

ASTM Engine Oil Gelation Test (EOGT) WK86363 Update

EOFT and EOWTT Surveillance Panel Meeting
September 16, 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



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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 and Original EOGT Results

- Savant update
- SwRI update
- Afton update
- ISP update
- Intertek update
- Valvoline update



Savant Labs Data for Afton Method – Sept 12

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: D4739: EOT 3.72	CP	Yes	24	-After addition of 2 nd 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 D4739 EOT 3.91	GM	Yes	24		
SAV 6	Oil F	CP	Yes	24		
SAV 7	Oil F	CP	Yes	24		
SAV 8	Oil K	CP	No	24		
SAV 9	Oil F	MB	Yes	24		9-12-24: 38C (up to 40C) mixing temp, run in duplicate, saved EOT for TBN:
SAV 10	Oil F	CP	Yes	24		Saved EOT for TBN
SAV 11	Oil K	CP	No	24	Saved EOT for TBN	

Notes 9-16-24:
 Savant focused on getting TBN results, low on Oils F and K
 -Recommendations: test to be pass/fail; look at heating in oven;
 -If Oil F does gel, the EOT will separate,
 -If Oil F doesn't gel, the EOT will not separate, behaving like Oil K



SWRI Updates 9-16-24 (tests mixed at 40C)

3 Techs,

- Oils In Duplicate
- Prepared Samples at the same time
- Interpreted Blending Procedure Independently.

Oil F

- Tech 1 – 2 Partial Successes
- Tech 2 – 1 Success, 1 Failure
- Tech 3 – 1 Success, 1 Failure

Oil F

- 4 Partial Successes
 - 1 Tech
 - Started to gel and then broke down in the last 15secs of blending (14,000rpm).
- 2 Failures
 - 2 Techs
 - Small particulates of gel on blending tool and jar walls.
 - No Complete gelling. >1ml/sec
- 2 Successes
 - 0.13 ml/sec
 - Blending Technique, Barista style with more air blending. More to follow.

Oil K

- No Particulates or Gelling



SWRI Updates 9-11-24

- Will Attempt additional runs to complete by Friday 13th
- Will film and get more a description on method. Can we compare videos to other labs technique? – Review in Root Cause Group



Afton Method V7

Version 7

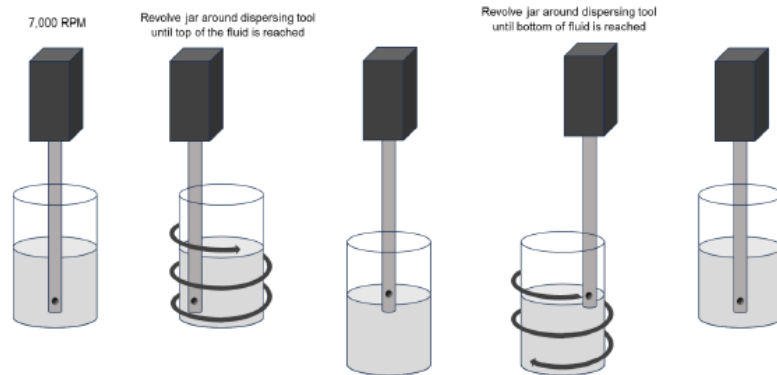


FIG. 1 Schematic of homogenization step 11.1.2

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.

11.3 Repeat steps 10.1 to 11.2.3 for duplicate runs.

12 TMC Reference Oil Testing

13 Report

- 13.1 Report the following information:
 - 13.1.1 Non-reference or reference oil identification.
 - 13.1.2 Time and associated volumes for each test.
 - 13.1.3 Time and associated volumes for the duplicate tests.
 - 13.1.4 For TMC-monitored tests, contact the TMC for reporting requirements.
 - 13.1.5 For tests not monitored by the TMC, an in-house quality assurance sample can be used to evaluate test acceptability.

14 Precision and Bias – to be established

Version 7.1

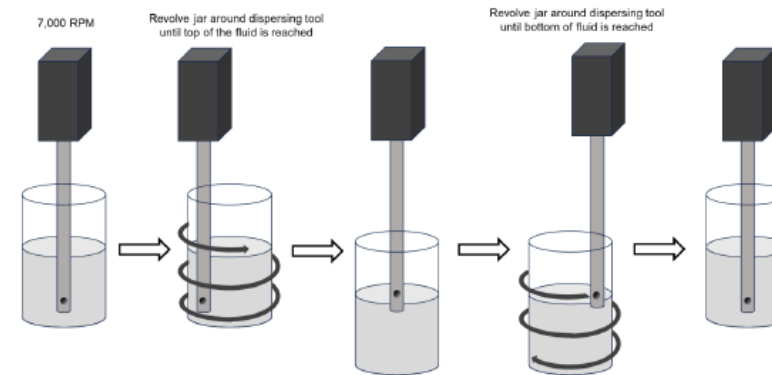


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EOGT METHOD 1/COLD STORAGE D4739 TBN BUFFER END POINT RESULTS



CMIRs	Average CFA, % (September 2023)	Average CFA, % (August 2024)	TBN Fresh Oil (mg KOH/g)	TBN EOT Oil (mg KOH/g)
182324 (Oil K)	-5.49	-11.29	5.30	1.92
182159/182160 (Oil K)	-8.03	-6.54	5.41	1.79
182325 (Oil F)	-7.62	-60.66	6.89	6.19
182161/182162 (Oil F)	-10.30	-72.13	6.95	6.18

- EOGT (large volume (600g)) method performed –September 2023
- 7-day cold storage-The retains were held at -5 to 0C for 7 days then filterability was run normally-August 2024
- TBN analysis on both the fresh oil and the post cold storage EOT oils was performed-September 2024

EOGT METHOD 1/COLD STORAGE OIL CONTAMINATION-OIL F



intertek

Total Quality. Assured.

TBN Results Sept 16

Lab	Method	Sample	TBN D4739 SOT	TBN D4739 EOT (homogenous sample)
ISP	Original	F (clog or gel)	7.4	6.3 (Hand mixed to be homogenous)
		F (clog or gel)	7.6	6.2
		F (clog or gel)	7.5	6.0
		K	5.5	5.1
		K	5.7	5.1
		K	5.7	5.0
ISP	Afton	F (no gel)	7.4	4.3
		K	5.5	3.2
Savant	Afton	F	5.41	3.91
			---	3.72
SwRI	Afton	K	5.68	3.29
		K	-	3.44
		K - 191493	5.75	3.28
		F (gel)	-	2.71
		F (gel)	7.48	4.46
		F (no gel)	-	4.39
		F- 191489 (no gel)	7.39	4.36
Intertek	Original + 11 months storage + 1 wk cold storage	K	5.30	1.02
		K	5.41	1.79
		F (clog or gel)	6.89	6.19
		F (clog or gel)	6.95	6.18

Notes 9/12/24:

Valvoline to provide TBN data on Original SOT and EOT samples

Savant to provide TBN results and suggestions for improving.

Intertek and Valvoline to set up to run Afton method

Infineum in process to try to run Afton method

Afton to run Afton method run at 25C and 35C mixing, and provide TBN on fresh and EOT gelling samples. To try different homogenizing step and let us know results.

ILS Planning (If time)

- Criteria for Afton method to moving forward to ILS: Root cause group to finalize method, specifically on shearing details; Ford to give recommendation to move forward
- Sample volume: 120 mL for duplicate run per oil
- Labs participating: Afton, Intertek, ISP, Savant, Southwest Research Institute, Valvoline
 - Others?



API comments on EOGT available for GF7 industry use

- 1. EOGT can correlate to the field issue of Engine Oil Gelation as described by Ford.
 - 2. EOGT must have an established test method with documented repeatability and reproducibility supported by statistical analysis.
 - 3. EOGT must be an ASTM Test Procedure.
 - 4. The EOGT Surveillance Panel would support API and AOAP in developing the Precision and BOI/VGRA Testing with Reference Oils to aid in Precision and BOI/VGRA testing.
 - 5. The AOAP would need to review and approve data from the EOGT Precision and BOI/VGRA testing.
 - 6. The EOGT Surveillance Panel exists as a functioning task force or surveillance panel to coordinate activities and analyze test data.
 - 7. The EOGT Surveillance Panel must provide a method for referencing and/or calibrating each bench test.
 - 8. EOGT shall be monitored by the TMC (or equivalent).
- Included Test Readiness Document from ACC comments that are relevant. Laura mentioned there will be an email from ACC email to Yongli on their comments.



Draft Timeline – updated Sept 13, 2024

Task	Date										
	5-6 2023	7-8 2023	9-10 2023	11- 12 2023	1-2 2024	3-4 2024	May-Jun 2024	Jul-Aug 2024	Sept-Oct 2024	Nov-Dec 2024	Jan-Feb 2025
Develop test procedure and ILS report form	█										
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			█	█	█	█	█	█	█	█	
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											█
Test available for industry use											█



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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)
 - 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.
 - 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
 - YM to draft ballot for changes to EOFT and EOWTT.
-
- SwRI and Afton to report on their results for Afton method
 - Savant to email Yongli list of recommendations for Afton method to share with group.
 - SwRI to take a video of the blending step for Root Cause group to review
 - Valvoline to report TBN results.
 - Labs to report TBN results.
 - Group to review TBN results on fresh and EOT samples.
 - Sample size: 120 mL? Jacob Z to let us know if adequate to run duplicates (as written in method)
 - Laura to help coordinate ACC response regarding EOGT readiness guidance.
 - Any other labs wanting to participate in ILS, please let Yong-Li know.
-
- Next Meeting: Monday Sept 30 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

