ASTM New Engine Oil Gelation Test (EOGT) WK86363 Update

EOFT and EOWTT Surveillance Panel Meeting

July 15, 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
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

EOFT D6795 and EOWT D6794 Method Review

- ASTM D6795 is up for 5-year review in 2024, ASTM D6794 is up for review in 2025
- Group met and have the following changes for D6794 and D6795:

1. Terminology section update, add D4175 to Section 2 Referenced Documents and wording to Section 3 Terminology.
   - Add to Section 2.1 D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants
   - Add to Section 3 Terminology, 3.1 Definitions: "3.1.1 For definitions of terms used in this standard, refer to Terminology D4175." Renumber.

2. Change order of Timer and Container in Section 6.

Current Wording:
6.2 *Blender,* capable of 18 000 r/min ±10 % without the container.
6.3 *Timer,* capable of timing 30 s ± 1 s.
6.4 *Container,* 250 mL, with blade compatible with the blender.

Proposed Wording:
6.2 *Blender,* capable of 18 000 r/min ±10 % without the container.
6.3 *Container,* 250 mL, with blade compatible with the blender.
6.4 *Timer,* capable of timing 30 s ± 1 s.

Note: Dispersing type tools, though readily available, were not part of the Precision study.

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. Group believes labs aren't able to tell which side should be up and would then remove words: "*installed in proper orientation (25 µm smooth side up).* Around 2000, change in filter paper made it difficult to determine smooth side.

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

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 7-12-24

- Root Cause Subgroup update (Cold Soak tests update and water type update)
- ISP rerun of performance oils estimate to complete by August 16
- 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)

Cold Soak Study

SOUTHWEST RESEARCH INSTITUTE®

SwRI Cold Temp Soak Previous 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

SwRI Cold Temp Soak Test Results		
Condition	**CMIR 183766 (Oil F)**	**CMIR 183768 (Oil K)**
7 days 20°C + 7 days 0°C	-10.17	-9.93
24 Hour Rotation 20°C + 0°C for 10 Days	-6.48	-9.77

If not seeing results, should additional tests continue or do modifications need to be attempted?

What was the difference from previous cold soak treatment?

Samples were homogenized before filtering and then placed into cold storage.
Could “seeding” be initiated by mixing of precipitate and further activated by cold?

Next Steps

-1wk+1wk samples continued with 1wk/1wk
-24Hr samples placed back into Cold storage for 1wk
-3wk/1wk homogenized before cold storage
-2wk/2wk homogenized before cold storage

SwRI Cold Temp Soak with Homogenization Test Results

Condition	CMIR 183766 (Oil F)	CMIR 183768 (Oil K)
7 days 20°C + 7 days 0°C + Homog/Filter + 7 days 20°C + 7 days 0°C	-20.22	-14.68
14 days 20°C + Homogenized + 14 days 0°C	-8.40	-7.42
21 days 20°C + Homogenized + 7 days 0°C	-9.08	-10.04
24 Hour Rotation 20°C + 0°C + Homog/Filter + 7 days 0°C	-14.28	-16.3

SwRI Cold Temp Soak Test Results

Condition	CMIR 183766 (Oil F)	CMIR 183768 (Oil K)
7 days 20°C + 7 days 0°C	-10.17	-9.93
7 days 20°C + 7 days 0°C + Homog/Filter + 7 days 20°C + 7 days 0°C	-20.22	-14.68
14 days 20°C + Homogenized + 14 days 0°C	-8.40	-7.42
21 days 20°C + Homogenized + 7 days 0°C	-9.08	-10.04
24 Hour Rotation 20°C + 0°C	-6.48	-9.77
24 Hour Rotation 20°C + 0°C + Homog/Filter + 7 days 0°C	-14.28	-16.3
* Ranges Observed to date	> 35%	No more than 18% in any experiment

Savant Test Results

Updated July 10, 2024

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

Test again July 30th -Week 8

Our cold and room temperature storage continues to show no discrimination between the two oils.

Notes 7-15-24: Could we control cooling ramp for storage with slower cooling ramp for more gelling?
Savant did run samples with D5133 cooling ramp. Root cause to review actions.
-Consider checking water content on 6 month retains and fresh runs.

EOGT Modified Procedure

Proposed Title: Measuring the Gelation Propensity of Engine Oils After Treatment with Acidified Water

07/12/2024

Passion for Solutions®

Introduction

- Existing methods have shown difficulty showing robust discrimination using below setup
  - Emulsify oil and water
  - Bubble CO2 at 50°C
  - Store at RT or cold storage
  - Homogenize and filter

- Afton tried more severe conditions
  - ~40% acidified water by mass
  - Mix 45°C for 1 hour
  - Store for 3 days
  - Shear mix to produce gel

- Main ideas
  - Destabilize detergent with acid
  - Simulate emulsion settling (time) with excess water
  - Apply shear to form gel network

General Procedure

1.

- 12g of acetic acid solution (pH 3.5)
- 40g Oil
- Stirred at 45°C for 1 hr

2.

- Storage at room temperature for 7 days

3.

- Additional 16g acetic acid solution is added

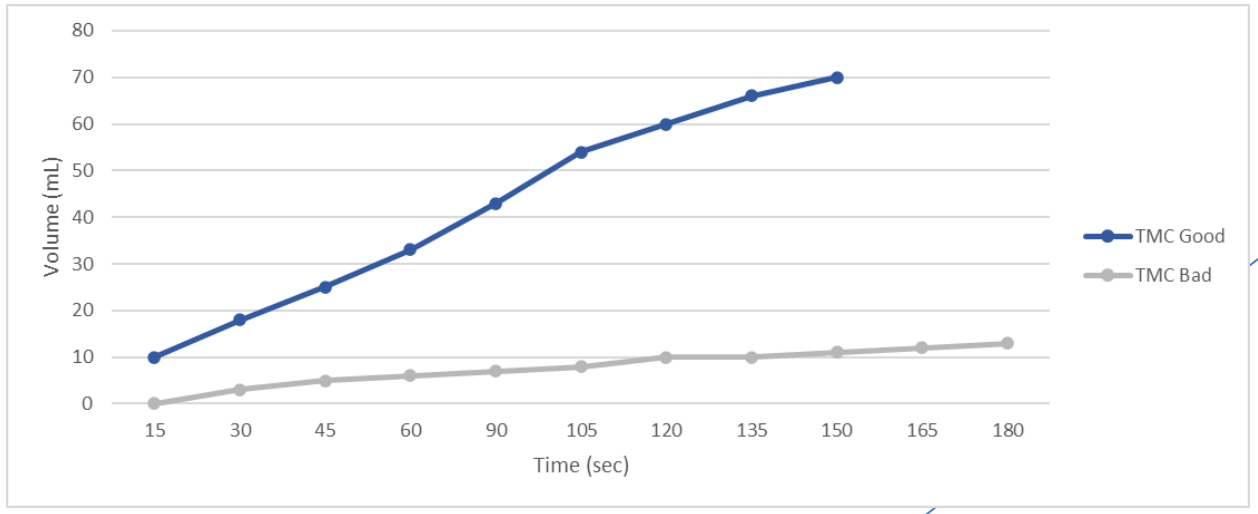
4.

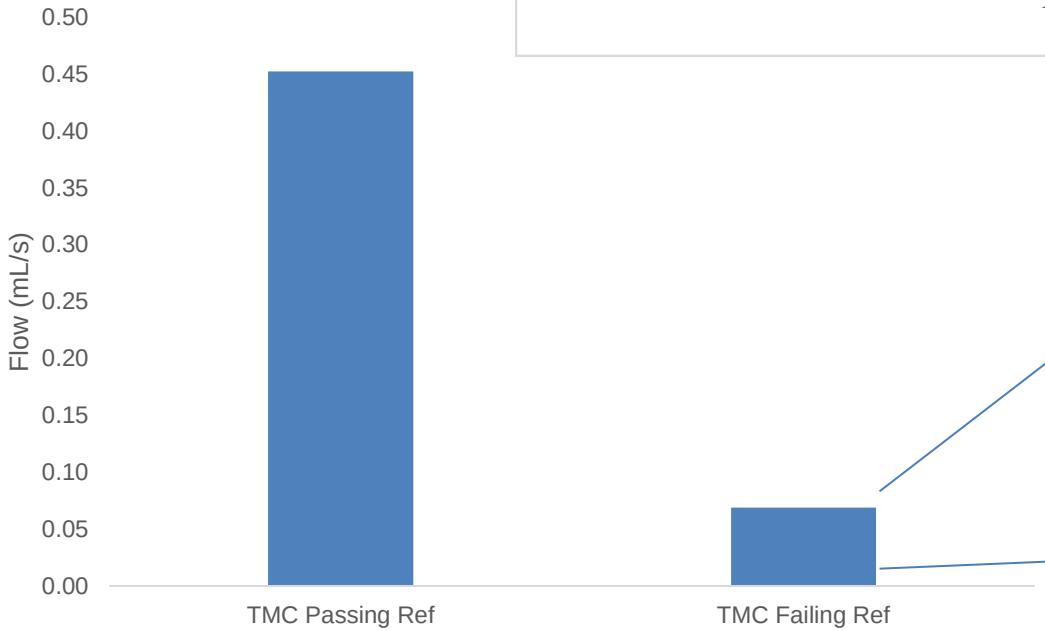
- Sample is shear mixed (7000 RPM 30 sec then 16000 RPM 90 sec)

5.

- **Flow rate** (mL/s) is measured

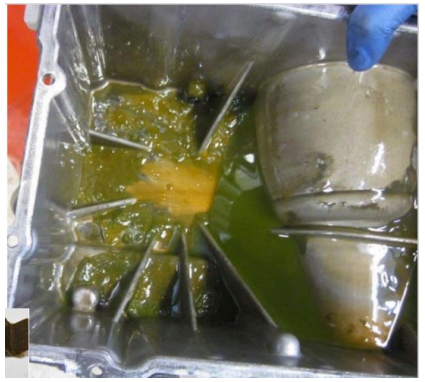
TMC Fluids Results





AftonChemical.com

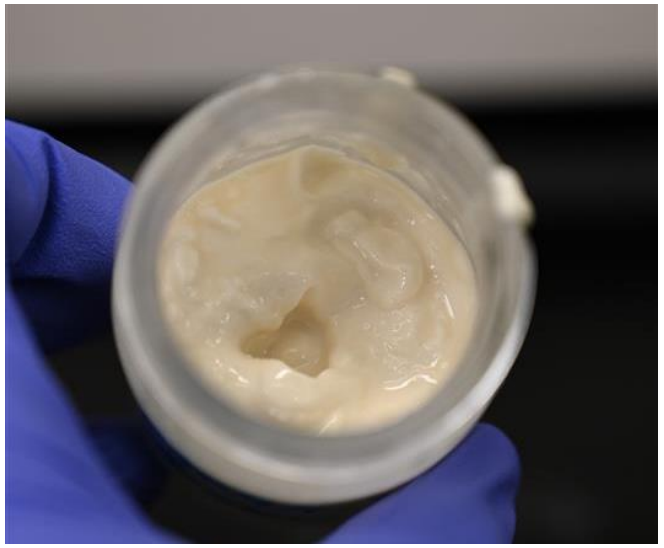
Pictures



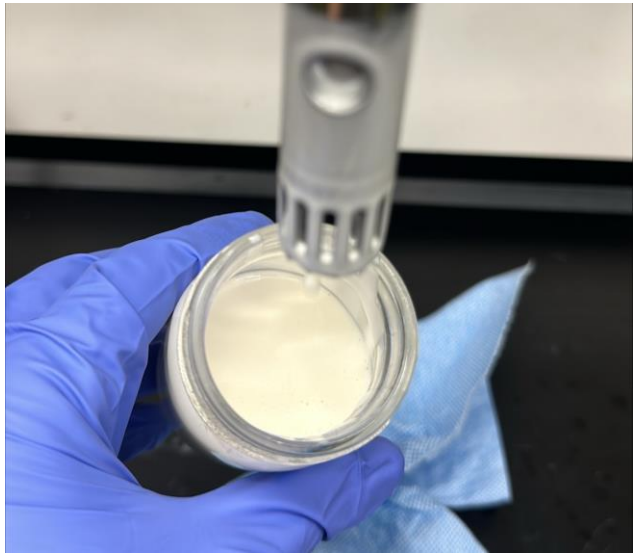
Initial Mass of water: 4%

~26% water in settled layer

Failing Ref



Passing Ref



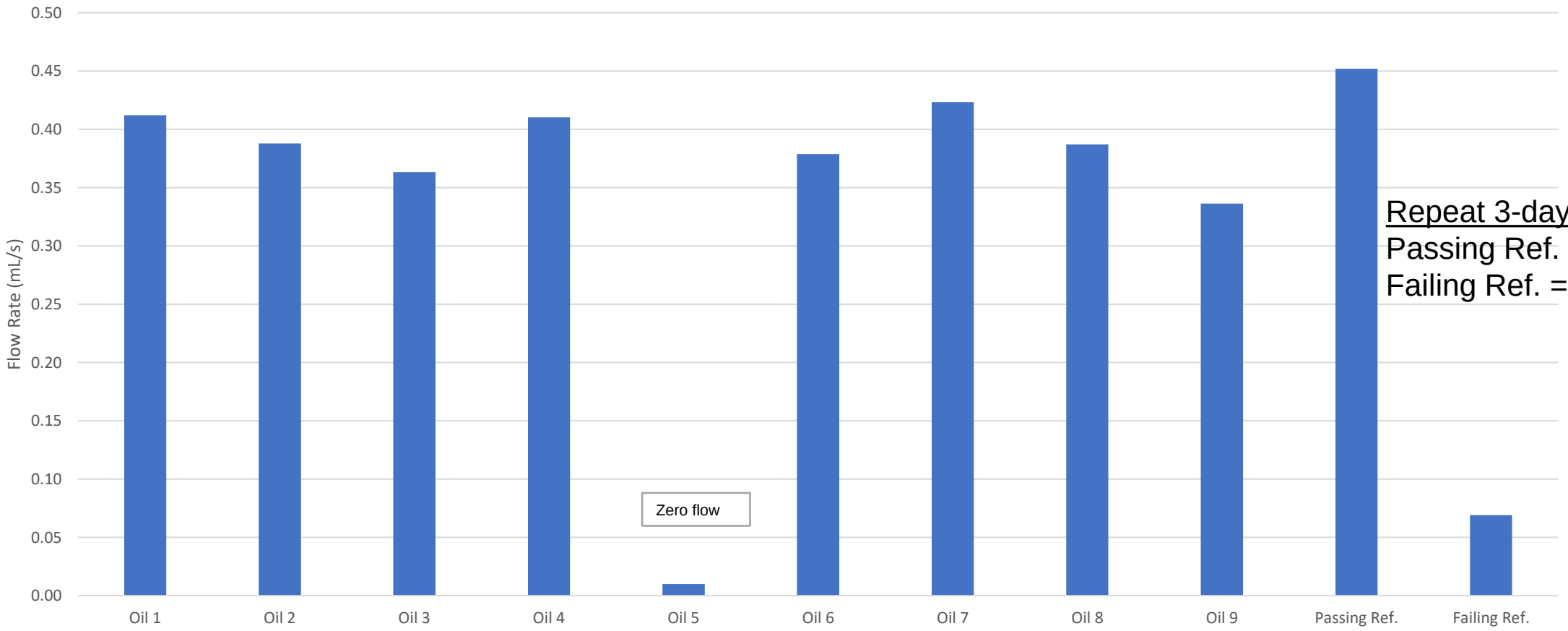
Passing Ref

Failing Ref

AftonChemical.com

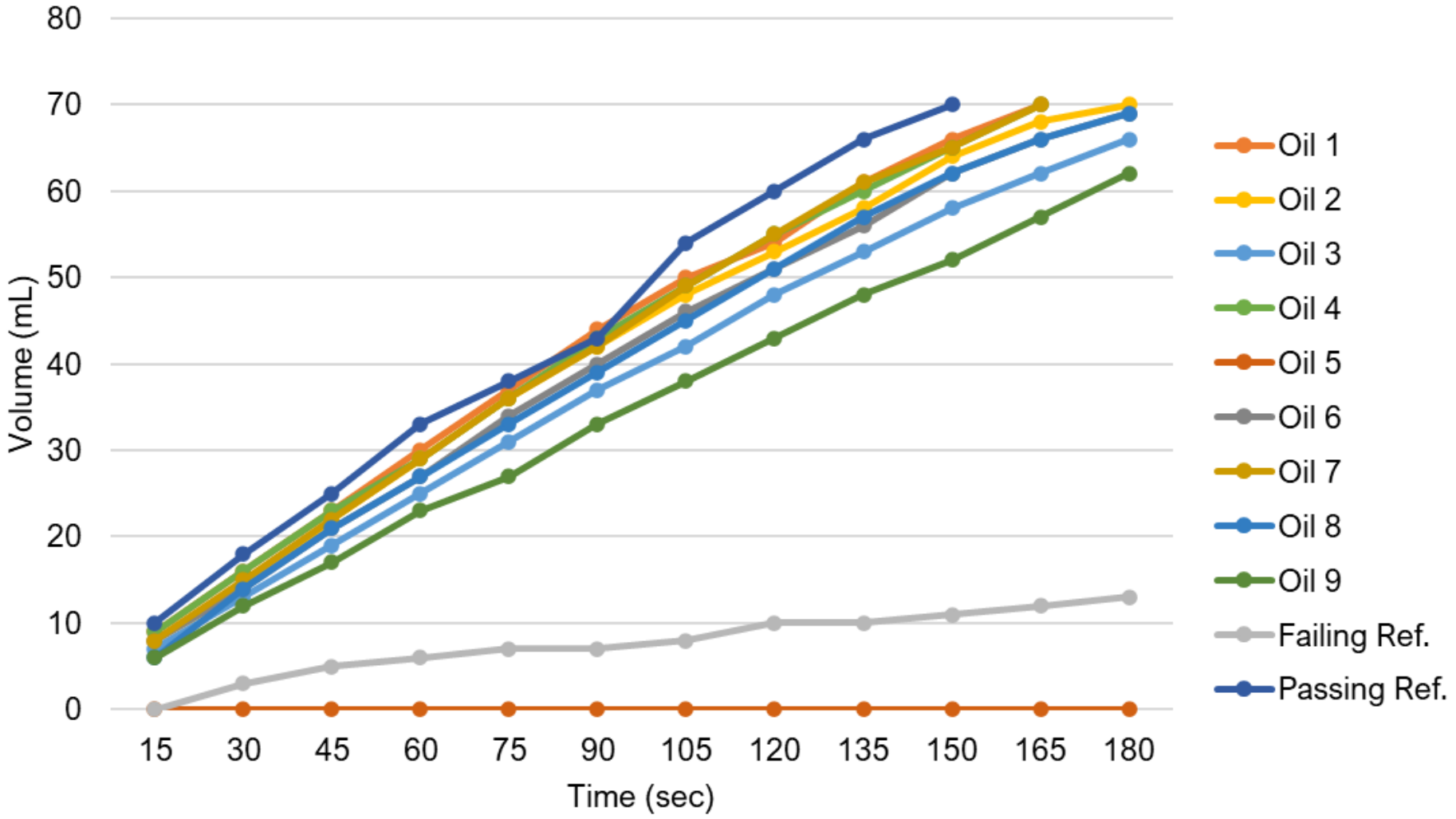
Afton CHEMICAL Passion for Solutions®

PCMO Gelation Results – Day 7 storage



Repeat 3-day storage
Passing Ref. = 0.6 mL/s
Failing Ref. = 0.06 mL/s

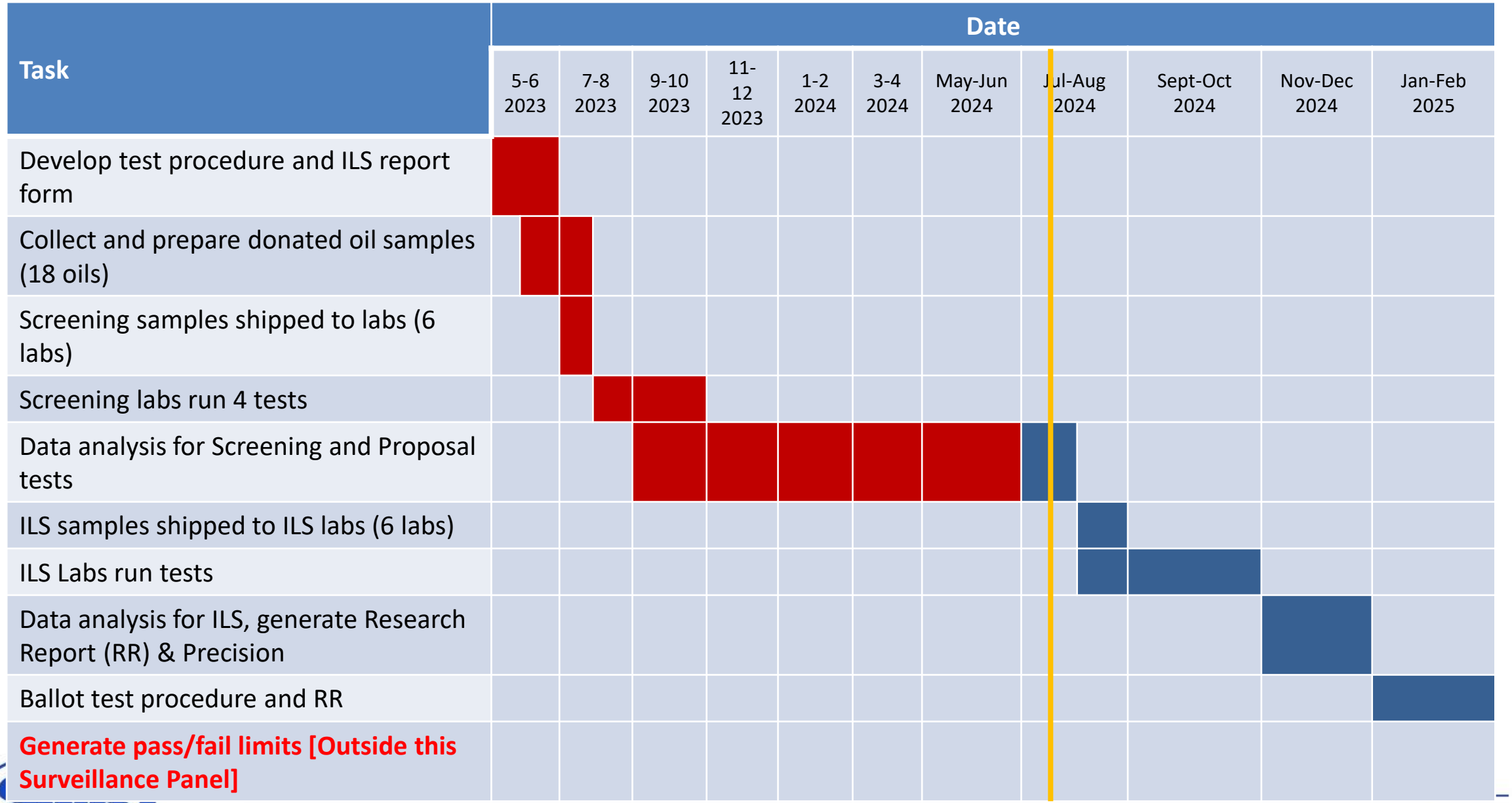
PCMO Gelation Results



Procedure Updates

- Recommend Acetic acid as proxy for carbonic acid (CO2 sequestered in engine)
  - Originally utilized carbonic acid by bubbling CO2 in water; determined pH was unstable over time
    - Each labs would need to make their own-introduces variability
    - pH rise due to low solubility of CO_2 in water
  - Acetic acid a better choice
    - Can be purchased as concentrate and diluted to proper pH
    - pH is stable over time
    - Drives same chemical mechanism – Amorphous $CaCO_3$ → Calcite

Draft Timeline – updated July 12



Action Items and Next Meeting

- Group to decide next steps for EOGT.
- SwRI, Savant, Afton (and any other labs) to report on their Afton EOGT modified procedure test results to Root Cause group.
- Labs to send TMC info on how to identify smooth side up filter.
- Please contact Afton or Yongli on questions for Afton EOGT modified procedure or test
- Ask if any lab with a retain willing to store in 0C or cold temp for 7 days and run filtration on sample and report out to group, please let Yongli know- Intertek possibly willing
- Labs to let TMC know on storage temperature (after blending and bubbling CO2) 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.

- Next Meeting: Monday August 5 at 9AM CDT for 1.5 hrs

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