FINAL TECHNICAL REPORT

POLYALKYLENE GLYCOL (PAG) BASED LUBRICANT FOR LIGHT & MEDIUM DUTY AXLES

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EXECUTIVE SUMMARY

The axle lubricant (SAE 75W-140) widely used in the market place over the last few years is primarily formulated from polyalpha olefin (PAO) basestock. This investigation focused on polyalkylene glycol (PAG) base stock which is significantly different chemically from PAO and because of their polar nature it is hypothesized that it can adsorb relatively easily on contacting surfaces resulting in significant friction reduction and improve fuel economy. Axle efficiency is generally quite high at high torque range but it could be quite low at low torque range representative of EPA drive cycles. Therefore, this project primarily focus on improving axle efficiency in the low torque range while maintaining all durability attributes.

The objective of this project is to develop novel lubricant formulations that are expected to improve the fuel efficiency of light, medium, heavy-duty, and military vehicles by at least 2% over Society of Automotive Engineers (SAE) 75W-140 axle lubricants (improvement based on comparative results from engine dynamometer testing, chassis dynamometer testing or test track, e.g., SAE J1321) without adverse impacts on vehicle performance or durability.

The proposed research project followed a structured approach beginning with lubricant formulations by varying base oil chemistry and additive components followed by physical property characterization. Lubricant performance evaluations started with simple laboratory friction, wear, oxidation, and corosion tests which guided into identification of performance areas requiring improvements. This required an iterative lubricant reformulation. Once a few acceptable formulations were identified, they are evaluated in more complex component tests including ASTM tests (L-37 - load carrying capacity under low speed and high torque, L42 – load carrying properties under high speed and shock loading, L-60 - thermal and oxidative stability, and L-33-1 moistre corrosion resistance). Two issues were observed following L-42 tests; toxicological and foamimg. This required reformulation and repeating all ASTM tests. During repeat tests it was observed that some of the additives precipitated out of solution. Since it was discovered quite late in the program, and the time it may need to find a solution to this issue, the team changed direction of the program to switch to use of an oil soluble PAG as a co-basestock (15-30%) with mineral oil used in a commercial formulation (SAE 75W-85). This formulation strategy helped passing all ASTM tests. This was followed by Ford proprietary axle efficiency, gear wear, and chassis roll dynamometer tests for fuel economy evaluations. Axle efficiency was improved upto 7% when a full PAG formulation was used but no such significant improvement was observed with PAG as co-basestock formulation. Chassis roll fuel economy improved by 1.9% with PAG co-basestock formulation (conforming to SAE 75W-85) compared to SAE 75W-140 baseline. However, it was observed that the improvement is mostly due to reduced viscosity of candidate formulation when compared to a full mineral oil based SAE 75W-85 formulation. PAG as co-basestock also exhibited unacceptable wear in proprietary gear wear test.

This investigation highlighted challenges with PAG formulations including identification of additve components that stay in PAG solution for intended duration, and meeting component durability requirement.

Introduction

The axle lubricant (SAE 75W-140) widely used in the market place over the last few years is primarily formulated from polyalpha olefin (PAO) basestock. This investigation focused on polyalkylene glycol (PAG) base stocks which are significantly different chemically from PAO and because of their polar nature it is hypothesized that they can adsorb relatively easily on contacting surfaces resulting in significant friction reduction and improved fuel economy. Axle efficiency is generally quite high at high torque range but it could be quite low at low torque range representative of EPA drive cycles. Therefore, this project primarily focuses on improving axle efficiency and thus fuel economy in the low torque range while maintaining all durability attributes.

Technology Background

Polyalkylene Glycol-based axle lubricants

Gear lubricants for use with hypoid gears such as those found in axles consist of a base stock and additives. The base stock comprises the majority of the lubricant. The base stock is typically a hydrocarbon oil and can be selected from API Groups II, III or IV, although occasionally API Group V base stocks such as esters are used. The chemistry of the base stock impacts its physical properties and therefore the overall performance of the formulated gear lubricant. Base stock chemistry impacts oxidative stability, viscosity index, pressure-viscosity coefficient, elastohydrodynamic film thickness, frictional properties, additive solubility, elastomer compatibility and low temperature viscosities.

Additives used in gear lubricants improve the performance of the base stock in a variety of areas. Oxidative stability is improved by the addition of aminic and phenolic anti-oxidants that act as free radical scavengers. Sulfur containing compounds can act as secondary anti-oxidants by decomposing organic peroxides. Anti-wear additives based on phosphorus containing compounds, such as phosphate esters, thiophosphate esters or amine phosphates, reduce wear by the formation of iron phosphate layers under contact pressure and temperature conditions found in the tribo-contact. Sulfur containing compounds are normally added to provide wear protection in boundary lubrication conditions. These are known as EP additives and are based on sulfurized olefins, sulfurized isobutylene, or sulfurized fatty acids. The effectiveness of the sulfurized EP additive is normally a function of the active sulfur found in the material, with higher levels of active sulfur resulting in higher EP loads before failure. ZDDP is also an anti-wear, secondary anti-oxidant component that can be used in gear lubricants. Corrosion inhibitors are required to protect ferrous and yellow metals. Typical yellow metal (copper) inhibitors are tolytriazole, benzotraizole, alkylated tolytriazole or mercaptobenzotriazole. Iron corrosion inhibitors include amines, overbased detergents, neutral or slightly basic salts of alkylated sulfonated aromatics, amine salts of borates and phosphates. Viscosity modifiers can be added to improve viscosity index, while pour point depressants can be added to improve low temperature properties. Other additives, such as anti-foams and fiction modifiers, can also be used.

The formulation of a gear oil can be divided into two steps. The first is the choice of base stock. The focus of this work will be on polyalkylene glycol base stocks. The second, and far more difficult step, is the selection of additives and concentration of additives required to meet the performance specifications for the gear lube. The Ford Motor company Engineering Material Specification *LUBRICANT, GENERATION III, FUEL EFFICIENT HYPOID GEAR LUBE WSS-M2C942-A* provided the primary performance requirements for this work and *ASTM D7450 Standard Specification for Performance of Rear Axle Gear Lubricants Intended for API Category GL-5 Service* provided additional industry wide performance requirements that must be met for a viable gear lube.

Each of the specifications has test requirements that use well defined test methods, such as ASTM D130 for copper corrosion, with very specific results, better than 2A in this case. System level tests required by the specifications, like the L-37 gear durability test, are well defined and have very specific pass/fail results, but are expensive and time consuming to run. To avoid needless expense, bench tests were developed to predict gear wear, oxidative stability and other properties measured by system level tests. Performance of experimental PAG-based lubricants were compared to commercially available SAE 75W-85 and SAE 75W-140 gear oils meeting the Ford specification in various bench tests. It was assumed that if the experimental fluid had equivalent or better performance than the commercial lubricants in a particular bench test, it would perform well in the corresponding system level test. Unfortunately, it was not always the case that the bench tests were predictive of performance in the system level test, requiring development of new bench tests that were more predictive of performance in the system level test. This was particularly true of the L-37 and L-42 tests.

When a fluid fails a system level test it is an indication that the additive package was not robust enough to protect the system components. This necessitated a change in the fluid formulation to pass the system level test. This typically involved adding or changing additive types or concentrations in an attempt to improve performance, wear for example, while not negatively impacting oxidative stability, corrosion, friction or other properties. Thus, formulation development was an iterative process consisting of performing system level test; if a fail, analyzing failed components to identify the failure mode; developing new bench test; proving that new bench test discriminates between good and bad oils; using new bench test to guide formulation revision; repeating other bench tests to insure no negative impacts on other performance characteristics; repeating system level test. Then to the next system level test, where a failure would trigger another formulation iteration.

This report contains a summary of PAG-based and mineral oil plus oil soluble PAG-based gear lubricant formulation development, focusing on lubricants evaluated in system level tests and omitting information on most of the intermediate formulations evaluated in bench tests during development.

Scope

The project contained three (3) budget periods; starting from October 2013 to September 2016. However, it was extended to March 31, 2018 to meet project deliverables.

Budget Period 1: Lubricant Formulation
In this first budget period work initiated with the formulation of new polyalkylene glycol (PAG) based axle lubricants. Preliminary testing conducted in an effort to identify those formulations with the greatest potential to meet project objectives.

Budget Period 2: Bench Testing
Second budget period focused on evaluation of fundamental properties of the new lubricant formulations, testing to make intrinsic property measurements, physical tests to determine performance characteristics in sliding and rolling friction and wear, micropitting, and analysis of lubricant additive-derived antiwear films using surface sensitive analytical tools will continue. In addition, oxidation and corrosion evaluations were performed.

Budget Period 3: System and Vehicle Evaluation
Performance characteristics were made in system level (axle) test rigs to evaluate axle efficiency improvements and gear/bearing durability performance of lubricants. Vehicle fuel economy was demonstrated on a chassis roll dynamometer.

ACCOMPLISHMENTS
Accomplishments are described under each of the key tasks outlined in the project summary above. Although project management efforts were present throughout, this report will focus on technical achievements. The technical areas include lubrication formulations, and different stages of friction and wear testing (i.e., bench, component and engine).

1. PROJECT MANAGEMENT
Project management activities were completed in accordance with the Federal Reporting Checklist as well as special requests such as Annual Merit Review and on-site meetings.

2. ACCOMPLISHMENTS

2.1 Experimental Methods

2.1.1 Viscosity Measurements:

- Viscosity of used fluids were measured with a glass capillary viscometer following ASTM D445 *Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)*. Used samples were filtered through a 5 micron syringe prior to measuring the viscosity.

- Low shear viscosities of fresh fluids were measured on an Anton Parr Stabinger SVM 3000 viscometer following ASTM D 7042 *Standard Test Method for Dynamic Viscosity and Density of Liquids by Stabinger Viscometer*.

- Low temperature viscosities were measured on a Brookfield DV-III Ultra rotational viscometer using the small sample adaptor and spindle/speed combination appropriate for the measured viscosity, ie torque readings of ~ 50% of scale.

- Viscosity Index was calculated per ASTM D 2270 *Standard Practice for Calculating Viscosity Index from Kinematic Viscosity at 40 and 100°C.*

- Fluid shear stability was measured per CEC L-45-T-93 *Viscosity Shear Stability of Transmission Lubricants (Taper Roller Bearing Rig).*

2.1.2 Corrosion Measurements

- Copper corrosion was measured per ASTM D 130 *Standard Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test*. Test conditions were 3h at 121 °C

- Ferrous metal corrosion was evaluated per ASTM D665 *Standard Test Method for Rust-Preventing Characteristics of Inhibited Mineral Oil in the Presence of Water,* Procedure A.

2.1.3 Friction and wear measurements

- 4-ball wear measurements were made with a Falex Multi-specimen instrument per ASTM D 4172 *Standard Test Method for Wear Preventive Characteristics of Lubricating Fluid (Four Ball Method)*. Test conditions used were 40 kg load, 600 rpm for 2 hours at 100 °C.

- 4-ball EP measurements were made per ASTM D-2783 *Standard Test Method for Measurement of Extreme-Pressure Properties of Lubricating Fluids (Four-Ball Method).*

- Pin and vee EP measurements were made per ASTM D 3233 *Standard Test Methods for Measurement of Extreme Pressure Properties of Fluid Lubricants (Falex Pin and Vee Block Methods)* Method A.

- Ball on disc wear and friction measurements were made with a PCS Instruments MTM2 Mini-Traction Machine using a disc and ball made of AISI 52100 steel (760 HV) with surface roughness Ra better than 0.01 micron. Test conditions were: load of 41 N (1 GPa max contact pressure); speed of 100 mm/s; SRR = 0 (pure sliding); temperature of 135 °C for 4 h.

- Pin on disk wear volume measurements were made using test conditions of 100 °C with a load of 10 N (1.05 GPa) at an entrainment speed of 100 mm/s using a 3/8" AISI 52100 steel ball with a smooth finish and a AISI 52100 steel disk with a smooth finish.

- Stribeck curves were measured with a PCS Instruments MTM2 Mini-Traction Machine using a disc and ball made of AISI 52100 steel (760 HV).with surface roughness Ra better than 0.01 micron. Test conditions noted in report.

- Wear by SRV was measured per ASTM D5707 *Standard Test Method for Measuring Friction and Wear Properties of Lubricating Grease Using a High-Frequency, Linear-Oscillation (SRV) Test Machine*. Test conditions were 400 N load at 80 °C, 50 hz, 1mm stroke for 2 h.

- Extreme pressure (EP) loads were measured per ASTM D6425 *Standard Test Method for Measuring Friction and Wear Properties of Extreme Pressure (EP) Lubricating Oils Using SRV Test Machine* at a test temperature of 80 °C.

2.1.4 Oxidative stability

- Oxidative stability was evaluated in a test apparatus consisting of a 500 mL 3 neck flask fitted with a water cooled condenser, thermocouple and an air sparge. Flask was heated with a mantle and the upper portion insulated, Figure 1. A cooper steel-coil was placed in the liquid and 300 mL of fluid were added. Test temperature was 162 °C for a duration of 50 h. Fluid samples were taken periodically during the test. TAN, kinematic viscosity at 100 °C and mass loss of the fluid were measured and observations of deposits and corrosion of the copper-steel coil and deposits on the glass flask were made. The conditions were chosen to mimic those found in ASTM D5704 *Standard Test Method for Evaluation of the Thermal and Oxidative Stability of Lubricating Oils Used for Manual Transmissions and Final Drive Axles* (L-60 test).

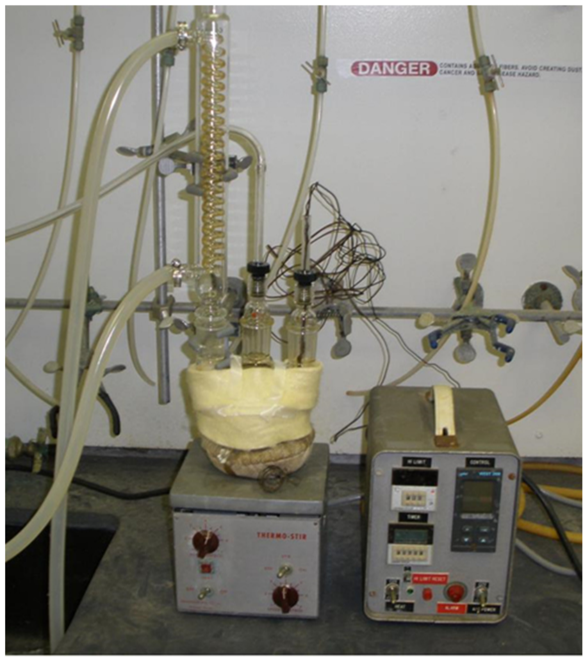

Figure 1 Oxidation test unit

2.1.5 Axle tests

- ASTM D5704 Standard *Test Method for Evaluation of the Thermal and Oxidative Stability of Lubricating Oils Used for Manual Transmissions and Final Drive Axles* (L60-1). Measures the change in viscosity, deposit forming tendencies of axle oils when subjected to temperatures of 163 °C in the presence of air and a copper catalyst for 50 h.

- ASTM D6121 *Standard Test Method for Evaluation of Load-Carrying Capacity of Lubricants Under Conditions of Low Speed and High Torque Used for Final Hypoid Drive Axles* (L-37). Evaluates the load carrying, wear and extreme pressure properties of an axle lubricant under test conditions of 24 h at 80 wheel r/min, 1740 lb.-ft. (2359 N-m) torque per wheel and an axle sump temperature of 275°F (135°C).

- ASTM D 7452 *Standard Test Method for Evaluation of the Load Carrying Properties of Lubricants Used for Final Drive Axles, Under Conditions of High Speed and Shock Loading* (L-42). Measures the anti-scoring properties of an axle lubricant when subjected to high speed and shock conditions.

- ASTM D7038 *Standard Test Method for Evaluation of Moisture Corrosion Resistance of Automotive Gear Lubricants (L-33)*. A test procedure for evaluating the rust and corrosion inhibiting properties of a gear lubricant while subjected to water contamination and elevated temperature in a bench-mounted hypoid differential housing assembly.

2.1.6 Axle efficiency tests

- Axle efficiency test was conducted in Ford proprietary test method using a production 8.8" conventional differential axles with 3.31 drive ratio under a variety of load speed combinations representative of FTP cycles.

2.1.7 Gear wear test

- Gear wear tests was conducted using a production axle. The test duration was 44 hours. Following tests, the gear contact pattern was evaluated. The test is representative of gear durability for the life of the axle.

2.1.8 Chassis roll dynamometer test

- The test was conducted with a human driver to assess the fuel economy benefit from one candidate fluid and two reference fluids using FTP city, highway and combined drive cycles.

2.1.9 Other fluid property measurements

- Total acid number (TAN) measured per ASTM D-664 *Standard Test Method for Acid Number of Petroleum Products by Potentiometric Titration using a Mettler Toledo DL-15 Autotitrator*.

- Foaming measured by ASTM D 892 *Standard Test Method for Foaming Characteristics of Lubricating Oils* and ASTM D6082 *Standard Test Method for High Temperature Foaming Characteristics of Lubricating Oils.*

- Fluid compatibility with FKM seal material determined following ASTM D471 *Standard Test Method for Rubber Property—Effect of Liquids.* See ASTM D1418 *Standard Practice for Rubber and Rubber Lattices—Nomenclature* for complete definition of FKM.

- XPS (X-ray photoelectron spectroscopy) measurements of gear sections were performed using a Thermo K-Alpha instrument using an Al Kα emission source. For depth profiling, an argon sputter beam accelerated to 1000 keV was used to etch a 2mm x 1mm region.

2.2 Base stock selection

Polyalkylene glycols have an inherently high VI, eliminating the need for a viscosity modifier. Polyalkylene glycols also have good low temperature properties, thus no pour point depressant is required. The elimination of the need for these high molecular weight polymeric additives allows the viscosity of the base stock to be chosen such that it is relatively close to the desired viscosity of the final formulation. It was desired that the gear lubricant meet the viscometric requirements of a SAE 75W-85 gear oil: 100°C viscosity between 11.0 mm^2/s and <13.5 mm^2/s with a -40 °C viscosity < 150,000 mPa s (1). It was also desired that the base stock have the highest VI to take advantage of the potential for fuel efficiency improvements due to lower viscosities at lower operating temperatures vs the benchmark lubricants.

Several PAG chemistries were considered, Table 1. EO/PO copolymers are water soluble, PO homopolymers are water and oil insoluble and PO/BO copolymers are oil soluble. Capping (2) any PAG base stock increase VI, improves low temperature properties and increases oil solubility. Diol initiated PAGs have high VI but poor low temperature properties.

Fuel efficiency improvements were also expected to come from lower friction in elastohydrodynamic and mixed lubrication contacts. Work done by Gangopadhyay et al. with PAG-base engine lubricants (3) showed that EO/PO copolymers had lower coefficients of friction in engine tribo contacts than other PAG chemistries and GF-5 SAE 5W-20 motor oils. This finding was in agreement with work by other researchers investigating the impact of PAG chemistry on friction in elastohydrodynamic and mixed lubrication contacts. These findings indicated that a preference should be given to EO/PO copolymers.

Table 1 PAG base stock chemistries

Fluid	Chemistry
PAG A	Alcohol initiated PO homopolymer
PAG B	Alcohol initiated PO/BO copolymer
PAG C	Proprietary blend of polymers
PAG D	Alcohol initiated EO/PO copolymer
PAG E	Alcohol initiated EO/PO copolymer
PAG F	Alcohol initiated EO/PO copolymer
PAG G	Diol initiated EO/PO copolymer
PAG H	Capped alcohol initiated PO homopolymer
PAG I	Capped alcohol initiated PO/BO copolymer
PAG J	Proprietary blend of polymers

PAG base stocks are manufactured by polymerization with the relevant oxide monomers to a target viscosity or molecular weight. It is relatively easy to manufacture PAG base stocks with a specific 100 °C viscosity or, alternatively, to blend two PAG base stocks to meet a viscosity specification. The viscometric of the PAG base stocks in Table 1 can be found in Table 2. Of the base stocks considered, PAG E had the desired EO/PO copolymer chemistry and the highest VI. PAG E was taken forward for shear stability testing. It was not anticipated that any of the PAG base stocks would suffer any viscosity degradation in the KRL shear stability test. The KRL shear stability test measures the shear stability of high molecular weight polymeric additives. These would not be present in any PAG formulation, thus it was felt that measurement of the shear stability of all the potential PAG base stocks would not be necessary.

Table 2 Viscometrics of base stock chemistries

Fluids	**Viscosity @ 100 °C mm²/s**	**Viscosity @ 40 °C mm²/s**	**Viscosity Index**	**Viscosity @ -40 °C mPa s**
PAG A	10.9	56.4	190	99400
PAG B	11.3	65.2	168	94000
PAG C	12.0	64.6	186	98861
PAG D	11.0	51.5	214	48100
PAG E	12.0	43.4	216	52800
PAG F	7.6	33.3	197	22900
PAG G	11.8	66.3	176	solid
PAG H	11.9	55.3	218	44500
PAG I	12.0	61.8	194	--
PAG K	11.7	54.4	216	--

Table 3 KRL shear stability at 150 °C for 20 h

Fluids	**Initial Viscosity mm²/s**	**Sheared viscosity mm²/s**	**Duration, h**	**Viscosity loss %**
75W-140	24.99	25.02	20	-0.12
75W-85	11.61	12.14	20	1.89
PAG E[1]	12.17	12.14	20	0.25

[1] contained antioxidant

The KRL results are in Table 3. The viscosity after 20 h was above the 10 mm^2/s required by the Ford specification. Since PAG E met all of the desired criteria for a base stock, it was selected as the base stock for formulation development.

3. RESULTS AND DISCUSSION

3.1 Formulation development with PAG E

Initially three formulation approaches were pursued. The formulation development focused on different combinations of anti-wear and extreme pressure additives to meet durability requirements. PAG E, anti-oxidants, and anti-foams were common to all three formulations. The viscosity of PAG E was adjusted to account for the viscosity contribution of the additives to the formulations. Formulation 1597-54-6 contained only phosphorus-based anti-wear additives; 2484-17-2 contained phosphorus anti-wear and sulfur containing EP additives and 2484-16-1 contained a zinc dialkyldithiocarbamate mixture that would act as both an anti-wear and EP additive. Fluids 1597-54-6 and 2484-16-1 contained a triazole copper inhibitor. The active sulfur in the EP additive used in fluid 2484-17-2 required the use of a sulfur scavenger in addition to the triazole copper inhibitor to obtain satisfactory copper corrosion test results.

Viscometrics of 2484-17-2 and 2484-16-1 met the viscosity requirements for a SAE 75W-85 gear oil with much higher VI than the benchmark SAE 75W-85 gear oil. The -40 °C viscosities of the PAG gear oil were essentially equivalent to the benchmark SAE 75W-85, Table 4.

Table 4 Fluid viscosities

	75W-85	**75W-140**	**2484-17-2**	**2484-16-1**
Viscosity @ 100 °C, mm^2/s	11.7	25	12.0	12.2
Viscosity @ 40 °C, mm^2/s	67.9	181.9	59.4	60.5
VI	168	170	203	204
Viscosity @ -40 °C, mPa s	66800	150000	68657	~70,000

Wear and EP measurements were made on the three fluids as well as the two benchmark gear oils. The PAG base oils gave 4-ball and SRV wear scars equivalent to or better than the benchmark oils, Table 5. Falex EP and 4-ball EP for 2484-17-2 were equivalent to or better than the benchmark oils and superior to the other two PAG lubricants. The SRV EP loads of 2484-17-2 were equivalent to the benchmark oils and lower than the EP load for 2484-16-1.

Table 5 Summary of tribology data

Test	**75W-85**	**75W-140**	**2484-17-2**	**2484-16-1**	**1597-54-6**
4 ball wear, mm	0.92	0.42	0.52	0.4	0.52
4 ball EP, kg	340	420	400	240	126
Falex EP, lb	2505	2807	3652	1850	2243
SRV wear, mm	0.75	0.72	0.75	0.57	--
SRV friction coefficient @ 2 h	0.129	0.128	0.102	0.103	--
SRV EP, N	500	600	600	900	--

The corrosion prevention and oxidative stabilities of the gear lubricants were measured. All three PAG-based lubricants satisfied the copper corrosion and rust preventative requirements. In the oxidation test, the PAG based lubricants had smaller Δviscosity and smaller ΔTAN than the benchmark oils, Table 6. The deposits and residues formed by 2484-17-2 on the glass flask and copper/steel coil, Figure 2, appeared to be less than those formed by the benchmark oils, Figure 3 and Figure 4.

While 1597-54-6 gave satisfactory performance in the corrosion and oxidation tests, the lower 4-ball EP loads than 2484-17-2 and 2484-16-1 suggested that the additive package was not robust enough and that a sulfur containing EP additive was likely necessary. Based on this, further development of1597-54-6 was stopped.

Table 6 Summary of corrosion and oxidation test results

Test	75W-85	75W-140	2484-17-2	2484-16-1	1597-54-6
Copper corrosion	1B	2A	1B	1A	1B
Rust Prevention	Pass	Pass	Pass	Pass	Pass
Oxidation, Δviscosity %	+20	+20	+4.7	+1.6	+2.5
Oxidation, ΔTAN, mgKOH/g	1.75	1.5	0.4	0	-0.3

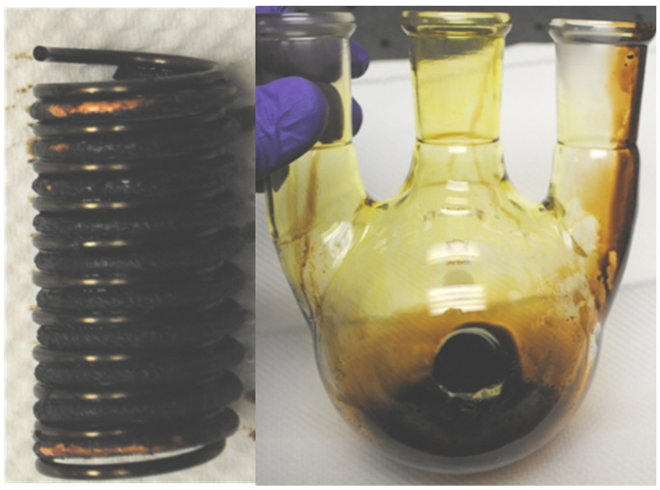

Figure 2 Copper/steel coil and flask after 50 h at 163 °C, 2484-17-2

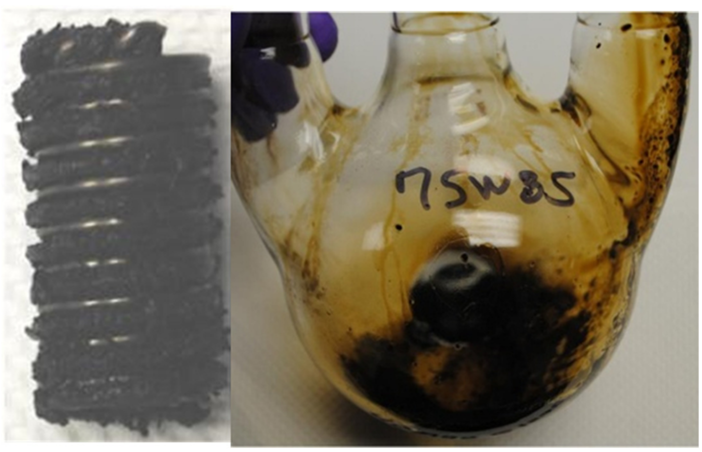

Figure 3 Copper/steel coil and flask after 50 h at 163 °C, 75W-85

Figure 4 Copper/steel coil and flask after 50 h at 163 °C, 75W-140

Stribeck curves were measured on both benchmark gear lubricants and 2484-17-2 and -16-1 at 40, 100 and 150 °C with a 100% SRR and a 50 N (1.1 GPa) contact load, Figure 5 to Figure 7. Each lubricant-test condition was repeated 12 consecutive times with the 12th repeat reported. Repetition at each test condition allowed tribo-layers to form, giving a better indication of frictional properties at equilibrium conditions. At all temperature conditions the SAE 75W-85 had the highest traction coefficient. The SAE 75W-140 had higher traction coefficients at the higher entrainment speeds while giving lower traction coefficients than the PAG-based fluids at lower entrainment speeds approaching the boundary lubrication regime. The SAE 75W-140 was the highest viscosity lubricant tested, likely explaining the shape of the Stribeck curve. 2484-17-2 and -16-1 gave roughly equivalent traction performance at 40 and 100 °C, while 2484-16-1 gave markedly lower traction values at 150 °C. 2484-17-2 was selected over 2484-16-1 to continue to system level testing based on the better 4-ball EP and Falex EP loads.

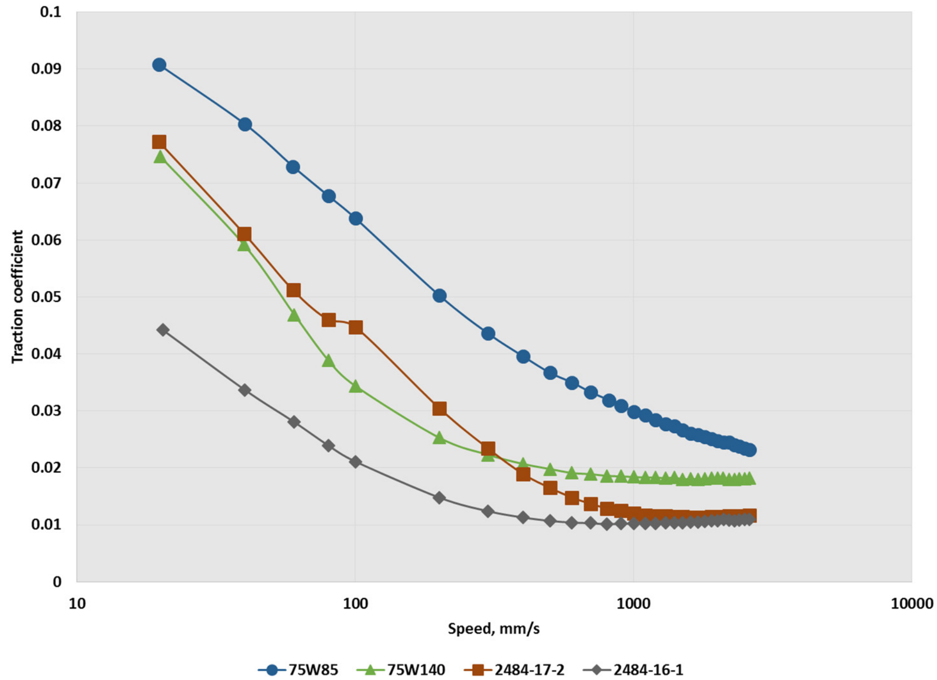

Figure 5 Stribeck curve at 150 °C, 50 N (1.1 GPa), 100% SRR

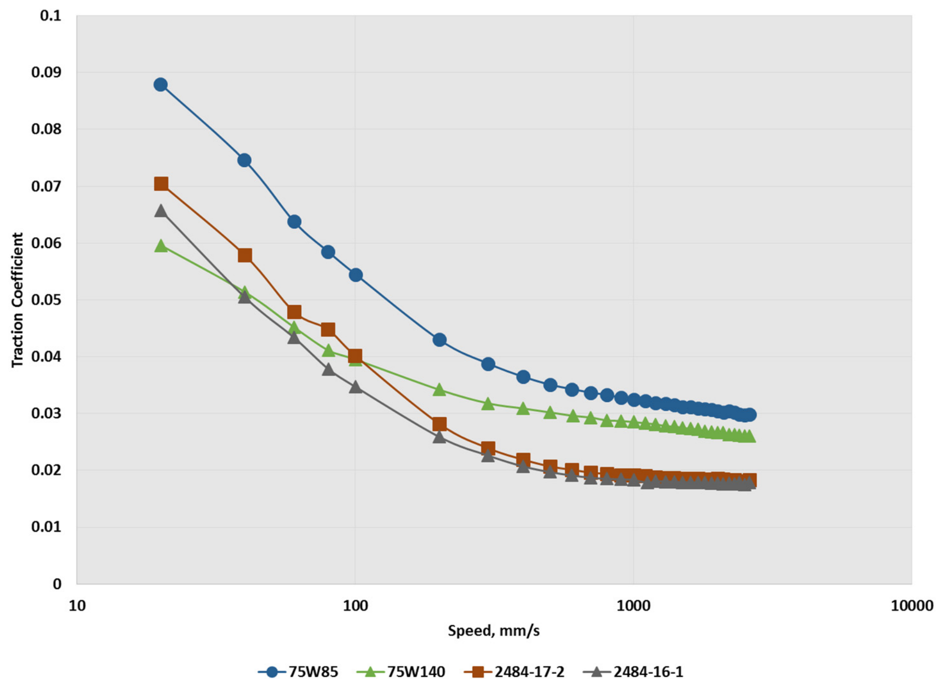

Figure 6 Stribeck curve at 100 °C, 50 N (1.1 GPa), 100% SRR

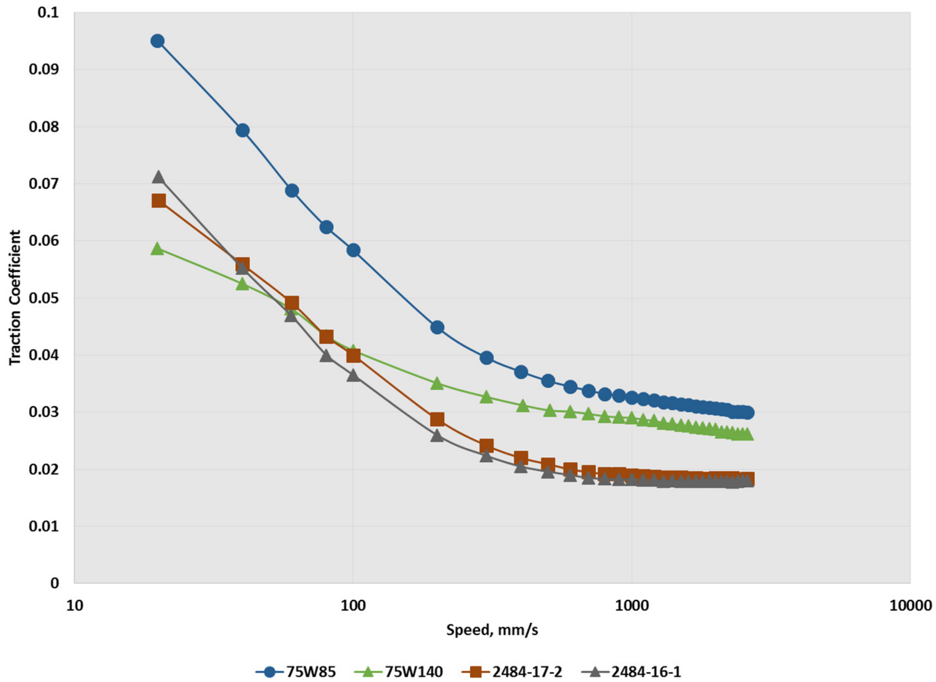

Figure 7 Stribeck curve at 40 °C, 50 N (1.1 GPa), 100% SRR

Elastomer seal compatibility of FKM material V41 with 2484-17-2 was measured. V41 was exposed to 2484-17-2 and SAE 75W-85 for 1000 h at 150^{o}C. Tensile strength, hardness and volume swell were measured at four time intervals, Figure 8 to Figure 10. After 1000 hours FKM exposed to PAG 2484-17-2 showed a 22% drop in tensile strength compared to an 18% drop for FKM exposed to SAE 75W-85. Both FKM samples showed similar change in hardness and volume after1000 hours. The change in mechanical properties of FKM when exposed to 2484-17-2 were considered acceptable.

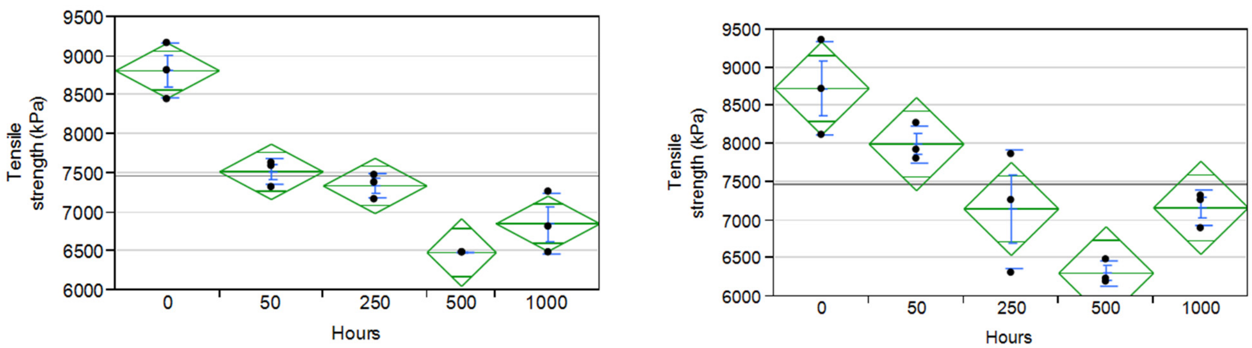

Figure 8 Tensile strength 2484-17-2 (left) and SAE 75W-85 (right)

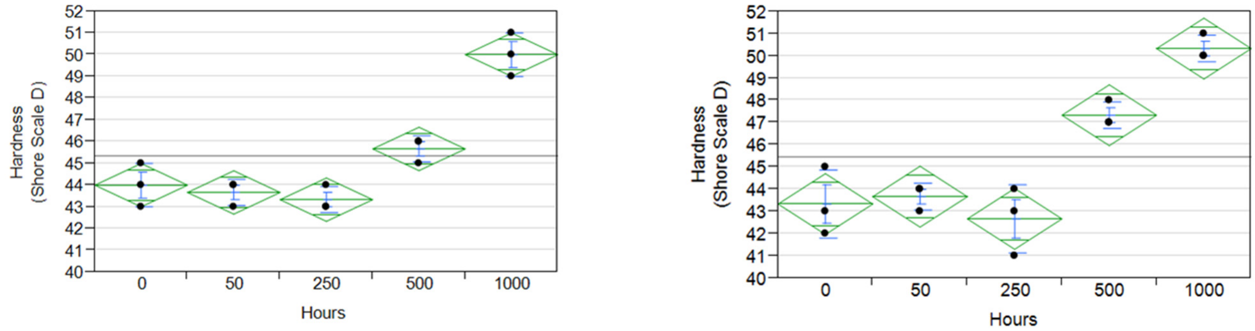

Figure 9 Hardness 2484-17-2 (left) and SA 75W-85 (right)

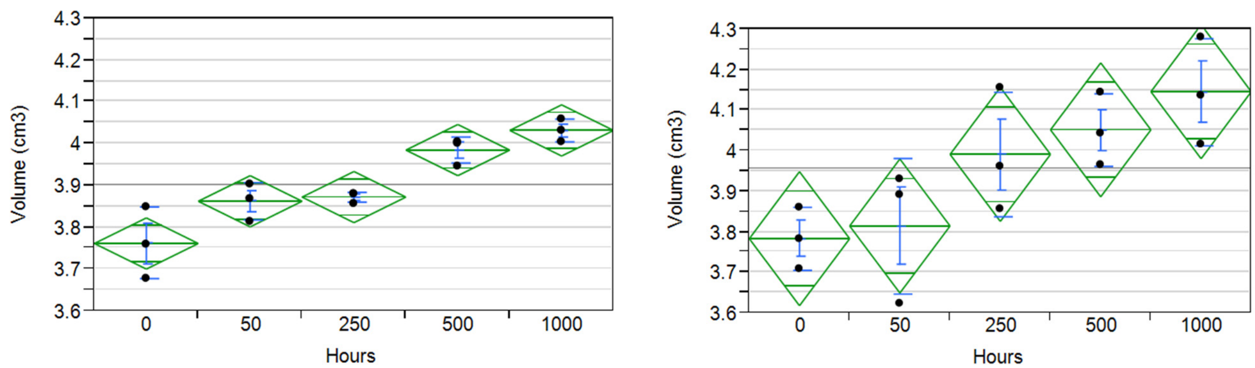

Figure 10 Volume 2484-17-2 (left) and SAE 75W-85 (right)

ACM (see ASTM D 1418 for definition of ACM) pinion shaft oil seals were exposed to 2484-17-2 and SAE 75W-85 for 168 h at 130°C with dimensional measurements made at the end of the exposure time, Figure 11. The ACM swelled substantially upon exposure to 2484-17-2 when compared to the seal exposed to SAE 75W-85. The damage to the ACM seal material was considered unacceptable, however it was noted that ACM would not be used in this particular service in future and should not stop further evaluation of 2484-17-2 in system level tests.

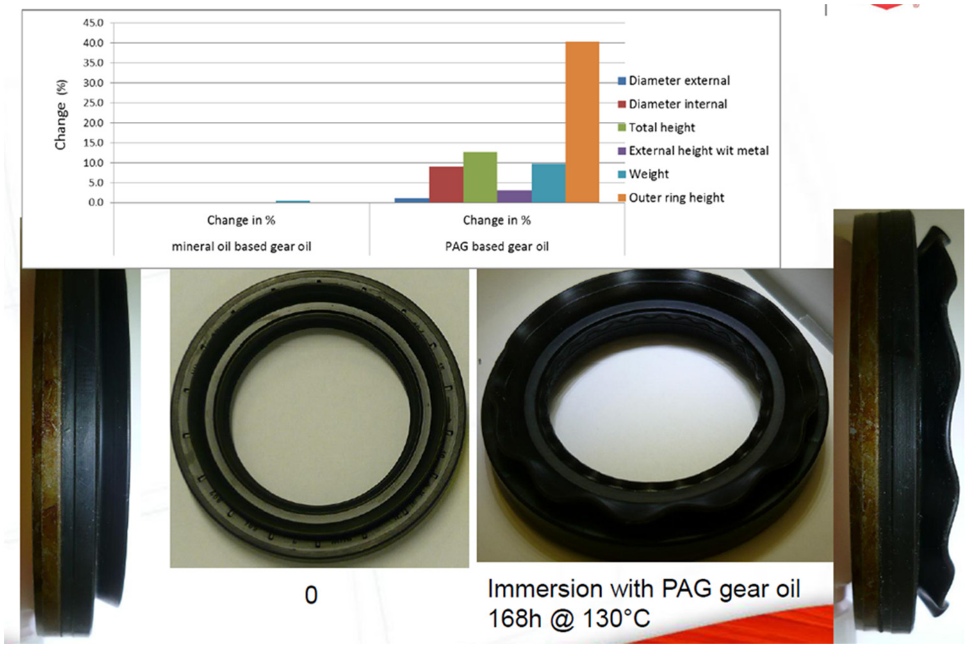

Figure 11 Dimensional changes of ACM seal after exposure to 2484-17-2

2484-17-2 was submitted to an third party testing facility for evaluation in an L-37 axle test. 2484-17-2 met or exceeded all of the ring and pinion gear rating requirements except for rippling of the pinion gear, Table 7.

Analysis of the end of test fluid found that the levels of sulfur and of phosphorus were unchanged as compared to the fresh fluid. Iron levels in the post test fluid were found to be 680 ppm. ^{31}P NMR found that the concentration of the phosphorus additive was essentially unchanged in the EOT fluid. LC/MS analysis of the EOT fluid found that one of the sulfur containing additives had been depleted.

Table 7 L-37 test results for 2484-17-2

	Ring rating	Pinion rating	Pass (ring/pinion) min
Wear	7.0	6.0	5/5
Rippling	8.0	5.0	8/8
Ridging	8.0	8.0	8/8
Pitting/Spalling	9.9	9.7	9.3/9.3
Scoring	10.0	10.0	--

Measurements by XPS on a pinion gear section were done on the rippled area of the gear, R1 in Figure 12, and the undamaged area of the gear, R2. XPS samples the top 7-10 nm of the surface, providing quantifiable elemental and chemical information. The XPS revealed that there were compositional difference in the two areas, Table 8. R1 showed presence of FeS and a reduced organic sulfur species with sulfur persisting beneath the surface. R2 showed only SO_x species. R1 showed both inorganic and organic oxygen species; R2 showed only organic oxygen-species. R1 and R2 showed PO_x species, being inorganic or organic phosphate. Carbon levels were higher and iron lower in R2, while the reverse was true for R1.

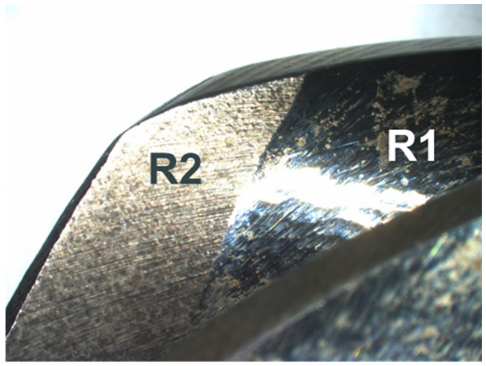

Figure 12 Pinion section analyzed by XPS. R1 is worn area, R2 is intact

Table 8 XPS results, elemental weight% forced to 100%

	C	Ca	Fe	N	O organic	O Inorganic	P PO_x	S SO_x	S FeS	S organic
R1	28.9	2.2	30.9	2.5	20.2	5.5	0.5	--	5.1	1.4
R2	62.2	2.6	5.8	2.3	18.8	--	1.2	2.5	--	--

McFadden et al. reported on rippling gear failures in the L-37 test (4). They reported that the phosphorus level in the fluid did not change over the course of the test for both passing and failing lubricants. They found phosphorus and little sulfur in tribo layers on the surface of passing gears and sulfide and little phosphorous in the tribo-layers on failing gear surfaces. They also found that carbon concentration increased with film depth on failing gears. McFadden's observations appear to apply to the ridging failure experienced by 2484-17-2 in the L-37 test. This suggested that the EP and AW additives in 2484-17-2 were not suitable and that a reformulation would be necessary to pass the L-37 test.

3.2 Reformulation of 2484-17-2 to AW-704-A

Tribological screening tests employed in the development 2484-17-2 gear lubricant did not predict performance in the L-37 test. Thus, the first step in reformulation of 2484-17-2 would have to be the development of a tribological screening test that would discriminate between gear lubricants that passed the L-37 test, the SAE 75W-85 and SAE 75W-140 bench marks, and those that failed the L-37 test, 2484-17-2.

Two test methods were proposed. One method involved measuring the wear volume using the lubricant under sliding conditions for 6 h at a temperature of 100 °C, a sliding speed of 100 mm/s and a contact pressure of 1.05 GPa using a pin on disk geometry. The other method measured wear scars on a ball using the ball on disk geometry after 4 h of sliding at a temperature of 135 °C at 100 mm/s under a contact load of 1 GPa. Friction coefficient as a function of time was also measured for the ball on disk geometry.

The wear volume of 2484-17-2 is markedly higher after 3 h of sliding than the benchmark lubricants after 6 h, Figure 13 and

Table 99. The ball on disk method did not find major differences in either wear or

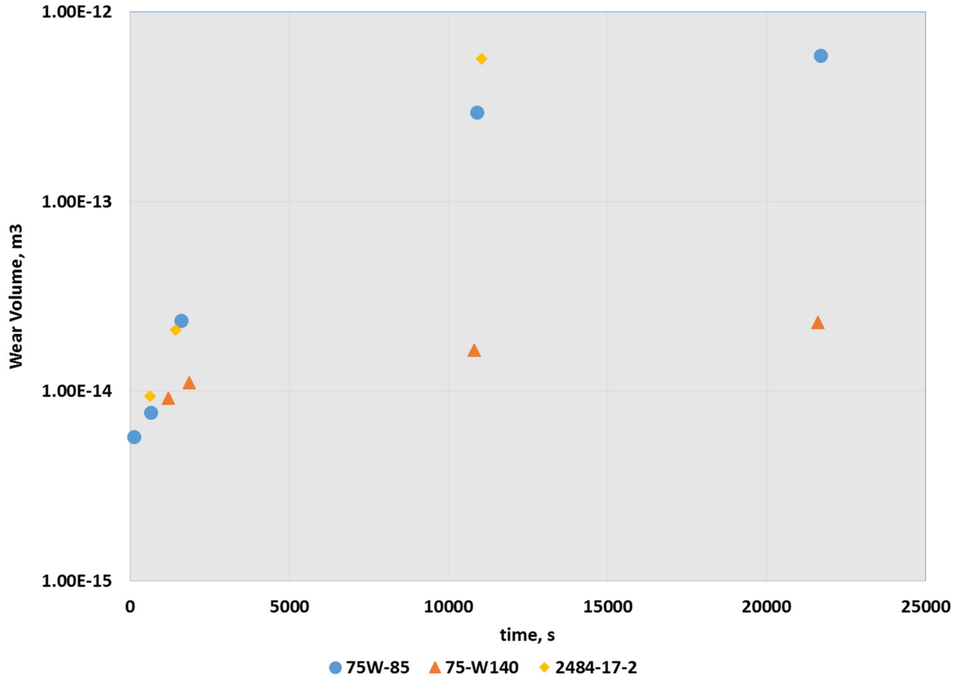

Figure 13 Pin on disk wear volumes for 2484-17-2

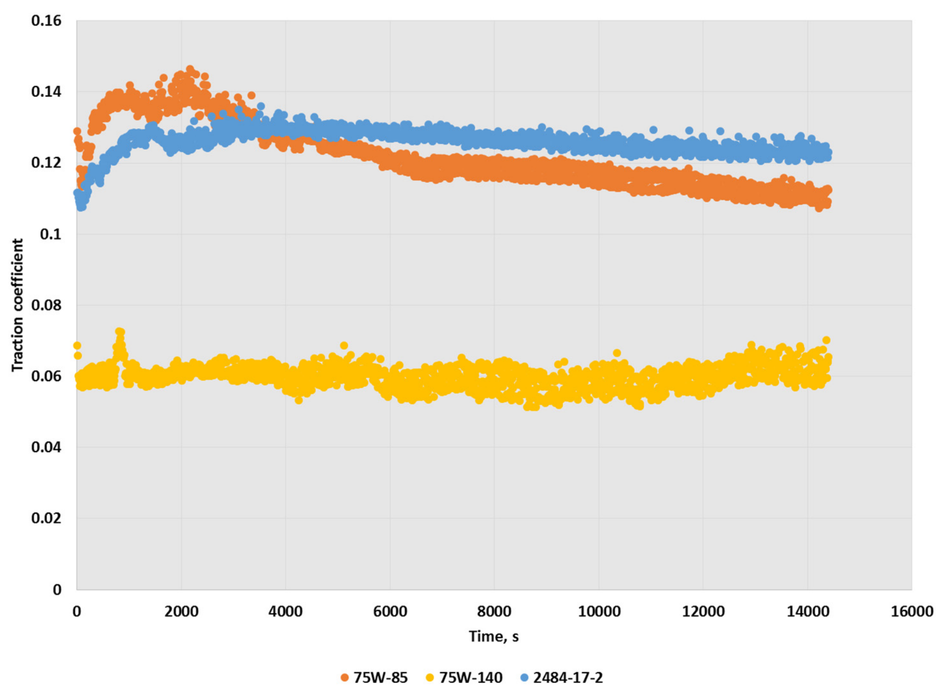

Figure 14 Ball on disk friction for 2484-17-2

Table 9 Summary of wear data

Tests	SAE 75W-85	SAE 75W-140	2484-17-2	AW-704-A
4 ball wear, mm	0.92	0.42	0.52	0.36
4 ball EP, kg	340	420	400	280
Falex EP, lb	2505	2807	3652	2885
Wear volume, m^3 @ 6 h[1]	$5.9*10^{-13}$	$2.3*10^{-14}$	$1.1*10^{-12}$	$5.3*10^{-14}$
Ball on disk wear of ball, mm	0.63	0.44	0.49	0.32
Ball on disk wear of disk, mm	0.54	0.41	0.46	0.31

[1]2484-17-2 wear volume at 3 h

Co-efficient of friction between the bench mark lubricants and 2484-17-2, Figure 14 and Table 99. Both methods were used to screen new formulations. The 2484-17-2 formulation was extensively modified to pass the L-37 test. The PAG base stock was changed, additional phosphorus-based AW compounds were added and their overall concentration increased, the sulfurized EP additive concentration was reduced, a friction modifier was added and the anti-oxidant package was modified.

PAGs generally have a higher liquid density than hydrocarbon lubricants (Table 10). Higher density lubricants can lead to overall efficiency losses due to liquid churning in the differential. The relationship between churning losses and liquid density can be illustrated with the following equation:

$$f = C_d \rho v^2 A$$

$$Where\ C_d = f(Re)\ and\ other\ factors$$

$$Re = \frac{\rho v D}{\mu}\ or\ \frac{vD}{\kappa}$$

Where *f* is friction; C_d is the drag coefficient, which is a function of the Reynolds number *Re*; *v* is the gear velocity; ρ fluid density; *A* the hydrodynamically wetted surface area; μ the absolute viscosity, κ kinematic viscosity and *D* the hydraulic diameter. Fluids with higher densities and similar C_d will tend to have higher drag losses leading to increased energy losses.

Base stock PAG E used to formulate 2484-17-2 and was replaced with PAG J, which lowered the density and dynamic viscosities of the new lubricant, AW-704-A, Table 10. The VI of AW-704-A was somewhat lower than that of 2484-17-2 while the -40 ° C was approximately 9000 mPa s higher,

Table 101.

Table 10 Viscosity and density of several lubricants

	SAE 75W-85		SAE 75W-140		2484-17-2		AW-704-A	
	40 °C	100 °C	40 °C	100 °C	40 °C	100 °C	40 °C	100 °C
Density, kg/m^3	858.6	820.0	849.3	812.2	1020.4	974.7	953.4	909.0
Viscosity, mm^2/s	67.7	11.7	181.9	25.1	59.4	12.0	57.8	11.2
Viscosity, mPa s	58.1	9.6	154.5	20.3	60.6	11.7	55.1	10.2

Table 101 Fluid viscosities

	SAE 75W-85	SAE 75W-140	AW-704-A	2484-17-2
Viscosity @ 100 °C, mm^2/s	11.7	25	11.2	12.0
Viscosity @ 40 °C, mm^2/s	67.9	181.9	57.8	59.4
VI	168	170	190	203
Viscosity @ -40 °C, mPa s	66800	150000	77500	68657

The wear properties of AW-704-A as measured by the 4-ball wear, pin-on-disk wear volume and ball-on-disk wear scar were all improved vs 2484-17-2 and were lower than those for the benchmark lubricants except for the wear volume of the SAE 75W-140 where it was somewhat higher,
Table 99. The wear volume of AW-704-A was initially higher than that of the benchmark lubricants but plateaued after 10000 s, Figure 15. The friction coefficient measured in the ball-on-disk of AW-704-A was lower than 2484-17-2 and SAE 75W-85, but was still higher than SAE 75W-140, Figure 16. The EP loads of AW-704-A were lower than those measured for 2484-17-2 in the 4-ball EP and Falex EP wear tests,
Table 99. Lower EP loads were likely due to the lower levels of sulfurized EP additives in AW-704-A.

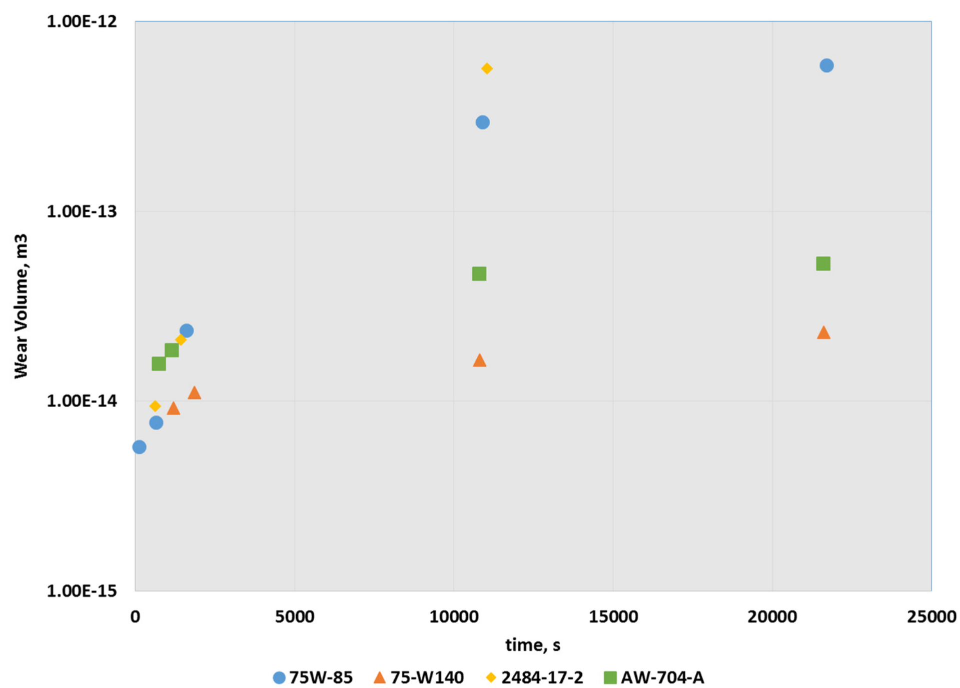

Figure 15 Pin on disk wear volumes for AW-704-A

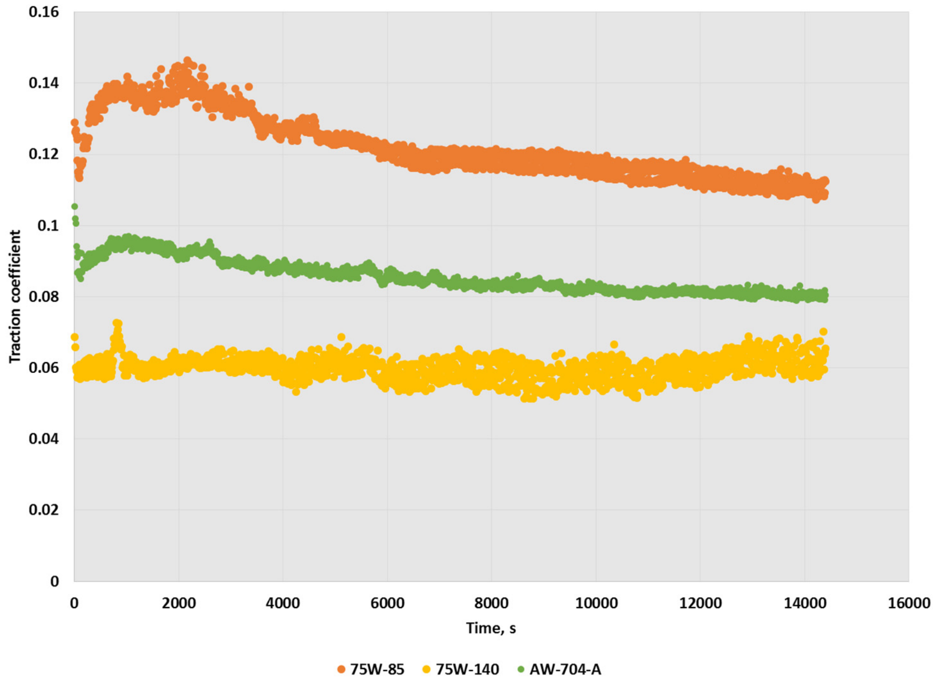

Figure 16 Ball on disk friction for AW-704-A

The copper corrosion and rust prevention properties of AW-704-A were similar to those of 2484-17-2 and the benchmark gear lubricants, Table 11. The oxidative stability of AW-704-A was comparable to 2484-17-2, though it did have greater ΔTAN than 2484-17-2. Deposits on the copper/steel coil and in the glass flask after the oxidation test, Figure 17, were similar to those observed for 2484-17-2, Figure 2.

Table 11 Summary of corrosion and oxidation test results

Tests	SAE 75W-85	SAE 75W-140	2484-17-2	AW-704-A
Copper corrosion	1B	2A	1B	1B
Rust Prevention	Pass	Pass	Pass	Pass
Oxidation, Δviscosity %	+20	+20	+4.7	+3.9
Oxidation, ΔTAN, mgKOH/g	1.75	1.5	0.4	1.26

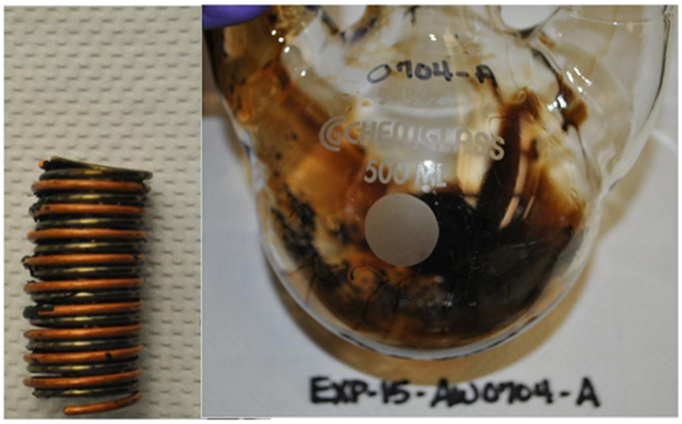

Figure 17 Copper/steel coil and flask after 50 h at 163 °C, AW-704-A

Stribeck curves were measured for AW-704-A at slightly different conditions than for 2484-17-2: 150% SRR vs 100% SRR. The measurement temperatures were also different, 80°C and 120°C instead of 100°C and 150°C. The same trends, however, can be observed. The traction coefficients for SAE 75W-140 and AW-704-A were lower than those of SAE 75W-85 at all temperatures and entrainment speeds,

to Figure 20. The traction coefficients for SAE 75W-140 were lower than those of AW-704-A at lower entrainment speeds.

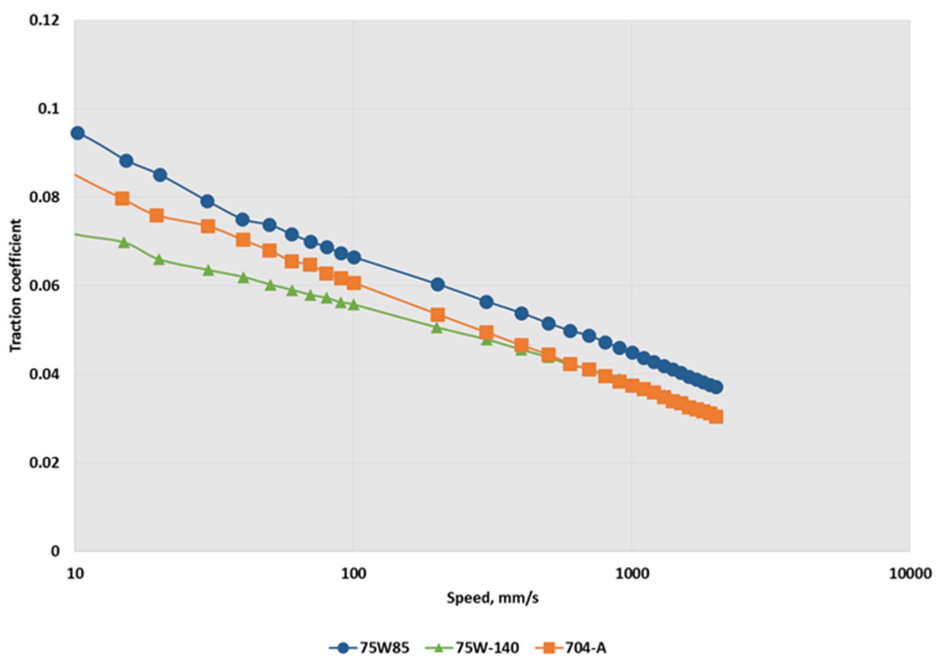

Figure 18 Stribeck curves, 40°C, 50 N (1.1 GPa), 150% SRR

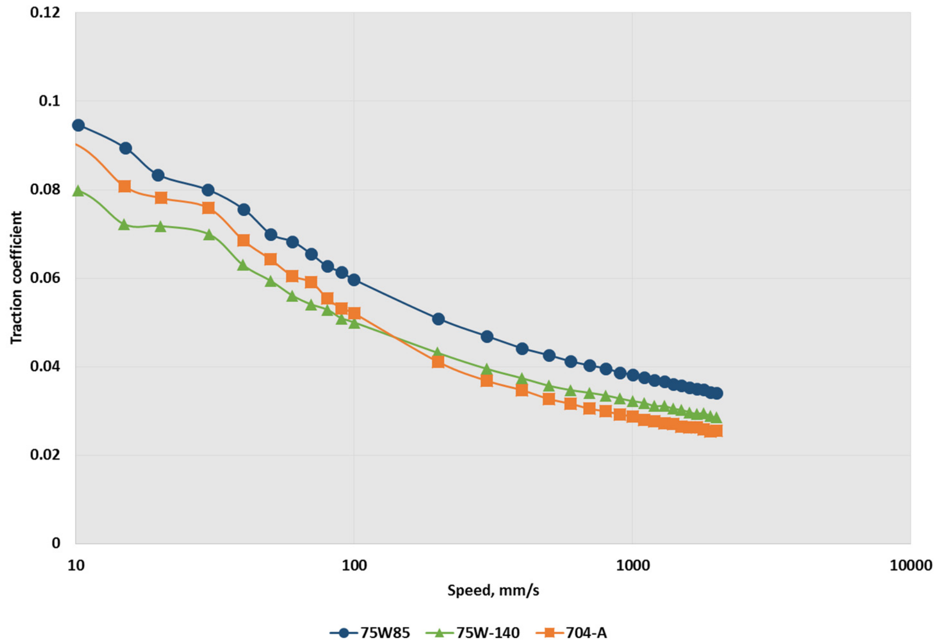

Figure 19 Stribeck curves, 80°C, 50 N (1.1 GPa), 150% SRR

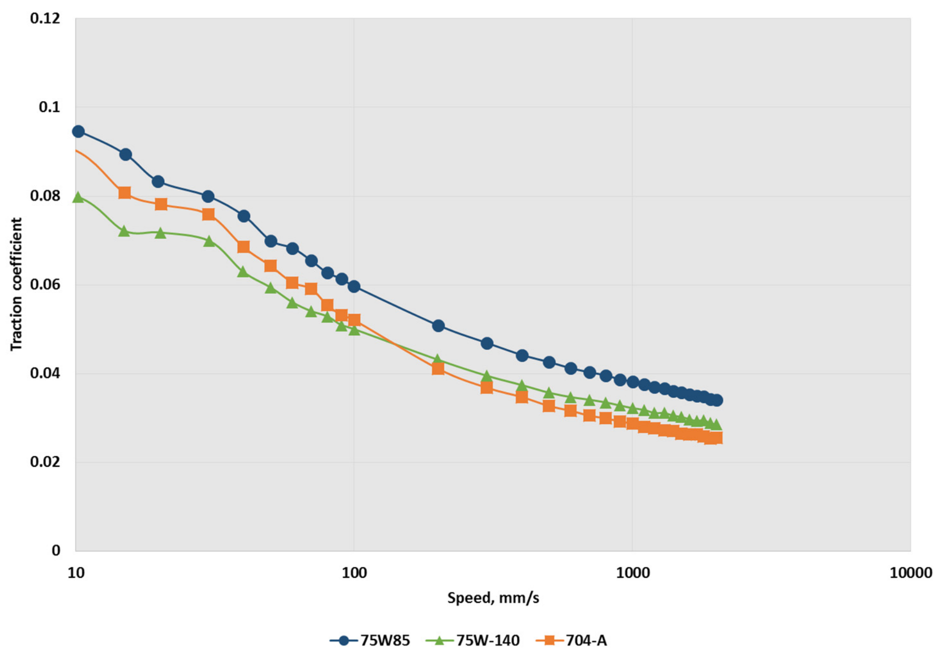

Figure 20 Stribeck curves, 120°C, 50 N (1.1 GPa), 150% SRR

AW-704-A was submitted for L-37 testing and passed, meeting or exceeding all gear rating requirements, Table 12. The pinion gear rating for rippling improved to 8.0 from 5.0 for 2484-17-2. All other ratings were either equivalent to or greater than those for 2484-17-2. The iron concentration in the EOT lubricant was significantly lower at 189 ppm vs 680 ppm measured for EOT 2848-17-2. Phosphorus and sulfur concentrations were reduced during the test from 1500 ppm to 1300 ppm for phosphorus and from 8590 ppm to 8120 ppm for sulfur. XPS analysis of pinion gear surfaces, Figure 21, indicated that changing the anti-wear and extreme pressure additives was effective in changing the composition of the tribo-film. The wear areas for the AW-704-A had a tribo-layer consisting of mostly phosphorus compounds with little or no sulfur present.

Table 12 L-37 test results for AW-704-A

	Ring rating	Pinion rating	Pass (ring/pinion) min
Wear	8.0	7.0	5/5
Rippling	10.0	8.0	8/8
Ridging	9.0	9.0	8/8
Pitting/Spalling	9.9	9.9	9.3/9.3
Scoring	10.0	10.0	--

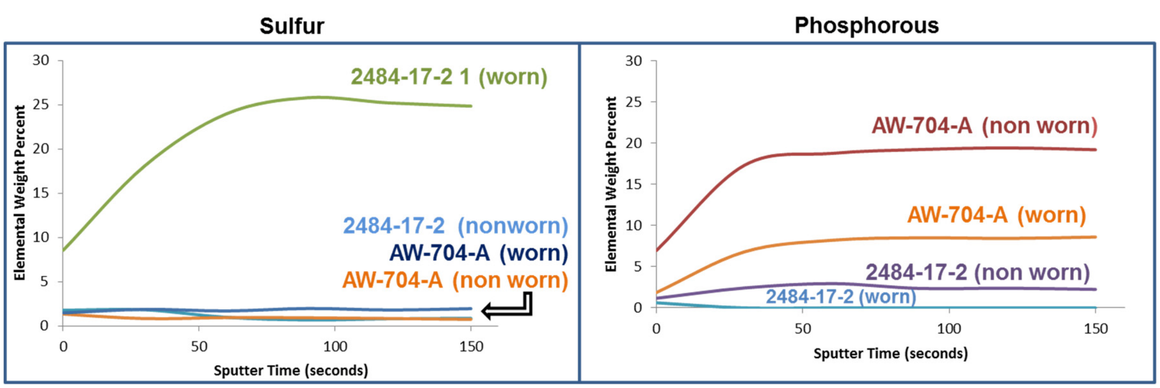

Figure 21 Sulfur and phosphorous on pinion gears as a function of sputter time

An L-60 test was performed with AW-704-A. The lubricant met or exceeded all the requirements, with the exception of pentane insoluble, Table 13. Solvent precipitation tests are normally not used with PAG fluids (5). One of the components in the PAG J blend used in AW-704-A is not soluble in pentane, likely contributing to a higher than acceptable result.

Table 13 L-60 test results for AW-704-A

	Reference Oil	AW-704-A	Pass
Viscosity increase, %	32	0	25 max
Pentane insolubles, %	1.5	4.9	1 max
Toluene insolubles, %	0.9	0	1 max
Avg Carbon/Varnish, merits	8.9	10	8.5 min
Avg Sludge. merits	9.4	9.5	9.4 min

Test results from an L-42 test with AW-704-A were unacceptable, Table 14. Significant scoring was observed on the drive and coast sides of both the ring and pinion gears. The failure of AW-704-A in this test indicated that the extreme pressure portion of the additive package was inadequate, necessitating another reformulation of the lubricant.

Table 14 L-42 results for AW-704-A

	AW704-A	Passing
Ring % scoring drive side	97	11
Ring % scoring coast side	92	11
Pinion % scoring drive side	77	18
Pinion % scoring coast side	88	18

Due to changes in Ford EH&S requirements during the project, the PAG J base stock used in AW-704-A was determined to be unacceptable. Different PAG base stocks were evaluated and one that met the rheological requirements was identified. Extensive work with this PAG to identify a viable additive package was unsuccessful. Timing and resource issues combined to force abandonment of the development of a PAG-based gear lubricant.

3.3 Development of a PAG –mineral oil gear lubricant

A second approach to developing an energy efficient gear lubricants was investigated. Oil soluble polyalkylene glycols have been shown to improve frictional properties of mineral oils when used as an additive at relatively low treat rates (6). The polarity of oil soluble PAG leads to enhanced surface activity, which allows them to act as friction modifiers by forming films on surfaces. Oil soluble polyalkylene glycols also have higher VIs, improving the viscometrics of the mineral oil, and can reduce deposit formation.

A capped alcohol initiated PO/BO copolymer, PAG K, was synthesized for this lubricant. The 100°C kinematic viscosity was 3.6 mm^2/s; the 40 °C kinematic viscosity 12.6 mm^2/s, giving a VI of 185. PAG K was mixed with an API Group III base stock and a proprietary additive package. Two concentrations of PAG K were evaluated, 30 wt% in AU-6615-E and 15 wt% in AU-6615-F. The additive concentrations were held constant and the concentration of the viscosity modifier adjusted to meet a 100°C viscosity of 11-12 mm^2/s, Table 15. The resulting VIs of AU-6615 –E and –F were somewhat higher at 186-187 than that of the SAE 75W-85's 168. The liquid densities of AU-6615-E and –F were higher than the SAE 75W -85, increasing with the concentration of PAG K in the lubricant.

Table 15 Viscosity of AU-66015-E and -F

	SAE 75W-85	SAE 75W-140	AU-6615-E	AU-6615-F
Viscosity @ 100 °C, mm^2/s	11.7	25	11.4	11.9
Viscosity @ 40 °C, mm^2/s	67.9	181.9	60.0	63.6
VI	168	170	187	186
Viscosity @ -40 °C, mPa s	66800	150000	<65000	<65000
Density @ 40 °C, kg/m^3	858.6	849.3	883.4	869.8
Density @ 100 °C, kg/m^3	820.0	812.2	843.7	831.0

The corrosion and oxidation performance of AU-6615-E was measured, Table 16. The viscosity and TAN increases were markedly higher than observed for 2484-17-2 and AW-704-A. However, positive results in later L-60 testing of AU-6615-E suggests that the conditions of the oxidation test may be too severe to accurately screen candidate gear lubricants for performance in the L-60 test.

Table 16 Summary of corrosion and oxidation test results

Tests	SAE 75W-85	SAE 75W-140	AU-6615-E
Copper corrosion	1B	2A	1B
Rust Prevention	Pass	Pass	Pass
Oxidation, Δviscosity %	+20	+20	+46
Oxidation, ΔTAN, mgKOH/g	1.75	1.5	7.0

Wear and extreme pressure properties were measured on AU-6615-E and –F, Table 17. Four ball, pin and disk wear scars are essentially equivalent to the SAE 75W-85 gear oil. The friction coefficients measured for the lubricants during the 4h ball on disk tests were also approximately the same as the SAE 75W-85 bench mark, Figure 22. Stribeck curves measured for SAE 75W-85 and AU-6615-E suggest that PAG K reduces the traction coefficient at higher temperatures and down to an entrainment speed of ~ 200 mm/s, Figure 23. The overall reduction in traction coefficient was not as great as observed for the PAG-based gear lubricants discussed above.

Table 17 Summary of tribology data for AU-6615-E

Tests	SAE 75W-85	SAE 75W-140	AU-6615-E	AU-6615-F
4 ball wear, mm	0.92	0.42	0.89	--
4 ball EP, kg	340	420	315	--
Ball on disk wear of ball, mm	0.63	0.44	0.67	0.62
Ball on disk wear of disk, mm	0.54	0.41	0.52	0.46

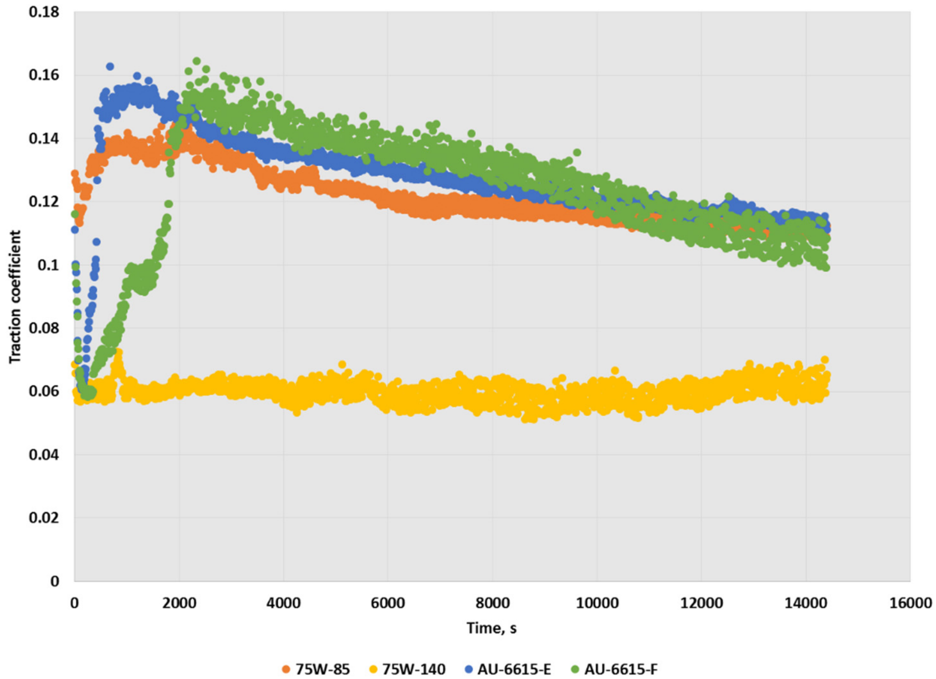

Figure 22 Ball on disk friction for AU-6615-E and -F

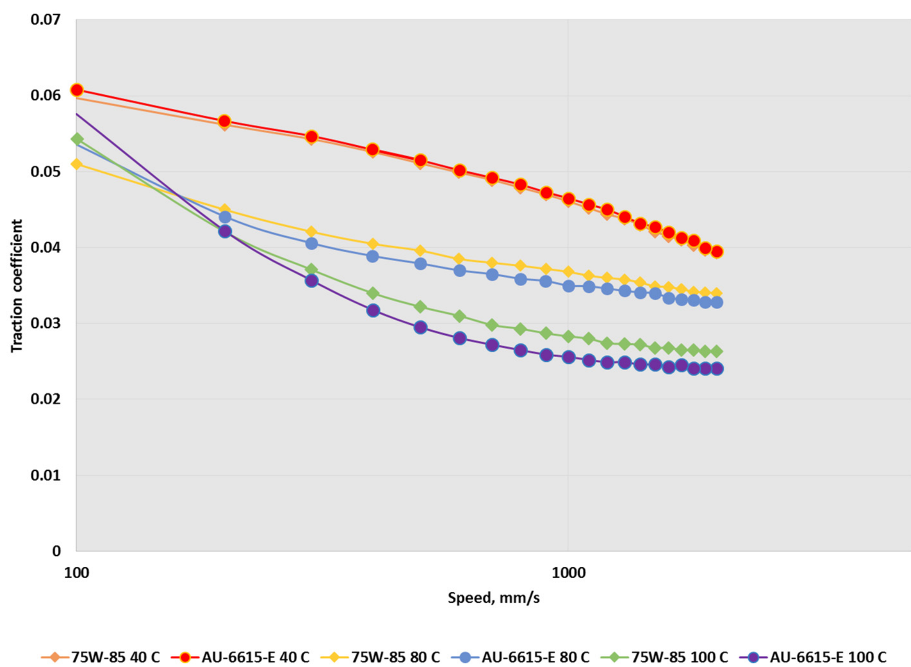

Figure 23 Stribeck curves of 75W-85 and AU-6615-E at 50 N (1.1 GPa), 100 % SRR and 40, 80 and 100 °C

AU-6615-F was submitted for L-37 testing. The lubricant met or exceeded all of the rating requirements, Table 18. The results were comparable to those obtained for AW-704-A, with improvements in the ratings for rippling and ridging on the pinon being noteworthy. XPS of the post-test pinion gear shows higher levels of phosphorus on the wear and non-worn surfaces and relatively small amounts of sulfur in these areas, Figure 24. The pinion gear from the AW-704-A L-37 test showed a relatively constant level of phosphorus in the tribo-surface after 150 s of etching, Figure 21. The XPS on the pinion gear from the AU-6615-F L-37 test shows that the phosphorus concentration in the wear area peaking after 50 s of etching and then beginning to decrease as etching continues, suggesting a somewhat thinner layer of phosphorus rich tribo-layer being formed during the test.

Table 18 L-37 test results for AU-6615-F

	Ring rating	Pinion rating	Pass (ring/pinion) min
Wear	7.0	7.0	5/5
Rippling	10.0	9.0	8/8
Ridging	10.0	9.4	8/8
Pitting/Spalling	9.9	9.9	9.3/9.3
Scoring	10.0	10.0	--

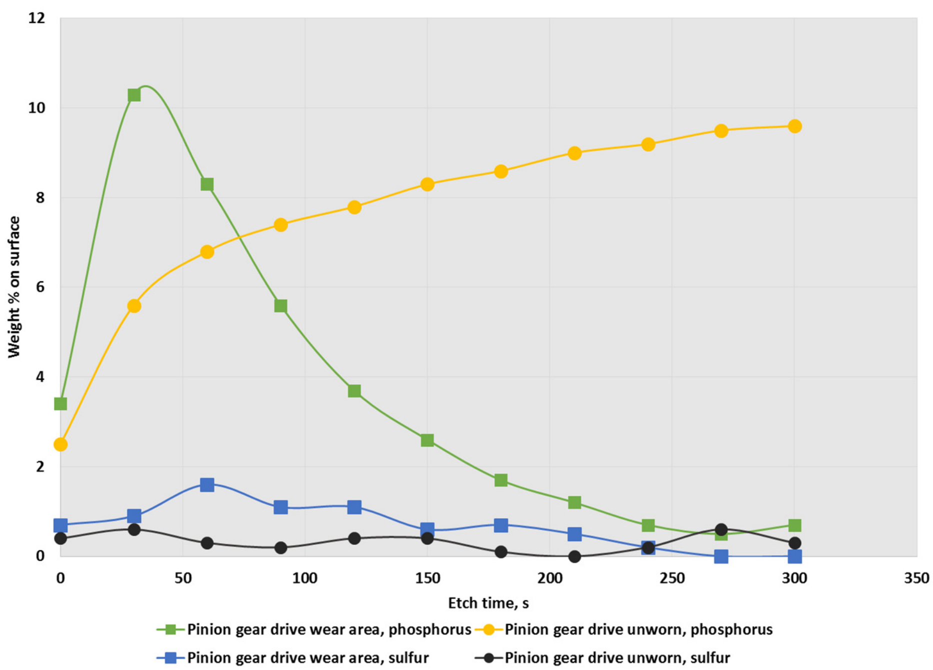

Figure 24 XPS for sulfur and phosphorus as a function of etch time on L-37 pinion gear

L-42 test results for AU-6615-E can be found in Table 19. Scoring measured for AU-6615-E was well below the maximum values allowed for both the ring and pinion gears, a significant improvement over the AW-704-A L-42 results.

Table 19 L-42 results for AU-6615-E

	AU-6615-E	Passing, maximum
Ring % scoring drive side	0	0
Ring % scoring coast side	8	15
Pinion % scoring drive side	0	0
Pinion % scoring coast side	14	22

L-60 test results for AU-6615-E are in Table 20. The AU-6615-E viscosity increase in the L-60 test was much higher than observed for AW-704-A, 0% vs 21%, but lower than the passing limit. The lab oxidation test for AU-6615-E suggested that the fluid would have a higher viscosity build than the PAG-based lubricants, however the actual viscosity build in the L-60 was not as dramatic as the lab oxidation test indicated it would be. The pentane insolubles for AU-6615-E was much lower than AW-704-A, suggesting that the oil soluble PAG was soluble in pentane, unlike the PAG found in AW-704-A. The varnish and sludge ratings for both fluids were similar. Photos of the gears from each test are in Figure 25.

Table 20 L-60 test results for AU-6615-E

	Reference Oil	AU-6615-E	Pass
Viscosity increase, %	29	21	25 max
Pentane insolubles, %	2.0	0.1	1 max
Toluene insolubles, %	1.7	0.2	1 max
Avg Carbon/Varnish, merits	8.9	9.9	8.5 min
Avg Sludge. merits	9.5	9.5	9.4 min

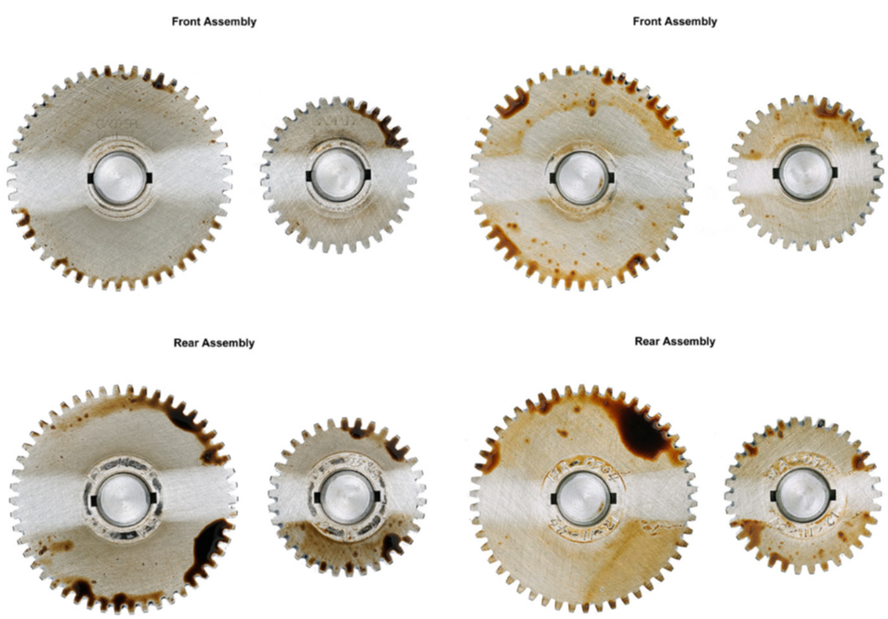

Figure 25 L-60 gears from AU-6615-E (left) and AW-704-A (right) tests

AU-6615-F was evaluated in L-33 test for rust and corrosion inhibiting properties for the lubricant. The results indicated a 9.6 rating vs a minimum required 9.0. Figure 26 is a photo of the differential cover from the L-33 test, showing clean, rust free surfaces.

Figure 26 Differential cover from L-33 test for AU-6615-F

3.4 Gear Wear Evaluation

AU6615-F was evaluated in Ford proprietary 44 hour gear wear test. Current production axles were used. The photos in Figure 27 and Figure 28 show the post-test gear mesh patterns for two ring gears in repeat tests. Figure 27 depicts the post-test drive side ring gear pattern of one sample axle tested with AU6615-F. This is a passing post-test gear pattern judging by the location of the contact area (gray area where the yellow paint worn away) in the middle of the

Figure 27. Gear mesh pattern on post test ring gear from axle PAG006(9597) tested with AU6615-F. This post test pattern represents typical gear wear hardware and denoted to be acceptable.

Figure 28. Gear mesh pattern on post test ring gear from axle PAG002(9602) tested with AU6615-F. This post test pattern indicates unacceptable wear, exhibiting a contact that is not consistent over the full land of the gear teeth

gear section. Figure 28 depicts another post-test drive side ring gear pattern of another sample axle test with AU6615-F. This is a non-passing post-test gear pattern. The contact between teeth has been away, yielding poor contact. This pattern would exhibit NVH issues in a vehicle. Figure 29 graphically displays the transmitted error attributed to each of the patterns shown in Figures 27 and 28. Because of the mixed results on production hardware, the co-base stock option did not meet the durability criteria for a viable product. The co-base stock option could be sensitive to assembly parameters and variation of current production axles, however changing axle assembly and design is outside the scope of this project.

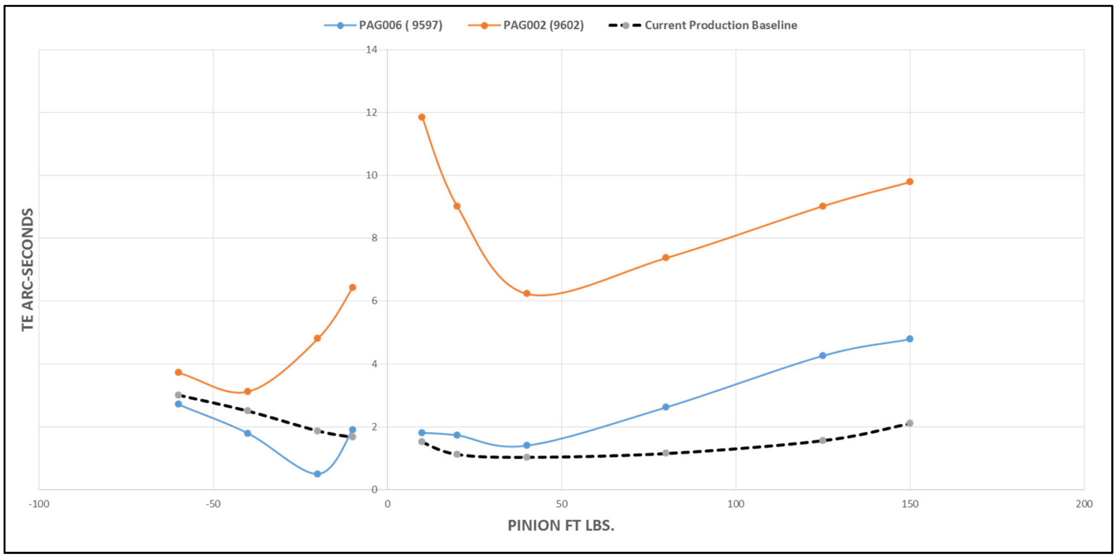

Figure 29**.** Ono Sokki Torque Sweep for AU6615-F providing transmitted error [TE] versus torque at the pinion gear [ft. lbs.]. Data is presented for both PAG006(9597) and PAG002(9602), as well as a virgin production axle. It can be seen that the PAG002(9602) deviates greatly from the production baseline. The curves on the left side of the graph represent the coast side of the gears and the curves on the right represent the drive side. High transmitted error translates to NVH issues.

3.5 Axle Efficiency Evaluation

Axle efficiency evaluation conducted on AU6615-F and AW-0704-A lubricants as an A-B-A study with 75W-140. Also included in the study was a reference fluid SAE 75W-85. Axle efficiency tests were conducted at different stages where each stage denote a combination of speed and load starting from lower load/speed to high load/speed points. Figure 30 shows that the PAG lubricant (AW0704-A) improved efficiency when compared to SAE 75W-140 and SAE 75W-85 lubricants. The data illustrates that the PAG exhibits indisputable improved efficiency to the baseline lubes throughout the low torque stages, and equivalent (and arguably improved) efficiency to SAE 75W-85 at throughout the mid and high torque stages. Test repeatability is

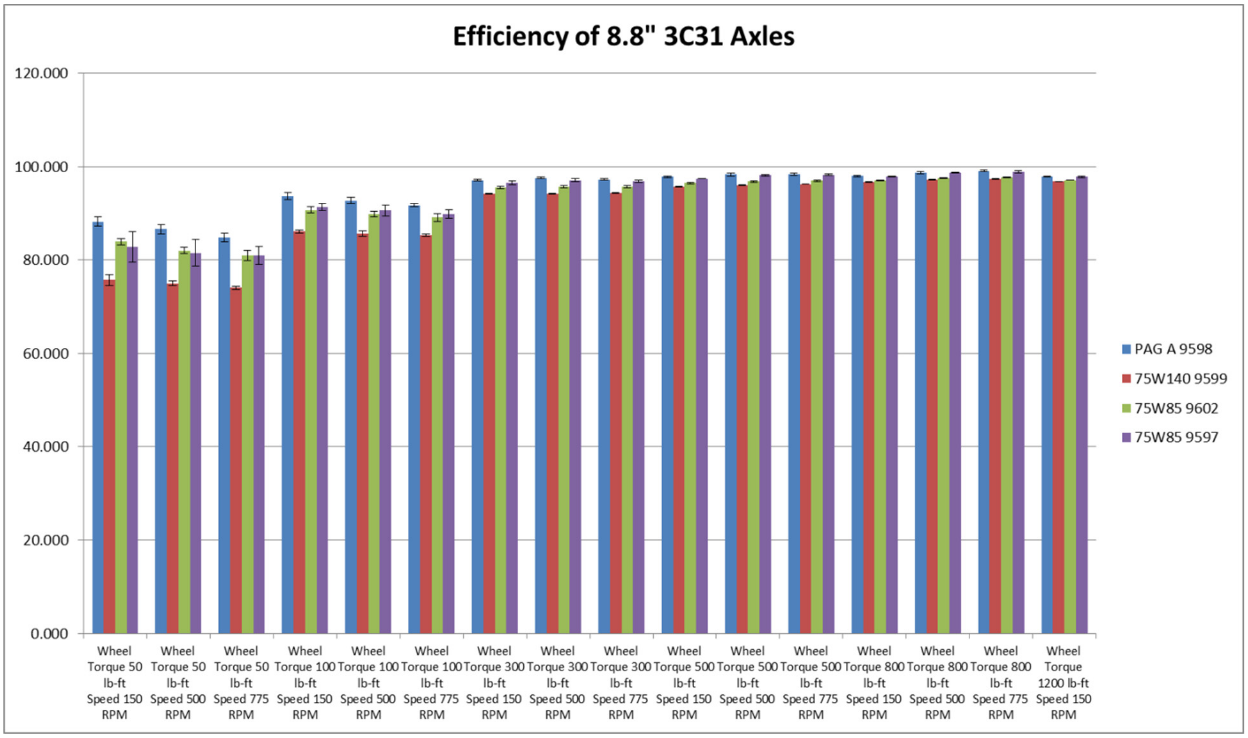

Figure 30. Efficiency data comparing AW0704-A (PAG A 9598) to SAE 75W-140 and SAE 75W-85. Each bar represents the average value of four replicates through the entire efficiency staging testing completed on 1 axle broken in with the respective fluid fill. The error bars represent the standard deviation of the 4 replicates.

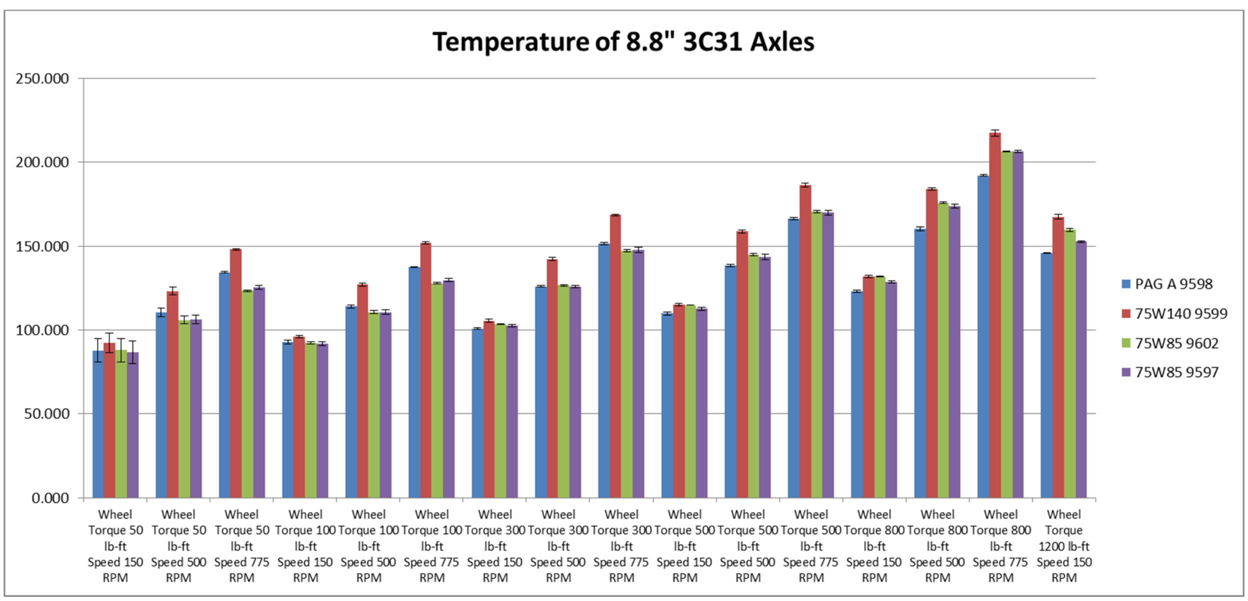

Figure 31.Temperature data from efficiency staging comparing AW0704-A (PAG A 9598)to 75W-140 and 75W-85. Each bar represents the average value four replicates through the entire shown with two runs on SAE 75W-85 lubricant. PAG lubricant exhibited comparable temperature profiles to SAE 75W-85 and vast improvement compared to SAE 75W-140 as efficiency staging testing completed on 1 axle broken in with the respective fluid fill. The error bars represent the standard deviation of the 4 replicates.

shown in Figure 31. In the low torque stages SAE 75W-85 has slightly lower temperatures than the PAG, however in the higher torque stages the PAG exhibits lower operating temperatures. Overall the efficiency and temperature data shows that PAG exhibits performance equivalent or better to SAE 75W-85 and is an all-around improvement to SAE 75W-140.

Figure 32 shows axle efficiency data for PAG co-basestock formulation AU6615-F in comparison to SAE 75W-140 and SAE 75W-85 on a similar production axle. Test repeatability is shown with two runs on reference lubricants, SAE 75W-140, and SAE75W-85 (OEM-1). Lubricant AU6615-F did show improved efficiency when compared to SAE 75W-140, and maintained cooler axle temperatures (Figure 33). A comparison with the reference lubricant SAE 75W-85 showed that the efficiency improvement of AU6615-F compared to SAE 75W-140 can be attributed to the viscosity reduction.

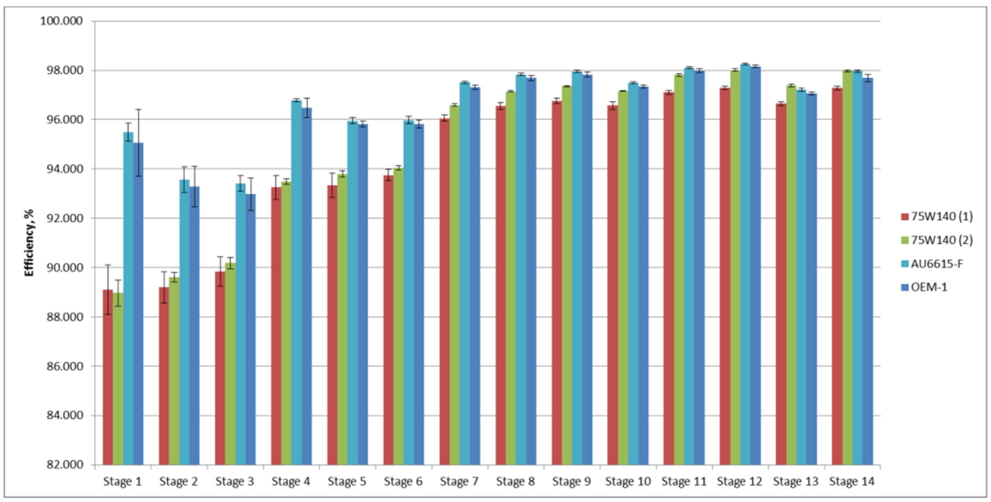

Figure 32. Axle efficiency percent for each stage of testing for the A-B-A style study conducted with SAE 75W-140 and AU6615-F.

3.6 Vehicle Fuel Economy Evaluation

Vehicle fuel economy was evaluated in a chassis roll dynamometer using a recent model year light duty truck equipped with a V-6 engine and six speed transmission under metro/highway cycles. The existing fluid from the vehicle was drained and the axle was thoroughly wiped to remove any residual fluid in the axle and also in the tubes connected to the axle prior to testing with candidate fluids. Six repeat tests were conducted for each fluid and the coefficient of variance (COV) was 0.30-0.40% except for AU6615-F fluid under city cycles, which was 0.8%. Figure 32 show the results in city, highway, and combined metro/highway cycles as a percentage improvement over reference fluid SAE 75W-140 lubricant. Lubricant AU6615-F nearly attains the 2% fuel economy improvement goal over SAE 75W-140 but falls just short. Reference gear oil OEM1 is a commercially available current production gear oil which has similar viscometrics to the AU6615-F. These additional data points serve to justify whether AU6615-F is providing fuel economy improvement based from friction reduction from PAG

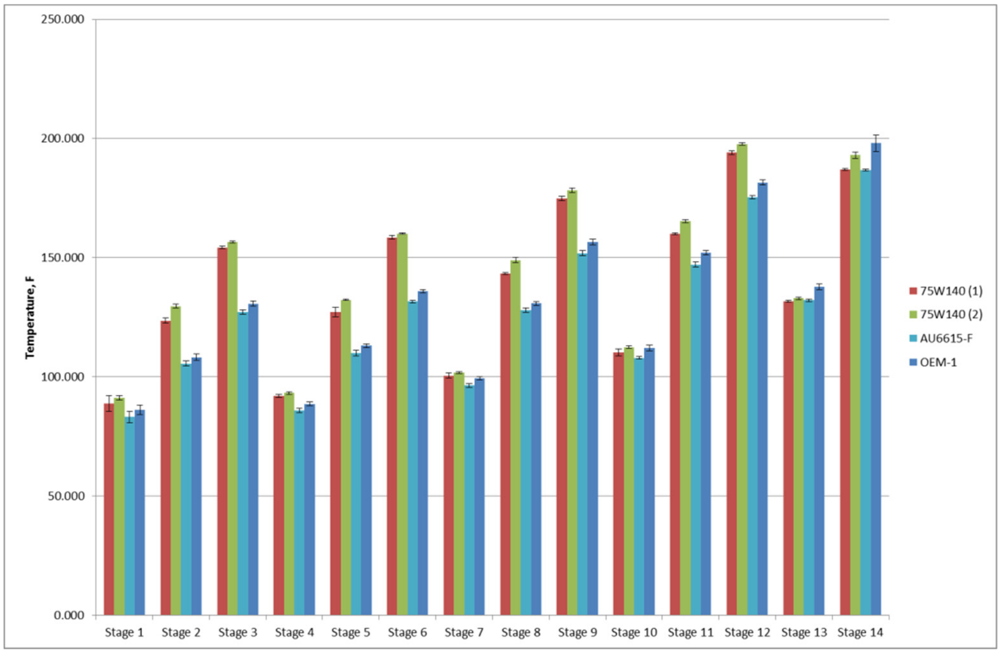

Figure 33. Axle temperature for each stage of testing for the A-B-A style study conducted with 75W-140 and AU6615-F.

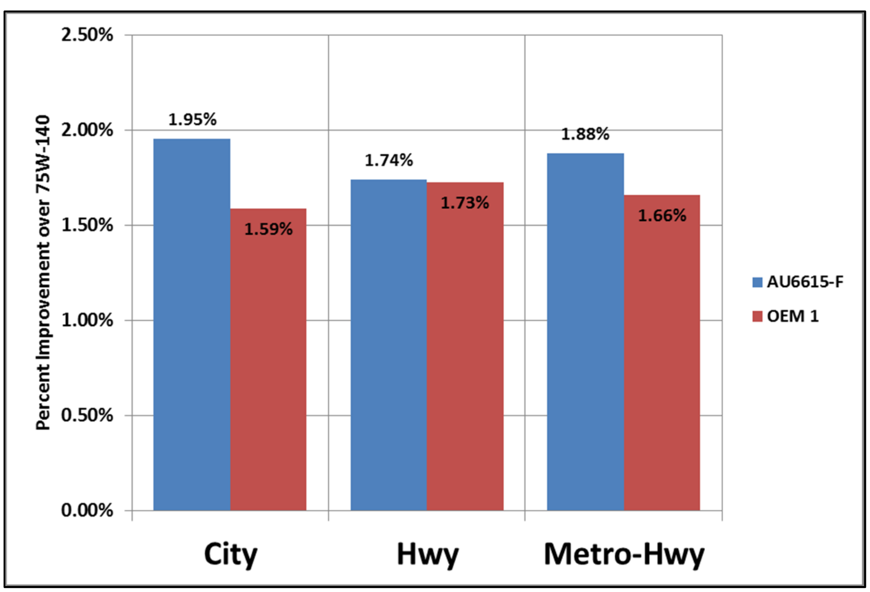

Figure 34. Percent fuel economy improvement over 75W-140 for candidate formulation Au6615-F and other reference fluid OEM 1. Percent improvement presented for City driving, Highway (Hwy) driving, and Metro-Highway (Metro-Hwy) categories.

chemistries or if improvement is from drag loss reduction from lower viscosity. It appears that the gains observed are from viscosity improvements and the PAG co-base option did not offer any additional significant advantage from its unique chemistry.

4. Conclusions

Several bench tests were developed to screen formulations for performance in the L-37 test. Both the pin-on-disk and ball-on-disk methods were able to distinguish between passing and failing L-37 tests. However, due to the limited number of tests run, no firm correlation between the bench tests and performance in the L-37 test could be established. The oxidation test used to screen formulations for performance in the L-60 test appeared to be somewhat predictive for PAG based lubricants, less so for lubricants consisting of blends of oil soluble PAGs and mineral oils.

Development of a PAG-based gear lubricant meeting all requirements of API GL-5 gear lubricants was not successful because of the inability to pass L-42 test. This is primarily due to the limited solubility of available additive component(s) in the PAG.

Formulations with PAG as a co-basestock (15%) with mineral oil conforming to SAE 75W-85 (AU6615-F) passed all GL-5 performance tests. There was up to 7% axle efficiency improvement compared to SAE 75W-140 reference oil but no significant improvement compared to SAE 75W-85 reference oil. Chassis roll dynamometer test showed 1.9%, 1.7%, and 1.8% fuel economy improvement compared to SAE 75W-140 oil in city, highway, and combined metro/highway cycles respectively. However, no significant improvement was observed compared to a full mineral oil based SAE 75W-85.

Formulations with PAG as a co-basestock (15%) with mineral conforming to SAE 75W-85 (AU6615-F) exhibited unacceptable gear durability in proprietary Ford tests.

RECOMMENDATIONS

Future research efforts should be directed in the following areas:
(a) developing a robust additive package for PAG oils to address issues related to wear, additive solubility while maintaining friction reduction benefits.
(b) exploring higher concentration ratio of PAG oil in mineral oil based formulations.

PRODUCTS

Publications/Presentations

"Optimization of a Polyalkylene Glycol Axle Lubricant Formulation" presented at STLE Annual Meeting, Atlanta, May 2017.

PATENT APPLICATIONS/INVENTIONS

No patent applications or invention disclosures were filed under this contract.

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