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Serving the Grease Industry Since 1933 - VOL. 85, NO. 6, JAN./FEB. 2022

In this issue:...

- 4 President's Podium
- 8 Overcoming Obstacles in Water Resistant H1 Specialty Greases Using Polymers
- 28 Modified Fatty Acids as Alternative Soap Thickeners for Lubricating Greases
- 38 NLGI Interviews Dr. William Tuszynski Partner, The Unami GroupQuakertown, PA and Co-Editor, The NLGI Grease Guide
- 50 Electric Motors Retrospective Part 2
- 60 High-Performance Multiuse (HPM) Grease Column



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President's Podium Jim Hunt, NLGI President

6

8

28

50

4

Industry Calendar of Events Welcome New NLGI Members Advertiser's Index

Overcoming Obstacles in Water Resistant H1 Specialty Greases Using Polymers Erik Willett, PhD Functional Products Inc., Macedonia, OH USA

Modified Fatty Acids as Alternative Soap Thickeners for Lubricating Greases Devin Granger, PhD and Shadaab Maghrabi, PhD Ingevity Corporation, Charleston, SC USA

- 38 NLGI Interviews Dr. William Tuszynski Partner, The Unami Group Quakertown, PA and Co-Editor, The NLGI Grease Guide By Mary Moon and Raj Shah
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ON THE COVER Happy Winter!

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PRESIDENT'S PODIUM

Happy New Year NLGI Family,

As always, we hope that you and your families are doing well. The holidays provided a great opportunity to spend quality time with our friends and loves ones and reflect on the blessings in our lives. Another

year has gone by, and a new one has just begun. Strangely enough, this year seems to be starting out the same as the last year ended. Although, we are still dealing with a different

version of the pandemic and possibly the worst global shortages we have experienced in our lifetimes, we must remain hopeful, grateful, and optimistic. The good times are not gone forever, they are just in limbo for now. We will see brighter days in the future and when they come, I truly believe that we will never take them for granted again.

Speaking of gratitude, NLGI would like to extend our appreciation once again to all our valued and loyal members. We want to thank you all for continuous support and commitment to renew your memberships again this year. We promise we are working diligently to ensure that every member receives tremendous value from their NLGI membership. In fact, the NLGI BOD will begin the process this month of revamping the NLGI strategic priorities for 2022. We will continue to work diligently to provide member value. Please don't hesitate to contact NLGI leadership with any areas NLGI can enhance for you or your business.

As most of you are aware, the 2021 NLGI Annual meeting was not only a success but, exceeded overall expectations. Our sincerest gratitude to all of you that attended the annual meeting. It was amazing to see the NLGI family back together again. The 2022 NLGI annual meeting will be held at the Westin Harbour Castle in beautiful downtown Toronto, Canada this year. The dates will be June 12-15th. We highly encourage everyone to attend this year's NLGI Annual Meeting. We anticipate another outstanding meeting with excellent opportunities to network, industry leading papers, highly informative educational classes and much more. Details on registration and hotel will be coming soon, so be on the lookout for additional information. We look forward to seeing you all there.

We wanted to take a moment to provide an update on the HPM program. It continues to be embraced by not only the grease industry end users but, the grease manufactures and marketers as well. We currently have six HPM certified greases from five companies. We anticipate a considerable increase in HPM certifications in 2022. If you have not submitted your grease for HPM, certification or need assistance, please feel free to contact the NLGI office and/or Mike Kunselman at The Center for Quality Assurance.

As we embark on a new year, there is never a better time than now to volunteer for NLGI committees. The NLGI committees are engines that drive the success of the NLGI and it is a great opportunity to not only support the NLGI but, put your special skill sets to great use for a truly great cause. All the committee members leave behind their own legacy of making the NLGI a great organization, you can leave you mark as well. We welcome everyone. Please feel free to reach out to the NLGI office for more details how you can volunteer.

In closing, we want to wish all of you a great year filled with joy, happiness, prosperity and all the blessings life has to offer. We remain grateful and humble for all your contributions to make the NLGI the amazing organization it has become over the years.

Wishing you all the best. Jim Hunt NLGI President



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Industry Calendar of Events 2022

Please contact Denise if there are meetings/conventions you'd like to add to our Industry Calendar, denise@nlgi.org (Your company does not have to be an NLGI membeer to post calendar items.)

2022 ILMA Engage	April 7 - 9, 2022	Ft Lauderdale, FL	ILMA Meetings
Asian Lubricants Industry Association (ALIA) Annual Meeting	April 25 - 27, 2022	Bangkok, Thailand	ALIA Annual Meeting
F&L Week Live! Conference & Exhibition	April 27 - 29, 2022	Bangkok, Thailand	F&L Week Live!
ELGI Annual General Meeting	April 30 - May 3, 2022	Hamburge, Germany	ELGI Meeting
76th STLE Annual Meeting & Exhibition	May 15 - 19, 2022	Orlando, FL	STLE Annual Meeting
NLGI 89th Annual Meeting	June 12 - 15, 2022	Toronto, ON Canada	
Lubricant Expo	September 6 - 8, 2022	Messe Essen, Germany	Lubricant Expo
ILMA 2022 Annual Meeting	October 1 - 4, 2022	Marco Island, FL	ILMA Meetings



Warm Welcome to our New NLGI Members

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Advertiser's Index

ALIA, page 43

ELGI, page 62

F+L, page 45

Lubricant Expo, page 7

Vanderbilt Chemicals, LLC, Inside Front Cover

Zschimmer & Schwarz Inc., page 5



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Overcoming Obstacles in Water Resistant H1 Specialty Greases Using Polymers

Erik Willett, PhD Functional Products Inc. Macedonia, OH USA

Abstract

Water resistance is a critical performance aspect of grease that often becomes an obstacle in developing non-industrial greases for the H1 incidental food contact or biobased grease markets. A specialty grease formulation may perform well in many categories but fail water resistance testing by methods such as ASTM D4049 (water sprayoff) or ASTM D1264 (water washout). Improving water resistance late in the grease development process can involve extensive changes to the formulation.

For example, HX1 base stocks are highly refined, which results in greases with low cohesion and little ability to resist the ingress of water as well as poor solubility for additives needed to fix the problem. This work shows how two HX1 grease polymers may be used in combination with a diverse range of HX1 base stocks to improve the water resistance of full PAO/mPAO base oil blends at significant cost reduction.

Conversely, greases formulated from biobased oils have high cohesion, but most natural or synthetic esters are limited to low ISO viscosity grades and have affinity for water. A higher treat rate of thickener is often used, but this can hinder fluidity and have a negative effect on the economics of the grease. This paper includes examples of how biobased viscosity modifiers can be used to build base oil viscosity, reduce thickener treat rates, and greatly improve water resistance of biobased grease.

In this work, the greases were made using hydrous calcium and calcium complex thickener from pre-formed salts, but the principles discussed in this paper apply broadly to other thickener types.

Introduction

Much of the longstanding experience in the grease industry is based on petroleum oils. Production-wise, 70% of reported grease production is lithium or lithium complex grease, and 86% uses conventional mineral oil with 7%, 5%, and 1% using synthetic, semi-synthetic, or biobased oils, respectively.¹ Best practices and starting points for high performance grease formulating are well established based on the structure and properties of paraffinic or naphthenic oils and bright stocks and their interactions with additive chemistries that were developed alongside greases.

Trends continue to push for reducing the use of petroleum oils in lubricants and greases for various reasons. Highly refined or synthetic base stocks offer improved oxidative and thermal stability and the potential for longer service life. Removal of organic substances containing aromatic carbon groups can greatly reduce the aquatic toxicity of greases, especially those used in environmentally sensitive applications.

Whichever the reason, formulators who wish to develop greases in the specialty areas of NSF H1 incidental food contact (which tend to include synthetic base oils such as polyalphaolefins or PAOs) or environmentally acceptable lubricants often find unique challenges that were easily solved under standard practices in petroleum oil. The body of knowledge and experience is largely focused on industrial lithium grease, while specialty grease formulators are often confronted by a constrained list of allowable raw materials plus a shortage of know-how when exploring new greases or markets.

Properties such as oxidation stability, dropping point, extreme pressure, or wear resistance can typically be adjusted with the use of a simple additive or solved by proper selection of thickener chemistry. For example, an antioxidant can be expected to perform in a wide range of base oil types. However, it can be a much greater challenge to improve water resistance, which is highly complex behavior, may involve many different test methods, and often does not have an easy answer. This work intends to provide formulators with various strategies for improving the water resistance of H1 and biobased greases where there is a limited offering of raw materials available. Fortunately, initial choices of base oil type or grease polymer can avoid water resistance problems that may appear later on when a project matures past basic testing on wear, corrosion, oxidation, etc.

Testing greases for water resistance dates back to the ASTM D4950 specification for automotive grease originally published in 1989. This specification contained water resistance testing according to ASTM D1264 (water washout) as shown in **Table A**. Decades later, the new NLGI High Performance Multipurpose (HPM) grease specification (described in ASTM D7594) adds ASTM D4049 water sprayoff testing with a limit of <40% for the Water Resistant (WR) subcategory.

Grease Specification (Year) NLGI Category ASTM D4049 Limit ASTM D1264 Limit Automotive Service Greases -GB ASTM D4950 (1989) GC <15% @ 79°C Not required GC-LB NLGI High Performance HPM <10% @ 79°C <5% @ 79°C Multiuse Grease (2020) HPM-WR <40% @ 38C

Table A: ASTM D4049 (water sprayoff) and D1264 (water washout) requirements for NLGI grease specifications.

Hydrous calcium grease is an ancient technology, and mixtures of lime and fat were used to make the first soap-based grease as opposed to straight lard.² While old technology, hydrous calcium thickener chemistry is low cost, acceptable for NSF H1 formulations, biobased, and biodegradable. Hydrous calcium has fair water resistance and a dropping point below 100°C, making it uncommon for today's demanding applications.^{3,4} However, these qualities make hydrous calcium grease an excellent platform to demonstrate the versatility of polymers for improving water resistance, specifically water sprayoff (D4049) at 40°C, which is the focus of this paper.

If a Bronze Age technology can be improved to equal or surpass the water resistance of modern calcium sulfonate or aluminum complex greases, then perhaps there is something to learn from antiquity. Higher dropping point and stability of the grease can be achieved by replacing 20 wt% of the calcium stearate with calcium acetate and adding a third temperature stage at 180-200°C to the process (see the Methods section) to allow the stearate and acetate to melt together and form calcium stearate-acetate complex (CaStAcX) grease. Preliminary work with CaStAcX grease was conducted to prepare for future work with the higher temperature water washout test. Results were similar in performance to the calcium stearate data here.

2. Materials and Methods

2.1 Materials

Hydrous calcium and calcium complex greases were prepared from kosher CaSt and CaAc powders. The St/Ac ratio is proprietary. **Table B** summarizes all base oils and viscosity modifiers used to prepare base oil blends for grease at a constant ISO 150 viscosity grade. ISO 150 was chosen because it is a typical viscosity for calcium greases and to allow the use of the popular 750 SUS naphthenic oil in the preliminary work.

Table B: Summary of base oils and polymers with their abbreviated names used in the Results and Discussion

Base Oil Studied (Abbreviated Name)	Supplier (Location)							
600 SUS Solvent Neutral Group ("600SN")	HollyFrontier (Dallas, TX)							
150 Bright Stock ("150BS")	HollyFrontier (Dallas, TX)							
750 SUS Naphthenic Oil ("750Naph")	Cross (Smackover, AR)							
500 SUS White Oil ("500WO")	Citgo (Houston, TX)							
8 cSt Group III, NSF HX1 listed ("8 cSt Group III")	Yubase (Seoul, Korea)							
6 cSt PAO ("PAO6")	Exxon Mobil (Irving, TX)							
10 cSt PAO ("PAO10")	Exxon Mobil (Irving, TX)							
12 cSt Farnesene-based PAO ("Bio PAO12")	Novvi (Alameda, CA)							
5 cSt Alkylated Naphthalene ("AN")	Exxon Mobil (Irving, TX)							
High Oleic Canola Oil, 80% Oleic	Cargill (Minnetonka, MN)							
Polymers Studied (Abbreviated Name)	Supplier (Location)							
HX1 styrene copolymer flake ("HX1 styrene grease	Functional Products Inc. (Macedonia, OH)							
polymer")								
HX1 polyolefin pellet ("HX1 polyolefin grease polymer")	Functional Products Inc. (Macedonia, OH)							
1000 MW PIB ("PIB1000")	Ineos (London, UK)							
2500 MW PIB ("PIB2500")	Ineos (London, UK)							
25 SSI olefin copolymer in white oil ("22 SSI OCP VM")	Functional Products Inc. (Macedonia, OH)							
75 SSI olefin copolymer in white oil ("75 SSI OCP VM")	Functional Products Inc. (Macedonia, OH)							
30 SSI biobased viscosity modifier ("High MW Bio VM")	Functional Products Inc. (Macedonia, OH)							
50 SSI biobased viscosity modifier ("Low Temp Bio VM")	Functional Products Inc. (Macedonia, OH)							
so so blobased theosie, mounter (con remp bio thin)								

** = Not H1 or HX1 but included to further prove certain points. **PIB** refers to polyisobutylene, **PAO** refers to copolymers of C6+ alpha-olefins, and **OCP** refers to olefin copolymer (of ethylene, propylene, butylene, etc.).

H1 CaSt greases prepared at 160°C were treated with 0.5 wt% of a powder form of NSF HX1 high molecular weight phenolic antioxidant. Biobased CaStAc complex greases prepared at 200°C were treated with 2.0 wt% of a mixture of the NSF HX1 phenolic antioxidant and a liquid aminic antioxidant in a proprietary ratio.

2.2 Equipment

500 gram batches of grease were prepared from pre-formed thickener in a three-speed Hobart C-100 mixer with a 10-quart bowl, a B-style Hobart agitator paddle, and an electronic heating mantle (Glas-Col, 600 W, 5000 mL, silicone-impregnated fiber glass, #100AO414). A 120 V Variac was used to regulate the heating mantle power and batch temperature. This equipment was operated in a closed fume hood. Production times and temperatures are described below based on type.

Greases were milled to good texture with a two-roll mill (Seattle Findings #28-281) adjusted to the finest gap setting that would allow material to pass through. The mill was modified with a motor (Dayton 6A198; 1/20 HP, 154 rpm, 20 ft-lbs torque) to power the 4:1 gear reduction. Grease was milled once then adjusted with extra base oil for consistency and grade. Adjusted grease was then milled two more times before screening through a 250 micron steel wire mesh to remove trace pieces of large particles that may interfere with sample preparation in ASTM D4049 water sprayoff. Samples were allowed to rest for 24 hours before testing.

2.3 Hydrous Calcium Stearate and Calcium Stearate-Acetate Grease Production

Hydrous CaSt and CaStAcX greases were prepared at NLGI #2 grade to evaluate water resistance with added polymers. **Table C** summarizes the overall formulations for CaSt and CaStAcX greases with wt % thickener based on base oil viscosity arranged in **Table D**. Water was used to induce and stabilize the unique structure of CaSt hydrate crystals.^{5–7} The low dropping point of hydrated CaSt grease was considered unsuitable for any further work with ASTM D1264 water washout testing at 79°C or preparation of biobased greases due to the water content. CaStAcX grease formulations were developed as an alternative.

CaStAcX greases were made in all biobased base oil blends to prevent the hydrolysis of the high oleic vegetable oil. The higher production temperature (200°C vs. 160°C) was offset by the use of 2 wt% of a phenolic/aminic antioxidant blend. The St-Ac ratio can be adjusted to optimize various properties such as yield, dropping point, and low temperature fluidity.^{2,8–11} No excess calcium hydroxide or other base was added.

All thickeners, base oil, and additives were charged to the kettle (i.e., no hold-out). Minor amounts of extra base oil blend were used to correct for consistency and obtain #2 grade greases.

Component	wt% in CaSt	wt% in CaX
	(White Oil/PAO Studies)	(Biobased VM Study)
Base Oil Blend	42.5-57.5%	34-74%
Liquid Viscosity Modifier	0-15%	0-40%
Calcium Stearate Powder	40%	
Calcium Stearate/Acetate Powder Blend		24%
High MW Phenolic Antioxidant	0.5%	
Phenolic/Aminic Antioxidant Blend		2.0%
Solid Grease Polymer	1.0%	
Water	1.0%	

Table C: Hydrous calcium stearate and calcium stearate-acetate grease formulations

 (note: VM refers to viscosity modifier)

Table D: Typical thickener usage vs. NLGI grade for calcium greases depending on thickener and base oil(note: veg oil refers to vegetable and bio refers to biobased oils)

	Calcium Stearate (CaSt)	Calcium Ste	arate-Acetate	e (CaStAcX)
Grade	in ISO 150 (any oil type)	in ISO 150 PAO	in ISO 32 veg oil	in ISO 150 bio
0	35%	23%	35%	19%
1	37%	26%	39%	21%
2	40%	28%	43%	24%
3	50%	30%	48%	30%

Order of operations:

- 1. Charge components to kettle at room temperature.
- 2. Set the Variac at 80°C and agitate on low speed until the target temperature is reached.
 - Batch will be paste- or dough-like.

- Set the Variac at 150-160°C and agitate on medium speed until the target temperature is reached.
 a. Batch will take on a gelatinous, viscous quality.
- 4. <u>If making CaStAc grease</u>: set the Variac at 180-200°C and agitate on medium speed until the target temperature is reached.
 - Batch will take on a slightly granular texture.
- 5. Mix for 30 minutes if using no grease polymer or using the flake form of HX1 styrene grease polymer. Mix for 90 minutes instead if using the pellet form of HX1 polyolefin grease polymer.
- 6. Inspect the batch every 15 minutes for residual grease polymer on the walls of the kettle to determine when grease is ready to cool.
- 7. Turn off the heat, remove the mantle, and mix on high speed until the temperature of the batch drops to 40°C.
 - Batch will have a grainy but paste-like consistency.
- 8. Mill the batch once on a grease mill.
- 9. Check the batch consistency and adjust with more base oil blend as needed until the target consistency is reached.
- 10. Mill the batch two more times until smooth texture is achieved.
- 11. Press the grease through a 250 micron steel mesh to remove particulates.
- 12. Allow the grease to rest overnight.

The water added to the initial kettle charge was sufficient to produce working hydrous Ca greases despite mixing at 150°C. Store hydrous Ca greases in airtight containers, and clean the kettle immediately before it cools and residual grease has a chance to set.

2.4 Physical Testing and ASTM Methods

Cone penetration and NLGI grade were assessed by ASTM D1403 using a quarter-scale cone penetration device, and the results were converted to full scale for NLGI grading.

Water resistance was evaluated by ASTM D4049 water sprayoff testing (WSO) under the default parameters: a 40 psi stream of 38°C/100°F water for 5 minutes.

Water washout data were not collected in this study. However, the specific grease polymers used in this study have historically performed very well in previous water washout tests with WSO performance. In contrast, some grease polymers are known to perform well in WSO but contribute little or no improvement to washout.

Base oil composition as % paraffinic, % naphthenic, and % aromatic carbon types was collected based on: 1) reported ASTM D2140 carbon profiles for most petroleum oils; 2) general values reported in the literature (Group III and V oils); and 3) known or approximate molecular structures for synthetic oils such as 5 cSt alkylated naphthalene, PAO, PIB, viscosity modifiers, etc.

Results and Discussion

Why do some polymers work differently in various greases?

This work was originally inspired by and intended to answer questions surrounding a specific grease polymer used to improve the water resistance of industrial petroleum-based greases. The styrene-modified HX1 grease polymer flake described in the Materials section has long been sold for use in petroleum greases. Over the years, different formulators reported starkly different experiences with the performance

and texture of finished grease that contained this HX1 styrene polymer. In various cases, the polymer provided exceptionally good (low) WSO and water washout results, had no major effect on water resistance, or resulted in excessive texture and graininess in the finished grease.

Why would one additive be the best and worst additive for petroleum greases at different laboratories?

Based on early findings from this study, it was determined that the type of petroleum oil was critical. Customers had great experiences when they added the HX1 styrene grease polymer to greases that were predominantly based on straight paraffinic oils such as 600SN Group I, 600N Group II, or bright stock. Other customers obtained poor results when they used naphthenic oils such as 750 SUS or roughly equal mixtures of heavy paraffinic and naphthenic oils.

Figure 1 demonstrates the variation in performance of the styrene grease polymer at 1 wt% in NLGI #2 CaSt greases seen prepared using ISO 150 base oil blends of varying composition. The higher solvency naphthenic (200Naph/3500Naph) and paraffinic (600SN/150BS) base oils showed significant reductions in WSO. However, the naphthenic based grease had an unacceptable rubbery texture. The more expensive and highly refined base oils (PAO and white oil/PIB) gave a slight improvement that would not justify the cost of adding the styrene grease polymer.

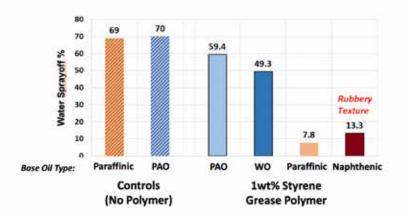


Figure 1: WSO for NLGI #2 CaSt greases using the styrene grease polymer at 1 wt% in various base oil blends (600SN = 600 SUS Group I; 150BS = 150 Bright Stock; PAO6 = PAO 6; mPAO100 = mPAO 100; 500WO = 500 SUS white oil; PIB2500 = 2500 MW polyisobutene; 750Naph = 750 SUS naphthenic oil)

Figure 1 corroborates the range of different experiences reported by formulators who used this styrene grease polymer to make different greases for different brands: excellent WSO improvement, no effect, or rubbery texture but good WSO. This explained *when* the HX1 styrene grease polymer performed differently but did not explain *why*. If the underlying reasons for the variations in performance in different base oil types could be explained, then the parameters of the base oil blends could be tuned to optimize the performance of the HX1 styrene grease polymer.

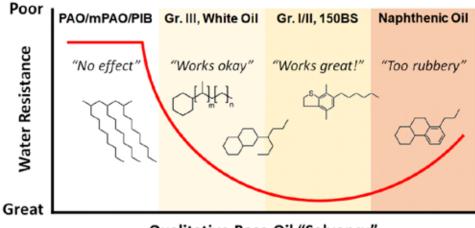
A difference in the properties or performance of any material ultimately depends on its composition and structure. **Table E** compares the composition of various base oils. Paraffinic oils are categorized under API Groups I, II, and III depending on basic properties such as viscosity index and percentage of saturates based on the level of refining. Several types of petroleum base oils derived from naphthenic crude oil are categorized as Group V. PAOs and some Group III (GTL) are prepared by polymerizing short runs of ethylene or CO/H₂ into synthetic crudes that are then distilled as cuts.

Table E: Composition and statistics of various paraffinic and naphthenic petroleum oils. Groups IV and V data were collected from publicly available datasheets from major brands. Group I-III rangeswere obtained from Espada, Jameel, and Ray.^{12–14}

Structure/Properties	Group I	Group II	Group III	PAO	Naphthenic Oil	White Oi
Saturates – Alkanes, %	70	70	79.5	100	40	70
Saturates – Naphthenes, %	25	28	20	0	50	30
Aromatics – %	5	2	0.5	0	10	0
API Group	I.	П	ш	IV	v	v
Typical Viscosity Index	80 - 100	90 - 115	120 - 140	120 - 200	30 - 70	60 - 90
Flash Point, D92,C (ISO 32)	193	226	240	246	170	200
Aniline Point, C	95	100	110	126	80	105

Based on **Table E**, the composition of paraffinic and naphthenic oil varies mostly in the high levels of naphthenic (cycloalkane) and aromatic hydrocarbons in the naphthenic oil. This is regarded as high solvency due to the greater solvating abilities of aromatic and ring-like molecules, similar to the effectiveness of benzene, toluene, turpentines, or limonene as industrial solvents.

The key between the varying experiences formulating greases with the styrene grease polymer is then the difference in solvent quality of the base oil affecting the cohesiveness or adhesiveness of the polymer in the final grease. **Figure 2** reimagines **Figure 1** and the range of customer experiences along a single axis parameter referred to as qualitative base oil "solvency".



Qualitative Base Oil "Solvency"

Figure 2: The range of formulators' experiences and styrene grease polymer performance are functions of base oil composition and solvency. Too much or too little solvency produce different results. The styrene grease polymer selected for this study is sensitive to the choice of base oil, which is true for many styrene polymers.

Figure 3 demonstrates the underlying mechanism why the performance of greases formulated with styrene polymers varies with increasing base oil solvency. Styrene grease polymers have high styrene content making them insoluble in low solvency base oils at ambient temperatures – no long-range interactions or

network is formed. Medium base oil solvency allows the polyolefin midblock of the styrene copolymer to dissolve – the insoluble styrene ends associate to form a mesh-like network. Increasing the solvency further causes the styrene terminal blocks to dissolve in the oil

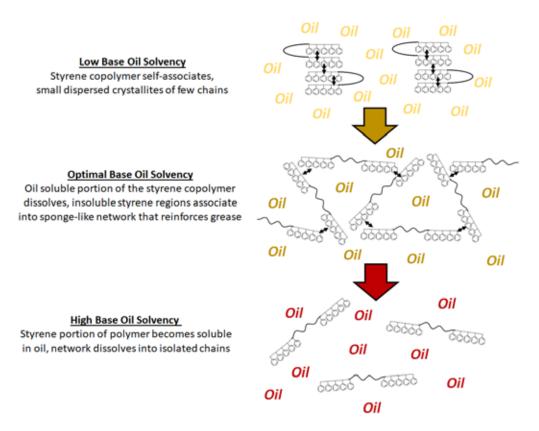


Figure 3: Mechanism for variations in observed performance of greases formulated with styrene polymer with increasing base oil solvency.

Ironically, the styrene grease polymer was found to meet NSF H1 criteria and received HX1 status although the results in **Figure 2** would suggest the polymer performs best in non-HX1 oils. However it was thought that if the solvency of the petroleum or HX1 base fluids could be quantified, measured, and controlled, then it was possible to engineer one or more HX1 oil blends with similar solvency to a Group I or II paraffinic oil. It was hypothesized that the HX1 styrene grease polymer's performance could be tuned with a mixture of HX1 base fluids similar to the examples in **Figures 1** and **2**.

Setting up base oil solvency calculation

Polymer solubility is complex and has been approached from many different levels of theory from "like dissolves like" to Hildebrand's and later Hansen's use of quantitative "solubility parameters" which must be matched closely between base oil and polymer. ^{15–18} Hildebrand solubility is a simple sliding scale like a ruler, while Hansen solubility is a dart board with the height and width representing hydrogen bonding and polarity. It is possible to have 'too much' solvency in one direction such that the solvent or polymer prefers to associate with itself and avoid forming a solution between the two different materials. Thus in **Figure 2** the best performance fell within a narrow range of optimal solvency (Group I/II/bright stock), and increasing the solvency further (naphthenic oil) caused a reversal in water sprayoff performance and poor grease texture.

A system of calculating solvency and estimating WSO based on the calculated solvency was developed here for the HX1 styrene polymer in the NLGI #2 CaSt with ISO 150 base oils.

First, one looks at **Figure 2** and compares the WSO performance by base oil type versus the structure of the base oils shown below in **Figure 4**. It is apparent that naphthenic oils have the greatest solvency arising from their excess aromatic carbon content. It is also apparent that Group III based oil blends gave noticeably better performance than pure PAO/mPAO blends. The major difference between Group III and PAO, where neither contains aromatics, is the naphthenic content. The naphthenic content of naphthenic oil vs. Group I/II paraffinic should also be considered in the first example. Although aromatic structures receive much attention for their solvency it lesser known that even non-aromatic ring structures (naphthenes) have appreciable solvency – think of limonene, a powerful non-aromatic degreaser.

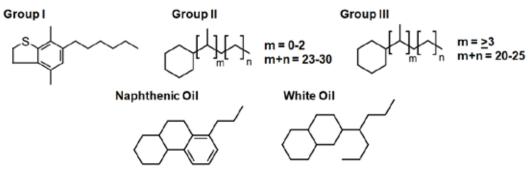


Figure 4: Idealized structures of different paraffinic^{12,14,19} and naphthenic²⁰ oils showing their paraffin and ring contents.

The first iteration of a mathematical model to relate the percentages of different hydrocarbons (alkanes, naphthenes, aromatics) began with plotting the % aromatic carbon of the base oil blends in **Figure 1** versus the WSOs of the greases.

A quick second iteration added a term for % naphthenic carbon but applied a factor to count the naphthenic content as lower solvency than the aromatics. Roughly, WSO is a function of (% aromatic carbon in base oil blend) + (% naphthenic carbon) * N, where N < 1. Microsoft Excel was used to plot this x/y relationship using different polynomial fits and varying the coefficient N until the R² coefficient of fit was optimized to >0.95. N was found to be 0.24 where approximately 1 wt% of aromatic carbon has the same solvency as 4 wt% of naphthenic carbon. Alkane carbon was assigned zero solvency. Molecular weight and size can affect solubility but were excluded; light or heavy oils were treated based only their carbon profile and not viscosity or molecular weight.¹⁸

Figure 5 shows the initial fit between solvency and WSO from the preliminary data points in **Figure 1**. This relationship can be used to engineer an HX1 base oil blend that will work better than other blends with the HX1 styrene grease polymer. The coefficients for % Aromatic C, % Naphthenic C, etc. shown in **Figure 5** and the following figures are based on fitting parameters determined from the final results of this study.

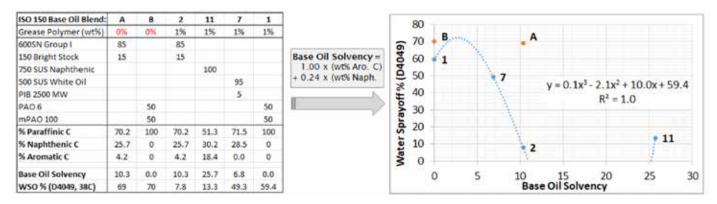


Figure 5: Preliminary fit of the WSO vs. base oil solvency relationship for the HX1 styrene grease polymer. Key features are: sharp reduction in WSO with an increase in base oil solvency from oil blends #7 to #2; and WSO slightly increases when base oil solvency is greatly increased from #2 (ISO 150 Group I) to #1 (ISO 150 naphthenic). More data points are needed to improve the correlation between base oil selection and WSO.

Figure 5 is a crude fit, but it captures the sharp decline in WSO with added base oil solvency and the plateau in WSO once a certain level of WSO performance is achieved. Actually, the WSO for formulation #11 (naphthenic oil) is higher than that of #2 (Group I), and the #11 grease exhibited an undesirable rubbery texture. Both findings support the idea of "too much solvency" established in **Figure 2**.

HX1 base oil solvency vs. optimization of grease water resistance

A rudimentary relationship between base oil solvency and water resistance (as WSO %) has been established. The hypothesis remains that this relationship will remain true for narrowing the range of base oils to only HX1 base stocks. To investigate, this plot and dataset must be explained with more HX1 base stocks. **Figure 6** compares the structures of various HX1 base stocks from petroleum and synthetic sources.

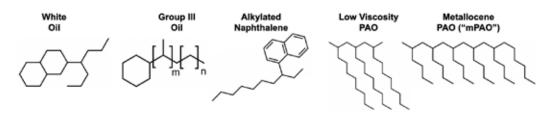


Figure 6: Common petroleum/synthetic HX1 base stocks and their structures. Certain brands of Group III paraffinic oil meet 21 CFR 178.3620(b)(1) regulations and are registered as NSF HX1.

The two sources of solvency, naphthenes and aromatics, must be found from HX1 sources that include: white oils, which are essentially dearomatized naphthenic oil (approximately 30% naphthenic carbon); HX1 Group III oils containing lesser amounts of naphthenes; and alkylated naphthalenes, which are PAOs attached to naphthalene cores and generally the only source of aromatics among the HX1 base stocks (with an estimated 45% aromatic carbon in the 5 cSt grade). Synthetic HX1 base stocks include PAO, mPAO, EPO (ethylene propylene oligomers), PIB (polyisobutylenes), and biodegradable farnesene-based PAO are all isoalkanes with no naphthenic or aromatic content for solvency.

Since aromatics have the highest influence on solvency, a 5 cSt grade of alkylated naphthalene (AN) was selected to add a controlled amount of aromatic carbon to the HX1 base oil blend. The difference in aromatic content was the largest difference in composition between the high performing Group I/bright stock blends (approximately 4-5% aromatic carbon) versus the low performing ISO 150 PAO and white oil blends (0% aromatic carbon).

The appendix contains the individual studies used to derive the Base Oil Solvency calculation. The optimization was performed by starting with % aromatic carbon + % naphthenic carbon, which were corrected by added coefficients. Naphthenic carbon was found to provide about 24% of the solvency of aromatic carbon, so a coefficient of +0.24 was multiplied by the % naphthenic carbon. Synthetic base stocks such as PAO, mPAO, and PB made negative contributions to solvency, so a -0.07 coefficient was applied. Light base oil carried over from using 5-20% of two high MW olefin copolymer viscosity modifiers (25 and 75 SSI OCP in ISO 22 white oil) was found to improve solvency, and a +0.10 coefficient was applied to the % of viscosity modifier.

The overall Base Oil Solvency equation was found to be: Base Oil Solvency = 1.00 x [% Aromatic Carbon] + 0.24 x [% Naphthenic Carbon] - 0.07 x [% PAO + mPAO + PB] + 0.10 x [% HX1 Viscosity Modifier]

Figure 7 compiles the individual formulations (referenced by lab notebook page number) used to map out the solvency versus water sprayoff curve. A third order polynomial produced the closest fit and produced a curve similar to the predicted model in **Figure 2** and the preliminary curve from in **Figure 5**. However, the exact relationship was better fit by a curve with the shape of a square root sign ($\sqrt{}$) due to the plateau in water sprayoff at approximately 70% from ≤ 6 solvency and V-shaped slope from 8 - 12 solvency. A base oil solvency of 10 - 10.5 was found to be ideal for the HX1 styrene grease polymer in this study.

	Pr	elimir	nary St	udy	Wh	ite Oil	+ PIB	+ AN	PA	0 + m	PAO +	AN		- w/	H1 Vi	scosity	Modifi	ers	
Lab Formula #:	Α	в	2	11	7	9	16	12	1	10	15	17	18	19	27	30	31	28	32
600SN Group I	85		85																
150 Bright Stock	15		15																
750 SUS Naphthenic				100															
500 SUS White Oil					95	82.5	69	68					86						
8 cSt Group III														75	70	68.06			
PIB 2500 MW					5	7.5	16												
PAO 6		50							50	40	31	15					52.5	50	
PAO 10																			57.4
mPAO 100		50						22	50	50	44	50							
22 SSI H1 OCP VM													6.7	12.5	12.6		17.1	17.1	
75 SSI H1 OCP VM																17			14.3
5 cSt Alkylated Naph.						10	15	10		10	25	33	7.3	12.5	17.4	14.9	30.4	32.9	28.3
Grease Polymer (wt%)	0%	0%	1%	1%	1%	1%	1%	1%	1%	1%	1%	1%	1%	1%	1%	1%	1%	1%	1%
% Paraffinic C	70.2	100	70.2	51.3	71.5	70.8	81.0	75.1	100	95.5	88.8	85.0	69.2	78.0	76.6	76.6	81.9	80.7	83.3
% Naphthenic C	25.7	0.0	25.7	30.2	28.5	24.8	12.0	20.4	0.0	0.0	0.0	0.0	27.5	16.2	15.3	16.4	4.5	4.5	4.0
% Aromatic C	4.2	0.0	4.2	18.4	0.0	4.5	7.0	4.5	0.0	4.5	11.3	15.0	3.3	5.9	8.0	6.9	13.7	14.8	12.7
Base Oil Solvency	10.3	0.0	10.3	25.7	6.5	9.9	8.7	7.9	0.0	0.0	6.0	10.3	10.6	11.0	13.0	12.6	12.8	14.1	11.1
WSO % (D4049, 38°C)	69	70	7.8	13.3	49.3	8.0	26.0	32.4	59.4	63.0	58.9	58.9	8.7	27.3	35.6	39.0	56.4	71.2	19.5

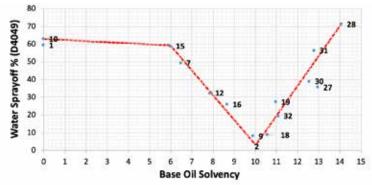


Figure 7: Top – Full table of NLGI #2 grade H1 CaSt grease formulations with 1 wt% HX1 styrene grease polymer and the resulting relationship between base oil solvency and performance of the grease polymer (i.e., water sprayoff). Bottom – Plot of water sprayoff vs. base oil solvency for the H1 CaSt greases. The general fit is a 3rd order polynomial, but the red line was added manually to show the 'square-root' shape of the solvency-WSO relationship. The Appendix contains individual studies that were used to determine the weighting coefficients for Base Oil Solvency. This general method can be applied to troubleshooting formulation vs. performance issues in a number of other grease applications. The goal of the work here is not to solve the problem of water resistance in CaSt for one specific polymer but to use this example as a simple framework for tackling problems in more complex grease systems. This process can be applied to solvency-based problems relating to both the compatibility of grease polymers and even grease thickeners in today's very wide range of base oil options.

The process consists of determining which factors of base oil composition affect grease performance, quantifying base oil solvency composition as a weighted sum of those factors, and then establishing a correlation between solvency and performance. This could prove invaluable for more complicated, multi-step grease thickeners like aluminum complex, calcium sulfonate complex, and polyurea.

One final note is that the solvency-WSO equation could be amended to include HX1 ester base oils such as vegetable oils, oleates/stearates, and polyols as additional sources of solvency. However, the scope of this work focused on only hydrocarbons as a simple example of this new approach, and thus, primarily white oils and alkylated naphthalenes were used. The same approach can be applied to esters, but it would be necessary to weight their different contributions to solvency by their Non-Polarity Index (i.e., the number of ester groups per molecular weight) or another measure of polarity.²³

Other Polymers I – HX1 polyolefin grease polymer for PAO

A second HX1 polyolefin grease polymer with different chemistry is available to improve mechanical stability and reduce oil bleed in certain H1 greases. This polymer is known to be less polar than the HX1 styrene grease polymer, and its performance varies between base oils like that of the HX1 styrene grease polymer, although the specific responses of the two polymers are different. The HX1 polyolefin grease polymer pellet was added to grease batches in the same manner as the HX1 styrene grease polymer, but the final stage of heating was extended to 90 minutes to allow the larger pellets more time to dissolve fully into the grease.

Optimization of the HX1 base oils was performed on this second polyolefin grease polymer using the solvency-WSO parameters established with the HX1 styrene grease polymer, i.e., WSO is a function of (wt% aromatic carbon) + 0.24 * (wt% naphthenic carbon) - 0.07 * (wt% PAO or mPAO) + etc.

If the approaches developed earlier in this work could be applied successfully to a second grease polymer system, then it is most likely that it could apply to many more systems. Since the HX1 polyolefin polymer lacks styrene, one can anticipate the required solvency and solvency-WSO relationship equation would be different from that of the styrene polymer.

Figure 8 demonstrates the effectiveness of the styrene-free HX1 polyolefin grease polymer in treating low solvency HX1 base oils without the complexity of adding alkylated naphthalene. This polymer appears to be less base oil selective than the HX1 styrene grease polymer, which allows for the use of higher performance PAO and mPAO. However, 1 wt% polymer was not sufficient to produce acceptable WSOs, while 2 or 3 wt% polymer was ideal.

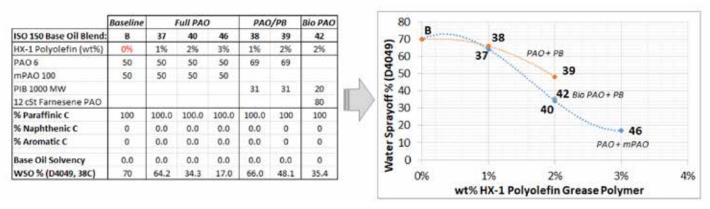


Figure 8: HX1 polyolefin polymer works well and reduces the WSO below the HPM-WR limit (<40%) at 2 wt% in PAO/mPAO base fluids, but performance lags in PAO/PIB. Using the highest viscosity non-metallocene PAO available (farnesene based 12 cSt PAO) to reduce the amount of PIB to replace mPAO to 20 wt% gave performance similar to that of the PAO/mPAO. There may be a critical limit to the PIB and its effect on reducing solvency in the 20-30 wt% range.

An interesting effect was noted by blending the HX1 styrene and HX1 polyolefin grease polymers at 2 wt%. The polyolefin grants high amounts of cohesion, and the styrene polymer gives high amounts of adhesion; the performance was best when both effects were both present. **Figure 9** shows the effects of various ratios of the two polymers at a total usage rate of 2 wt%. This gave performance similar to that of the HX1 polyolefin polymer at 3 wt%. Thus, the mixed polymer approach would yield lower treat rate and cost to achieve very low WSO.

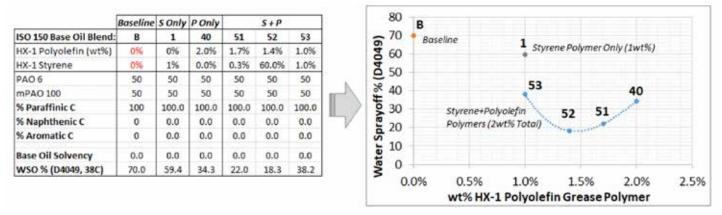


Figure 9: Investigating the synergy between two different types of semicrystalline HX1 grease polymers

Thermoplastics such as the HX1 styrene polymer or polyolefin are known to have synergies when mixed in applications, e.g., engineered materials or thermoplastic sealants and adhesives. This approach appears to also be valid in a grease application. These are known "polymer alloys" where two different polymers preferentially mesh to form a mixture with properties that differ from those of the two components.²⁴ Regardless of whether this effect is used to improve a grease, this study showed that using two grease polymers can result in a unique interaction. When the HX1 styrene polymer was used by itself in the previous study, it had no effect; the combination of the HX1 styrene polymer with the HX1 polyolefin copolymer in a full PAO/mPAO grease halved the WSO value.

Other Polymers II – HX1 polar grease polymer for HX1 vegetable oil based grease

Existing work with biobased esters showed that one should use polar or hydrogen bonding polymers for treating HX1 biobased esters.²⁵ Figure 10 compares a variety of possible polar and hydrogen bonding

functional groups that may be used to treat ester fluids. CaStAcX grease was prepared for biobased grease production to avoid the inclusion of water at high temperature that could risk hydrolysis of the vegetable oil base fluid.

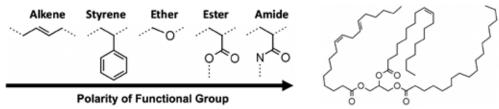


Figure 10: Structural features of ester-soluble polymers and viscosity modifiers (left) versus vegetable oil (right).²⁵ Electron density from C=C or C=O double bonds plus heteroatoms like N or O attached to C induce polarity.

The primary concern with vegetable oil based greases is the abnormally low base oil viscosities, ISO 32–36, compared to traditional petroleum grease made using ISO 100–460 or higher. The low base oil viscosity can be corrected by adding high MW polymer viscosity modifiers or low-medium MW polymeric base oils.

An NLGI #2 CaStAcX grease was produced in ISO 32 vegetable oil without the addition of polymers. The WSO was approximately 96%. Since the very low base oil viscosity was the most significant difference from prior grease formulations, the base oil viscosity was increased using several commercial biobased viscosity modifiers.

CaStAc grease was made using an ISO 150 blend of high oleic canola oil and one of three biobased viscosity modifiers to observe the effects of higher base oil viscosity:

- 50 SSI biobased viscosity modifier with slight tackiness, referred to as High MW VM;
- 30 SSI biobased viscosity modifier with excellent cold temperature fluidity, referred to as Low Temperature VM; and
- 29 SSI biobased viscosity modifier with NSF HX1 registration, referred to as HX1 Bio VM.

Grease Formulations by wt%	No VM (Control)	High MW Bio VM	Low Temp Bio VM	HX1 Bio VM
Calcium Stearate/Acetate Powder	40.0	30.0	28.5	22.5
Antioxidant Blend	2.0	2.0	2.0	22.5
Biobased VM (various)		11.6	12.7	12.8
High Oleic Canola Oil	58.0	56.4	56.8	62.7
-				
NLGI Grade	#2	#2	#2	#2
Base Oil Blend ISO VG	32	150	150	150
Water Sprayoff % (D4049)	99.0%	95.5%	74.8%	31.2%
wt% Thickener + wt% VM	40.0	30.0	28.5	35.3
Meets European Ecolabel?	Yes	Yes	Yes	Yes
Total "A" Content	89-91%	90-92%	89-91%	90-92%
(Biodegradable)				
Total "C" Content	9-11%	8-10%	9-11%	8-10%
(Non-Biodeg., Non-Accumulative)				
Total "D" Content	100%	100%	100%	100%
(Non-Toxic)				

Table F: Comparison of different biobased CaStAc greases prepared with ISO 150 base oilblends vs. a straight ISO 32 vegetable oil

All four formulations meet the European Ecolabel standard for biodegradability and aquatic toxicity of environmentally acceptable grease. Up to 15 wt% "C"-rated content (non-biodegradable but non-bioaccumulative) is allowed in Ecolabel greases. Both the CaX thickener and active polymers in the biobased VMs contain some "C" content, but the cumulative sum is well within the limits. These starting point formulations leave room for roughly 5 wt% more "C" content that may be taken up by extra performance additives like AW/EP and corrosion inhibitors.

Three polymer chemistries all produced different results. Increasing the base oil viscosity had a dramatic effect on the efficiency of the CaX thickener and lowered the thickener usage by 25–45%. In all three cases, the biobased viscosity modifiers replaced more than their wt % of CaX thickener (i.e., the total content of thickener plus VM was lower than the control).

The high molecular weight biobased VM produced negligible improvement in WSO. While the consistency was #2 according to the cone penetration test (ASTM D1403), the grease behaved similar to a fluid (or 'thin') when a sample was manipulated or loaded onto the WSO jig. It appears this polymer chemistry may be non-Newtonian as it provides high consistency under sudden impacts (yielding a #2 grade under in the impact of the cone) but is very soft or flows while resting on the plate in the WSO instrument. This rheological phenomenon implies a dilatant material.

The low temperature biobased VM was most economical in that the usage of both the thickener and biobased VM were lowest. WSO was slightly improved to 75%, and the consistencies of the grade in cone penetration testing and at rest were more consistent.

The HX1 biobased VM was most effective in lowering WSO to 35.3% and within HPM-WR performance requirements. This chemistry resulted in a tougher grease than all other biobased greases prepared in this study. Comparing the molecular weight, inherent toughness of the polymers, and the amount of polymer delivered between the three different biobased VMs showed that the toughness of the pure polymer and % polymer delivered were the key factors. The HX1 biobased VM was most effective in those two categories.

A second study to achieve very low ($\leq 20\%$) WSO with ISO 150 base oils and CaX thickener was not successful. Instead, increasing the base oil viscosity to very high levels was effective. A #3 CaX grease with vegetable oil was cut back to a #2 grade with 35–40 wt% biobased VM. The results were referred to as high MW VM: 35% WSO at ISO 1500 base oil viscosity; low temp VM: 12% WSO at ISO 3200 base oil viscosity; and HX1 bio VM: 8% WSO at ISO 12000. This follows the same trend in VM chemistry vs. WSO performance as the ISO 150 biobased study above. Ultimately, using very high viscosities to control water resistance is probably not feasible due to the potential impact on low temperature fluidity. This small high viscosity study showed diminishing returns on ISO VG vs. WSO after ISO 2200.

Further work was performed to achieve very low WSO with CaX at a reasonable ISO 150–460 viscosity range including modifications to the St-Ac ratio, inclusion of calcium sulfonate as a pseudo-calcium sulfonate complex, and more. 35% WSO using the HX1 biobased VM appeared to be the lower limit for CaX in vegetable oil. The high polarity and hydrophilic nature of both CaStAc and vegetable oil appeared to be major obstacles.

A graphical summary of the three studies

Overall, the approach to selecting HX1 grease polymers for water resistant H1 greases follows an axis similar to the sliding scale, ruler-like Hildebrand solubility parameter theory. **Figure 11** summarizes the

spectrum of base oil solvency and types in the HX1 or biobased specialty markets and the optimal type of polymer to choose when trying to improve water resistance for specifications such as the new HPM-WR and D4049 WSO test method. These matches are presented along one axis in a Hildebrand-style sliding scale of polymer compatibility with different base fluids.

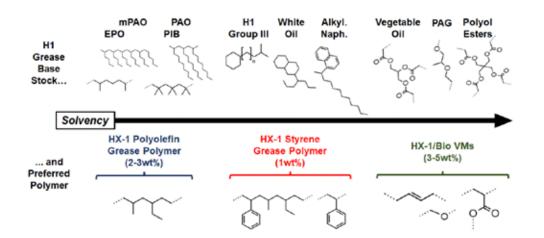


Figure 11: Structural similarities between available HX1 base fluids and HX1 grease polymers that are suitable to reduce WSO based on the findings of this study. This plot holds true for non-HX1 materials but would be greatly expanded and made more complex by the inclusion of industrial options.

Conclusions

Grease formulation is a complicated process, especially when a project falls outside of the established knowledge that was predominantly based on petroleum oils. Certain performance aspects can be changed easily with the addition of less than a few percent of an antioxidant or a corrosion inhibitor. However, when the water resistance of a grease is deficient, addressing this issue can involve complete reformulation, which can be problematic in the late stages of a project when this problem tends to arise. This work is intended to extend the knowledge base of how to incorporate water-resistant grease polymers at the initial stage of formulating greases for NSF H1 or biobased applications.

It may be unrealistic for formulators to choose base oils in order to make 0.5–3 wt% of an HX1 grease polymers perform correctly in their incidental food contact greases. However, the number of overall polymer chemistries suitable for NSF H1 applications is small, and the number of actual suppliers producing those grades of polymer to NSF/FDA tolerances is smaller still.

The next steps of this work are to take the opposite approach and design polymers to suit specific base oils using the relationships developed in this paper. Almost a dozen structurally different NSF HX1 polymers have now been made available to investigate and treat water resistance for a wide range of greases.

Another area to investigate is sources of solvency other than alkylated naphthalenes for NSF H1 formulations. Many natural and synthetic esters are strong polar solvents and routinely used to improve the solvency of PAO gear oils to enable high teat rates of additives. This H1 grease study was limited to hydrocarbons to establish the groundwork for this approach without involving the complexity of different kinds of solvency associated with polarity and hydrogen bonding from esters.

APPENDIX A - Data Fitting the Coefficient for Base Oil Solvency Calculation

Figure 12 begins to include formulations using white oil + AN or PAO + AN as higher solvency HX1 base fluid blends to properly dissolve the HX- styrene grease polymer according the hypothesized solvency-WSO response curve established in **Figure 2**. White oil and AN blends worked well to correct the deficiencies in HX1 base stock solvency and improve WSO. However PAO and AN, specifically with high amounts of mPAO, did not perform as expected in the solvency-WSO model.

Base oil blends containing mPAO performed as if lower solvency. Another correction to the solvency-WSO model equation was added with a negative coefficient for the wt% mPAO. After retesting the fit, the remaining low viscosity PAO also appeared to reduce overall base oil solvency. The negative coefficient was extended to both the low viscosity PAO and mPAO. WSO is a function of (wt% aromatic carbon) + 0.24 * (wt% naphthenic carbon) -0.07 * (wt% PAO or mPAO); if the calculated base oil solvency is less than zero then it becomes zero. In effect, adding 14% PAO or mPAO removes the equivalent of 1 wt% aromatics in solvency. This is interesting as the choice to use PAO or mPAO are actively resulting in a net loss of solvency versus petroleum and white oils.

Figure 13 recalculates the Base Oil Solvency based on these observations and replots WSO vs. solvency with an added correction factor for the reduction in solvency when using mPAO or PIB (synthetic polyolefin oligomer base stocks).

	in/t/a/ (Pata Po	ints		White	Oil + PB	+ AN Se	wles	PAC +	mPAO +	AN Seri	les	22	_											
ISO 150 Base Oil Blend:	A		2	11	7	9	16	12	1	10	15	17].	80	1										
Grease Polymer (wt%)	054	056	1%	1%	1%	1%	1%	1%	1%	15	15	15											A		
600SN Group I	85	-	85										1	a 70	•			1				*	•		
150 Bright Stock	15		15										1	(640) 0	1				10				. 15	- 1	17 .
750 SUS Naphthenic				100									1	8		S								1.5	20
500 SUS White Oil					95	82.5	69	68						0 50		1.1	Sec. 1			• 7					
P18 2500 MW					5	7.5	15						1	₩ 40)			See.	10						
PA0 6		50							50	40	31	15		A 30					110	******	(+)+)	• 12			
mPAG 100		50						22	50	50	44	50										+ 16			
5 cSt Alkylated Naph.						10	15	10	3420.00	10	25	33		5 20	¥							-			
% Paraffinic C	70.2	100	70.2	51.3	71.5	70.8	81	75.1	100	95.5	\$3.8	85	Base Oil Solvency =	6	y = -0	0.0x ³	+ 0.8	kx2 - 1	9.2x	+68	0				
% Naphthenic C	25.7	0	25.7	30.2	28.5	24.8	12	20.4	0	0.0	0.0	0	1.00x(wt% Aro. C)	10 Mar			R2 =	0.3				9=	2		
% Aromatic C	4,2	0	4.2	18.4	0.0	4.5	7	.4.5	0	4.5	11.3	15	+ 0.24x(wt% Naph. C)	- 0)	11.	-	-		12.1		-1-			-
Base Oil Solvency	10.3	0.0	10.3	15.7	6.8	10.4	9.8	9.4	0.0	4.5	11.3	14.9			0 1	2	3 4	5	6		8 9		1 12	13 1	4 15
WSO % (D4049, 18C)	69	70	7.8	15.3	49.3	8.0	26	32.4	59.4	63.0	58.9	60.2						- 1	Base	OILS	olve	ncy			

Figure 12: Expanded WSO-solvency relationship including alkylated naphthalene as a source of HX1 aromatic carbon. Correlation with white oil based formulas was good but the inclusion of later PAO based formulas skewed the relationship. Some effect of the synthetic base oils has not been factored into the WSO-solvency equation. Data point #11 has been removed as an outlier due to the odd texture of the grease and it's being the only naphthenic formula.

	Initial I	Data Po	ints		White	01 + P18	+ AN S	eries	PAC +1	mPAO +	AN Seri	es														
ISO 150 Base Oil Blend:	A	B	2	11	7	9	16	12	1	30	15	17		80												
Grease Polymer (wt%)	0%	0%	1%	1%	1%	1%	1%	1%	1%	1%	1%	1%			B	10	1.0	Sec					A			
6005N Group I	85		85											(600bd)	12			100	S							
150 Bright Stock	15		15											8 60	1	0			1	15			- 17			
750 SUS Naphthenic				100										8	1				- 0							
500 SUS White Oil					95	82.5	69	68						× 50						1.7						
PI8 2500 MW					5	7.5	15							₩ 40						1						
PAO 6		50							50	-40	31	15	1								- 12					
mPAD 100		50						22	50	50	44	50										16				
5 cSt Alkylated Naph.						10	15	10	10000	10	25	33	Base Oil Solvency =	\$ 20							12	C.				
% Parattinic C	79.2	100	70.2	51.3	71.5	70.8	BI	75.1	100	.95.5	\$8.8	85	1.00x1wt6 Arp. C)		y =	0.2x ¹	- 4.2	x2 +	17.5	x + 6	1.2	N.				
6 Naphthenic C	25.7	0	25.7	30.2	28.5	24.8	12	20.4	0	0.0	0.0	0	+ 0.24x(ht% Naph. C)	10 Mate	(T)		R ²	- 1.	0			9 .	- 2			
% Aromatic C	4.2	0	4.2	18.4	0.0	4.5	7	4.5	0	4.5	31.3	15	- 0.07x(PAO/mPAO/PIB)	- 0		1.1.1										
Base Oil Solvency	10.3	0.0	10.3	23.7	6.5	9.9	8.7	7.9	0.0	0.0	6.0	10.3	n		0 1	1 2	3	4 5	6	7	-		0 11	12	13 14	1 15
WSO % (D4049, 38C)	69	70	7.8	13.3	49.3	B.0	26	32.4	59.4	63.0	58.9	60.2							Base	Oil	Solve	ency				

Figure 13: Corrected version of Figure 12 by adding a -0.07 * (wt% PAO + wt% mPAO + wt% PIB) correction factor. Data point #17 was a gross outlier likely due to the very high amount of alkylated naphthalene used to counteract the insolvency of mPAO 100. The span between data points #1 / #10 and #15 should be treated as a plateau of 60% WSO until a base oil solvency of at least 6.5 is exceeded.

<u>APPENDIX B – A Closer Look at the Effect of Viscosity Modifiers</u>

By limiting or eliminating the usage of mPAO (ISO 1000+) it became difficult to formulate the HX1 base oil blends to ISO 150. 500 SUS white oil with 5 cSt alkylated naphthalene (ISO 32) requires a thickener to meet ISO 150. If using HX1 Group III oil then the problem becomes worse, as Group III is only available up to the ISO 46 "8 cSt" grade.

HX1 polyisobutylenes ("PIB") were sourced as an mPAO replacement. After fixing several new formulations with Group III + PIB + AN, it was found that PIB causes faster loss of solvency than PAOs. WSO is a function of (wt% aromatic carbon) + 0.24 * (wt% naphthenic carbon) - 0.07 * (wt% PAO or mPAO) - 0.084 * (wt% PIB). However, PIBs are ISO 5000 to 100,000+ and can increase the base oil viscosity at lower wt% than the PAO. This results in PIBs producing a higher solvency ISO 150 than mPAO due to the lower wt% PIB than mPAO although the penalty to solvency per wt% is higher on PIB.

If using high amounts of polymer (mPAO and PIB) to increase viscosity reduces base oil solvency and WSO performance then a traditional viscosity modifier or viscosity index improver may be more effective. VI improvers are typically 5-10 wt% of a high molecular weight polymer (10K-300K Mw) in light oil.^{21,22} Often less than a percent of active polymer is in the final product. This would greatly reduce the usage of polymer in the base oil as seen with replacing mPAO with PIB.

Two concentrated commercial HX1 viscosity modifiers of greatly different shear stability (SSI by ASTM D6278) and MW were used to replace PIB in preparing HX1 ISO 150 base oils in white oil or Group III plus the 5 cSt alkylated naphthalene:

- 22 SSI olefin copolymer in white oil (3000 cSt @ 100°C) "Low MW OCP VM"
- 75 SSI olefin copolymer in white oil (3000 cSt @ 100°C) "High MW OCP VM"

Figure 14 demonstrates the new fit using the rules set for aromatic, naphthenic, and PAO/mPAO/PB content after including the OCP VM as a means of minimizing the content of high viscosity synthetic base stocks like the mPAO and PB. The fit becomes closer than **Figures 12 and 13** but around Base Oil Solvency 10 there are some poor fits to the data.

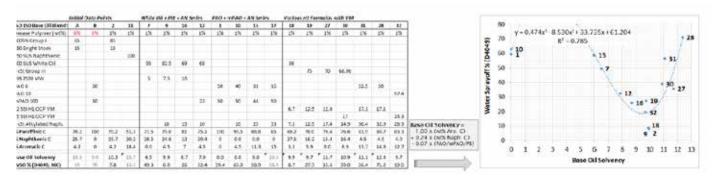


Figure 14: Initial fit of the VM modified base oil blends in an attempt to keep the use of PIB and mPAO low but still achieve ISO 150 oil blends.

A variety of formulations in 500 white oil, 8 cSt Group III, and PAO 6 were prepared at ISO 150 using 22 SSI and 75 SSI liquid OCP VMs. Formulations were built using the establish base oil solvency calculation results established so far and to achieve an optimal 9-10 solvency rating for a target WSO of <20%. Formulations which should have been rated around 10 base oil solvency did not perform as expected. It was apparent than the OCP VMs required their own factor.

Initially, it was thought that the addition of viscosity modifier (VM) was further reducing solvency since calculated solvency was not correlating well with the addition of OCP VM in early models. More AN was added to compensate which further worsened WSO performance. Above 30% AN caused high WSO which was observed previously in the data point #17 outlier. This also supports the earlier hypothesis that the increase in WSO and unpleasant rubberiness in the full naphthenic oil formulation was due to too much solvency in the base oil. However, too much solvency in these later cases with high treat of OCP VM resulted only in higher WSO and not poor texture.

Rather than applying a solvency penalty, the data was best fit by adding a positive contribution to solvency with wt% OCP VM. WSO is a function of (wt% Aromatic C) + 0.24 x (wt% Naphthenic Carbon) – 0.070 x (wt% PAO + mPAO + PIB) + 0.10 (wt% VM). Figure 10 shows the new solvency-WSO equation and fit using this new factor.

This positive contribution of solvency and addition of too much solvency is most likely due to the light mineral oil (ISO ~22) in the viscosity modifier. Most commercial viscosity modifiers are a few percent of very high MW polymer (30K - 200K Mw) in a light ISO 20-25 oil. While all base oil blends were formulation to a constant ISO 150 the oil blends with added OCP VM at 5-20wt% will contribute very light base oil which has a greater capacity for solvency than higher MW heavy base stocks. The fitting in **Figure 10** (using the final coefficients in **Table G**) and prior plots was based on a 3rd order polynomial regression. Looking closely at **Figure 10** is apparent that the funnel shape from base oil solvency 6 to 14 is more of a sharp V or cone shape rather than a gentle curve. The response between WSO and base oil solvency is flat from 0 to 6 base oil solvency. This gives an overall shape like a square root sign ($\sqrt{}$) to the relationship between too little solvency, optimal solvency, and too much solvency vs. WSO performance of the HX1 styrene grease polymer.

Table G: Summary of contributions to solvency in the base oil solvency-WSO relationship established for theHX1 styrene grease polymer at 1 wt% in ISO 150 NLGI #2 calcium stearate grease. Multiply each row by itscoefficient and take the sum for the calculated base oil solvency. Some blends (100% PAO) will result in negative

Base Oil Composition	Multiply by
wt% Aromatic Carbon (by ASTM D2140)	1.000
wt% Naphthenic Carbon (by ASTM D2140)	0.240
wt% PAO or mPAO or PIB	-0.07
wt% Viscosity Modifier (in light oil)	+0.10

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Modified Fatty Acids as Alternative Soap Thickeners for Lubricating Greases

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Abstract

In 2020 and 2021, the COVID-19 pandemic has highlighted the fragility of global supply chains. Port closures in supply hubs have the potential to disrupt the 12-hydroxystearic acid (12-HSA) supply chain due to the vast majority of the supply used for grease thickeners passing through these hubs.[1] The introduction of effective alternatives to 12-HSA for use as grease thickeners can help grease manufacturers by alleviating their dependence on imports and reducing the possibility of production disruptions caused by supply chain breakdowns.

This paper describes initial experimental results that indicate that modified fatty acids (MFAs) not derived from castor oil can be used to form thickeners that are shear stable. The developmental MFAs can be used as obtained or blended with other fatty acids to form grease thickeners. Lithium soaps of the developmental MFA mixtures were used to produce greases with texture similar to that of reference lithium 12-HSA greases formulated with the same base oil. Although a slightly higher thickener percentage may be required in the developmental MFA formulations to obtain the same consistency as the reference greases, both types of greases exhibited similar shear stability as measured by prolonged working of 10,000 and 100,000 strokes in a mechanical grease worker.

The thickener efficiency of developmental MFA greases was nearly identical to that of reference lithium complex formulations (12-HSA and azelaic acid complex lithium soap). Worked penetration measurements of developmental MFA greases were comparable to the reference lithium complex formulations. These MFA-thickened greases were also evaluated for rheological characteristics, roll stability, oil bleed and dropping point, then compared to standard lithium 12-HSA and lithium complex formulations produced inhouse with the same base oil.

Introduction

For the past 70 years, 12-HSA has been the dominant fatty acid used for lithium soap thickeners in lubricating greases, and today, lithium soap greases make up more than 70% of the commercially available greases.[2] Greases produced from 12-HSA tend to have smooth texture and good thixotropic properties. A prior investigation into alternatives to 12-HSA as a thickener found that to a large degree, other stearic acid derivatives were not as well suited for grease use.[3] In most cases, the thickener efficiency of alternative fatty acids was very low compared to that of 12-HSA, requiring a larger concentration of thickener to achieve the same consistency of grease. Many of the other properties of the experimental greases, including shear stability, were inferior to those of lithium greases.

Complexing dicarboxylic acids are used in small portions in lubricating grease formulations to improve high temperature tolerance. Commonly used dicarboxylic acids are typically produced from oleochemicals such as oleic and ricinoleic acids by a small number of sources.[4] Some long-chain dicarboxylic acid derivatives of fatty acids were investigated as co-thickeners in lithium soap lubricating greases.[5] These dicarboxylic acids performed comparably to standard complexing acids, e.g., azelaic acid, in lubricating grease formulations. Even though there has been enough 12-HSA to meet grease production demand, recent shipping disruptions and potential port closures have highlighted the fragility of this supply chain that relies on sources from one region, South Asia.[1] This paper revisits the idea of alternatives to 12-HSA, focusing on fatty acid derivatives as potential thickener precursors. Modified fatty acid 1 (MFA1) and modified fatty acid 2 (MFA2) are used as obtained and blended with a fatty acid for use as a grease thickener. Lithium soaps are the focus of this investigation, because 12-HSA is widely used for lithium soaps, and lithium soaps are the most widely adopted grease technology.

Experimental

Materials:

Base oils and chemical reagents were used as received. 12-HSA acid was purchased from Tokyo Chemical Industry (TCI) America (Portland, OR). Azelaic acid was purchased from Sigma-Aldrich (Burlington, MA). Lithium hydroxide monohydrate was purchased from Oakwood Products, Inc. (Estill, SC). The MFAs were developmental products produced at Ingevity.

Equipment

Soap reactions to form lithium greases were conducted on the bench scale using a standard glass kettle (Hammett Glass, North Augusta, SC) housed in a heating mantle (VWR, Radnor, PA) [**Figure 1**]. The reaction temperature was monitored with a K-type thermocouple attached to a programmable controller (J-KEM Scientific, St. Louis, MO). The reaction was agitated with a tilted disk impeller with alternating angled teeth (IKA Works Inc., Wilmington, NC). A nitrogen blanket delivered via sparging tube was used to avoid flashing of the base oil or other components and facilitate the removal of reaction water through a Dean Stark apparatus.



Figure 1: Processing equipment for grease sample production: (left) kettle-style chemical reactor and peripherals with mechanical overhead stirrer, and (right) 3-roll mill.

The grease samples were milled on a 3-roll mill (Exakt Technologies Inc., Oklahoma City, OK) [**Figure** 1]. Each sample was passed through the mill 3 times with manual mixing between passes to ensure homogeneity. The grease penetration was determined using a digital manual penetrometer (Humboldt Mfg. Co., Elgin, IL) [**Figure 2a**] per ASTM D217. If grease penetration was less than the target value, then the grease was diluted with base oil and remixed in the glass kettle with an anchor shaped impeller. The penetration was remeasured, and the process repeated until the desired penetration range was achieved.

Finished samples were shear tested by two methods. Method 1 employed a mechanical grease worker (Koehler Instrument Co. Inc., Holtsville, NY) [**Figure 2b**] to shear the sample, followed by the standard penetration measurement (ASTM D217). Method 2 used a roll stability cell with a 5 kg internal pin (Linetronic Technologies SA, Mendrisio, Switzerland) [**Figure 2c**] turned in a roller oven (ASTM D1831), followed by penetration measurements using the ¹/₄-scale penetration method (ASTM D1403). Dropping point temperature was measured according to ASTM D566 using a stirred hot bath with thermometer and sample insert (Koehler Instrument Co. Inc.) [**Figure 2d**]. Oil separation was measured according to ASTM D6184 using a mesh cone suspended from a steel plate on a beaker (Koehler Instrument Co. Inc.) [**Figure 2e**]. Rheology and viscoelastic properties were measured according to DIN 51810-2 using a modular compact rheometer (Anton Paar USA Inc., Ashland, VA) with a 50 mm parallel plate geometry [**Figure 3**] and a 0.5 mm gap at 40°C.

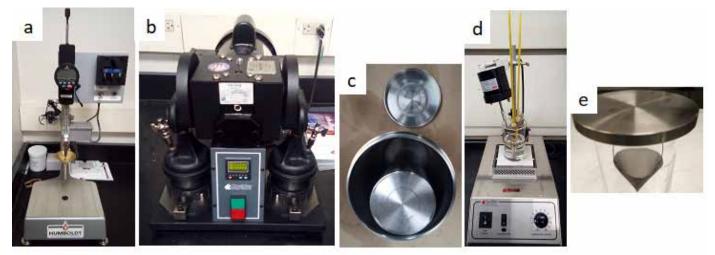


Figure 2: Standard grease testing apparatus: (a) electronic manual penetrometer, (b) mechanical grease worker, (c) roll stability testing cell, (d) dropping point apparatus, and (e) oil separation test apparatus.



Figure 3: Modular compact rheometer (left) and full instrument profile (right) sample stage with a 50 mm parallel plate geometry.

Personal Protective Equipment

Fume hoods were utilized as engineering controls for all work at high temperatures discussed above, including formulation of grease samples in the glass reactor and dropping point tests. The 3-roll mill was equipped with various safety shut-off devices and safety guards for safe usage. Safety glasses, laboratory coats and chemically resistant gloves were always used when handling raw materials and greases. Heat-resistant gloves were used in addition to chemically resistant gloves when handling hot equipment.

Formulation

In this study, greases were formulated from only soaps and base oils, without any performance additives. Percent thickener was calculated from the combined masses of fatty acid and lithium hydroxide monohydrate divided by the total mass of grease components. The initial base oil and fatty acid were charged to the reaction kettle, heated and stirred under a N_2 blanket.[Figure 4] The kettle contents were heated to 72°C until all fatty acid was completely dissolved in the base oil. If the fatty acid would not dissolve in the given base oil, the temperature was increased by 5°C increments until solvation occurred. A slurry of lithium hydroxide monohydrate in water (2x the mass of LiOH:H₂O) was added to the kettle and the temperature profile shown in Figure 4 was followed. Once the sample temperature was below the flashpoint of the base oil, the quench oil was added to further cool the grease.

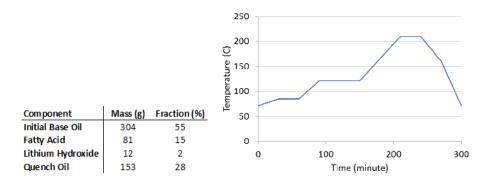


Figure 4: (left) Example formulation of 12-HSA grease, and (right) temperature ramp profile for soap reactions.

Results and Discussion

MFAs were tested against 12-HSA and 12-HSA/azelaic acid mixtures in the same base oil. We used two types of naphthenic base oils (NBO1 and NBO2) and two types of paraffinic base oils (G1BO and G2BO) in the study. The investigation started with the two naphthenic base oils;

- NBO1 was International Standards Organization viscosity grade (ISO VG) 22 and NBO2 was ISO VG 220.
- 2. G1BO and G2BO were both ISO VG 22.

Instead of a specific thickener concentration, we targeted a penetration range (in units of 0.1 mm) for the resultant greases. We intended to produce grease samples with similar physical properties, allowing thickener efficiency to be determined in each base oil.

Performance in Naphthenic Base Oils:

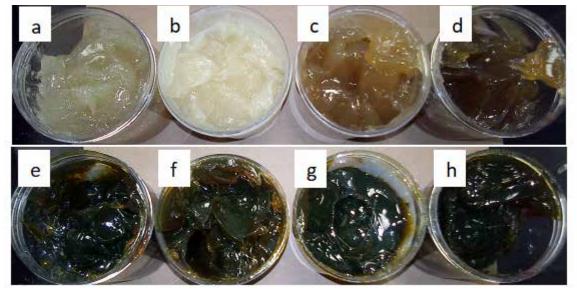


Figure 5: Grease samples using (top) NBO1 with (a) 12-HSA, (b) 12-HSA with azelaic acid, (c) MFA1, and (d) MFA2, and (bottom) NBO2 with (e) 12-HSA, (f) 12-HSA with azelaic acid, (g) MFA1, and (h) MFA2.

The MFA greases were very similar to the 12-HSA greases in appearance and texture, although the MFA greases were darker in color compared to the 12-HSA greases [Figure 5]. MFA1 produced similar grease properties to 12-HSA greases in both base oils at similar thickener concentrations [Table 1], suggesting that thickener efficiency was comparable for MFA1 and 12-HSA formulations in these base oils. MFA2 required considerably more thickener to achieve similar penetration measurements versus 12-HSA in both base oils. Oil bleed for greases prepared using naphthenic base oils was less than 1%. MFA thickened greases had lower dropping points compared to 12-HSA thickened greases.

	NBO1						NBO2					
Thickener	Thickener Conc. (%)	Pentration (0.1 mm)	NLGI Grade	Dropping Point (°C)	Oil Bleed (%)	Thickener Conc. (%)	Pentration (0.1 mm)	NLGI Grade	Dropping Point (°C)	Oil Bleed (%)		
12-HSA	12	253	2.5	196	0.2	7	239	3	198	0.1		
12-HSA + Azelaic Acid	13 (11+2)	251	2.5	224	0.7	8 (7 + 1)	241	3	223	0.7		
MFA1	13	245	3	184	0.2	8	249	2.5	183	0.0		
MFA2	16	246	3	191	0.2	12	284	2	182	0.3		

Table 1: Thickener concentration of grease formulations with unworked penetration and NLGI grade, dropping point and oil bleed measurements in naphthenic base oil NB01 (left) NB01 and NB02 (right). In NB01, the thickener concentration of 12-HSA + azelaic acid was 13% total with 11% from 12-HSA and 2% from azelaic acid.
 In NB02, he thickener concentration of 12-HSA + azelaic acid was 8% total with 7% from 12-HSA and 1% from azelaic acid.

Worked penetration measurements of MFA1-thickened greases in both NBO1 and NBO2 showed slightly more shear softening compared to 12-HSA-based greases in the same base oils [**Table 2**]. The effect was more pronounced with NBO1 where an additional 9% difference was observed for the MFA1-thickened grease compared to the 12-HSA grease. MFA2 behaved differently than MFA1 in the naphthenic base oils, where MFA2-thickened greases were more shear stable under mechanical grease worker conditions compared to their 12-HSA grease counterparts. MFA2-thickened greases had only an 11% difference in penetration between 60 and 100,000 strokes, compared to 25% for 12-HSA greases. MFA2-thickened greases made in NBO1 stiffened under shear, while those formulated in NBO2 softened under shear.

	NBO 1						NBO2				
Thickener	60x	10Kx	% Change	100Kx	% Change	60x	10Kx	% Change	100Kx	% Change	
12-HSA	251	280	12	314	25	240	273	14	304	26	
12-HSA + Azelaic Acid	246	276	12	296	21	243	260	7	291	20	
MFA1	251	304	21	337	34	247	288	16	318	29	
MFA2	257	252	2	230	11	271	295	9	302	11	

Table 2: Worked penetration of grease formulations in naphthenic base oils, (left) NBO1, and (right) NBO2.Samples were worked using a mechanical grease worker, and penetration was measured using an electronicmanual penetrometer (ASTM D217).

The difference in shear stability for greases formulated in naphthenic base oils was more pronounced after roll stability testing (ASTM D1831). Samples were rolled for 16 hours at 25 rpm using a standard roller cell with a 5 kg internal pin. The results of rolling NBO1 samples were analogous to the effects of using the mechanical grease worker [**Table 3**], though the MFA1-thickened greases softened more due to rolling than working. All NBO1 samples were negatively affected to a considerable extent by the addition of 10% water to the rolling cell, but MFA2-thickened greases performed much better than all other samples under this condition with only a 25% difference in penetration before and after rolling. The structure of the grease thickener broke down for greases formulated with MFA1 in both naphthenic base oils and for MFA2 in NBO2, while the 12-HSA thickener performed better in NBO2 then NBO1.

		NBO1		NBO2						
Thickener	Before Rolling	Rolled	% Change	Rolled + 10% Water	% Change	Before Rolling	Rolled	% Change	Rolled + 10% Water	% Change
12-H5A	245	289	18	411	68	239	303	27	297	24
12-HSA + Azelaic Acid	228	285	25	443	95	241	287	19	351	45
MFA1	231	341	47	Breakdown		229	338	48	Breakdown	
MFA2	223	250	12	278	25	258	357	38	Breako	lown

Table 3: Roll stability testing of grease formulations in naphthenic base oils, (left) NBO1, and (right) NBO2.Samples were rolled in a sealed steel cylinder with 5 kg internal steel pin (ASTM D1831), and penetration was
measured using a ¼-scale cone (ASTM D1403).

Performance in Paraffinic Base Oils

Two paraffinic base oils were used to formulate greases, a Group I (G1BO) ISO VG 22 and a Group II (G2BO) ISO VG 22. Only MFA1 and 12-HSA thickeners were tested in both paraffinic oils; a blend of 12-HSA and azelaic acid was also formulated in G1BO. The appearance and texture of these greases were very similar to that of the greases formed in NBO1, and MFA1 greases were very similar to 12-HSA greases in both paraffinic base oils [**Figure 6**], although the MFA1 greases were amber compared to the off-white 12-HSA greases.

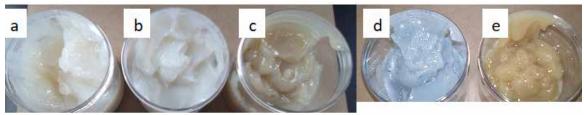


Figure 6: Grease samples formulated using (left) G1BO with (a) 12-HSA, (b) 12-HSA with azelaic acid, and (c) MFA1, and (right) G2BO with (d) 12-HSA, and (e) MFA1.

In G1BO, almost equal concentrations of MFA1 thickener and 12-HSA thickener were used to achieve greases with similar consistencies [**Table 4**] much like the performance of these thickeners in naphthenic base oils. However, in G2BO, MFA1 had considerably lower thickener efficiency than 12-HSA. The dropping point was depressed in the paraffinic base oils versus 12-HSA, with approximately a 20°C lower dropping point observed for MFA1 greases, which was consistent with the results for the greases prepared in naphthenic base oils. Oil bleed was similar for all samples, and values were typically between 1% and 3%, which was slightly higher than the naphthenic grease examples where the typical oil bleed was <1%.

Worked penetration results for greases prepared in paraffinic base oils showed some parallels with the results for the greases formulated in the naphthenic base oils. The greases prepared with MFA1 thickener showed better shear stability at 10,000 and 100,000 double strokes compared to both 12-HSA-thickened greases made in G1BO [**Table 5 left**]. MFA1-thickened grease demonstrated lower shear stability versus 12-HSA-thickened grease in G2BO at 10,000 double strokes, but similar stability at 100,000 double strokes [**Table 5 right**]. The penetration of the MFA1-thickened grease increased by 18% after 10,000 double strokes, whereas the 12-HSA-thickened grease increased by 10% and 22% after 10,000 and 100,000 double strokes, respectively.

Roll stability tests confirmed the results from the work stability tests when compared to the dry rolled samples [**Table 6**]. In G1BO, MFA1 and 12-HSA thickeners performed similarly when comparing percent change in penetration before and after the roll test. Similar changes in penetration were measured for worked and rolled samples of greases prepared with all thickeners in G2BO. Large differences in shear stability were observed for MFA1- versus 12-HSA-based greases when rolled with 10% water. The 12-HSA-based greases softened to grades 00 to 000 when water was introduced, while the MFA1-thickened greases softened (G1BO) or thickened (G2BO) by only 1 NLGI grade on rolling with water.

			G1BO					G2BO		
Thickener	Thickener Conc. (%)	Pentration (0.1 mm)	NLGI Grade	Dropping Point (*C)	Oil Bleed (%)	Thickener Conc. (%)	Pentration (0.1 mm)	NLGI Grade	Dropping Point (*C)	Oil Bleed (%)
12-H5A	12	273	2	201	1.1	12	273	2	205	1.3
12-HSA + Azelaic Acid	12 (10 +2)	255	2.5	220	3.4	-	-	-	-	-
MFA1	13	298	1.5	177	0.7	16	332	1	183	2.2

Table 4: Thickener concentration of grease formulations with unworked penetration and NLGI grade, droppingpoint and oil bleed measurements in paraffinic base oils (left) for G1BO. The thickener concentration of 12-HSA + azelaic acid was 12% total with 10% from 12-HSA and 2% from azelaic acid. (right) for G2BO. 12-HSA andazelaic acid mix was not used in this comparison for G2BO.

	G1BO						G2BO					
Thickener	60x	10Kx	% Change	100Kx	% Change	60x	10Kx	% Change	100Kx	% Change		
12-HSA	239	280	17	320	34	271	299	10	330	22		
12-HSA + Azelaic Acid	252	279	11	296	17	-	-	-	-	-		
MFA1	287	313	9	322	12	327	386	18	408	25		

Table 5: Worked penetration of grease formulations in paraffinic base oils, (left) G1BO, and (right) G2BO. The12-HSA and azelaic acid blend was not used as comparison for G2BO. Samples were worked using a mechanicalgrease worker, and penetration was measured using an electronic penetrometer (ASTM D217).

	G1BO						G2BO				
Inickener	Before Nolling	Rolled	% Change	Rolled + 10% Water	% Change	Before Rolling	Rolled	% Change	Rolled + 10% Water	% Change	
12-HSA	254	293	15	416	64	225	272	21	390	74	
12-H5A + Azelaic Acid	267	299	12	487	82	-		-	-	-	
MFA1	294	340	16	312	6	334	429	28	287	14	

Table 6: Roll stability testing of grease formulations in paraffinic base oils, (left) G1BO, and (right) G2BO. A blend of 12-HSA and azelaic acid was not used in this comparison for G2BO. Samples were rolled in a sealed steel cylinder with 5 kg internal steel pin (ASTM D1831), and penetration was measured using a ¼-scale cone (ASTM D1403).

Rheology Analysis

Rheological properties were measured for the grease samples formed in NBO1 and NBO2 [Figure 7] according to DIN 51810-2 Method A. The method requires a slow heat ramp to the desired test temperature (0.4°C/minute) and a long equilibration time at the test temperature (30 minutes). This procedure is due to the low thermal conductivity of grease, and insufficient temperature equilibration time can result in inconsistent results.

Rheology measures the physical response (glow and deformation) of materials under stress. Greases belong to a special subset of non-Newtonian viscoelastic materials.[6] An example of a rheology curve as measured by the rheometer [**Figure 7 left**] shows the linear viscoelastic (LVE) range where the storage modulus (G') and loss modulus (G'') remain relatively flat (constant) over a range of shear stresses. The G' values were used to compare the consistency of the grease samples.[7] The G' values within each base oil set were all very similar, which agreed well with the penetration measurements for these samples.

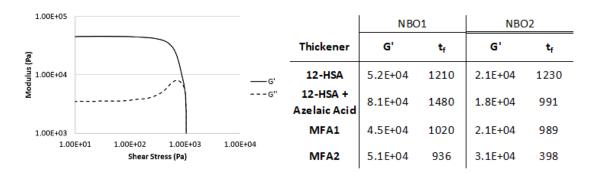


Figure 7: (left) Rheology data for greases thickened with MFA1 and lithium soap in NBO1. The storage modulus (G', ---) and loss modulus (G', - -) are shown on the same graph with both axes on a logarithmic scale. The values of G' and G'' intersect at the flow stress (τf) at which the sample becomes more liquid-like than solid-like and the shear stress where the linear viscoelastic region ends; G' deviates more than 10% at the yield stress (τγ). (right) Rheological values of G' and τf for grease samples in NBO1 and NBO2.

The flow stress (τ_f) of the grease gives a measure of the sample behavior under shearing conditions. τ_f is the shear force required to effectively liquify the sample and is represented in rheology measurements as the point where the storage and loss moduli curves cross. Flow stress was consistently lower for MFA-thickened greases compared to 12-HSA-thickened greases, but not all thickeners had equivalent flow stress in both naphthenic base oils.

One of the most important properties of lubricating grease is its ability to shear thin and return to its solid state after shear is removed, thus keeping the lubricant in place. This behavior can be observed via a rheometer, allowing us to measure and compare the responses of greases. Each grease sample was conditioned in the same manner as DIN 51810-2, with a slow heating ramp followed by a long equilibration at constant. Samples were tested at 40°C using the oscillation mode of the rheometer by adjusting the shear strain to observe specific sample properties [**Figure 8**]. In stage 1, the shear strain was set to a value that was well within the LVE range for the sample (0.1% strain) to measure the baseline modulus. In stage 2, the shear strain was stepped up to a value well above τ_f for the sample (100% strain) in order to break down the solid structure. Then, in stage 3, the shear strain was set back to 0.1% to observe the regeneration of the solid structure of the sample.

Only the results for greases formulated with 12-HSA and MFA1 in NBO1 are is shown [**Figure 8**] for clarity. The 12-HSA- and MFA-based greases exhibited similar solid regeneration behavior. Both storage and loss moduli values were consistent with DIN 51810-2 measurements in stage 1 for all samples. During stage 2, the storage modulus dropped below the loss modulus as expected for grease sheared beyond its flow stress. When the high shear condition was stopped at the beginning of stage 3, the storage modulus increased strongly over the course of a few seconds, while the loss modulus fluctuated and eventually increased. Both moduli increased to below their initial values in stage 1 but continued to increase slowly over time. The initial recovery of the moduli was the same for both sets of samples, but in the MFA-thickened greases, the moduli increased more quickly after the initial recovery than the 12-HSA-thickened greases. All samples fully regained their moduli after several hours.

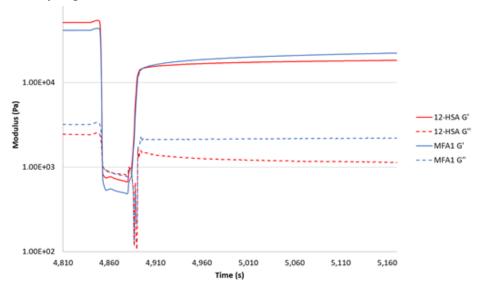


Figure 8: Thixotropic traces of greases formulated with 12-HSA (---) and MFA-1 (---) in NBO1. Low shear strain (0.1%) was used in stage 1 (4,810–4,850 s) to obtain baseline moduli; high shear strain (100%) was used in stage 2 (4,850–4,880 s) to break down the solid structure of the grease; and low shear was used in stage 3 (4,880 s and up) to observe rebuilding of the solid structure of the grease.

Conclusions

Soap thickened grease characteristics are often formulation dependent, and this behavior was observed in this study. Lithium soaps thickened with 12-HSA, 12-HSA complexed with azelaic acid and MFA soaps were prepared in two naphthenic base oils (ISO VG 22 and ISO VG 220) and two paraffinic base oils (Group I ISO VG 22 and Group II ISO VG 22). Greases were produced in a target penetration range (240 – 280 penetration units, 0.1 mm), and then dropping point, oil separation, shear stability and rheology were measured.

- Oil separation was similar for all samples in all base oils, but a small increase was observed for greases formulated with the Group II base oil.
- The dropping points of the MFA-thickened greases were in the range of 180-190°C.
- Results of mechanical working;
 - MFA1 greases shear thinned a little more, and MFA2 greases shear thinned less, than 12-HSA greases in the naphthenic base oils.
 - MFA1 greases shear thinned a little less than 12-HSA greases in the Group I base oil, and but were similar in the Group II base oil.
- Roll stability testing was similar for all the greases in the absence of water.
- Roll stability testing in the presence of water produced severe negative effects on the greases tested with some exceptions:
 - MFA2-thickened greases in NBO1 were more shear stable than 12-HSA-thickened greases.
 - $\circ~$ 12-HSA-thickened greases in NBO2 showed no difference in roll stability in the presence or absence of water.
 - MFA1-thickened greases in both paraffinic base oils demonstrated improved roll stability versus 12-HSA-thickened greases in the presence of water.
 - MFA1-thickened greases in both paraffinic base oils demonstrated improved roll stability in the presence of water versus dry conditions.
- Rheology measurements showed that all tested greases had similar G', but the MFA-thickened greases had lower flow stress values than the other greases.
- Thixotropic measurements revealed that the MFA-thickened greases recovered their structure after shear degradation on the same time scale and magnitude as the 12-HSA-thickened greases.
- The MFA greases had smooth and buttery texture that was similar to the texture typically observed for 12-HSA soap greases.

Although only NLGI grade 2 greases were studied here, a broad range of NLGI grades was achieved by varying the concentrations of the thickeners.

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NLGI Interviews Dr. William Tuszynski Partner, The Unami Group Quakertown, PA andCo-Editor, The NLGI Grease Guide

By Mary Moon and Raj Shah



Dr. William Tuszynski, Principal, The Unami Group (Quakertown, PA) and Editor, The NLGI Grease Guide

Fortunately for NLGI, Bill Tuszynski decided not to go to dental school. Instead, Bill earned his doctorate in physical organic chemistry at Cornell and then decided to become a fox instead of a hedgehog. He rolled up the sleeves of his lab coat and worked at Pennwalt. Atochem. and Inolex. Surfing the waves of M&A, Bill joined Ivanhoe Industries, Inc. a distributor, and assumed responsibility for marketing specialty chemicals to the lubricants industry. This decision led to new career as an entrepreneur and co-owner of Ivanhoe and involvement in NLGI. His contributions to NLGI have ranged from organizing an unbelievably difficult trivia contest of potato-related facts to editing The NLGI Grease Guide (twice!) To learn more about Bill and Mr. Potato, read on!

Education

NLGI: Please tell us a little bit about where you grew up.

BILL: I grew up in Maspeth, a community in the borough of Queens in New York City. It was a blue-collar neighborhood near the Elmhurst Gas Tanks, two natural gas storage tanks that stood alongside the Long Island Expressway. They were well known landmarks, as every AM traffic report in the '60s and 70's started with a description of the build-up of traffic headed to Brooklyn and the Midtown Tunnel starting at the Elmhurst Tanks.

NLGI: How did you develop your interest in engineering and science?

BILL: I was always interested in math and science as far back as my days in elementary school. I'd start my Saturday morning by watching an episode of "Modern Farmer" which aired on the NYC PBS station. Who knows why PBS showed it in New York City?

NLGI: When you were growing up or a student, were you interested in business or becoming an entrepreneur?

BILL: While I would like to say yes, that implies a degree of self-awareness that was not there. Truth be told, I wanted to be a Major League baseball player but learned early on that the only thing holding me back was a lack of athletic talent.

NLGI: Where did you go to college, and what did you study?

BILL: I attended Manhattan College which, oddly enough, is located in the Bronx. I started as a biology major and planned to become a dentist but decided to pursue chemistry after taking organic chemistry. My BS was in biochemistry because I would have had to make up too many courses if I became a chemistry major. Biochemistry has undergone some amazing changes over the 45 years since I graduated. We barely touched on genes in our senior level course, and now so much of biochemistry is driven by genomics such as research with CRISPR gene editing.

NLGI: What was your favorite course or professor?

BILL: My favorite course was organic chemistry with Paul Newman (the professor, not the actor). My favorite professor was Gerry Macri. He left the College while I was a junior to take a job as a company that recovered silver from spent photographic negatives. He hired two of us to work parttime and summers during my junior and senior years, giving me plant experience early on. My thesis advisor, Dr. Charles Wilcox, not only was an excellent scientist but also a great example of managing work-life balance.

NLGI: What inspired you to continue your education? Where did you earn our doctorate, and what was your field of study?

BILL: The job market for BS chemists in 1976 was uninspiring. I went to grad school on a teaching assistantship and earned almost what I would have made working in industry while still living like a student. I would up at Cornell University and



The potato crop in the field

eventually got my PhD in Physical Organic Chemistry studying organic reaction mechanisms.

NLGI: Did you study lubrication or tribology?

BILL: Not at all, save for internal lubrication at the local pub after hours in the lab.

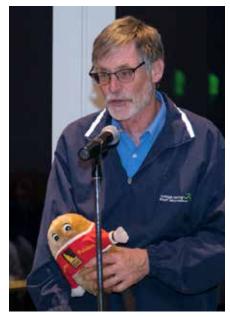
NLGI: How did you become a guru of potato-related facts? What inspired you to co-host a trivia contest at the 2015 NLGI Meeting? Do you remember some of the quiz questions (and answers)? Who won Mr. Potato? What's next?

BILL: We decided to grow potatoes in our home garden in 2002 and unexpectedly wound up with over 100 lbs. We decided to have a potatotasting party to help get rid of some which quickly became our annual "Spudfest" complete with a cheesy quiz and equally cheesy prizes.

It seemed a natural to have a quiz when NLGI went to Potato-Nirvana, aka Idaho, in 2015. Tyler Housel won that year



A portion of the harvest



Bill T and his side-kick, Mr. Potato, were co-MCs of a mind-boggling trivia contest about their favorite vegetable at the 2015 NLGI Meeting Coeur D'Alene

and Martin Keenan was the champion when the meeting and quiz returned to Coeur D'Alene in 2018. Fortunately, I still have the answer keys to both quizzes because I typically forget the answers shortly after making the quiz.

Career

NLGI: How did you begin your career?

BILL: I started at Pennwalt Corp. (now part of Arkema) in an Applications Lab position for the thiochemical and amine businesses. It was a great experience being exposed to diverse applications of these chemicals including metalworking, gas odorants, natural gas processing, coatings, water treatments, refining catalysts, and electroplating among others. The philosopher Isiah Berlin described people as being either hedgehogs or foxes. Hedgehogs know everything about one thing, while foxes know something about many things. I learned early on that I was happier working as a fox than as a hedgehog.

My colleagues and I worked closely with Sales, Marketing, and Technical Service and I became a Group Leader in 1985. Pennwalt was acquired by Elf Aquitaine to form Atochem in 1987, and I joined a New Business Development Group in 1989 where I worked on a project for producing high H2S natural gas. I also played a small role in developing di(t-butyl)trisulfide as an EP additive.

I then joined Inolex in 1995 and worked on lubricant esters and polyester polyols for polyurethanes. During my time there until 2002, I worked closely with Tyler Housel, including carpooling, and now I work with him as an agent for Zschimmer & Schwarz.

I was "rightsized" by Inolex in 2002 and started a consulting practice while looking for a new position. During that time, I started working with Ivanhoe Industries, Inc. (Tullytown, PA) and, together with two former Atochem colleagues, Jeff Snyder and Rafael Valle, bought the Company from John Hoegl, the founder, in 2005.

NLGI: How did you become involved in the grease industry?

BILL: When we purchased Ivanhoe, we divided up responsibilities for various markets. I took responsibility for lubricants, as I was the only one of the three partners who had experience with lubricants. although I had none in grease at the time. Ivanhoe was and still is a significant player in sebacic acid for LiX greases and in disodium sebacate for H1 grease, and we had a number of grease customers. That was my first exposure to greases. I attended my first NLGI Annual Meeting was in 2005, and I've been to all of them since then.

NLGI: What inspired you to become an entrepreneur?

BILL: The flippant answer is that I needed a job. In reality, I don't think of the three of us as true entrepreneurs. With Ivanhoe, we purchased an ongoing company with healthy sales and profits. We never had to go through the start-up and pre-revenue stages. Those who manage those phases are the true entrepreneurs.

NLGI: Please tell us a little bit about Ivanhoe. When and how did you become a partner and what were your responsibilities?

BILL: As I said, I was doing project work for Ivanhoe starting in late 2002 and,

together with my future partners, approached the owner in 2003 to express our interest in buying the company if he was interested in selling. We negotiated for the better part of two years before closing the deal in June 2005. In addition to dividing market responsibilities, we divided operating responsibilities and I became CFO and handled regulatory affairs. That being said, the three of us obviously consulted each other and came to an agreement before making any changes in the business.

NLGI: What were some milestones in the growth of Ivanhoe?

BILL: Our biggest accomplishment was tripling sales in 10 years. Granted that this is easier to accomplish for a small company than for a multi-national. but is something in which we took great pride. We added a number of products such as benzoic acid and several preformed thickeners for greases, and we became the Northeast distributor for Functional Products in 2010. Our biggest growth area was in personal care products where we arranged toll manufacturing and became the only domestic supplier of a non-paraben preservative. We also became a significant supplier of raw materials for polyurea foam.

I'm also proud to say that all but one of Ivanhoe's employees in 2005 are still working there today.

NLGI: What are some of the pros and cons of being an entrepreneur or owning a business?

BILL: If you own a small business, there is a level of risk that you don't have as a company employee. On the other hand, you have control of the decisions that affect your future. If you have partners, better that there is an odd number to avoid deadlocked decisions.

You also have to look forward, especially if you have partners. One of the smartest things we did when we bought the company was make a "prenuptial" agreement that outlined how shares would be valued and paid out if one of us resigned, died, or became disabled. This allowed my departure in 2016 to be smooth and without conflict.

NLGI: Do you have any words of wisdom for readers who might be thinking about becoming an entrepreneur?

BILL: Be prepared and consider all possible scenarios, both good and bad. This will help you avoid surprises. There will be ups and downs, and you have to maintain an even keel. Understand your value proposition but don't fall in love with it to the point that you grossly overestimate yourself and underestimate your competitors. Be prudent financially and do fall in love with cash flow.

Whatever the size of your company, treat your customers, employees, and suppliers fairly and with respect. Not only is it the right thing to do, but others have the same products that you do and being decent to people goes a long way. Be honest. They should always hear bad news from you first.

Finally, be diligent about paying your suppliers on time. Nothing sours a relationship, particularly a distributorprincipal relationship, faster than unpaid invoices.

NLGI: Do you have any advice for readers who might be interested in working in Sales?

BILL: Be yourself. Some customers will like you, and others will not. You can't be a chameleon trying to please everybody, and that behavior will be readily perceived as fake. Don't be afraid to give bad news. Anyone working in sales today has gotten plenty of practice advising customers about supply shortages. Customers should always hear updates from you, not the rumor mill or a competitor.

NLGI: When and how did you decide to retire from Ivanhoe?

BILL: Simply, it was the time of life where both Becky, my wife, and I were looking to back away from some things and spend more time together. We started to talk about it in 2014 and we spent some time making sure our finances were in order. I advised my partners in early 2015 and separated from the company on 12/31/2015.

NLGI: Please tell us a little about The Unami Group.

BILL: Becky and I are partners in The Unami Group. It includes my sales/consulting activity and her technical writing. My original preference was to be The White Eagle Group, the white eagle being the Polish national emblem, but the name was taken by a business consultancy located 20 minutes from my house. The Unami were our local Native American tribe in the Lenape Confederation, and the Unami Creek is our main local waterway.

NLGI: What are your current work activities with The Unami Group?

BILL: I work out of a home office which cuts wear and tear on both me and my car although cats and dogs interrupting Zoom calls are a constant threat. I am a commissioned sales agent for Ivanhoe, Functional Products, and Zschimmer & Schwarz, handling accounts for each. I have a few customers who buy from more than one of my principals, which is nice. In addition to the sales part of my job, I do consulting projects for clients. I've done business development/product introduction projects, market research, and competitive assessments, as well as supporting due diligence activities for M&A work.

NLGI: What were some pivotal or learning experiences at Ivanhoe?

BILL: Being a distributor is significantly different than being a manufacturing company. You spend as much time "selling" to your principals as you do to customers as the relationship with them is of equal importance. We had some nervous moments with principals being acquired or changing their philosophy on distribution but survived all of them. Our product portfolio changed significantly during my time at Ivanhoe. Identifying complementary products and new principals is an ongoing process for any distributor.

NLGI: Do you have a sales or management philosophy?

BILL: Again, be yourself and be honest. Always be willing to say "I don't know" and "I was wrong about that." And shut up and listen to what the other person is saying.

NLGI: Please describe a typical day or week working at The Unami Group.

BILL: (Home) office days are the same as for everyone else. Keep the cats off the keyboard and don't open the refrigerator too often. I do try to fit in either a cycling workout or a session with weights when I can and have been known to spend summer lunches working in our vegetable garden. When I'm on the road visiting customers or at a conference, the work flow is no different than at any of the jobs I've had except that my boss is always with me.



Bill regularly attends NLGI Annual Meetings

Grease Industry

NLGI: What are your thoughts about the lubricating grease industry? What do you think about its future?

BILL: Greases are subject to the same trends as the entire lubricant industry. The drive

for higher performance and longer life means that tonnage will stay flat or decline, but unit pricing should go up. I think we'll see more specialized products in the mix taking volume away from commodity products. Sustainability and decarbonization are macro trends that will have strong future influences on greases.

NLGI: Are there new or future applications of greases?

BILL: Everyone wants a bigger piece of the EV pie, and all that needs to be sorted out. Manufacturing will continue to become more automated, and greases will be the lubricant of choice for robotics in some applications.

NLGI: Are there new chemistries for greases?

BILL: I attended a recent ACS webinar where the presenter pointed out that one consequence of EVs is that gasoline demand will plummet, leading to refinery closures. Besides having less (lower quantities of) basestock available, there will less production of the refinery streams that the petrochemicals industry depends on for synthetic basestocks and to manufacture additives. I think the landscape of chemistries available to formulators will be markedly different in the next 10-20 years. Although more problematical for greases,



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recycling and re-refining will grow in importance.

NLGI: Are there new tests and specifications for greases?

BILL: NLGI'S HPM specs and the need for a new ASTM long-life test are obvious answers.

NLGI: Do you have a favorite grease chemistry or grease test?

BILL: Any grease chemistry for which I get a customer order is my favorite of the day!



Scott Crawford (Primrose Oil), Bill, and Juan-Louis Bessette (Triboscience)) enjoying the Welcome Reception at the 2019 NLGI Annual Meeting (Las Vegas)

NLGI

NLGI: How did you become involved in NLGI?

BILL: With Ivanhoe having some activity in greases, I went to my first NLGI Annual Meeting in 2005. NLGI is a very welcoming organization, and I felt at home right away. I think it is important to be active in any organization you join. Experience has shown that you get back ten-fold what you put in.

NLGI: How have you benefitted by attending NLGI meetings?

BILL: The obvious benefits are networking and learning. NLGI meetings are a great forum for meeting who's who in the industry and the presentations and exhibits are valuable for learning both industry basics and new trends in the industry. There are any number of people in the industry I consider friends.

NLGI: Why is NLGI important? How does NLGI add value to the lubricating grease industry?

BILL: Along with the benefits of the meetings, NLGI provides value through education and professional development through the Grease Courses and the CLGS certification. The work that NLGI and ELGI did on addressing REACh registration for common thickeners was a real service along with the new HPM specifications.



Brooke Martin and Bill cycled to the 2019 Rotary District 7430 Conference in Reading PA

NLGI Grease Guide

NLGI: How did you become involved with editing the new edition of The NLGI Grease Guide?

BILL: I had been editor of the previous edition published in 2015, so it was natural to come back to the project. This time, Raj Shah and I are working together as co-editors. Raj has brought some exciting new ideas that we are incorporating into the new edition.

NLGI: Why is NLGI revising The Grease Guide?

BILL: The current edition has a discussion of the GC-LB specifications, and that part needed to be updated in light of the new HPM specs. It was decided to pursue a full rewrite of the book rather than preparing an addendum to the current volume.

NLGI: What is special or improved about the new edition of The Grease Guide?

BILL: The scope of the book has been greatly expanded. There are new chapters covering Formulation Strategy and Environmentally Acceptable Lubricants. Two topics, H1 greases and NLGI Certifications, which were sections of or add-ons to existing chapters, are now stand-alone chapters raising the total number of chapters from eight to twelve. In



Leading in times of uncertainty

Conference Theme: "Disruption & Transformation in the Fuels & Lubes Industry"



addition to bound copies, NLGI will be selling both the full volume and individual chapters online.

NLGI: When will NLGI publish the new edition of The Grease Guide?

BILL: Our goal is to have it ready for the 2022 Annual Meeting.

NLGI: What are your responsibilities as Editor of The Grease Guide?

BILL: Raj and I are charged with putting together a quality volume that addresses the important aspects of the industry and keeping to our deadline. The Grease Guide serves a dual purpose. It is both the course text book for the Basic Grease Course and it serves as a valuable reference for more experienced practitioners; and addresses the needs of both audiences.

NLGI: What are some of your activities as Editor?

BILL: After Raj and I worked out the chapter structure and order, we had to find authors. Fortunately, we had a deep pool of talented volunteers to draw from. We also were cognizant of the desire to involve members, especially younger members, who have attended annual meetings but have not been asked to volunteer.

We worked with the authors on expectations for content

and timing and gently noodged them to keep to their deadlines. Once we had draft chapters, we needed to find qualified reviewers to make comments and suggestions that were then turned over to the authors for consideration. Upon receiving the revised final drafts from the authors, the final step was to make sure that the formatting was consistent from chapter to chapter and then submit them to NLGI for publishing.

NLGI: What are some pros and cons of working as an editor?

BILL: The best part was working with Raj and our amazingly talented authors and reviewers. I learned tons about grease this time and last time although not enough to pass the CLGS exam. It is obviously a significant time commitment, and there is some drudge work in harmonizing the formatting. Fortunately, our authors by and large respected the guidance we gave them at the start.



Stefanie Clark, Habitat Bucks Family Program Director, receiving an award from Bill for participating in all 20 Habitat 5K races.

Perspectives

NLGI: Do you still volunteer for Habitat for Humanity? Please tell us a little about Habitat for Humanity and your volunteerism.

BILL: I joined Rotary in 2000 and became involved with Habitat when our local affiliate began a 73-townhome development about a mile from my house. Our Rotary club did monthly on-site work days during the early part of the construction. I then got involved with the Family Selection Committee on which I still serve. We vet and select families to buy Habitat homes. There is a misconception that Habitat gives away homes when the truth is that we sell them at an affordable price to working families.

As Rotary, we sponsor an annual 5K race as a fundraiser for Habitat on Thanksgiving weekend. We just held our 20th race. I promised to be race director as long as the tux still fits. We reward our runners with post-race pizza and wings.

I'm also on the Board our school district's Education Foundation leading our STEM initiatives.

Professionally, I am on the Scholarship Committee of our local STLE Section and am currently Treasurer. NLGI: Do you still volunteer for the Science History Institute (formerly the Chemical Heritage Foundation) and the Joseph Priestley Society? Please tell us a little about these organizations, their activities, and your role.

BILL: The Joseph Priestley Society (JPS) is a volunteer-led program at the Science History Institute. For those who aren't familiar, Priestley was a late 18th century English scientist who invented carbonation and co-discovered oxygen with Lavoisier, although Priestley called it "dephogistenated air". He was forced to flee England because of his radical views and wound up living in eastern Pennsylvania for the later years of his life.

At the JPS, we put on seven programs per year covering the intersection of innovation and entrepreneurship. There are nine of us on the Program Committee, which I chair. We all have different backgrounds in chemicals, materials, and life sciences (pharma and ag), which allows us to organize programs touching on a wide variety of topics. We've covered everything from gene therapy to alternative energy to indoor farming and plant-based meats and cheese.

We did a program on GF-6 some years ago and plan on covering EALs and EVs in the fall of 2022. We did all virtual programming for 2020-21 and 2021-22 seasons and will do hybrid programs when we can return to in-person meetings.

All programs are recorded for the Institute's You Tube channel, and a number of our programs have gotten thousands of views.



Claire Lutz (Croda), Prof. Michael Azarian (University of Maryland), Deepak Bondra (University of Maryland, 2021 Philadelphia STLE Scholarship awardee), and Bill

NLGI: Where is your favorite place to travel?

BILL: Calgary and Banff Alberta. Back when we were working in the sour gas industry in Alberta, our customers were based in Calgary. Whenever we visited them, we always made time for a side trip to Banff. Becky and I took a vacation in Calgary after the STLE Meeting that was held in Calgary. The scenery in the Canadian Rockies is spectacular, and it's a unique experience to soak outdoors in the hot sulfur spring when the air temperature is -30 °C!

Becky and I also do an annual pilgrimage to Knoebel's Grove amusement park in Elysburg PA, two hours west of us in Quakertown. Our daughter, her husband, and our grandsons come down from Massachusetts for a couple of days at the park. I also go into Queens to see my accounts on Long Island and go fishing for fluke and striped bass with my brother.

NLGI: If NLGI members travel to Quakertown or Bucks County, PA, do you recommend special things to do and places to visit?

BILL: We have a lot to offer in Bucks County from artsy communities like New Hope along the Delaware River to the Mitchner and Mercer Museums in Doylestown and interesting live music in Sellersville. In Bucks County, you're no more than an hour away from both Philadelphia and the Lehigh Valley, which have a lot to offer. New York is only two hours away. If you like quirky experiences, we have the Quakertown Farmer's Market aka Q-Mart, which has all the local butchers, delis, grocers, and bakeries plus some unique vendors and an outdoor flea market. Our son-in-law and grandsons can get lost in there for hours.

NLGI: Please recommend some of your favorite books or technical journals to NLGI members.

BILL: I recently finished "The Smell Detectives" about the odor landscape in 19th century NYC. My favorite line from the book was a reporter's comments after taking a boat trip down the heavily polluted Gowanus Canal in Brooklyn. He stated that "The stench was so bad that no amount of profanity could alleviate it."

This interview series, started in 2019 by Dr. Moon and Dr. Shah, gives NLGI members a bit of insight into the professional and personal lives of their colleagues, developments in the grease industry, and the role of NLGI worldwide. If you would like to suggest the name of a colleague for an interview (or volunteer to be considered as a candidate), please kindly email Mary at <u>mmmoon@</u> <u>ix.netcom.com</u> or Raj at <u>rshah@</u> <u>koehlerinstrument.com</u>.

Dr. Mary Moon is Technical Editor of *The NLGI Spokesman*. She consults, edits, and writes scientific and marketing features published in Lubes'n'Greases and Tribology & Lubrication Technology magazines, book chapters, specifications, and other technical literature specific to lubrication and condition monitoring. Her R&D and project management



Becky and Bill (right) enjoyed a Viking River Cruise in Germany (2016).

experience in the lubricant, polymer, and specialty chemicals industries includes inventions, formulation, product development, marketing, and applications of tribology, electrochemistry, rheology and spectroscopy. She served as Section Chair of the Philadelphia Section of STLE. She is a member of the National Association of Science Writers.

Dr. Raj Shah is currently a Director at Koehler Instrument Company, Long Island, NY where he has lived for the last 25 years. An active NLGI member and he served on the NLGI board of directors from 2000 to 2017. A Ph.D in Chemical Engineering from Penn State University and a Fellow from the Chartered Management Institute, London, Dr. Shah is a recipient of the Bellanti Sr. memorial award from NLGI. He is an elected fellow by his peers at NLGI, IChemE, STLE, INSTMC, AIC, MKI, Energy Institute and the Royal Society of Chemistry. He has over 300 publications and is currently an Adjunct Professor at the Dept. of Material Science and Chemical engineering, State University of New York, Stony Brook. Currently active on the board of directors of STLE he volunteers on the advisory boards of several universities. More information on Raj can be found at

https://www.nlgi.org/nlgiveteran-member-raj-shahpresented-with-numeroushonors-in-2020/





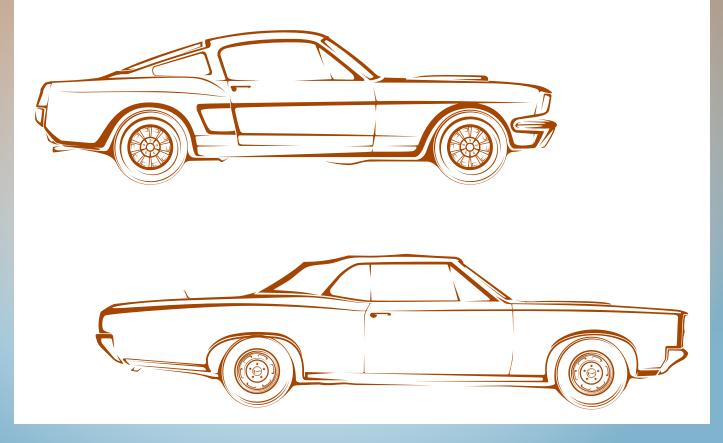
The NLGI SPOKESMAN is pleased to announce the launch of a new section within its publication titled "VALUE -ADD." The theme of this new section is to highlight changes, advancements, best practices in lubrication and maintenance, as well as challenges in the grease industry as they relate to customer centricity, general grease issues, suppliers, supply chain, education and other non-traditional technical related topics that are current to the grease industry. NLGI leadership is excited to provide additional value to *The NLGI Spokesman* readers and welcome future articles that bring insight into our industry.

Contact <u>nlgi@nlgi.org</u> for more information on how to submit.

Electric Motors Retrospective Part 2

The Electric Motors Retrospective continues with a technical paper published in November, 1965 by H. Eldridge titled "Lubrication Requirements of 1966 Cars." This paper focuses on passenger car models, changes in lubricants and the way cars are serviced.

The production and use of electric vehicles has increased dramatically over the last decade and there has been much interest in the lubrication industry with regard to the impact on performance requirements of lubricating oils and greases. Certainly, the use of lubricating greases for electric motors will be an area of great interest as this technology continues to evolve and grow. While the future performance needs of lubricating greases in these new generations of electric vehicles are still not yet fully understood, it is interesting to look back at work done in this area to understand how much has changed and how much remains the same.



- 50 -NLGI Spokesman | VOLUME 85, NUMBER 5 | November/December 2021

Lubrication Requirements of 1966 Cars

By: H. Eldridge, The Chek-Chart Corporation

Presented at the NLGI 33rd annual meeting in San Francisco, October, 1965

A S THE 1966 passenger car models are introduced, we find the usual interesting changes in lubricants . . . in intervals . . . and in the way cars are to be serviced.

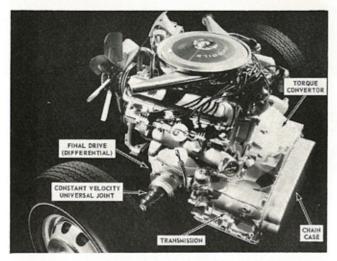
We'll turn our attention to all of these matters after this general look at the 1966 line-up for each auto maker.

Engineering News

We observe initially the almost-total disuse of the word "compact" in the tons of descriptive material attending 1966 introductions. Though the cars that once were hailed as compacts may not have grown more than a couple of inches in wheelbase over the past few years, it is now patently true that the big word in today's market is Performance-Not Economy.

There have been three very interesting engineering developments at the two largest auto makers: (1) Oldsmobile's front-wheel drive design for its new Toronado model; (2) Pontiac's overhead-cam 6-cylinder engine that's now standard in the 1966 Tempest, and (3) Ford's new *Bronco* vehicle, a passenger car with many light-truck uses.

About *Toronado*: Front-wheel drive, of course, hasn't appeared on a U.S. production model since the Cord of the 1930's. For its venture into a power-train design that has been the almost-exclusive domain of racing-car manufacturers, Oldsmobile is using a 425-cu. in. V-8 engine mounted in normal longitudinal fashion, but offset slightly to right of center.





At the left side of the engine is a standard automatic transmission unit which is driven by chain from an engine-mounted torque converter. Figure 1 shows, among other elements, the Turbo Hydra-Matic transmission fill pipe and dipstick. Note that it is just below and very close to the crankcase dipstick—a location that may create unfortunate problems if stationmen are not given adequate notice of this situation.

Automatic transmission fluid introduced through the transmission fill pipe serves the torque converter, the chain case and, of course, the transmission itself. Type A, Suffix A fluid is recommended for the Toronado, and transmission draining is specified at 24,000-mile or two-year intervals under normal service conditions and at 12,000 miles under severe service conditions.

To drain the Toronado's transmission, which has a 5-qt. refill capacity, the oil pan must be removed. Before it's replaced, the oil strainer should be cleaned and a new oil-pan gasket must be installed. The torque converter, incidentally, is not to be drained. It would be dismantled in the course of any necessary transmission overhaul, following which 10½ qts. of fluid are required to refill the unit.

The Toronado's differential (a small part of which can be seen in Figure 1) is mounted ahead of the transmission. It's unique in that the differential gear is of planetary design—rather than being a conventional bevel-gear type. This differential is *not* to be drained. There is, however, a fill and level-check plug at the left front of the stamped cover on the front of the unit. Lubricant should be maintained to the level of the plug hole.

Oldsmobile recommends as differential lubricant a part-numbered product (1050015), for field addition and for post-overhaul refill, that has a bit more "slipperiness" than is normal for a limited-slip differential lubricant. As an alternate to this part-numbered product, Oldsmobile permits the use of multi-purpose lubricants designed for use in limited-slip differentials.

The 345-hp Toronado engine has a crankcase capacity of 5 qts. On the steering linkage, there are six lubrication fittings—only one less than on other largeseries Oldsmobiles.

The two front-axle drive shafts are open in design, and there are two constant-velocity universal joints in each drive-axle section. These U-joints are packed with lubricant at assembly and require no further service.

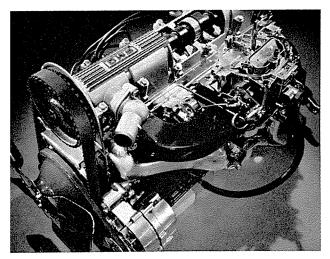
Wheel bearings on the front axle also are sealed and

in't be serviced. If they are defective, or in need of ibrication, they'll have to be replaced. (And for such placement, we might add, special tools will be needl.) The Toronado's rear-wheel bearings *will* require packing only if wheels and brake drums are removed or brake service.

Before leaving Oldsmobile, we should mention that hey have dropped their V-6 engine for this year's todels in favor of a straight-6.

eoprene Drive Belt

Pontiac's decision to go with an overhead-cam enine in their 1966 Tempest line makes them only the cond U.S. passenger-car manufacturer to employ this



GURE 2.

ngine design (Figure 2). The first, of course, was eep' with its Tornado-OHC 6-cylinder engine. This 'as introduced in 1962 and is now being phased-out 1 favor of a conventionally-designed engine.

This new Tempest engine has many features, not re least of which is a separate accessory drive for the istributor, the fuel pump and the oil pump. The most nusual feature, however, would have to be the beltriven cam shaft, because the belt is made of neoprene, einforced with fiber glass cords. Cogs are molded into re belt to help insure proper timing and synchronizaon of the cam shaft. Timing is also greatly aided by n essential quality of the belt material: it won't stretch. The neoprene drive belt can be properly tensioned by posening six bolts and sliding the accessory drive housig up or down (Figure 3).

The overhead-cam engine does not make use of the ydraulic valve lifter generally used on conventional ngines. Instead, Pontiac has incorporated the autonatic hydraulic lash adjuster. This unit provides for ero lash at the valve stem by acting on the rocker arm.

Principal advantage of an overhead-cam engine is he elimination of some valve-train parts-notably the

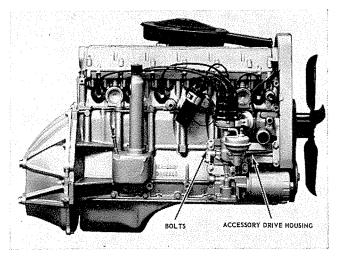


FIGURE 3.

push-rods—and the subsequent reduction in the inertia problem they created. Thanks to the more precise valve action which results, there is far less overlap in valve timing—and this, in turn, leads to improved performance at both low and high rpm's.

Ford Enters Growing Field

Ford Division, fond parent of the Mustang, announced another off-spring a few months ago--this one cast in a different mold. The *Bronco* for 1966 is a passenger car with four-wheel drive to give it lighttruck capabilities. Thus, Ford invades an increasinglyprofitable field pioneered by 'Jeep' and the Land Rover, and enlarged most recently by International's Scout.

A Bronco feature that's particularly worthy of note is the fact that, unlike other Ford passenger cars over the past five years, this vehicle has four lubrication

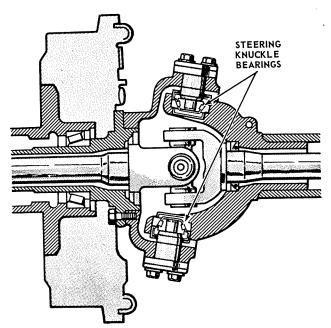


FIGURE 4.

fittings on the steering linkage, plus one each on the universal-joint splines. A Lithium/1% Moly lubricant is to be applied to these fittings at Ford-recommended intervals of 6,000 miles or six months.

Another noteworthy Bronco feature is the design of front-axle universal joints and the steering knuckle. A conventional, cross-type U-joint is used on each front wheel, as we can see in Figure 4. This represents a departure from the enclosed-type U-joints that have been standard in the past on front-wheel drive units. The universal joint is packed at assembly and is an open type, as illustrated in this figure.

The steering knuckle bearings are prepacked with lubricant, and no additional service is required. The double Cardan, or constant-velocity, joints incorporated at the transfer-case end of the drive shafts are prepacked, but they are to be repacked with Lithium/1% Moly lubricant at the 36,000-mile or 36-month interval recommended by Ford.

More New Names

Other new names among the 1966 models this fall are just that: they are *not* new cars. We're speaking of the *VIP* at Plymouth, a top-of-the-line Fury . . . the *DPL* at American Motors, now the top Ambassador model . . . the Chevrolet *Caprice*, introduced during 1965-model production as an extra-plush Impala sports sedan . . . and over at Ford, the 7 *Litre*, a very sporty addition to the Galaxie 500 line. (This latter car will have the Thunderbird 428-cu. in. V-8 with 4-barrel carburetion as its standard power plant.)

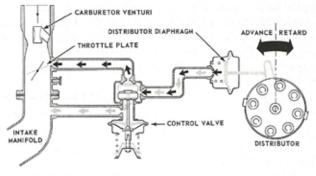
Exhaust Control

One more interesting item, before we turn our attention to lubricants, is a development affecting all car makers—and ultimately, all servicemen. We're talking about the exhaust control devices that must be installed in all 1966 cars and light trucks to be sold here in the state of California.

Largely because of the Los Angeles smog problem, California was instrumental in promoting the use of now-common Positive Crankcase Ventilation—or PCV —systems just a few years ago. But even with PCV, it was apparent that more control of unburned hydrocarbons and carbon monoxide from engine exhausts was needed.

That greater degree of control has been promised by American Motors, with its *Air-Guard* system . . . by Chrysler, with the *Cleaner Air Package* . . . by Ford, with its *Thermactor* system . . . and by General Motors, with the *Air Injection Reactor*.

Actually, among the four systems used by the major car makers, there are but two basic types of operation-one represented by Chrysler's CAP (the *Cleaner Air Package*), and the other by air-injection systems used on AMC, Ford and GM cars.





Chrysler's CAP system uses continuous control of both carburetion and ignition to burn fuel more completely at deceleration—the period in which most noxious gases are emitted in the exhaust. Figure 5 shows a simple representation of Chrysler's newly-designed distributor at right, and the throat of its modified carburetor at left. Two vacuum hoses are connected to the carburetor throat—one above the throttle plate, and one below it. These hoses are joined at their other end to what Chrysler terms a "vacuum advance control valve." A single hose then connects this valve to the distributor diaphragm chamber, which is fitted with linkage to adjust distributor setting.

Essentially, the CAP system provides the same *advanced* distributor setting at deceleration that exists during acceleration, and it does it in this way:

1. At idle, the throttle plate is in almost-closed position. There is very weak carburetor and manifold vacuum-not enough to either open the control valve diaphragm against its spring, nor to activate the distributor diaphragm. Thus, the control valve stays closed, and the distributor stays in retarded position.

2. During acceleration, the throttle plate is in the open position shown in black, and carburetor vacuum is strong enough to pull the distributor diaphragm against its spring, advancing spark timing. However, manifold vacuum is still not powerful enough to unseat the control valve diaphragm.

3. At deceleration, when manifold vacuum is at its peak, the CAP system puts it to work. Exerting enough force (as illustrated by the light gray arrows) to overcome spring tension at the control valve diaphragm, manifold vacuum succeeds in opening this valve. Then, the vacuum can exert enough force to keep the distributor in its advanced position.

The carburetor modifications we mentioned as being part of the CAP system serve to produce a leaner airfuel mixture, and as the engine warms up, to open the automatic choke more quickly.

Furthering Combustion

The other basic type of exhaust control device—as its various tradenames imply—injects fresh air right at the exhaust valves to keep the air-fuel mixture burning

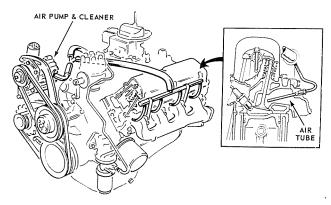


FIGURE 6.

after it leaves combustion chambers (Figure 6). Cars having this system type are fitted with a belt-driven air compressor to pump air through a network of tubes right up to the exhaust valves. The air then maintains combustion in that portion of the air-fuel mixture that didn't get a chance to burn in the combustion chamber.

While the air-injection systems are similar in principle, each must be engineered for the particular car model it serves. Most of them also contain an antibackfire valve, a check valve and a relief valve—all three of which serve to protect the engine.

System Service

Cars fitted with exhaust control devices and sold in California should emit only one-half the amount of undesirable exhaust fumes that would normally be produced by a new car without such device. To insure that this will be the case during the car's service life, the car manufacturers have generally agreed on 12,000 miles and/or 12-month intervals for system service. Here are the elements comprising this service:

For Chrysler's CAP: Engine tune-up, using specifications that differ from those for non-system equipped cars, and including special checks of engine idle speed, ignition timing, carburetor air-fuel ratio with an exhaust gas analyzer, and the distributor vacuum advance control valve.

For all air-injection systems: Inspection of the air pump and driving belt. No service is required on either part unless it's worn or defective, in which case it must be replaced. Regular air cleaner inspection and/or replacement also is recommended. Some cars employ a separate air cleaner for the unit; others utilize the carburetor air cleaner. (And we can't help but observe that increased air use by the latter systems is certainly going to hasten the contamination of air cleaners.) Also recommended for cars with air-injection systems is periodic engine tune-up (again, with extra-ordinary specifications). Tune-up, however, actually is advised no more frequently than for non-system equipped cars.

It's too early to know just what effect these exhaust

control systems will have on the over-all car-service picture in California. However, it's never too early to prognosticate, and one thing is sure: Servicing personnel will have to be properly trained, and equipment for testing cars with exhaust control devices will have to be more sophisticated than is usually the case. For instance, in checking the operation of Chrysler's CAP system, it is necessary to use an exhaust gas analyzer to make sure that the carburetor is properly adjusted.

Exhaust control devices are going to have some slight effect, too, on the service outlook of stationmen elsewhere in the country as they begin to encounter touring Californians in system-equipped cars.

These other stationmen, in fact, may one day soon be servicing cars with exhaust control devices sold right in their own locality. Other states are studying California's progress intently, and it seems safe to assume that several of them may enact exhaust-control legislation if these systems prove successful in California.

Then, too, there is the possibility that car makers will want to keep the advantage by continuing development of these systems and, eventually, by installing them in all cars as a standard engine accessory or component.

Lubrication Status: 1966

And now to the lubricant picture for the 1966 models, which we'll tackle in order of products, then intervals, and finally methods of application.

As Figure 7 shows, the number of chassis lubricants recommended for 1966 car models by the various manufacturers totals *nine*. This is the same *number* as for the 1965 models, but there are changes in the list and we'll call them out for you now.

Lincoln-Mercury has reinstated No. 5 Calcium Cup Grease for lubrication of the steering linkage. For its 1965 models, they had recommended the Lithium-with Moly lubricant for these points. With its current recommendation, Lincoln-Mercury is simply calling for field use of the same type of lubricant employed at the factory during assembly of steering linkage components.

Oldsmobile now states that steering linkage should be lubricated with a "commercially available Multi-Purpose chassis lubricant."

At Buick and Chevrolet, though their listed lubricants have rather lengthy names, they are essentially conveying the same information we've just cited for Oldsmobile.

Now, briefly, here are what the other car makers are recommending:

Chrysler continues with a Barium EP type;

Ford is sticking with a Lithium/1% Moly lubricant; Both Cadillac and Pontiac recommend Barium grease with 10% Moly;

1966 CHASSIS LUBRICANTS

Туре	Part or Spec. No.	Make	Application
No. 2 Barium– 10% Moly	1474829 1050411	Cadillac Pontiac	All All
No. 2 Barium EP	2525035	Chrysler	All
No. 2 Lithium –1% Moly	ESA-M1C75A ESA-M-1C47-	Ford	All
-	A or ESA-M1C75A	Lincoln- Mercury	Ball Joints
No. 2 Lithium	8991416	'Jeep' Universal Rambler	All All (ex. upper trunnion)
	MS-939	Studebaker	All
No. 5 Calcium Cup	934571	'Jeep' Wagoneer	Steering Linkage
	ESA-M1C48-A	Lincoln- Mercury	Steering Linkage
No. 2 Sodium	8991229	Rambler	Classic, Marlin, Ambassador upper trunnion
Long Effective- ness Lubricant	• 9985024	Buick	All
Multi-Purpose Grease	_	Oldsmobile	All
Water Resist- ant E.P. Chas- sis Lubricant		Chevrolet	All

FIGURE 7.

'Jeep' remains unchanged, specifying No. 5 Calcium Cup Grease for Wagoneer steering linkage, and recommending No. 2 Lithium Grease for king pins and all chassis points of the Universal model;

Rambler and Studebaker both specify a No. 2 Lithium Grease for chassis lubrication. In addition, a No. 2 Sodium Soap Grease is called for in the upper trunnion on Ambassador, Classic and Marlin models. This latter recommendation is due to a continuation of 1965 construction, in which the Sodium Grease is supposed to leach out gradually and lubricate the trunnion bearings.

Looking at the intervals for chassis lubrication of 1966 models (Figure 8), we find that Cadillac once again is in a class by itself. There is a change in the Chrysler line, which last year recommended a 32,000mile interval with inspection every six months. They have deemed it advisable this year to settle on a 36,000mile or 36-month recommendation. This, as you can see, duplicates the continuing recommendation of Ford, Lincoln and Mercury, as well as that of Oldsmobile for its initial lubrication of ball joints.

Pontiac and Tempest, Buick and Studebaker continue with what we might term "alternative" recommendations. That is, if chassis lubrication is performed with the factory-specified product, a longer—and sometimes surprisingly longer—interval can be observed.

1966 CHASSIS LUBRICATION INTERVALS

Make	Miles	or	Months
		07	
BUICK	6,000		6
Reg. Chassis Lub.	2,000		
CADILLAC In	spect: 6,000		2
CHEVROLET	6,000		6
CHRYSLER LINE	36,000		36
FORD, MERCURY	36,000		36
Thunderbird (Ball Joi	nts) 100,000		36
'JEEP' Universal	1,000		
Wagoneer			
2 Wheel Drive, Steer	ing		
Linkage	- 30,000		_
King Pins	6,000		
4 Wheel Drive, Steer			
Linkage	12,000		
LINCOLN CONTINEN	NTAL 36,000		36
OLDSMOBILE Ball Joi	nts 36,000	(initial; then	
		12,000 or 12 n	
Steering Linkage	12,000		12
PONTIAC	30,000		12
Reg. Chassis Lub.	6,000		6
PONTIAC TEMPEST	12,000		12
Reg. Chassis Lub.	6,000		6
RAMBLER	32,000		36
STUDEBAKER			
Factory Lub.:			
Fittings	6,000		_
Plugs	24,000		
Reg. Čhassis Lub.	1,000		

FIGURE 8.

But if what the car makers choose to call "regular chassis lubricant" is used, a much shorter interval must prevail.

Again this year, Buick and Chevrolet are adhering to the type of lubrication interval that we'd all like to see more of.

Application of Chassis Lubricants

The means by which the various chassis lubricants illustrated earlier can be applied on 1966 car models is shown in Figure 9. Generally, it can be seen that General Motors cars, except Cadillac, employ lubrication fittings on front suspension ball joints and/or steering linkage. That "and/or" has reference to largeseries Buicks, which have prepacked bearings with threaded metal plugs on the steering idler arm, and to Oldsmobile, which uses prepacks with threaded metal plugs on ball joints. Cadillac, which has sealed steering linkage, employs a push-in type plastic plug on its prepacked ball joints.

All Chrysler and Rambler models are provided with threaded metal plugs on prepacked bearings. Ford uses a mixture of prepacked bearings that are sealed and that have threaded metal plugs. The notable exception is Bronco, with its six lubrication fittings on front suspension and U-joint splines. Studebaker also has a mix, with fittings on the front suspension and threaded

1966 CHASSIS LUBRICATION METHODS

1966	5 CHASSIS LUBRICATIO	N METHODS
Make	Part	Method
BUICK	Ball Joints & Steering Linkage	Fittings
	Steering Idler Arm (ex. Special & Skylark)	Threaded Metal Plugs
CADILLAC	Ball Joints Steering Linkage	Plastic Plugs Sealed
CHEVROLET	Front Suspension & Steering Linkage	Fittings
CHRYSLER, Imperial DODGE	Ball Joints & Steering Linkage	Threaded Metal Plugs
FORD	Ball Joints & Steering Linkage	Threaded Metal Plugs
Bronco	Steering Knuckles Steering Linkage	Sealed Fittings
Fairlane	Ball Joints	Threaded Metal Plugs
Falcon Mustang	Tie Rods Pitman Arm Stud (Power Steering only)	Sealed Threaded Metal Plug
Thunderbird	Ball Joints Steering Linkage	Threaded Metal Plugs Sealed
'JEEP'	All Chassis Points	Fitting
Universal Wagoneer	King Pins & Steering Linkage	Fittings Fittings
LINCOLN CONTINEN- TAL	Ball Joints & Steering Linkage	Threaded Metal Plugs
MERCURY	Ball Joints & Steering Linkage	Threaded Metal Plugs
Comet	(Ball Joints Tie Rods	Threaded Metal Plugs Sealed
	Pitman Arm Stud (Power Steering only)	Threaded Metal Plug
OLDSMO- BILE	Ball Joints Steering Linkage	Threaded Metal Plugs Fittings
PLYMOUTH	Ball Joints & Steering Linkage	Threaded Metal Plugs
PONTIAC	Ball Joints & Steering Linkage	Fittings
RAMBLER	Ball Joints & Steering Linkage	Threaded Metal Plugs
STUDE- BAKER	Front Suspension Steering Linkage	Fittings Threaded Metal Plugs

FIGURE 9.

metal plugs on the prepacked steering linkage. 'Jeep' Wagoneer uses lubrication fittings on both king pins and steering linkage.

Lube gun adapters presently available from equipment suppliers will be adequate for the lubrication of all 1966 car models. This includes the Buick Riviera model, on which the centering ball in the constantvelocity U-joint and adjacent spline is still hard to reach because of its position in the "tunnel" created by the X-frame. A standard needle-nosed adapter, and some patience in alignment, will readily do the job. Another hard-to-reach area that doesn't even require an adapter—just patience in alignment—occurs on the Bronco. A protective shield mounted under the drive shaft partly conceals the spline forward of the transfer case.

Manual Transmission Lubricants

The gear lubricant picture for 1966 manual transmissions (Figure 10) has not changed greatly. All General Motors Divisions, except Cadillac, now recommend MIL-L-2105B lubricants—achieving consistency, while at the same time expressing their desires in a specific and generally-understood term. Buick and Oldsmobile last year had recommended Multi-Purpose lubricant, with Buick adding motor oil as an alternative.

1966 /	MANUAL TRAN	SMISSION LUBRICANTS
Make	Speeds	Gear Lubricant
BUICK	3 or 4	MIL-L-2105B
CHEVROLE	T 3 or 4	MIL-L-2105B
CHRYSLER		.AT Fluid (90 MP may be used in warm climates)
DODGE	\{4	.140 MP (80, 90, or AF may be used for hard shifting in cold-
PLYMOUTH		used for hard shifting in cold- weather operation)
FORD,	3 or 4	ESW-M2C83-A
MERCURY		
'JEEP'	3 or Overdrive	GL4
OLDSMO- BILE PONTIAC	}3 or 4	MIL-L-2105B
	J	
RAMBLER	3, 4 or Overdrive	Straight Mineral Gear Oil, Mo- tor Oil or AT Fluid
STUDE- BAKER	\int_{1}^{3} or Overdrive	Straight Mineral Gear Oil or Motor Oil
DAKER	<u></u> [4	Straight Mineral Gear Oil

FIGURE 10.

The four Chrysler makes specify either Multi-Purpose lubricant or automatic transmission fluid for their 1966-model manual transmissions. Conditions for alternative use of these products are spelled-out in the figure. A three-speed heavy-duty transmission for which we listed a lubricant recommendation in 1965 has been dropped at Dodge and Plymouth.

You can see that Ford Divisions specify a partnumbered product that can be characterized as a mild EP-type lubricant.

Both Rambler and Studebaker follow the straight Mineral Gear Oil route (including alternative engine oil or automatic transmission fluid use, except for Studebaker's four-speed transmission).

Only 'Jeep' recommends transmission drain and refill in normal service—and that at a 30,000-mile interval. But over at Chrysler, there is a recommendation for a 36,000-mile drain interval, followed by 12,000-mile drains, for all cars in severe service or equipped with a trailer-towing package.

The use of cars to haul trailers carrying boats, camping equipment, or what-have-you is increasing at such a rate that these car-trailer combinations now constitute—quite literally—a full-blown service market. The car makers are recognizing this fact in warranty provisions, but Chrysler has taken the lead, not only by setting this transmission-drain interval for cars towing trailers, but also in publishing a 16-page booklet that details the complete range of modifications it feels are necessary to suit Chrysler Corp. cars for towing trailers.

As more and more car owners engage in the hauling of boats and campers, either of which can weigh several hundred pounds, it goes almost without saying that transmissions and transmission lubricants are going to bear close watching. And the same will hold true for chassis and engines—and the lubricants used there.

Automatic Transmissions

There have been no essential changes in types of automatic transmission units employed for 1966 models (Figure 11). There is some modification involved,

1966 AUT	OMATIC	TRANSMISSION	RECOMMENDATIONS

Make	Type Fluid Drain Intervals
CHRYSLER	AT Fluid Type A, Normal-None Suffix A Severe or trailer towing package-Initially 36,000 miles, 12,000 thereafter.
FORD	AT Fluid Meeting None Ford Spec. M2C33D
GM 'Jeep'	ATFluid Type A, Normal–24,000 miles or 24 Months Severe–12,000 or 12 months
RAMBLER	AT Fluid Type A, None Suffix A
STUDE- BAKER	AT Fluid Type A, None Suffix A

FIGURE 11.

however, in the Toronado's Turbo Hydra-Matic transmission. And over at Chevrolet, home of the two-speed Powerglide transmission, they are making available the Turbo Hydra-Matic units for the 396- and 427-cu. in. optional engines in large-series Chevrolets.

To cover the rest of the field, we can state that all 1966 car models will retain the three-speed automatic transmissions they had in 1965 *except*: Buick Special, Skylark and some LeSabre models . . . the Olds F-85 . . . some Pontiacs and the Pontiac Tempest. These exceptions are equipped with two-speed units.

There occurred-effective last spring-an increase in the number of vehicles offering automatic transmissions. 'Jeep', for the first time in its history, made available both a V-8 engine and Turbo Hydra-Matic transmission as optional equipment for its Wagoneer and Gladiator models.

Transmission fluid recommendations remain constant, with all manufacturers except Ford specifying a qualified Type A, Suffix A fluid. Ford continues to specify a fluid meeting its specification M-2C33D for topping-off transmissions, but does not recommend draining and refilling.

All GM Divisions except Chevrolet continue with their recommendation to drain and refill automatic transmissions on all cars in normal service at a 24,000mile interval, and at 12,000 miles if service is severe. Chevrolet does better than this with its 12,000-mile drain interval for automatic transmissions on cars in either normal or severe service. 'Jeep' follows GM's 24,000-mile normal-service, and 12,000-mile severeservice drain intervals. Chrysler Divisions retain their 32,000-mile severe-service drain recommendation.

Neither American Motors nor Studebaker recommend draining of automatic transmissions.

Rear-Axle Lubricants

In the category of rear-axle lubricants, we find that the most part-numbered products are recommended for limited-slip axles (Figure 12). However, most recommendations for the conventional type of differential now are couched in terms the car-service industry

19	66 REAR AXLE L	UBRICANTS
Make	Regular	Limited Slip
BUICK	1	[1050081
CADILLAC		1050189
CHEVROLET	MIL-L-2105B	Positraction Lubricant
CHRYSLER		2585318
LINE		
FORD	-	
MERCURY]
170, 200 engs.	ESW-M2C50-A	FSW M2C50 A plan 4 or
240, 289, 352, 390 except with		ESW-M2C50-A plus 4 oz. additive
axle models	ESW-M2C57-A	ESW-M2C58-A per axle
WDT	J	1
410, 427, 428)	
engs., 390 with axle models	ESA-M2C80-A	
WDT	Lott-macou-it	1 - Max2011
'JEEP'	MIL-L-2105B	94557
LINCOLN CON-	ESA-M2C80-A	∫ESW-M2C50-A plus 4 oz.
TINENTAL		additive
		ESW-M2C58-A per axle
OLDSMOBILE	MIL-L-2105B or	1
except Toronado	1050081	∫1050081
PONTIAC	1050015 MIL-L-2105B	1050001
RAMBLER		1050081
STUDEBAKER	MP	AMC Approved Lub.
STUDEBAKER	GL4	Twin-Traction Lubricant

FIGURE 12.

NLGI SPOKESMAN

recognizes—rather than in part or specification numbers. This point is best illustrated, as we mentioned a few minutes ago, by the MIL-L-2105B gear lubricants specified by GM Divisions in place of part-numbered products.

Actually, the new differential (or "final drive") unit on Oldsmobile's Toronado is excepted from this otherwise-general GM recommendation, and we have already cited the part-numbered product specified for it.

Draining and refilling of rear axles continues to be recommended *only* by Chrysler and 'Jeep'. Just as they specified for manual transmissions, all Chrysler Divisions set a 36,000-mile interval, with subsequent 12,-000-mile intervals, for differential drain on cars in severe service, or equipped with the trailer-towing package. 'Jeep' has a normal-service differential drain interval of 30,000 miles.

Crankcase Lubricants

The only alterations in the crankcase-capacity picture for 1966 are the addition of Oldsmobile's Toronado, with a 5-qt. crankcase, and of Ford's Bronco, which has a 6-qt. capacity.

1966 CRANKCASE DRAI	n intervals	
Make	Drain In	terval
	Months	Miles
CHRYSLER	3	4,000
FORD	6	6,000
GM	2	6,000
'Jeep'		
F-4 engine		2,000
6-cyl. 232, V-8 327		4,000
V-6	2	6,000
RAMBLER		
Favorable conditions		
Over 10 miles per trip	_	4,000
Summer (over 32° average)		
Less than 10 miles per trip	_	2,000
Winter (below 32° average))	
Less than 10 miles per trip		1,000
or		
Dusty Driving Conditions)	
STUDEBAKER	2	6,000

FIGURE 13.

About the Author

H. ELDRIDGE, a veteran contributor to the NLGI SPOKESMAN, received his BA degree from Yale University in 1928. He was associated with the Alemite Div., Stewart-Warner Corp., from 1928 to 1932, and with the Chek-Chart Corp. from 1932 to 1941. He spent four years in the Army Air Force,

serving overseas as an intelligence officer. In 1946, Mr. Eldridge returned to Stewart-Warner where he remained until 1957 when he joined Chek-Chart as executive vicepresident. Mr. Eldridge became president of the Chicago, Ill., company after the death of Ray Shaw.



NOVEMBER, 1965

Crankcase drain intervals, seen in Figure 13, indicate that the Big Three are continuing their recommendaitons of last year: 4,000 miles or three months for Chrysler; 6,000 miles or six months at Ford; 6,000 miles or two months at General Motors.

'Jeep', which is now using a Buick V-6 engine, is following that manufacturer's 6,000-mile or two-month crankcase drain interval. Studebaker, with Chevroletbuilt power plants, is doing likewise.

We've cited the full scope of American Motors Corp.'s crankcase drain intervals for Rambler vehicles principally because we've taken a liking to that "over" or "less than 10 miles per trip" statement.

This is precisely the kind of ammunition that servicemen need to convince car owners that what they may construe as "moderate" car-use conditions — that is, short-trip driving—actually imposes a severe hardship on the car engine and its lubricant.

Outlook: 1966-67

All told for 1965, there were more than 8.6 million U.S. passenger cars sold, together with about a half-million imported cars. In round figures, let's call it 9,200,000 cars.

Car makers reportedly are optimistic about the chances for a 9 million-unit sale of U.S. cars alone during the 1966 production year.

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The facts that we've reviewed for you point out many opportunities available to dealers. We've turned a spotlight on two certain future developments: exhaust control devices and the growing car-trailer service market. Had we the time, we could also have taken up the matter of PCV service that still is not receiving the attention it deserves.

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Castrol Molub-Alloy 860/460-1 ES	BP Lubricants USA, Inc.	CORE				
Gadus [®] S3 V220C 2	Shell	CORE+			+HL	
Mobilgrease XHP™ 222	ExxonMobil Oil Corporation	CORE+		+WR		
MOLYKOTE [®] Multilub Synthetic High Performance Grease	Molykote Specialty Lubricants	CORE+				+LT
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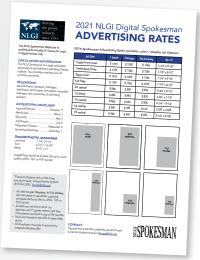
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