# ILSAC GF-5 Development - Better Late Than Never

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**Abstract**

On December 3, 2004, the International Lubricant Specification and Approval Committee (ILSAC), which represents U.S. and Japanese automobile manufacturers, issued a letter to the chairman of the ILSAC/Oil Committee requesting the development of the next ILSAC Minimum Performance Standard for Passenger Car Engine Oils, known as ILSAC GF-5, scheduled to be commercialized on July 1, 2009. Since that time, a GF-5 Needs Statement has been written (but not finalized), several work groups have been formed to develop new performance tests, a draft GF-5 specification has been issued by ILSAC, and the GF-5 timeline for commercialization has been delayed one year into 2010. This paper discusses the development of ILSAC GF-5 from January 2005 to the present time, as well as the challenges which lie ahead in formulating an engine oil that can pass all engine and bench tests at the proposed limits defined in the GF-5 specification.

**ILSAC/Oil Committee**

ILSAC specifications are developed through a committee known as the ILSAC/Oil Committee, which is composed of representatives from ILSAC member companies, the API (American Petroleum Institute) Lubricants Committee, and the ACC (American Chemistry Council) Petroleum Additives Panel member companies[1]. The membership of the ILSAC/Oil Committee consists of 50% representation from the auto industry (ILSAC) and 50% from the oil/additive industry (API/ACC). Currently, there are six voting members, three from ILSAC, two from API, and one from ACC; there are also three alternate members, one from each trade association. Decisions are made by consensus, which is defined as approval by two thirds of ILSAC representatives and by two thirds of the Oil/Additive representatives[2].

**Process for New Engine Oil Category Development**

With the advent of ILSAC GF-4 and its sister category, API SL, new passenger car engine oil categories are now developed according to the ILSAC/Oil performance category development process. This process is designed to accomplish the following[3]:

a. Validate the need for a new category
b. Achieve stakeholder consensus early in the process
c. Optimize the process for developing and approving new categories

There are ten (10) steps in the process, which can be summarized as follows:

1. Define category need & timing
2. Develop test needs & alternatives
3. Develop new test methods
4. Formalize new tests, e.g. test precision
5. Issue draft specification
6. Industry review of draft specification
7. Review of industry comments
8. Provide demonstration oil data
9. Finalize specification
10. Approve API licensing of ILSAC specification

[1] American Petroleum Institute website, http://committees.api.org/downstream/lubricants/ilsacoil.
[2] API Publication 1509, 16th Edition (April 2007), Appendix C, Section C.2.1.
[3] API Publication 1509, 16th Edition (April 2007), Appendix C, Section C.3.

Most of these steps are managed by the ILSAC/Oil Committee, with the exception of test formalization (Step 4, managed by the ASTM PCEOCP[4]) and API licensing (Step 10, managed by API Lubricants Committee). The provision of demonstration oil data (Step 8) is also managed by the API Lubricants Committee. A demonstration oil is necessary to demonstrate the technical and commercial viability of the proposed new engine oil category: an engine oil formulated with base stock and additive components that are expected to be commercially available when licensing of the new category begins. The oil shall have been tested and passed, at the proposed limits, all tests required in the draft specification[5].

**Definition of GF-5 Needs**

In its December 2004 letter, ILSAC cited three general areas of improvement for GF-5 engine oils[6]:

1) Fuel economy and fuel economy retention;
2) Emission system compatibility; and
3) Robustness of engine oils to provide increased engine durability.

These three areas of improvement are the same as those announced for the earlier engine oil categories, ILSAC GF-4 and GF-3. Besides the need for fuel economy and emission system compatibility improvements, ILSAC identified the following improvements over the current GF-4 category:

a. Replacement of Seq. VIII Cu/Pb bearing corrosion test with a bench test
b. Protection against high temperature black sludge
c. Reduction of high temperature piston deposits
d. Replacement of Seq. IIIG high temperature oxidation test with a new test (Seq. IIIH)
e. Protection against soot-induced abrasive wear (e.g. JAMA chain wear)
f. Reduction of engine oil phosphorus volatility (related to catalyst compatibility)
g. Replacement of the Seq. VIB fuel economy retention test with a new test (Seq. VID)
h. Protection against turbocharger bearing coking (TEOST 33C from GF-2)
i. Reduction in aeration of new and used oil
j. Evaluation of elastomeric compatibility

In June 2005, a small task force was assembled to develop a draft GF-5 Needs Statement for approval by the ILSAC/Oil Committee. The final draft was presented to the ILSAC/Oil Committee on June 29, 2006. As outlined in API 1509 Appendix C, the task force justified the need for a new engine oil category, citing impending government regulations, the introduction of new engine hardware and consumer-driven needs[7]. No data concerning field problems with current oils were presented; however, it was stated that increased sludge protection is necessary to address the low temperature sludge concerns associated with current (mostly GF-3) oils in the market.

*Impending Government Regulations*

- CAFE – the U.S. Corporate Average Fuel Economy standard for light duty trucks is currently at 21.6 mpg and will possibly increase to 29.5 mpg (moving target) by the 2011 model year.
- U.S. Energy Policy Act of 2005 – requires increased use of renewable fuels through 2012.
- U.S. EPA Mobile Source Air Toxics – will require non-methane hydrocarbon (NMHC) standards for cold temperatures (20$^{o}$F) by the 2011 model year
- California CAA section 177 – will adopt increasingly more stringent non-methane organic gas (NMOG) standards for LEV II (low emission vehicles) through the 2010 model year

[4] Passenger Car Engine Oil Classification Panel, ASTM D02.B0.01.
[5] API Publication 1509, 16th Edition (April 2007), Appendix C, Section C.3.3.1.
[6] Letter from Michael L. McMillan, ILSAC Chairman to R. Olree. ILSAC/Oil Chairman, “Request to Begin Development of ILSAC GF-5”, December 3, 2004.
[7] Murray, H., “GF-5 Needs Statement”, presentation to ILSAC/Oil, June 29, 2007.

In response to the regulations outlined above, ILSAC GF-5 must incorporate increased fuel economy and improvements in both FEI retention and durability throughout the oil change interval, relative to GF-4. The development of the Sequence VID Fuel Economy Test is expected to address this need. ILSAC GF-5 must also include requirements ensuring the compatibility of GF-5 oils with emission system components capable of meeting the stringent federal and California emission regulations. The reduced phosphorus and sulfur poisoning of exhaust gas catalysts and oxygen sensors is expected to allow OEM's to meet current and future tailpipe emission requirements for the full useful life of the vehicle.

*New Engine Hardware Introduction*

- Multi-fueled Vehicles (MFV) –Approximately 50% of General Motors engines will be fuel-flexible by 2011[8], meaning that they will be able to run on gasoline as well as E85 (a blend of 85% ethanol and 15% gasoline). Approximately 25% of Chrysler vehicles are E85-capable in the current model year. Currently, U.S. EPA allows CAFE credits up to 1.2% as an incentive for automakers to produce E85-capable vehicles. MFV fuel demands emulsibility and rust control.
- Turbocharging – Currently, only 2% of all gasoline-powered engines in North America are turbocharged; however, CAR (Center for Automotive Research in Detroit) estimates that the percentage of turbochargers in new vehicles will grow from 13% in 2011 to 18% in 2016[9] as automakers work to meet tougher fuel economy standards. Chrysler has been the most vocal concerning the need for improved protection against turbocharger bearing coking deposits, proposing to include the TEOST 33C bench test in the GF-5 standard. Chrysler reported that turbocharger coking is not necessarily a problem in the field, as failures have not occurred when drivers follow the oil recommendations and change their oil on time[10].
- Cylinder Deactivation – Virtually all current General Motors engines use variable valve timing. This is accomplished using camshaft phasing, which can also provide the capability to deactivate the cylinder for improved fuel economy. These VVT systems with hydraulic valve actuation require sufficient engine oil aeration control in order to function properly.
- Gasoline Direct Injection – Although not specifically mentioned in the latest GF-5 Needs Statement, nearly all automakers currently have at least one vehicle using GDI engine technology, and CAR estimates that the market penetration of GDI engines will increase from 12% of new vehicles sold in 2011 to nearly 20% in 2016[9], since GDI can provide 5-10% better fuel economy (10-15% better with turbocharging), and better driving performance, than conventional gasoline engines. GDI engines have shown an increased level of oil contamination by soot, resulting in engine durability and performance issues such as increased timing chain wear and elongation. This issue was raised by JAMA (Japanese Automobile Manufacturers Association) late in the development of ILSAC GF-4, resulting in the adoption of a minimum phosphorus limit (0.06%) for GF-4. It was expected that a Valve Chain Wear test would be developed for GF-5 to address this concern.

Consumer-driven needs identified in the GF-5 Needs Statement include improved vehicle performance, fuel efficiency, and towing capability.

In response to the drafted GF-5 Needs Statement, the Oil members of the ILSAC/Oil Committee, represented by API, stated that GF-5 should be a core standard designed to meet the needs of the "typical" engine and driver, with special requirements (e.g., turbocharging, GDI) best served

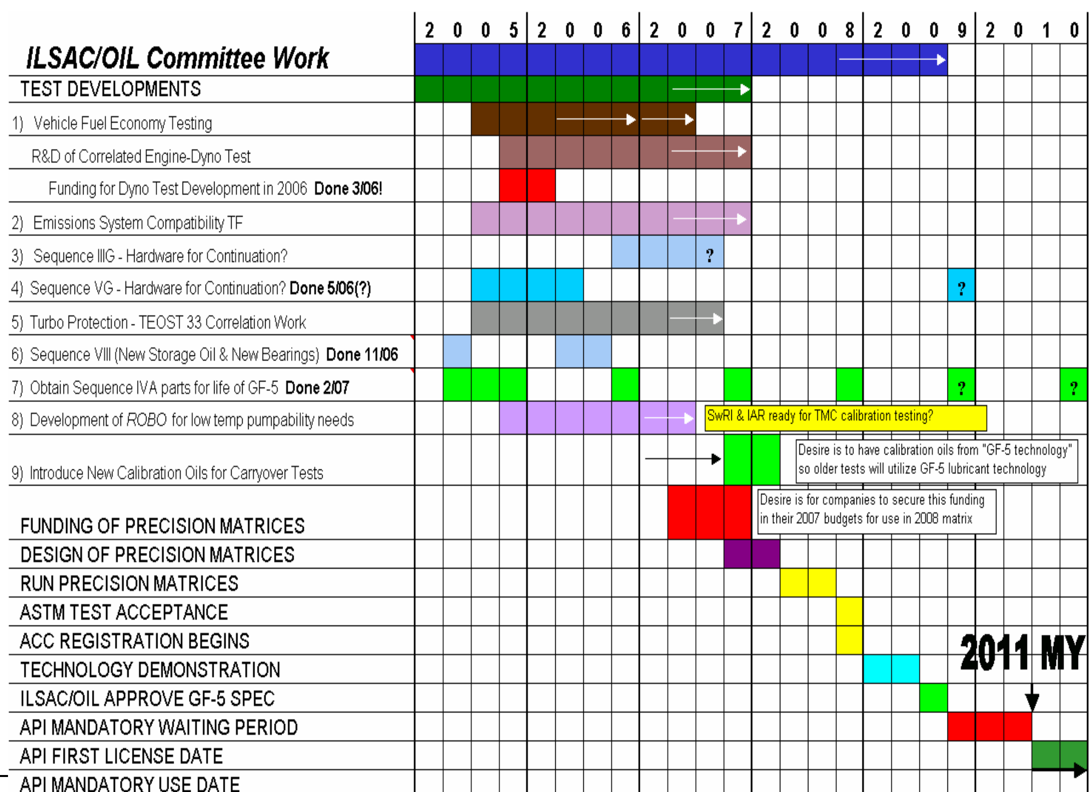
[8] Olree, R., Draft ILSAC/Oil Committee Meeting Minutes, Nov. 21, 2006.
[9] Center for Automotive Research "Automotive Powertrain Forecast U.S. Market 2011 and 2016", July 20, 2007, presented at ILSAC/Oil Committee Meeting, August 10, 2007.
[10] Olree, R., Draft ILSAC/Oil Committee Meeting Minutes, Apr. 12, 2006, page 2.

by different products[11]. API agreed with the need for fuel economy and catalyst durability/protection; however, more information was requested from ILSAC in order to understand the significance of turbocharger deposits, MFV requirements, oil aeration and soot-induced wear. ILSAC was specifically requested to provide estimates of the future use of certain new engine technologies.

In a letter to API on February 16, 2007, ILSAC responded that no information on the production volumes of the future engine technologies in question (e.g., turbocharging, GDI, MFV, VVT) could be made available by ILSAC member companies without divulging highly proprietary information, given the limited number of companies supplying data[12]. This prompted API to hire CAR (Center for Automotive Research) to conduct a survey of automakers and parts suppliers in order to estimate the extent of market penetration of new engine hardware technology during the lifetime of GF-5. A draft final report was provided to API by CAR on July 20, 2007, and shared with ILSAC on August 10, 2007. No agreement has yet been reached between ILSAC and the Oil members concerning the GF-5 Needs Statement, due to different interpretations of the data in the CAR report.

**GF-5 Category Timing**

The original timeline for GF-5 was issued by the ILSAC/Oil Committee in April 2005, proposing that the first date for API licensing be July 1, 2009, in order that GF-5 oils be available for the introduction of 2010 model year (MY) vehicles. In September 2006, the ILSAC/Oil Committee revised the GF-5 timeline to account for delays in the development of the Sequence VID Fuel Economy test. The current GF-5 timeline calls for the first API licensing one year later on July 1, 2010, in time for the introduction of 2011 model year vehicles. Automakers have been adamant that the revised timing cannot be delayed any further, primarily due to the timing of U.S. governmental regulations regarding tailpipe emissions and fuel economy. The current GF-5 timeline is shown in Figure 1 below[13]:




[11] Newsom, J., "API Lubricants Committee comments on draft ILSAC GF-5 Needs Statement", API letter to Hannah Murray and Bob Olree, Sept. 19, 2006.
[12] Linden, J., ILSAC letter to Kevin Ferrick, Feb. 16, 2007.
[13] Weber, B., "GF-5 Timeline – June 2007", presentation to ILSAC/Oil, June 21, 2007.

The Test Development phase is the critical stage for the timely introduction of GF-5 to the marketplace. All test development must be complete by the end of 2007 or the first quarter of 2008, so that test precision matrices can be conducted for the new GF-5 tests, and so the new tests can be accepted by ASTM by the end of 2008. This will be followed by a six month technology demonstration period when formulators will evaluate the newly accepted GF-5 tests in order to understand the response of new additive technologies and determine the formulation tradeoffs that may exist. The final ILSAC GF-5 specification, with all tests and limits, must be approved by the ILSAC/Oil and API Lubricants Committees by October 1, 2009, in order to allow API licensing by July 1, 2010. This provides a nine month mandatory waiting period after final specification approval to allow all API licensees sufficient time to develop their GF-5 product offerings.

**ILSAC GF-5 New Test Development**

There are three new tests being developed specifically for GF-5:

1. A fuel economy engine dynamometer test (Sequence VID), which will replace the Sequence VIB test.
2. A phosphorus volatility test (yet to be selected), which is intended to protect the exhaust gas catalyst from phosphorus poisoning.
3. A used oil low temperature viscosity bench test, called the Romaszewski Oil Bench Oxidation test (ROBO), which is intended to replace the Sequence IIIGA engine test.

*Sequence VID Development*

At its October 2004 meeting, ILSAC agreed unanimously that GF-5 will require a new engine dynamometer test, responsive to both viscometric and friction modifier effects, to evaluate the engine oil fuel efficiency of gasoline-powered passenger cars and light-duty trucks. ILSAC also agreed that baseline data using the U.S. Federal Test Procedure (FTP) fuel economy test cycle should be obtained to serve as the field correlation database for the development of the new test[14].

At its January 11, 2005 meeting, the ILSAC/Oil Committee established a GF-5 Fuel Economy Task Force to oversee the development of both the FTP data and the new Sequence VID test. General Motors agreed to act as sponsor for the Sequence VID Test, and offered its 3.6L V6 engine as the test development platform.

At the April 12, 2006 meeting of the ILSAC/Oil Committee, the GF-5 Fuel Economy Task Force gave way to the Sequence VID Consortium for the development of the new fuel economy test for GF-5. Ten companies signed a “letter of intent” to join the Consortium, including Afton Chemical, Infineum, Lubrizol, Chevron Oronite, R.T. Vanderbilt Company, Chevron Products Company, ExxonMobil, Shell Oil, General Motors, and Ford Motor Company[15].

The two parts of the Consortium’s Sequence VID development are (1) FTP testing to provide field correlation, and (2) VID dynamometer engine mapping, data collection, stage selection, and prove-out. A matrix of 10 engine oils was assembled to cover a range of oil viscosities, types and levels of friction modifiers. These engine oils for FTP testing and VID development are shown in Figure 2. The vehicles used for FTP fuel economy testing include engine platforms from General Motors (3.6L V6, 3.5L V6, 6.0L V6, and 2.3L L4), Ford (5.4L V8, 2.3L I4), and Nissan (2.5L L4). All 10 oils were run in three of four GM platforms, while 3-5 selected oils were run in all the other platforms from GM, Ford, and Nissan. FTP testing was conducted after mileage accumulation of 2000 and 6500 miles on the test oils (500 and 5000 miles for Ford testing).

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[14] McMillan, M., “Invitation to Join Consortium to Develop New Sequence VID Fuel Efficiency Test for Engine Oils”, May 10, 2006.

[15] Linden, J. & Leverett, C., “Consortium to Develop A New Sequence VID Fuel Efficiency Test for Engine Oils”, report to ASTM PCEOCP, June 19, 2007.

A summary of the FTP test results was presented to the ASTM PCEOCP on June 19, 2007[13]. Over 600 FTP/Highway tests were conducted during an 18-20 month period ending in June 2007. With regard to the average FTP, highway, and combined fuel economy results from all vehicles, statistical evidence indicated that all 10 matrix oils were different than the baseline oil (20W-30 monograde oil); however, there was no statistical evidence that the matrix oils could discriminate from each other. FTP fuel economy differences between the matrix oils ranged from 0.2 mpg to 0.5 mpg. There was no statistical evidence that, among the matrix oils, the lighter viscosity grade oils (e.g. 0W-20, 5W-20, 5W-30) showed any FE improvement; however, there was some observed FE benefit from friction-modified oils.

***Figure 2. Sequence VID Matrix Oils***

| | DI Package –1 |
|---|---|
| **A** | 5W-20 (No FM) |
| **B** | A + Organic FM-1 |
| **C** | A + Moly-type FM-1 |
| **D** | 5W-30 |
| **E** | 10W-30 |
| **F** | Z + 5X Detergent |
| **Z** | Baseline Oil (20W-30) |

| | DI Package –2 |
|---|---|
| **G** | 5W-30 (No FM) |
| **H** | G + Organic FM-2 |
| **I** | G + Moly-type FM-2 |

| | DI Package –3 |
|---|---|
| **J** | 0W-20 + Moly-FM-3 |
| **K** | 5W-20 + Moly-FM-3 |

With regard to the effects of oil and vehicle aging during FTP testing, there was statistical evidence that fuel economy improves with increased vehicle miles, but this relationship was different for each vehicle. Not surprisingly, since the matrix oils could not be statistically distinguished from each other, there was no statistical evidence of an oil aging effect on fuel economy.

On a positive note, preliminary VID engine mapping tests have been carried out in the proposed VID engine (GM 3.6L HFV6) using a subset of matrix oils. The testing suggests that this engine recognizes whether or not oils contain a friction modifier and, to a lesser extent, can discriminate between oils of different viscosity grades.

It is expected that the VID engine dynamometer test will be finalized (final stage conditions selected and final test development runs completed) in early 2008.

*Phosphorus Volatility Test*

The GF-5 Emissions System Compatibility Improvement Team (ESCIT) was formed in early 2005 with the mission to evaluate potential methods to determine the impact of GF-5 engine oil formulations on emission system function and durability. The team is focusing on the impact of phosphorus on catalysts and oxygen sensors[16]. In April 2006, ESCIT defined the development of a phosphorus volatility test as its primary goal[17]. Phosphorus volatility has been studied as a potential problem for catalyst compatibility in modern engine oils by T. W. Selby et al[18], using a modified version of the ASTM D 5800 (Noack) Evaporation Loss of Lubricating Oils test. The

[16] Sherwood, C., "GF-5 Emissions System Compatibility Improvement Team Charter", March 11, 2005, presented to ILSAC/Oil Committee April 14, 2005.

[17] Engel, C., "GF-5 ESCIT Report to ILSAC/Oil", April 12, 2006.

[18] Selby, T. W., Bosch, R.J., and Fee, D.C., "Phosphorus Additive Chemistry and Its Effects on the Phosphorus Volatility of Engine Oils", presented at ASTM D02 Symposium, December 7, 2004.

analysis of phosphorus in the collected volatiles indicates that phosphorus volatility is not closely related to engine oil volatility. Furthermore, the level of phosphorus volatilization in the Noack test is strongly affected by the chemistry of the ZDDP (zinc dialkyldithiophosphate) used in the engine oil formulation.

ESCIT expects to finalize their recommendation to the ILSAC/Oil Committee for a specific GF-5 phosphorus volatility test by the end of 2007. Current tests under consideration include the Sequence IIIG engine test, the Selby-modified Noack bench test, referred to as PEI-165, and the TEOST MHT-4 test (ASTM D 7097). PEI (Phosphorus Emissions Index) is the measured amount of phosphorus volatilized in the modified Noack test, expressed in milligrams per liter of engine oil. The modified test conditions involve a test temperature of 165°C and a test length of at least 16 hours, compared to the standard D 5800 test conditions of 250°C for 1 hour. Test lengths of 32 hours and 48 hours are also being studied to establish the optimum test length where most of the volatile phosphorus has been released and the PEI value (mg/L) stabilizes. The TEOST MHT-4 test was proposed as a phosphorus volatility test by R.T. Vanderbilt Company in October 2007. This test method will be the subject of a future technical paper.

Studies by ExxonMobil[19] investigating the effects of alcohol type (primary vs. secondary) and chain length support this conclusion. In this work, significant differences in phosphorus volatility were observed by measuring the amount of phosphorus in the crankcase at the end of a standard, 100-hour Sequence IIIG engine test. The test oils, all 0W-30 with 0.075% phosphorus, contained either a long chain primary ZDDP or a short chain secondary ZDDP. In the Sequence IIIG test, the long chain primary ZDDP demonstrated a significant advantage over the short chain secondary ZDDP: 97% vs. 78% phosphorus retention in the crankcase at the end of the test. The same 0W-30 oils were tested in the PEI-165 16 hour bench test, yielding PEI values of 48 mg/L and 4 mg/L, respectively, which is consistent with the Sequence IIIG results.

A taxi test was conducted by Lubrizol and Ford[20] using eight 2003 Crown Victoria™ vehicles (with 4.6L V8 engines) to investigate the effect of ZDDP chemistry on 3-way exhaust catalyst durability. The test ran for 100,000 miles with oil changes every 5000 miles. The two lubricants used were GF-4 5W-20 oils containing 0.076% phosphorus, one with conventional ZDDP, the other with "low impact" ZDDP. The evaluations included EOT engine parts inspection, used oil analysis, aged catalysts emissions efficiency assessment by FTP-75, and post-mortem aged catalyst analysis. The "low impact" ZDDP caused approximately 50% less phosphorus loss than did conventional ZDDP, based on measurements of phosphorus and calcium in the fresh and drain oils. This can be seen in Figure 3 *(courtesy of Lubrizol)*, a plot of average phosphorus retention (%) versus miles traveled.

***Fig 3. ZDDP Taxi Test Results***

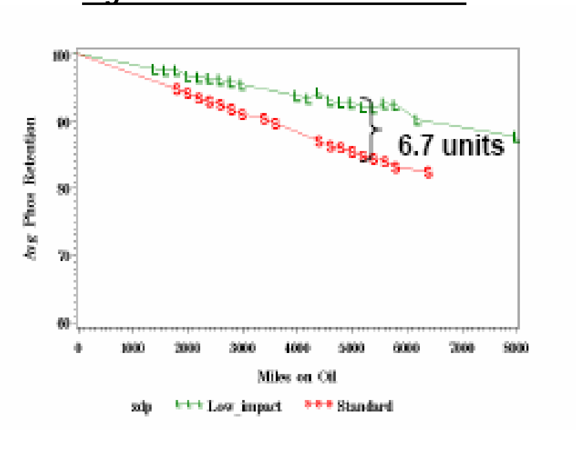
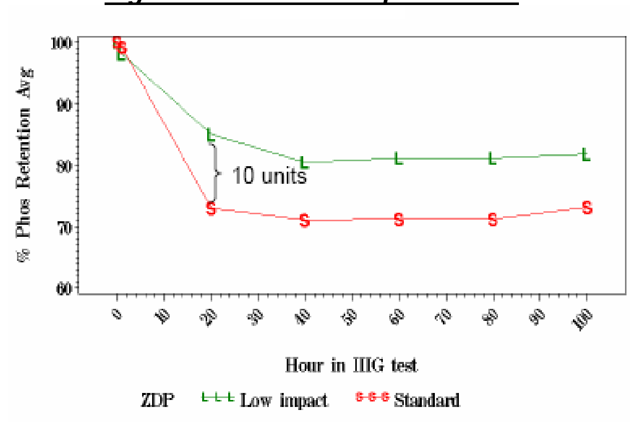



***Figure 4. ZDDP in Sequence IIIG***




[19] Deckman, D., "ExxonMobil Phosphorus Volatility Studies", presented to ESCIT, February 22, 2007.
[20] Williams, L., "P Retention in Sequence IIIG Engine Test vs. Field Performance", presented to ESCIT, Sep. 28, 2006.

The difference in total phosphorus loss over 100,000 miles by mass balance around the crankcase was measured and calculated to be 9-10 grams for "low impact" ZDDP, as opposed to 14-15 grams for conventional ZDDP. A 30% reduction in phosphorus concentration in the first inch of catalyst brick was also measured. A phosphorus mass balance showed that a very high percentage of the phosphorus that disappeared from the crankcase ended up deposited on the catalyst. Figure 4 *(courtesy of Lubrizol)* shows the percent phosphorus retention in the Sequence IIIG test using the same oils as in the Lubrizol/Ford taxi test, indicating that the Sequence IIIG test can be used to measure phosphorus volatility and that it properly ranks the taxi test oils.

Another test to measure the catalyst compatibility of engine oils that is also being considered by ESCIT is the Catalyst Poisoning Test being developed by Southwest Research Institute[21]. This is an engine test using a 2002 Chevy Malibu 3.1-liter V6 which is run for 250 hours with a heated oil sump at 150°C. The test oil is completely changed each day (10 times during the test). The engine exhaust is piped to a test catalyst block containing 900 cpi Pd/Rh washcoat. Catalyst poisoning is measured by using the FTP-75 emissions test and determining $T_{50}$, the temperature at which 50% conversion of HC and CO is achieved across the catalyst. Initial test runs indicate that the engine test has a reasonable potential to discriminate oils with different levels of ZDDP, but it most likely won't be ready for GF-5.

*ROBO as Used Oil Low Temperature Viscosity Test*

The Romaszewski Oil Bench Oxidation test (ROBO) is named after a Rohmax scientist who developed the test; the development began around the time of the approval of the ILSAC GF-4 specification. Rohmax was attempting to develop a laboratory method to simulate the oil aging that occurs in the Sequence IIIGA test in order to determine aged oil low temperature viscosity. The Sequence IIIGA test was added to the ILSAC GF-4 specification as a new requirement to protect engines against oil pumping failures at low ambient temperatures in situations where the vehicle owner may have extended the oil change interval beyond the manufacturer's recommendation. In the Sequence IIIGA test, used oil from the Sequence IIIG engine test is collected and MRV TP-1 low temperature viscosity is measured using ASTM D 4684. The D 4684 viscosity of the EOT sample must meet the requirements of the original grade or the next higher grade[22].

The ROBO test was introduced by Rohmax to the ILSAC/Oil Committee in October 2005 as a possible bench test replacement for the Sequence IIIGA engine test, in an effort to reduce product approval testing costs. In setting out to duplicate Sequence IIIG with respect to its oxidative environment, Rohmax determined that the following factors were important components of the ROBO test[23]:

1) bringing together the main reactants oil and oxygen (air);
2) accounting for catalytic effects caused by the wear metals (iron) and blow-by gas ($NO_2$); and
3) setting the appropriate conditions (time, temperature, mixing, and vacuum).

Many of these factors have been used to simulate Sequence IIIG conditions using a bench test in prior attempts[24]; however, the ROBO test is the first test to include evaporative loss (related to oil consumption) as a very significant physical process in engines and, in particular, Sequence IIIG. This is important for any procedure attempting to reproduce used oil rheology. Without removing the lighter oil fractions it is not possible to achieve correct rheology measurements that are in line with each other.

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[21] Ellis, E., "Catalyst Poisoning Test", presented to ESCIT, June 14, 2007.
[22] "ILSAC GF-4 Standard for Passenger Car Engine Oils", January 14, 2004, page 4.
[23] Kinker, B.G., Romaszewski, R., Palmer, P., "ROBO – A Bench Procedure to Replace Sequence IIIGA Engine Test", presented at ASTM, June 2007.
[24] Roby, S.H., Mayer, R.J., Ruelas, S.G., Martinez, J.G., and Rutherford, J.A., "Development of a Bench Test to Predict Oxidative Viscosity Thickening in the Sequence IIIG Engine Test", SAE Paper 2004-01-2965.

The ROBO test employs a one liter glass reactor with an outer heat element rated at 400 watts, an electric stirrer with a 45° pitch blade turbine, a nitrogen dioxide delivery system, an air drier and flow meter for subsurface feed, temperature and vacuum controls. Operating conditions are as follows:

- 200 g of test fluid
- 15 ppm of iron ferrocene catalyst
- 2 mL of high purity, liquid nitrogen dioxide delivered over a 12 hour period
- 185 mL/minute dry air flow
- 200 rpm agitation
- 18 inches Hg vacuum with velocity of 2 cfm
- Reaction temperature of 170°C
- Reaction time of 40 hours

In addition to three ASTM reference oils, 34 engine oils having Sequence IIIG and IIIGA engine test results were donated by various companies, conditioned by ROBO testing, and the resulting used oil rheologies compared to the EOT used oil rheologies from Sequence IIIG. The comparison between the Sequence IIIG MRV results and the ROBO MRV results is displayed graphically in Figure 5 *(courtesy of Rohmax)* along with linear regression statistics. Pass/fail lines at the 60,000 mPa-s maximum limit have been placed on the graph for reference.

***Figure 5. ROBO Test Correlation to Sequence IIIGA***

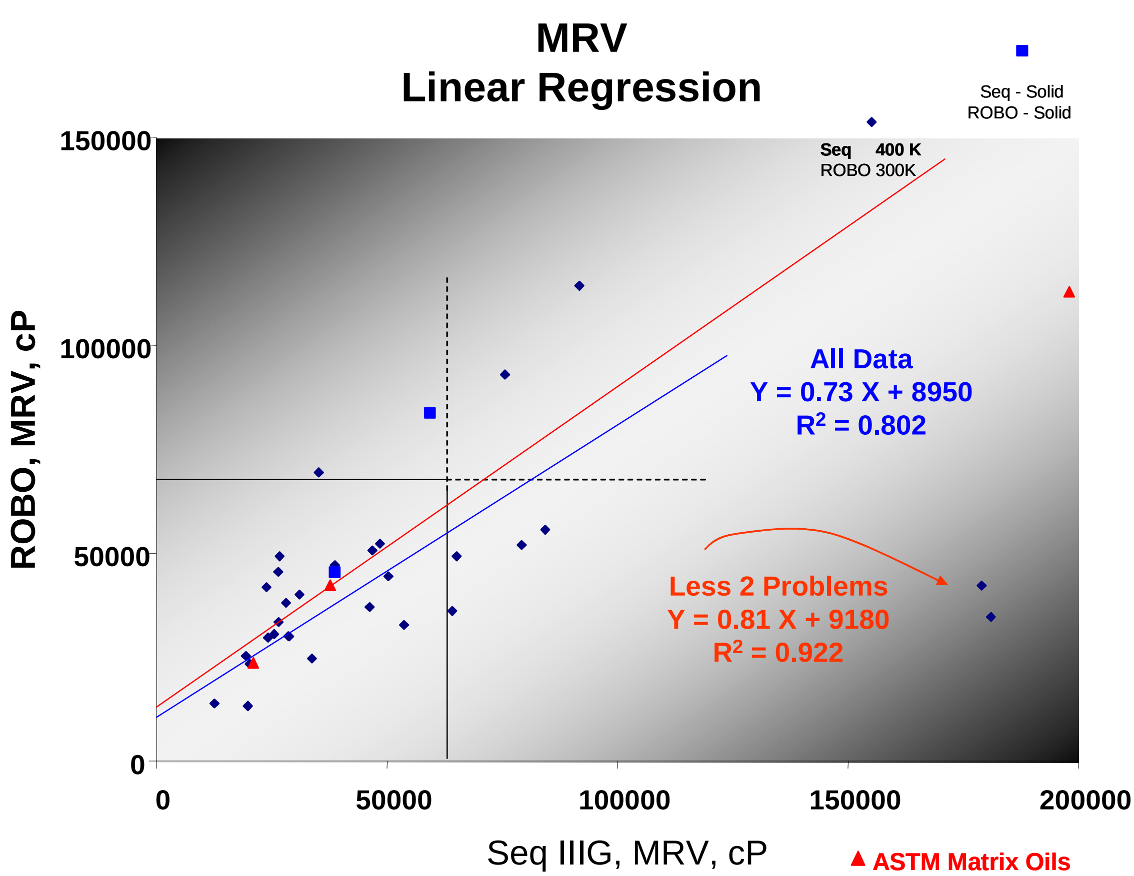



Figure 5 shows that the MRV data from 37 different engine oils run in both the Sequence IIIG test and the ROBO test appear to be reasonably correlated. These oils represent a broad range of engine oil technologies, indicating that no chemistry bias exists. In addition to replacing the Sequence IIIGA test, the ROBO test also has the capability to be an excellent screening tool for high temperature oxidation in the Sequence IIIG test, as measured by the percent kinematic viscosity increase at 40°C. In December 2006, ASTM established a ROBO task force under Subcommittee D02.09 which was charged with developing an official test method. Approximately seven laboratories are currently running the test.

**ILSAC Draft GF-5 Specification**

On February 21, 2007, ILSAC issued a draft GF-5 specification for industry comment. As of August 2007, no response from API or ACC has been received by ILSAC, as both organizations have refused to comment until the GF-5 Needs Statement has been finalized and approved by the ILSAC/Oil Committee. So the chess game continues between automakers and oil marketers. In the meantime, the draft GF-5 specification can be reviewed.

*Fuel Economy & Exhaust System Compatibility*

The two areas where all stakeholders agree that GF-5 improvements are necessary are fuel economy and exhaust system compatibility. For fuel economy improvement, the Sequence VID engine dynamometer test is currently under development, as described earlier in this paper, and ILSAC has proposed that the minimum FEI limits for GF-5 will be at least 0.5% higher than the GF-4 limits for comparable viscosity grades[25]. In addition, ILSAC has separated 0W-XX oils from 5W-XX oils in the draft specification, assigning different (higher) FEI limits for 0W-XX oils. Specifically, 0W-30 oils are expected to provide 0.3% higher FEI than 5W-30 oils, and 0W-20 oils are expected to provide 0.5% higher FEI than 5W-20 oils. The relationship between 5W-20, 5W-30, and 10W-30 oils will remain the same in GF-5 as in GF-4, that is, 5W-30 oils are expected to provide 0.7% higher FEI than 10W-30 oils and 5W-20 oils are expected to provide 0.5% higher FEI than 5W-30 oils.

With regard to exhaust system compatibility, the ESCIT group is currently in the process of selecting a phosphorus volatility test which will be recommended to the ILSAC/Oil Committee, as described earlier in this paper. Either a bench test (e.g. Selby Noack PEI-165) or an engine test (e.g. Sequence IIIG), or both, will be selected. ILSAC has proposed that the reduction in phosphorus volatility, as measured by these tests, will be equivalent to the reduction that could be achieved by reducing the phosphorus content of engine oils containing conventional ZDDP technology from 0.07% to 0.05%[25].

*Chemical Limits for GF-5*

Early in the ILSAC GF-4 engine oil development process, ILSAC indicated its desire to reduce the phosphorus content from 0.10% to 0.05%. Many companies invested heavily in the development of engine oil formulations containing 0.05% phosphorus, only to have to start over when the maximum phosphorus limit was set at 0.08% in the ILSAC GF-4 specification, mainly to ensure backwards serviceability of GF-4 oils in older vehicles still on the road. When ILSAC agreed to a 0.08% phosphorus maximum for GF-4, it was expected that it would return to a 0.05% phosphorus maximum for GF-5. However, late in the GF-4 development process, the Japanese Automobile Manufacturers Association (JAMA) expressed serious concern with engine oils containing 0.05% phosphorus that lead to excessive timing chain elongation caused by soot-induced wear in modern gasoline engines. In the field, modern gasoline engines can generate up to 2% soot in the engine oil[26]. Since it was too late in the GF-4 development process to develop an engine test method to provide protection against soot-induced wear and timing chain elongation, the most expedient solution was to set a minimum phosphorus requirement for ILSAC GF-4 oils at 0.06% phosphorus.

This minimum phosphorus level of 0.06% has carried over to GF-5 for protection against soot-induced wear. JAMA spent three years developing an engine test to measure soot-induced wear resulting in timing chain elongation, using 1% carbon black in the engine oil. In January 2006, ILSAC announced that JAMA had stopped development of the chain wear test due to poor test

[25] "Draft ILSAC GF-5 Standard for Passenger Car Engine Oils", February 21, 2007, page 5.
[26] Olree, R., Draft ILSAC/Oil Committee Meeting Minutes, Oct. 4, 2005, page 2.

reproducibility, but that it remains concerned about soot-induced wear with oils below 0.06% phosphorus content [27].

Recognizing that the only sure way to reduce exhaust catalyst poisoning from phosphorus is to reduce the phosphorus content of engine oil, ILSAC in its draft GF-5 specification has reduced the maximum allowed phosphorus content of GF-5 engine oils to 0.07% from the current GF-4 level of 0.08% P. This leaves a very narrow window for the formulation of GF-5 engine oils when using phosphorus-containing anti-wear compounds (e.g., ZDDP).

The 0.5% maximum sulfur content of GF-5 5W-20 and 5W-30 engine oils will remain the same as in GF-4, and ILSAC has proposed that the sulfur limit for 10W-30 oils be reduced to a maximum of 0.5% from the current GF-4 level of 0.7% .

*Engine Oil Robustness for Increased Engine Durability*

This third and final area of need for GF-5 improvements is the most controversial, as many of the new engine technologies included by ILSAC in the GF-5 Needs Statement are being questioned by the Oil members of the ILSAC/Oil Committee with regard their relevance to a core GF-5 specification (e.g., cam phasing and cylinder deactivation, turbocharging, and E85 capability). ILSAC has proposed five bench tests in the draft GF-5 specification to protect the engine hardware associated with these new technologies, which, although they are not part of the current ILSAC GF-4 specification, already have test methods developed. These bench tests and limits are shown in Table 1 below.

***Table 1. New GF-5 Bench Tests (with methods previously developed)***

| Test Method | Description | Parameter | GF-5 Limit | Comment |
|---|---|---|---|---|
| ASTM D 6894 | Aeration Resistance of Diesel Engine Oils | Aeration volume | 6% maximum | Known as EOAT in API CJ-4/CI-4 with 8% limit |
| ASTM D 6335 | High Temperature Deposits (TEOST 33C) | Deposit weight | 30 mg maximum | Last used in ILSAC GF-2 specification with 60 mg limit |
| ASTM D 1748 | Rust Protection in the Humidity Cabinet | Sandblasted panel after 100 hr exposure | No rust | Used for metal preservative oils |
| DCX procedure | Emulsion Retention of Oil Mixed with 10% Water, 10% E85 | 24 hour exposure at 0°C and 25°C | No separation. No precipitate when heated above 110°C | Chrysler procedure |
| SAE J2643 & ASTM D 7216 | Automotive Engine Oil Compatibility with Typical Seal Elastomers | Volume, Hardness, Tensile Strength, Elongation at Break, Tensile Stress at 50% Elongation | Varies by elastomer type; measured after 336 hr immersion | Includes nitrile, polyacrylate, silicone, and fluorocarbon rubber |

The EOAT test was selected to measure used oil aeration in order to protect new cam phasing and cylinder deactivation engine hardware. It is a diesel engine test and its relevance to gasoline engines has been questioned. General Motors initially looked at the Dexron® ATF Oil Aeration Test for this purpose, but determined that the test cannot measure aeration in used engine oil[28].

[27] Olree, R.,"Soot-Induced Wear", presented at ILSAC/Oil Committee Meeting, Jan. 11, 2006.
[28] Olree, R., Draft ILSAC/Oil Committee Meeting Minutes, Oct. 4, 2005, page 4.

The TEOST 33C test was selected to measure high temperature deposits in order to protect turbocharger bearings from coking. Photographs of water-cooled turbochargers, run on a dynamometer by Chrysler using two different oils with TEOST 33C deposit results of 45 mg and 65 mg, showed coking deposits on the turbocharger inlet and outlet. These engine oils were labeled as "Synthetic Blend" API SL/CF/GF-3 SAE 5W-20 and "All Synthetic, Turbo-Recommended" API CH-4/SL SAE 10W-30, respectively, and are considered performance failures by Chrysler. A third engine oil with API SM/GF-4 SAE 5W-20 credentials gave TEOST 33C deposit results of 22 mg and passed the dynamometer turbocharger coking test, although no photos were available from Chrysler. It was advised by Chrysler that 15% of ILSAC GF-4 engine oils do not pass the TEOST 33C at the proposed limit[29].

Two test methods added to GF-5 measure the protection against the corrosive nature of ethanol (E85) fuel, which can be used in vehicles manufactured with MFV capability. These tests are the ASTM D 1748 Rust Protection test and the DCX Emulsion Retention test. The DCX procedure is an in-house procedure that will be provided to the ILSAC/Oil Committee by Chrysler. The relevance of the ASTM D 1748 humidity cabinet test has been questioned.

*GF-4 Engine Tests in GF-5*

There are four existing GF-4 engine tests that will be carried over to GF-5. The ILSAC/Oil Committee is working to ensure that sufficient engine hardware will be available for these tests through the expected end of GF-5 in 2016. The GF-5 draft specification proposes pass/fail limit changes (severity increase) in two of these tests, as described in Table 2. These more severe limits are being questioned by the Oil members, who are unaware that current GF-4 oils may be deficient in these areas.

***Table 2. GF-4 Engine Tests carried over to GF-5 with increased severity***

| Test Method | Description | Parameter | GF-4 Limit | GF-5 Limit |
|---|---|---|---|---|
| Sequence IIIG (ASTM D 7320) | High Temperature Wear and Oil Thickening | WPD | 3.5 min. merits | 5.0 min. merits |
| Sequence VG (ASTM D 6593) | Sludge and Varnish | - AES<br>- ARCS<br>- Oil Screen Sludge | 7.8 min. merits<br>8.0 min. merits<br>20% area max. | 8.3 min. merits<br>8.5 min. merits<br>5% area max. |

ILSAC considers the proposed improvements in deposit and sludge performance in the Sequence IIIG and VG tests as vital in order to contend with the environment created in modern engines by higher power densities, higher speeds, extended oil change intervals practiced by many owners, and concerns about low temperature sludge associated with current market oils[30].

The draft GF-5 specification proposes no changes in Sequence IV-A (D 6891) Average Cam Wear limits or Sequence VIII (D 6709) Bearing Weight Loss limits. Also, for the first time in nearly two decades, no increase in Sequence III high temperature oxidation severity (PVIS) has been proposed by ILSAC.

At the first GF-5 ILSAC/Oil Committee meeting in January 2005, ILSAC announced that the Sequence IIIG engine test would be replaced by a new Sequence IIIH test being developed by General Motors using their new ECO TECH 4-cylinder engine. Work on the Sequence IIIH test was, however, abandoned in September 2006.

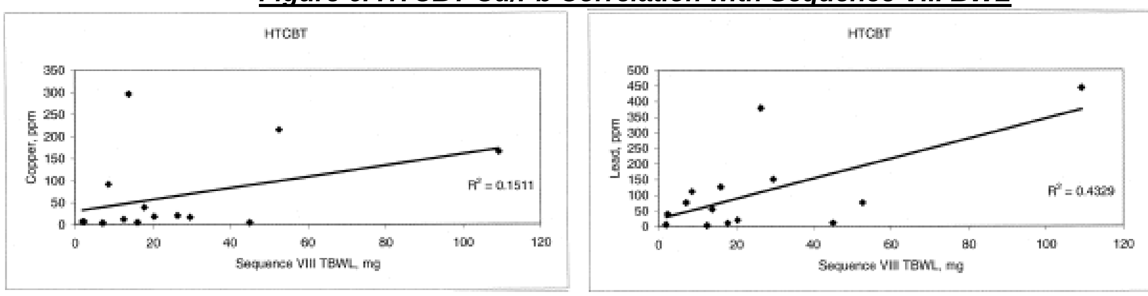
One of the original desires of ILSAC for GF-5 was to replace the Sequence VIII Cu/Pb Bearing Corrosion engine test with a bench test because of the problem of lead leaching from the

[29] Olree, R., Draft ILSAC/Oil Committee Meeting Minutes, April 12, 2006, pp. 1-2.
[30] Murray, H., "ILSAC GF-5 Needs Statement", presented at ILSAC/Oil meeting on June 29, 2006.

Sequence VIII test bearings despite the fact that new bearings are stored in an EF411 (Group I) bath under an argon blanket[31]. Significant lead leaching approaches the 25 mg limit. The Sequence VIII Surveillance Panel subsequently changed the storage fluid from Group I to Group IV (PAO), but this appears not to have solved the problem[32].

The Alliance of Automobile Manufacturers funded a study to measure the correlation of two copper/lead corrosion bench tests against the Sequence VIII engine test. Data evaluating the results of 14 engine oils with regard to copper and lead corrosion in ASTM D5968 (CBT) and ASTM D6594 (HTCBT), and the Sequence VIII engine test Bearing Weight Loss (BWL), were reviewed in April 2005 (see Figure 6).

***Figure 6. HTCBT Cu/Pb Correlation with Sequence VIII BWL***




The limited analysis gave a low $R^2$ value, indicating poor correlation for both CBT and HTCBT with the Sequence VIII test. Based on the results, ILSAC determined that the Sequence VIII test will be the test requirement for bearing corrosion until 2016. GF-5 performance requirements will likely require higher levels of friction modifiers, which can corrode copper/lead bearings. As a result, the Sequence VIII corrosion test will become more important in GF-5.

*GF-4 Bench Tests in GF-5*

In addition, there are 11 existing GF-4 bench tests that will be carried over to GF-5. The GF-5 draft specification proposes the following pass/fail limit changes (severity increases) in two of these tests as described in Table 3.

***Table 3. GF-4 Bench Tests carried over to GF-5 with increased severity***

| Test Method | Description | Parameter | GF-4 Limit | GF-5 Limit |
|---|---|---|---|---|
| TEOST MHT-4 (ASTM D 7097) | High Temperature Deposits | Deposit Weight | 35 mg maximum | 30 mg maximum |
| ASTM D 892 | Fresh Oil Foaming | Settling time | 10 minutes | 1 minute |

Nine other bench tests will be carried over to GF-5 from GF-4 with no change in limits:

- SAE J300 Fresh Oil Viscosity requirements
- Gelation Index, ASTM 5133
- Evaporation Loss, ASTM D 5800
- Filterability, EOWTT, ASTM D 6794
- Filterability, EOFT, ASTM D 6795
- Fresh Oil High Temperature Foaming, ASTM D 6082 (Option A)
- Shear Stability, ASTM D 6709
- Homogeneity and Miscibility, ASTM D 6922
- Engine Rusting, Ball Rust Test, ASTM D 6557

[31] Olree, R., Draft ILSAC/Oil Committee Meeting Minutes, April 14, 2005, page 1.
[32] Mazzamaro, G., ASTM D.02.B.01 Passenger Car Engine Oil Classification Panel minutes, June 19, 2007, page 2

## GF-5 Formulation Challenges

Based on the draft GF-5 specification issued by ILSAC, the level of engine oil performance proposed for GF-5 oils is known. In order to formulate an engine oil that can pass all engine and bench tests at the proposed GF-5 limits, a formulator will select additives that are known to be beneficial in one or more performance tests. In many cases, these additives can be antagonistic in other performance tests. When this occurs, other additives may be added or rebalanced in order to compensate for the antagonistic effect. Some examples of these formulation challenges for GF-5 oils are discussed below.

*Fuel Economy and Friction Modifiers*

A formulator may choose to increase the friction modifier level in order to achieve the required fuel economy increase in the Sequence VID engine test. Many friction modifiers are antagonistic towards Cu/Pb bearing corrosion in the Sequence VIII engine test[33]. Certain organic, vegetable oil-derived friction modifiers are aggressive towards lead[34]. This effect will be exacerbated in GF-5 if ZDDP is lowered from 0.075% to 0.065%, as ZDDP is a good metal protectant and helps to protect Cu/Pb bearings from corrosion. In this case, other additives can be used to passivate the Cu/Pb surface[35,36]. Certain molybdenum friction modifiers can also be corrosive but, unlike organic friction modifiers, molybdenum compounds also bring strong oxidation, wear and deposit control benefits to the formulation[37].

*Exhaust Catalyst Compatibility and ZDDP*

Reducing catalyst poisoning by reducing the amount of phosphorus that reaches the catalyst will likely be accomplished in GF-5 in two ways: by lowering the maximum phosphorus content and by introducing a phosphorus volatility limit. This means using less ZDDP and shifting from mostly short chain, secondary ZDDP to more long chain, primary ZDDP. It is well known that short chain, secondary ZDDPs are more active as anti-wear compounds than long chain, primary ZDDPs in gasoline engines[38]. By reducing the total ZDDP and shifting to a less active type, one would expect that the oil's anti-wear performance will be compromised. This can be compensated for by the use of supplemental anti-wear additives which do not contain phosphorus. Molybdenum and boron compounds are good examples[39], as well as sulfur compounds like dithiocarbamates[40].

*Piston Deposit Control and Detergents*

The ILSAC proposal to increase the minimum weighted piston deposit rating in the Sequence IIIG engine test from 3.5 merits to 5.0 merits for GF-5 oils represents a significant performance improvement, which will most likely be achieved through the use of additional metallic detergents and/or detergent rebalancing. Because of the strong surface activity of metallic detergents like calcium and magnesium sulfonates, these additives will compete with other surface active (polar) compounds on the metal surface. When the other polar compounds are friction modifiers, an antagonistic effect can be expected in the Sequence VID fuel economy test[41]. In order to minimize the negative effect on fuel economy, additional detergents must be carefully selected and balanced. The deposit-controlling properties of detergents can be supplemented by the use of additional phenolic antioxidants[42] and organo-molybdenum compounds[43]. Both of these

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[33] Olree, R., Draft ILSAC/Oil Committee Meeting Minutes, April 14, 2005, page 1.
[34] Chasan, D.E., and Difrancesco, S., U.S. Patent Application 20040038835, February 26, 2004.
[35] Stachew, C.F. and Shaklin, J.R., U.S. Patent 6,010,986, January 4, 2000.
[36] Waddoups, M., Hartley, R.J., and Miyoshi, T., U.S. Patent 6,333,298, December 25, 2001.
[37] Karol, T.J., U.S. Patent 5,137,647, August 11, 1992.
[38] Fujitsu, T., Kubo, K., and Nagakari, M., U.S. Patent 6,114,288, September 5, 2000.
[39] Karol, T.J. and Donnelly, S.G., U.S. Patent Application 20040138073, July 15, 2004.
[40] Nakazato, M., Magarifuchi, J., Mochizuki, A., and Tanabe, H., U.S. Patent 6,531,428, March 11, 2003.
[41] Boffa, A., U.S. Patent 6,790,813, September 14, 2004.
[42] Kristen, U., Muller, K, and Rasberger, M., U.S. Patent 5,523,007, June 4, 1996.
[43] Gatto, V.J., U.S. Patent 6,914,037, July 5, 2005.

additives have deposit control as well as fuel economy benefits, either by reducing the increase in used oil viscosity and/or by reducing friction.

*Engine Sludge Control and Dispersants*

The ILSAC proposal to increase the minimum sludge ratings in the Sequence VG engine test for GF-5 oils also represents a significant performance improvement, which will most likely be achieved by a higher dispersant dosage and/or more active dispersant types. Ordinarily, this would not be an issue, but the draft GF-5 specification has added an elastomer compatibility test which involves elastomer immersion in the test oil at high temperature for 2 weeks. The active nitrogen in most dispersants is aggressive towards fluorocarbon rubber elastomer (e.g., Viton®)[44]. This will likely affect which dispersant, or how much, is used to improve sludge performance in GF-5, although less ZDDP will be helpful in reducing negative effects on Viton seals.. The dispersancy retention of engine oils can be improved by the addition of organo-molybdenum compounds and ashless antioxidants[45].

*High Temperature Deposits and Antioxidants*

There are two TEOST bench tests in the draft GF-5 engine oil specification, the TEOST MHT-4 test and the TEOST 33C test. Chrysler believes that the TEOST MHT-4 test simulates high temperature piston deposits in one of their critical engines. The test involves oil and air flowing down a vertical hot metal rod at 285°C for 24 hours. ILSAC has proposed a decrease in the maximum deposit weight in the TEOST MHT-4 test from 35 mg in GF-4 to 30 mg in GF-5. The test responds well to antioxidants[46] and will require an increased level of these additives (e.g., alkylated diphenylamines, hindered phenols, sulfur compounds and/or organo-molybdenum compounds), especially if less ZDDP is used as the maximum phosphorus limit reduced.

Chrysler believes that the TEOST 33C test simulates the formation of coking deposits in the turbocharger bearings of one of their critical engines. This test involves oil and air flowing up a vertical rod for 2 hours in the presence of iron and nitrous oxide catalysts, with 12 temperature cycles between 200°C and 480°C. In this test, organo-molybdenum compounds, generally known as excellent antioxidants in lubricants, are antagonistic when used at high levels (above 500 ppm of molybdenum). Further studies of these formulation effects will be the subject of a future technical paper.

**Author's Note**

After reviewing the key formulation challenges expected with ILSAC GF-5, it is interesting to note that the use of organo-molybdenum compounds is a recurring theme, as they are beneficial for fuel economy, wear control, deposit control, oxidation control, and dispersancy retention. For companies that manufacture and market molybdenum and organo-molybdenum compounds, ILSAC GF-5 is indeed better late than never. 02/2008

[44] Fenoglio, D.J., Vettel, P.R., and Eggerding, D.W., U.S. Patent 5,080,815, January 14, 1992.
[45] Gao, J.Z., Fyfe, K.E, Elnicki, J.D., U.S. Patent 6,150,309, November 21, 2000.
[46] Esche, C.K, Gatto, V.J., Lam, W.Y., U.S. Patent 6,599,865, July 29, 2003.

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