

# ASTM New Engine Oil Gelation Test (EOGT) WK86363 Update

EOFT and EOWTT Surveillance Panel Meeting

November 20, 2023

Yong-Li McFarland, Chair



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# EOFT and EOWTT Surveillance Panel Membership

## 20 members

Beth Schwab, Afton Chemical

Michael Kunselman, Center for Quality Assurance

Robert Stockwell, Chevron Oronite

Dennis Gaal, Exxonmobil

Michael Deegan, Ford

Ron Shah, Infineum

Joe Franklin, Intertek

Karina Gil, Intertek

Michael Johnscher, ISP

Litchi Xie, Lubrizol Additive (Zhuhai) Co., Ltd.

Victoria Fein, Lubrizol

Jason Bowden, OH Technologies Inc

Greg Miiller, Savant Group

Maggie Smerdon, Savant Labs

Sean Alston, SGS North America

Jared Cavaliere, SwRI

Becky Grinfield, SwRI

Yong-Li McFarland\*, SwRI

John Loop, TMC

Amy Ross, Valvoline

\*Chair



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# New EOGT WK86363, ILS# 1854

- Ford request for a new Engine Oil Gelation Test (EOGT): request to add a new test filterability test to better screen oils for field issues
- Current status
  - Method: 2 drafts (large volume (600g) and small volume (200 g)) uploaded on ASTM Collaboration Area, email Jared with procedure questions
  - Oils: **11** potential reference oils offered; 17 oils received at TMC
  - Screening Tests, Pilot Study, and ILS: Screening Test completed, start additional Proposal testing
  - Timing: Pilot Study tests run by February, ILS tests to be run by June, and final method ballot in July or August 2024



# Testing Status – updated Nov 16 [EOGT report form Rev 6](#)

Test Phase	Description	Status	Comments
1. Screening Tests	<p>-4 tests; 2 oils: low performance oil in duplicate, high performance oil in duplicate; 6 labs</p> <p>-Run LV method</p> <p>-Intent: check labs can run test as set up</p>	All 6 labs submitted data	<p>Run SOT &amp; 14 day EOT only.</p> <p><b>-REVIEW data</b></p>
2. Pilot Study	<p>-? Tests; ? Oils; 4 labs</p> <p>-Run SV and LV methods, all intervals</p> <p>-Intent: determine samples, method, and intervals for procedure and ILS</p>	<p>Waiting to determine samples:</p> <p>-Plan to include either 6 or 10 oils including 2 performance oils, and then each of the other oil participants select 1 or 2 “borderline” oil for total of 6 or 10 oils to run. “Borderline” is something that would fall in-between the 2 performance oils (less Ca than Oil F?)</p> <p>-Pros/cons: 6 oils – shorter to run, but may not find a borderline reference and may need another Pilot run; 10 oils-longer to run, but more likely to find a borderline reference</p>	<b>Group to decide as we move forward</b>
3. Interlaboratory Study (ILS)	<p>-? Tests; ? Oils; 6 labs</p> <p>-Run _ method</p> <p>-Intent: generate repeatability and reproducibility data on samples, determine at least 2 reference oils</p>	Waiting to determine samples	

# Screening Data Plots

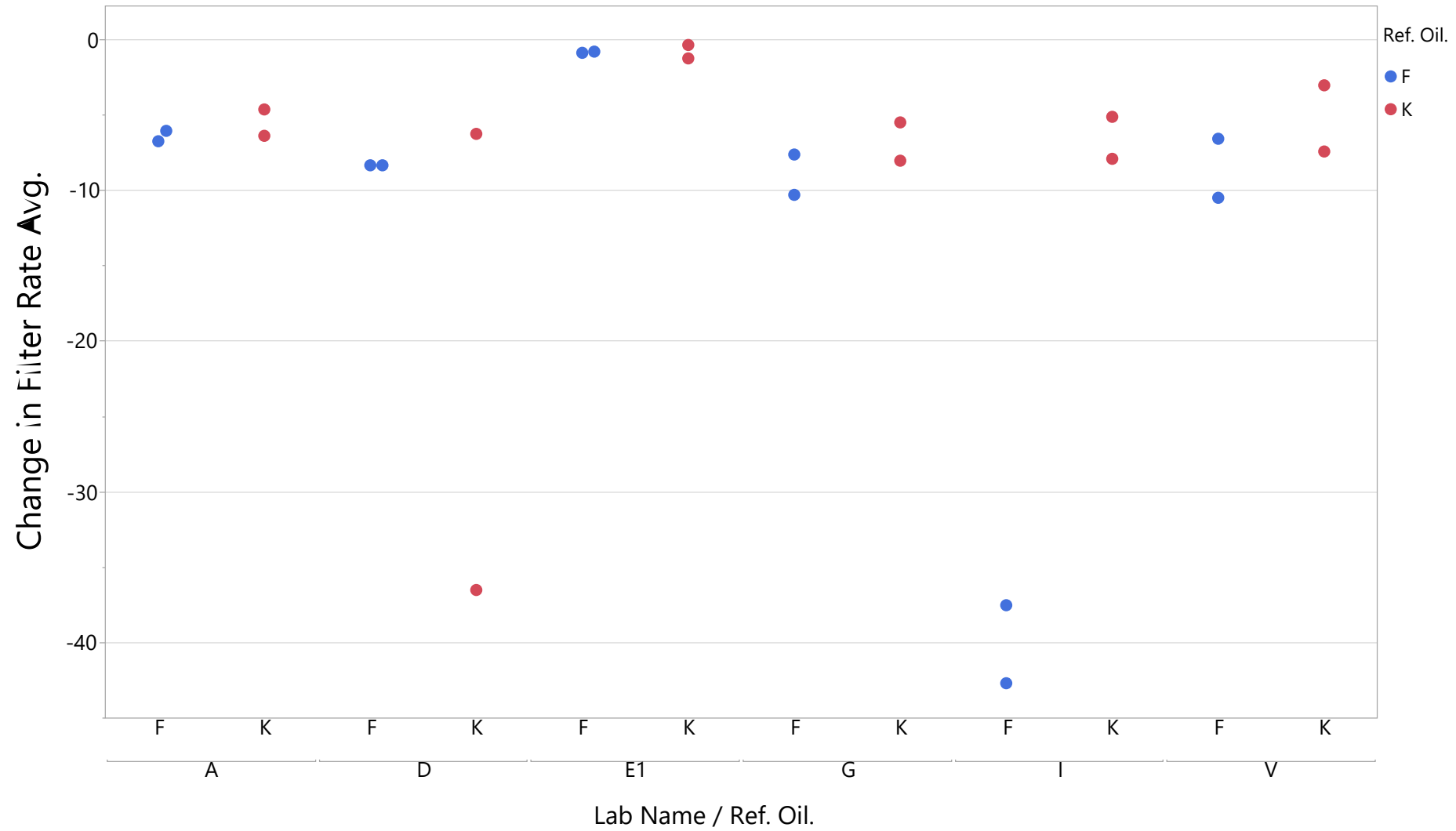
Updated Nov 17, 2023



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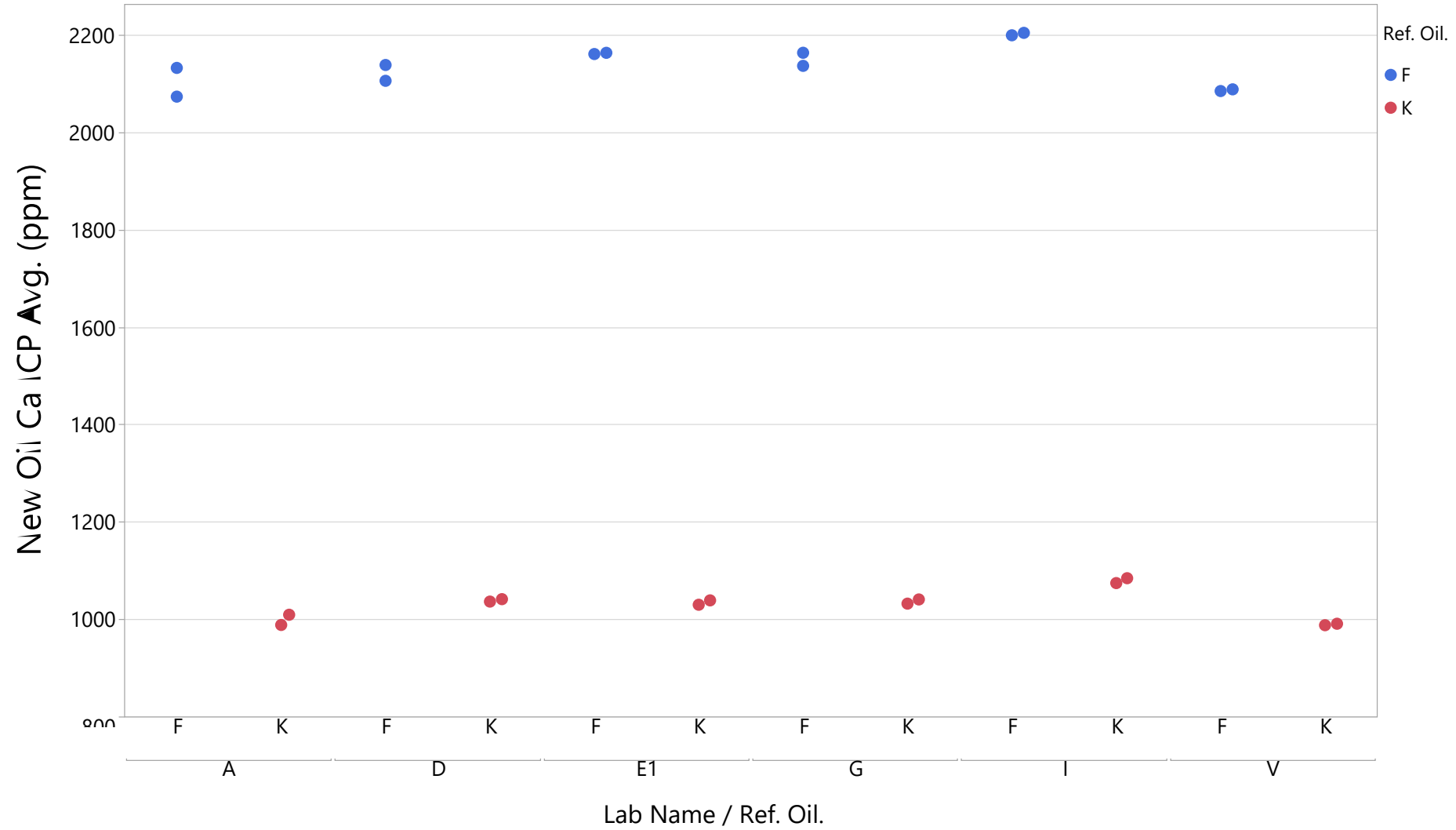
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# Change in Filter Rate Avg. by Lab and Oil



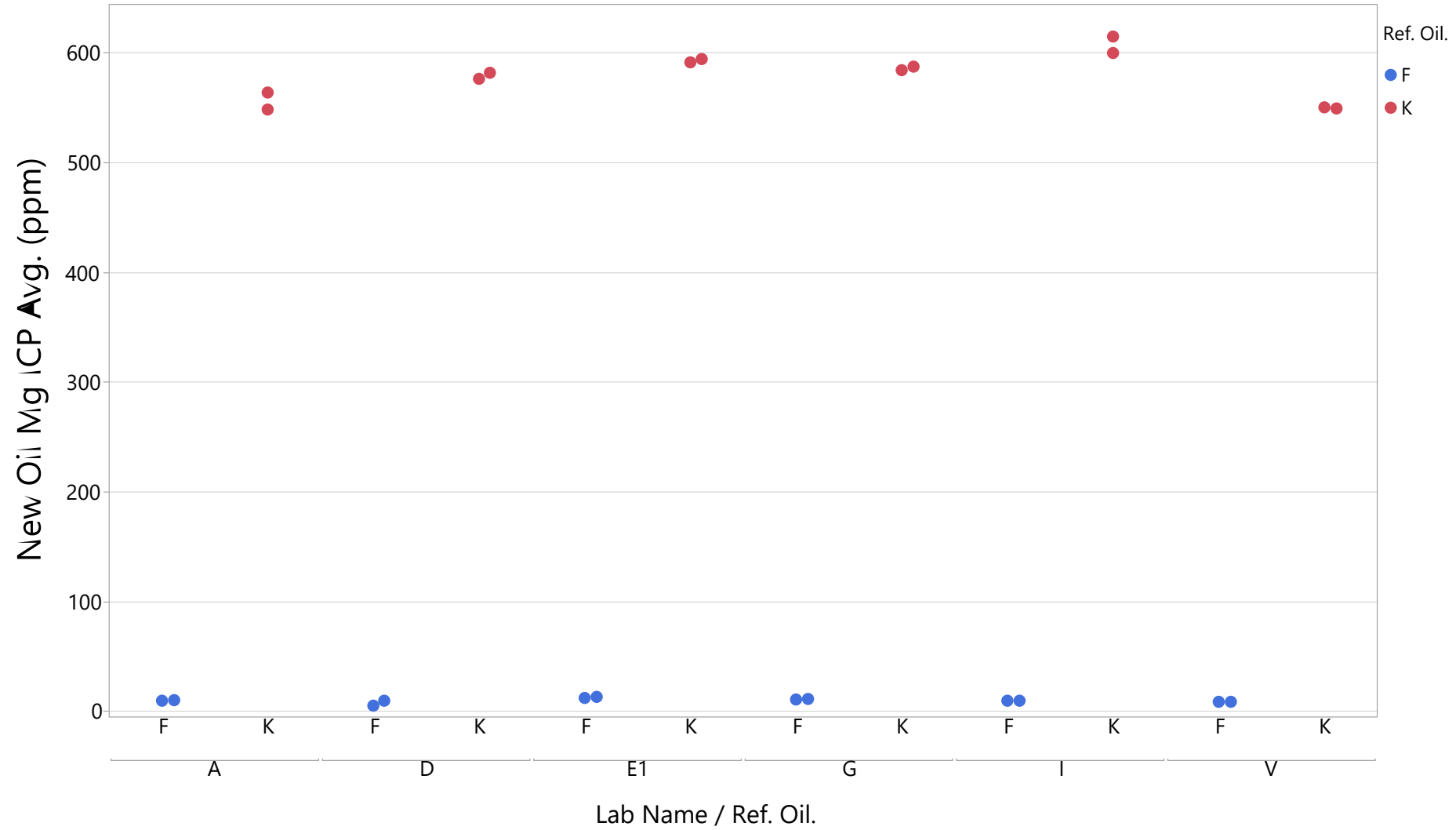
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# New Oil Ca Avg. by Lab and Oil



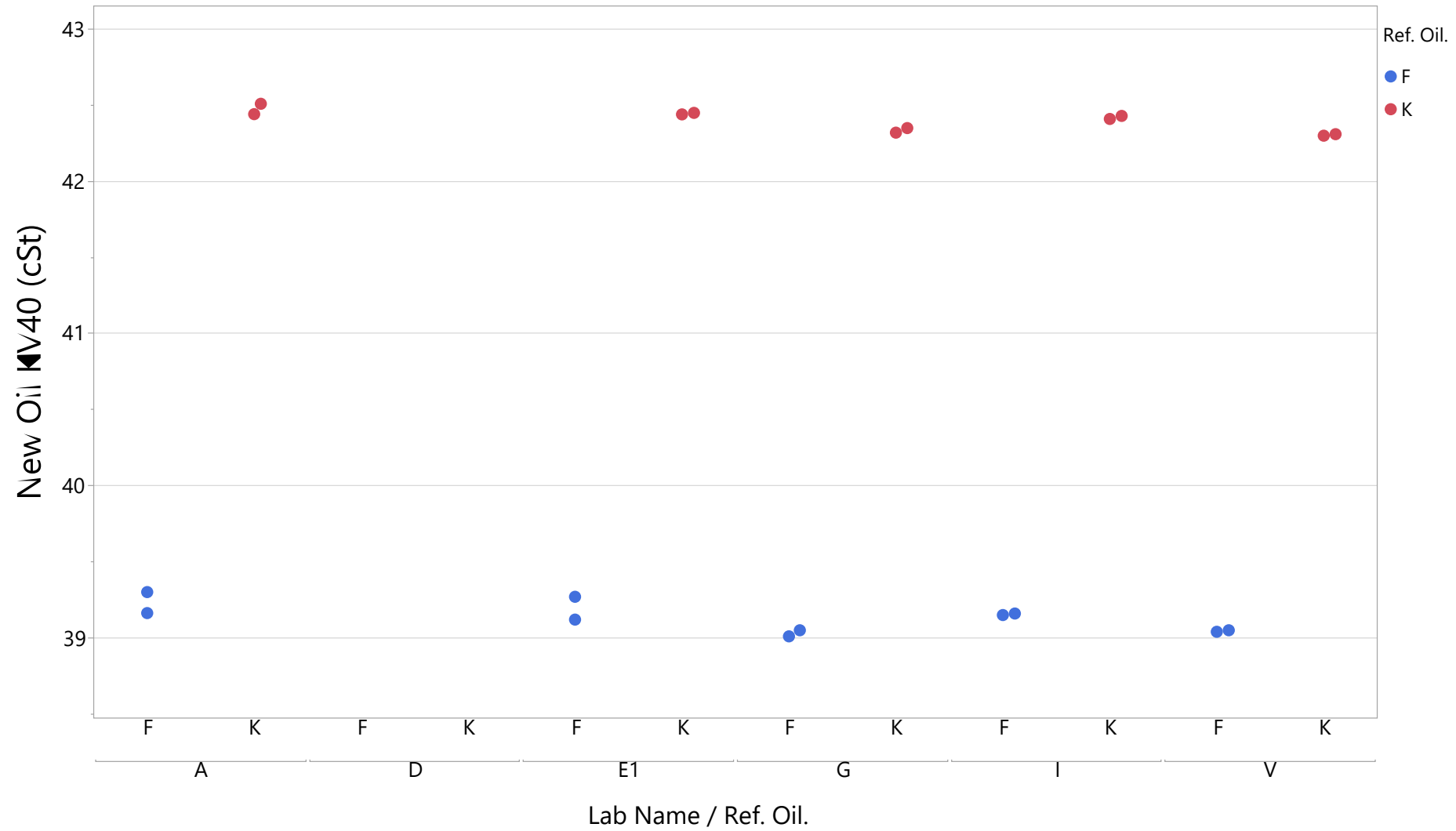
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# New Oil Mg Avg. by Lab and Oil



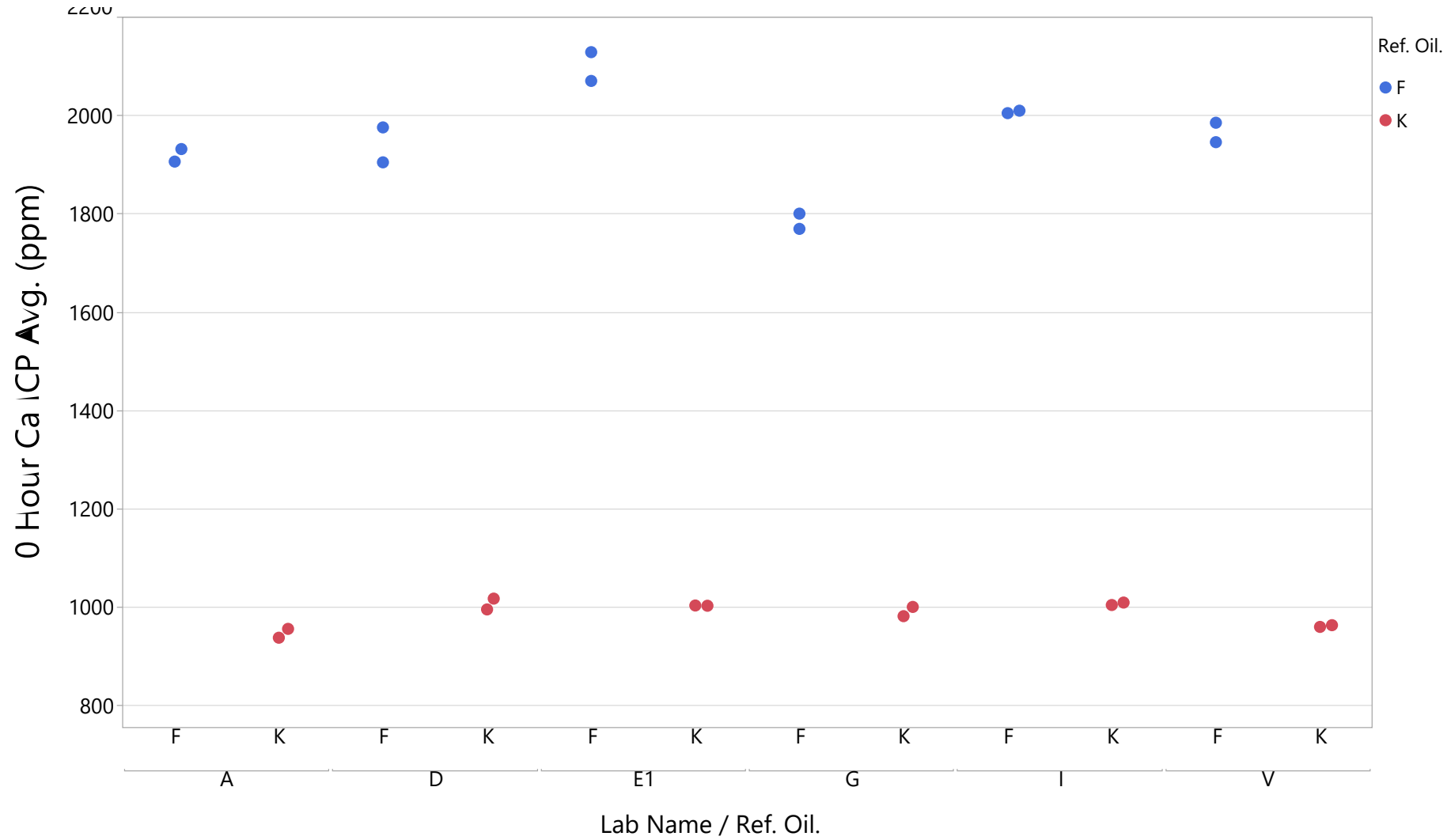
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# New Oil KV40 by Lab and Oil



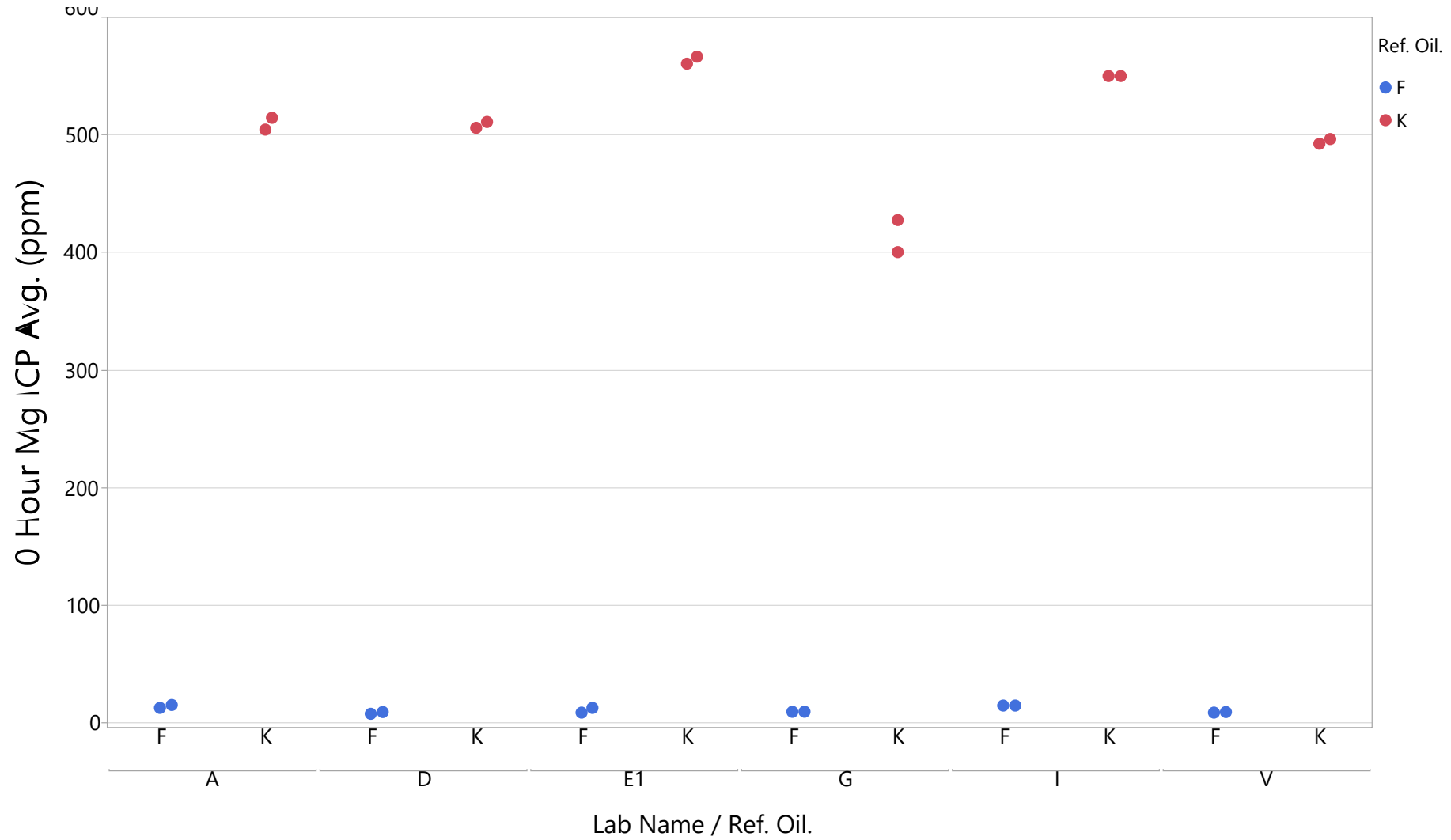
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# 0 Hour Ca Avg. by Lab and Oil



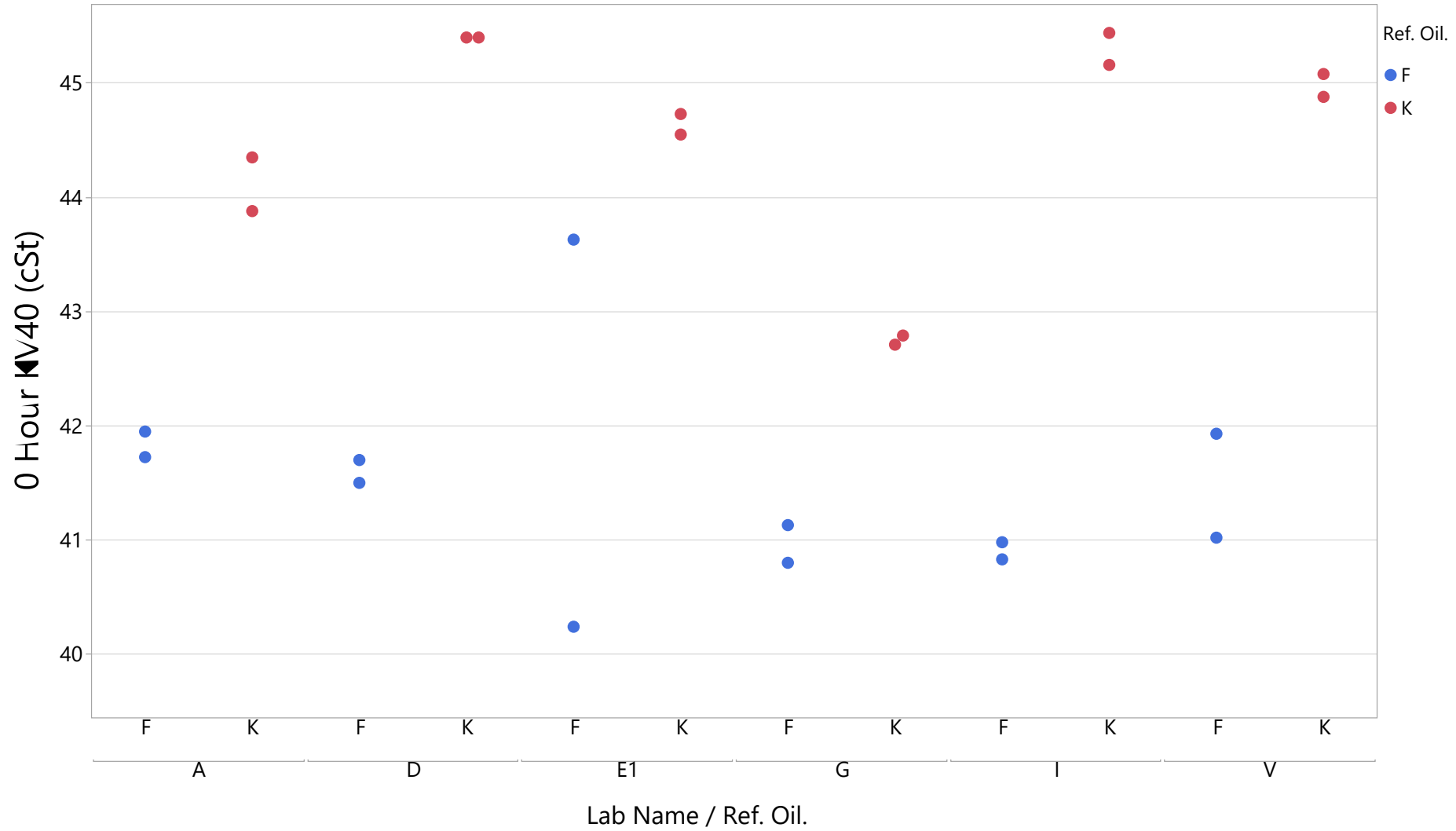
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# 0 Hour Mg Avg. by Lab and Oil



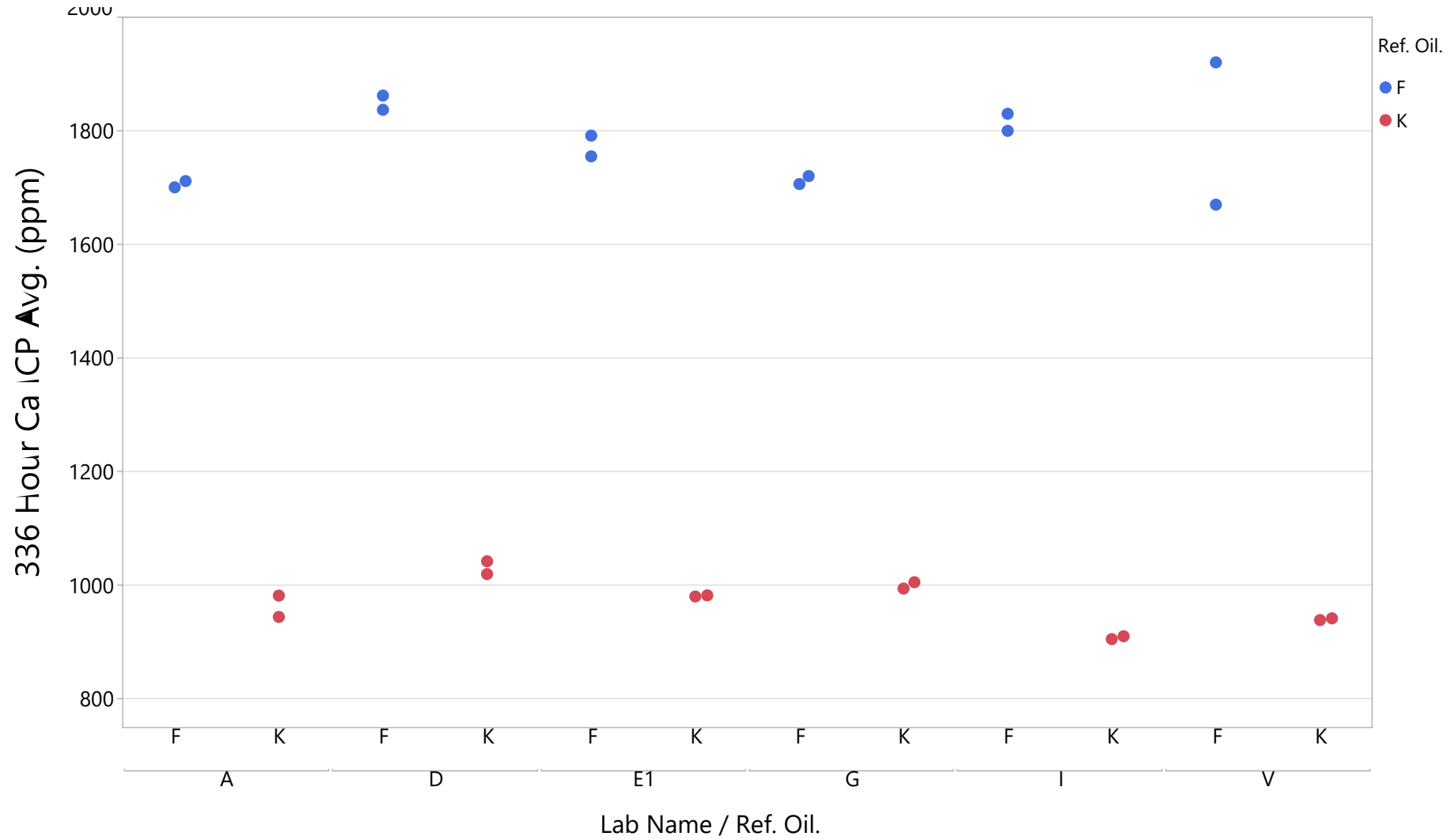
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# 0 Hour KV40 by Lab and Oil



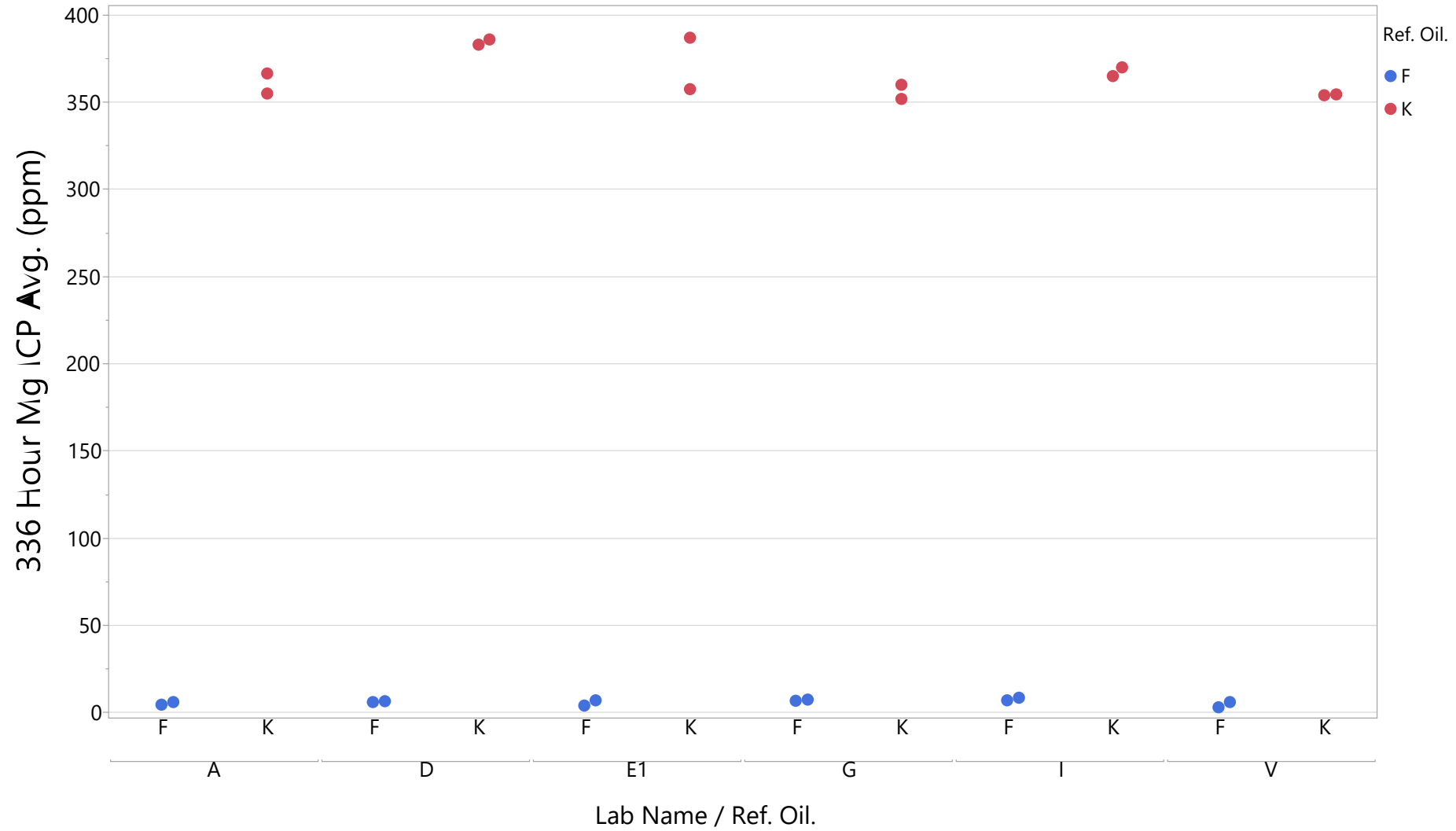
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# 336 Hour Ca Avg. by Lab and Oil



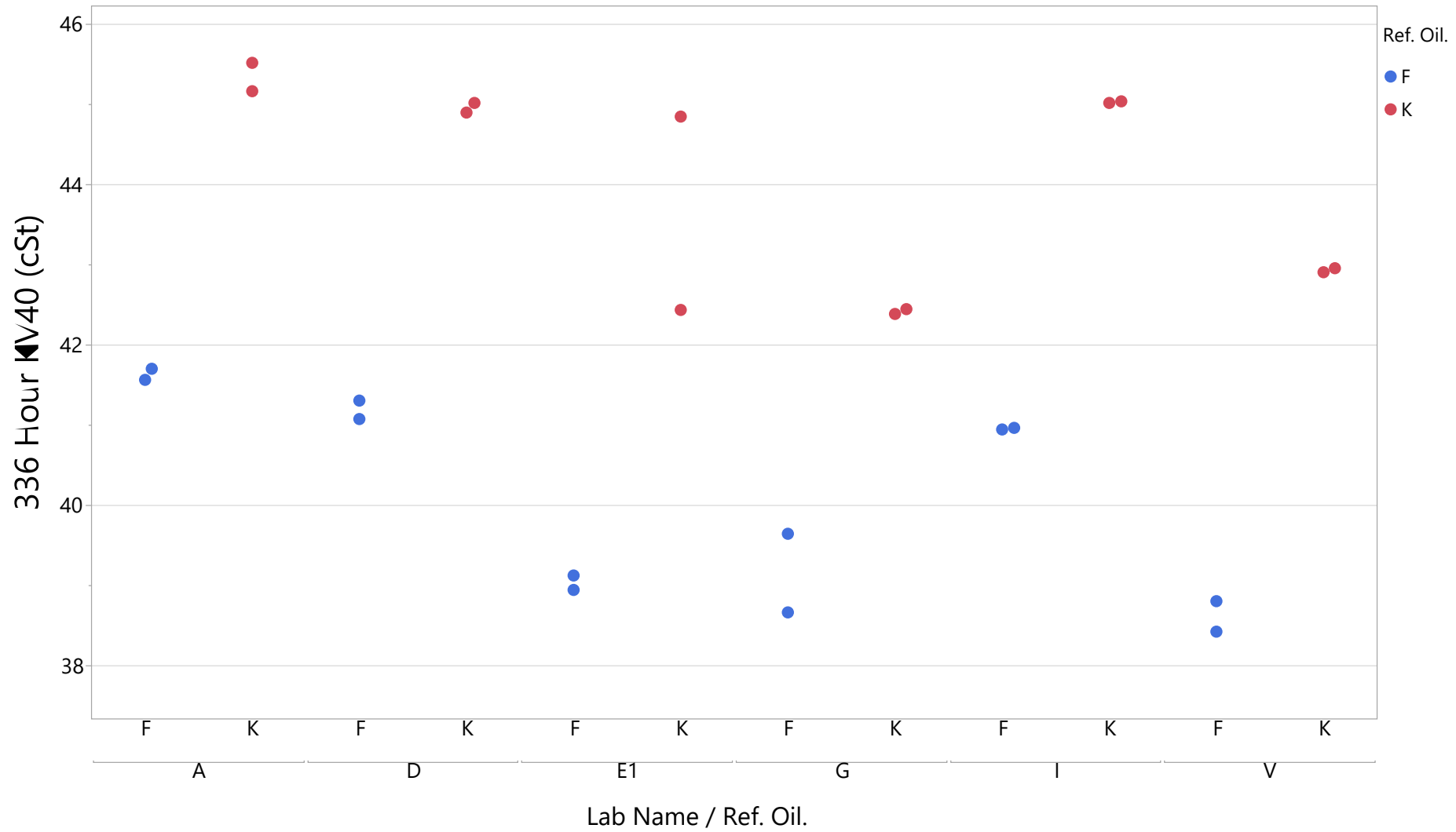
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# 336 Hour Mg Avg. by Lab and Oil



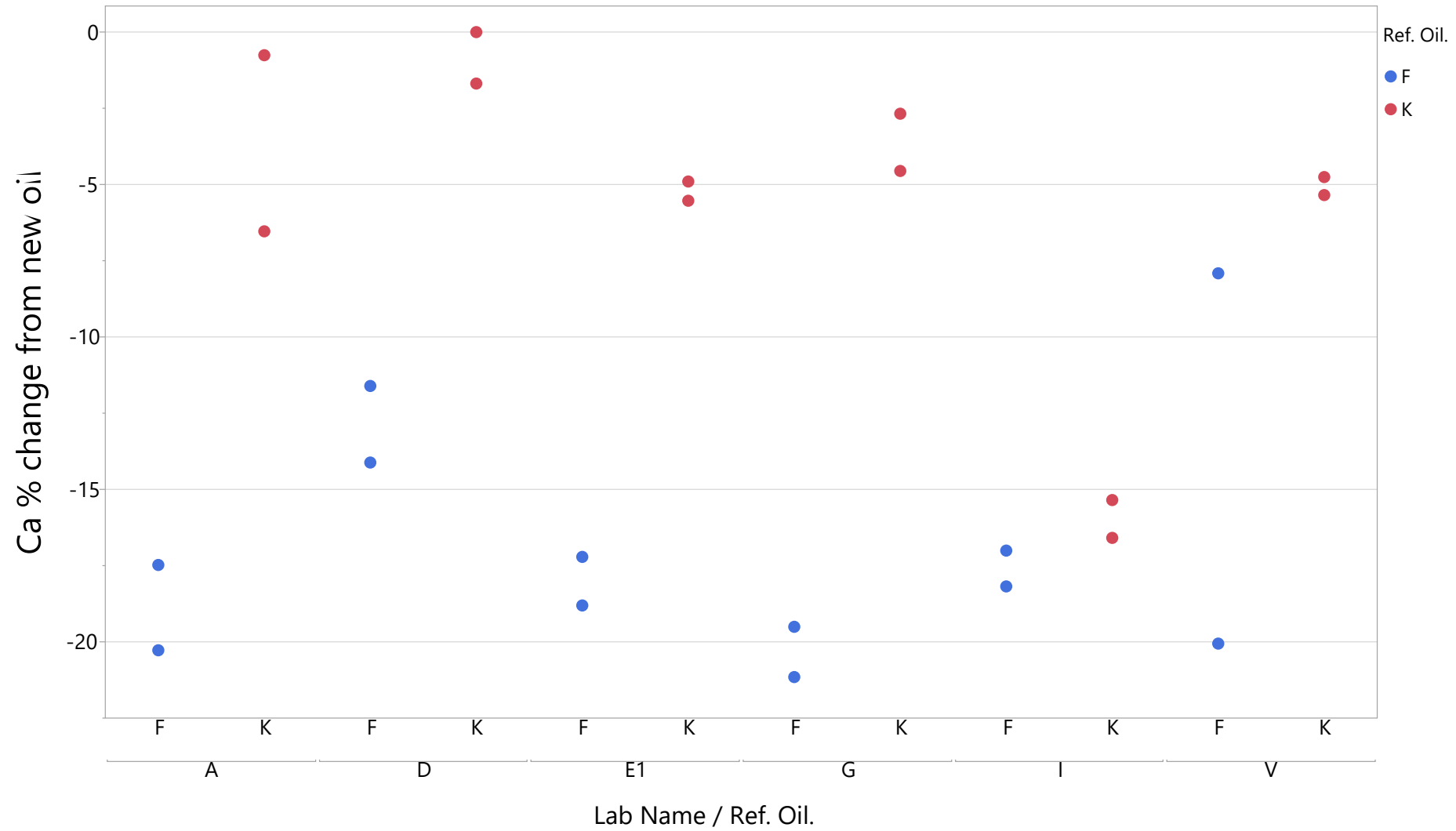
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# 336 Hour KV40 by Lab and Oil



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# Ca Percent Change from New Oil



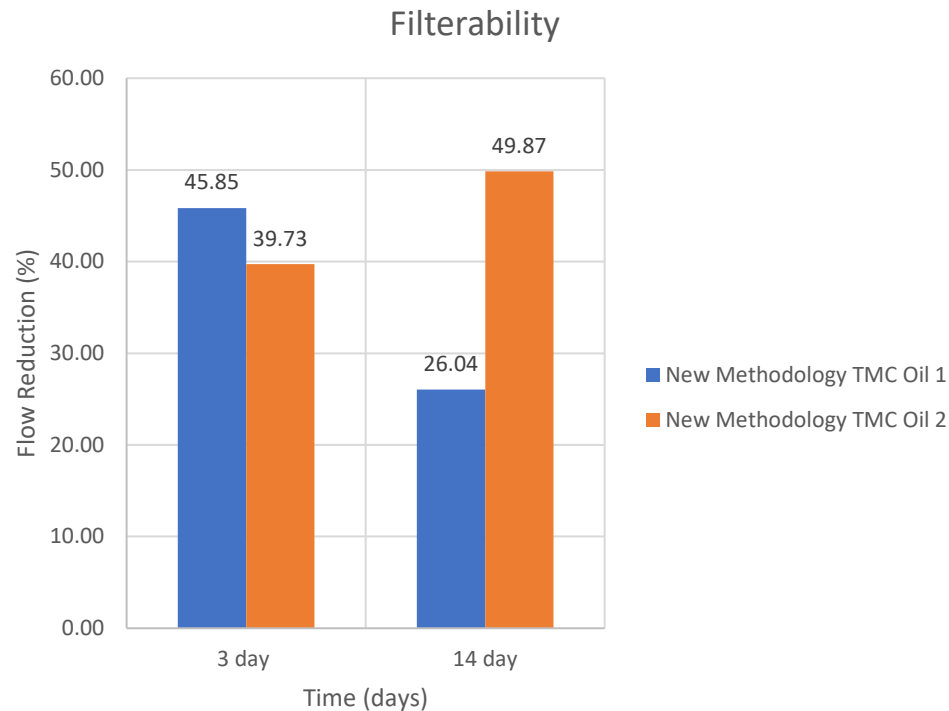
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# Proposal Experiments – November 20

Proposal Experiments	Lab(s)	Status	Next Step
1. Sediment-only Filterability	Afton	Completed	GROUP to make comments on if this is helpful
2. Include 0 Hr filterability	ISP	Completed	No need to include 0 Hr filterability currently
3. Run EOGT on Ref Oil 79 and 77-3	ISP and Afton	ISP: Complete Dec 8 Afton: Complete Nov 20	
4. Run EOGT with 8% water	Savant and Intertek	Savant: Completed Intertek: Completed	No need to use 8% water
5. Characterize gel	Savant and Valvoline	Savant: Completed IR Valvoline: tried microscopy, will try IR on 4% water as well	Unsure how results will be helpful in test currently
6. Centrifuge sample before filterability	SwRI	Complete Nov 17	
7. No homogenizing before filterability	Intertek	Completed	GROUP to make comments on if this is helpful
8. Run EOGT with 4 hr CO2 bubbling and heating (run with 2 flow tubes)	SwRI	Complete Nov 22	

# I. Sediment-only filterability results

Afton data



Include run times for experiments- next slide

Methodology Procedure of Filterability with Decanting

- Mix ( $600 \pm 1.0$ ) g of new oil and ( $24 \pm 0.5$ ) g of deionized water in the blender for ( $30 \pm 1$ ) s at 18 000 rpm  $\pm 10$  %. Cover the top of the container loosely to prevent oil spattering.
  - Actual Amounts
  - Container 1
    - Oil: 600.1 g
    - Water: 24.0 g
  - Container 2
    - Oil: 600.2 g
    - Water: 24.1 g
- Transfer the blended sample to a glass vessel and place in an oil bath or mantle with condenser, gas tube 12-24 mm from bottom, and thermo-probe. Sample oil level should be below the bath oil or top of the heating mantle.
- When sample temperature has reached ( $50.0 \pm 1$ ) °C, start the CO<sub>2</sub>-flow ( $18 \pm 0.5$ ) L/h and maintain sample temperature at ( $50.0 \pm 1$ ) °C for ( $60 \pm 1$ ) minutes.
- Remove sample from heat after 60mins. Homogenize sample and transfer equal aliquots to 2 inert lined containers. A non-metallic spatula was used to help mix any precipitate before transferring sample as equally as possible to the multiple containers.
- Store the containers in dark at room temperature (20 to 24) °C for 72hrs and 336hrs.
- At the desired timepoint, sample is decanted to removed most of the top layer (75 to 85% of top layer typically removed)
  - Container 1 (3 day) decanted 256.1 g of fluid (82.1% of container)
  - Container 1 (14 day) decanted 261.6 g of fluid (83.9% of container)
  - Container 2 (3 day) decanted 257.9 g of fluid (82.7% of container)
  - Container 2 (14 day) decanted 258.4 g of fluid (82.9% of container)
- Determine Filterability at the end of 72hrs and 336hrs ( $\pm 2$  hours).

# I. Sediment-only filterability results cont.

Afton data

Date			10/9/2023		
		Fresh	3 day	Fresh	14 day
Fluids	Volume (mL)	Time (sec)	Time (sec)	Time (sec)	Time (sec)
RIC23-09047	0	0	0		
	5	6.05	11.4	6.54	13.54
	10	6.09	13.17	6.67	10.13
	15	6.17	13.52	6.33	9.16
	20	6.05	13.01	6.53	8.4
	25	6.27	11.58	6.59	8.91
Filterability		Fresh	3 day	Fresh	14 day
(Flow Reduction)	Change		45.85		26.04
	Average				35.95
Flow Rate					
	0	0.00	0.00	0.00	0.00
	5	0.83	0.44	0.76	0.37
	10	0.82	0.38	0.75	0.49
	15	0.81	0.37	0.79	0.55
	20	0.83	0.38	0.77	0.60
	25	0.80	0.43	0.76	0.56
		Wt of oil decanted	256.14		261.62
		% of oil decanted	82.10		83.85

Date			10/9/2023		
		Fresh	3 day	Fresh	14 day
Fluids	Volume (mL)	Time (sec)	Time (sec)	Time (sec)	Time (sec)
RIC23-09049	0	0	0		
	5	5.49	12.27	5.18	15.14
	10	5.71	10.65	5.46	11.96
	15	5.73	10.84	5.3	12.08
	20	5.8	9.34	5.52	10.81
	25	5.72	9.49	5.61	11.19
Filterability		Fresh	3 day	Fresh	14 day
(Flow Reduction)	Change		39.73		49.87
	Average				
Flow Rate					
	0	0.00	0.00	0.00	0.00
	5	0.91	0.41	0.97	0.33
	10	0.88	0.47	0.92	0.42
	15	0.87	0.46	0.94	0.41
	20	0.86	0.54	0.91	0.46
	25	0.87	0.53	0.89	0.45
		Wt of oil decanted	257.9		258.39
		% of oil decanted	82.66		82.82



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# 2.0 Hr Filterability

ISP data

Sample	Change in Flow Rate Run 1, 0 Hr	Change in Flow Rate Run 2, 0 Hr	Change in Flow Rate Average, 0 Hr	Change in Flow Rate Average, 336 hr
182320	-8.80	-7.09	-7.95	-42.69
182319	10.89	6.86	8.88	-5.12

-No need to include 0 hr filterability in test currently based on these results.



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# 4. Run EOGT with 8% water results

- Savant Labs data

Samples were treated with 18L/hr of CO2 for one hour at 50C.							
Customer ID	Results after 336hrs hold			Fresh Viscosity at 25C	Viscosity 25C 2Wk Retain Top Layer	Viscosity 25C 2Wk Retain Bottom Layer	Comments
	Run 1 %	Run 2 %	Change In Flow Rate - Average %	cP	cP	cP	
CMIR 183758	-17.33	-17.45	-17.39	56.9	69.5	80.7	Sample mixed together well and flowed readily
CMIR 183760	-15.55	-14.72	-15.13	68.8	78.6	87.0	Sample did not mix together easily and was more difficult in testing.



## 4. Run EOGT with 8% water results cont.

- Intertek Data – full data also attached

Sample	Change in Flow Rate, Run 1	Change in Flow Rate, Run 2	Change in Flow Rate Average, 336 Hr	KV40, cSt, New oil	KV40, cSt, 336 Hr	Ca ICP average, ppm, New Oil	Ca ICP average, ppm, 336 Hr	Mg ICP average, ppm, New Oil	Mg ICP average, ppm, 336 Hr
183756	-13.23	-14.56	-13.9	42.07	42.25	1024	934.5	583.5	258.5
183757	-12.7	-12.69	-12.7	42.09	42.29	1032	938.5	583	260

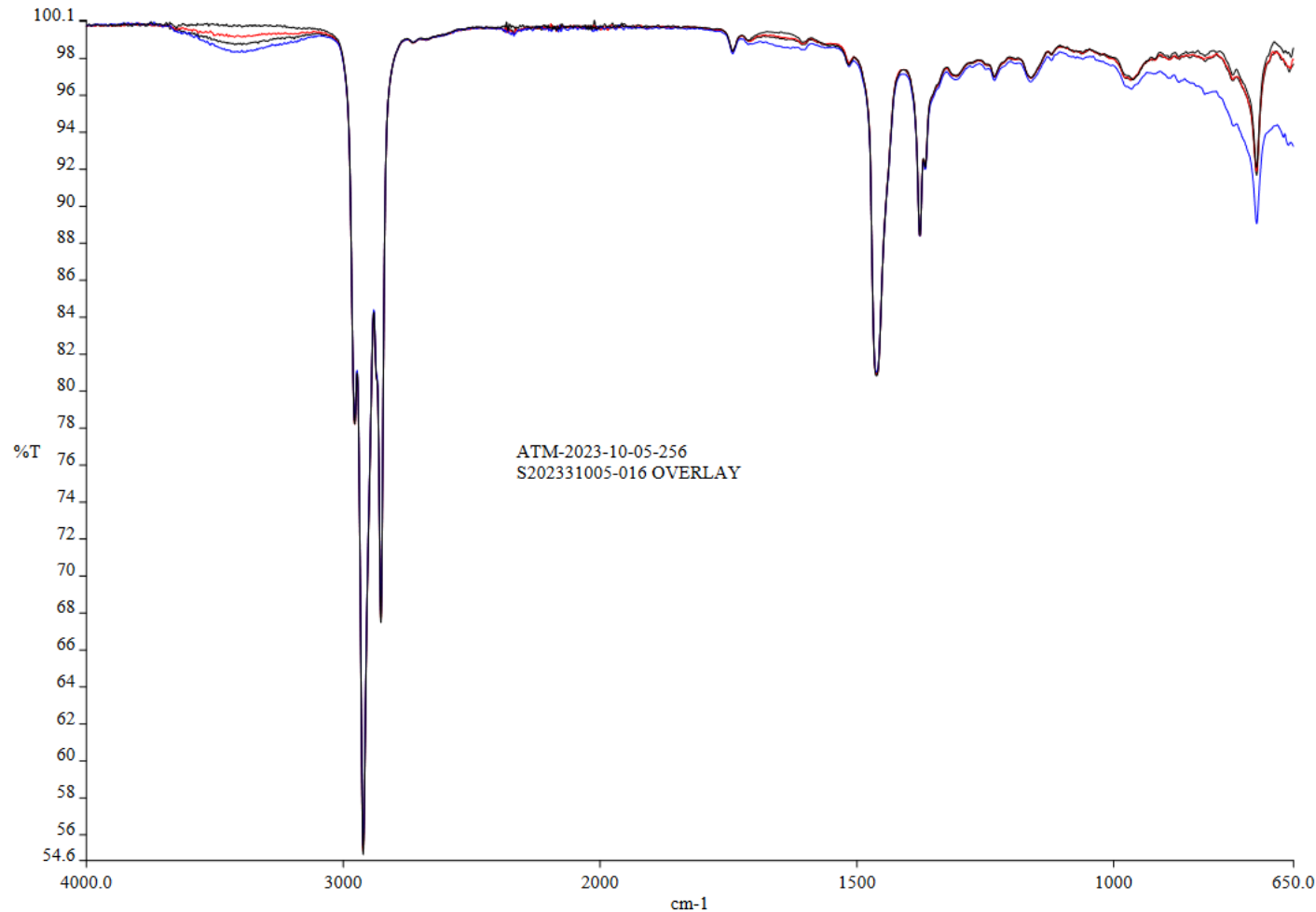
-No need to increase water % in EOGT, results don't show difference in 2 oils performance.



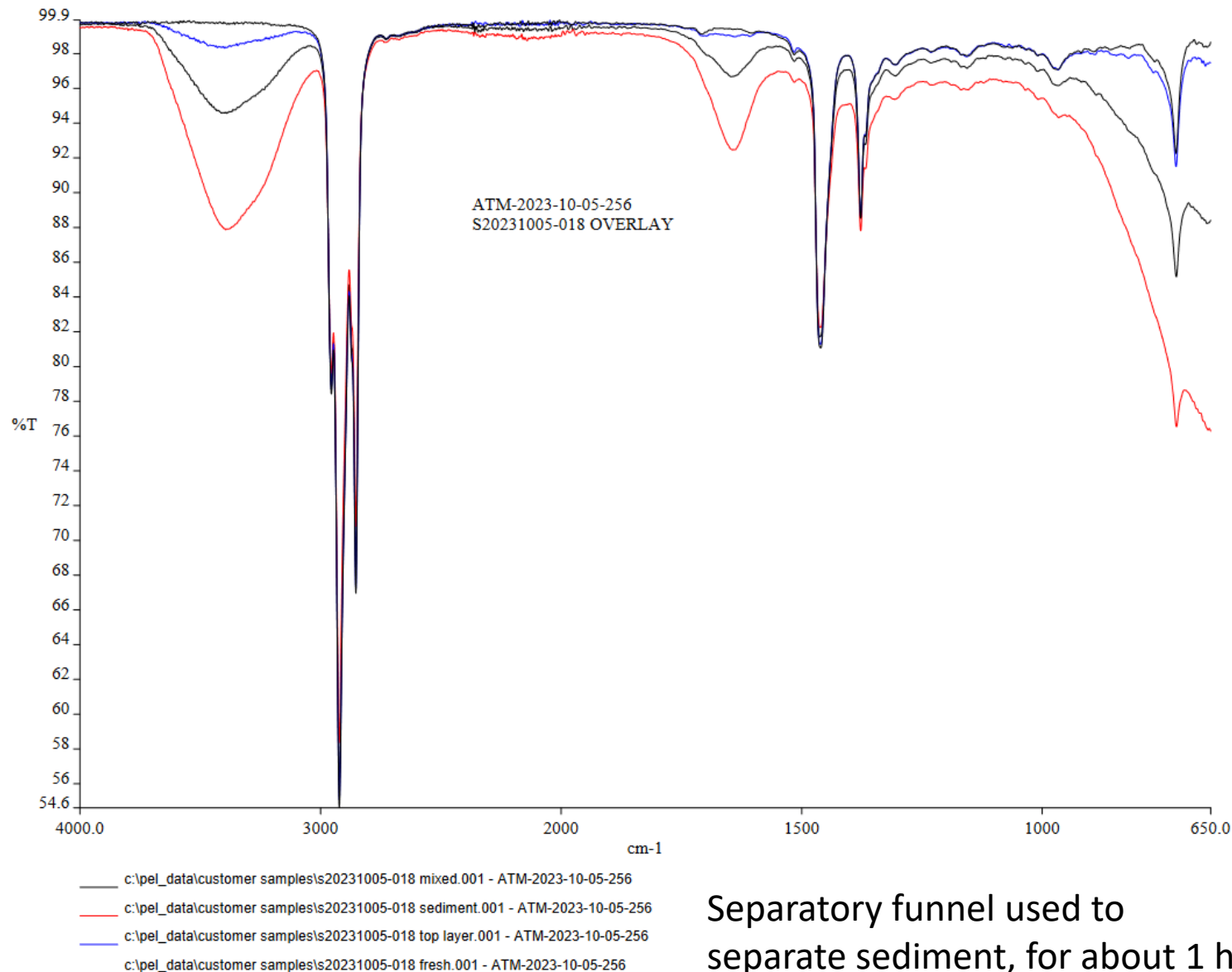
# 4 / 5 Run EOGT with 8% water cont.

## Savant Labs data

- FTIR Analysis for EOGT Samples
- Both samples S20231005-016 (CMIR 183758) and sample S20231005-018 (CMIR 183760) were analyzed using the FTIR. The fresh, bottom sediment layers, top oil layer, and the mixed samples all were scanned on the FTIR. The sediment layer and the top layer of the treated oils were separated using a separatory funnel and placed into separate containers. The mixed samples were shaken for 30 seconds immediately prior to being scanned on the FTIR.
- Sample S20231005-016 (CMIR 183758) seemed to have the water incorporated better into the emulsion of the sample. Sample S20231005-018 (CMIR 183760) had the water fall out of the sample way more prominently. The O-H stretch, and the O-H-H bend are visibly apparent in sample S20231005-018 (CMIR 183760) showing that the water is separating way more in the sample than in sample S20231005-016 (CMIR 183758). In sample S20231005-018 (CMIR 183760) the water peak is the most intense at the sediment layer. This is as expected due to the greater density of the water, whereas the water peak is less intense in the top layer of the oil.



# 4 / 5 Run EOGT with 8% water cont.



## Savant Labs data

- FTIR Analysis for EOGT Samples
- Both samples S20231005-016 (CMIR 183758) and sample S20231005-018 (CMIR 183760) were analyzed using the FTIR. The fresh, bottom sediment layers, top oil layer, and the mixed samples all were scanned on the FTIR. The sediment layer and the top layer of the treated oils were separated using a separatory funnel and placed into separate containers. The mixed samples were shaken for 30 seconds immediately prior to being scanned on the FTIR.
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Separatory funnel used to separate sediment, for about 1 hr

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# 7. No homogenization before filterability

- Intertek data – full data also attached

Sample	Change in Flow Rate, Run 1	Change in Flow Rate, Run 2	Change in Flow Rate Average, 336 Hr	KV40, cSt, New oil	KV40, cSt, 336 Hr	Ca ICP average, ppm, New Oil	Ca ICP average, ppm, 336 Hr	Mg ICP average, ppm, New Oil	Mg ICP average, ppm, 336 Hr
183754	6.19	-6.57	-0.19	38.83	38.71	2117.5	1719	11.53	5.46
183755	5.2	4.63	4.92	38.85	38.69	2106.5	1703	10.65	7.67



# ISP Filterability Setup – Nov 20



Requested a video of heating and bubbling CO<sub>2</sub> steps, and filterability to ISP  
-Will work on video with next tests to end Dec 6

# Draft Timeline – updated Nov 17

Task	Date									
	May-Jun 2023	Jul-Aug 2023	Sept-Oct 2023	Nov-Dec 2023	Jan-Feb 2024	Mar-Apr 2024	May-Jun 2024	Jul-Aug 2024	Sept-Oct 2024	
Develop test procedure and report form (ILS)	█									
Collect and prepare donated oil samples (18 oils)	█	█								
Screening samples shipped to labs (6 labs)		█								
Screening labs run 4 tests			█	█						
Data analysis for Screening and Proposal tests			█	█	█					
Pilot Study samples shipped to labs (4 labs)					█	█				
Pilot Study labs run ? tests						█	█			
Data analysis for Pilot Study							█			
ILS samples shipped to ILS labs (6 labs)							█			
ILS Labs run tests							█	█		
Data analysis for ILS, generate Research Report (RR) & Precision									█	
Ballot test procedure and RR										█
<b>Generate pass/fail limits [Outside this Surveillance Panel]</b>										



# Next Steps

- Labs to run additional Proposal Experiments and group to review results
- Request TMC to send 2 samples each of both Performance oils to ? Labs for additional testing; ask labs to let John Loop know how much sample remaining by Nov 29. John to check on remaining fluids at TMC
- If change in flow rate not showing difference between the 2 performance oils, will group consider IR or other data as criteria for test?
- On Hold-Determine Pilot Study samples (# and which samples, and # repeats), and method (LV, SV, all intervals)
- On Hold- Send out Pilot Study samples to Pilot Study labs
- On Hold- Labs run Pilot Study





# Thank you for your support!

Participants		
Method Development	Oil Donations	Testing Labs
Afton ExxonMobil Ford Infineum Intertek ISP Lubrizol Oronite Savant SwRI TMC	Afton Ford Infineum Lubrizol OH Technologies (donate filters only) Oronite Subaru TMC (collection, shipping only) Toyota	Afton (Screening, Pilot, and ILS) Intertek (Screening, Pilot, and ILS) ISP (Screening, Pilot, and ILS) Savant (Screening and ILS) SwRI (Screening, Pilot, and ILS) TMC (monitoring system only) Valvoline (Screening and ILS)

