ASTM New Engine Oil Gelation Test (EOGT) WK86363 Update

EOFT and EOWTT Surveillance Panel Meeting

February 12, 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
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

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 May, ILS tests to be run by September, and final method ballot in November or December 2024

Testing Status – updated Nov 16

EOGT report form Rev 6

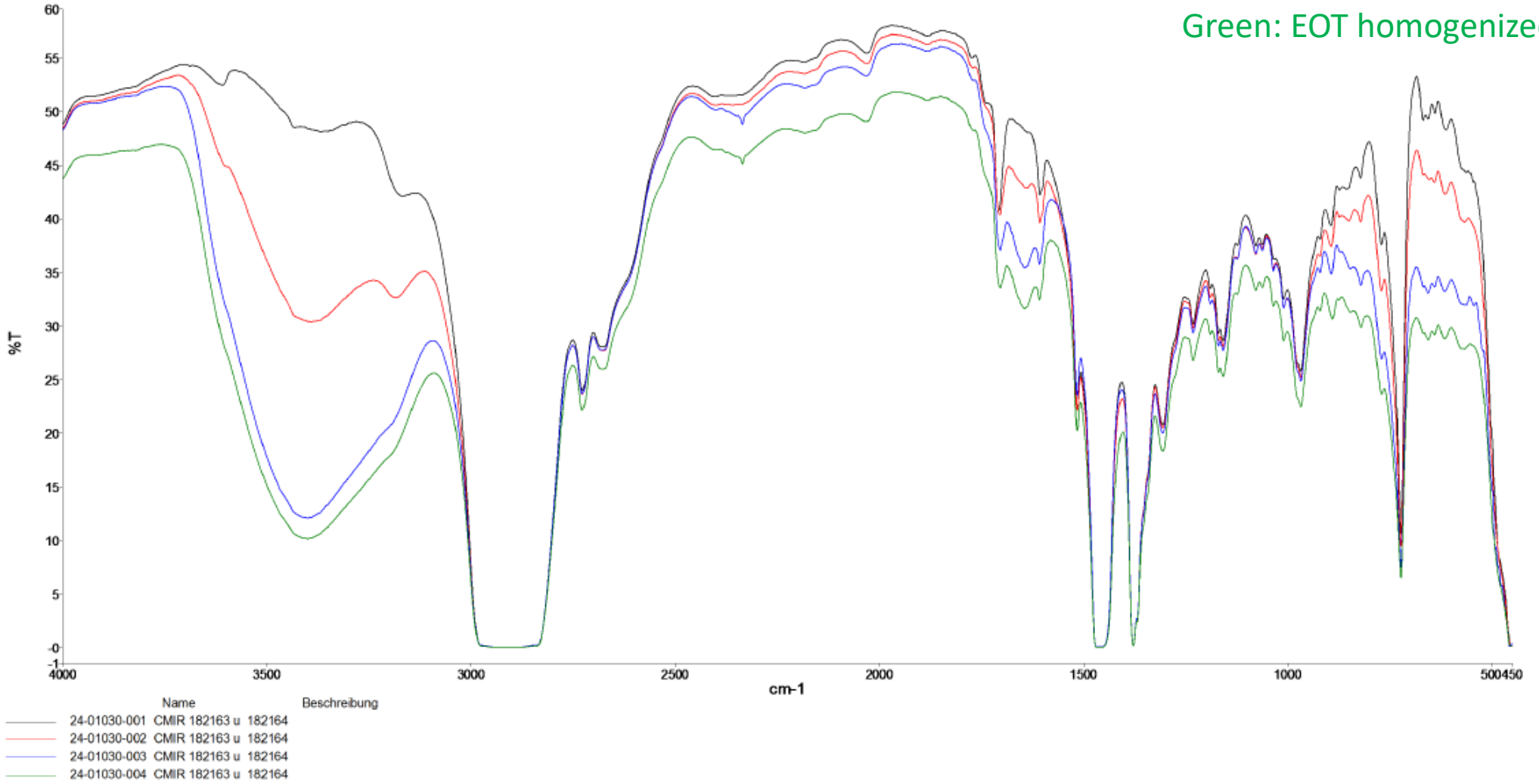
Test Phase	Description	Status	Comments
1. Screening Tests	-4 tests; 2 oils: low performance oil in duplicate, high performance oil in duplicate; 6 labs -Run LV method -Intent: check labs can run test as set up	All 6 labs submitted data	Run SOT & 14 day EOT only. **-REVIEW data**
2. Pilot Study	-? Tests; ? Oils; 4 labs -Run SV and LV methods, all intervals -Intent: determine samples, method, and intervals for procedure and ILS	Waiting to determine samples: -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?) -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	**Group to decide as we move forward**
3. Interlaboratory Study (ILS)	-? Tests; ? Oils; 6 labs -Run _ method -Intent: generate repeatability and reproducibility data on samples, determine at least 2 reference oils	Waiting to determine samples	

Proposal Experiments (1 of 2) – updated Feb 9

Proposal Experiments	Lab(s)	Status	Next Step
1. Sediment-only Filterability	Afton	Completed	GROUP to make comments on if this is helpful – no feedback to use
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: Completed Afton: Completed	Group to make comments
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 – ran on ATR Valvoline: completed IR, microscopy did not work	Group to review data, Savant and Valvoline to add any data related to 870 wavelength, Ford to give feedback on how useful IR maybe in EOGT-No comments, ISP to possibly provide IR for previous EOT samples
6. Centrifuge sample before filterability	SwRI	Completed	
7. No homogenizing before filterability	Intertek	Completed	GROUP to make comments on if this is helpful- no feedback to use
8. Run EOGT with 4 hr CO2 bubbling and heating (run with 2 flow tubes)	SwRI	Completed	Additional CO2 not necessary, running with 2 tubes helps, gel not sticking to plastic jars compared to sticking to glass

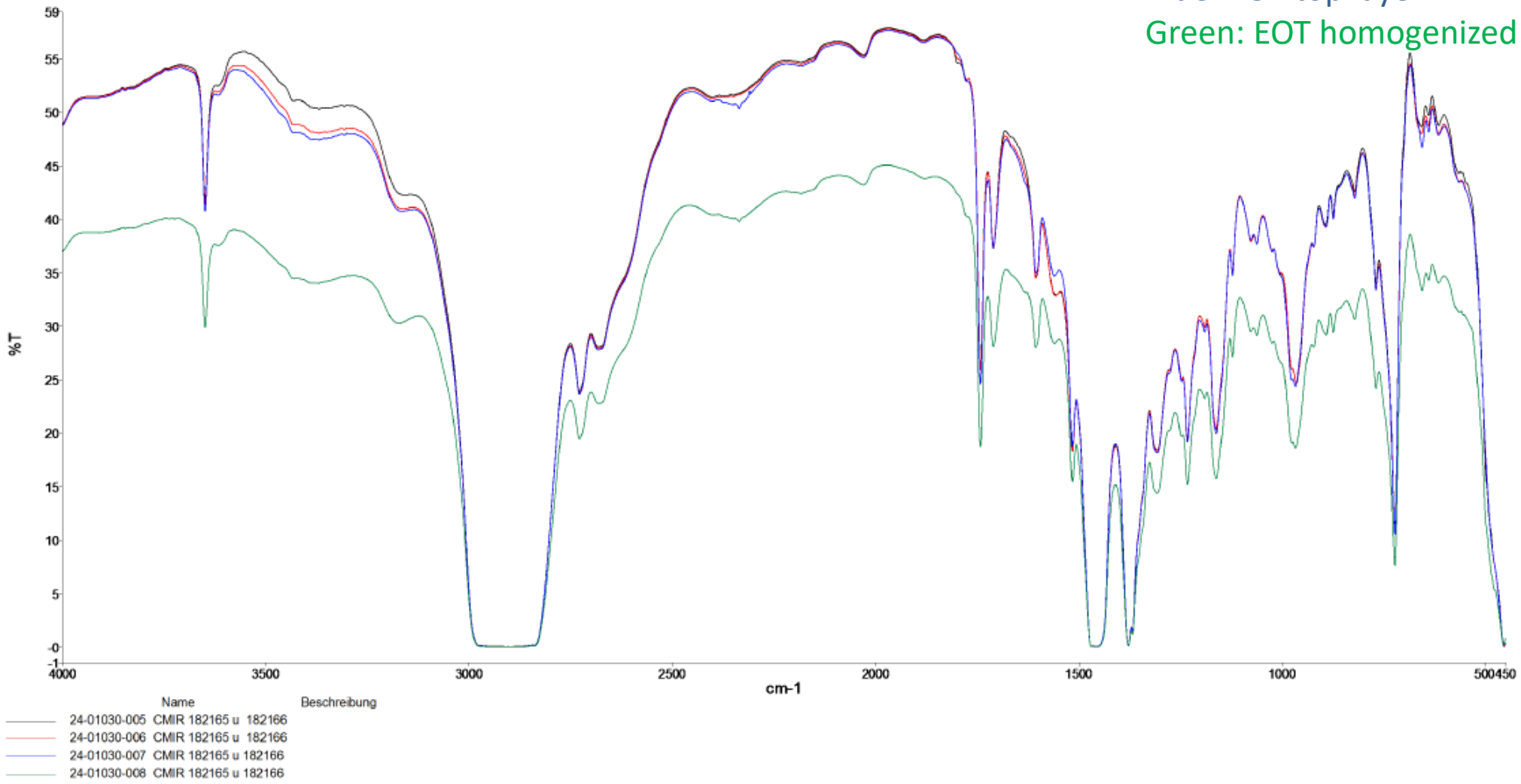
ISP IR data 182163 & 182164 (Oil K)

Black: New oil
Red: 0 hr
Blue: EOT top layer
Green: EOT homogenized



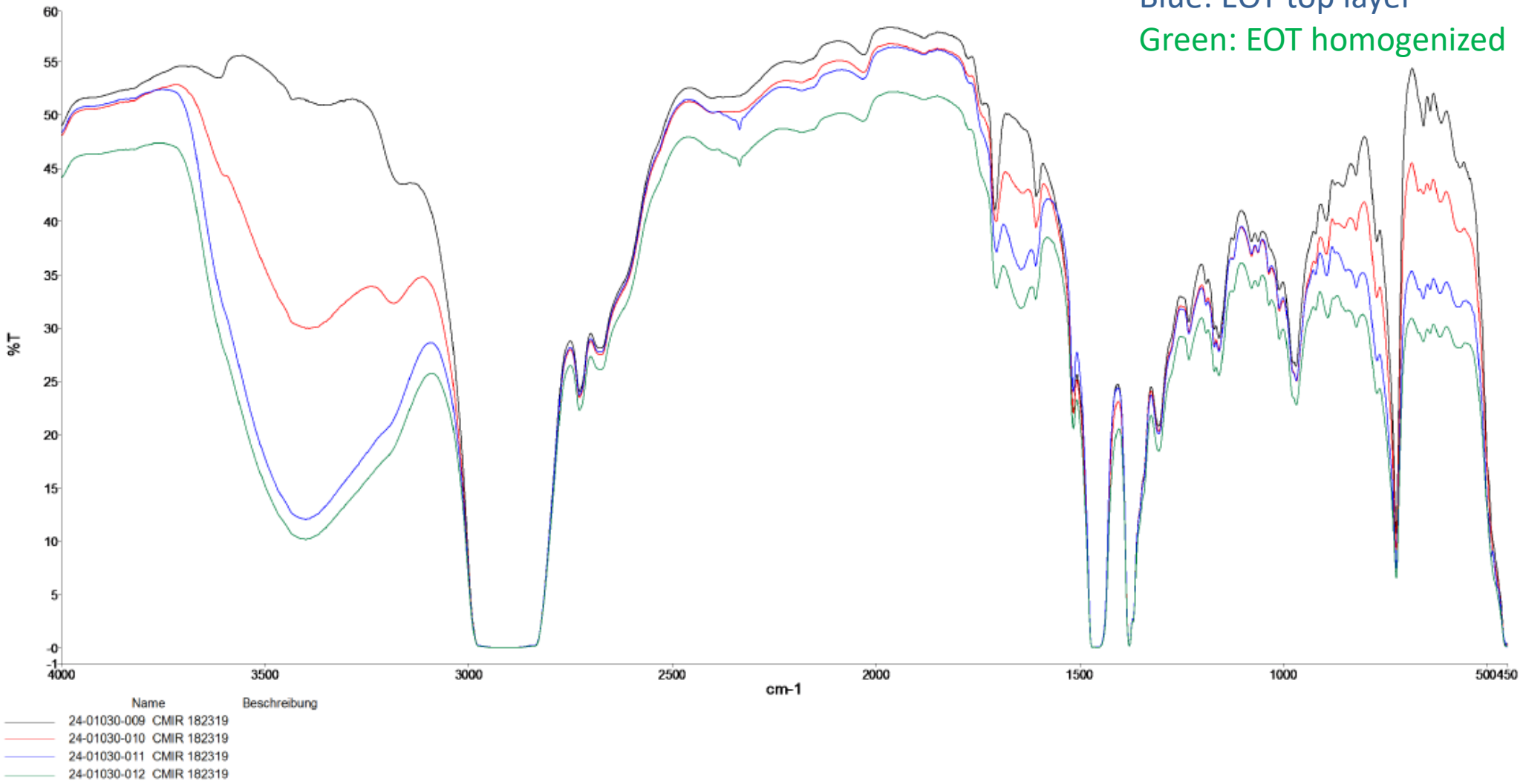
ISP IR data 182165 & 182166 (Oil F)

Black: New oil
Red: 0 hr
Blue: EOT top layer
Green: EOT homogenized



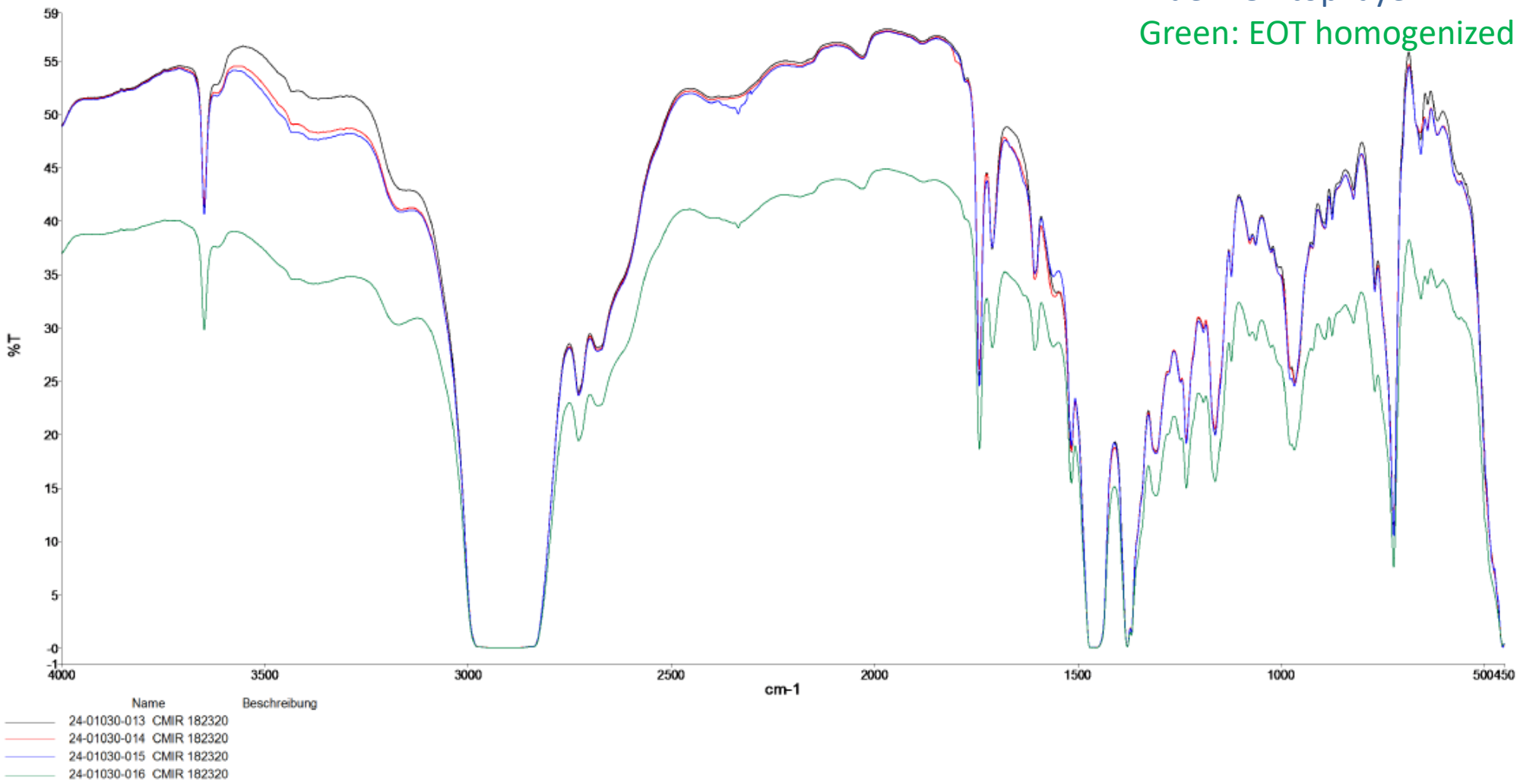
ISP IR data 182319 (Oil K)

Black: New oil
Red: 0 hr
Blue: EOT top layer
Green: EOT homogenized



ISP IR data 182320 (Oil F)

Black: New oil
Red: 0 hr
Blue: EOT top layer
Green: EOT homogenized



Proposal Experiments (2 of 2) – updated Feb 9

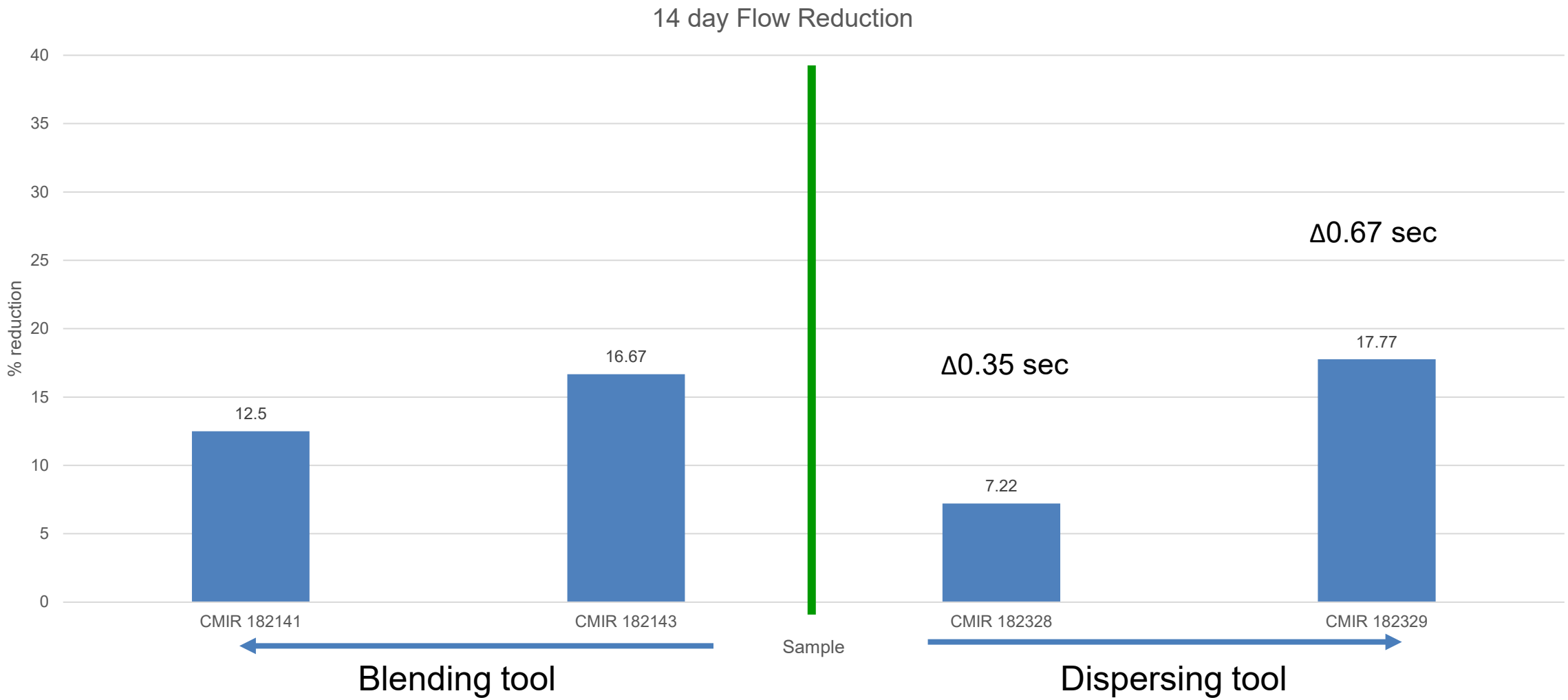
Proposal Experiments	Lab(s)	Status	Next Step
9. Run EOGT with 1 CO2 tube, plastic jars for storage, and dispersing tool to report Change in filtering rate results on 2 performance oils	Afton	Completed Feb 9	(Include IR on fresh oil, top layer, sediment layer, and mixed EOT spectra) Results didn't show sufficient differentiation between the 2 oils using the 1 CO2 tube, dispersing tool, and plastic jars.
10. Run EOGT with 2 CO2 tubes, plastic jars for storage, and waring blender setup to report Change in filtering rate results on 2 performance oils	ISP	Expected to complete Feb 16	(Include IR on fresh oil, top layer, sediment layer, and mixed EOT spectra)
11. Afton to help write an IR detection method for calcite	Afton	Currently working on procedure and will share when ready.	

Gelation Methodology

Background

- Does type of mixing affect filterability results?
  - Shearing vs Dispersing
- Dispersing unit and tool
  - Description - T 25 digital ULTRA-TURRAX® (ika.com)
  - Description - S 25 N - 18 G Dispersing tool (ika.com)
- Stored in plastic bottle

Results



Raw data (CMIR 182328/CMIR 182329)

Volume	Fresh (sec)	14 day (sec)
0		
5	4.59	4.24
10	4.32	4.55
15	4.46	4.57
20	4.39	4.86
25	4.50	4.85

Volume	Fresh (sec)	14 day (sec)
0		
5	3.04	3.08
10	2.94	3.23
15	3.18	3.67
20	3.03	3.49
25	3.10	3.77

Other updates

- Review of lab equipment information (blade, blender, cup, filtering system)
- What is the next step?
  - Notes: Labs have reviewed water types, CO2 sources, and as many variables of the EOGT test and have tried to determine what would make results more similar to ISP lab results. Without more direction from OEMs, there aren't any other actions to take (other than wait for ISP results).

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	*Cole-Parmer Item# EX-00111-KK* BLADE FOR 250 ML CONTAINER from Cole-Parmer (coleparmer.com)	*Model: HGB2WTS3 Supplier Part# 7011HS Fisher Catalog No. 14-509-54* Conair Waring Two-Speed 1L Blenders Stainless-steel jar:Mixers, Quantity: | Fisher Scientific	*Mfr # MC3 Cole-Parmer Item# EW-04248-30* Waring MC-3 Mini containers; 50 to 250 mL capacity from Cole-Parmer (coleparmer.com)		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. BLADE FOR 250 ML CONTAINER from Cole-Parmer (coleparmer.com) Cole-Parmer Item# EW-00111-KK	Conair Waring Two-Speed 1L Blenders Stainless-steel jar:Mixers, Quantity: | Fisher Scientific 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 Waring MC-3 Mini containers; 50 to 250 mL capacity from Cole-Parmer (coleparmer.com) 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 https://www.coleparmer.com/i/blade-for-250-ml-container/00111kk?searchterm=EW-00111-KK	Conair Waring 700G Model: 700G Catalog No: 14-509-10 Supplier: Fisher Sci https://www.fishersci.com/shop/products/waring-laboratory-blenders-single-speed-2/1450910#conair%20waring%20700G	Conair CAC64 Supplier: Fisher Sci Catalog No: 14-509-42 https://www.fishersci.com/shop/products/waring-laboratory-blenders-single-speed-2/1450910#conair%20waring%20700G	Airgas 12FM244B320-AG Part#: Y12ASN245E320 Supplier: Airgas Tube: Y21B65L1	Yes
I	https://www.ika.com/de/Produkte-LabEq/Dispergierer-pg177/S-18-N-19-G-Dispergierwerkzeug-L004640/	No link available for the basic version. It is now a digital one: https://www.ika.com/de/Produkte-LabEq/Dispergierer-pg177/T-18-digital-ULTRA-TURRAX-3720000/	https://si.vwr.com/store/product/568695/flat-flange-beaker-duran		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

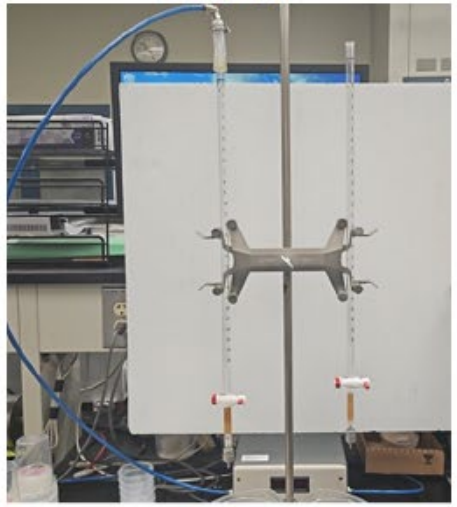
Lab V

Lab I

Lab D

No Photo

Lab A



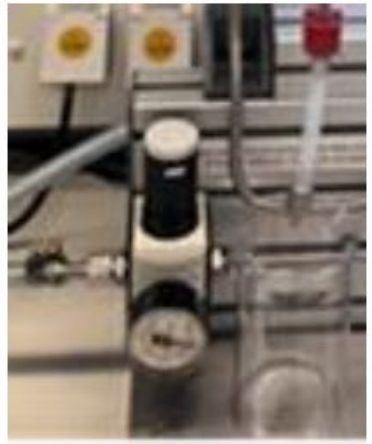
Lab G

Lab E1

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 Feb 9

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 / Nov-Dec 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	
Generate pass/fail limits [Outside this Surveillance Panel]	

Action Items and Next Meeting

- ISP to complete additional proposal test
- Group to let Yongli know on direction for EOGT
- Group to review existing data and share any comments for next meeting

- Next Meeting: Monday February 26 at 8:30AM CDT and every ? weeks

Thank you for your support!

Participants		
Method Development (11)	Oil Donations (9)	Testing Labs (7)
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)