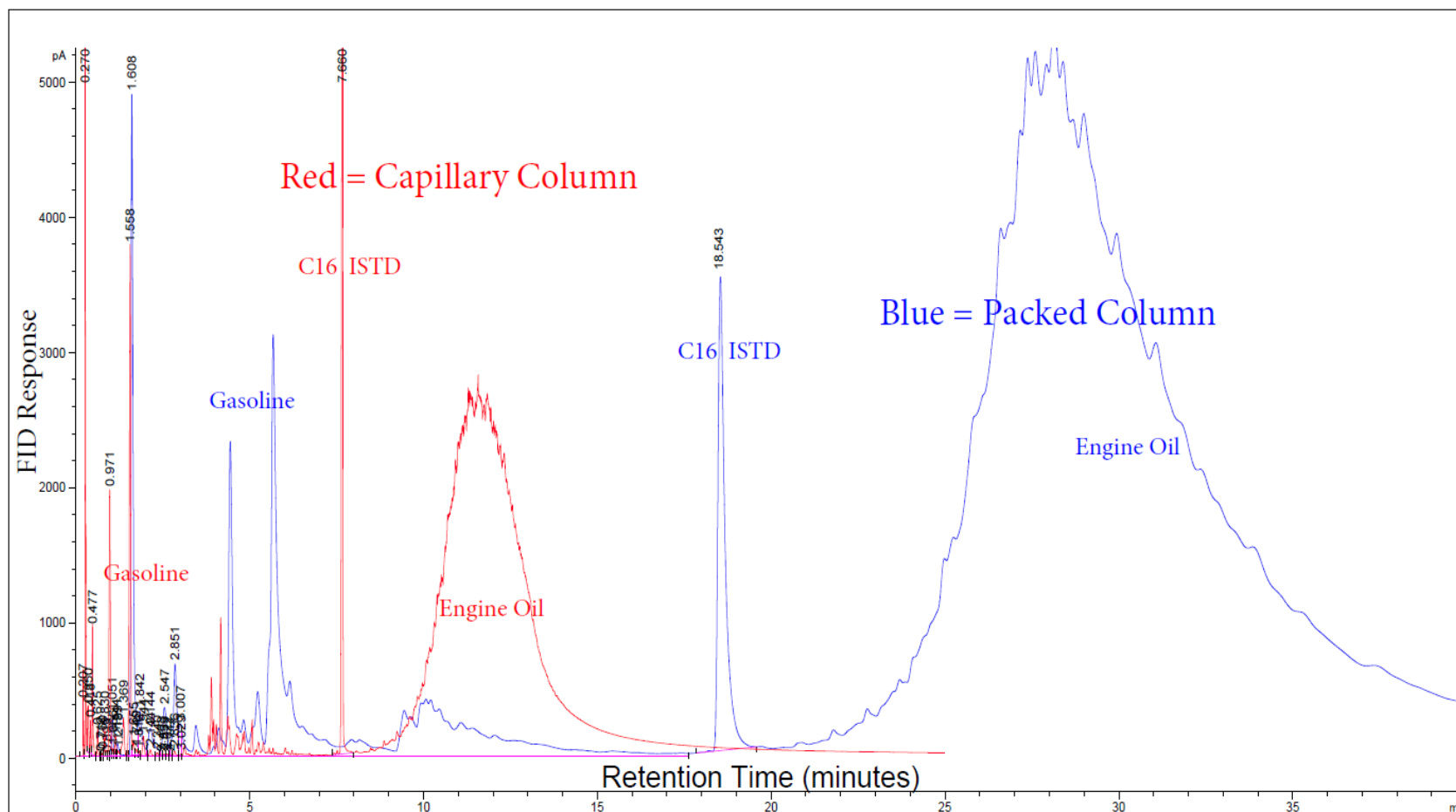
- 0.53 mm Open Tubes made of Fused Silica
- ~ 5 - 30 meters long
- Stationary Phase is coated on the inside of the tube





# D3525 Packed vs. Capillary Columns

## 8% Gasoline in Engine Oil



# Capillary Column GC is Better!



- **Capillary Columns** outperform Packed Columns:
  - Better Resolution
  - Faster
  - More Consistent (better precision)
- NOTE: The *Injection Volume* for the Capillary Column is *Smaller*.
  - Consequently, the Gasoline, C16 ISTD, and Engine Oil profiles are all *smaller* in the Capillary Chromatogram.
  - However, the *ratio* of Gasoline to C16 ISTD peak areas is the same as on the Packed Column giving the same calculated concentration.

# Appeal to Sequence IV & V Surveillance Panels



- Either Specify or Allow the use of Capillary Columns when specifying Fuel Dilution in used oil samples by Gas Chromatography in D6891 and D6593.

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1-440-347-6589





## Working together, achieving great things

When your company and ours combine energies, great things can happen. You bring ideas, challenges and opportunities. We'll bring powerful additive and market expertise, unmatched testing capabilities, integrated global supply and an independent approach to help you differentiate and succeed.

***Draft of the wording that was generated during the August 30, 2017 Technical Guidance Committee Meeting Conference Call:***

**Alternate Supplier Protocol**

ASTM International policy is to encourage the development of test procedures based on generic equipment. It is recognized that there are occasions where critical/sole-source equipment has been approved by the technical committee (surveillance panel/task force) and is required by the test procedure. The technical committee that oversees the test procedure is encouraged to clearly identify if the part is considered critical in the test procedure. If a part is deemed to be critical, ASTM encourages alternate suppliers to be given the opportunity for consideration of supplying the critical part/component providing they meet the approval process set forth by the technical committee.

An alternate supplier can start the process by initiating contact with the technical committee (current chairs shown on ASTM TMC website). The supplier should advise on the details of the part that is intended to be supplied. The technical committee will review the request and determine feasibility of an alternate supplier for the requested replacement critical part. In the event that a replacement critical part has been identified and proven equivalent the sole-source supplier footnote shall be removed from the test procedure.