ASTM Engine Oil Gelation Test (EOGT) WK86363 Update

EOFT and EOWTT Surveillance Panel Meeting

August 19, 2024

Yong-Li McFarland, Chair

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EOFT and EOWTT Surveillance Panel Membership

21 members

Beth Schwab, Afton Chemical
Michael Kunselman, Center for Quality Assurance
Robert Stockwell, Chevron Oronite
Quanchang Li, ExxonMobil
Michael Deegan, Ford
Melissa Chu, Infineum
Angela Willis, 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: 3 drafts (large volume (600g), small volume (200 g), and Afton method) uploaded on ASTM Collaboration Area
  - 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 8-19-24

- Root Cause Subgroup update (Afton method update, water type update, Cold Soak tests update)
- ISP rerun of performance oils estimate to complete by August 16, please confirm lab running Oils F and K before starting
- New Afton EOGT modified procedure
- 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 CO2) (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)

MEASURING THE GELATION PROPENSITY OF ENGINE OILS – AFTON TECHNIQUE
8/14/2024

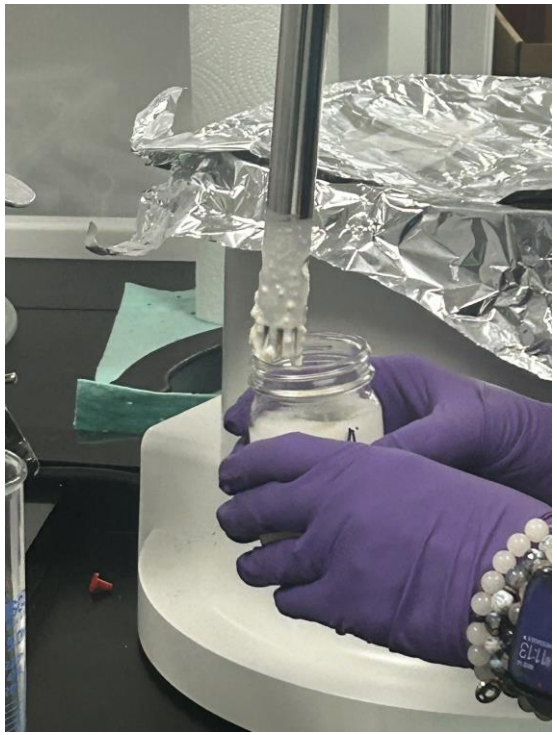
AFTON TECHNIQUE SCOPE

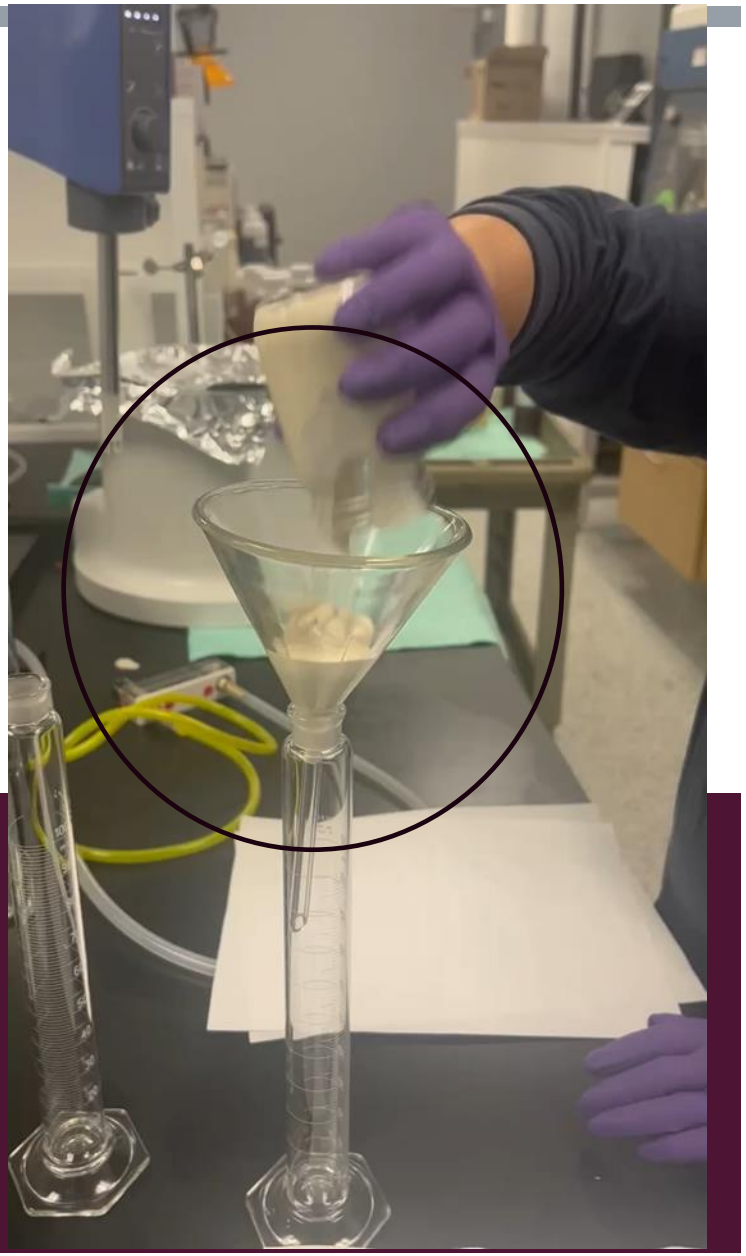
- Method covers the determination of the tendency of an oil to form a precipitate that causes an abrupt viscosity increase due to gelation. It simulates a problem that may be encountered after multiple cold starts or short drive times, resulting in a buildup of water contamination in the oil. After long storage periods, gelation of the oil/water/precipitate mixture can be observed.

PROCEDURE

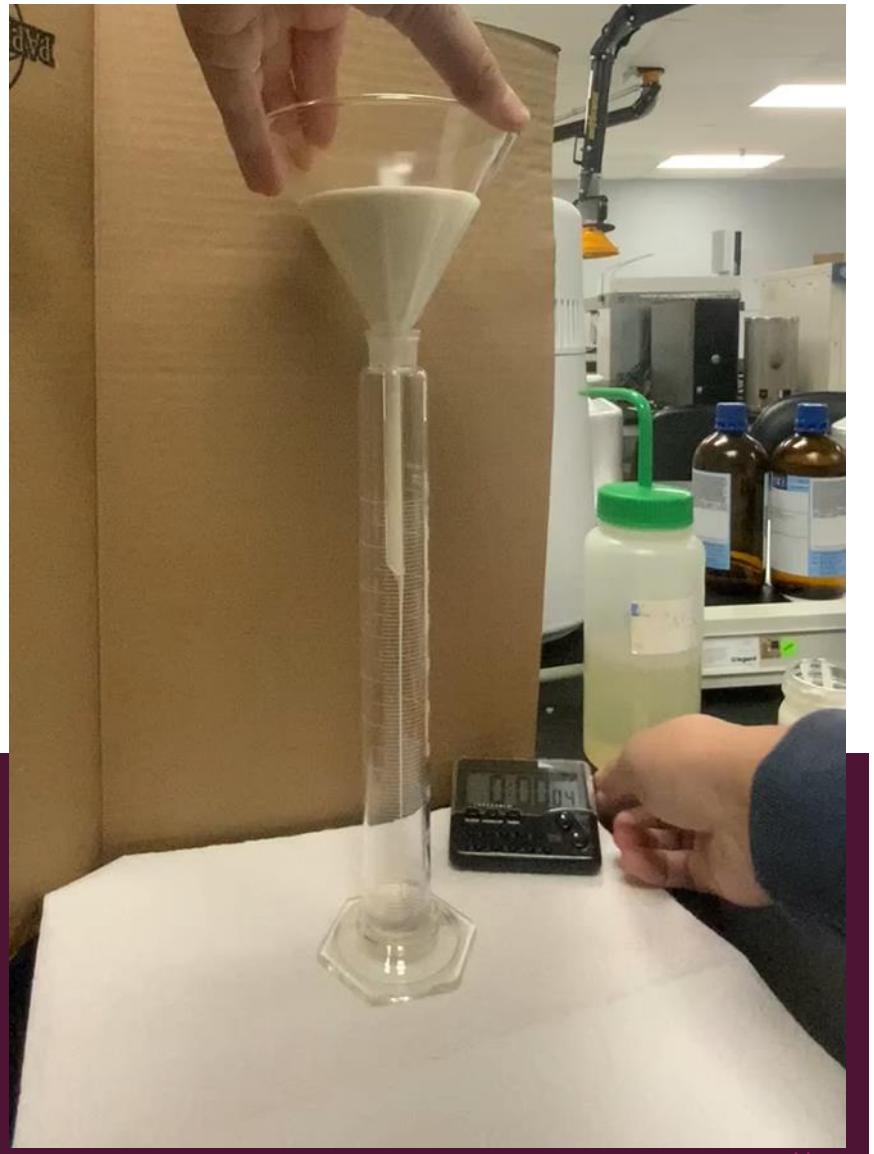
- 8.1 Take samples in accordance with the instructions in Practice D4057.
- 9 Preparation of the Acetic Acid Solution
- 9.1 Transfer 1 L of deionized water to a glass container and add glacial acetic acid under mixing until a pH of (3.5 ± 0.2) is reached.
- 9.2 After 24 hours, test the pH of the water using a digital pH probe and ensure the pH is still (3.5 ± 0.2). If the pH is too high, add additional glacial acetic acid. If the pH is too low, dilute with deionized water until the appropriate pH is reached.
- 10 Preparation of the Test Oil Sample
- 10.1 Add (40 ± 0.1) g of the new oil to a 120 mL glass container with a lid.
- 10.2 Add (12 ± 0.1) g of the acetic acid solution to the oil with an appropriate stir bar.
- 10.3 Mix the test oil for at (400 RPM) for 1 hour at (45 ± 5)°C. Close the glass container and store at room temperature in the dark for (3 ± 0.25) days.
- 10.4 After the storage period, add (16 ± 0.1) g of the acetic acid solution to the test oil sample.
- 11 Procedure
- 11.1 Propagation of the gel structure through shear mixing
- 11.1.1 Submerge the shear mixer approximately halfway down the fluid volume and turn the shear mixer to 7,000 RPM. Manually swirl the test oil container slowly in a circular motion for 30 secs.
- 11.1.2 Increase the shear mixing speed to 16,000 RPM and continue to swirl the container slowly in a circular motion for 90 secs.
- 11.2 Determine the fluid flow rate
- 11.2.1 Place the glass funnel atop the graduated cylinder opening.
- 11.2.2 Quickly pour in the entire volume of the test oil into the funnel and start the timer once the first drop has dispensed from the funnel stem.
- 11.2.3 Record the volume of test oil in the graduated cylinder every 15 secs for 180 secs.

11 AM SAMPLE



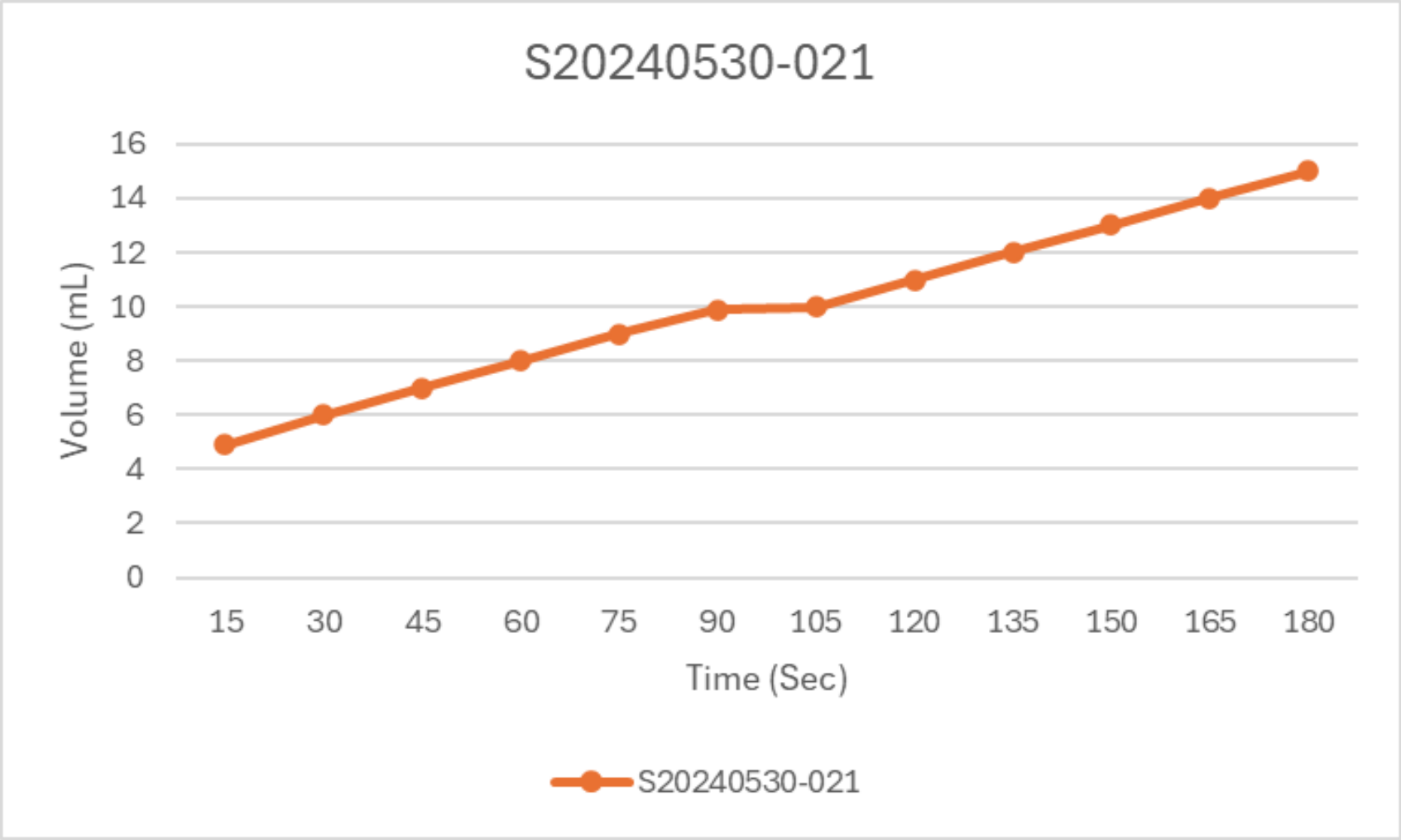


11 AM SAMPLE (HEATING DIFFERENT WITH FLASKS)

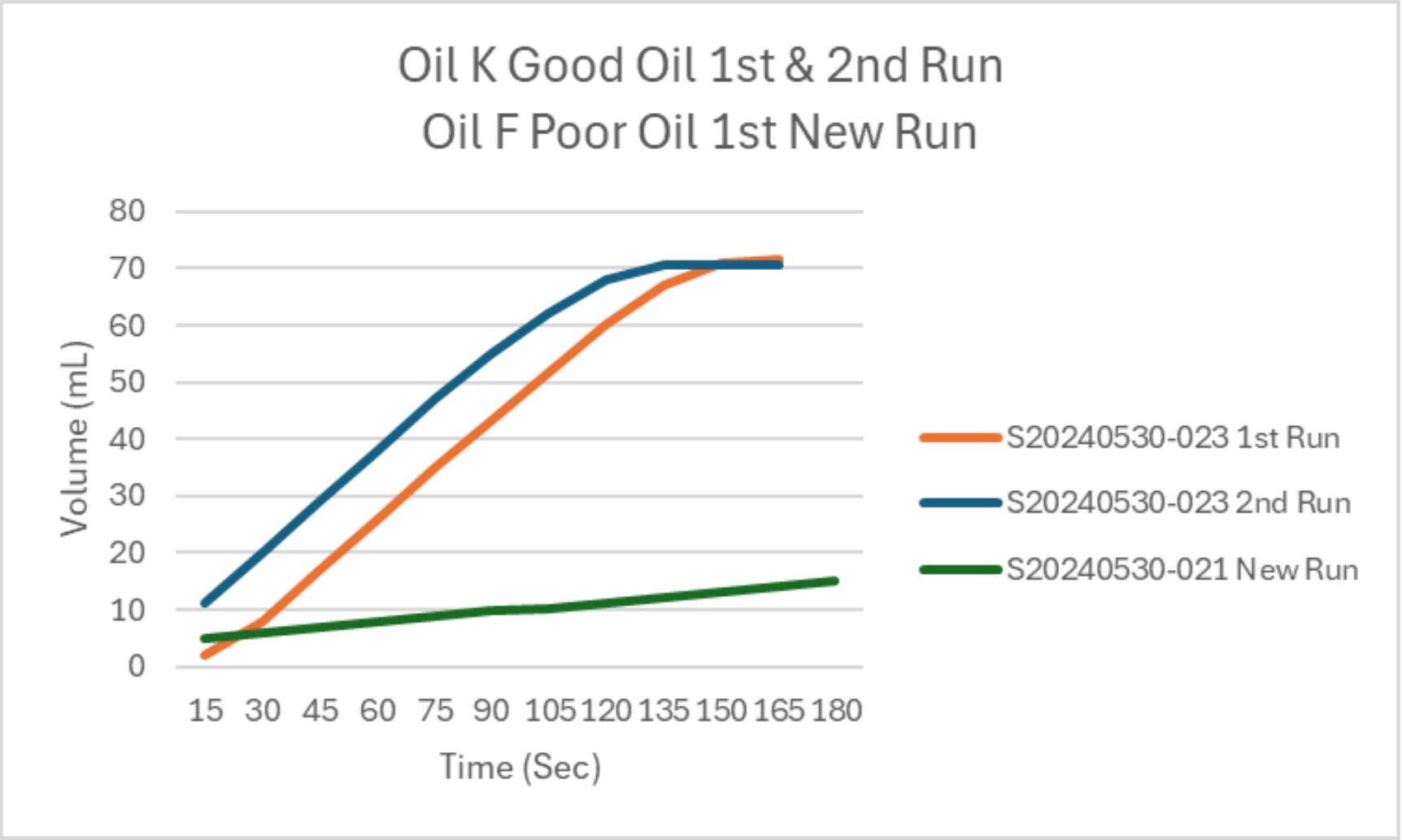


12 NOON SAMPLE – HEATING WITH FINAL FLASK ONLY. DID NOT FAIL, TOO FAST TO HEAT.

Poor Sample - Test 11 am

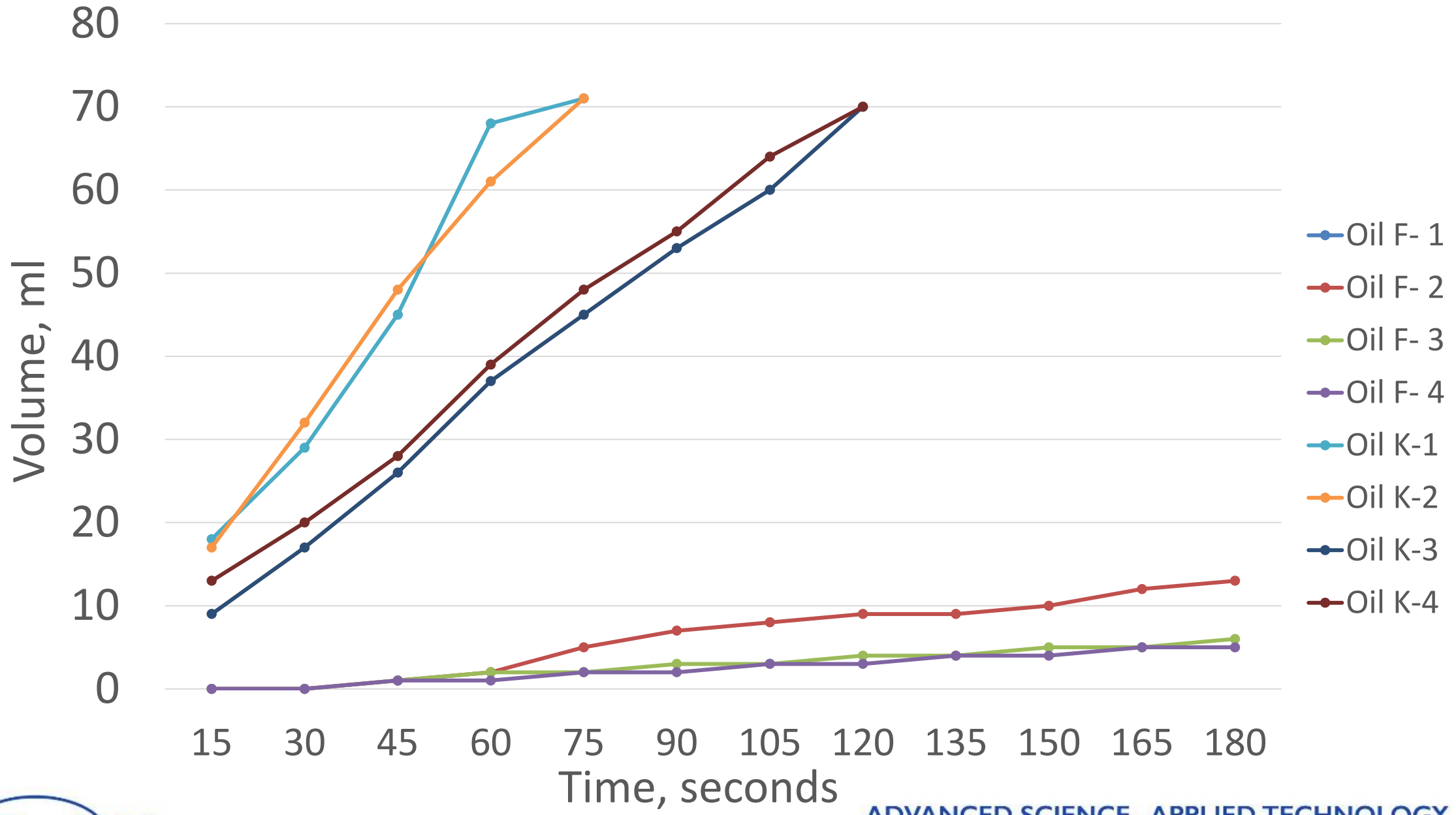


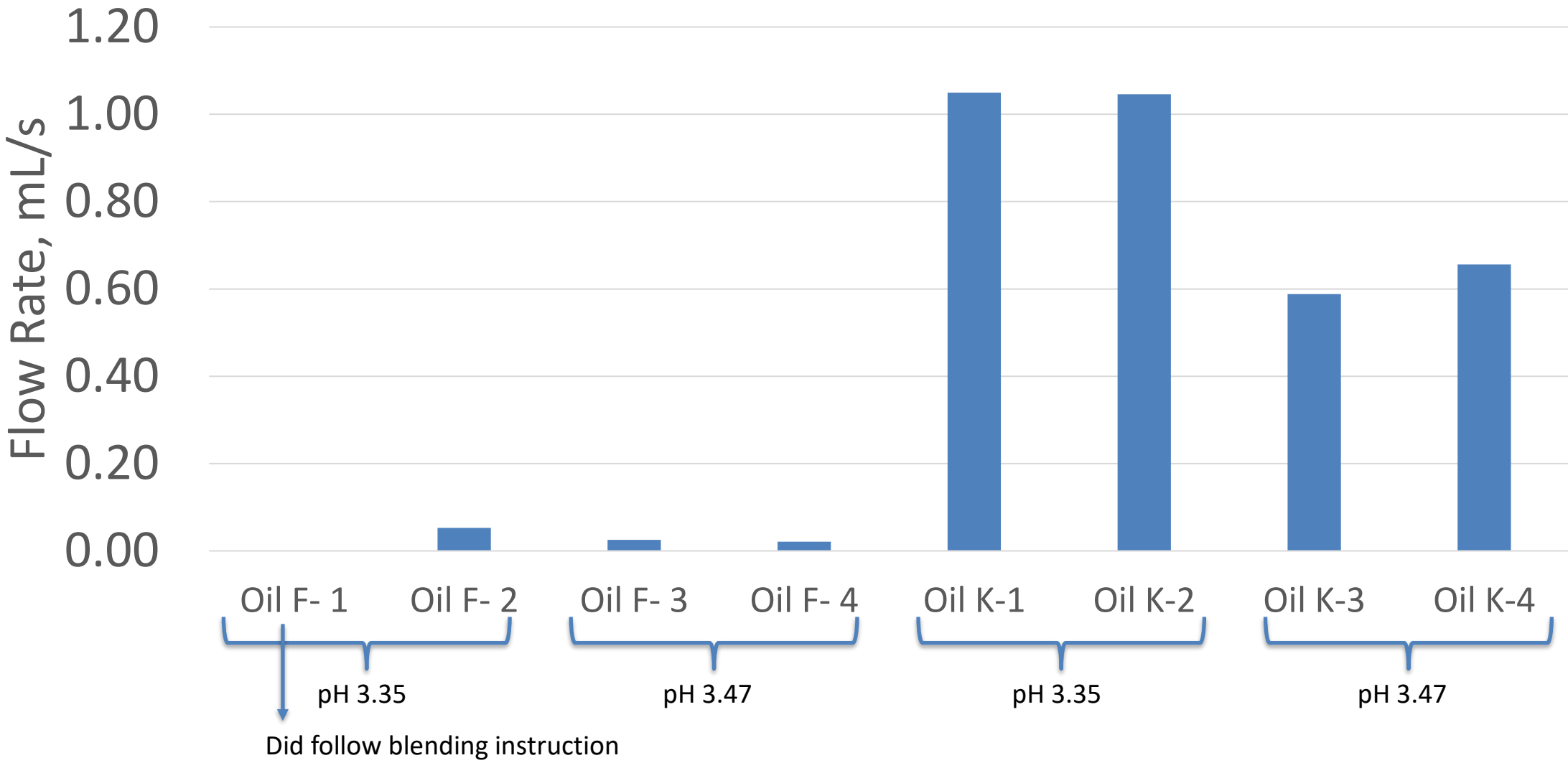
Good and Poor Samples

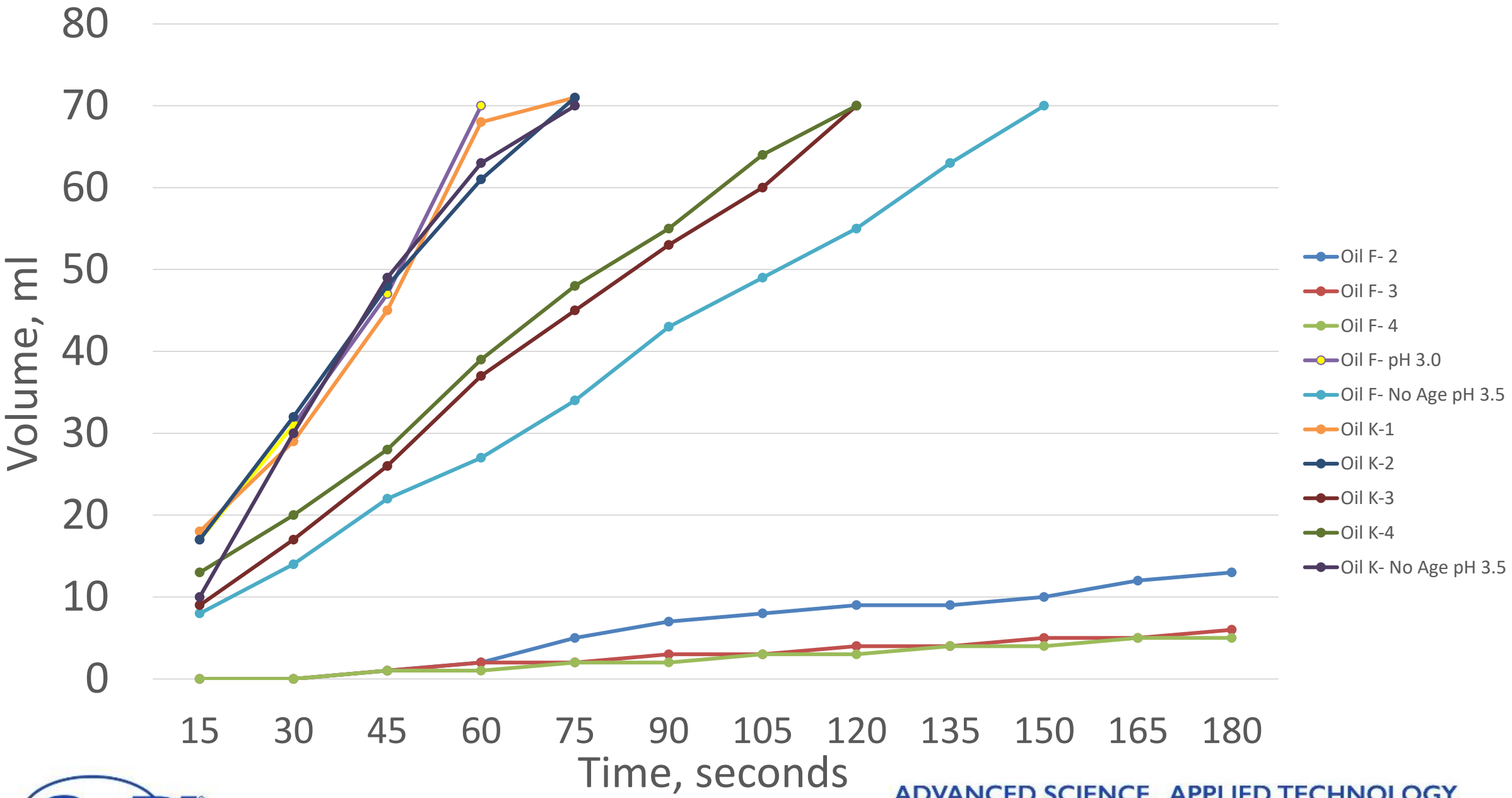


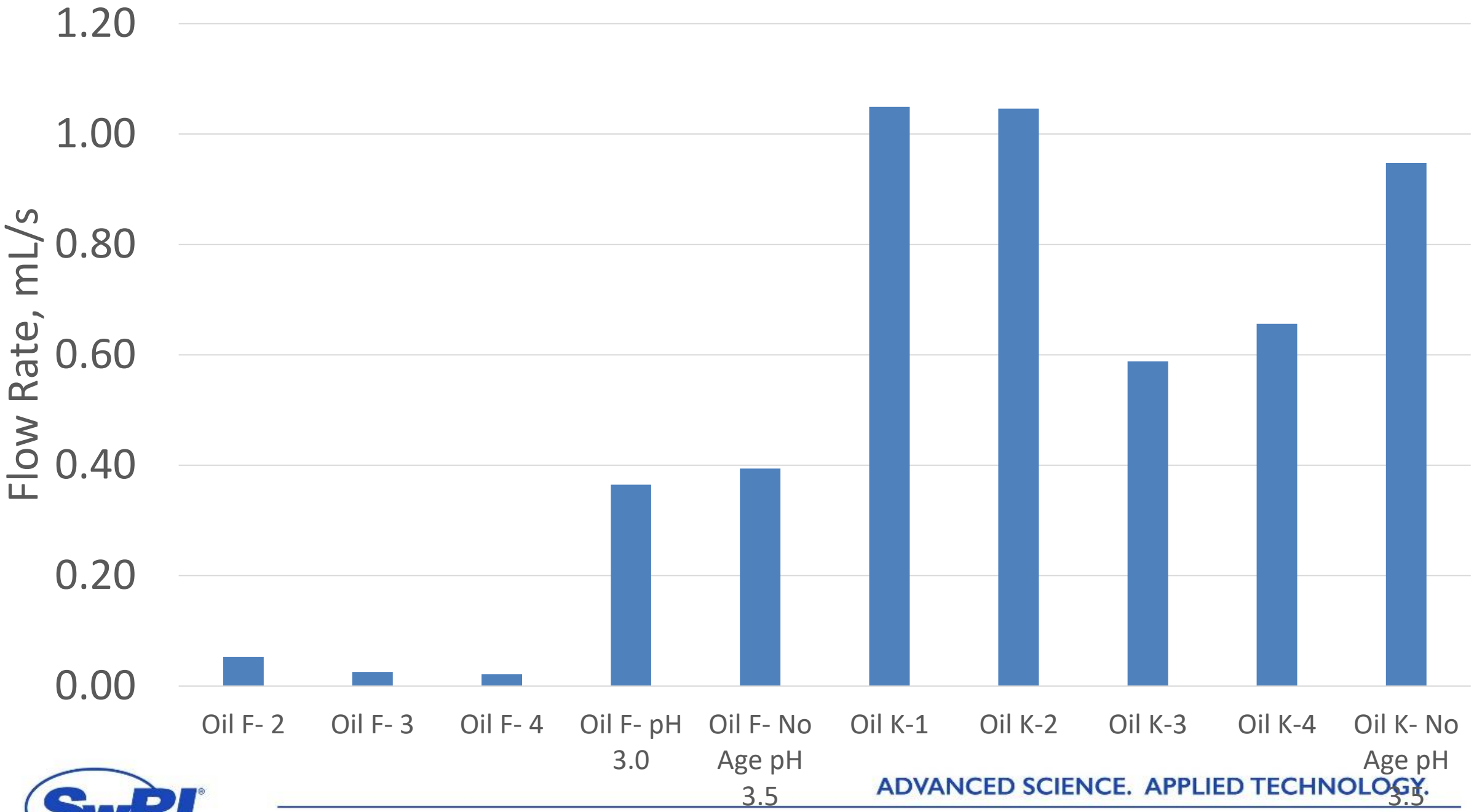
SwRI Results on Afton Method

Jared Cavaliere
August 16, 2024



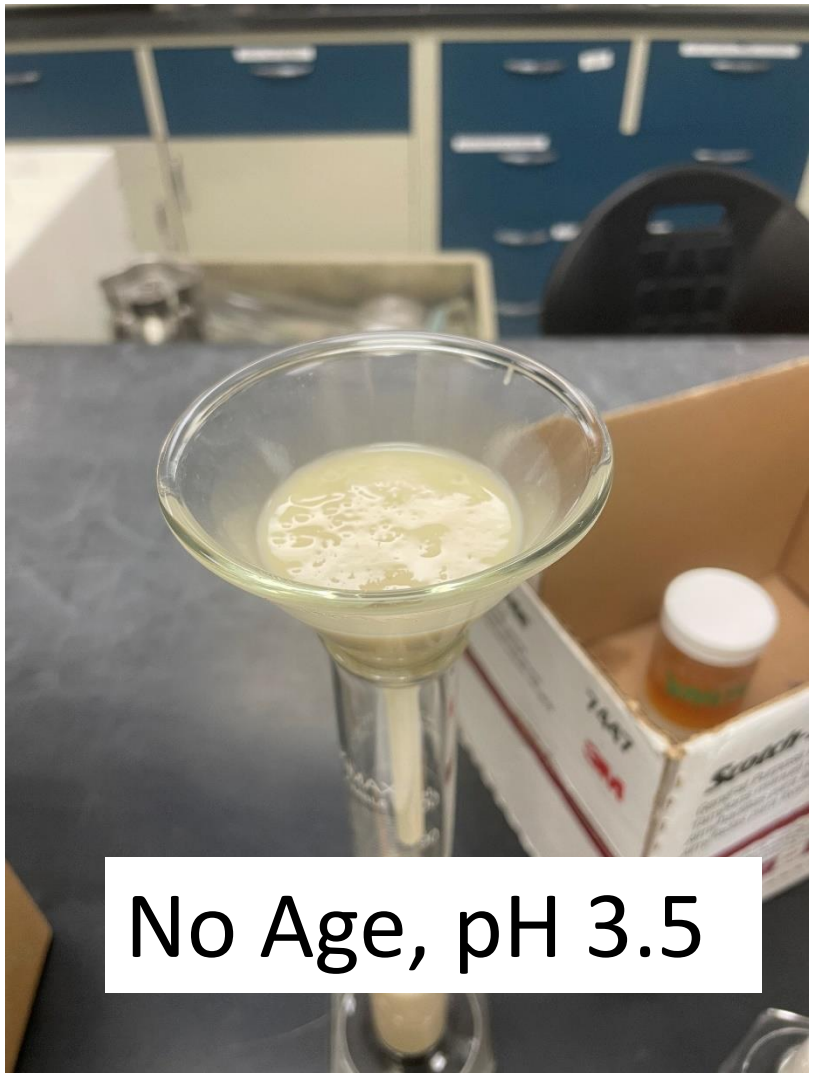


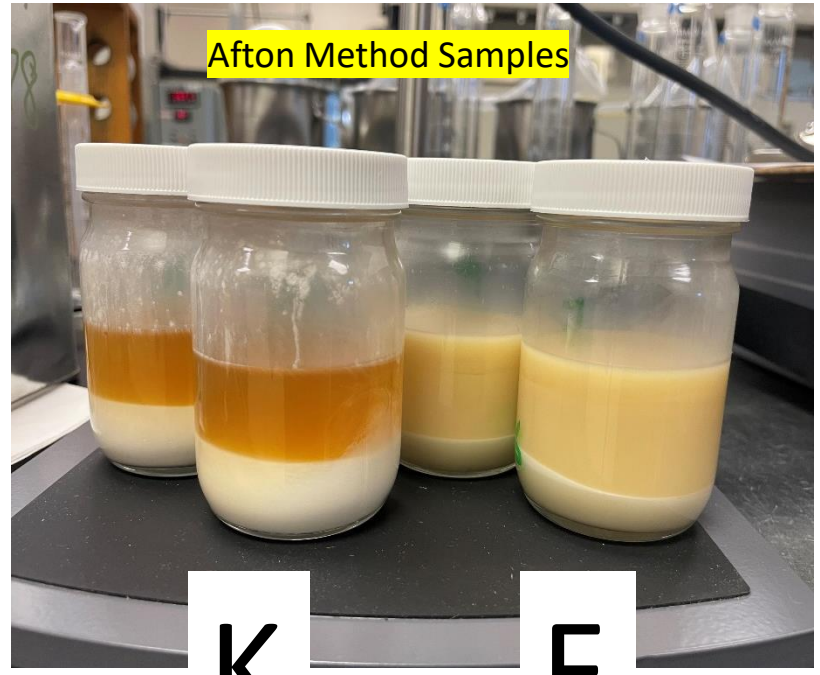




Observations

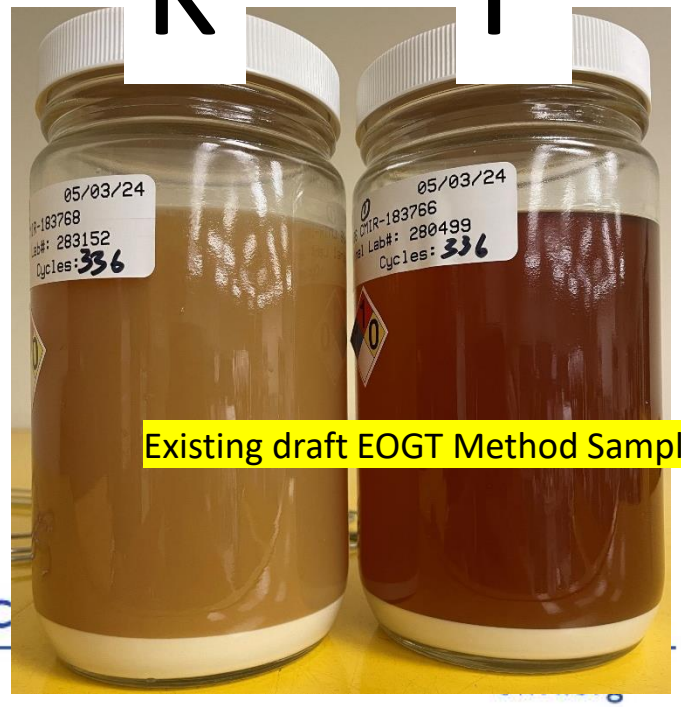
- pH dependent
  - 3.5 is the magic number
  - Suggest checking pH before each use
- Shear/blending technique dependent
  - Constant movement
  - Do not keep stationary as heat is quickly generated.
- No aging still shows differentiation





K

F



Afton Method Test Results

- Savant and SwRI, and probably Afton will run 1 day storage test with target temperature of 40C +/- 5C, check pH using pH meter, run in duplicate, mix at 15,000 rpm, and report the volume, in mL, at 90 seconds for run 1 and 2 and the average.
- Group to consider what test would report: average volume at 90 sec (of 2 runs), flow rate, or something else? Go forward with avg volume at 90 sec result for now, can change if needed.
- Other labs checking on equipment needs (Intertek and Valvoline). ISP thoughts?
- Propose to move to ILS with edits after next meeting

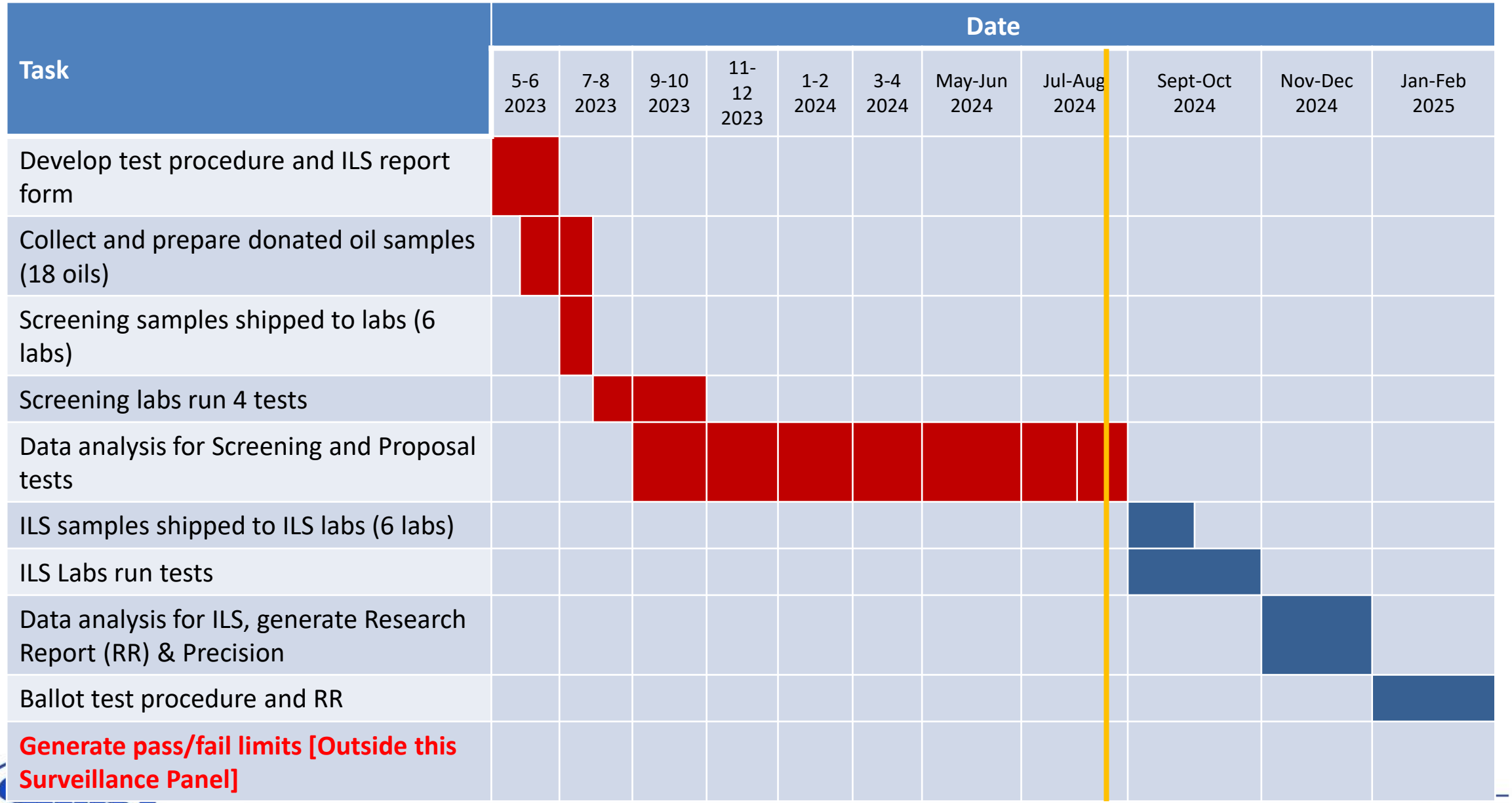
Savant Cold Storage Tests

Sample IDs		Time Inteval	ROOM TEMP Percent Change			0C Percent Change		
		Week	Run 1	Run 2	Average	Run 1	Run 2	Average
CMIR 183759	S20231005-017 (Oil F)	Week 1	-12.29	-9.57	-10.93	-7.40	-8.05	-7.73
CMIR 183761	S20231005-019 (Oil K)		-9.40	-6.50	-7.95	-8.96	-5.37	-7.17
CMIR 183759	S20231005-017 (Oil F)	Week 2	-17.61	-15.85	-16.73	-6.47	-9.96	-8.22
CMIR 183761	S20231005-019 (Oil K)		-7.65	-6.86	-7.26	-8.29	-7.61	-7.95
CMIR 183759	S20231005-017 (Oil F)	Week 4	-15.62	-1.78	-8.70	1	-6.85	-2.93
CMIR 183761	S20231005-019 (Oil K)		-6.69	-11.67	-9.18	-13.25	-5.32	-9.29
CMIR 183759	S20231005-017 (Oil F)	Week 8	-36.75	-49.94	-43.35	-17.18	-27.9	-22.54
CMIR 183761	S20231005-019 (Oil K)		-20.69	-16.32	-18.51	-16.43	-6.64	-11.54
CMIR 183759	S20231005-017 (Oil F)	Week 9 (Aug 6)	-50.52	-57.92	-54.22	-16.32	-34.58	-25.45
CMIR 183761	S20231005-019 (Oil K)		-21.23	-21.8	-21.52	-13.25	-3.78	-8.52

Our cold and room temperature storage continues to show discrimination at 9 weeks

Notes 8-19-24: Ask Savant to verify pH of EOT samples (9 week samples) using pH meter, after separating water from oil phase.

Draft Timeline – updated August 19



EOFT D6795 and EOWT D6794 Method Review cont.

- 3. Consider rewording for which side to test on filter. The method says "filter smooth side up". It isn't always easy to see which side is "smooth".
  - Current wording: *Section 10.1: Assemble apparatus as shown in Fig.1 with filter installed in proper orientation (25 µm smooth side up).*
  - Request labs to respond to TMC on how they determine smooth side up. If no comment, group would then remove words: "*in proper orientation (25 µm smooth side up).*
  - *Notes 8-19-24: No additional lab feedback, move forward with proposed changes to the 2 methods.*

- WK91396 opened to revise D6795
- WK91397 opened to revise D6794

Action Items and Next Meeting

- Root Cause group to consider if pH was root cause for lack of differentiation in original EOGT method; ask labs to run pH on EOT sample (ISP, SwRI, Afton, Savant)
- SwRI, Savant, and Afton to report on their Afton method for 1 day storage.
- YM to draft ballot for changes to EOFT and EOWTT.
- YM to check with Intertek on additional EOT sample analyses
- Group ready to move onto ILS pending next meeting's results?
- Include acetic acid vs carbonic acid chemical reaction slide
- Sample size: 120 mL? Jacob Z to let us know if adequate
- Request labs to let John know how much volume remaining of Oil F and K
- Ask Savant to verify pH of EOT samples (9 week samples) using pH meter, after separating water from oil phase
- Next meeting to discuss how to include running oils F and K runs in ILS, also to discuss with Root Cause group.

- Next Meeting: Wednesday Sept 4 at 9:00 AM CDT for 1 hr

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 Intertek ISP Savant SwRI TMC (monitoring system only) Valvoline