

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

EOFT and EOWTT Surveillance Panel Meeting

June 10, 2024

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

Quanchang Li, ExxonMobil

Michael Deegan, Ford

Melissa Chu, 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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# EOFT D6795 and EOWT D6794 New Item

- ASTM D6795 is up for 5-year review in 2024, ASTM D6794 is up for review in 2025
- Looking for participants for suggested edits and improvements- Maggie, Rachelle, Victoria, John L volunteered
- Group will meet in late June/early July for more discussion



# New EOGT WK86363, ILS# 1854

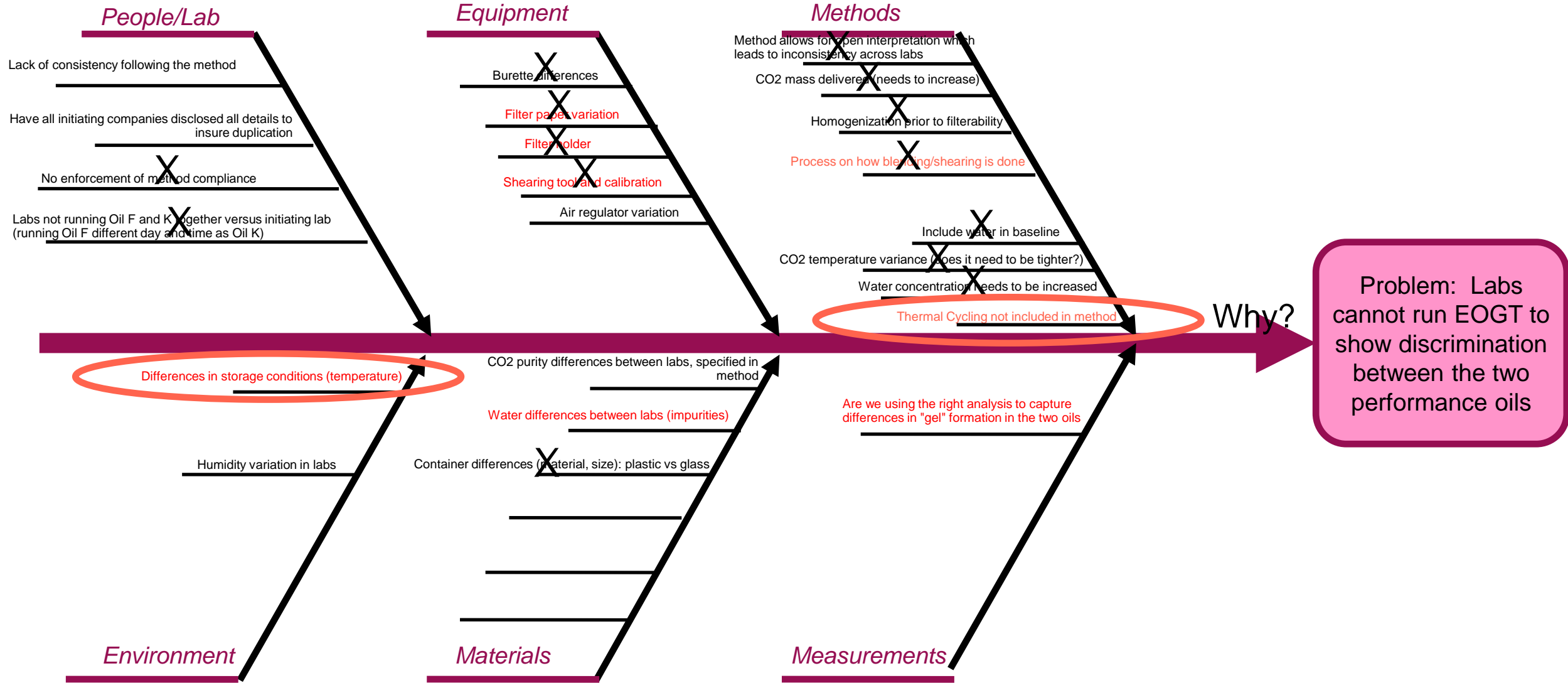
- Ford request for a new Engine Oil Gelation Test (EOGT): request to add a new test filterability test to better screen oils for field issues
- Current status
  - Method: 2 drafts (large volume (600g) and small volume (200 g)) uploaded on ASTM Collaboration Area, email Jared with procedure questions
  - Oils: 11 potential reference oils offered; 17 oils received at TMC
  - Screening Tests and ILS: Screening Test and additional tests ongoing
  - Timing: ILS tests to be run by October, and final method ballot in January or February 2025

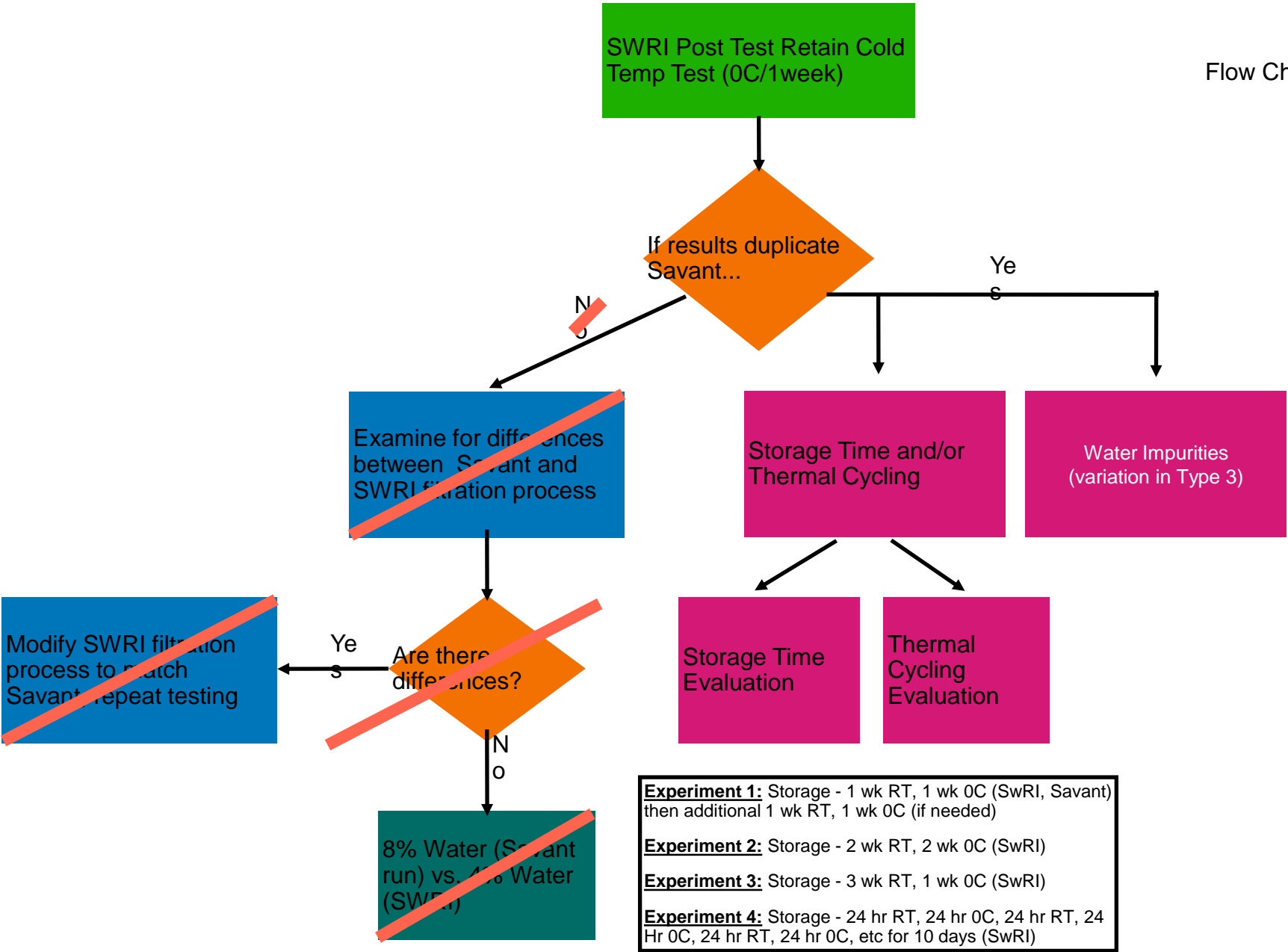


# Updates 5-22-24

- Root Cause Subgroup update
- ISP rerun of performance oils estimated to complete in August
- TMC update on labs' response for burette size opening (missing 1 lab response)
- TMC update on labs' response for storage temperature (after blending and bubbling CO<sub>2</sub>) (missing 4 labs response)
- TMC update on labs' response for part number, vendor, and photo of filter holder to TMC; OHT also offered info on part (missing 4 labs response)
- TMC update on labs' response if homogenization step description is accurate and add details to Data Excel (no changes from any lab)
- Eugene (Afton) to look into FTIR peak in ISP data around 2300 wavenumber to see what peak may be. This peak is related to CO<sub>2</sub>, and Afton does a correction for CO<sub>2</sub> so not seen in Afton IR graphs. Not sure if ISP does a CO<sub>2</sub> correction in their FTIR.







# Cold Temperature Soak Tests – Savant Tests

- Procedure
- Savant Tests: Retain EOT samples CMIR 183758 and 183760 were stored for 2 weeks in -2.5C freezer. They were removed and stabilize to room temperature (~72.8F) for 2 hours. Samples were hand shaken and stirred with rubber stopper until appeared homogeneous and then filterability run.
- Note: These were samples with 8% water (EOGT method is for 4% water) and original test date was October 25, 2023. After testing, samples were stored in sample check room.

- Results

Savant Cold Temp Soak Test Results		
CFA = Change in Flow Rate	CMIR 183758 (Oil F)	CMIR 183760 (Oil K)
CFA Run 1, %	-71.51	-14.14
CFA Run 2, %	-76.56	-12.21
CFA Average, %	<b>-74.04</b>	<b>-13.18</b>
Original EOT CFA Average, %	-17.39	-15.13



# Cold Temperature Soak Tests – SwRI Tests

- Procedure
- SwRI Cold Soak Tests #1: Newly prepared samples CMIR 183766 and 183768 were stored for 14 days in 0C freezer, 9C cabinet, or 20C cabinet. They were removed and stabilize to room temperature (~22C) for 2 hours. Samples were inverted and mixed with rod until appearing homogeneous and then testing filterability.
  - Notes: These CMIR were stored in a non-climate-controlled space for 6 months. Prepared by EOGT Preparation (Section 9) and 2 CO2 tubes
- SwRI Cold Soak Tests #2: Retains from Cold Soak Tests #1 and previous retain EOT were stored for 7 days in 0C freezer. They were removed and stabilize to room temperature (~22C) for 2 hours. Samples were inverted and mixed with rod until appearing homogeneous and then testing filterability.
  - Notes: Retains from Cold Soak Tests #1 were stored in 20C cabinet for additional 9 and 7 days before storing at 0C.

\* Used for additional testing

Notes: consider how labs are homogenizing samples and differences

## Results

### SwRI Cold Temp Soak Test #1 Results

Condition	CFA = Change in Flow Rate	CMIR 183766 (Oil F)	CMIR 183768 (Oil K)
14 days 0C	CFA Average, %	-10.4	-8.55
14 days 9C*	CFA Average, %	-8.86	-8.22
14 days 20C*	CFA Average, %	-10.23	-6.93

### SwRI Cold Temp Soak Test #2 Results

Condition	CFA = Change in Flow Rate	CMIR 183766 (Oil F)	CMIR 183768 (Oil K)
14 days 9C + 9 days 20C + 7 days 0C	CFA Average, %	-19.44	-14.82
21 days 20C + 7 days 0C	CFA Average, %	-25.38	-17.17
6 months +7 days 0C	CFA Average, %	-36.98	-9.58

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# Cold Temperature Soak Tests – SwRI Tests

- Procedure
- SwRI Cold Soak Tests #3: Newly prepared samples CMIR 188836 and 188837 were stored for 7 days in 20C cabinet and then 7 days in 0C freezer. After these conditions, they were removed and stabilize to room temperature (~22C) for 2 hours. Samples were inverted and mixed with rod until appearing homogeneous and then testing filterability. If needed, an additional 7 days at 20C and 7 days at 0C storage may be repeated before an additional filterability run.
  - Notes: These CMIR were received from TMC May 30, 2024. Prepared by EOGT Preparation (Section 9) and 2 CO2 tubes.
  - Savant may run these conditions as well, 2 weeks complete on June 18, and will continue to run until see difference in 2 oils
- SwRI Cold Soak Tests #4: Newly prepared samples CMIR 188836 and 188837 were stored for 14 days in 20C cabinet and then 14 days in 0C freezer. After these conditions, they were removed and stabilize to room temperature (~22C) for 2 hours. Samples were inverted and mixed with rod until appearing homogeneous and then testing filterability.
  - Notes: These CMIR were received from TMC May 30, 2024. Prepared by EOGT Preparation (Section 9) and 2 CO2 tubes.

## Results

### SwRI Cold Temp Soak Test #3 Results

Condition	CFA = Change in Flow Rate	CMIR 183766 (Oil F)	CMIR 183768 (Oil K)
7 days 20C + 7 days 0C	CFA Average, %	To complete by June 14	
7 days 20C + 7 days 0C. If Needed, additional 7 days 20C + 7 days 0C	CFA Average, %	To complete by June 28	

### SwRI Cold Temp Soak Test #4 Results

Condition	CFA = Change in Flow Rate	CMIR 183766 (Oil F)	CMIR 183768 (Oil K)
14 days 20C + 14 days 0C	CFA Average, %	To start June 10	



# Cold Temperature Soak Tests – SwRI Tests

- Procedure
- SwRI Cold Soak Tests #5: Newly prepared samples CMIR 188836 and 188837 were stored for 21 days in 20C cabinet and then 7 days in 0C freezer. After these conditions, they were removed and stabilize to room temperature (~22C) for 2 hours. Samples were inverted and mixed with rod until appearing homogeneous and then testing filterability.
  - Notes: These CMIR were received from TMC May 30, 2024. Prepared by EOGT Preparation (Section 9) using Waring blender and 2 CO2 tubes.
- SwRI Cold Soak Tests #6: Newly prepared samples CMIR 188836 and 188837 were stored for alternating 1 day at 20C cabinet and 1 day at 0C freezer for 10 days. After these conditions, they were removed and stabilize to room temperature (~22C) for 2 hours. Samples were inverted and mixed with rod until appearing homogeneous and then testing filterability.
  - Notes: These CMIR were received from TMC May 30, 2024. Prepared by EOGT Preparation (Section 9) using Waring blender and 2 CO2 tubes.

## Results

### SwRI Cold Temp Soak Test #5 Results

Condition	CFA = Change in Flow Rate	CMIR 183766 (Oil F)	CMIR 183768 (Oil K)
21 days 20C + 7 days 0C	CFA Average, %	To start June 11	

### SwRI Cold Temp Soak Test #6 Results

Condition	CFA = Change in Flow Rate	CMIR 183766 (Oil F)	CMIR 183768 (Oil K)
20C and 0C 24 hour rotation for 10 Days	CFA Average, %	To start June 13	



EOGT FISHBONE ACTION ITEMS LIST - Revised 23May2024

Category		Cause	Action to Evaluate	Due Date	Status	Date Closed
Method/Environment		Storage Time Temperature/Thermal Cycling	Savant performing "cold soak" on post test retains (repeat storage at 32F), bring to room temperature, then repeat filtration. Want additional tests on performance oils with cold soak: SwRI to run at cold storage -5C and 7C with waring blender with 2 CO2 tubes on 2 performance oils. Savant to consider a second run.	May 10	Savant reported differentiation on change in filter rate after cold soak for the 2 samples (retains from 8% water). <b>Waiting for additional results from SwRI and Experiments 1-4.</b>	
Environment		Differences in storage conditions from initiating lab	Request participating labs to monitor and report storage temperatures, as well as confirm they are in a controlled environment (set temperature, no fluctuations due to company energy savings)	May	YM to ask labs to provide response to TMC, <b>missing 4 labs response</b>	
Equipment		Shearing tool and calibration	ISP ask to share how rpm is calibrated; <b>Group to review the different blade types used:</b> Afton: IKA T25, IKA S25N-18G (rotor and stator gap: 0.3mm); ISP: IKA T18 now digital T18 , IKA S18N-19G (rotor and stator gap: 0.25 mm); SwRI: IKA S25N-18G; Savant: Waring blender	May	tool used hasn't been calibrated and uses setting 6 for 18,000 rpm; a new digital version has a connection to "calibrate"; ISP is using new head: <a href="https://www.ika.com/de/Produkte-LabEq/Dispergierer-pg177/T-18-digital-ULTRA-TURRAX-3720000/">https://www.ika.com/de/Produkte-LabEq/Dispergierer-pg177/T-18-digital-ULTRA-TURRAX-3720000/</a> , same blade. <b>Group decide to put this on hold.</b>	
Materials	different water types used		Angela to share differences between Type I, II, III and share links to papers on silica forming more carbonate; <b>Check silica content of DI water from different labs (ISP, Savant, SwRI)</b>	May	With increasing silica content, may form more carbonate; Type III has most silica (up to 500 ppb). <b>Group decide to put this on hold.</b>	
Materials	filter paper variation	understand batch variability and if any differences in cutting filters	YM to ask OHT to provide 1) tolerances for pore size and beta efficiency from manf, 2) could share with main group their procedure for cutting filter paper and quality protocol to ensure integrity and compliance; 3) C of A possible to be shared. Rob to check on magnification to review filters and any suggestions on inspection process for filters from filter expert. <b>Ask if all labs would look at 3 filters under microscope under 75X magnification and provide photos and observations on pore structure (ie signs of pore plugging or collapse due to damage). Request all labs to provide photos of new filter and filter in holder.</b>	20-Jun	Ask: how is inspection done (microscope or by eye), detail on inspect: what happens to die if there are bent, slugs or debris; assume 13 mm diameter? For 1 batch cutting, what is inspection frequency for batch? How many total parts are inspected and how are they selected for inspection (are they representative of batch). Reviewed Ford filter expert comments on process.	
Equipment	Filter holder		YM ask labs to share part #, vendor and photo of holder to TMC	May	Missing 4 labs response	
Methods	homogenizing step		YM ask labs to check if homogenization step is accurately described in Data excel		<b>No changes mentioned from any lab, assuming no response is there were no changes from what was previously submitted in Data Excel</b>	
	<b>Humidity Conditions</b>		<b>Will be requesting labs to monitor humidity conditions for lab area and storage, in the future</b>		<b>Angela to add to fishbone</b>	
			<b>Next meeting: June 14 at 9am CDT</b>			



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## **BLENDING EQUIPMENT SUMMARY EOGT**

20240522

# BLENDING EQUIPMENT SUMMARY

LAB ID	Blade Name/Part (weblink if avail)	Blender Name/Part (weblink if avail)	Cup Name/Part (weblink if avail)	Air Regulator Distance	Photos (Y/N)
D	<i>Cole-Parmer Item# EX-00111-KK</i> <a href="#">BLADE FOR 250 ML CONTAINER from Cole-Parmer (coleparmer.com)</a>	<i>Model: HGB2WTS3</i> <i>Supplier Part# 7011HS</i> <i>Fisher Catalog No. 14-509-54</i> <a href="#">Conair Waring Two-Speed 1L Blenders Stainless-steel jar:Mixers, Quantity:   Fisher Scientific</a>	<i>Mfr # MC3</i> <i>Cole-Parmer Item# EW-04248-30</i> <a href="#">Waring MC-3 Mini containers; 50 to 250 mL capacity from Cole-Parmer (coleparmer.com)</a>		Yes
A	Blade for 250 mL Container. We think this is the blade we use, but we are not 100% sure. We are waiting for the order to come in later this week. <a href="#">BLADE FOR 250 ML CONTAINER from Cole-Parmer (coleparmer.com)</a> Cole-Parmer Item# EW-00111-KK	<a href="#">Conair Waring Two-Speed 1L Blenders Stainless-steel jar:Mixers, Quantity:   Fisher Scientific</a> Model: HGB2WTS3 Supplier Part# 7011HS Fisher Catalog No. 14-509-54	The Cup we use is a Waring MC-3 Mini containers; 50 to 250 mL capacity <a href="#">Waring MC-3 Mini containers; 50 to 250 mL capacity from Cole-Parmer (coleparmer.com)</a> Mfr # MC3 Cole-Parmer Item# EW-04248-30	2 stands, Stand to test regulator 3.5 and 4 ft.	Yes
G	Blade for 250mL Supplier: Cole Parmer Item: EW-00111-KK <a href="https://www.coleparmer.com/i/blade-for-250-ml-container/00111kk?searchterm=EW-00111-KK">https://www.coleparmer.com/i/blade-for-250-ml-container/00111kk?searchterm=EW-00111-KK</a>	Conair Waring 700G Model: 700G Catalog No: 14-509-10 Supplier: Fisher Sci <a href="https://www.fishersci.com/shop/products/waring-laboratory-blenders-single-speed-2/1450910#conair%20waring%20700G">https://www.fishersci.com/shop/products/waring-laboratory-blenders-single-speed-2/1450910#conair%20waring%20700G</a>	Conair CAC64 Supplier: Fisher Sci Catalog No: 14-509-42 <a href="https://www.fishersci.com/shop/products/waring-laboratory-blenders-single-speed-2/1450910#conair%20waring%20700G">https://www.fishersci.com/shop/products/waring-laboratory-blenders-single-speed-2/1450910#conair%20waring%20700G</a>	Airgas 12FM244B320-AG Part#: Y12ASN245E320 Supplier: Airgas Tube: Y21B65L1	Yes
I	<a href="https://www.ika.com/de/Produkte-LabEq/Dispergierer-pg177/S-18-N-19-G-Dispergierwerkzeug-L004640/">https://www.ika.com/de/Produkte-LabEq/Dispergierer-pg177/S-18-N-19-G-Dispergierwerkzeug-L004640/</a>	No link available for the basic version. It is now a digital one: <a href="https://www.ika.com/de/Produkte-LabEq/Dispergierer-pg177/T-18-digital-ULTRA-TURRAX-3720000/">https://www.ika.com/de/Produkte-LabEq/Dispergierer-pg177/T-18-digital-ULTRA-TURRAX-3720000/</a>	<a href="https://si.vwr.com/store/product/568695/flat-flange-beaker-duran">https://si.vwr.com/store/product/568695/flat-flange-beaker-duran</a>		Yes
V	Waring Commercial Blender 51BL30(7010) 120V		N/A	3-ft	Yes
E1	Waring Variable Speed Laboratory Blender		N/A	~2.5 ft	Yes



# Blender

## Lab V



## Lab I



## Lab D



## Lab A



## Lab G



## Lab E1



# Mixer Impeller

Lab V



Lab I



Lab D

No Photo

Lab A



Lab G

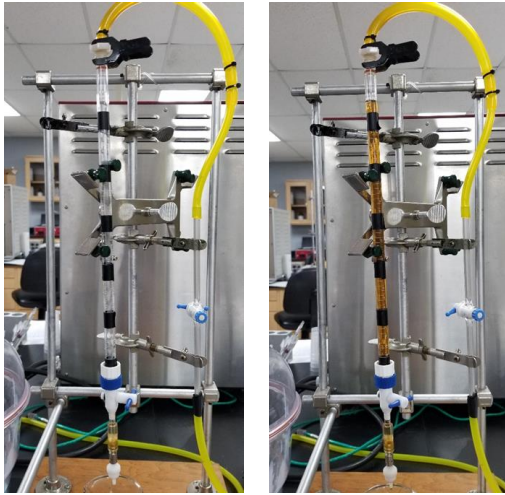


Lab E1



# Filtration Burette

Lab V



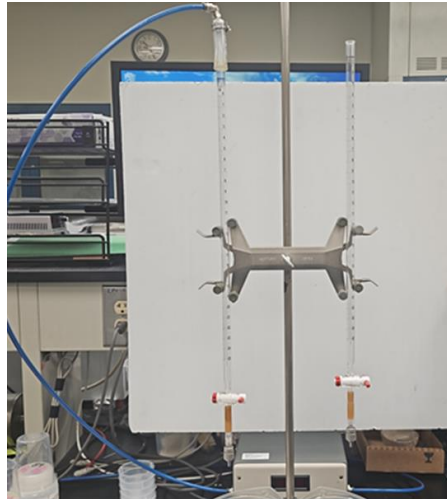
Lab I



Lab D

No Photo

Lab A





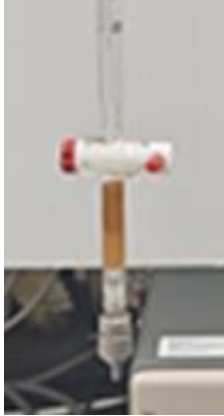
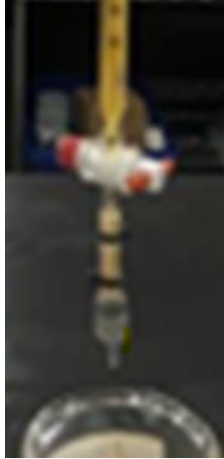

Lab G





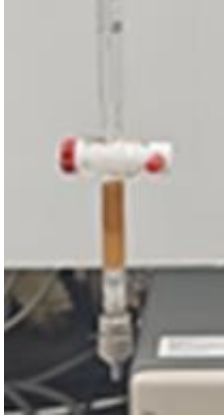
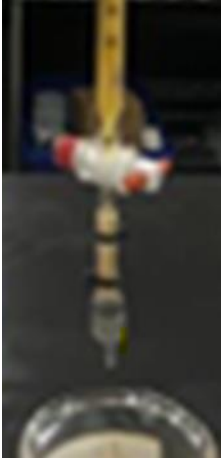

Lab E1



# Filtration Burette TIP SIZE

<p><b>Lab V</b></p> <p>it is ~1.5 mm.</p> 	<p><b>Lab I</b></p> <p>Between 1.8mm And 1.9mm</p> 	<p><b>Lab D</b></p> <p>1.32mm</p> <p><b>No Photo</b></p>
<p><b>Lab A</b></p> <p>32 burettes. Half at 1.8, half 1.7 at least 1.7</p> 	<p><b>Lab G</b></p> <p>Metal Tip 1.8mm</p> 	<p><b>Lab E1</b></p> <p>1.8 mm</p> 

# Filter Holder: ID#, Type, and Photo

<p><b>Lab V</b></p> <p>ID# I'm not sure of the part numbers for the filter holders but I'm pretty sure we would have gotten it from OHT long ago.</p> <p>TY</p> 	<p><b>Lab I</b></p> <p>ISP uses a 13 mm SST Filter holder from EMD Milipore Corporation. Article number is XX3001200.</p> 	<p><b>Lab D</b></p> <p>ID#</p> <p>TYPE</p>
<p><b>Lab A</b></p> <p>ID#</p> <p>TYPE</p> 	<p><b>Lab G</b></p> <p>ID#</p> <p>TYPE</p> 	<p><b>Lab E1</b></p> <p>ID#</p> <p>TYPE</p> 

# Storage Temperature Variation over Length of Storage Time

<p style="text-align: center;"><b>Lab V</b></p> <p>H We store our samples in one of our main labs and track the temperature throughout the day but not in direct relation to this testing. The temperature spans from 68F – 73F</p> <p>SI throughout the year.</p> <p>L Reading Frequency</p>	<p style="text-align: center;"><b>Lab I</b></p> <p>High</p> <p>SET Point</p> <p>Low</p> <p>??? Hot or cold storage??</p> <p>Reading Frequency</p>	<p style="text-align: center;"><b>Lab D</b></p> <p>High</p> <p>SET Point</p> <p>Low</p> <p>Reading Frequency</p>
<p style="text-align: center;"><b>Lab A</b></p> <p>High</p> <p>SET Point</p> <p>Low</p> <p>Reading Frequency</p>	<p style="text-align: center;"><b>Lab G</b></p> <p>High</p> <p>SET Point</p> <p>Low</p> <p>Reading Frequency</p>	<p style="text-align: center;"><b>Lab E1</b></p> <p>Hi The samples were stored in a box, in a cabinet on the main floor of our facility. I do not expect significant</p> <p>SE temperature swings above or below ambient during the</p> <p>Lc time period in which the testing was conducted</p> <p>Reading Frequency</p>

9.3 When sample temperature has reached  $(50.0 \pm 1)^\circ\text{C}$ , start the  $\text{CO}_2$ -flow  $(6 \pm 0.5) \text{ L/h}$  and maintain sample temperature at  $(50.0 \pm 1)^\circ\text{C}$  for  $(60 \pm 1)$  minutes.

9.4 Remove sample from heat after 60mins. Homogenize sample and transfer equal aliquots to 4 inert lined containers. A policeman or non-metallic spatula may be used to help mix any precipitate before transferring sample as equally as possible to the multiple containers.

9.5 Store the containers in dark at room temperature  $(20 \text{ to } 24)^\circ\text{C}$  for 72hrs and 336hrs.

9.6 Dry filters in an oven at  $(70 \pm 2)^\circ\text{C}$  for  $(30 \pm 2)$  min and store in a desiccator until used.

9.7 Determine Filterability at the end of 72hrs and 336hrs ( $\pm 2$  hours), after removing from heat.

9.8 Determine ICP within 24 hrs at the end of 72hrs and 336hrs.

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# Air Regulators

Lab V



Lab I



Lab D

No Photo

Lab A

No Photo

Lab G



Lab E1

2-Stage Air regulation System





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# Draft Timeline – updated May 22

Task	Date											
	5-6 2023	7-8 2023	9-10 2023	11- 12 2023	1-2 2024	3-4 2024	May-Jun 2024	Jul-Aug 2024	Sept-Oct 2024	Nov-Dec 2024	Jan-Feb 2025	
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			█	█	█	█	█	█				
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>												



# Action Items and Next Meeting

- Group to decide next steps for EOGT.
  - Labs to let TMC know on storage temperature (after blending and bubbling CO<sub>2</sub>) from Labs I, D, A and G.
  - Labs to let TMC know on part number, vendor, and photo of filter holder; OHT also offered info on part from Labs D, A, G, EI.
  - Anyone have concerns with sending out TMC summary of lab equipment, please let John and Yong-Li know by COB Tue June 11.
  - SwRI and Savant to report their Cold Soak Tests to Root Cause group.
  - Yong-Li to work on slides for API AOAP, PCEOCP, and B.07.
- 
- Next Meeting: Monday July 15 at 8:30AM CDT



# Thank you for your support!

Participants		
Method Development (11)	Oil Donations (9)	Testing Labs (7)
Afton	Afton	Afton (Screening, Pilot, and ILS)
ExxonMobil	Ford	Intertek (Screening, Pilot, and ILS)
Ford	Infineum	ISP (Screening, Pilot, and ILS)
Infineum	Lubrizol	Savant (Screening and ILS)
Intertek	OH Technologies (donate filters only)	SwRI (Screening, Pilot, and ILS)
ISP	Oronite	TMC (monitoring system only)
Lubrizol	Subaru	Valvoline (Screening and ILS)
Oronite	TMC (collection, shipping only)	
Savant	Toyota	
SwRI		
TMC		

