NLGI SPOKESMAN

Serving the Grease Industry Since 1933 - VOL. 87, NO. 5, NOV./DEC. 2023

In this issue:…

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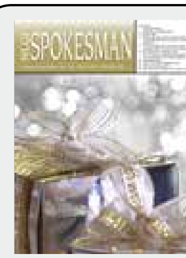

ON THE COVER

Happy Holidays!

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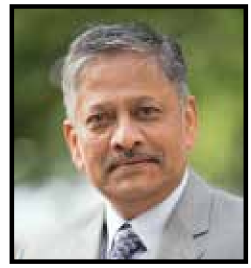
PRESIDENT'S PODIUM



Anoop Kumar, Ph. D
Chevron Products Company
NLGI President
2022-2024

As we are heading towards the end of the year 2023, it gives me a great sense of pride and satisfaction to count on the progress we have made on our six strategic priorities. This year has been very important for NLGI, being 90th anniversary year that we celebrated in grand way. Our various activities and outreach clearly demonstrate NLGI a true global organisation addressing the needs of the grease industry. We also started our quarterly Grease World E-Newsletter which is being well received and appreciated in the industry. I take this opportunity to acknowledge the volunteers in our various committees for their dedication and guidance without that we would have not reached to such heights.

Membership value, engagement and growth is one of our six strategic priorities. Jennifer Foreman, NLGI's Membership Services Manager has spent the year visiting and connecting with NLGI members in order to understand the needs of our valued members. Thank you to all that have connected with Jen to share what you like about NLGI membership as well as potential growth areas.

This is also the time to remind our members to renew their membership. As a reminder, you receive the following benefits under your company's membership:

- Discounted pricing on annual meeting registration, certification marks, reference grease, advertising, sponsorships and more.
- Repository of HPM webinars, each featuring a test within the HPM specification
- Over $350,000 funded in research grants for industry related topics, available to NLGI members only
- Complimentary Spokesman technical papers, dating back to 1941
- Complimentary annual Grease Production Survey including global grease consumption data
- Complimentary publications including *The NLGI Spokesman* and Grease World E-Newsletter
- Complimentary employment listings on NLGI website
- Complimentary membership directory with primary contact details
- Affordable education and training
- Recognition and influence
- Profitable partnerships
- Various networking opportunities
- Build lasting relationships in the industry
- Ability to increase your network

NLGI is also focused on global expansion, including enhancing membership value to our Latin America members as well as other members around the globe. Stay tuned for more details.

I wish you and your families Happy Holidays,
Anoop Kumar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HIGH PERFORMANCE
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Marine and Eco-Friendly Applications.
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Industry Calendar of Events 2024

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 member to post calendar items.)

6th International Metalworking Fluids Conference host by ILMA	**Jan 8 – 10, 2024**	Atlanta, GA, USA	6th International Metalworking Fluids Conference
NLGI India Chapter 26th Annual Conference	**Feb 22 – 24, 2024**	Kolkata, India	NLGI-IC 26th Lubricating Grease Conference
ILMA Engage	**Apr 11 – 13, 2024**	Coronado, CA, USA	ILMA Engage
ELGI 34th Annual General Meeting	**Apr 20 – 23, 2024**	Madrid, Spain	ELGI 34th AGM
78th STLE Annual Meeting and Exhibition	**May 19 – 23, 2024**	Minneapolis, MN USA	78th STLE Annual Meeting and Exhibition
NLGI 91st Annual Meeting	**June 10 – 13, 2024**	San Antonio, TX, USA	NLGI 91st Annual Meeting
2024 ILMA Annual Meeting	**Sept – Oct 1, 2024**	Colorado Springs, CO, USA	2024 ILMA Annual Meeting

NLGI COMMITTEE UPDATE

Membership Committee Update

Committee Chair: Tom Schroeder, AXEL Americas, LLC

The Membership Committee focuses on retaining current members as well as membership growth including international expansion. Additionally, this committee focuses on membership engagement, benefits, and value.

2024 Dues Renewals

The 2024 Dues Renewal process is underway! All members have received their invoices for their 2024 Membership Dues via email and a hard copy was mailed to all members within the U.S. ***All renewals are due by January 31, 2024!***

Pay your dues today by clicking HERE!

Global Expansion

The Membership Committee is supporting the efforts of the International Sub Committee to ensure that our international members and industry professionals from across the world are receiving benefits that they can utilize to the full extent. The sub-committee is looking at targeting benefits and services to the Latin America region for 2024 with plans to expand to other region in the future.

Membership Category Review

The Membership committee will be reviewing all membership categories and our current application to ensure that our process is serving our organization effectively.

***If interested in serving on a committee/sub-group, complete the volunteer form on the NLGI website.**
Please don't hesitate to contact NLGI HQ with any questions: 816.524.2500 or nlgi@nlgi.org.

FOLLOW NLGI ON LINKEDIN

Looking to stay up-to-date with the latest NLGI news, connect with fellow grease professionals, and expand your professional network? Look no further than the NLGI LinkedIn page! Follow us for updates, industry insights, and exclusive content. Join the NLGI community today and join the conversation.

Please Join us in Welcoming NLGI's Newest Members

- MANUFACTURER -

MOL-LUB Ltd.

MOL-LUB is one of the leading producers and distributors of premium automotive, commercial, and industrial lubricants, auto chemicals and additives, as well as a provider of superior lubrication services in the CEE region. They are located in Hungary and supply customers in over 50 countries worldwide.

https://mollubricants.com/en/

- SUPPLIERS -

Omni Specialty Products

Omni Specialty Products is in Shreveport, Louisiana and manufactures, markets, and distributes lubricants and chemicals to automotive and industrial markets in the United States and internationally. Omni Specialty Packaging serves mass merchandisers, retailers, wholesale distributors, manufacturers, industrial users, and the oil and gas industry and they sell their products through distributors and suppliers.

www.omnisp.com

CDA-USA

CDA-USA is a filing, capping and labeling machine manufacturer that has been operating since 1991. To meet integral packaging needs, CDA offers complete packing lines, as monobloc or custom-made for the filling, the capping / screwing and labelling of every product. Located in Richmond, Virginia, they are a sister company to CDA France, and they service multiple industries.

https://cda-usa.com/

NLGI Member Visits
November 2023

ExxonMobil – Manufacturer

NLGI's Member Services Manager, Jen Foreman, met with Luca Silva and Sherif Shawky, at the remarkable ExxonMobil Houston Campus. The visit provided a front-row seat to witness ExxonMobil's commitment to innovation. Jen took an extensive tour and learned about the history of the campus. She was also given further insight into the roles that Luca and Sherif have taken within Exxon. She learned how the NLGI has been able to assist them with valuable resources and has provided them the opportunities to network and collaborate with other industry professionals. The experience left a lasting impression, highlighting the crucial role played by forward-thinking members within the NLGI. The company's dedication to staying at the forefront of industry trends is indicative of their dedication to cutting-edge initiatives. www.exxonmobil.com

Univar Solutions – Supplier

Jen Foreman met with Brett Miller, Elizabeth Zwit, Jesse Ziobro and Cara Loewen at Univar Solutions' impressive facility in Houston. They provided her with a brief presentation about their company and wide range of services, then gave her a tour of their facility and laboratories. Univar Solutions is a global leader in specialty chemical and ingredient distribution, with a vast supplier network and a provider of value to customers among many various industries. From state-of-the-art equipment and technology to sustainable business practices, each aspect of their operations reflected an advanced approach and focus on creating a full well-rounded experience for their customers, suppliers, and products. The NLGI has been helpful with bridging partnerships for Univar Solutions and continues to provide resources and opportunities to connect them with their customers within the industry. https://www.univarsolutions.com/

NLGI Member Visits Continued
November 2023

Soltex Inc. – Supplier

Jen Foreman met with Jeff Truong and Chole West at Soltex's Baytown location. They provided her with a tour of their extensive distribution facility and laboratories used for product testing and R&D.. As a family-owned company, they provide effective and efficient service to their customers. With the continued growth of their company, they still maintain the ability to customize requests and showcase their dedication to their customers at a detailed level. http://www.soltexinc.com

Chevron Phillips Chemical Company, – Supplier

Jen Foreman had a brief visit with Greg Henke, Ken Hope and members of their team at the corporate headquarters in Woodlands, TX. They discussed resources and information regarding current industry trends, and how they can continue to utilize the NLGI. Chevron Phillips Chemical LLC is one of the world's top producers of olefins and polyolefins and a leading supplier of aromatics, alpha olefins, styrenics, specialty chemicals, plastic piping and polymer resins. They been a valuable, contributing member of the NLGI for decades! http://www.cpchem.com/

T.S. Molylubricants Inc. – Manufacturer

Jen Foreman met with the President of T.S. Moly-Lubricants., Inc. Jonell Salin, and her team at their facilty in Houston, TX. T.S. Moly-Lubricants Inc. is a manufacturer and supplier of industrial lubricants. Some of the products that they sell include greases, oils, antiseiz compounds, aerosols, and additives and has been operating since the sixties. It was interesting to learn about the history of the company and the roll that Jo Nell has been in since the early eighties when she took over the position from her father. This member visit offered an exclusive peek into the core values, innovation, and collaborative spirit of their operations. It is clear that her team takes pride in their products and customer relationships! www.tsmoly.com

Grease Lubrication of Motor Operated Valves – A Sustainable Approach for the Nuclear Industry and Beyond

Solongo Lewis, CLS, Dr. Henry Sapiano, Dr. Gamil Alhakimi – Canoil Canada Ltd.

Valves are a crucial component needed to control the flow of fluid in a process system. From the simple on/off valve found in a water faucet to more sophisticated motor-operated throttling valves, the principle remains the same – to control the flow of fluid, be it crude oil, water or a gas. While some systems simply require a valve to perform a quick open/close function, others require precision to incrementally decrease or increase the rate of flow or to change the direction of flow. In large system processes, it is rarely practical to have manually operated valves; there are often a large number required, with some being in locations that are difficult to access or that would expose operators to harsh or dangerous environments (heat, excess moisture, etc). Others may need to operate when plants or installations are unmanned or when a quick automatic response is needed. Hence, valves operated by an actuator – pneumatic, hydraulic, or electric – are a natural choice. Motor-operated valves fall into the third category, and their lubrication will be the focus of this paper.

In motor-driven valves, a gear box and motor are attached to the valve stem via a stem nut as show in the illustration below (Figure 1). Many of the rotating components require proper lubrication to minimize metal-to-metal contact and to prevent ingress of moisture and contaminants. Since continuous operation is not expected, excess heat generation is not a concern and oil lubrication would be deemed unnecessary. Furthermore, the use of oil can increase the risk of leakage out of the gearbox. Grease, on the other hand, would cling to the parts of the actuator where oil might run off and allow corrosion to begin. Specifically, the use of a calcium sulfonate complex (CSC) grease has proven to be ideal for the lubrication of all components of a motor operated valve. This is because it offers inherent wear protection, corrosion protection, resistance to water and shear stability. However, this has not always been the grease used when it comes to one of the major actuator manufacturers. For example, prior to 2002 the three main parts of one of their heavy-duty actuators were often lubricated with three different greases: 1) the main gearbox 2) the limit switch gearbox and 3) the stems. However, after detailed evaluation and rigorous testing, it was determined by the Electric Power Research Institute (EPRI) [see footnote] that one specially formulated CSC grease could replace the three separate lubricants, thereby reducing inventory, minimizing error and providing better performance.

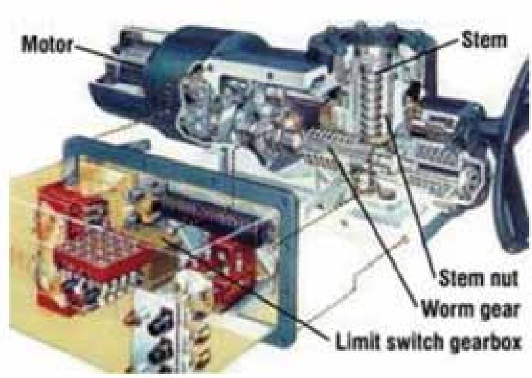

Figure 1 – Breakaway drawing of a Motor Operated Valve (MOV) actuator with main parts labelled

Footnote 1: (EPRI) conducts research, development, and demonstration projects for the benefit of the public in the United States and Internationally. It is an independent, nonprofit organization for public interest energy and environmental research, focusing on electricity generation, delivery, and use in collaboration with the electricity sector, its stakeholders, and others to enhance the quality of life by making electric power safe, reliable, affordable, and environmentally responsible. [1]

The major wear areas of the actuator components are [2]:

1) Bearings on the drive sleeve, spring pack, and worm shaft
2) Sliding surfaces such as the drive sleeve splines, worm shaft splines, and worm and worm gear teeth
3) The motor pinion and drive gear

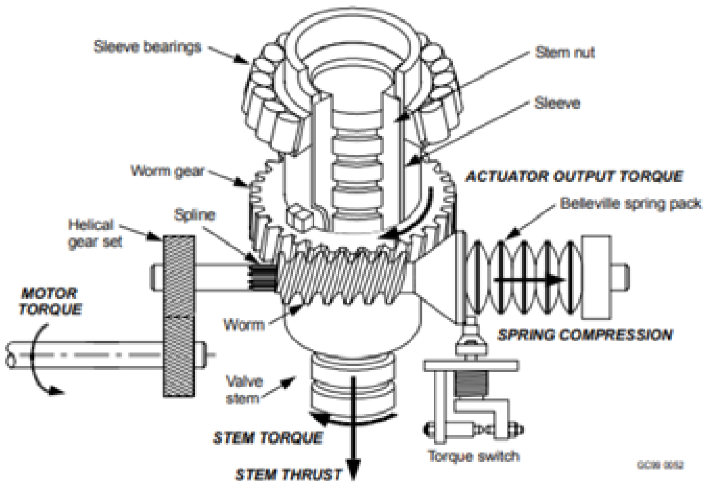

Figure 2 – Cutaway showing the gears and stem of an MOV actuator

The predominant lubrication mechanisms in the main gearbox are boundary lubrication – mainly on the worm gear interfaces – and hydrodynamic and elasto-hydrodynamic lubrication on helical gears and bearings. For boundary lubrication, the critical characteristics of the lubricant are its anti-scuffing properties. For the other two lubrication modes, the critical property is base oil viscosity. In addition, the selected grease should also have good thermal and oxidative stability, resistance to water washout, non-corrosive characteristics, low oil bleeding tendency and good compatibility with elastomers and other materials. [3]

Another important area to consider is the valve stem area. There can be very high loads on the threads of rising stem valves and running them dry or with hardened grease can quickly wear both the steel stem and the bronze stem nut, creating excessive loads. Proper lubrication of the stem/stem nut interface is therefore crucial to ensuring the right actuator output thrust. Significant changes in the coefficient of friction at this interface due to a change in the lubrication properties could result in the valve not operating properly. Too low a friction can mean that the torque setting bottoms out the valve possibly causing severe damage, while too high can mean that the torque switch stops the valve movement before it is fully closed.

To that end, the US Nuclear Research Commission (NRC) conducted studies to research the behavior of MOV stem lubricants to evaluate their performance under normal conditions, elevated temperature, and aging using a MOV load simulator. [4] The study concluded that the frictional performance of the calcium sulfonate complex grease sample (labelled "CSC Grease A" in this paper) [footnote] was stable and repeatable over a wide load range and over the simulated aging period. A series of lubricant aging tests simulating actual MOV operational strokes and flow isolation strokes was also performed. [Footnote 2] 'CSC Grease A' is made from a calcium sulfonate thickener and hydro-treated Group II base oil (95 mm^2/s at 40°C)

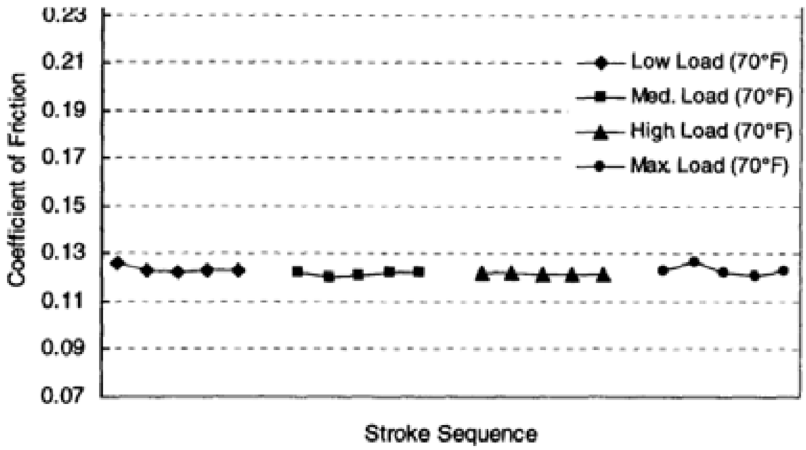

Figure 3 – coefficient of friction vs stroke sequence at various loads

Figure 3 above shows stable and repeatable stem nut friction values between 0.120 and 0.130 for each of the load levels. Even at elevated temperatures, CSC Grease A showed excellent repeatability among each set of five strokes (a single step series (70, 250, and 70°F/ 21, 121, 21°C) and a multiple step series (70, 130, 190, 250, and 70°F/21, 54, 88, 121°C) as shown in Figure 4 below. The stem nut friction drops significantly as the temperatures increase from 70 to 250°F. However, little change is seen in the friction value at 130°F.

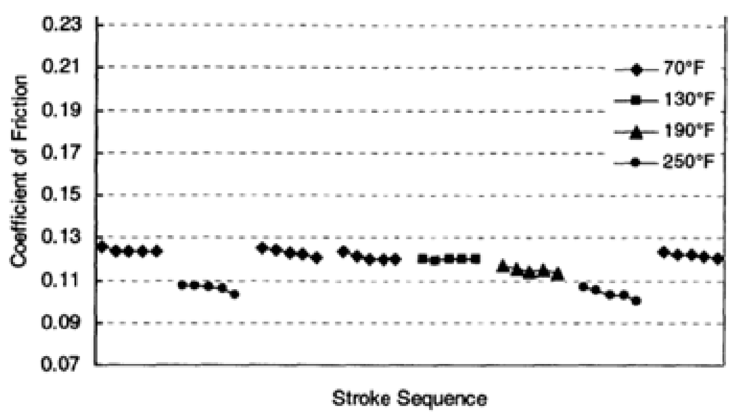

Figure 4 – coefficient of friction vs stroke sequence at varying temperatures

When it comes to the performance of the aged grease, the stem nut friction remains consistent with the initial (hot and cold) baseline tests. The overall scale of the graph in Figure 5 below relates to a simulated five-year aging period with the vertical grid lines representing a simulated one-year interval. The data show a slight increase in stem nut friction over the first 2.5 years, but no increase beyond the 2.5 years. Therefore, stem nut friction appears to be stable over the simulated 5-year period.

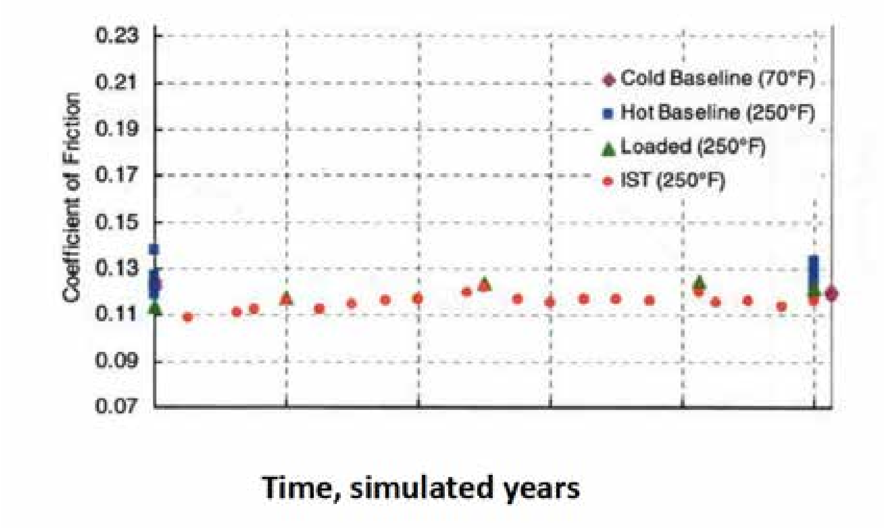

Figure 5 – Aging test timeline

While the above data shows that the friction coefficient using CSC Grease A remains stable with varying loads, temperature and aging, it doesn't give direct indication of how much wear protection it provides at the stem/stem nut interface. Using a slightly modified version of the standard ASTM D2266 4-ball wear test, Brown et al [5] showed that when compared to several competitor lubricants, CSC Grease A resulted in the smallest wear scar. To better duplicate actuator components, this modified test was run using three balls made of brass and one of 302 stainless steel under 15kg load and similar test conditions as the standard test which uses four ½" 52100 steel balls, i.e. 1200 rpm, 75°C (162°F) for 60 minutes. The results are summarized in Table 1 below.

Lubricant	Wear Scar, mm
Anti-seize lubricant paste A	2.63
Polyurea grease	2.60
Anti-seize lubricant paste B	2.40
Clay-thickened grease	1.73
Lithium complex grease A	1.55
Lithium complex grease B	1.52
Lithium complex grease C	1.43
Calcium sulfonate A	1.05

Table 1 – Wear scar results of various lubricants using a modified 4-ball wear method

The relatively low wear scar with CSC Grease A (calcium sulfonate grease A) can be attributed to the small amount of specialized EP additive present in its formulation, but more importantly to the inherent lubricity properties of the crystalline form of calcium carbonate (calcite) found within the grease's micelles. The grease thereby meets the first of eight minimum lubricant qualities required by one major OEM:

1. Should contain an "EP" *(extreme pressure)* additive.
2. Must be suitable for the temperature range intended.
3. Resistant to water, heat and separation

4. Must not create more than 8% swell in Buna N or Viton.
5. Must not contain any grit, abrasive, or fillers.
6. Must slump - prefer NLGI grade 0 to 1.
7. Must not be corrosive to steel gears, ball or roller bearings
8. Dropping point must be above 316°F (157.8°C) for temperature ranges of -20°F (-28.9°C to 150°F (65.6°C).

Due in part to the inherent properties of the thickener itself, CSC Grease A has in fact been shown to fulfill all eight of these requirements [6]. In order to demonstrate the suitability of the grease for the specific demands of the nuclear industry, however, further specialized and costly testing was required.

While actuators such as the one discussed above can find application in a variety of industries such as oil & gas, in fossil-fired plants such as coal and natural gas etc., the focus of this paper will be on their use in the nuclear power industry – one of the largest sources of reliable carbon-free electricity. There are more than 440 commercial reactors worldwide, including 93 in the United States [7] and 19 in Canada [8]. All commercial nuclear reactors in the United States are light-water reactors (either pressurized- or boiling water reactors). This means they use normal water as both a coolant and neutron moderator [9]. On the other hand, Canada and a handful of other countries use CANDU reactors. CANDU stands for CANada Deuterium Uranium, since deuterium (heavy water), is used as the reactor's neutron moderator. The reactors also differ from other reactors because they are designed to use natural uranium as a fuel (as opposed to enriched uranium). In fact, CANDU reactors use 30–40 percent less mined uranium than light-water reactors per unit of electricity produced. So not only does it require less fuel, but since the fuel does not have to be enriched, it is much less costly as well.[10]

Regardless of the type of reactor in question, safety is an important concern. Whether or not the grease in service will continue to do its job within safety-related (SR) valves during upset conditions is an important consideration. One possible failure situation is a main steam line break (MSLB) where a main steam pipe to a turbine fails, releasing hundreds of kg per second of superheated steam into the reactor or turbine building, exposing equipment to high temperatures, pressure and moisture. In addition to these stressors, radiation is another factor in nuclear facilities. Radiation leads to breakage of chemical bonds within the grease. In normal, everyday operation it will face low doses over an extended period. However, accident conditions may expose it to 200 Mrad or more radiation in a very short period of time. A Loss of Coolant Accident (LOCA) is a containment failure of the primary heat transport system to/from the reactor core, which can release hundreds of kg per second of high temperature, supersaturated and radioactive water into the reactor building, or elsewhere, which flashes to steam [11]. In the event of such accidents will the grease present in the valves be able to stand up to the harsh conditions?

This was an important consideration taken into account by EPRI before recommending a valve actuator grease for nuclear applications. In the late 90s, they began work to qualify a calcium sulfonate complex grease in thermal and radiation exposures simulating service, MSLB and LOCA conditions. Around the same time, the maker of one of the commonly used valve actuator greases was set to discontinue its manufacture. Hence, a replacement was needed. EPRI arranged for testing of a CSC grease to be conducted at Herguth Laboratories following a Quality Assurance Program (10 CFR50, Appendix B) prescribed by the USNRC. Regulation 10 CFR50 outlines the requirements binding on any license holder to operate a nuclear facility. Among other things, the Environmental Qualification (EQ) program qualifies nuclear safety-related equipment to guarantee operation under accident conditions, i.e. to guarantee safe shutdown and core cooling of a nuclear reactor in case of an accident event. EQ looks at equipment as a whole, materials of construction, and lubricants to ensure equipment operates for a mission time.

Equipment such as valves, actuators, pumps and motors, switchgear, conduit, instrumentation, etc need to be evaluated. EQ looks at cumulative normal operating and accident conditions on equipment, such as temperature, pressure, moisture and radiation, and qualifies equipment and material based on testing and/or analysis. [11]

The EPRI report found that CSC Grease A performed as well as or better than the calcium complex grease in essentially all respects [12], particularly in oxidation testing as shown below in Figure 6. While the other thickeners became severely hardened, CSC Grease A is seen to remain grease-like.

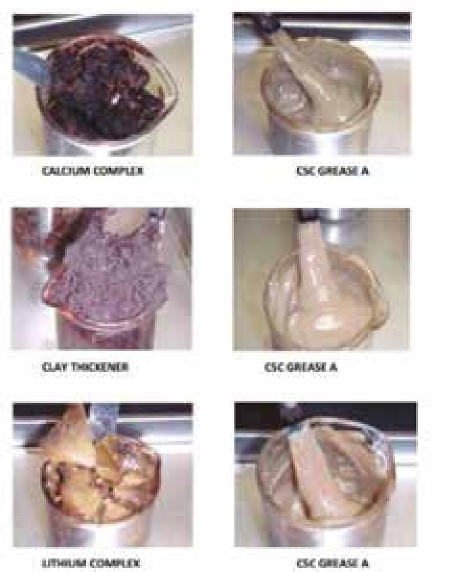

Figure 6 – Bulk aged samples – 66 hours at 177°C (350°F)

Another important report was issued around the same time by the CANDU Owners Group (COG). In their report COG-JP-01-009, CSC Grease A and CC Grease B (calcium complex) were subjected to the following sequence of stresses in a simulation of worst-case service in CANDU stations: oven aging at 130C for 660h, gamma irradiation to 70Mrad and LOCA steam exposure including 6h exposure to 171C, 105kPag steam [3]. Table 2 below shows that even after a simulated 5 year aging, 70MRad exposure and LOCA conditions (bulk), the CSC Grease A sample only stiffened by one-half NLGI grade. The corresponding thin film sample (LOCAVP) only stiffened by one grade. In comparison, CC Grease B stiffened by two NLGI grades in a shorter aging period as shown in Table 3. This data corresponds well to the results of the EPRI report mentioned above.

Sample Description[1]	Full scale Penetration	NLGI Grease Grade
NEW UNAGED	326	1
1.6 YR	317	1
3.8 YR	314	1
5YR	318	1
5YR + 70 MR	326	1
5YR+70MR+LOCACAN	309	1.5
5YR+70MR+LOCAVP	283	2

Table 2 – 'CSC Grease A' penetration (consistency) data

<u>Note:</u> YR = years of aging , MR = Mrad exposure, LOCACAN = grease packed in perforated can during LOCA, LOCAVP = grease mounted on vertical plates during LOCA.

Sample Description [1]	Full Scale Penetration	NLGI Grease Grade
NEW	312	1
1.6 YR	281	2
- 3.8 YR	215	3.5
3.8 YR + 70MR	248	3

Table 3 – 'CC Grease B' penetration (consistency) data

Another interesting test result is that the post-LOCA wear scar for CSC Grease A was the same as for the new grease (0.40mm), indicating no loss of wear protection due to aging. With regard to the effect of LOCA steam on the appearance of the grease, it is of note that the aged CSC Grease A sample did not slump from the vertical panels or become hard; the only significant change in appearance was a darkening of the material as shown in Figure 7 below.

Figure 7 – Appearance of 5-year aged & 70 Mrad irradiated CSC Grease A before and after exposure to LOCA conditions

Similarly, the aged CC Grease B sample also suffered from darkening but did not slump from the panels (Figure 8).

Figure 8 – Appearance of 3.8-year aged & 70 Mrad irradiated CC Grease B before and after exposure to LOCA conditions

Figure 9 shows a similar pair of CC Grease B panels for in-service grease removed from an actuator gearbox, then LOCA tested. In addition to the effects noted above, the grease 'bubbled' during LOCA, presumably due to water evaporation. It appears that prolonged use in an actuator can result in pickup of water by the calcium complex grease sample.

Figure 9 - In-service CC Grease B before/after LOCA showing bubbles

Other test results reported included evaporation loss D972, dropping point and IR analysis. The CSC Grease A sample experienced less than a 2 percent change in weight due to evaporation, whereas the CC Grease B sample saw a 6 percent weight change and turned stiff with bubbles, similar to Figure 12. The dropping point of CSC Grease A was not significantly affected by the thermal aging, irradiation and LOCA exposure. IR analysis showed minor changes before and after irradiation and LOCA. The final findings of the report was that 'CSC Grease A' was recommended for use in the OEM gearboxes in nuclear applications of CANDU design stations and was also recommended as a top-up to existing OEM gearboxes filled with a calcium complex grease, since compatibility was established.

Thus far, CSC Grease A has been shown to meet both OEM and nuclear EQ requirements. Beyond that, this grease also has to meet the audit and QA requirements for nuclear stations, including the need for low halide content. This means that in addition to the standard certificate of analysis provided with each grease batch, a complete ICP metal analysis and halogen content report has to be included. The main concern with the presence of halides is that they can contribute to stress corrosion cracking (SCC) of steel alloy fasteners. Although sulfur is also a concern when it comes to SCC, it must be noted that the sulfur within the sulfonate of CSC grease is 'bound' and not free, and therefore not reactive.

Although this paper has focused on the performance of CSC grease A in MOV actuators, it would also perform very well in electric motor bearings despite possible concerns about its relatively higher thickener content [13]. Additionally, it is suitable in applications where the spent grease eventually ends up in waterways, such as travelling screens or trash rakes of hydroelectric plants since it is relatively harmless to aquatic life, contains no dyes and is not expected to bioaccumulate. In aquatic toxicity testing, the 48 hour acute static non-renewable toxicity value was >1000ppm (test EPR Method 2021.0 and ASTM D6081-98 using *Daphnia pulex* (water flea)) [14]. While some toxicity data is available on the grease's major components, further testing on the final product itself is needed to give a complete picture of its impact on the environment.

Beyond just the environmental considerations, however, this grease product can be said to contribute to sustainability as a whole. Although, the word "sustainability" is sometimes thought of only in terms of environmentalism, it actually deals with the intersection of the environment, society, and the economy [15]. When a product or process contributes to the balance of these three factors, it can be termed "sustainable". Improved machine life as a result of the longevity of CSC Grease A certainly contributes to an environmentally-conscious product since it translates into increased life expectancy of the actuator and other lubricated components. This means less waste generation of metal parts and of the grease product itself. Furthermore, it may be useful to consider which industry is the largest consumer of a particular product. In this case, the calcium sulfonate complex grease product has been geared towards the nuclear industry; one which protects air quality, has a small land footprint and produces minimal waste [16].

From an economic standpoint, sustainability takes into consideration the ability to sustain economic and financial goals at both the macro (e.g. entire industries) and the micro level (e.g. individual businesses). On a micro level, a manufacturer remains sustainable by making a reliable, high-quality product which creates value to the end user. For instance, a large nuclear station may have several hundred valve actuators requiring grease maintenance and/or replacement. Such a plant would see considerable savings in cost and time due to the extended life of its grease. These savings could then be used to positively impact sustainable practices in other areas.

Calcium sulfonate complex grease continues to play an important role in several industries, including the nuclear industry. It offers inherent wear protection, corrosion protection, resistance to water and mechanical stability. This paper has demonstrated that a CSC grease is well suited to lubricating the three major areas of a motor-operated valve actuator – the main gearbox, the limit switch gearbox and the stems. The stem area, in particular, is prone to severe loads but extensive testing has demonstrated that even with varying loads, temperature and aging, stable friction coefficients were obtained. Furthermore, when the grease was subjected to conditions simulating those that would be experienced under accident conditions (exposure to hot, pressurized, irradiated steam) only mild changes were observed in its consistency, colour, IR spectrum and weight due to evaporation. The grease has also been shown to have a low order of toxicity in one aquatic test. While further testing is needed to gain a better understanding of the grease's environmental impact, it can be argued that it contributes to sustainability based on its longevity and its prevalence in the carbon-free nuclear industry.

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The Three Categories of Lubricating Grease Formulation Work: A Forty-Six Year Perspective

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Abstract
Formulation of new products is a key activity of research scientists that work in all product areas. This is no less true for lubricating greases. Using the perspective of forty-six years as a research chemist, the author has determined that all lubricating grease formulation work can be considered to fall primarily into one of three categories.

1. Using only existing componentry (base oils, thickeners/thickener reactants, additives), and using them in the manner that is well-known to achieve results that are also well-known and expected, based on information available in the open literature.

2. Using only existing componentry but using at least one existing component in a novel way that achieves a beneficial result that has not been previously documented in the open literature.

3. Using at least one component that has not been previously used in lubricating greases, but that nonetheless provides a beneficial result, either by itself or in combination with other components.

These three categories are not always mutually exclusive. Some formulation work may be considered to have aspects of more than one category. Nonetheless, understanding all formulation work within this formalism can help the chemist to reach the desired balance of performance and formula cost in a more time-efficient manner.

Using work done by the author during the past forty-six years, examples of all three of these categories are provided. These examples showcase not only the way these three formulation categories work, they also demonstrate the value that each category can provide when correctly used.

Introduction
Chemists who work for science and technology-based companies that manufacture and market lubricating greases can perform a range of activities. Some will work in technical service, assisting sales personnel or actual end-use customers. Others work within a more literal research and development environment where new product development or product reformulation/improvement are the primary activities.

Some of this work will be borne out of necessity due to the unavailability of one or more raw materials. Other work will be required due to a change in the hazard classification of one or more raw materials. These two activities have become more frequently required during the last few years.

However, the core activity of lubricating grease research and development has been and continues to be the formulation of new products to meet new and more stringent performance demands, or the reformulation of existing products for the same reasons. For forty-six years, the author has been heavily involved in such formulation activities in the areas of fuels, fuel additives, fluid lubricants, and especially lubricating greases. Regardless of the area of formulation work, it has been noted that all such work can be placed primarily into one of three categories. Some formulation work may have aspects of more than one of these three categories. The boundaries between the categories may sometimes be fuzzy. Nonetheless, the persistence of these three categories has always been apparent in all the formulation work done by this author over his entire career.

The remainder of this paper provides examples spanning most of the author's career. These examples demonstrate how the three categories apply to lubricating grease formulation. The experimental details of each example, as needed, are provided as the example itself is discussed. The examples themselves are taken from information previously published within the open literature, primarily U.S. patents where the author was involved as named inventor. Obviously, no research paper of typically acceptable length could adequately cover all the formulation work done over a forty-six year career. However, the examples included herein are representative of that body of work. They are also representative of what can be expected by any lubricating grease formulation chemist during their career.

FORMULATION CATEGORY 1: *Using only existing componentry (base oils, thickeners/thickener reactants, additives), and using them in the manner that is well-known to achieve results that are also well-known and expected, based on information available in the open literature.*

Example 1 – Motor Oil Formulation

Perhaps the clearest example of a Category 1 formulation is a lubricant that is not a grease: SAE and ILSAC licensed motor oils. The Society of Automotive Engineers (SAE) and the International Lubricant Specification Advisory Committee (ILSAC) oversee the testing and performance requirements for licensed motor oils used in both gasoline and diesel vehicles. Both organizations use the American Petroleum Institute (API) Engine Oil Licensing and Certification System (EOLCS) that specifies the test requirements for all defined viscosity grades. These requirements are detailed in API 1509 [1]. Additional details of how motor oils must be formulated and tested to verify full compliance with all requirements are provided in the American Chemistry Council Petroleum Additives Product Approval Code of Practice [2]. For any viscosity grade, both Level 1 and Level 2 requirements must be met. Level 1 includes laboratory test requirements such as viscosity, volatility, and compositional limits. Level 2 includes the engine tests that measure the various performance requirements.

The suppliers of the performance additive packages generally do the Level 2 tests. One or more motor oil formulations using the additive package are submitted to an approved outside laboratory for Level 2 engine testing. Once the performance additive supplier obtains documented passing results on all Level 2 tests, they can market their additive package to motor oil manufacturers. Those manufacturers must use the Level 2 approved additive package at the concentration for which it was approved. Additionally, the additive package must be used in either the base oils for which the Level 2 engine tests were validated, or in base oils that read across the base oil interchangeability information as defined in API 1509 Annex E. The only other additives that can be included in such a licensable motor oil are a VI improver and a cold flow improver (if one was not included in the Level 2-approved performance additive package). Both additional additives must also be approved based on the requirements of API 1509. No new additive components are allowed in the motor oil formulation. Failure to meet all these formulation requirements will invalidate the expensive Level 2 engine test approvals obtained by the performance additive supplier.

Because of these stringent requirements on formulation chemistry, formulators of licensable motor oil only have to make adjustments in the amounts of the various allowed base oils and VI improver so as to simultaneously meet all the Level 1 requirements. Often, the performance additive supplier will provide guidance in doing this. Once this is done, the motor oil formulation is ready for submission for SAE and ILSAC licensing. Clearly, such formulation work is quintessentially Category 1.

Example 2 – Strategic Cruise Missile Engine Thrust Bearing Grease

In the late 1980's, the U.S. Air Force noted that the thrust bearings in engines that power their strategic cruise missiles were experiencing corrosion during prolonged storage. Such corrosion could adversely affect the operation of such missiles, should they need to be launched. The cause of this corrosion was determined to be due to the bearing grease's inability to provide adequate corrosion protection during extended storage, especially near humid saltwater environments. The author's company was contracted by the U.S. Air Force to develop an improved engine thrust bearing grease that would provide the required performance properties. Besides greatly improved corrosion resistance, the new grease required good extreme pressure/antiwear (EP/AW), low oil bleed, passivity to ferrous and non-ferrous metals, and good performance from about -54 C (-65 F) to about 135 C (275 F).

Based on these performance requirements, a lithium 12-hydroxystearate (Li 12HSt) grease in polyalphaolefin (PAO) was chosen as the thickener/base oil combination. Table 1 provides the final formulation information on the new grease.

TABLE 1
Cruise Missile Engine Thrust Bearing Grease Formulation

	wt%
PAO (40 cSt at 100 C)	47.0
PAO (6 cSt at 100 C)	31.3
Lithium 12-Hydroxystearate	12.0
Ashless Dithiocarbamate	3.0
Tri-Aryl Phosphate Ester	3.0
Barium Dinonyl Naphthylene Sulfonate (in PAO)	1.5
Borated Alkenyl Amide	1.5
Phenyl Alpha-Naphthylamine (PANA)	0.5
Alkyl Tolutriazole	0.1
Excess Lithium Hydroxide	0.1

During laboratory development, the grease was made using a procedure that is typical of such simple lithium soap-thickened greases. The grease was made in a 40-pound capacity kettle. The kettle had double action mixing and could be sealed and heated under pressure. The grease was made a follows:

1. First, the 12-hydroxystearic acid (12HSA) was added to a portion of the blend of the two PAO's as per the ratios indicated in Table 1.

2. The blend was heated to about 82 C (180 F) to melt and dissolve the 12HSA.

3. Then enough lithium hydroxide monohydrate was added to fully neutralize the 12HSA plus a 0.1 wt% excess lithium hydroxide (anhydrous) in the final grease. A small amount of water was also added to help facilitate the reaction.

4. The kettle was sealed and heated under pressure until the temperature was about 121 C (250 F).

5. The kettle was vented and opened. FTIR indicated that the reaction was complete.

6. The grease was heated to about 204 C (400 F) to fully dehydrate and melt the thickener. During this heating process, once the water had been removed (as indicated by FTIR), the PANA was added.

7. After reaching top temperature, the mixture was then cooled to reform the grease.
8. The remaining PAO was added once the grease cooled to about 149 C (300 F).

9. The additives were added once the grease had cooled to about 93 C (200 F).

10. The grease was milled using a laboratory-scale colloid mill.

The Table 1 formulation is deceptive in its apparent simplicity. In fact, there are several interactions and cases of multiple functionality that contribute to its performance.

First, combination of the ashless dithiocarbamate and tri-aryl phosphate ester provide mild EP and excellent AW performance. However, both these materials are also well-known hydroperoxide decomposing (Class 2) antioxidants that work cooperatively with the chain-breaking (Class 1) high temperature antioxidant PANA [3].

Second, the barium di-nonyl naphthalene sulfonate and the borated alkenyl amide are both good ferrous (rust) corrosion inhibitors that work by different mechanisms that do not interfere with each other. This provides an excellent cooperative functionality. Additionally, the borated amide also provides a small boost to the dropping point of lithium soap-thickened greases and helps to restrict oil bleed. This latter property will be discussed in greater detail in the section on Category 2 formulations.

Finally, the tolutriazole is an excellent sulfur-free metal deactivator that provides cleanup to any mild aggression to yellow metals that might be caused by the dithiocarbamate.

The slight excess of lithium hydroxide ensures that no free 12-hydroxystearic acid remains in the final grease, thereby providing better shear stability. The very low amount of this excess basicity should also avoid a deleterious effect to rust inhibitor performance that may sometimes result when larger amounts of unreacted lithium hydroxide are present in the overall lithium soap thickener [4]. The excess lithium hydroxide also provides another functionality that will be described when Category 2 formulations are discussed.

Despite the intricate and multiple interactions of the various additives, all such interactions were well-known when this grease was formulated. Accordingly, this grease was essentially an example of a Category 1 formulation.

Laboratory test results for the formulation of Table 1 are provided below in Table 2.

TABLE 2 Cruise Missile Engine Thrust Bearing Grease Test Results	
Worked 60 Stroke Penetration, ASTM D217, 0.1 mm	307
Worked 100,000 Stroke Penetration, ASTM D217	
Final Value, 0.1 mm	329
% Change from Worked 60 Stroke Value	7.2
Dropping Point, ASTM D2265, C (F)	206 (403)
Oil Separation, FTM 791-321, % Loss	
24 hr, 100 C	2.5
100 hr, 100 C	5.9
Copper Corrosion Protection, ASTM D4048, 24 hr, 100 C	1B
Four Ball Wear, ASTM D2266, mm	0.4
Four Ball EP Load Wear Index, ASTM D2596	40.6
Optimol SRV Stepload Test, GM Procedure, Maximum Pass, N	1000
Corrosion Prevention, ASTM D1743, 5% Synthetic Sea Water	Pass, Pass
Water Washout at 38 C (100 F), ASTM D1264, % Loss	2.5
Low Temperature Torque at -40 C (-40 F)	
Starting, N-m	0.320
Running, N-m	0.058
Low Temperature Torque at -54 C (-65 F)	
Starting, N-m	1.06
Running, N-m	0.23

It should be noted that FTM 791-321 is the cone sieve oil bleed test procedure from which ASTM D6184 was derived. The SRV procedure was the one used by General Motors during the time this work was done. The two current ASTM SRV procedures (D5706 and D5707) had not yet been developed or standardized. Similarly, the ASTM D1743 synthetic sea water bearing corrosion prevention test is similar to what is now ASTM D5969. This test was run in duplicate, and the results for each replicate are accordingly reported.

The formulation of Table 1 was further evaluated by the U.S. Air Force using a test rig designed to closely approximate actual engine experience in the cruise missiles during startup and flight. Actual cruise missile ball bearings were used. The test rig was operated at 30,000 rpm and 396 pounds of thrust. Such tests were run on bearings packed with fresh grease, and on bearings packed with fresh grease and then stored for one, three, and six months in a cabinet at 71 C (160 F) and 100% relative humidity. The new grease was found to provide such a high level of performance that a new specification, MIL-PRF-32014, was created specifically for it. The grease was also evaluated for another military application and found to work well [5]. At the request of the U.S. Air Force, the grease formulation was submitted as a U.S. patent application. The patent was issued on July 28, 1992 [6].

Example 3 – An Ultra-Low Temperature Grease Requiring Diester Base Oil

In the 1980’s, MIL-G-23827B had one requirement that most obviously distinguished it from most other existing military grease specifications: a stringent requirement for performance at -73 C (-100 F). The reason for such extreme low temperature performance requirements was due to the use of the grease in actuators and other lubricated components in high-altitude, supersonic military aircraft.

The author’s company had been making a grease approved for MIL-G-23827B for years. However, the volume demand for the grease was small due to the very small amount of grease needed for the specified aircraft components. One or two commercial batches each year was sufficient to meet that demand.

The grease was a lithium 12-hydroxystearate grease made entirely in a diester synthetic base oil. The diester base oil was necessary owing to its ultra-low temperature flowability. PAO did not provide the necessary low temperature properties; neither did polyol esters available at that time.

The procedure for making this grease involved the usual reaction of lithium hydroxide with 12HSA in a portion of the diester base oil. Although such a reaction is, in theory, possible, it was nearly impossible to achieve in actual practice. Unless the temperature was held within a very tight range with only the minimum critical amount of water, the diester base oil would significantly hydrolyze. Additionally, the carboxylic acids formed by the hydrolysis of the diester base oil could also react with the lithium hydroxide. Some of the 12HSA could also exchange with the acid moieties of the diester base oil via the well-known base-catalyzed transesterification reaction. The result of such unwanted reactions would be a ruined and unrecoverable batch. Since only one or two such batches were made each year, the grease makers were never able to obtain the necessary experience to maintain the tight control required to successfully make a batch. The result was frequent slopped batches and the resulting adverse impact on operating costs.

The author was given the task to develop a reformulated grease that would meet all the MIL-G-23827G requirements but have none of the manufacturing issues associated with the existing formulation. Table 3 provides the final formulation information on the new ultra-low temperature grease.

TABLE 3
Ultra-Low Temperature Grease Requiring Diester Base Oil

	wt%
PAO (4 cSt at 100 C)	40.1
Di-2-Ethyl Hexyl Azelate	40.0
Lithium 12-Hydroxystearate	12.0
Ashless Dithiocarbamate	3.0
Tri-Aryl Phosphate Ester	3.0
Barium Di-Nonyl Naphthylene Sulfonate (in PAO)	1.0
Phenyl Alpha-Naphthylamine (PANA)	0.5
Alkyl Tolutriazole	0.1
Calcium Hydroxide	0.1
Excess Lithium Hydroxide	0.2
Orange Dye	0.01

Comparing Table 3 with Table 1, it is apparent that this new ultra-low temperature grease formulation is very similar to the previous cruise missile engine thrust bearing grease. The purpose of the very small amount of calcium hydroxide that was added with the other additives will be discussed in the next example. The discussion of the additive functionalities and interactions of the previous example apply to this grease and will not be repeated.

The primary feature of this grease formulation was its inclusion of a 4 cSt PAO as 50% of the total added base oil. Preliminary viscometric evaluation of this base oil blend had indicated that a simple lithium soap grease in such a base oil blend should satisfy the stringent low temperature requirements of MIL-G-23827B. PAO is a hydrocarbon and is therefore hydrolytically stable. This allowed an initial concentrated grease to be made entirely in the PAO, thereby avoiding the problems associated with trying to make such a grease in a diester base oil. The procedure by which this was done was comparable to what was described in the previous example. Once the initial grease had been heated to top processing temperature and cooled below about 149 C (300 F), the diester base oil was added. Then the grease was additized and milled in a manner like the previous example grease.

This manufacturing process solved the hydrolysis problem of the earlier grease. However, it did so by utilizing base oil properties that were already well-established. Since the additive chemistry was also well-established, as described in the previous example, this ultra-low temperature grease was essentially an example of Category 1 formulation. Even so, both the grease formulation and the process by which is was made was eventually covered by U.S. patents [7, 8].

Laboratory test results for the formulation of Table 3 are provided below in Table 4.

TABLE 4 Ultra-Low Temperature Grease Test Results	
Worked 60 Stroke Penetration, ASTM D217, 0.1 mm	289
Shear Stability, FTM 791-313, Final Value, 0.1 mm	335
Dropping Point, ASTM D2265, C (F)	192 (378)
Oil Separation, FTM 791-321, 30 hr at 100 C, % Loss	3.4
Copper Corrosion Protection, ASTM D4048, 24 hr, 100 C	1B
Four Ball EP Load Wear Index, ASTM D2596	30.0
Water Washout at 38 C (100 F), ASTM D1264, % Loss	3.0
Oxidation Resistance at 99 C (210 F), ASTM D942,	
Pressure Drop after 100 hr, kPa.	7.0
Pressure Drop after 500 hr, kPa	17.5
Low Temperature Torque at -73 C (-100 F)	
Starting, N-m	0.59
Running, N-m	0.043

Full testing of this grease was performed by the U.S. Navy. Accordingly, the grease was approved against MIL-G-23827B.

FORMULATION CATEGORY 2: *Using only existing componentry but using at least one existing component in a novel way that achieves a beneficial result that has not been previously documented in the open literature.*

Example 4 – Restricting Oil Bleed With Very Small Excess Of Hydroxide Base

Of the formulation work that falls outside of Category 1, the majority will be in Category 2. As previously mentioned, formulation work will sometimes include aspects of more than one category. This is especially true in the case of work that is primarily Category 1 but which also contains a minor aspect of Category 2. This first example of Category 2 formulation work is an example of this.

In the previous Example 2 grease, a very small amount of excess lithium hydroxide was used to insure complete reaction of the 12HSA with no remaining free acidity. In the Example 3 grease, a very small amount of excess lithium hydroxide *and* calcium hydroxide was used. During the formulation work resulting in the final Table 3 grease, it was determined that having small amounts of both metal hydroxides helped restrict oil bleed.

A grease was made similar to the Example 3 grease. The only difference was that no excess lithium hydroxide was used. Portions of this grease were added to small steel cans and additized with combinations of lithium hydroxide, calcium hydroxide, and a high molecular weight polyisobutylene polymer. The latter was a very commonly used polymer to impart extreme stringiness and restrict oil bleed in greases. The greases were mixed by hand and heated to about 100 C in a forced air convection oven. Then each grease was mixed again and given three passes through a three-roll mill with both gaps set at 0.01 mm (0.003 inch).

Compositions and test results are provided below in Table 5. Note that the grease with both hydroxides has essentially the same composition as the grease of Table 3.

TABLE 5
Restricting Oil Bleed With Very Small Excess Hydroxide Base

Grease No.	5-1	5-2	5-3	5-4
Example 3 Grease Without Excess Lithium Hydroxide, wt%	99.9	99.8	99.7	96.0
Lithium Hydroxide Monohydrate, wt%	0.0	0.2	0.2	0.0
Calcium Hydroxide, wt%	0.1	0.0	0.1	0.0
Polyisobutylene, wt%	0.0	0.0	0.0	4.0
Oil Separation, FTM 791-321, 30 hr at 100 C, % Loss	4.3	3.8	3.5	2.8

Test results are the average of three determinations. Adding the very small amount of calcium hydroxide appears to impart a small additional restriction of oil bleed. Note that the oil bleed of Grease 5-3 with both hydroxides is essentially the same as the value reported in Table 4. However, the most interesting aspect is that the oil bleed of Grease 5-3 is only slightly higher than the value of Grease 5-4 with 4.0% polymer. The cost of this polymer was more than 10 times the cost of either metal hydroxide at the time this work was done. That fact combined with the concentrations of each material resulted in a much higher total additive cost for the polymer grease compared to the grease with both hydroxides. Additionally, Grease 5-4 had a significantly tacky texture that would be expected to be even greater had the polymer been added to a milled grease without further milling. Although it is sometimes desirable to have an extremely stringy, tacky grease, such a rheology is not always appropriate. When such situations exist, being able to restrict oil bleed without using polymers can be an important formulation tool.

The use of very small amounts of excess lithium hydroxide and unreacted calcium hydroxide in lithium soap greases to restrict oil bleed was not known by the author at the time the Example 3 grease formulation work was done. Accordingly, this aspect of the Example 3 formulation could be considered to fall into the Category 2. The next example provides a clearer case of a Category 2 formulation.

Example 5 – Restricting Oil Bleed in Polyurea Grease by Using Borated Chemistry

The author's first major grease formulation project was to develop a new and higher performance grease for initial-fill Constant Velocity Joints (CVJ) as used by a major U.S. automobile manufacturer. That work was ultimately successful and resulted in lubricating grease technology covered by several U.S. patents [9, 10]. This example and the next one illustrate clear cases of Category 2 formulation in the same final grease composition.

In developing an improved CVJ grease, the final formulation was a polyurea grease with the composition as indicated below in Table 6.

TABLE 6 Polyurea Grease For CVJ Applications	
	wt%
Paraffinic Base Oil (850 SUS Oil at 100 F)	47.6
Paraffinic Severely Hydrotreated (White) Base Oil (350 SUS at 100 F)	31.2
Polyurea Thickener	9.5
Tri-Basic Calcium Phosphate	5.0
Calcium Carbonate	5.0
Barium Dinonyl Naphthalene Sulfonate (Neutral)	1.0
Borated Alkenyl Amide	0.5
Mixed Aryl Amine Antioxidant	0.2
Dye	0.02

The two base oils are specified using the viscosity units commonly used by refineries at the time this formulation was developed. Using the current metric conventions, they would approximately correspond to ISO 150 and ISO 68 viscosity grades. The two inorganic solids had a mean particle size of about 3 microns and were used to provide EP/AW. The barium sulfonate and aryl amines are a rust inhibitor and antioxidant, respectively. Details of how the polyurea grease was made is provided in the next example.

During the early work to develop the improved CVJ grease, the Table 6 formulation without the borated amide was used. That formulation gave excellent EP/AW as determined by a stepload test using the Optimol SRV machine. The U.S. automobile manufacturer had incorporated this SRV stepload test into the specification requirement for their initial-fill CVJ grease. (This was before the ASTM developed standardized SRV test procedures.) The early formulation without the borated amide also met the other specification requirements. However, oil bleed by the automobile manufacturer's specification cone sieve method (similar to FTM 791-321) was at best marginally acceptable. The incorporation of 0.5 wt% of the borated amide reduced the oil bleed to acceptable levels. Additionally, the oil bleed was controlled even when the oil bleed test was run at 149 C (300 F) and 178 C (350 F). Although the CVJ grease specification did not set a limit on oil bleed at such higher temperatures, it was believed that this was a desirable property since one of the inboard CVJ's experienced much higher operating temperatures owing to its very close location to the fuel intake manifold.

To evaluate the borated amide's ability to restrict oil bleed, two polyurea greases, Greases 7-1 and 7-2, were prepared with essentially the composition of Table 6. The polyurea thickener content was 9.6 wt%. The concentration of the other additives was as indicated in Table 6. The only difference between the two greases was that one did not contain the borated amide whereas the other contained it at the Table 6 value of 0.5 wt%.

So as to evaluate the effect of the borated amide when the consistency of the grease was much softer, two additional polyurea greases, Greases 7-3 and 7-4, were similarly prepared. The polyurea thickener content of these two greases was 6.0 wt%, significantly less than the previous two greases. The concentration of the other additives were as indicated in Table 6. The only difference between the two greases was that one did not contain the borated amide whereas the other contained it at the Table 6 value of 0.5 wt%.

The oil bleed test results of these four polyurea greases are provided below in Table 7. The two sets of greases are color coded to make the presentation clearer.

TABLE 7 Restricting Oil Bleed in Polyurea Grease by Using Borated Chemistry				
Grease No.	7-1	7-2	7-3	7-4
Polyurea Thickener, wt%	9.6	9.6	6.0	6.0
% Borated Alkenyl Amide	0.0	0.5	0.0	0.5
Worked 60 Stroke Penetration, ASTM D217, 0.1 mm	312	315	383	384
Dropping Point, ASTM D2265, C (F)	255 (491)	258 (497)	ND	ND
Oil Separation, SDM 433, % Loss				
6 hr, 100 C	5.5	3.3	ND	ND
24 hr, 100 C	8.7	6.0	9.6	6.9
24 hr, 149 C	9.7	7.9	12.1	5.6
24 hr, 178 C	15.7	8.1	34.3	30.0

The test results in Table 7 demonstrate the ability of this borated additive to restrict oil bleed. The U.S. automobile manufacturer required oil bleed by their test procedure to be no more than 6.0 wt% after 6 hours at 100 C. For the two greases with 9.6 wt% polyurea, the one with the borated amide improved the oil bleed from near the limit of the specification to the middle of it.

However, the improvement (decrease) in oil bleed was most apparent when the test was run at the higher temperatures. For the greases with 9.6 wt% polyurea, the borated amide had its largest effect at 178 C (350 F). For the greases with only 6.0 wt% polyurea thickener, the borated additive reduced oil bleed by more than 50% when the test was run at 149 C (300 F). However, this ability to reduce oil bleed was essentially lost when the test was run at 178 C (350). Given the extremely soft consistency of the 6.0 wt% polyurea greases, the ability of the borated amide to reduce oil bleed at 149 C is remarkable. Such soft greases are essentially pourable. Dropping point is essentially meaningless for such greases, which is why that test was not run on them.

At the time this work was done, the borated alkenyl amide was marketed by its supplier as a rust inhibitor. Its use in the CVJ formulation provided additional rust protection to that imparted by the barium dinonyl naphthalene sulfonate. However, until this work had been done, this borated additive was not known to provide reduction in grease oil separation. Now that the U.S. patent that covered such use has expired, the supplier now claims oil separation improvement as a property of this additive. Thus, the original work using this borated additive as a method to restrict oil separation clearly meets the definition of a Category 2 formulation. Other borated additives were also shown to provide such oil separation reduction [9].

Example 6 – Using a Different Base Oil to Solve a Dropping Point Problem

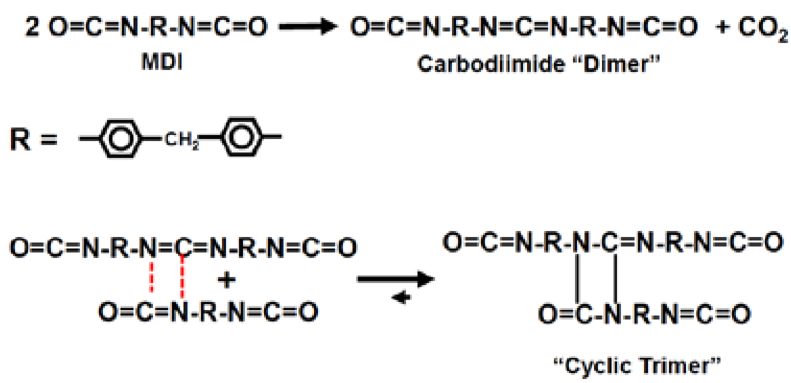
During the early stages of the formulation of the CVJ grease, as described in the previous example, the base oil blend was not what is provided in Table 6. Specifically, the severely hydrotreated white oil was not used. Instead, a lightly hydrotreated base oil of the same viscosity grade as the white oil was used. Why was that base oil ultimately replaced with a more expensive white oil? The answer had nothing to do with improved oxidation stability or any other property that was then associated with severely hydrotreated white mineral oils. Instead, the answer involved a dropping point problem.

To understand the dropping point problem, a brief discussion of the polyurea thickener chemistry is required. Polyurea grease was first developed in the 1950's [11]. By the time the CVJ grease formulation project described in the previous section was done, one way to make polyurea grease thickener involved the reaction of a di-isocyanate, diamine (usually ethylene diamine), and fatty amine in a portion of the base oil. However, the polyurea grease thickener of the previous Example 5 grease was not made that way.

The author's company manufactured polyurea grease by reaction of a di-isocyanate, fatty amine, and water. The details of this process can be found in several U.S. patents [9-10] and will not be repeated here. The reaction was done under pressure and required other measures to insure a correct balanced reaction profile was achieved. When properly done, the water reacted with a portion of the di-isocyanate to form the corresponding aryl diamine in-situ. In this way, the use of a diamine as an explicitly added reactant was avoided. The di-isocyanate used was 4,4'-diphenylmethane di-isocyanate, more commonly known as MDI.

MDI is a solid at room temperature. Large batches of such polyurea grease required bulk storage of MDI in its molten form. The melting point of MDI is about 40 C (104 F). Like all di-isocyanates, MDI is highly toxic. To avoid bulk storage of MDI at elevated temperature, the decision was made to use a modified material based on MDI. That modified material was a liquid at room temperature. The chemistry of that modified material is provided below in Figure 1.

FIGURE 1



The structure of MDI is provided in the upper left corner of Figure 1. When molten MDI is heated to a sufficient temperature, it irreversibly reacts with itself to eliminate carbon dioxide and form a carbodiimide "dimer". This is shown as the top reaction in Figure 1. As the carbodiimide forms, it further reacts with yet unreacted MDI. This reaction proceeds by a concerted pericyclic mechanism known as a 1,2-cycloaddition reaction. The product formed by this reaction can be considered a cyclic trimer (minus one molecule of carbon dioxide). This is shown as the bottom reaction of Figure 1. The reaction to form the cyclic trimer is an equilibrium reaction. At 25 C, the position of the equilibrium is far to the right. The result is a liquid eutectic mixture containing a small but significant amount (20 wt%) of cyclic trimer, virtually no carbodiimide, and about 80 wt% unreacted MDI.

Polyurea greases can be made using this MDI-based eutectic mixture by reaction under pressure with water and fatty amine. The polyurea greases discussed in the previous example, this example, and the following example were made using that material. All such polyurea greases were made in the same 40-pound kettle mentioned in Example 2. After base greases were made, they were additized at about 110 C (230 F). Then the greases were mixed/cooled to 77 C (170 F) and milled at 7,000 psi using a laboratory Gaulin homogenizer.

However, when making polyurea greases using the eutectic MDI-based material, the final reacted thickener will not be *only* polyurea. It will also have some more complex compounds. These more complex compounds are formed because the cyclic trimer is not exactly a trimer since a molecule of carbon dioxide was lost during its formation. The hydrolysis of the cyclic trimer and resulting further reactions with MDI, fatty amine, and yet un-hydrolyzed cyclic trimer will produce more complex products with higher molecular weights, cross linking, and more aromatic character than true polyurea.

When early versions of the CVJ grease as described in the previous Example 5 were made using the MDI-based eutectic product, it was noted that the dropping points were extremely low. Values ranged from 104 C (220 F) to 161 C (321 F). In contrast, polyurea greases should typically have dropping points of at least about 260 C (500 F). Additionally, it was noted that the entire grease was sliding out of the bottom of the dropping point test cup. No oil separation was observed during the test. Additional work with similarly made polyurea greases using naphthenic base oils gave even worse dropping points. Even more significant was the fact that when the CVJ grease was made using pure MDI, excellent dropping points were always obtained.

Naphthenic base oils had a much higher aromatic content than the lightly hydrotreated paraffinic base oils used in the 1980's. The author reasoned that when the MDI-based eutectic blend was used, the complex non-polyurea components formed during the thickener reaction were interacting with the aromatic moieties of the base oil in a manner similar to the "like dissolves like" rule. Such an interaction could be causing the entire grease to slide out of the cup as the temperature is raised.

To test this theory, the author replaced the lower viscosity lightly hydrotreated base oil with an equal amount of a severely hydrotreated white base oil of comparable viscosity. The white oil had an almost zero aromatic content. Since this base oil represented 40% of the total added base oil, the total aromatic content of the base oil blend would be dramatically lowered. When a CVJ grease was made using the white oil, the dropping points were consistently around 260 C (500 F) or higher. When the final formulation as indicated in Table 6 was commercialized, the first 20 full-scale commercial batches were evaluated. Dropping point was consistently above 260 C. It remained at that level for the entire commercial history of the grease.

This use of a paraffinic white base oil to solve a polyurea grease dropping point is a good example of using an existing lubricant component to impart a new, previously undocumented benefit. Accordingly, this is a classic example of Category 2 formulation work.

Example 7 – Extremely Long-Life Sealed-For-Life Wheel Bearing Grease

Another example of Category 2 formulation involved the development of polyurea greases with extremely long high-temperature bearing life. During the 1980's and much of the 1990's, ASTM D3336 run at 178 C (350 F) was the gold standard test for determining a lubricating grease's high temperature bearing life. The two most used polyurea greases for sealed-for-like automotive wheel bearings published bearing life values of 500 hrs and 800 hrs when tested by this method. Both of those values were considered excellent results, and both greases provided excellent performance in automotive wheel bearings and high temperature, high speed electric motor bearings. However, even higher levels of performance were desired.

A series of polyurea greases were made using the eutectic MDI-based material. The equipment and manufacturing method are as previously described. The composition and test results for the first five such greases, Grease 8-1, 8-2, 8-3, 8-4, and 8-5 are provided below in Table 8.

TABLE 8 Extremely Long-Life Sealed-For-Life Wheel Bearing Grease					
	Composition, wt%				
Grease No.	8-1	8-2	8-3	8-4	8-5
Paraffinic Base Oil (850 SUS Oil at 100 F)	45.5	51.4	50.8	49.3	50.8
Paraffinic White Base Oil (350 SUS at 100 F)	30.3	34.2	33.9	32.9	33.9
Polyurea Thickener	12.5	9.5	9.5	11.0	11.0
Tri-Basic Calcium Phosphate	2.0	0.5	0.5	0.8	0.8
Calcium Carbonate	2.0	0.5	0.5	0.8	0.8
Methacrylate Polymer	4.0	0.0	0.0	0.0	0.0
Styrene-Alkylene Copolymer	0.0	1.0	1.0	0.0	0.0
Potassium Triborate Dispersion	1.0	0.0	0.0	0.0	0.0
Zinc Naphthenate	1.0	0.0	0.0	0.0	0.0
Barium Dinonyl Naphthylene Sulfonate (Neutral)	1.0	0.0	0.0	0.0	0.0
Barium Dinonyl Naphthylene Sulfonate/Polyalkenylene Succinate	0.0	1.5	1.5	2.5	0.0
Micronized Sodium Nitrite	0.0	0.0	1.0	0.2	0.2
Borated Alkenyl Amide	0.5	0.0	0.0	0.1	0.1
Mixed Aryl Amine Antioxidant	0.2	0.0	0.0	0.0	0.0
Alkylated Aryl Amine A	0.0	1.5	1.5	2.5	2.5
	Test Results				
Worked Penetration, ASTM D217, 0.1 mm	318	315	304	306	303
Dropping Point, ASTM D2265, C (F)	258 (496)	271 (520)	258 (496)	262 (503)	263 (505)
Bearing Life, ASTM D3336 at 178 C, hr	650	518, 781	875, 1,100+	1,049, 1,237	614, 742

All five greases used the same base oil blend of the CVJ grease of Table 6. To provide a clearer presentation of this data, grease compositions and data are color-coded.

Grease 8-1 used a formulation approach that had some similarity to the CVJ grease of Table 6. The same two solid additives, barium sulfonate, and borated amide are used. A significant level of a methacrylate pour point depressant was added to provide a moderate amount of adherence without significantly reducing the low temperature mobility. The zinc naphthenate was a supplemental rust inhibitor, and the potassium triborate dispersion provided additional antiwear. Note that Grease 8-1 was run only once on ASTM D3336. All other greases in this example were run in duplicate.

The ASTM D3336 bearing life of Grease 8-1 was within the range previously stated as being typical for good high temperature grease. This is significant since at the time of this work it was thought that such levels (4.0%) of inorganic solids would be detrimental to high temperature bearing life. Clearly, in Grease 8-1 this was not the case.

Greases 8-2 and 8-3 had significant changes in their formulation compared to Grease 8-1. Much lower levels of the two inorganic solids were used. The zinc naphthenate, potassium triborate, barium sulfonate, and borated amide were eliminated. The barium sulfonate was replaced by a barium sulfonate/succinate additive marketed as a rust inhibitor that did not decrease high temperature performance. The methacrylate polymer of Grease 1 was replaced with a much lower amount of a styrene-alkylene copolymer. Finally, the mixed aryl amine antioxidant was replaced with a specific alkylated aryl amine antioxidant.

The only difference between Greases 8-2 and 8-3 was the inclusion of 1.0% sodium nitrate in Grease 8-3. The sodium nitrite had been micronized by the supplier to provide a mean particle size of about 1 micron. Sodium nitrate was a well-known rust inhibitor at the time this work was done.

As can be seen, the ASTM 3336 bearing life of Grease 8-2 was not significantly different from Grease 8-1. Thus all the formulation changes of Grease 8-2 relative to Grease 8-1 did not significantly that test result. However, the ASTM D3336 bearing life of Grease 8-3 was significantly improved compared to Grease 8-2 or 8-1. Since the only compositional difference between Greases 8-2 and 8-3 was the inclusion of 1.0 wt% sodium nitrite in Grease 8-3, that difference had to be the reason for the improved bearing life.

It was also noted that Greases 8-2 and 8-3 had a very glassy smooth texture that was absent in Grease 8-1. This visual difference was extremely obvious. Also, by comparing the polyurea content and penetration values of Greases 8-2 and 8-3 with Grease 8-1, it is apparent that Greases 8-2 and 8-3 had a significantly better thickener yield. One possible reason for this could be the styrene-alkylene copolymer used in Greases 8-2 and 8-3. Other work done by the author has shown that this polymer can improve thickener yield by slowing the drop of the cone during the penetration test. The methacrylate copolymer in Grease 8-1 does not have this property to the same extent.

Greases 8-4 and 8-5 had some changes in their formulation compared to Greases 8-2 and 8-3. The level of the two inorganic solids was increased from 0.5 wt% to 0.8 wt%. The antioxidant level was increased, and the sodium nitrite level was dropped from 1.0 wt% to 0.2 wt%. The styrene-alkylene copolymer was not added. Finally, a small amount of the borated amide was added. The only difference between Greases 8-4 and 8-5 was that Grease 8-5 did not contain the barium sulfonate/succinate.

Grease 8-4 had a significantly improved ASTM D3336 bearing life compared to Grease 8-5. Also, Greases 8-4 and 8-3 (containing both the barium sulfonate/succinate and sodium nitrite) had improved bearing life compared to Greases 8-5, 8-2, or 8-1. These results explain why Grease 8-3 had an improved ASTM D3336 bearing life compared to Greases 8-2 (no sodium nitrite) or Grease 8-5 (no barium sulfonate/succinate). One might be tempted to attribute the improved ASTM D3336 bearing life of Grease 8-4 to its significantly increased antioxidant level. But if that was true, then Grease 8-5 would be expected to have a similar improved bearing life since it also has the same increased antioxidant level. Likewise, the styrene-alkylene copolymer cannot be the cause of the improved ASTM D3336 bearing life for similar reasons. The only explanation that is consistent with all the ASTM D3336 bearing life test results of Table 8 is a very strong cooperative, perhaps synergistic, effect of *both* the barium sulfonate/succinate and sodium nitrite on ASTM D33236 bearing life.

Another interesting result is that the very glassy smooth texture of Grease 8-3 was also observed in Grease 8-4. This effect was not observed in Greases 8-1, 8-2, or 8-5. Obviously, the barium sulfonate/succinate is the cause of this extremely smooth texture. Since Grease 8-1 used the same barium sulfonate that was part of the barium sulfonate/succinate, the barium succinate component must be the cause of the obvious visual difference when it was present. Alkenylene succinate salts are well-known as detergent/dispersants. The succinate component of the barium sulfonate/succinate may be more effectively dispersing the polyurea thickener and inorganic solid additive, thereby improving thickener yield and imparting a glassy smooth appearance.

Two more greases were made to determine if other metal sulfonate/succinate additives, when combined with sodium nitrate, would provide extremely high ASTM D3336 bearing lives. The compositions and test results of these two greases are provided below in Table 9.

TABLE 9 Extremely Long-Life Sealed-For-Life Wheel Bearing Grease		
	Composition, wt%	
Grease No.	9-1	9-2
Paraffinic Base Oil (850 SUS Oil at 100 F)	50.0	50.0
Paraffinic White Base Oil (350 SUS at 100 F)	33.3	33.3
Polyurea Thickener	10.0	10.0
Tri-Basic Calcium Phosphate	0.9	0.9
Calcium Carbonate	0.9	0.9
Styrene-Alkylene Copolymer	0.6	0.6
Magnesium Dinonyl Naphthylene Sulfonate/Polyalkenylene Succinate	1.8	0.0
Calcium Dinonyl Naphthylene Sulfonate/Polyalkenylene Succinate	0.0	1.8
Micronized Sodium Nitrite	0.3	0.3
Borated Alkenyl Amide	0.1	0.1
Alkylated Aryl Amine B	2.2	2.2
	Test Results	
Worked Penetration, ASTM D217, 0.1 mm	337	323
Dropping Point, ASTM D2265, C (F)	266 (511)	261 (502)
Bearing Life, ASTM D3336 at 178 C, hr	1,640 1,464	1,594 1,864

Greases 9-1 and 9-2 were made using the same equipment and manufacturing process as the previous greases of this example. Grease 9-1 used a magnesium sulfonate/succinate; Grease 9-2 used a calcium sulfonate/succinate. Other compositional aspects of the two greases were the same. As can be seen, both greases gave ASTM D3336 bearing life values as good as or better than the highest value in Table 8.

In retrospect, the action of sodium nitrite to improve high temperature bearing life may be related to the fact that sodium nitrite is an odd electron compound. As such, it might be acting as a carbon free radical trap where antioxidants would be useless. However, the use of a combination of metal dinonyl naphthenate sulfonate/alkenyl succinate and sodium nitrate in polyurea grease to achieve extremely long ASTM D3336 bearing life values was not taught anywhere in the open literature at the time this work was done. Likewise, it was not anticipated by any published literature. The polyurea compositional technology summarized in this example was covered in a U.S. patent that issued in 1993 [12] and is another example of Category 2 formulation.

Example 8 – Calcium/Magnesium Sulfonate Complex Greases

In a paper published in 2021, a new method was described to make lithium complex greases by initial inclusion of a very small amount of overbased magnesium sulfonate [13]. This new method and the related lithium complex greases required much less lithium hydroxide when making an NLGI No. 2 grease while maintaining or improving the test properties associated with such greases. This new technology had already been covered by a U.S. patent [14] when the paper was first presented. Such formulation work is clearly an example of Category 2 since overbased magnesium sulfonates had not previously been shown to provide such improvements in the cost/performance properties of lithium complex greases. Details of this work will not be covered here since it was documented in the aforementioned paper and patent.

However, the use of overbased magnesium sulfonates has been the defining aspect for the recent development of another entirely new class of lubricating greases: calcium/magnesium sulfonate complex (Ca/Mg Sulf-X) greases. The landscape of Ca/Mg Sulf-X grease covers a wide range of compositions, each with its own unique set of beneficial properties. To fully discuss all such greases would require a series of papers. This example provides a brief overview of just one specific type of Ca/Mg Sulf-X grease: extremely rheopectic Ca/Mg Sulf-X grease.

Except when otherwise noted, all Ca/Mg Sulf-X greases discussed in this example were made in the laboratory using a tilt-head stand-style kitchen mixer with a planetary stirrer and electric heating mantle. All such greases had the same composition as provided below in Table 10.

Table 10
Calcium/Magnesium Sulfonate Complex Grease

Chemical Component Name	%(wt)
Solvent Neutral Group I Base Oil (113 cSt @ 40 C)	47.6
Overbased Calcium Sulfonate (400 TBN)	32.0
Overbased Magnesium Sulfonate (400 TBN)	3.3
Dodecylbenzene Sulfonic Acid (DDBSA)	3.2
Powdered Calcium Carbonate	7.7
Alkylene Glycol	1.6
12-Hydroxystearic acid (12HSA)	2.5
Glacial Acetic Acid (HOAc)	0.4
Phosphoric acid (75 wt% aqueous solution)	1.7

The procedure for making the first Ca/Mg Sulf-X grease described in this example was as follows:

1. Add 70.0 wt% of the base oil, all the overbased magnesium sulfonate (OMgS), and all the overbased calcium sulfonate (OCaS) to the mixer and mix for about 20 minutes.
2. Add all the DDBSA and mix for at least 20 minutes.
3. Add all the powdered calcium carbonate and mix for at least 20 minutes.
4. Add 33% of the 12HSA and 35% of the HOAc. Mix for at least 20 minutes.
5. Add 4.0% water based on the final weight of the batch. Mix and begin heating to 88 C (190 F). Adjust the rheostat controlling the heating mantle so that it takes about 1 hour to reach 88 C.
6. Once 88 C is reached, hold at that temperature for 30 minutes. Then add all the alkylene glycol.
7. Once FTIR confirms that the conversion process has finished, add the remaining 12HSA and HOAc. Allow the grease to mix for at least 20 minutes.
8. Slowly add the phosphoric acid. Once visible reaction has ceased, begin heating to 199 C (390 F). Adjust the rheostat controlling the heating mantle so that it takes about 2 hours to reach 199 C.
9. Once 199 C is reached, remove the heating mantle and continue mixing while the batch cools.
10. Once the batch reaches 77 C (170 F), remove a small portion of it, and give it three passes through a three roll mill with both gaps set at 0.03 mm (0.001 inch).
11. Record the unworked penetration (ASTM D1403) of the milled portion as an estimate of the consistency of the grease before addition of the remaining base oil,
12. Return all the milled grease to the mixer, add remaining base oil and mix at 77 C for 20 minutes. Then mill the entire batch by the same procedure as in step 10.

It should be noted that the above procedure uses a “delayed addition of non-aqueous converting agent technique” that was disclosed a few years ago [15, 16].

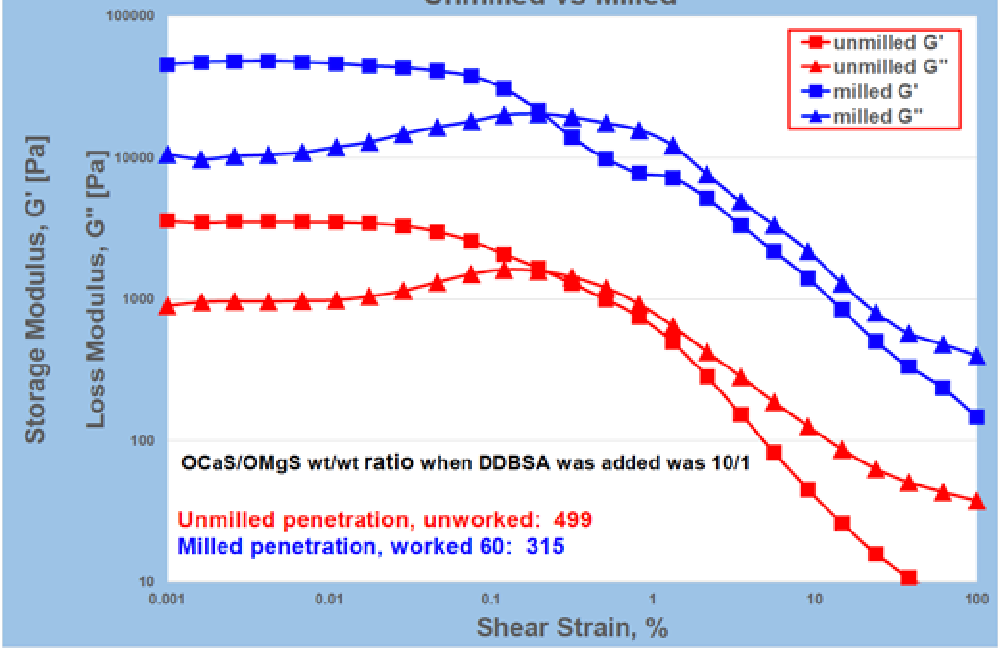
When the first laboratory batch of the Table 10 grease was made by the above procedure, the initial visual progression appeared typical for a calcium sulfonate complex grease. However, as the batch approached 88 C, it began to visibly thicken. During the 30-minute holding delay at 88 C (step 6), the batch continued to thicken. FTIR showed that conversion was well underway even though the primary non-aqueous converting agent (alkylene glycol) had not yet been added. In less than 10 minutes after the glycol was added, FTIR indicated that the conversion process was finished. However, the batch did not continue to thicken with the addition of the glycol. Instead, it began to soften. This softening continued as the post-conversion acids were added, and as heating to top temperature began. Well before reaching 199 C, the batch appeared completely fluid and had lost all visual appearance of a grease. Upon cooling, the fluid appearance remained. A grease structure was not visually apparent. However, when a small portion of the batch was milled (step 10), the unworked penetration (step 11) was 189. The milling had changed a visually fluid product to an extremely hard grease.

When the remaining 30% of the total required base oil was added and the entire batch was milled, it had a worked 60 stroke penetration of 290 (ASTM D217). The dropping point was 325 C (617 F).

The extremely rheopectic behavior of this batch was unlike anything previously reported in the calcium sulfonate-based grease literature. This grease was made again several more times to ensure that the highly unusual results were repeatable. In each case, essentially the same result was obtained. Note that in these greases the ratio of OCaS to OMgS when the DDBSA was added was 10/1.

One such repeat batch was evaluated by oscillatory rheometry using a controlled shear strain sweep at 25 C. Oscillatory rheometry is a much more revealing test for grease structure than penetration [17]. Results are provided in Figure 2.

Figure 2: Extreme Rheopectic Ca/Mg Sulf-X Grease
Unmilled vs Milled



As can be seen, even though the unmilled grease appeared fully fluid, it still had a grease structure that was detectable by the shear strain sweep test. For typical calcium sulfonate greases (with no OMgS), the G' (storage modulus) and G" (loss modulus) curves for the unmilled and milled greases will overlap. The significant gap between the G' and G" curves of the unmilled and milled greases in Figure 2 is an indication of the extreme rheopectic behavior of the unmilled grease. (Remember that both axes are logarithmic.)

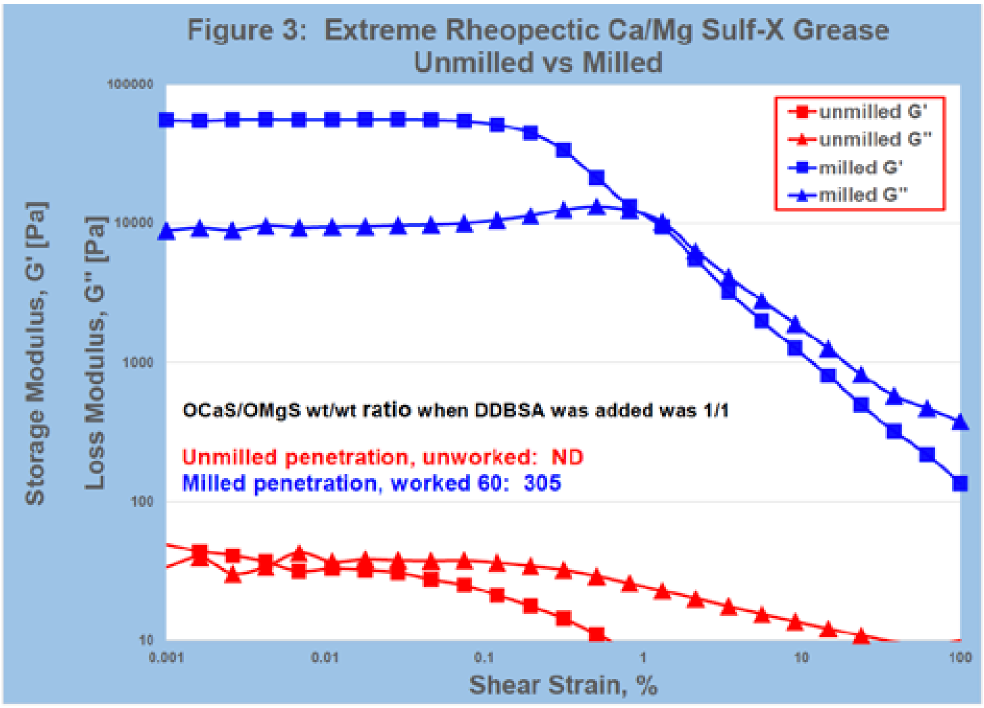
Another grease was made with essentially the same composition of Table 10. However, one change was made in the step-by-step procedure by which it was prepared. The OMgS was not added with the initial base oil and OCaS. Instead, the DDBSA was added to the blend of only the initial base oil and OCaS and allowed to mix for at least 20 minutes. Then OMgS was added to the batch. The remaining process steps were the same as the previous extremely rheopectic batches. When this batch was made, it progressed identical to the previous batches until the glycol was added. However, when the glycol was added, the batch continued to thicken. Upon post-conversion steps and heating to top temperature, the batch did not significantly thin out like the previous batches did. Upon cooling, the batch retained its grease consistency. Milling hardened the batch by about 40 points, which is typical for calcium sulfonate complex greases.

This observation's significance cannot be understated. Simply by changing the order of addition of two components – the OMgS and the DDBSA – a huge change in grease rheology resulted. When the DDBSA was added with both OCaS and OMgS being present, extreme rheopectic behavior resulted. When the DDBSA was added with only the OCaS being present (and with the OMgS being added later), normal calcium sulfonate complex grease rheology resulted.

DDBSA is a strong organic acid, orders of magnitude stronger than any of the other acids used in the Table 10 formulation. The huge difference in rheology as described above is most likely due to the formation of magnesium dodecyl benzene sulfonate *and* its incorporation into the final grease structure as it forms.

To test this hypothesis, another Table 10 grease was made. However, in step 1, most of the required OCaS was not added. Instead, the amount of OCaS added in step 1 was the same as the full amount of OMgS that was added. Then the DDBSA was added as per step 2. After the minimum 20 minutes of mixing, the remaining OCaS was added. From that point on, the procedure to make the grease was the same as the first Table 10 greases where extreme rheopectic behavior was observed. Note that this change meant that when the DDBSA was added, the ratio of OCaS to OMgS was 1/1 instead of 10/1. This change should increase the formation of magnesium dodecyl benzene sulfonate when the DDBSA is added. If the formation of magnesium dodecyl benzene sulfonate is a cause for the extreme rheopectic behavior, this change in the ratio of OCaS to OMgS when the DDBSA is added should increase the rheopectic behavior.

The final unmilled grease was too fluid to test by penetrometer. The milled grease had a worked 60 penetration of 305. The dropping point was greater than 343 C (650 F). The grease was evaluated by oscillatory rheometry using a controlled shear strain sweep at 25 C. Results are given below in Figure 3.



By comparing Figures 2 and 3, three things are most noteworthy:

1. The G' and G" curves of the milled greases are nearly identical regarding the actual initial values of G' and G".
2. The viscoelastic portion of the G' curve for the Figure 3 grease is more stable than the corresponding portion of the G' curve for the Figure 2 grease.
3. The primary result of increasing the concentration of the OMgS relative to the OCaS concentration when the DDBSA was added was to further destabilize the unmilled grease, thereby increasing the rheopecticity of the Figure 3 grease relative to the Figure 2 grease.

These results are consistent with the formation and incorporation of magnesium dodecyl benzene sulfonate into the grease structure as being a trigger for the extreme rheopectic behavior of these Ca/Mg Sulf-X greases.

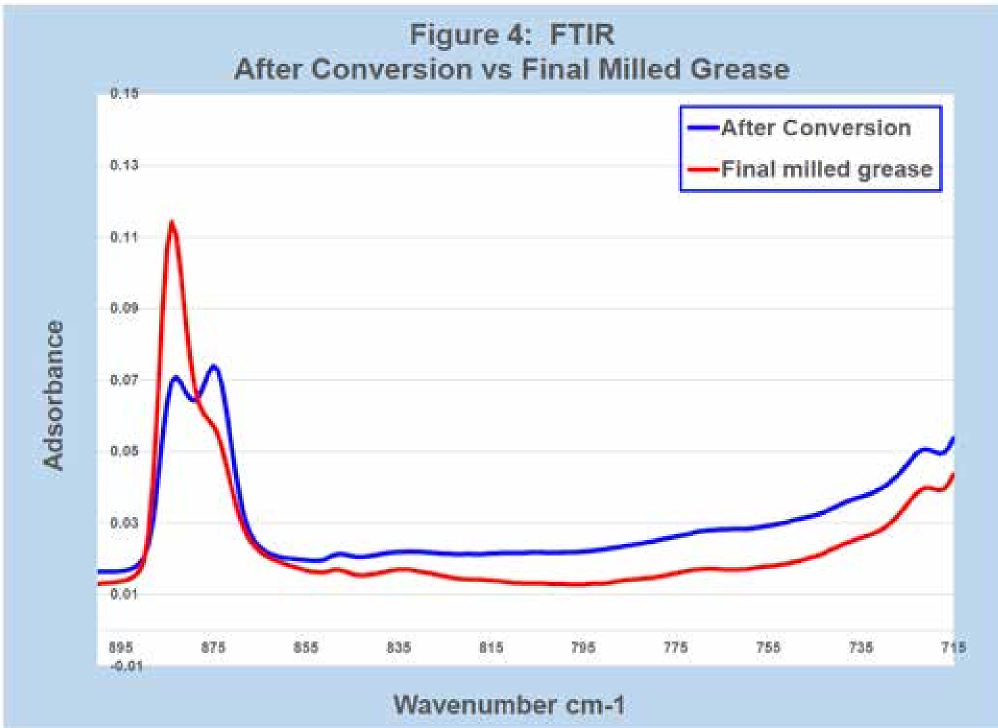
Greases with the compositions similar to that of Table 10 have been made in a jacketed stainless steel, 1,000-pound grease kettle with double action mixing paddles. A hot oil/cold oil heat transfer fluid was used for heating and cooling. Milling was accomplished using a colloid mill that was part of the kettle system. The unmilled and milled penetration values for three such batches are provided below in Table 11.

TABLE 11
Kettle Batches of Ca/Mg Sulf-X Grease

Kettle Batch Number	1	2	3
Unmilled Grease			
Unworked Penetration, ASTM D217, 0.1 mm	ND	557	445
Milled Grease			
Unworked Penetration, ASTM D217, 0.1 mm	293	305	286
Worked 60 Stroke Penetration, ASTM D 217, 0.1 mm	303	307	294
P0 (Unmilled) - P60 (Milled)	ND	250	151

The results provided in Table 11 show that the extremely unusual rheopectic behavior of the laboratory greases can be duplicated in actual grease kettles. Note that Grease 1 was considered too fluid to evaluate its penetration. It gave controlled shear strain sweep oscillatory rheometry essentially identical to Figure 3.

Finally, the FTIR data from Grease 1 of Table 11 is provided below in Figure 4.



The curve in blue represents the grease once the conversion process was complete as indicated by no further progress. In typical calcium sulfonate grease production, such a conversion spectra would be indicative of poor conversion. But this is not the case with the extremely rheopectic Ca/Mg Sulf-X greases as discussed in this example. During both the numerous laboratory and kettle production batches of such greases, this doublet conversion peak pattern was the norm. This doublet FTIR conversion spectra are a characteristic feature of Ca/Mg Sulf-X greases that exhibit extremely rheopectic behavior. X-ray diffraction confirmed no vaterite to be present. Only calcite was detected.

Figure 4 also shows that once milled, the final grease had a more normal conversion spectra within the conversion area near 882 cm^{-1}. The resolved intermediate peak at about 877 cm^{-1} in the "after conversion" spectra was reduced to a prominent shoulder in the "final milled" spectra.

The doublet in the "after conversion" spectra is best interpreted as indicating that the calcite in the final grease is surrounded by two different chemical environments, each differently affecting the asymmetrical stretching mode that is being measured by that peak. A bimodal calcite particle size distribution might also explain the doublet peak. However, all such unmilled greases appeared smooth with no visible grainy texture. Regardless, larger particles of calcite would not have as many other chemical species surrounding it per mass of calcite. Larger particles have a lower surface area/mass ratio. Being surrounded by less of other chemical species per mass of calcite would amount to the calcite being surrounded by a different chemical environment. This would be expected to change how the calcite stretching mode is altered relative to much smaller calcite particles.

Also, the final milled grease had the additional 30% of the total base oil present. That additional 30% base oil was not present when the "after conversion" spectra was taken. If that additional oil had *not* been present in the "final milled" sample, the height of its peak at 882 cm^{-1} would have been much higher. The "after conversion" spectra would have appeared even smaller by comparison. Thus, the "after conversion" spectra represent a collapsed structure that is reconstituted upon milling. It is important to remember that milling does not create more calcite. The amount of calcite in the "final milled" grease will be identical to the amount in the "after conversion" grease.

Based on the information provided in the data of this example, the magnesium dodecyl benzene sulfonate formed during the addition of DDBSA, and subsequent heating apparently affects the species that surround the calcite as it is formed and dispersed during the conversion process. Different calcite-related structures are formed that are not normally formed when calcium sulfonate-based greases are properly made (without OMgS).

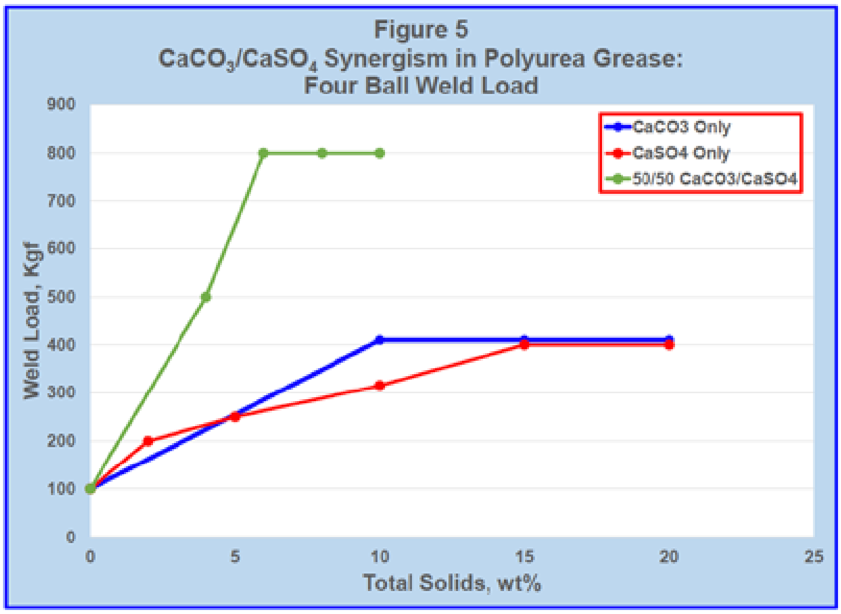
In summary, Ca/Mg Sulf-X greases have different FTIR spectral data and a unique and highly unusual rheometry that can vary between both extremes (depending on how they are made). There are other interesting and unique properties of these greases that have been determined that will require additional papers to fully discuss. (Such additional papers are being planned, and this example serves as a foundation for those papers.) For all these reasons, Ca/Mg Sulf-X greases should be properly regarded not as simply calcium sulfonate complex greases with OMgS as an additive. Instead, Ca/Mg Sulf-X greases comprise an entirely new thickener category.

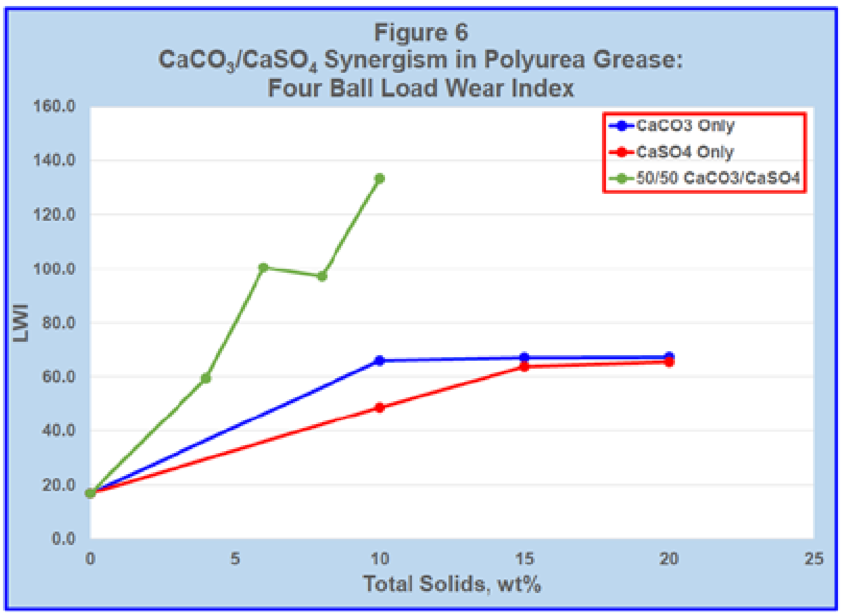
Obviously, the use of OMgS to generate the unique and wide-ranging properties of Ca/Mg Sulf-X greases was not indicated by anything in the existing published literature at the time of its first discovery. Ca/Mg Sulf-X greases are now covered by several U.S. patents [18-23]. The development of such greases is another clear example of Category 2 formulation work.

FORMULATION CATEGORY 3: *Using at least one component that has not been previously used in lubricating greases, but that nonetheless provides a beneficial result, either by itself or in combination with other components.*

Example 9 – Calcium Carbonate and Anhydrous Calcium Sulfate Synergy

As the polyurea grease work described in the previous Examples 5-7 was being done, various combinations of powdered solids were also being investigated as potentially useful additives. The combination of powdered calcium carbonate and anhydrous calcium sulfate exhibited the highest level of synergistic performance as EP/AW additives. Figures 5 and 6, below, show the response by the ASTM D2596 Four Ball EP test for weld load and load wear index, respectively.





All greases represented in Figures 5 and 6 were made from a common polyurea base grease. The polyurea base grease was made from the same eutectic MDI-based material that was described in Example 6. Each of the greases were prepared by adding the various combinations of the two solids to portions of the polyurea base grease. Then each additized grease was mixed and heated to about 77 C (170 F). Finally, each grease was given three passes through a three-roll mill with both gaps set at 0.03 mm (0.001 inch).

As Figures 5 and 6 clearly show, the combination of calcium carbonate and anhydrous calcium sulfate provides a very large synergistic response when evaluated by the Four Ball EP test. Both weld load and load wear index (LWI) are positively affected by this synergism. The grease with 3 wt% of each of the two solids gave weld loads and LWI values higher than a grease with 20 wt% of either solid alone.

Additional work evaluating the use of the calcium carbonate/anhydrous calcium sulfate combination in polyurea grease demonstrated excellent results when evaluated by ASTM D2266 Four Ball Wear and by Optimol SRV stepload and wear tests. Frictional heat generated during prolonged Four Ball runs was much less than what was generated when typical sulfur-phosphorus additive packages were used. This combination of the two solids in polyurea greases also inhibited oil bleed by the cone sieve method even when borated additives were not used. All this information was documented in the U.S. patent that covered this technology [24].

When this work was done, powdered calcium carbonate was recognized as an EP/AW additive in greases. However, anhydrous calcium sulfate was not. Additionally, the other beneficial properties imparted by this use of anhydrous calcium sulfate were not documented or anticipated by the existing literature when this work was originally done. This is an example of Category 3 formulation work.

Example 10 – Carbon Nanotubes as a Grease Thickener

Within the last 20 years, the author collaborated with Dr. Haiping Hong to develop and document greases thickened by carbon nanotubes for the first time. This technology has been described in four papers [25-28] and one U.S. patent [29]. Accordingly, the details of this work will not be presented in this paper. However, this work clearly falls within Category 3 formulation work.

Conclusions

The work described above supports the following conclusions:

1. All lubricating grease formulation work falls within one or more of the three formulation categories as described.
2. Some formulation work will have aspects of more than one category. This is especially true for formulation work where aspects of both Category 1 and 2 are present.
3. Most lubricating grease formulation work will mostly, if not entirely, fall into Category 1.
4. Even when only Category 1 formulations are being done, the results can be extremely significant.
5. Category 2 formulation work represents perhaps the most frequent means to provide new paths of innovation in the chemistry and technology of lubricating greases. Such paths can sometimes yield important and even ground-breaking advances in lubricating grease performance potential.
6. Category 3 work is the least frequently encountered of the three formulation categories since it requires the discovery of a grease component not previously documented or anticipated by the established literature. However, when such discoveries are made, the results can be noteworthy.

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NLGI Interviews Dr. Maureen Hunter
Vice President and Global Business Manager
Lubricant Additives Division King Industries, Inc.
Norwalk, CT and NLGI Board of Directors

By Mary Moon and Raj Shah

Dr. Maureen Hunter
Vice President and Global Business Manager
Lubricant Additives Division
King Industries, Inc.

The historic landing of the Apollo 11 Lunar Module and the first steps taken by an astronaut on the surface of the moon inspired millions of people worldwide. Maureen Hunter was motivated by this inspiration to graduate with a doctorate in Chemical Engineering and pursue a career in the lubricating grease industry. Her journey along a road less travelled has involved a rewarding career at King Industries and many fulfilling hours of volunteer activities on behalf of NLGI and STLE. To learn more about Maureen's favorite chemicals, her experiences as a volunteer, board member, officer and president and the role of pizza in her career, read on!

Education

NLGI: When did you become interested in science? Did a specific person or experience encourage your interest?

MAUREEN: I remember the exact day I became interested in science. It was July 20, 1969 – that famous day in history when Neil Armstrong and "Buzz" Aldrin emerged from the Apollo 11 Lunar Module to become the first men to step on the surface of the moon.

Although I was only a child, I fondly remember the transformative hold it took on me. The house was buzzing with excitement and anticipation. My aunt, uncle, and five cousins from New Jersey were visiting us for the big day. All day long, my cousins, younger sister and brother, and I pretended to be astronauts. Wearing space suits made with layers of clothes and winter coats, we jumped up and down on the beds in an attempt to defy gravity. We kept running outside onto the beach where I grew up in Vermont and looking up into the sky.

What I remember most vividly is the lunar landing. That night, we kids were allowed to stay up late. We all sat around our TV set with "rabbit ear" antennae. The images transmitted from the lunar surface looked ghostly. My father kept jumping up to adjust the rabbit ear antennae, trying to make the picture clearer. I could sense that the adults felt anxious. They hesitated between teaching us the significance of the historic moment and hushing us so that everyone could hear the anchorman, Walter Cronkite, walk us step-by-step through the exciting event.

Just before Neil Armstrong came down the ladder from the Module, you could have heard a pin drop. I looked at my mother. She was praying. As Neil Armstrong stepped from the bottom of that ladder to the surface of the moon, he uttered his famous words, "One small step for [a] man, one giant leap for mankind." We all cheered, and my aunt cried. It was the most powerful moment and display of the human spirit I had ever seen. That's the day I became interested in science.

My mind was really opened to the many career possibilities available to chemical engineers.

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

MAUREEN: Growing up in Burlington, Vermont, the logical choice for college was the University of Vermont (UVM). I really liked math and chemistry, so I entered their Engineering Chemistry program. But at the end of my sophomore year, UVM decided to discontinue this major. Even though I loved going to UVM, I transferred to the State University of New York at Buffalo as a Chemical Engineering major. It was there that my mind was really opened to the many career possibilities available to chemical engineers. I got to know many excellent professors. I made many good friends, and I am still very close to some of them.

NLGI: Please tell us about your first job after getting your bachelor's degree. And what made you decide to go to graduate school?

MAUREEN: After graduating, I worked for three years at Wyeth-Ayerst Laboratories in Rouses Point, New York. It was a great experience. I joined an ambitious, fun-loving group of young professionals in the pharmaceutical industry, one of whom became my husband. I greatly enjoyed my job developing and writing HPLC methods for testing drug formulations, investigational compounds, and raw materials. Working there in a company where many people had doctorates inspired me to go to graduate school.

Maureen in her laboratory in the Chemical Engineering building (Fenske Laboratory) on the day that she graduated with her doctorate from The Pennsylvania State University, December 1992. She is holding a vessel that she used to run the Penn State Microoxidation Test to evaluate the thermal and oxidative stability of oils.

NLGI: Where did you go to graduate school, and with whom did you study?

MAUREEN: I attended the Graduate School of Chemical Engineering at the main campus of The Pennsylvania State University at University Park, Pennsylvania. That is where I entered the lubricant industry by chance, like so many other people that I know in the lube industry.

When interviewing with professors who had open positions in their research groups, I connected with Dr. Elmer Erwin Klaus (professor emeritus) and Dr. John Larry Duda (head of the department) who became my co-advisors in the Tribology Group, and I never looked back. They both became great mentors to me and advised me through both my master's and Ph.D. degrees.

It was during this time that I made close friendships with several students who became colleagues. Dr. Vasu Bala (Tiarco, LLC) and I defended our research and dissertations back-to-back on the same day. Several months later, Dr. Raj Shah (Koehler Instrument Company) defended his dissertation. After graduating from Penn State, I started working for King Industries, Inc.

Maureen and Dr. Vasu Bala (right) with Dr. J. Larry Duda (center) on their graduation day at Penn State (1992)

Dr. Raj Shah and Maureen at the NLGI Annual Meeting in Coeur D'Alene, Idaho (2015)

Career at King Industries, Inc.

NLGI: How did you find your job at King Industries?

MAUREEN: On a snowy night in 1989, I was on my way to attend my first meeting of the local section of STLE in Pittsburgh. I was attending the meeting because my advisor in grad school, Dr. Klaus, had submitted my name for the Pittsburgh Section STLE scholarship that year. Little did I know that the meeting would set my life's course into motion.

At that STLE meeting, I met Thomas Pane, who served on the NLGI Executive Committee for many years. During dinner, he talked a lot about King Industries, and he took great pride in working there. I gave my presentation and received my $500 check (not bad for a graduate student whose yearly stipend was $9000). Then Tom gave me his business card and told me, "Call us when you're done with school and looking for a job." I decided to do just that.

In January 2024, I will have worked at King Industries for 31 years.

> ***I entered the lubricant industry by chance, like so many other people that I know in the lube industry.***

NLGI: Please tell us a little about King Industries.

MAUREEN: For more than 90 years, King Industries, Inc. has been owned and operated by the King family in Norwalk, Connecticut. King Industries designs and manufactures additives for many companies, from small to large, throughout the world that make their own branded products including industrial and automotive greases and oils, rust preventives, paints, coatings and rubber goods.

NLGI: Please tell us a little about the products and technologies in the Lubricant Additives Division at King Industries.

MAUREEN: King Industries offers a robust product portfolio, and we pride ourselves on our innovative chemistries and technical support. We work closely with our customers to implement our visionary approach to developing multifunctional additives to meet demanding industrial challenges.

In the Lubricant Additives Division, we manufacture and sell rust inhibitors, antiwear additives, extreme pressure additives, friction modifiers, antioxidants, yellow metal deactivators, additive packages and alkylated naphthalenes. We offer a broad range of high-performance rust inhibitors based on unique sulfonate and non-sulfonate chemistries with advantageous secondary features. These advantages include improved lubricant demulsibility, hydrolytic stability, wet filtration, antiwear performance, oxidation resistance, dispersancy, detergency, acid neutralization and color stability.

We have also designed specialized additive blends and packages offering alternatives to individual additive formulations. We have a product line specifically designed to provide outstanding rust protection to greases exposed to severe salt water conditions. And we have the industry's widest viscosity range of alkylated naphthalene synthetic base oils. Many of our additives and alkylated naphthalenes have HX-1 food grade approvals and are on the Lubricant Substance Classification (LuSC) list as suitable for use in products that bear the European Ecolabel as environmentally acceptable.

NLGI: What is your current role?

MAUREEN: Since October 2021, I have been the Vice President and Global Business Manager for the Lubricant Additives Division. It is a busy and fulfilling position that I find very rewarding. Previously, I was the Technical Service Manager for 26 years, and when I joined the Company, I was an Applications Specialist for 3 years. I am very grateful to Mr. King for the years of support and opportunities he has given to me in both my career and industrial society positions, especially with NLGI and STLE. I truly believe that I could not have found a better company or person to work for.

Maureen working on the SRV at King Industries (2007)

Science and Technology

NLGI: You've recently given many presentations on alkylated naphthalenes. What are they, and why are they important?

MAUREEN: I have always enjoyed writing and speaking about our products. Over the years, I have written many papers and book chapters and given many industry presentations on all our product lines.

King Industries, Inc., Norwalk, Connecticut

At King Industries, the core technology and expertise involve the alkylation of naphthalene. My favorite products are our unique sulfonates and alkylated naphthalene base oils. Alkylated naphthalenes are high performance Group V base oils that are primarily used as co-base oils. That is, they are added to other synthetics or Groups II and III base stocks.

Alkylated naphthalenes enhance thermal and thermo-oxidative stability in blends with other base oils, improve their additive response and extend the operating life of high-performance industrial and automotive oils and greases.

Maureen giving a presentation (2015)

NLGI: How did you learn about grease formulations and testing?

MAUREEN: At the start of my career, I attended the basic and advanced lubricating grease courses at NLGI annual meetings. Taking those education courses along with attending presentations of technical papers at NLGI annual meetings for many years has served me well.

But the best ways to learn about grease formulations and testing are working hands-on in the lab and having an outstanding mentor. I am grateful to have had both experiences at King. King industries has an extensive grease testing lab with equipment ranging all the way from a cone penetration device to a rig for FE8 testing of grease tribology in roller and ball bearings.

> ***The best way to learn about grease formulations and testing is working hands-on in the lab.***

At King, I had an outstanding mentor, my colleague Robert Baker, who was both a technical mentor and industrial society mentor as the 2004-2005 STLE President. Bob worked at King for many years until his retirement. Together, we formulated several synergistic rust inhibitor systems specifically for greases used under severe conditions.

Maureen and Bob Baker at King Industries

NLGI: Do you have favorite chemistries for greases?

MAUREEN: One of the challenges of working with greases is that their performance is very dependent on the specific thickener. Even when greases are formulated with the same thickener type, their performance can differ from one company to another. This has allowed us to work closely with many grease manufacturers and formulators to customize solutions specific to their grease products. We offer a full product line of additives and alkylated naphthalenes for greases formulated with various thickener types, i.e., soaps and inorganic thickeners.

My favorite additives for greases are our unique complexes that combine both sulfonate and carboxylate chemistries for applications under normal requirements and our unique synergistic blends for severe conditions. My other favorite products for greases are our alkylated naphthalenes, especially our unique higher viscosity products. Over the years, we have generated a lot of data on these products for greases and shared them in our many papers and presentations.


ALKYLATED NAPHTHALENES

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Greases are used in numerous applications requiring a broad range of operating temperatures and special requirements, and the base oil used can impart improved characteristics to the grease. Alkylated naphthalenes are a unique class of synthetic fluids with outstanding thermo-oxidative and hydrolytic stability, low volatility, and good solubility characteristics. The flexibility of this technology to achieve a balance of physical and chemical properties will be discussed. Physical property and performance test data will be presented for both liquid lubricants and greases with alkylated naphthalenes as the sole base fluid and as a modifier for other primary base fluids. Grease work has focused on the advantages of using alkylated naphthalenes to reduce the amount of thickener, improve grease clarity and smoothness, and impart significant resistance to oxidation.

INTRODUCTION

Alkylated naphthalenes are high performance synthetic fluids that were first developed during WWII but were never commercialized. In the last 20 years, with advanced processing technology and raw material availability, alkylated naphthalenes have emerged as cost-competitive, high-performance basestocks. These unique synthetic fluids were first used in engine oils and more recently in all types of industrial lubricants and greases. They are primarily used as base oil modifiers with other synthetic base oils or Group II and III mineral oils. They are used to enhance the thermal and oxidative stability and/or additive performance to extend the lifetime of high performance lubricants. The basic structure of an alkylated naphthalene is shown in figure 1, where the core consists of two fused six-membered rings with the alkyl groups attached. It is because of the ability of this electron-rich conjugated naphthalene ring to absorb energy, resonate, and disperse energy, much like antioxidants do, that alkylated naphthalenes inherently have excellent thermo-oxidative stability. To make alkylated naphthalenes, naphthalene is reacted with an alkylating agent (alcohols, alkyl halides, or olefins) in the presence of an acid catalyst and this produces a mixture of alkylated naphthalenes having different numbers of alkyl groups on the naphthalene ring. The reaction is shown in figure 2.

The physical properties of alkylated naphthalenes depend on:

- The number of carbons in the alkyl group, which is controlled by raw material selection.
- The degree of branching of the alkyl group, which is controlled by raw material selection.
- The number of alkyl groups on the naphthalene ring, which is controlled by chemical processing.

Figure 3 shows by gas chromatograph the distribution of an alkylated naphthalene mixture showing MAN (mono alkylated naphthalene), DAN (di alkylated naphthalene).

The NLGI Spokesman, May/June 2015

STLE Experience

NLGI: You served as President of STLE in 2014-2015. Please tell us a little about what it entailed and what you learned as President.

MAUREEN: As the President of STLE, I travelled from one time zone to another and learned to stand tall and simply enjoy the experience. I had opportunities to meet CEOs and presidents of large international companies and organizations as well as high-ranking government officials. This is not a world where most of us live.

Near the end of my presidency, I told my 10-year-old daughter that I couldn't watch a video with her because I needed to write letters to the Minister of Energy in Trinidad, who I'd met on a recent trip, and to a NASA astronaut. She laughed, then paused and said "Really?"

When serving as President of STLE, I learned about the true importance of the lubricant industry. We need outspoken advocates for our professions as tribologists and lubrication engineers. I became aware that we need to let the world know who we are. I realized the benefit of working together with other international organizations to share ideas and help solve energy and tribological issues that can improve our lives, the environment and the global economy.

> ***We need outspoken advocates for our professions as tribologists and l ubrication engineers.***

NLGI: How did you come to the role of being the President of STLE.

MAUREEN: I had absolutely no idea what I was getting myself into when I joined STLE in 1986 as a student. I have to admit that I initially joined STLE for the fun of attending a student pizza party! I enjoyed being a student member of STLE while earning a master's degree and a doctorate studying tribology in the chemical engineering department at Penn State.

When I joined King Industries in January 1993, I became very involved with STLE. Through the years, I have been on many committees and held many positions on both the local and national levels, including roles at Lubrication Engineering (the *journal that preceded Tribology and Lubrication Technology* or *TLT*) as an author and Associate Editor (1997-2003), Northeast/Mid-Atlantic Regional Vice President on the Board of Directors (2001-2005), and Editor of TLT (2007-2009). During 2011-2016, I rose through the ranks of the Executive Committee of STLE and served as President (2014-2015). Since 2016, I have been the STLE STEM Ambassador and spearheaded efforts to make students more aware of opportunities in the fields of tribology and lubrication.

NLGI

NLGI: Have you attended many NLGI Meetings?

MAUREEN: In 1993, I joined King Industries and attended my first NLGI annual meeting. Since 1993, I have attended all but one NLGI annual meeting. King Industries is very supportive of NLGI. Through the years, many colleagues and I have attended annual meetings, taken education courses, instructed at the hands-on training course, presented papers, published technical papers in The Spokesman, sponsored events and been Board members.

Bob Baker and Maureen on the cover of The NLGI Spokesman, March 1999

NLGI: What are your activities as a member of the Board of Directors of NLGI?

MAUREEN: I joined the NLGI Board of Directors in 2017. We are both a strategic and a working Board. I am the Academic Committee Chair and serve on the Governance Committee. Board members are periodically asked to join brainstorming meetings and events. I've participated in new member receptions, a networking event for women in grease, HPM certification and sustainability meetings, planning and site selection for annual meetings and membership recruitment. Being on the NLGI Board has been a wonderful experience for me.

NLGI Board of Directors (2023) Front row (from left to right): Tom Schroeder, Jim Hunt, Anoop Kumar, Wayne Mackwood, Chad Chichester; Second row: David Turner, Ray Zhang, Simona Shafto, Chuck Coe, Joe Kaperick, David Cardy; Third row: Muibat Gbadamosi, John Sander, Maureen Hunter, Dwaine Morris; and Fourth row: Matt McGinnis, Tyler Jark, Pat Walsh

NLGI: What are some of the benefits of being involved with NLGI?

MAUREEN: Colleagues at NLGI member organizations can read The Spokesman and Grease World e-newsletter, attend presentations and education courses at annual meetings, and learn a lot about grease chemistry, additives, manufacturing, testing, applications, selection and specifications, and that's great.

> ***You'll gain skills and experiences you didn't think possible - and have fun while doing it!***

I'd like to encourage colleagues to take a more active role in NLGI. I can guarantee that if you volunteer for NLGI, then you'll gain skills and experiences you didn't think possible - and have fun while doing it! You'll gain experience that will differentiate you from your peers. You'll learn to be successful in strategic planning, creative thinking, public speaking, writing and leading your colleagues into new ventures. Volunteering for NLGI will allow you to connect with high-level industry professionals and academics. You will learn valuable and insightful details about the grease industry, and you'll be better equipped to achieve new and exciting goals in your career.

Becoming more involved with NLGI will help you to uncover what makes you exceptional. You will gain confidence that will enable you to make solid career choices and deliver outstanding value to your company. It's a no-risk proposition that promises high returns.

Maureen at the NLGI Annual Meeting on Coronado Island, California, 2023

Perspectives

NLGI: Is it difficult to balance professional success and family life?

MAUREEN: We have only 24 hours in a day, so most of life is a balancing act. Each of us needs to personally assess the importance of family, work, volunteering, sports, etc. Setting priorities and time management are key to reaching one's goals.

Being a family member (a spouse, mother, daughter and sister) has its challenges and responsibilities indeed. But for me, my family provides support and balances the stresses of everyday life and a hectic workplace environment. I typically leave the house before 8 a.m. and do not return home until 7 p.m., but nightly dinner as a family has always been a priority. Our three children are living on their own now, so eating dinner together is a special time for my husband and me to catch up and share the important events of our daily lives.

The Hunter Family at a garden-themed wedding in Vermont, 2023

NLGI: Do you have a favorite pastime activity?

MAUREEN: One of my all-time favorite things to do is travel in our RV motorcoach. I love visiting friends, touring cities and boondocking off-grid in the wilderness. On a recent adventure, we stopped at a lubritorium in Hampton Beach, New Hampshire. A lubritorium is a room where automobiles are greased and oiled. The word was coined in 1928 from "lubrication" and "auditorium". The ending, -torium, was an overworked trade suffix in the late 1920s.

New Hampshire, 2023

NLGI: Do you have a favorite poem?

MAUREEN:
My favorite poem is "The Road Not Taken" by Robert Frost, which was published in *The Atlantic Monthly* in August 1915 and his collection, *Mountain Interval* (1916). It describes the way I feel about having a wonderful and fulfilling career in the lubricant industry!

The Road Not Taken
by Robert Frost

Two roads diverged in a yellow wood,
And sorry I could not travel both
And be one traveler, long I stood
And looked down one as far as I could
To where it bent in the undergrowth;

Then took the other, as just as fair,
And having perhaps the better claim,
Because it was grassy and wanted wear;
Though as for that the passing there
Had worn them really about the same,

And both that morning equally lay
In leaves no step had trodden black.
Oh, I kept the first for another day!
Yet knowing how way leads on to way,
I doubted if I should ever come back.

I shall be telling this with a sigh
Somewhere ages and ages hence:
Two roads diverged in a wood, and I –
I took the one less traveled by,
And that has made all the difference.

Connecticut, 2023

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 mmmoon@

ix.netcom.com or Raj at rshah@koehlerinstrument.com.

Dr Mary Moon is Technical Editor of *The NLGI Spokesman*. She writes scientific and marketing features published in *Lubes'n'Greases* and *Tribology & Lubrication Technology* magazines, book chapters, specifications, and other literature. She graduated with a Ph.D. in Chemistry from the University of Chicago, an MBA from Rider University (Lawrenceville, NJ), and bachelor's degrees in chemistry and physics from Lafayette College (Easton, PA). Her experience in the lubricant and specialty chemicals industries includes R&D, project management, and applications of lubricants, tribology, and electrochemistry. She currently works in publishing; she served as Section Chair of the Philadelphia Section of STLE. She received the Clarence E. Earle Memorial Award (2018) and the Golden Grease Gun Award (2022) from NLGI.

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 2015. 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 Golden Grease gun award, the Clarence Earle Memorial award, and the J. Bellanti Sr. memorial award from NLGI. He is an elected fellow by his peers at NLGI, IChemE, STLE, INSTMC, AIC, IOP, CMI, the Energy Institute and the Royal Society of Chemistry. He has over 525 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://bit.ly/3QvfaLX

www.nlgi.org/nlgi-sustainability-page/

NLGI SUSTAINABILITY

Crystal O'Halloran, NLGI Executive Director attends second annual ELGISTC meeting in Amsterdam, Netherlands. The meeting was held on October 24, 2023 and included updates from all five taskforces including:

- **Regulation & Communication Task Force**
- **Co2, Carbon Footprint Task Force**
- **Life Cycle Analysis Task Force**
- **End User Task Force**
- **End of Life Task Force**

Please contact carol@elgi.demon.nl if interested in joining the ELGISTC.

The NLGI Sustainability webpage is now live.

VISIT IT HERE.

NLGI and the Sustainability Committee is excited to announce the launch of the NLGI Sustainability website. This website is a resource for members to access sustainability related information such as the Sustainability Series, Sustainability Committee Updates, Sustainability Technical Articles, along with other resources and presentations. The Sustainability Committee will continue to update this website with additional information to support members sustainability efforts. If you are interested in exploring a sustainability related topic, please reach out to NLGI@nlgi.org.

SUSTAINABILITY COMMITTEE

Interested in Joining? **Contact** NLGI@nlgi.org.

Current Initiatives:

1. Three task forces were created, including:
   - Identifying and measuring footprint, handprint, and life-cycle analysis
   - Getting regular updates from experts in the field of sustainability
   - Environmental, Health, and Safety implications in the grease industry
2. Working with API on Lubricants Life Cycle Assessment and Carbon Footprinting- Methodology and Best Practice
3. Working with other industry organizations on universal, global initiatives

SUSTAINABILITY DEFINITION:

"Meeting the needs of the present without compromising the ability of future generations to meet their own needs."

COMMITTEE MEMBERS

Amber Dzikowicz | Joshua Sheffield | Ravi Menton | Casey Budd | Martin Birze | Dwaine (Greg) Morris
Jim Hunt | Joe Kaperick | Chad Chichester | Cassie Fhaner | Staff liaison: Crystal O'Halloran

NLGI GREASE WORLD

NLGI Grease World E-Newsletter Update

The Q4 *NLGI Grease World E-Newsletter* is accessible to everyone, offering a wealth of valuable information and updates. Sign up online to make sure you get the latest issues in your inbox as soon as it releases.

See the latest articles here.

2023 Q4 ARTICLES

- Where did my Grease Go? Why Water Resistance Can Be Very Important
- Strategic Priority Update: Year-End Recap
- New Member Profiles Q4 2023
- People and Places Q4 2023
- NLGI Grease World E-Newsletter Survey

The *NLGI Grease World E-Newsletter* aims to cover current events in and around our industry, NLGI strategic priorities updates, and highlight new members who have joined the organization.

22nd CLGI GREASE CONFERENCE

October 9-13, 2023 | TaiYuan, China

Dr. Anoop Kumar, NLGI President attended the CLGI meeting in TaiYuan, China October 10-14, 2023.

SCHEDULE INCLUDED:

October 10, 2023	October 11-13, 2023	October 14, 2023
VIP Dinner	Technical Papers	Tours to Historical Locations

300 participants

55 technical papers

CHINA INDUSTRY FACTS:

Over 200 grease manufacturing plants are in China.

China grease market is 38% of the global market.

NLGI leadership will continue to evaluate ways to enhance NLGI & CLGI partnership.

Women in Grease
INTEREST NETWORK

Connecting, Sustaining and Educating
Industry Experts Around the Globe

WIGIN's First Leadership Series Webinar Event

November 7, 2023 | 109 attendees

"From Parts Writer to President"

Keynote Speaker: Jenny Bush, President, Cummins Inc.

"Thank you for hosting this phenomenal Webinar! Jenny Bush's story is so inspirational! So many applicable wisdom nuggets as a leader, mentee, and peer."

"Trailblazer!!"

"This is definitely so inspirational; I am thankful for the opportunity to be here. I needed this uplift!"

"I had to reach out to you, and please pass this along to Lindsey and anyone else who coordinated, THANK YOU! Jenny was fantastic to hear from and the Q&A was amazing. What a journey and what an empowering moment. Going back into my day refreshed and renewed! Thank you, again, and can't wait to attend the next one!"

Access the recorded webinar HERE.

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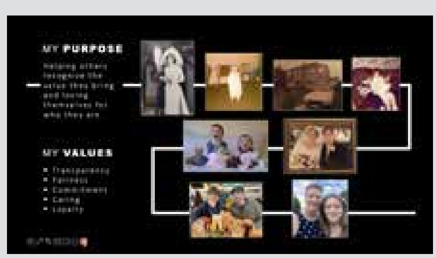


To learn more about the Women in Grease Interest Network visit the NLGI website

High-Performance Multiuse (HPM) Grease Column

LICENSED PRODUCTS

Registered Branded Product	Supplier	CORE	Corrosion Resistance	Water Resistance	High Load	Low Temperature
Acinol 152 HQS (US) – All Colors	Axel Americas	CORE+		+WR		+LT
Axellence 652 HQ (US) – All Colors	Axel Americas	CORE+				+LT
Castrol Molub-Alloy 860/460-1 ES	BP Lubricants USA, Inc.	CORE				
Castrol Tribol™ GR SW 460-1	BP Lubricants USA, Inc.	CORE+	+CR		+HL	+LT
DEWALT Bio Synthetic Lithium Complex #2 Grease	Dynamic Green Products, Inc.	CORE+			+HL	
DGP Bio Synthetic Lithium Complex #2 Grease	Dynamic Green Products, Inc.	CORE+			+HL	
Gadus® S3 V220C 2	Shell	CORE+			+HL	
GLEITMO 680 XT	FUCHS Lubricants Co.	CORE				
JMX-007	Calgary L&G Inc.	CORE+				+LT
LML Lithium Complex Grease	Loadmaster Lubricants, LLC	CORE+		+WR		
MAC TOOLS Bio Synthetic Lithium Complex #2 Grease	Dynamic Green Products, Inc.	CORE+			+HL	
Mobilgrease XHP™ 222	ExxonMobil Oil Corporation	CORE+		+WR		
MOLYKOTE® Multilub Synthetic High Performance Grease	Molykote Specialty Lubricants	CORE+				+LT
Peerless™ LLG	Petro-Canada Lubricants, an HF Sinclair Brand	CORE				
Peerless™ OG2 Red	Petro-Canada Lubricants, an HF Sinclair Brand	CORE				
RENOLIT CXS BGR	FUCHS Lubricants Co.	CORE+			+HL	+LT
RENOLIT CXS CRM 1	FUCHS Lubricants Co.	CORE				
RENOLIT LX 2	FUCHS Lubricants Co.	CORE				
STABYL LX 460 SYN	FUCHS Lubricants Co.	CORE+			+HL	
Valvoline™ Cerulean Plus #2	Valvoline Global Operations	CORE+		+WR	+HL	
Valvoline™ Extreme Red	Valvoline Global Operations	CORE				
XPG 15	49 North Lubricants	CORE+				+LT

*as of November 21, 2023

2023 HPM Survey

Thank you to those who participated in our recent HPM survey. We are excited about the progression of the HPM certification and look forward to new registrations in 2025.

86% of respondents are **currently working on or planning to work** on a HPM product

58% of respondents **currently manufacture/market** 1-5 greases that can potentially meet the HPM specification

50% of respondents **plan to submit 1-2 HPM products** over the next 12 months

74% of those with a current HPM product **rate the ROI as valuable**

As a reminder, NLGI members may access a webinar repository of videos of each HPM specification test. To view these videos, log into the Members' Only section of the NLGI website. For questions about the HPM specification and how to register, please visit: https://www.centerforqa.com/hpm-grease/

To register for an HPM product, please click here and follow the steps below:

FIVE STEP CERTIFICATION PROCESS
for Grease Manufacturers and Marketers

1. Submit Application for Sample Approval & Branded Product Registration
   - Manufacturers include product data
   - Rebrands submit Supplier Affidavit
2. Submit Qualification Sample
3. Submit the signed License Agreement
4. Submit Payment
5. Submit additional Branded Grease Product names

Serving the grease industry since 1933
NLGI

NLGI Year End Recap 2023

2023 NEW MEMBERS

Consumer:
Cummins-Meritor

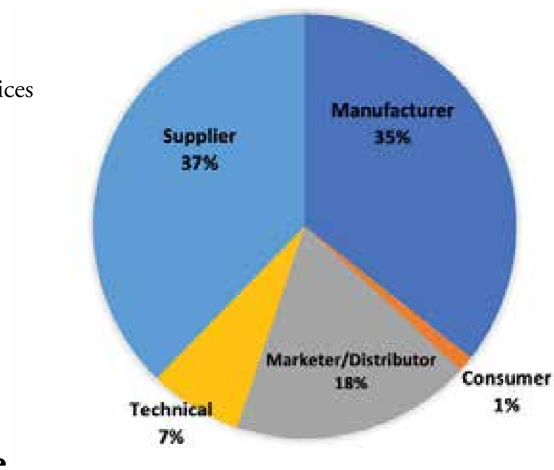
Manufacturer:
KAJO GmbH
MOL-LUB Ltd.
SI Group
Sinopec Lubricant Co., Ltd.
Syn-Tech Ltd.
M/S Manak Petro-Chem

Marketer/Distributor: 49
North Lubricants Marco
Peruana S.A.
Van Horn Metz & Co Inc.

Supplier:
CDA – USA
CDF Corporation
Compass-Instruments Inc.
Four R Marketing
INVISTA
Lion Elastomers
Omni Specialty Products
Polyethylene Containers Inc.
Ravago Chemicals North America
Xinxiang Richful Lube Additive Co. Ltd.
Yasho Industries Limited

Technical:
H F Webster
Engineering Services

94% member retention rate



RESEARCH GRANT

Auburn University for their research proposal titled
'Electrically Conductive Nanoparticle Additives for Greases Used in Electric Vehicles and Other Applications'
The grant will take place over a one year period with expected completion in July, 2024.

2023 DEVELOPMENTS

The year for the Women in Grease Interest Network commenced with a reception held during the NLGI 90th Annual Meeting. This autumn WIGIN had a remarkable turnout of 109 members for the inaugural event of the WIGIN Leadership Series, a webinar featuring the esteemed keynote speaker Jenny Bush, who serves as the President of Power Systems at Cummins Inc.

Jenny Bush's journey is inspiring, from being the first female graduate in Shell Oil's apprenticeship program to her beginnings as a Parts Writer at Cummins to the President of Power Systems with Cummins Inc., making significant strides in the grease industry and establishing herself as a true trailblazer.
Learn More About WIGIN.

Women in Grease
INTEREST NETWORK
Connecting, Sustaining and Educating Industry Experts Around the Globe

WIGIN's First Leadership Series Webinar Event
November 7, 2023
109 attendees
"From Parts Writer to President"
Keynote Speaker: Jenny Bush, President, Cummins Inc.

FINANCIAL

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NLGI remains financially healthy with a robust investment account and operating budget, overseen by the Finance Committee and Board of Directors.

2023 BOARD MEMBERS

President
Anoop Kumar
Chevron Products Company, a division of Chevron U.S.A. Inc.

Vice President
Wayne Mackwood
LANXESS Corporation

Secretary
Tom Schroeder
AXEL Americas, LLC

Treasurer
Chad Chichester
Molykote by DuPont

Immediate Past President
Jim Hunt
Tiarco Chemical

Technical Committee Co-Chair
David Turner
CITGO Petroleum Corporation

David Cardy
Italmatch Chemicals

Chuck Coe
Grease Technology Solutions LLC

Gareth Fish
The Lubrizol Corporation

Muibat Gbadamosi
Calumet Branded Products, LLC

Maureen Hunter
King Industries, Inc.

Tyler Jark
AOCUSA

Joe Kaperick
Afton Chemical Corporation

Matthew Mcginnis
Daubert Chemical Company

Dwaine G. Morris
Shell Global Solutions (US) Inc.

John Sander
Lubrication Engineers, Inc.

Simona Shafto
Koehler Instrument Company, Inc.

Joshua Sheffield
Livent Corporation

Ruiming "Ray" Zhang
Vanderbilt Chemicals, LLC

2023 ANNUAL MEETING

The NLGI 90th Annual Meeting was held at the Hotel Del Coronado in San Diego, CA, USA. | June 4-7, 2023.

NLGI Celebrates 90 YEARS

Total Attendees: **496**

Basic Course Participants: **41**

Advanced Course Participants: **30**

CLGS Exam Participants **6**

Number of Technical Presentations: **31**

Golf Tournament Participants: **136**

Fun Run Participants: **85**

- Dual track of technical presentations
- Industry Speakers Andy Waynick and Bill Tuszynski co-presented a detailed overview of the industry trends that have occurred since 1933, while in contrast, Dr. Erik Willett focused on emerging and future trends in the industry, providing a glimpse into what the future holds.
- Various Networking Opportunities
- General Session
- Awards Ceremony
- Kickoff WIGIN reception
- Partnered with Wounded Warriors for a golf tournament at the renowned Torrey Pines golf course

CERTIFICATION MARKS

22 HPM certifications held by 13 companies
HPM Core: 7
HPM + Water Resistance (WR): 2
HPM + High Load (HL): 5
HPM + Low Temperature (LT): 4
HPM +WR +LT: 1
HPM +HL +LT: 1
HPM +WR +HL: 1
HPM +Corrosion Resistant (CR) + HL +LT: 1

Total Certification Marks for 2023
270 GC, LB, GC-LB certifications held by 78 companies
GC-LB: 229
GC: 12
LB: 29

NLGI High-Performance Multiuse Grease HPM SAMPLE

NLGI COMMITTEES

Education

The education committee focuses on the overall education strategy for NLGI education including education courses and the certified lubricating grease specialist certification.
**Consists of two sub-groups*

✓ **Courses** – Courses Focuses on fine-tuning the Basic & Advanced Lubricating Grease Courses

✓ **CLGS** – Must be CLGS certified to participate on this committee. Focuses on the test given at the Annual Meeting

Editorial

The Editorial Committee collaborates on content circulated to NLGI members and non-members including the NLGI Spokesman, Annual Production Survey and Ask the Expert Q&A. **Consists of two sub-groups*

✓ **Spokesman Sub-Group** – Improve content and readership for Spokesman

✓ **Editorial Review Sub-Group** – Review papers that are ultimately included in the Spokesman as articles for readers. Work with authors on changes

Technical

The technical committee focuses on the technical aspects within the industry and organization. The technical committee incorporates NLGI's certification marks, working groups, reference grease and the annual grease production survey.
**Consists of four sub-groups*

✓ **Certification Marks** – HPM, GC-LB, GC & LB certification marks

✓ **Working Groups** – Bio-Based, Food Grade, Grease Specification and Grease Particle working groups

✓ **Reference Grease** –Oversee current reference grease, including round robin and certificate of analysis

✓ **Grease Production Survey** – Annual survey provided complimentary to NLGI members containing an array of industry information, including global grease production

Membership

The Membership Committee focuses on membership growth by recruiting new member companies including international expansion. Additional focuses of the Membership Committee include member benefits, membership value and retaining current member companies.

Annual Meeting

The Annual Meeting Committee serves as the advisory group for the Annual Meeting including selecting speakers, award recipients, solidifying technical sessions and direction on the site selection process. **Consists of four sub-groups*

✓ **Speakers Sub-Group** – Help select keynote and industry speakers Site Selection Sub-Group Help determine future locations/hotels for upcoming meetings

✓ **Awards Sub-Group** – Determine award winners

✓ **Technical Sessions Sub-Group** – Oversee technical presentations presented at Annual Meeting, collecting papers that will be used in Spokesman issues as articles

✓ **Site Selection** – Help determine future locations/hotels for upcoming meetings

Academic

The Academic Committee seeks to strengthen the grease industry by fostering relationships with universities containing tribology programs as well as evolving the organization's research grant program. **Consists of two sub-groups*

✓ **Outreach Sub-Group** – Foster partnerships with Universities and professors regarding NLGI membership and attending Annual Meeting

✓ **Research Grants Sub-Group** – Help determine deserving University for annual NLGI research grant

If interested in serving on a committee/sub-group, complete the volunteer form on the NLGI website.

NLGI 816.524.2500 | fax: 816.524.2504 | nlgi@nlgi.org


Education and Networking Opportunities
Sustainability
Industry Trends and Challenges
NLGI 2022-2024 STRATEGIC PRIORITIES
Effective Governance and Leadership
Membership Value, Engagement and Growth
Global Outreach