ASTM Engine Oil Gelation Test (EOGT) WK86363 Update

EOFT and EOWTT Surveillance Panel Meeting

September 4, 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 November, and final method ballot in February 2025

Afton Method Test Results

- Savant latest results
- SwRI latest results:
  - Focus on shearing technique. Another tech tried shearing Oil F, it partially gelled. Following written method, Oil F did not gel. Using the moving motion for shearing, Oil F did gel. Incorporate air/top layer into shearing step helps create gel. Currently out of fresh Oil F and K. Will request more oil F and K to confirm another tech can also create gel on Oil F and no gel on Oil K.
  - TBN D4739 Buffer end point Results: EOT Oil K: 3.44 mg KOH/g; EOT Oil F (did gel): 2.71 mg KOH/g
- Afton latest results: taking note on the importance of temperature and shearing method currently. Will provide more info on TBN and possibly pH on previous samples. Will review and help better document in method the specific measurements or conditions needed in method. Jacob to send out latest draft of method.
- ISP results: did not see any gel form on Oil F. Will try different shearing techniques on Oil F and take TBN measurements of fresh and EOT samples.
- Go forward with avg volume at 90 sec result for now, can change if needed.
- Other labs checking on equipment needs (Intertek and Valvoline): Intertek is waiting for materials to arrive.
- Propose to move to ILS with edits after next meeting?

Savant Labs Data for Afton Method - Aug 30

Test #	Sample (oil F or K or CMIR#)	Technician (who ran)	Did sample gel? (Yes or no)	Storage Time (24 hrs or 72 hrs or ?)	Any other comments
SAV 1	Oil F	MB	Yes	24	
SAV 2	Oil F	MB	No	24	
SAV 3	Oil F	GM	No	24	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.
SAV 4	Oil F	CP	Yes	24	-After addition of 2nd aliquot of acetic acid, hand stir the sample with glass rod for 5 seconds (or try mixing with magnetic stir bar). -Submerge the shear mixer to the bottom of the fluid volume and turn the shear mixer to 7,000 RPM. Move the oil container slowly in a circular motion for 30 secs. -Ramp to increase the shear mixing speed to 14,000 RPM and continue to swirl the test oil container slowly in a circular motion for 30 secs. -Hold the shear mixing speed at 14,000 RPM and continue to swirl the test oil container slowly in a circular motion and from top to bottom of the sample for 30 secs
SAV 5	Oil F	GM	Yes	24	
SAV 6	Oil F	CP	Yes	24	
SAV 7	Oil F	CP	Yes	24	
SAV 8	Oil K	CP	No	24	

Notes: Savant will be repeating SAV 7 with another tech. Will have 4 more tests coming on oils F and K. Plan to take TBN (D4739) of fresh and EOT oils F and K.

Acetic Acid vs CO_2

Le Chatelier's principle:

If a dynamic equilibrium is disturbed by changing the conditions, the position of equilibrium shifts to counteract the change to reestablish an equilibrium.

Similar mechanism but different rate

Acetic Acid Detergent Dissolution Pathway

- $CaCO_3(s) + 2CH_3COOH \leftrightarrow Ca(CH_3COO)_2(aq) + H_2O(l) + \boldsymbol{CO_2(g)}$
- $Ca(CH_3COO)_2(aq) \leftrightarrow Ca^{2+}(aq) + 2CH3COO^-(aq)$

Carbonic Acid Detergent Dissolution Pathway

- $CO_2(g) + H_2O(l) \leftrightarrow H_2CO_3(aq)$
- $H_2CO_3(aq) \leftrightarrow \boldsymbol{HCO_3^-}(aq) + H^+(aq)$
- $HCO_3^-(aq) \leftrightarrow CO_3^{2-}(aq) + H^+(aq)$

- $CaCO_3(s) + H_2CO_3(aq) \leftrightarrow Ca(HCO_3)_2(aq)$
- $Ca(HCO_3)_2 \leftrightarrow Ca^{2+}(aq) + \boldsymbol{2HCO_3^-}(aq)$

Carbonic acid inhibitory effects

1. Common ion effect: Both dissociation and neutralization produce HCO_3^-
2. No CO_2 product to drive reaction forward
3. Lower pKa: At equal pH, more bicarbonate ions will be present (see point 1)

Updates 9-4-24

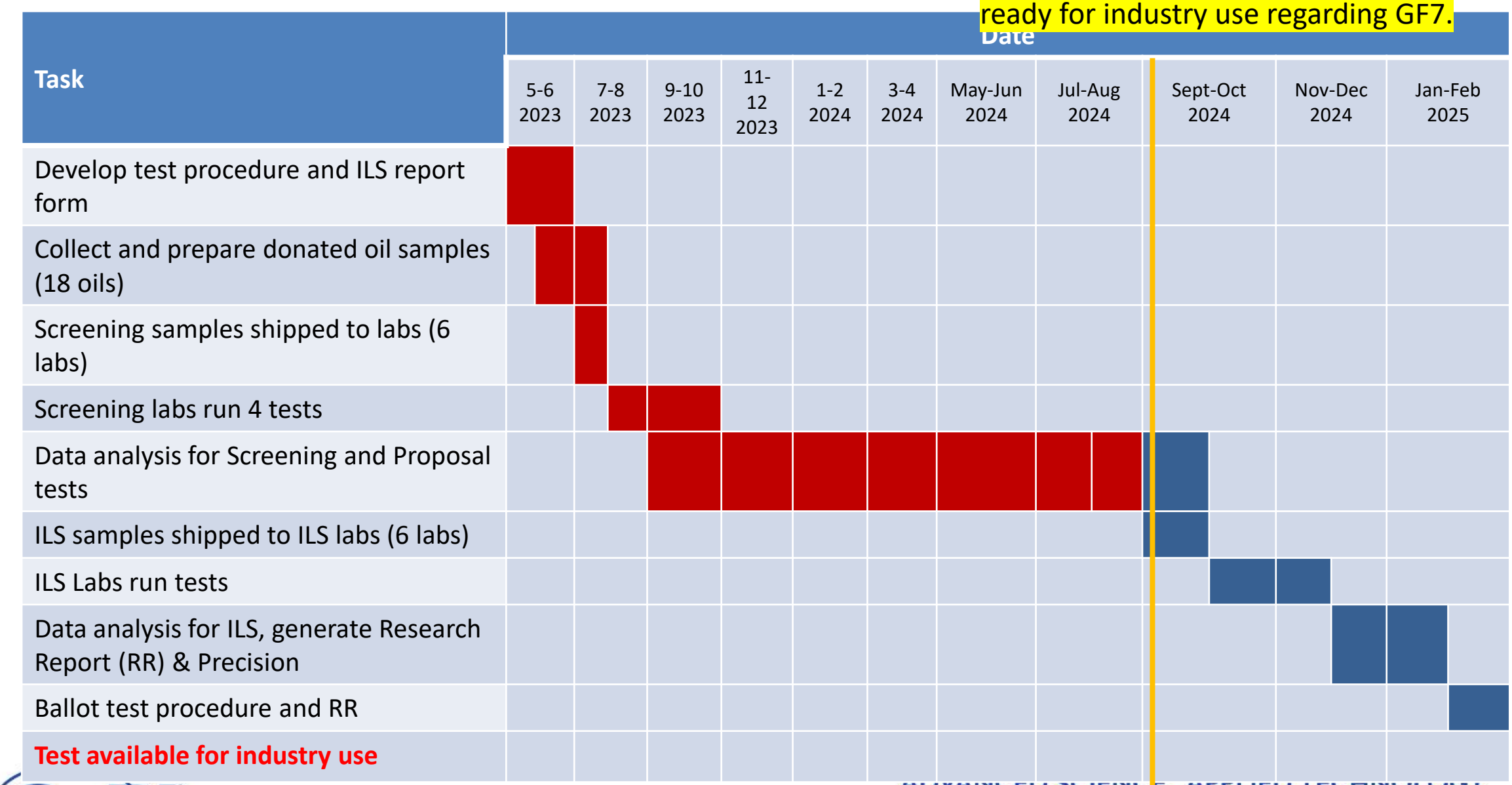
- Root Cause Subgroup update (Afton method update, water type update)
- ISP completed rerun of performance oils with original EOGT method. Will take TBN on fresh and EOT samples from this test. TBN info may help root cause group to determine reason for lack of discrimination in this method.

Recent Test	Change in Filterability (CFA, %)		
CMIR	Run 1	Run 2	Average
185167 (Oil F)	-26.83	-28.60	-27.70
185168 (Oil K)	-8.08	-10.12	-9.11

Previous Tests	Change in Filterability (CFA, %)		
CMIR	Run 1	Run 2	Average
182165 + 182166 (Oil F)	-35.77	-39.26	-37.52
182320 (Oil F)	-42.98	-42.40	-42.69
182163 + 182164 (Oil K)	-9.57	-6.24	-7.91
182319 (Oil K)	-4.30	-5.94	-5.12

Draft Timeline – updated Sept 3, 2024

Notes: Yongli to reach out to API and Darryl to understand requirements for test to be ready for industry use regarding GF7.



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 or TBN on fresh EOT sample (ISP, Intertek)
- SwRI, Savant, and ISP to report on their results for Afton method with 1 day storage
- YM to draft ballot for changes to EOFT and EOWTT.
- YM to check with Intertek on additional EOT sample analyses- run TBN on cold storage fresh and EOT samples.
- Afton: Will provide more info on TBN and possibly pH on previous samples. Will review and help better document in method the specific measurements or conditions needed in method. Jacob to send out latest draft of method.
- Yong-Li to reach out to API and Darryl to understand requirements for test to be ready for industry use regarding GF7
- Sample size: 120 mL? Jacob Z to let us know if adequate to run duplicates (as written in method)
- Any other labs wanting to participate in ILS, please let Yong-Li know.
- TMC supply of Oils F and K (John to provide estimate of how much remaining and estimate if additional oil is needed). Oil F has 7.085 gallons remaining (from 25 gallons initially provided). Oil K has 2.925 gallons remaining (from 20 gallons initially provided).

- Mike to email Yongli and Jacob request for clarification for ILSAC

- Group ready to move onto ILS pending this meeting's results?

- Next Meeting: Monday Sept 16 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