Study of the ROBO Oxidation Test Technique and Instrumentation: Current and Future Potential for use with Upcoming Oil Specifications

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Abstract

With an increasing demand for superior emission and fuel economy standards in modern engines, the automobile industry must be prepared to supplement these changes with the development of higher performance engine oils. Before an engine oil can be introduced to the consumer market, however, the oil must undergo significant testing for several properties, according to standards set by the American Petroleum Institute (API) and International Lubricant Standardization and Approval Committee (ILSAC). One such property is the oil's viscosity at low temperatures, which may cause catastrophic engine failure from poor pumpability and deposit formation.

The Romaszewski Oil Bench Oxidation (ROBO) test, developed for the API SN and ILSAC GF-5 specification, is a strong alternative to the Sequence IIIGA test that has long been used to evaluate cold-temperature viscosity of an aged oil. The ROBO test is able to duplicate the oil aging mechanism of Sequence IIIGA test results in a fast and cost-effective manner, while allowing for the assessment of oil viscosity with several other test methods. This paper is an attempt to be a comprehensive literature review on the work done on the ROBO test method so far. This paper will therefore take a close analysis into the development and applications of the ROBO test, as well as present a comparison with the Sequence IIIGA test method. We will discuss the various advantages and any possible drawbacks regarding the use of ROBO. We will also discuss the current and future potential of the ROBO test, including its use in the development of future oil specifications such as ILSAC GF-6.

- 1996 — ILSAC GF-2 Defined Max. Gelation Index
- 2001 — Development of Sequence IIIG
- 2004 — ILSAC GF-4 Introduced Aged Low-Temp Viscosity
- 2006 — ASTM Task Force Created for ROBO
- 2010 — ROBO Listed for Use in ILSAC GF-5
- 2016 — First Licensing of ILSAC GF-6
- 2017 — Sequence IIIHA Accepted as Alternative to IIIGA
- 2018 — ROBO Listed for Use in API SN Plus
- 2020 — Implementation of ILSAC GF-6

Figure Courtesy of Charles Dustman of Evonik Oil Additives [1] and Lubrizol Additives 360 [2]

Procedure and Significance

The ROBO test is its able to simulate an engine oil's oxidation and nitration processes, all within efficient laboratory conditions [3]. This process is significant as it allows for the development of various oil formulations. ROBO can be used quickly and inexpensively in the assessment of oil additives, viscosity modifiers, pour point depressants, and other formulation changes. In order to calibrate and use the ROBO test, the ASTM TMC (Test Monitoring Center) has set parameters for use with standardized reference oils. These oils must be aged by ROBO and obtain a certain range of MRV and pVis values for calibration. The pass/fail criteria for such reference oils are listed below.

Critical Pass/Fail Parameters for ROBO Aged Reference Oils - MRV Viscosity Acceptance Bands
Unit of Measure: Ln(MRV)

Oil	n	Natural Log Transformed Mean (ln)	Mean in Original Units	Standard Deviation (ln)	95% Band in mPa*s Min	95% Band in mPa*s Max	95% Bands Min (ln)	95% Bands Max (ln)
434-1	13	10.6599	42,612	0.1672	30,706	59,136	10.3322	10.9876
434-2	36	10.9284	55,737	0.1551	41,126	76,008	10.6244	11.2386
435	15	11.4895	97,685	0.2932	60,000	173,546	11.0021	12.0642
435-1	22	11.0416	62,420	0.20295	44,470	92,910	10.7048	11.4394
438	14	10.2676	28,785	0.2037	19,308	42,912	9.8683	10.6669
438-2	10	10.4421	34,273	0.2322	21,742	54,025	9.9870	10.8972

Figure Courtesy of ASTM Test Monitoring Center [4]

The ROBO procedure replicates the volatility and oxidation/nitration mechanisms within an automobile engine. To do so, 200 grams of test oil is first combined with iron ferrocene catalyst, to account for wear debris in the engine. This mixture is stirred and heated at 170 °C for 40 hours in a 1-L reaction vessel [5]. A feed of liquid nitrogen dioxide and air is then introduced below the reaction surface, while a feed of dry air is flowed across the surface of the liquid at negative pressure. Nitrogen dioxide is a critical component of the ROBO test, used to simulate the effects of blow-by gas within the engine [6]. Currently, however, there are proposals to replace pure liquid NO_2 with a dilute NO_2 air feed. As shown below, the dilute NO_2 setting produces similar MRV and pVis relationships to the current pure NO_2 feed. After the 40-hour oxidation process, the concentrated oil then undergoes viscometric and low-temp pumpability testing through ASTM D5293 and D4684. Lastly, the evaporated oil is condensed and weighed in order to calculate evaporative loss.

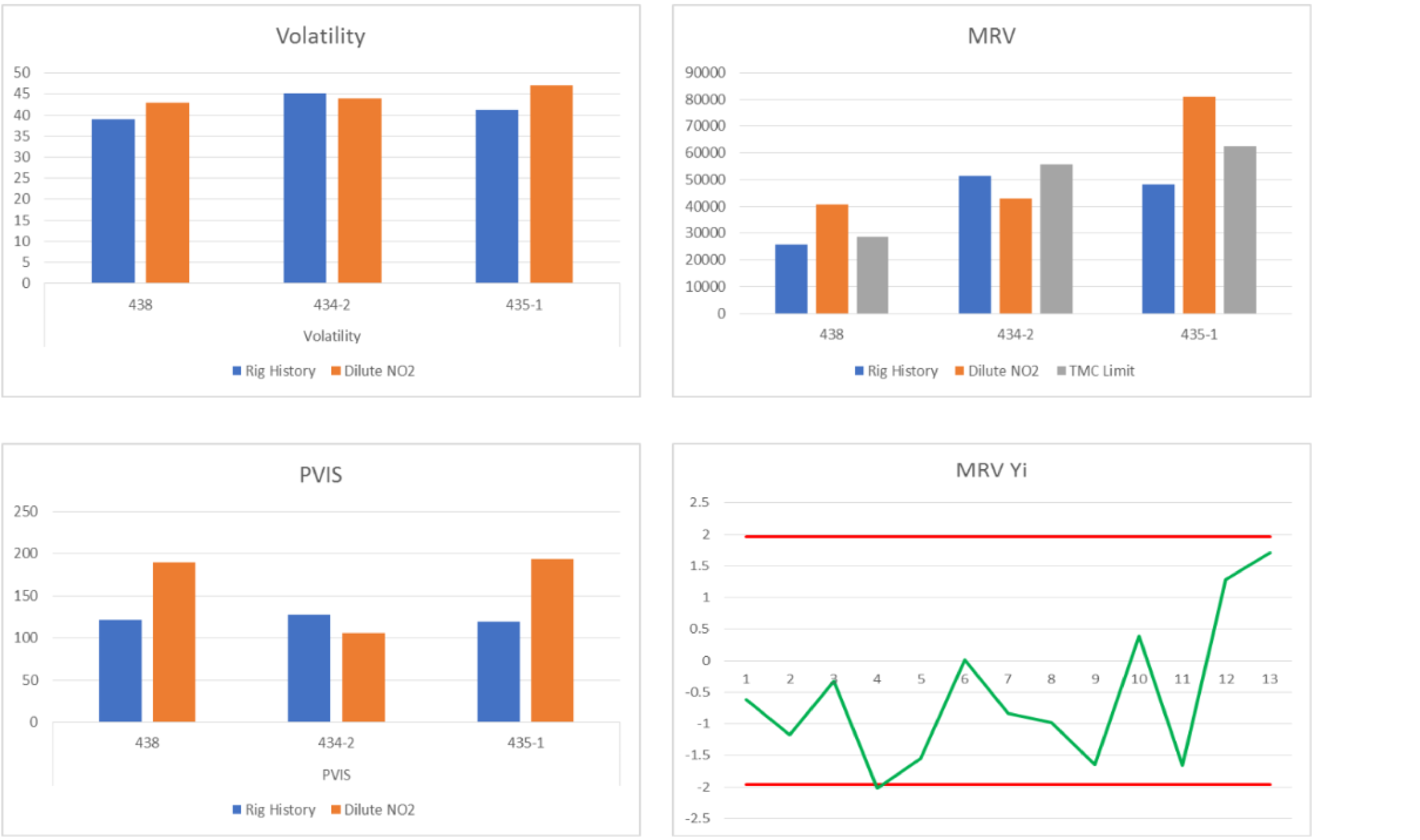

Figure Courtesy of April 2019 Surveillance Panel Meeting (Intertek) [7]

Figure Courtesy of Koehler Instrument Company Inc. [8]

Ingredient	Current ROBO Setting	Proposed Dilute NO_2 Setting
Test Fluid	200 grams	200 grams
Iron ferrocene	15 PPM	15 PPM
Nitrogen Dioxide	**2 mL "pure" NO_2 fed over first 12 hours**	**1.13% NO_2 in air fed at 185 mL/min over first 12 hours**
Dry air	**185 mL/min (entire test)**	**185 mL/min (12 hours – EOT)**
Agitation	200 RPM	200 RPM
Vacuum	0.61 Bar 56.6 L/min	0.61 Bar 56.6 L/min
Temperature	170°C	170°C
Time	40 hours	40 hours

Figure Courtesy of Justin Mills October 2018 [9]

Comparison to Sequence IIIGA

In 2010, ROBO became accepted for use in the ILSAC GF-5 specification, effectively rendering the need for Sequence IIIGA obsolete. Sequence IIIGA uses a GM 3.8L V6 engine, operates 60 hours longer, and costs about 22 times as much as the ROBO test [10]. After a careful analysis of reference oils across both test methods, ROBO has been shown to correlate well with the engine conditions of Sequence IIIGA. Preliminary data analysis of 34 reference oils reveals that ROBO successfully predicts the outcome of Sequence IIIGA pass/fail results 81% of the time [11]. This accuracy, though low, will continue to improve as the ROBO test method is further developed. Additionally, further linear regression analysis yields r^2 values of 0.802. This value increases to 0.922 after eliminating two outlier values. These values, along with the good MRV and pVis correlation, allows ROBO to be a strong alternative to Sequence IIIGA.

Test	Sequence IIIGA (ASTM D7320)	Sequence IIIHA (ASTM D7320)	ROBO (ASTM D7528)
Equipment	1996/1997 GM 3.8L V6 Engine	2014 Chrysler Pentastar 3.6L V6 Engine	Laboratory equipment and hardware from manufacturers
Aging Mechanism	Volatility and oxidation/nitration	Volatility and oxidation/nitration	Volatility and oxidation/nitration
Operation Conditions	100 hours at 150°C	90 hours at 150°C	40 hours at 170°C
Criteria	MRV ≤ 60,000 cP, with < 35 Pa yield stress	MRV ≤ 60,000 cP, with < 35 Pa yield stress	MRV ≤ 60,000 cP, with < 35 Pa yield stress
Test Cost	~$55,000	~$55,000	~$2,500

Figure Courtesy of Alan Flamberg [12] and ASTM International [5, 10, 13]

The Future of ROBO

In upcoming years, the authors hope that ROBO will be used in the development of oil formulations. A key component of oil additives that directly effects aged oil low-temperature rheology and performance are pour point depressants (PPDs). At the cloud point temperature of untreated oils, wax crystals appear and begin to conglomerate into large arrays as temperatures continue to decrease. These large wax crystals begin to rapidly increase viscosity, causing poor oil flow and yield stress. Oils treated with certain PPDs, however, control the size and formation of wax crystals, significantly reducing the rate of viscosity increase [14]. Therefore, PPDs maintain optimal oil flow and pumpability within the engine, without causing any yield stress.

The use of PPDs and other such oil additives will be increasingly important in future oil development and specifications. The upcoming ILSAC GF-6 specification aims to improve upon the performance standards of engine oils, while having a key focus on fuel economy, engine cleanliness, reduced engine wear, and aged low-temp oil performance. Such regulations call upon the need for improved oil formulations in order to create more reliable and robust oils. The ROBO test, therefore, allows the oil industry to efficiently screen PPDs and further develop formulations that meet the standards of ILSAC GF-6 and other such specifications.

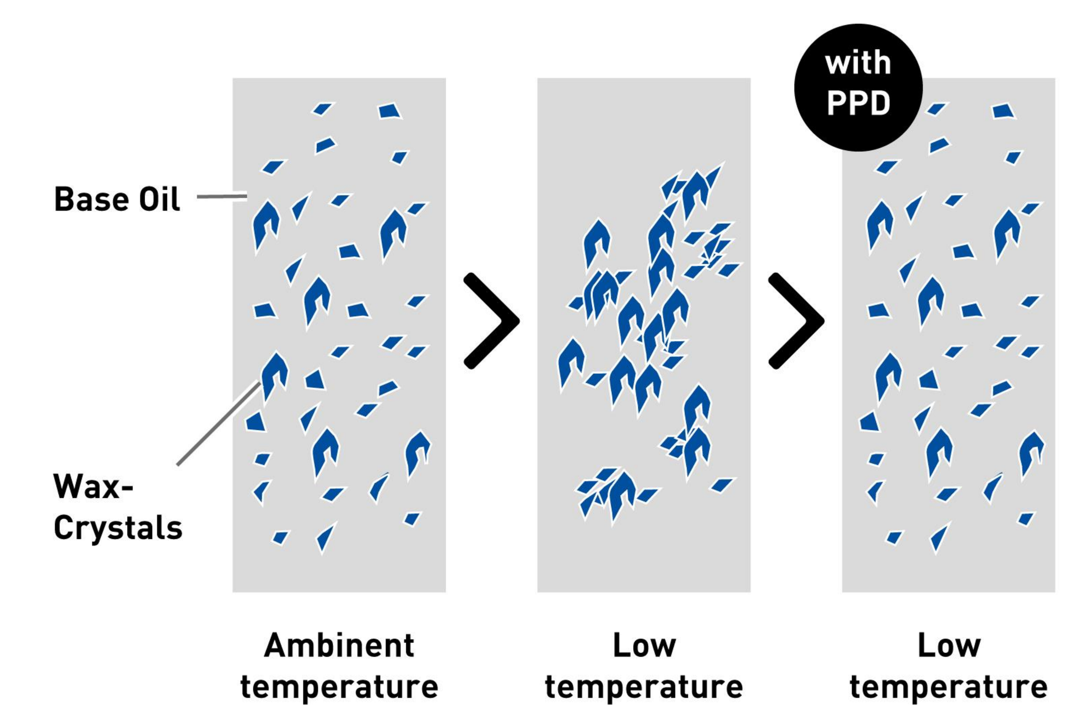

Figure Courtesy of Liqui Moly [15]

ILSAC GF-6 and API SP

The ILSAC GF-6 and API SP specifications are currently being developed by OEMs to improve oil performance in modern engines. The ROBO test is considered equivalent to the new Sequence IIIHA (ASTM D8111) test, which replaces Sequence IIIGA. A key difference between IIIHA and IIIGA is the use of a more modern 2014 Chrysler Pentastar engine, running at 90 hours instead of 100 [13]. ILSAC GF-6 is also split into two subcategories: GF-6A and GF-6B. GF-6A will be backward-compatible for previous SAE standards, while GF-6B adopts the new SAE 0W-16 viscosity grade, which is incompatible with older engines [16]. The API SP standard includes all ILSAC requirements, while adopting new standards to specify spark-ignited internal combustion engines. This is an effort to combat low-speed pre-ignition, which may cause catastrophic engine failure.

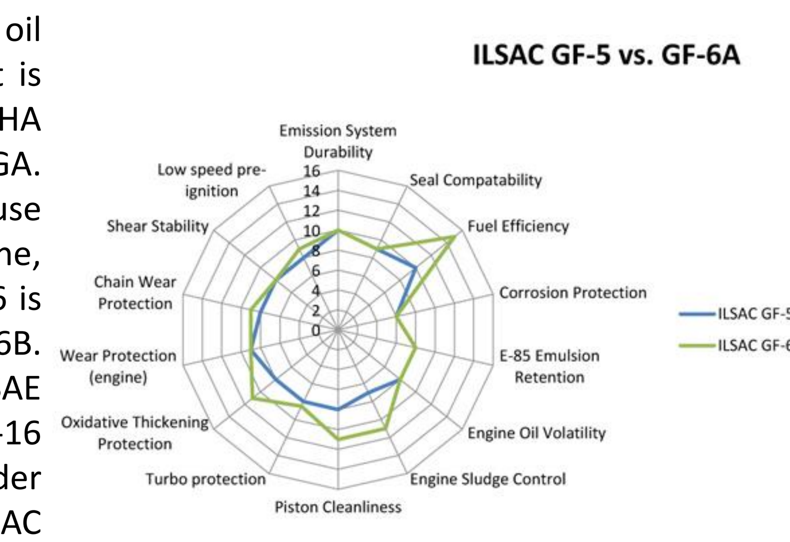

Figure Courtesy of MOTOR Magazine [17]

Conclusion

The ROBO test is able to efficiently and cost-effectively reproduce the oxidative oil aging process of Sequence IIIGA. Originally developed to replace IIIGA in the ILSAC GF-5 specification, the ROBO test utilizes laboratory equipment to reproduce results 60 hours faster at a significantly cheaper cost. ROBO plays a significant role in modern engine oil performance, especially in robust low-temperature performance and oil pumpability. Therefore, by closely analyzing the current improvements in ROBO's development, it is clear that ROBO will play a significant role in the development of future oil formulations and specifications such as ISLAC GF-6 and API SP.

References

1. Dustman, Charles K. "Low Temperature Assessment of Current Engine Oils." Evonik Oil Additives USA, 14 Jun. 2016.
2. Lubrizol Additives 360. "ILSAC GF-6 Timeline." Lubrizol Corporation, 26 Oct. 2017.
3. Gill, George. "ROBO Is Ready to Roll." *Lube Report*, Vol. 17, No. 52. 22 July 2009.
4. ASTM Test Monitoring Center. "ASTM Test Monitoring Center Requirements for Engine Test Stand/Laboratory Calibration." ASTM International, Apr. 2019.
5. ASTM D7528-17a. "Standard Test Method for Bench Oxidation of Engine Oils by ROBO Apparatus." ASTM International, 2017.
6. Canter, Neil. "Proper Additive Balance Needed to Meet GF-5." *Tribology & Lubrication Technology September 2010*, STLE, 26 Aug. 2010.
7. Mills, Justin. "April 2019 Surveillance Panel Meeting – Intertek's Configuration for Dilute NO2." *ROBO Surveillance Panel*. 11 Apr 2019.
8. Koehler Instrument Company Inc. "K83400 ROBO Apparatus." 2017.
9. Mills, Justin. "October 2018 Surveillance Panel Minutes and Presentation." *ROBO Surveillance Panel.* 25 Oct 2018.
10. ASTM D7320-18. "Standard Test Method for Evaluation of Automotive Engine Oils in the Sequence IIIG, Spark-Ignition Engine" ASTM International, 2018.
11. Kinker, B. G., Romaszewski, R., and Palmer, P. A. "ROBO – A Bench Procedure to Replace Sequence IIIGA Engine Test." *Journal of ASTM International*, Vol. 4, No. 10, pp. 1-13, 2007.
12. Flamberg, Alan. "ROBO: A Method to Generate Aged Oil for Low Temperature Viscosity Assessment for Current and Future Engine Oil Categories." Evonik Industries. May 25, 2017.
13. ASTM D8111-19. "Standard Test Method for Evaluation of Automotive Engine Oils in the Sequence IIIH, Spark-Ignition Engine." ASTM International, 2019
14. Covitich, Micheal J. and Dimitrakis, William J. "Cold Comfort – Pour Point Depressants Keep Things Flowing." Lubes'N'Greases Magazine.
15. Liqui Moly. "FAQ: 4.3 Pour-Point-Depressant (PPD)" Liqui Moly GmbH, 2019.
16. Infineum Insight. "Why ILSAC GF-6?" Infineum International Limited, 2019
17. Chabot, Bob. "A Well-Oiled Edge." *Alternative Fuels & Lubricants*, MOTOR Magazine, 21 Nov. 2013.