ASTM New Engine Oil Gelation Test (EOGT) WK86363 Update

EOFT and EOWTT Surveillance Panel Meeting

March 25, 2024

Yong-Li McFarland, Chair

ASTM Antitrust and Recording Policy

ASTM International is a not-for-profit organization and developer of voluntary consensus standards. ASTMs leadership in international standards development is driven by the contributions of its members: more than 30,000 technical experts and business professionals representing 135 countries.

The purpose of antitrust laws is to preserve economic competition in the marketplace by prohibiting, among other things, unreasonable restraints of trade. In ASTM activities, it is important to recognize that participants often represent competitive interests. Antitrust laws require that all competition be open and unrestricted.

It is ASTMs policy, and the policy of each of its committees and subcommittees, to conduct all business and activity in full compliance with international, federal and state antitrust and competition laws. The ASTM Board of Directors has adopted an antitrust policy which is found in Section 19 of ASTM Regulations Governing Technical Committees. All members need to be aware of and compliant with this policy. The Regulations are accessible on the ASTM website http://www.astm.org/COMMIT/Regs.pdf).

Electronic recording of ASTM meetings is prohibited.

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

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
- Let Yong-Li know if can help by April 3

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

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

Proposal Experiments (2 of 2) – updated March 22

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	Completed 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.	
12. Run EOGT with 2 CO_2 tubes, dispersing tool (IKA S18N-19G), and plastic jars, 600 g volume on 2 performance oils	SwRI	Completed March 21	

12. EOGT with dispersing tool, 2 CO_2 tubes, plastic jars, and 600 g volume for 336 hours - SwRI

Sample	Run 1 Change in Flow Rate (%)	Run 2 Change in Flow Rate (%)	Average Change in Flow Rate (%)	Run 1 New Oil Flow Time (s)	Run 1 EOT Flow Time (s)	Run 2 New Oil Flow Time (s)	Run 2 EOT Flow Time (s)	Notes
CMIR-185163 (Oil F)	-12.19	-6.44	-9.32	3.53	4.02	3.92	4.19	Change Ca: -15.9%, Change Mo; -3.44%, Change in Mg: -50%
CMIR-185164 (Oil K)	-6.30	-6.32	-6.31	4.61	4.92	4.74	5.06	Change Ca: -2.95%, Change Mo; -23%, Change in Mg: -38.8%

Left: Dispersing tool
Right: Photo of EOT 336 hr samples 185163 and 185164

Other Notes/updates - March 22

- Previously FTIR on filter was looked at – Ford to check if there's method on how IR was done (ATR, combined FTIR-EDS, can data be shared or quantified?- Rob to look into)
- Concern about variation in results due to different extraction processes
- Is XRF/XRD possible?
- Was Ca sulfonate grease used in previous system? – No grease was used in manufacturing process

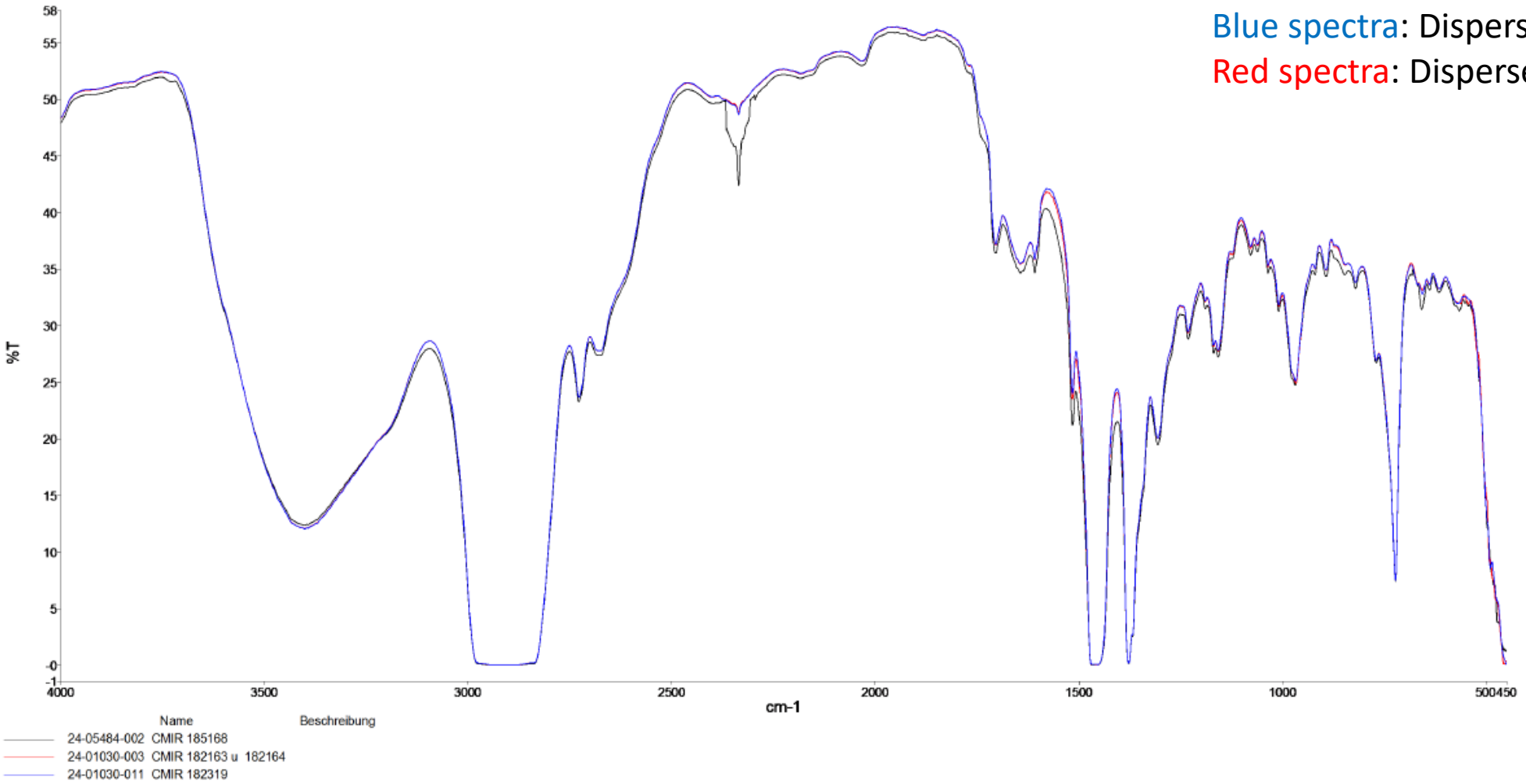
Notes 3-25-24:
dispersing procedure consistent; Filter media questions; particle count, particle size on EOT and SOT?, TMC to confirm on burette tip size; request Type III water contaminant analysis from ISP and SwRI; Angela, Rachelle, (ask Philipp to join) and YM to set up fishbone diagram; filter confirmation; consider changing porosity; ask ISP to repeat differentiating run-Confirm if possible; used about 50% of Oils F and K from TMC

ISP Update - March 22

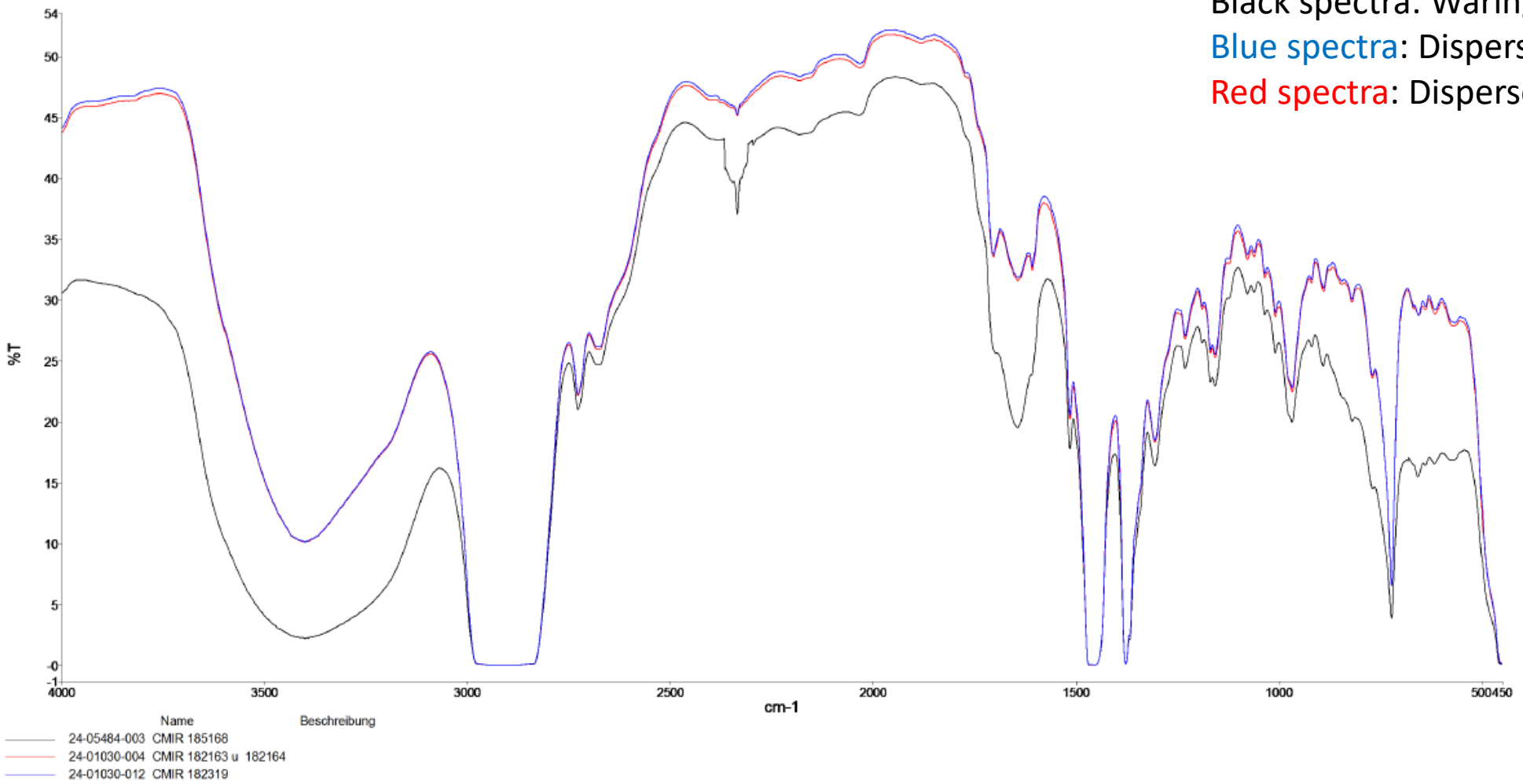
		Change in Filterability (%)			Flow Time New Oil 20-25 mL (s)			Flow Time Aged 20-25 mL (s)		
	CMIR	Run 1	Run 2	Avg.	Run 1	Run 2	Avg	Run 1	Run 2	Avg
EOGT-F	185167	-8.62	-5.69	-7.16	4.30	4.05	4.18	4.72	4.31	4.52
EOGT-F Dispersing Tool	182165+182166	-35.77	-39.26	-37.52	4.07	3.7	3.89	6.31	6.08	6.2
EOGT-F Dispersing Tool	182320	-42.98	-42.4	-42.69	4.4	3.99	4.2	7.75	6.96	7.36
EOGT-K	185168	-3.12	-7.22	-5.17	5.20	5.14	5.17	5.39	5.55	5.47
EOGT-K Dispersing Tool	182163+182164	-9.57	-6.24	-7.91	5.31	5.22	5.27	5.88	5.58	5.73
EOGT-K Dispersing Tool	182319	-4.3	-5.94	-5.12	5.38	4.93	5.16	5.6	5.25	5.43

ISP FTIR Graph Sample K @ 336 hour oil phase

Black spectra: Waring Blender run
Blue spectra: Disperser run
Red spectra: Disperser run



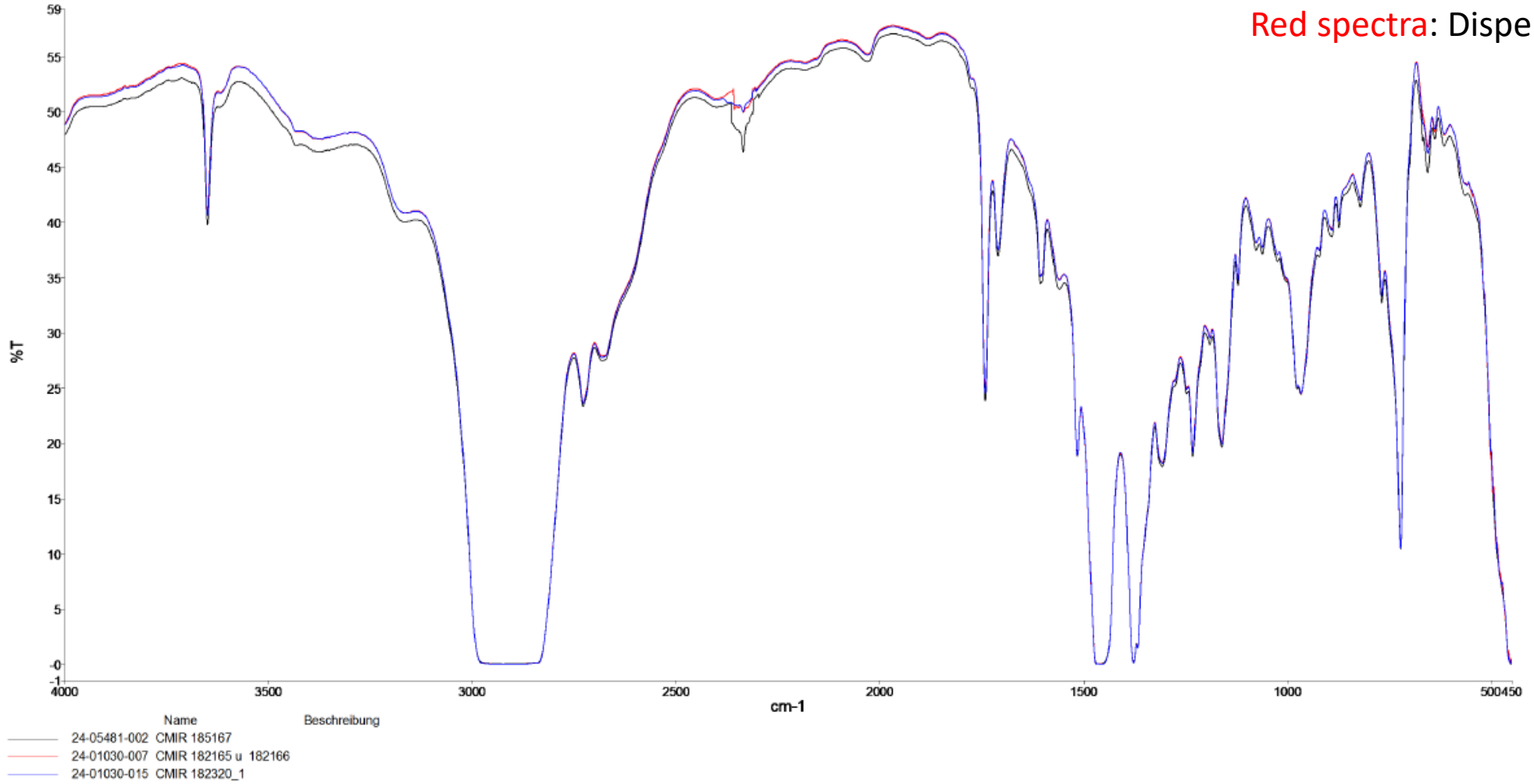
ISP FTIR Graph Sample K @ 336 hour homogenized



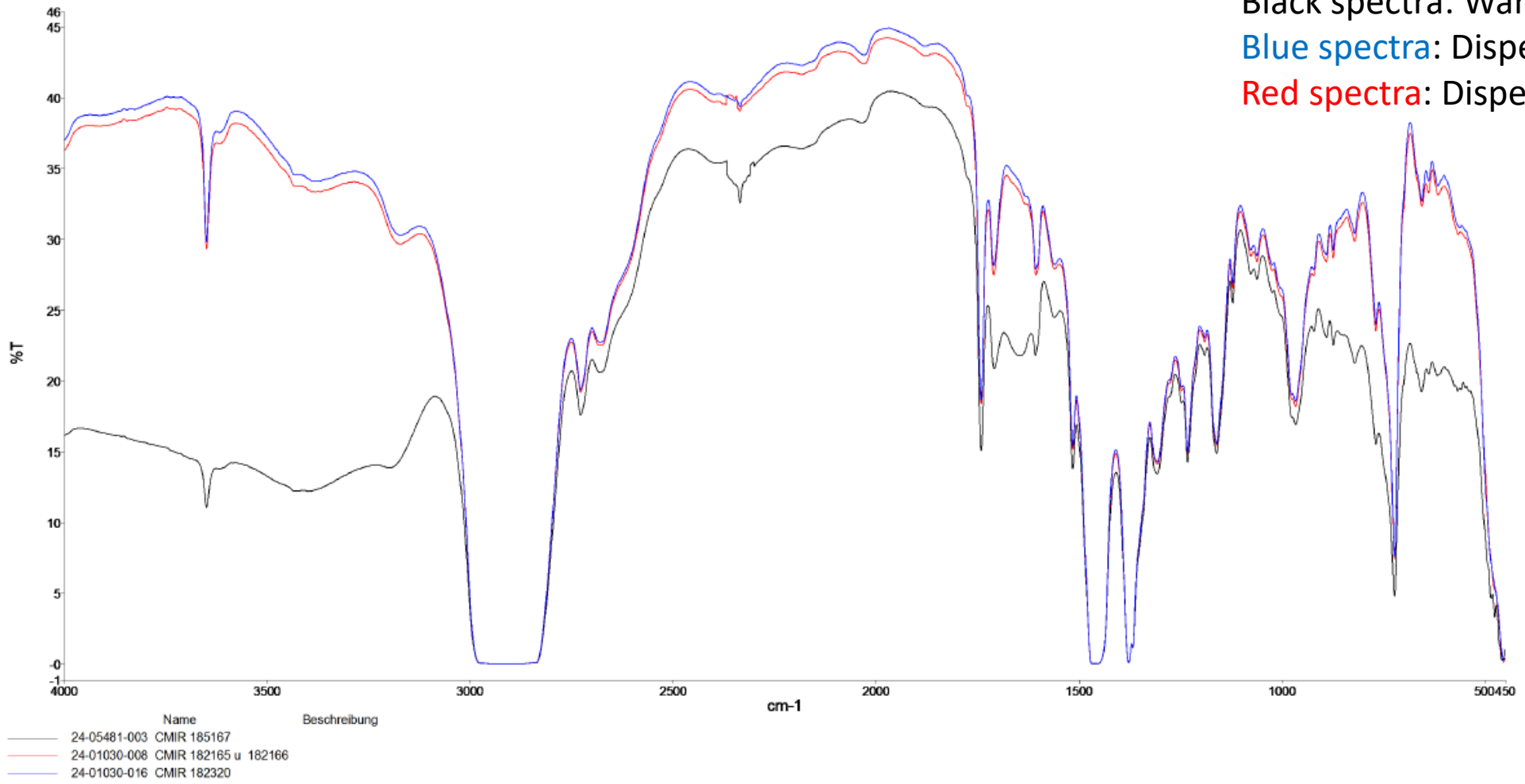
Black spectra: Waring Blender run
Blue spectra: Disperser run
Red spectra: Disperser run

ISP FTIR Graph Sample F @ 336 hour oil phase

Black spectra: Waring Blender run
Blue spectra: Disperser run
Red spectra: Disperser run

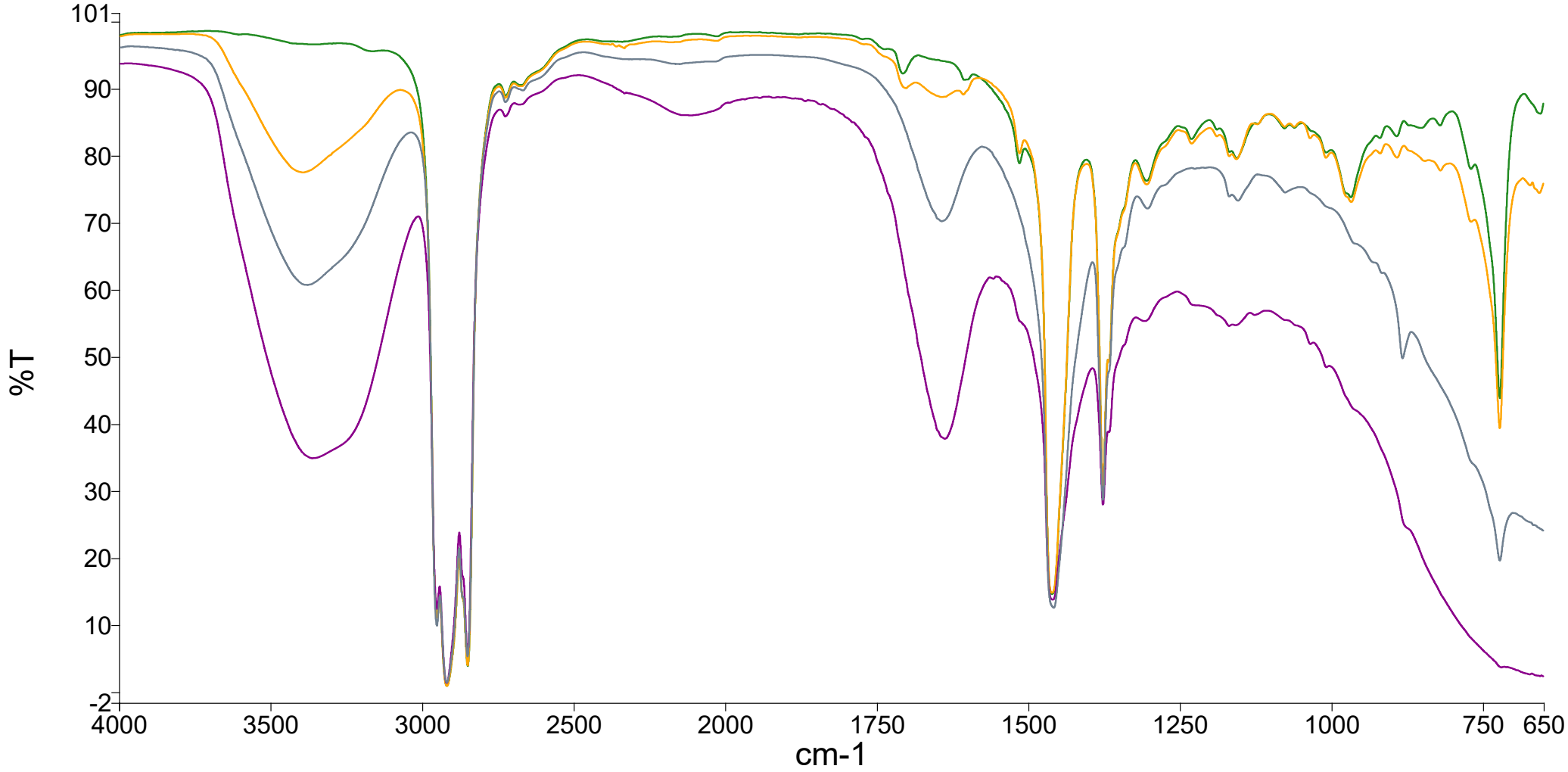


ISP FTIR Graph Sample F @ 336 hour homogenized



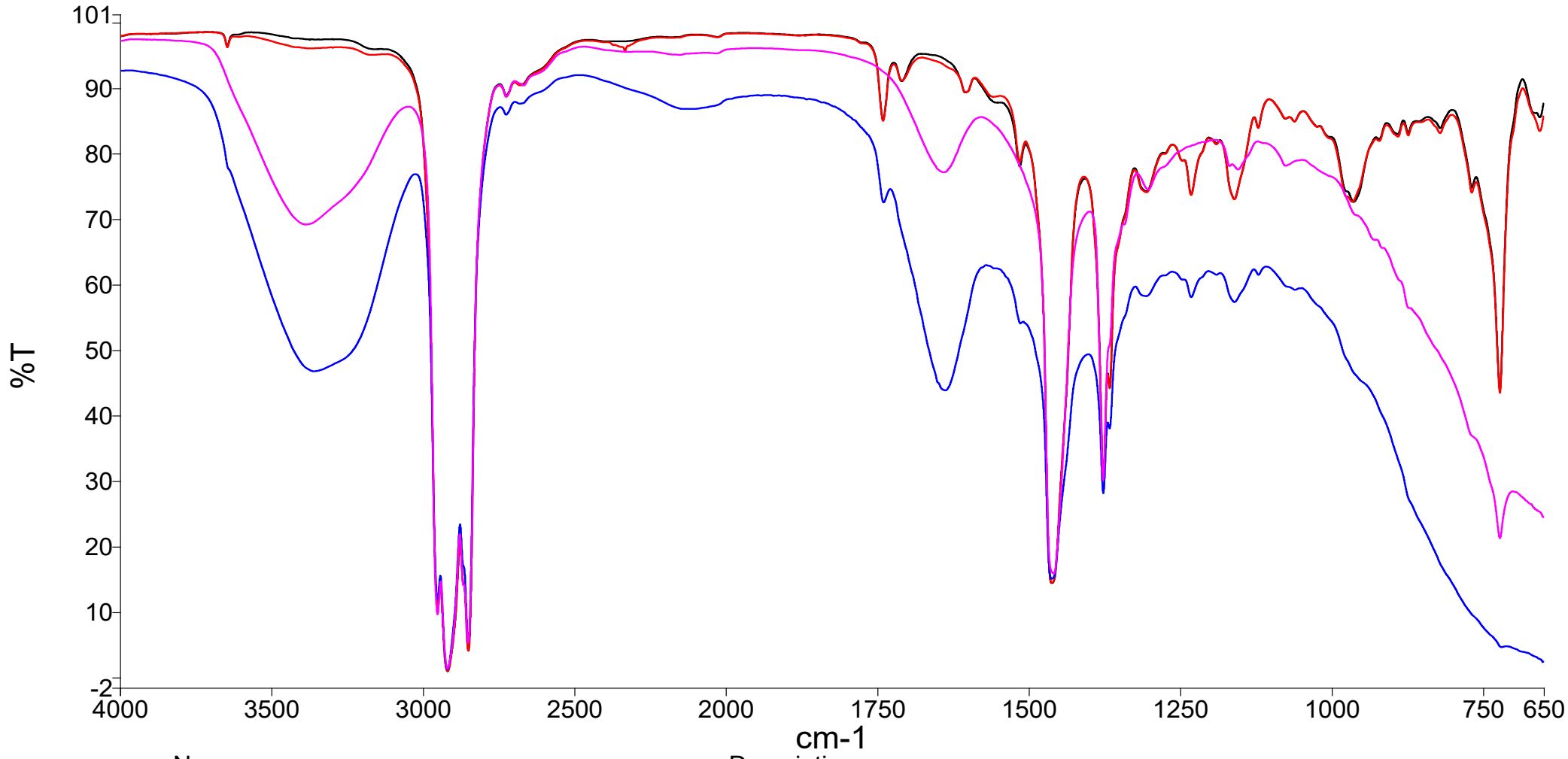
Black spectra: Waring Blender run
Blue spectra: Disperser run
Red spectra: Disperser run

Afton FTIR Set 1: 1.1 – 1.4 (CMIR 182328- Oil K)



Name				Description
R000201073	RA2405109001	RIC23-06857	RIC23-06857	TMC REFERENCE OIL
R000201075	RA2405111001	RIC24-01436	RIC24-01436	RIC23-06857 Post CO2 Oil
R000201076	RA2405112001	RIC24-01437	RIC24-01437	RIC23-06857 Post CO2 Emulsion
			-02008	RIC23-06857 Post CO2 Emulsion Wash

Afton FTIR Set 2: 2.1 – 2.4 (CMIR 182329- Oil F)



Name			Description	
R000201074	RA2405110001	RIC23-06858	RIC23-06858	TMC REFERENCE OIL
R000201077	RA2405113001	RIC24-01438	RIC24-01438	RIC23-06858 Post CO2 Oil
R000201078	RA2405114001	RIC24-01439	RIC24-01439	RIC23-06858 Post CO2 Emulsion
[illegible]	[illegible]	[illegible]	[illegible]-02009	RIC23-06858 Post CO2 Emulsion Wash

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

- Group to let Yongli know on direction for EOGT
- Group to review existing data and share any comments for next meeting
- Ford (Rob) to get back on filter IR method details.
- Group to let Yongli know if can help with EOFT and EOWT method review and revision by April 3.
- TMC to confirm burette tip opening with labs.
- Root Cause subgroup will meet and report back to group on fishbone diagram.
- ISP to see if willing to run another EOGT run with dispersing tool on the 2 performance oils to confirm their first run's results. If possible, to provide timing estimate.
- Look into water analysis. –Angela? Group?
- Eugene (Afton) to look into FTIR peak in ISP data around 2300 wavenumber to see what peak may be.

- Next Meeting: Monday April 15 at 8:30AM CDT

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)